have been in use for centuries as ceramic pottery. The Chinese, as well as Mediterranean cultures, have been making enamelware for jewelry and objets d'art since about 2000 B.C. In our modern society both the glass and silicate industries are becoming more and more closely involved with the metals industries with the development of composite materials and have long been associated with refractory ceramics, an off-shoot industry of the basic silicates and still a part of that industrial complex. The jewelry trade uses natural gem stones, such as amethyst, as well as amorphous glass as beads or cut stones, etc. It is interesting to note that the Egyptians were making fake glass "paste" gem stones as early as 3000 B.C.

Technical Application: Pure silicon dioxide, SiO_2 was initially grown on silicon wafers as a (1) "wet" thermal oxide using nitrogen or argon gas bubbled through DI water into a furnace at 900—1300°C, or (2) "dry" thermal oxide using O_2 gas only, under similar temperature conditions. In both cases, the silicon wafer was the source of silicon. Both methods are still used and a common temperature is 1000°C. For such "oxide" deposition on silicon and other metallic compounds that do not contain silicon, the now called Chemical Vapor Deposition (CVD) process was developed from the original pyrolytic process which used liquid chemical compounds. CVD uses gas combinations, such as SiH_4/O_2, with deposition temperatures ranging from 350 to 900°C. The AMT Sylox systems are similar with 350 to 500°C temperatures. RF (magnetron) sputter systems using a solid glass or pressed powder target as the silica source are now in wide use with deposit temperatures as low as 200°C.

Finely powdered silica, as a glass-frit, can be painted or sprayed on surfaces and then fired to form the oxide coating between 600—800°C. Silicon monoxide, SiO, can be vacuum evaporated, 5×10^{-5} Torr vacuum or better, then densified by bake-out in oxygen or air at 400 to 800°C.

A thin piece of glass, such as a glass coverslide, can be fired and fused onto a semiconductor surface between 500 to 800°C as a method of cladding and has been used to fill channels, via-holes, and pits in surfaces.

Boiling water, nitric acid, or hydrogen peroxide, depending upon the semiconductor material, has been used to form a hydrated surface oxide that can then be stripped with HF as a method of surface cleaning. Potassium hydroxide, KOH, both as a boiling solution or electrolytic solution, has been used to grow a hydrated silica on silicon wafers in the study of oxidation reactions and as a method of surface cleaning and can be used to form similar oxides on germanium, Ge, and compound semiconductors, such as gallium arsenide, GaAs, or indium phosphide, InP. These are all porous, hydrated oxides and require special postbake treatments if they are going to be used for other than surface cleaning applications.

The hard silica coatings . . . evaporated, sputtered, CVD . . . are used for surface passivation; mechanical and chemical protection; as a mask against diffusion, metallization, ionization, etching (wet chemical of dry — gas), and as doped oxides PSG, BSG, ASG, etc., as either surface protection or as an element diffusion source.

As a mechanical operator, glass has been deposited in channels, "V" slots or pits, both for strength as well as for dielectric isolation of diffused areas or separation of epitaxy layers and similar structures. In other cases, as in fabricating MOS and MNOS devices, the oxide thin film is an active element in the device operation. In most of these applications the thin film is amorphous and used as such, though it may be partially crystallized by special processing, even to single crystal form by substrate orientation or by laser annealing.

Silica also is being deposited as a colloidal thin film, such as hydrogenated a-SiO_2:H, or a-$SiON_x$:H compound for special purpose application.

As glass plates — microscope slide, soda-lime, pure quartz— the glass is used as substrates for thin film evaporation of metals or the epitaxy growth of materials which, after deposition, are removed with an HF float-off technique from the glass for material morphological study as with a transmission electron microscope (TEM). Such glass slides also are used in both vacuum metal evaporation and epitaxy growth as thickness monitors, or to

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cover portions of a wafer during metal evaporation, epitaxy, etc., for measurement purposes. The microscope slide/metal evaporation is also used in transmitted light evaluation for pinhole density in the metal oxide thin films and in studying the growth step mechanisms of thin films as deposited by evaporation, epitaxy etc.

In addition to the direct applications in semiconductor device fabrication, quartz single crystal, fused quartz, or fused silica blanks are used to fabricate circuit substrates, and some thin film oxides on such substrates used in making resistor, capacitor or other dielectric elements as planar devices, to include integrated circuits (ICs). This also may include doped glassy electrical contact pads or fingers.

Semiconductor packages that require external lead contacts use glass-isolated feed-thru or stand-off pins as well as isolation plates or coatings and many test holders include such pins and plate isolation parts. The "glass diode" package is a sleeve or tube of glass into which the device is fused or the lead fabricated device may be bead-fused or oxide/nitride sputter coated prior to epoxy encapsulation.

Both quartz and silica have long been used for the fabrication of a wide variety of lenses — microscopes and telescopes — as well as light frequency modulators, resonators, filters, and similar devices. The "quartz wedge" is standard to most metallurgical microscopes for material evaluation — a tapered and oriented single crystal piece of quartz that produces frequency color steps, or, as pair lenses, for polarized light by rotation of the two lenses with the thinned specimen between, and as surface polarization for direct illumination. In many applications, such as in telescope fabrication, the lens is coated with aluminum and then overcoated with SiO_2 (evaporation as SiO with densification). In vacuum systems, glass microscope slides or plates are coated with either aluminum, gold, or nickel — or other reflective metals and used as sighting mirrors to observe metal evaporation in progress; similar mirrors are used in observing a cobalt-60 radiation source indirectly as direct observation is hazardous.

Needless to say, quartzware and glassware are construction materials in much laboratory equipment — diffusion furnaces, epitaxy systems, vacuum systems, work stations — in addition to the beakers, measuring cylinders, stirring rods, and so forth used in chemical processing. This includes glass-wool as a packing material in furnaces, as fiber sheeting for electrical and temperature isolation, goggles — deep red or dark blue — to observe welding and high temperature material processing.

Single crystal alpha-quartz has been in use since the early 1920s for the fabrication of radiofrequency crystals, taking over from the original point contact galena, PbS, crystals. The best natural stone comes from Brazil, but artificial quartz is coming into wider use as it is of higher purity or more defect-free, e.g., no twinning. By oriented cutting of blanks, mostly, off z-axis, zones of frequency are obtained; AT, BT, DT, X cut and others. The AT-cut produces the highest frequencies as, by polish thinning, these circular blanks (about $^3/_8$" diameter) can reach as high as 5th order 100 MHz frequency. Alpha-quartz, under electric load, produces a physical motion and, with proper cut orientation, the motion is translated into a controlled frequency. When combined with semiconductor and electronic circuitry modulators, resonators and many other similar device applications are possible.

Other products from glass include glass lasers and fiber optics. The latter not only used for light transmission but for telephone transmission. Light fibers are widely used in conjunction with microscope study and have major application in medicine where the fiber can be inserted into the body for inspection without major surgery. TV and fiber optics are now common in all surgical procedures.

As already mentioned, silica powder is still widely used as a lapping and polishing abrasive. The material can be natural single crystal/artificial and may have specific compound name which are associated with the mixture, physical structure or fracture characteristics. As a general name it is called White Rouge or Glassite; as an amorphous mixture containing less than 0.5% quartz — a natural mineral — it is called white "coloring compound" or

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"Tripoli"; the natural colloid is opal, and the artificial is colloidal silica. There also is sand, as loose grains or sandstone, and pumice or tufa as solid sanding compounds, and natural flint is still used as an abrasive and so named. In the artificial growth of single crystal alpha-quartz, natural quartz chips are used in a slurry of KOH/water rather than sand, in a temperature gradient autoclave capable of 40,000 psi — the ingot grown from a single crystal seed held above the silica vapor bottom melt pot. High purity sand, on the other hand, is the chief source of semiconductor silicon.

Both natural single crystal quartz in its various forms as well as artificial quartz have been the subject of general morphological study and thin film silicas are under ongoing studies in the semiconductor and Solid State fields in addition to the continual development work in the glass and other silicate industries.

The following section includes both silica used as a Solid State thin film coating, as well as some of the glasses in present use.

Etching: HF and other fluorine containing compounds; hot H_3PO_4. Alkalies and dry chemical etching (DCE).

SILICON DIOXIDE ETCHANTS

SIO-0001a
ETCH NAME: Sodium carbonate
TYPE: Acid, removal
COMPOSITION:
x .... *N*/50 $NaCO_3$
DISCUSSION:
Vycor 7913 glass. This is a slow etch on specific types of glass.
REF: Brochure: OVB-4 11/i/64 — Corning Glass Works
TIME: 9 h
TEMP: 100°C
RATE: 70 mil/6 h

SIO-0001b
ETCH NAME: Sodium hydroxide
TYPE: Alkali, removal
COMPOSITION:
x .... 5% NaOH
DISCUSSION:
Vycor 7913 glass. This is a slow etch on specific types of glass.
REF: Ibid.
TIME: 6 h
TEMP: 100°C
RATE: 900 mils/6 h

SIO-0001c
ETCH NAME: Hydrochloric acid
TYPE: Acid, removal
COMPOSITION:
x .... 5% HCl
DISCUSSION:
Vycor 7913 Glass. This is a slow etch on specific types of glass.
REF: Ibid.
TIME: 24 h
TEMP: 95°C
RATE: $^1/_2$ mil/24 h

SIO-0002
ETCH NAME: Potassium hydroxide
TYPE: Alkali, removal
COMPOSITION:
x .... 2 *M* KOH
TIME:
TEMP: RT (21°C +/− 5°C)
RATE: 0.05 Å/min

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DISCUSSION:

Pyrex glass beaker. Authors say that hydroxides used at room temperature etch so slowly the reaction products introduced into the solution will not affect the etching of silicon. (*Note:* These authors have used boiling solutions of KOH and NaOH with a variety of chemical additives in Pyrex beakers. One beaker pinholed after daily use for a period of about 6 months.)

REF: Bergman, I & Paterson, M S — *J Appl Chem*, 11,369(1961)

SIO-0003

ETCH NAME: Hydrofluoric acid — TIME:

TYPE: Acid, removal — TEMP:

COMPOSITION:

(1) x .... HF, conc. (2) 1 .... HF
20 .... H_2O

DISCUSSION:

SiO, thin film. Deposits evaporated on NaCl (100) substrates and used in a study of etch rates of SiO. Concentrated HF removal is rapid. The dilute solution shown was slow removal. SiO deposits were 8 nm thick and were removed from NaCl by the float-off technique using water, and called "wet" stripping.

REF: Kaito, C & Shimizu, T — *Jpn J Appl Phys*, 23,L7(1984)

SIO-0004: Menth, M — personal communication, 1979

SiO thin films evaporated over aluminum thin films on telescope lenses of various sizes. Stabilized as SiO_2 by heat treatment densification. SiO stripped with HF as required for lens re-work.

SIO-0038: Fahr, F — personal communication, 1979

SiO, thin films evaporated from tungsten boats with cover containing an exit hole to control evaporation. Used as a thin film surface coating on silicon devices. Densification to SiO_2 in an air oven at 125°C with variable time. HF used to strip or in thickness studies and density evaluation.

SIO-0004

ETCH NAME: Hydrofluoric acid — TIME:

TYPE: Acid, removal — TEMP: RT

COMPOSITION:

x .... HF, conc.

DISCUSSION:

SiO_2, thin film deposits on silicon wafers, (111), (100), and other orientations, both p- and n-type with various doping concentrations. For general, rapid removal. Etch rate varies with method of SiO_2 deposition, annealing times and temperatures, type doping and doping concentrations. Also deposited on germanium, compound semiconductors and metal surfaces as a surface protectant, as a surface mask against diffusion and metallization or as an active device element. Many clean/etch sequences include concentrated HF as the final step in sequences as an oxide remover prior to further processing. It also acts as a polish liquid on glasses.

REF: N/A

SIO-0005a: Walker, P — personal application, 1956—1959

SiO_2 thin films deposited on silicon (111) and (100) n-type, 5 to 150 Ω cm resistivity, and with or without phosphorus and boron doping. A study of both wet and dry thermal oxidation deposited on wafers at various temperatures and thicknesses in a general oxidation evaluation. Concentrated HF used to: (1) remove deposits for oxide defects induced on silicon surfaces; (2) as a pinhole etch through oxide for depth measurement; (3) as a step-etch for depth measurements, and redeposition to step-etch and observe possible SiO_2/SiO_2

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interface reactions. Establishing optimum thickness of oxide on n-type diode wafers using wet thermal oxidation for use as a mask against boron, phosphorus and gold diffusion. Concentrated HF was used as the base line solution in developing compatible solutions for photo resist processing with DC sputtered SiO_2, Si_3N_4, and $Si_2N_xO_{x-1}$. Also in developing n-type silicon mesa diodes with Si_3N_4/SiO_2 coatings to prevent degradation of exposed boron diffused p-n junctions. And in an etch rate study of CVD (Silox) vs. RF magnetron sputter (MRC system) of deposited oxides on both (111) silicon and (100) gallium arsenide with and without epitaxy layers.

SIO-0007b: Ibid.

SiO_2, as glass microscope slides used as substrates for thin film metal evaporation. HF used for float-off technique to remove metal thin films for TEM study.

SIO-0065: Tomozawa, M & Takamori, T — *J Am Ceram Soc*, 60,301(1977)

SiO_2, as borosilicate glasses of different compositions. A study of the effects of HF etching rates on phase separation. Anneal time and temperature used to establish phase separation.

SI-0057a: Atalla, N M et al — *Technol J Bell Syst*, 38,2(1959)

SiO_2, thin films grown on silicon wafers by thermal oxidation. A major study of oxide stability on silicon. Oxide deposited at 1000°C as both wet and dry oxidation. Wafers were FZ (n-type) and CZ (p-type) as acceptor and donor wafers, respectively. Washed in HF to produce a hydrophobic silicon surface prior to oxidation. The following gas and etch evaluations were studied: (1) Cl_2 at 900°C will etch silicon at 0.001/min, but does not etch oxide. Oxides were deposited at 920°C, 1—30 min for oxides 250—300 Å thick and quenched to air at RT. (2) Hydrophobic wafer surface: etch is fresh HF:HNO_3 solutions; in boiling H_2O; organic solvents; or water with detergents. (3) Hydrophilic wafer surface: soak in hot HNO_3, 10 min, follow with boiling H_2O, and surface remains hydrophilic. If HF vapors are present it becomes highly hydrophobic, and if exposed to air also becomes hydrophobic but nonuniform. (4) Deposited oxide surfaces are hydrophilic. After HNO_3 soak and exposure to air goes from hydrophobic to hydrophilic by washing in organic solvents.

SIO-0008
ETCH NAME: Hydrofluoric acid, dilute
TYPE: Acid, removal
COMPOSITION:
1 .... HF
1 .... H_2O
TIME:
TEMP: RT
DISCUSSION:

SiO_2, thin films and native oxides. A widely used mixture of HF for native oxide removal, it is usually the last step in a clean/etch sequence followed by DI water rinse immediately before placing wafers/substrates in a vacuum system, diffusion tube or epitaxy system. Used on silicon and other compound semiconductors as the final native oxide remover.

REF: N/A

SIO-0009
ETCH NAME: BHF
TYPE: Acid, removal
COMPOSITION:
1 .... HF
100 .... NH_4F, sat. sol.
TIME:
TEMP:
RATE: 0.14 nm/sec
DISCUSSION:

SiO_2 and Si_3N_4 thin films deposited on silicon, (100), p-type, 5—9 Ω cm resistivity substrates used in an etch study. Substrates were pre-cleaned in H_2SO_4:H_2O_2, followed by an HF dip and DI water rinse. SiO_2 deposited at 950°C and annealed in N_2 for 30 min.

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Si_3N_4 was then grown at 925°C with NH_3 for 2 h. Authors say that etch rate increases with nitride deposition temperature. Solution shown was used to establish etch rates.
REF: Wong, S S et al — *J Appl Phys*, 30,1139(1983)
SIO-0010: Walker, P — personal application, 1969

SiO_2, as single crystal quartz as AT and BT cut blanks were either frequency tuned in NH_4F:HF solutions at RT containing undissolved flakes of NH_4F in etch containers to improve repetitive frequency etching time control with long time solution use. The addition of glycerin to the solutions for improved surface wetting was also used to improve etch planarity results.

SI-0011a
ETCH NAME: BHF
TYPE: Acid, removal
TIME:
TEMP: RT
COMPOSITION:
1 .... HF
6 .... NH_4F (40%)
DISCUSSION:

SiO_2, deposited as CVD thin films on (100) silicon substrates with boron and phosphorus doping, as BSG or PSG, respectively, and used in a study of etch rates as deposited or after densification at 700°C, 30 min. All coatings show tensile stress, as deposited, with BSG highest — decreases with shelf-time and converts to compressive stress under high humidity conditions. Refractive index, n, changes after 700°C anneal. Show variation in etch rates between as deposited and after annealed. The following etch solutions will leach deposits: (1) H_2SO_4:H_2O_2:H_2O; (2) NH_4OH:H_2O_2:H_2O and (3) H_2O, only. These three solutions are of interest as they are used in clean/etch sequences on a number of semiconductor materials. Rates with the solution shown were compared to those of the P-etch (SI-0011b). The P-etch appears to be more sensitive to oxide deposit composition than the BHF solution shown.
REF: Kern, W & Schnable, G L — *RCA Rev*, 43,423(1982)

SIO-0011b
ETCH NAME: P-etch
TYPE: Acid, removal
TIME:
TEMP:
COMPOSITION:
3 .... HF
2 .... HNO_3
60 .... H_2O
DISCUSSION:

SiO_2, thin films deposited on (100) silicon wafers. This solution developed by AMT Corp. in conjunction with their SILOX deposition system is a widely used standard for study of pinhole density in oxide thin films.
REF: Ibid.
SIO-0087: Maissel, L L & Glang, R — *Handbook of Thin Film Technology*, McGraw-Hill, New York, 1970.

SiO_2, thin films. Described as a controlled etch for silica.
SI-0012: Balazs, M K & Swanson, T B — AMT SM-102R Brochure, June 1970.

SiO_2, thin films grown by the AMT-2600 SILOX system using $SiH_4/O_2/N_2$ gas source. P-etch was developed for use in establishing etch rates and pinhole density in silica films. Refractive index n = 1.44 for a gas mixture of 2:18 (O_2:SiH_4) with temperature between 350 and 475°C. (*Note:* Opal, n = 2.1—2.3 and quartz, n = 1.54—1.55.)
SI-0061: Walker, P & Gomez, A — personal application, 1978

SiO_2, thin films deposited on (100) GaAs:Cr (SI) wafers used in the fabrication of LN-FETs. Oxide was deposited from a SILOX system at 450°C for a royal blue color (about 2000 Å) for use as a metallization mask for Au/AuGe/Ni evaporation. After photo resist

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patterning, the P-etch was used to remove SiO_2 in the open pattern areas prior to metal evaporation. After metallization and device forming, the wafers were given a passivation oxide coating at 350°C.

SIO-0020
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
15 ml .... HF
10 ml .... HNO_3
300 ml .... H_2O
TIME:
TEMP: RT
RATE: 2.5 Å/sec
DISCUSSION:

SiO_2, thin films deposited on silicon wafers at 1000°C by wet oxidation. This form of a P-etch was used in a properties study of the oxide.
REF: Plisken, W A & Lehman, H S — *J Electrochem Soc*, 113,872(1964)
SIO-0086: Plisken, W A & Lehman, H S — *IBM J Res Dev*, 8,43(1966)

SiO_2, thin films deposited on silicon wafers used in a study of the oxide properties.

SIO-0013
ETCH NAME: BHF
TYPE: Acid, removal
COMPOSITION:
100 ml .... HF
860 ml .... NH_4F (40%)
TIME: 5 sec
TEMP: 30°C
DISCUSSION:

Si, (100), p-type 10 Ω cm resistivity wafers used as substrates for RF magnetron sputter of SiO_2 thin films in argon or Ar/H_2. Deposit thicknesses were 0.1 to 2.5 μm. The solution shown was used in studying oxide etch rates which vary with deposit temperature and gas pressure used.
REF: Serikawa, T & Yachi, T — *J Electrochem Soc*, 131,2105(1984)

SIO-0058
ETCH NAME: BSG
TYPE: Silica, diffusion source
COMPOSITION:
"A" x .... *K_2SiO_3
"B" 10 g .... $K_2B_4O_7.5H_2O$
3.5 g .... KOH
15 g .... H_2O
TIME:
TEMP:

*As a 1.25 specific gravity solution

Mix: 1 "A":1 "B" when ready to use.
DISCUSSION:

SiO_2, as a BSG glassy layer on silicon used as a diffusion source for boron. Coat the mixture shown on silicon surfaces with a camel hair brush, dry under a heat lamp, then furnace drive-in for boron diffusion. Remove BSG remaining after drive-in with HF soak.
REF: Cline, J E & Seed, R G — *J Electrochem Soc*, 105,700(1958)

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SIO-0006
ETCH NAME: Hydrofluoric acid, vapor
TIME:
TYPE: Acid, removal
TEMP: Hot
COMPOSITION:
x .... HF, vapor
DISCUSSION:

SiO_2, as thermal oxidation on silicon wafers in fabrication of Al/SiO_2/Si capacitors in a study of high field phenomena (HFP) in silicon dioxide. Si, (100), n-type, phosphorus doped substrates were used with four different thicknesses of thermal oxides. Aluminum was evaporated on the SiO_2 for CV characterization and forced voltage-current-time studies. HF fumes were used to remove the oxide from the back of wafers.
REF: Shirley, C G — *J Electrochem Soc*, 132,488(1985)
SIO-0007c: Ibid.

Glass, as microscope slides. One side of the slides was etched in hot HF vapors to roughen the surfaces for better adhesion of thin film gold evaporated in a study of gold morphology.
SIO-0005b: Ibid.

SiO_2, thin films grown on (111) silicon n-type, 5—20 Ω cm resistivity wafers by wet thermal oxidation at 1000°C for thickness up to about 10,000 Å maximum. Hot HF vapors used to step-etch oxide by color steps in establishing oxide layer color order thicknesses, and in studying stacking faults at the SiO_2/Si interface.

SIO-0060
ETCH NAME: Hydrofluoric acid, dilute
TIME:
TYPE: Acid, removal
TEMP:
COMPOSITION:
1 .... HF
100 .... H_2O
DISCUSSION:

SiO_2, grown on IC devices. Oxide called an intermediate dielectric film. Solution used as a very light oxide surface cleaning etch.
REF: Hess, D W — *J Vac Sci Technol*, A2(2),243(1984)

SIO-0014
ETCH NAME: BHF
TIME: 30 sec
TYPE: Acid, step-etch
TEMP: RT
COMPOSITION:
1 .... HF
10 .... NH_4F
15 .... H_2O
DISCUSSION:

Si, (100) wafers used as substrates for RF sputtered oxide thin films under various argon pressures. The solution shown was used to establish etch rates of the various films.
REF: Yachi, T & Serikawa, T — *J Electrochem Soc*, 131,2720(1984)

SIO-0015
ETCH NAME: BHF
TIME:
TYPE: Acid, removal
TEMP:
COMPOSITION:
(1) x .... 1.2 *M* HF
x .... 10.3 *M* NH_4F
(2) x .... 1 *M* HF

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DISCUSSION:

SiO_2, thin films deposited on (100) silicon wafers. Solutions shown were used in a general etch rate study of deposits.

REF: Nielsen, H & Hackleman, D — *J Electrochem Soc*, 130,708(1983)

SIO-0016

ETCH NAME: TIME:

TYPE: Acid, chlorination TEMP:

COMPOSITION:

(1) x .... HCl, vapor (2) x .... TCA, vapor

DISCUSSION:

SiO_2, thin films deposited on silicon (100), n-type, 8—12 Ω cm and p-type 14—22 Ω cm resistivity wafers used in a study of the differences between using HCl or TCA as additives to the gas stream during growth of the oxides. Wafers were "Piranha" cleaned prior to SiO_2 deposition. **CAUTION:** TCA at elevated temperatures with the presence of phosphorus will form phosgene gas. (*Note:* TCA is 1-1-1 trichloroethane. There is a "Piranha" clean/etch system commercially available . . . it is a system name — not an etch/clean solution.)

REF: Cosway, R & Wu, C-E — *J Electrochem Soc*, 132,151(1985)

SI-0017

ETCH NAME: BOE TIME:

TYPE: Acid, removal TEMP:

COMPOSITION:

1 .... BOE (a 6:1 mixture of ?)

6 .... H_2O

DISCUSSION:

SiO_2, as thin film deposits, with substrates not shown. A study of flow characteristics of doped oxides between APCVD and LPCVD conditions of deposition. After initial deposition "flowing" was done in steam with Cl_2 present during processing BSG films. With >4%/wt concentration of boron in the films, blisters form during annealing. Etch rates were similar to those of Kern & Schnable (SI-0011).

REF: Foster, T et al — *J Electrochem Soc*, 132,505(1985)

SIO-0018

ETCH NAME: Alcohol TIME:

TYPE: Alcohol, pinhole TEMP: RT

COMPOSITION:

(1) x .... alcohol (2) 1 .... HF

19 .... HNO_3

DISCUSSION:

SiO_2, thin films deposited on (100) silicon wafers used in a study of pinhole development on the oxides on the silicon substrates. A Narvonic Leak Detector was used. The wafer is placed under alcohol and a SST wire loop at an electrical potential of 20—80 V is submerged and passed over the oxidized wafer surface. The electrical potential will cause the alcohol to bubble at pinhole and defect points in the SiO_2 film. Etch solution shown is used to enhance the observed defects. It was shown that brush scrubbing or jet washing of wafer surfaces in cleaning prior to oxidation — washing from wafer center toward periphery — causes a high pinhole density that is propagated into both the grown oxides and nitrides that are most prominent at wafer center.

REF: Goodwin, C A & Brossman, S W — *J Electrochem Soc*, 129,1066(1982)

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SIO-0019
ETCH NAME:
TYPE: Acid, oxidation
TIME: 10 min
TEMP: 90°C
COMPOSITION:
3 .... HCl
1 .... H_2O_2
1 .... H_2O
DISCUSSION:
SiO_2, thin films deposited on (111), p-type, 1—3 Ω cm resistivity wafers used as substrates for silicide formation. A hydrated oxide was grown on substrate surfaces in the solution shown. Follow with metal evaporation of Pt:Pd or Pt and Pd, only. Subsequent annealing used SiO_2 deposit as the source of silicon to form silicides.
REF: Kawarada, H — *J Appl Phys*, 57,244(1985)

SIO-0021a
ETCH NAME: EPD
TYPE: Acid, selective
TIME:
TEMP:
COMPOSITION:
20 ml .... ethylenediamine
20 g .... pyrocatechol
44 ml .... H_2O
DISCUSSION:
SiO_2, thin films deposited in etched grooves of (100) silicon wafers. A thin film Al_2O_3 thin film was deposited and used as a mask. After photo resist patterning grooves were etched with ⟨110⟩ orientation prior to SiO_2 RF plasma deposition. Solution shown used to etch pattern the grooves.
REF: Ho, V & Seguano, T — *Solid Thin Films*, 95,315(1982)

SIO-0021b
ETCH NAME:
TYPE: Acid, dislocation
TIME:
TEMP:
COMPOSITION:
200 ml .... HF
9 g .... CrO_3
100 ml .... H_2O
DISCUSSION:
SiO_2, thin films deposited on (100) silicon substrates with RF plasma CVD and by thermal oxidation. Used in a study of stacking faults developed in Si by SiO_2. No stacking faults were observed in plasma CVD deposited surfaces but were present in thermal oxidized surfaces (wet oxidation at 1150°C, 3 h). Refractive index, n = 1.46, for both types of oxide. Dielectric constant: e = 3.5—4.0, for both types of oxide. Solution used to remove SiO_2 and develop stacking faults in silicon substrates.
REF: Ibid.

SIO-0021c
ETCH NAME: Pliskin etch
TYPE: Acid, preferential
TIME:
TEMP: RT
COMPOSITION:
1 .... HF
1 .... 1 *M* Cr_2O_3

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DISCUSSION:

SiO_2, thin films deposited on (100) silicon wafers. CVD SiO_2 was deposited on Al_2O_3 films used as photo resist patterned masks. Solution shown used to pattern grooves in silicon. See SIO-0021a-b.

REF: Ibid.

SIO-0022

ETCH NAME: Sulfuric acid

TYPE: Acid, cleaning

TIME: $^1/_2$ h minimum

TEMP: Hot

COMPOSITION:

x .... H_2SO_4

DISCUSSION:

Glass, soda-lime, blanks used to fabricate photo resist chrome masks for silicon and other compound wafer processing. After lap polishing glass surfaces with a CeO_2 slurry and water washing, blanks are soak/cleaned with sulfuric acid prior to leaching for Na replacement with K in a $KMnO_4$ at 350°C bath for 6 to 8 h. This treatment produces high quality glass blanks.

REF: Tarn, W H — personal communication (Optifilm Co., 1978)

SIO-0023

ETCH NAME: DE-100

TYPE: Ionized gas, selective/removal

TIME: 15—20 min

TEMP: 25°C start

GAS FLOW: 300 cc/min

PRESSURE: 0.14 Torr

POWER: 300 W

COMPOSITION:

x *DE-100

*DE-100 = CF_4:O_2 (8.5%)

DISCUSSION:

SiO_2, thin films deposited by SILOX system method on (100) silicon and GaAs:Cr (SI) wafers in processing microwave devices. Used in a general study of SILOX deposited SiO_2 and oxide removal. Solution also used on germanium, (111) wafers to remove SiO_2 deposits. Prior to DE-100, SiO_2 surfaces can be treated with 300 cc/min O_2 flow, 100 W power and 1.2 Torr pressure for 1—2 min. (*Note:* PDE-100 [CF_4:17.5% O_2] is 20—30% faster than DE-100.)

REF: Scientific Gas Products Company — personal communication, 1979

SIO-0024

ETCH NAME: Dry chemical etching

TYPE: Ionized gas, selective

TIME:

TEMP:

GAS FLOW:

PRESSURE:

POWER:

COMPOSITION:

x .... CHF_3

x .... O_2

DISCUSSION:

SiO_2, thin film deposits. A study of RIE selective etching of SiO_2 thin films for controlled structuring of deposits.

REF: Steinbruchel, C H et al — *J Electrochem Soc*, 132,180(1985)

SIO-0029: Castellano, R N — *Solid State Technol*, May 1984, 203

SiO_2, thin film grown as a cap deposit in an SiO_2/poly-Si/PSG layered structure. All three layers were deposited by LPCVD and used in study of the selectivity etch reactions between the three layers.

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SIO-0025
ETCH NAME: TIME:
TYPE: Oxide, growth TEMP: ~1000°C
COMPOSITION:
x .... SiO_2
DISCUSSION:
SiO_2, thin film coatings of different types deposited on (100) GaAs:Cr and InP:Fe (SI) wafers as substrates for evaluation of the various films for device encapsulation for cap type annealing. Passivating films grown were SiO_2, Si_3N_4, BSG, PSG, BPSG, and ASG on GaAs and Si_3N_4 on InP. On the InP, In out-diffuses into the nitride and Si in-diffuses to the InP, but there is no cross-diffusion with PSG coatings. Also, deposition of AlN on silicon.
REF: Oberstar, J & Streetman, B G — *Thin Solid Films,* 103,17(1983)

SIO-0026
ETCH NAME: Steam TIME:
TYPE: Steam, oxide reflow TEMP: >100°C
COMPOSITION: PRESSURE:
x .... steam
DISCUSSION:
SiO_2, deposited on silicon wafer substrates as doped PSG thin film oxide. PSG with 7—12/g wt phosphorus deposited by CVD at 430°C. Anneal and reflow deposits in steam at 950°C, 15 min. A major study and analysis of reflow reactions of PSG coatings.
REF: Bowling, R A & Larrabee, G B — *J Electrochem Soc,* 132,141(1985)

SIO-0027
ETCH NAME: Oxide, growth TIME:
TYPE: Oxide, growth TEMP: ~1000°C
COMPOSITION:
x .... SiO_2
DISCUSSION:
SiO_2, thin films deposition on (100) silicon wafers at an oblique deposition angle. SiO_2 source was 5° off perpendicular toward wafer surface. Produces a columnar, slanted oxide structure as observed after cleaving the wafer ⟨110⟩. Refractive index, n = 1.47—1.49. (*Note:* This is the only reference to artificial growth of columnar SiO_2. The natural mineral chalcedony grows as an admixture colloidal/amorphous SiO_2 matrix with columnar growth sections. Columnar growth also observed in a number of epitaxy metallic compound thin films.)
REF: Levy, Y — *J Appl Phys,* 57,2600(1985)

SIO-0028
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... HF, conc.
DISCUSSION:
SiO_2, thin films deposited by a special technique. Fine grains of silica deposited from a crucible at 1500°C through a nozzle with the grains ionized by E-beam bombardment just as they reach the deposit substrate surface. The refractive index varies with the degree of ionization with n = 1.46 to 2.01, ion current, Ie, 0—1000 mA. Reaction: $2SiO_2$ to $2SiO + O_2$ and SiO are stable as a gas mixture. SiO_2 as deposited is metastable with n = 1.99.

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Deposited under low pressure O_2 deposit conditions is SiO_x. Increase O_2 pressure, and n = ~2. (*Note:* SiO_2 natural quartz refractive index is n = 1.54—1.55.)
REF: Wong J et al — *J Vac Sci Technol,* B3(1),453(1985)

SIO-0030
ETCH NAME: TIME:
TYPE: Oxide, growth TEMP:
COMPOSITION:
x .... SiO_2
DISCUSSION:
SiO_2, thin films deposited on (100) silicon wafers by LPCVD. Gas flow was directed upward to silicon wafers held in a "cage" in a vacuum bell jar with outside wall heating through water cooled coils. Deposition was by pyrolysis of $SiH_4/O_2/N_2$ at 400°C and 100 mTorr pressure. This was a study of this method of silica deposition.
REF: Learn, A J — *J Electrochem Soc,* 132,390(1985)

SIO-0031
ETCH NAME: TIME:
TYPE: Oxide, growth TEMP: <1000°C
COMPOSITION:
x .... SiO_2
DISCUSSION:
SiO_2, thin films deposited on (HgCd)Te wafers at <100°C. Pyrolysis of SiH_4 + NO_3 gases passed over liquid mercury as they enter the deposition chamber that was heated using a mercury resonance lamp for heating. Used to deposit SiO_2 without out-gassing of Hg or Cd from the substrates.
REF: *Microwaves & RF,* August 1984, 55

SIO-0032
ETCH NAME: TIME:
TYPE: Acid, selective TEMP:
COMPOSITION:
1 .... HF
19 .... HNO_3
14 .... H_2O
DISCUSSION:
SiO, deposition on aluminum and quartz blanks or silicon wafers. Deposition of the monoxide was from molybdenum boats in fabricating MIM devices as Al/SiO/Al/substrate. Various annealing temperatures evaluated. The solution shown was selected as the best for uniform MIM structure etching.
REF: Shabalon, A L & Feldman, M S — *Thin Solid Films,* 110,215(1983)
SIO-0033: Ablov, S H & Feldman, M S — *Thin Solid Films,* 110,225(1983)
SiO, monoxide thin films. Similar work as in SIO-0032.

SIO-0088
ETCH NAME: Hydrofluoric acid, dilute TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
1 .... HF
15 .... H_2O
DISCUSSION:
SiO_2, as a residual PSG surface film from poly-Si growth on (100) silicon wafer substrates

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with heavy phosphorus doping which leaves the P-glass. Etch surface with solution shown to remove the residual PSG glass.
REF: Chang, C C et al — *J Appl Phys,* 130,1159(1983)

SIO-0034
ETCH NAME: Hydrofluoric acid TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... HF
x .... H_2O
DISCUSSION:
SiO_2, thin film deposited on InP, (100) wafer substrates in a study of CVD oxides on indium phosphide.
REF: Bertrand, P A et al — *J Vac Sci Technol,* B(1),832(1983)

SIO-0015b
ETCH NAME: BHF TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... 1.2 *M* HF
x .... 10.3 *M* NH_4F
DISCUSSION:
SiO_2, thin film deposits used in a study of the etching mechanisms of hydrofluoric acid solutions.
REF: Ibid.

SIO-0015c
ETCH NAME: Hydrofluoric acid TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... 1 *M* HF
DISCUSSION:
SiO_2, thin film deposits used in a study of the etching mechanisms of hydrofluoric acid.
REF: Ibid.

SIO-0037
ETCH NAME: TIME:
TYPE: Phosphor, defect TEMP:
COMPOSITION:

(1)	(2)	(3)
x .... Zn_2SiO_4:Mn	x .... CdS:Zn	x .... YV_2
x .... Freon, TF	x .... Freon, TF	x .... Freon, TF

DISCUSSION:
SiO_2, thin films deposited on silicon wafers and devices used in a study of defect recognition using fluorescent tracers. Both phosphor powders and dyes — organic and inorganic — used under a variety of conditions including electrovoltaic. Phosphors and dyes "plate" out at defect points. Solution (1) fluoresces green/yellow in short-wave UV; (2) fluoresces yellow in long-wave UV and (3) fluoresces bright red. Reaction is electrostatic or electrophoric.
REF: Kern, W et al — *RCA Rev,* 43,310(1982)

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SIO-0038a
ETCH NAME:
TYPE: Acid, cleaning
COMPOSITION:
40 .... HF
15 .... H_2SO_4
DISCUSSION:
Corning glass 7720. This solution will leave a white precipitate on the glass surface.
REF: Teeg, R O et al — Final Rep (15 April 1966), Control No. 65-0388f

TIME:
TEMP:

SIO-0038b
ETCH NAME:
TYPE: Acid, cleaning
COMPOSITION:
53 .... HF
37 .... HCl
DISCUSSION:
Corning glass 7720. This solution will not leave any precipitate on the glass surface. (See ALO-0038a.)
REF: Ibid.

TIME:
TEMP:

SIO-0039a
ETCH NAME: Nitric acid, dilute
TYPE: Acid, cleaning
COMPOSITION:
x .... HNO_3
x .... H_2O
DISCUSSION:
Glass — various types. Different solution concentrations of nitric acid can be used as general glass surface cleaners. May leave craters in the glass surface depending upon glass formulation.
REF: Tichane, R M & Carrier, G B — *J Am Ceram Soc*, 44,606(1961)

TIME:
TEMP: Hot

SIO-0039b
ETCH NAME:
TYPE: Acid, cleaning
COMPOSITION:
(1) x .... 1% NaOH (2) x .... 5% HCl
DISCUSSION:
Corning glass 7740 (borosilicate). Mechanically polish surfaces with calcium carbonate, $CaCO_3$. Etch clean sequentially, first in (1) and then in (2) at temperatures shown. DI water rinse after each etch cycle at RT. Both Nonex (Corning 7720) and Kimble (K-772) are attacked by moisture which leaches alkalies producing a frosted surface. Wash these glass surfaces in NH_4F 10 sec to counteract the frosting effect.
REF: Ibid.

TIME: (1) 10 min (2) 2 min
TEMP: (1) 95°C (2) 50°C

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SIO-0040
ETCH NAME: TIME: 30 sec
TYPE: Acid, cleaning TEMP: RT
COMPOSITION:
40 .... H_2SO_4
20 .... HF
40 .... H_2O
DISCUSSION:
Glass — various types. Solution used in a study of stress in glass. The following procedure was used: (1) etch in solution shown; (2) running DI water rinse, 15 sec; (3) repeat etch and (4) repeat rinse. Continue the three-step procedure for a total of 5 min etching time (10 cycles).
REF: Kistler, S S — *J Am Ceram Soc*, 45,59(1962)

SIO-0041a
ETCH NAME: TIME:
TYPE: Acid, cleaning TEMP: RT
COMPOSITION:
5 .... HF
33 .... HNO_3
60 .... H_2O
2 .... Dreene (wetting agent)
DISCUSSION:
Glass — various types, as both sheet and rods. Used as a general glass cleaning solution. It is called a variable solution. Also used on molybdenum.
REF: Crawley, R H A — *Chem Ind*, 45,1205(1953)

SIO-0039c
ETCH NAME: TIME:
TYPE: Acid, cleaning TEMP: Hot
COMPOSITION:
x .... NaOH
x .... $NaCO_3$
x .... H_3PO_4
DISCUSSION:
Glass — soda-lime. Solution used to remove contamination on soda-lime glass that is not removed by hot water washing, only.
REF: Ibid.

SIO-0041b
ETCH NAME: TIME:
TYPE: Acid, cleaning TEMP: RT
COMPOSITION:
1000 ml .... H_2SO_4
35 ml .... $Na_2Cr_2O_7$, sat. sol.
DISCUSSION:
Glass — various types. This has been a standard glass cleaning solution for a number of years. It can be mixed, used, stored and re-used for several months without loss of efficiency even though it turns coal-black in color. **CAUTION:** It is a severe oxidizing solution and should be handled with extreme care. According to Crabbs (SIO-0042), chromium ions are difficult to remove from cleaned surfaces even with extended water washing

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(3 or 4 days). Because of this, it is no longer used for cleaning quartz diffusion tubes and parts where residual chromium ions can affect semiconductor electrical parameters.
REF: Ibid.
SIO-0042: Crabbs, G — personal communication, 1962

SIO-0043
ETCH NAME: AB etch (RCA) TIME: 15—20 min
TYPE: Acid, cleaning TIME: 80°C
COMPOSITION:

"A"	"B"
1—2 .... NH_4OH	1—2 .... HCl
1—2 .... H_2O_2	1—2 .... H_2O_2
4—6 .... H_2O	4—6 .... H_2O

DISCUSSION:
Glass — thin film deposition and growth in bevelled moats of high power silicon transistors. Electrophorically deposit glass frit in isopropanol (IPA) containing NaOH or HF additives. Form borosilicate in ethyl acetate plus acetone or methanol. Add yttrium or magnesium nitrates to the glass and melt in a platinum boat at 1450°C, 4 h. Pour between water-cooled rollers as a thin sheet and then pulverize in a ball mill. Clean powdered glass in the solutions shown above. Silicon, (100) p-type wafers, 2—3 Ω cm resistivity were used in the transistor fabrication. Fill wafer moats formed to separate individual transistors with the glass frit described and fire at 700°C as a surface protectant coating of p-n-p junctions.
REF: Shimbo, M — *J Electrochem Soc*, 132,393(1985)

SIO-0044
ETCH NAME: Hydrofluoric acid, vapor TIME: 10 min
TYPE: Acid, removal TEMP: 30°C
COMPOSITION:
x .... HF, vapor
DISCUSSION:
Glass — microscope slides. Solution vapors used to roughen microscope surface to produce irregular protrusions of variable height (angstroms). The following process was used: (1) pre-clean with soap and water; (2) mask off one side; (3) heat to 100°C and then (4) immerse in a sealed enclosure of HF vapors at 30°C. After etching, remove and DI water rinse. Slides were used as-polished and as roughen etched in a study of metal thin films deposited on these slides. The roughening was used to improve adhesion of 3000 Å thick metal films. Metals studied were electrically (1) good conductors — noble metals; (2) moderate conductors — Sn, Pb, and Ni and (3) semi-metal — Bi.
REF: Boyd, G T et al — *Phys Rev*, B30,519(1984)

SIO-0039d
ETCH NAME: Hydrofluoric acid, dilute TIME:
TYPE: Acid, cleaning TEMP: Boiling
COMPOSITION:
1 .... 1% HF
DISCUSSION:
Glass — borosilicate and soda-lime. The solution as applied will roughen borosilicate glass surfaces. Soda-lime surfaces remain smooth.
REF: Ibid.

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SIO-0045a
ETCH NAME: Hydrofluoric acid, dilute
TYPE: Acid, removal
COMPOSITION:
x .... HF (1—25%)
DISCUSSION:
Glass — various types. Processing produces micro-cracks on glass surfaces and under exposure to atmospheric conditions alkali hydrates are formed which show long-term outgassing characteristics. By etch removal of 0.002 to 0.003″ from surfaces with HF, glass strength is improved as much as by a factor of 10, though tension stress remains.
REF: Kohl, W H — *Handbook of Materials and Techniques for Vacuum Devices,* Reinhold, New York, 1967, 15
TIME:
TEMP:

SIO-0045b
ETCH NAME: Heat
TYPE: Thermal, gas removal
COMPOSITION:
x .... heat
DISCUSSION:
Glass — lead, soda-lime, borosilicate, etc. Water vapor and CO_2 primarily adsorbed on surfaces; H_2, N_2, O_2, and CO secondary. Bake out at 150°C (soda-lime); 200°C (lead), and 250°C (borosilicate and others) to desorb gases.
REF: Ibid.
TIME:
TEMP: See discussion

SIO-0046
ETCH NAME: Hydrogen peroxide
TYPE: Acid, cleaning
COMPOSITION:
x .... 5% H_2O_2
+ .... NH_3 for pH 11
DISCUSSION:
Glass — various types. The following procedure was used in cleaning surfaces: (1) scrub in hot tap water +0.5% Igipal wetting agent; (2) rinse in running, hot tap water; (3) rinse in running DI water and (4) boil in H_2O_2 and rinse in DI water and N_2 blow dry.
REF: Kern, H E & Graney, E T — *ASTM Special Tech Pub,* #246(1959)
TIME: 15 min
TEMP: Boiling

SIO-0047
ETCH NAME: Laser
TYPE: Ionic, preferential
COMPOSITION:
x .... H^+ ions
DISCUSSION:
Glass — Pyrex substrates. A pulsed Excimer laser beam using H_2 used to pattern etch Pyrex glass at its softening point. An Al/O film deposited on SiO_2/silicon turns black under laser etching with major reduction of sheet resistance. Author says that color change is representative of different chemical etching properties.
REF: Ehrlich, D J — *J Vac Sci Technol,* B(3),1(1985)
TIME:
TEMP:
POWER:

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SIO-0048
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
x .... acids
TIME:
TEMP:
DISCUSSION:
Glass — various types. This is a major reference for the etching, cleaning, and surface treatment available for glass.
REF: Holland, L — *The Properties of Glass Surfaces*, John Wiley & Sons, New York, 1964.

SIO-0049a
ETCH NAME:
TYPE: Alkali, lap/etch
COMPOSITION:
x .... Ce_2O_3
x .... H_2O
x .... glycerin
x .... 5% NaOH (KOH)
TIME: Variable
TEMP: RT
DISCUSSION:
Glass — soda-lime. Cerium oxide is a standard lapping powder for preparing chrome glass masks. Acid slurry shown was developed to reduce the amorphous surface smearing common to final polishing of glass blanks.
REF: Walker, P — personal development, 1975

SIO-0050
ETCH NAME:
TYPE: Alkali, lap/etch
COMPOSITION:
(1) x .... emery
x .... glycerin
x .... water
(2) x .... alumina
x .... glycerin
x .... water
x .... 10—30% KOH (NaOH)
TIME: Variable
TEMP: RT
DISCUSSION:
Glass — various types. Mixture (1) is a standard lapping slurry for glass (SIO-0050). Mixture (2) was developed for polishing vacuum glass ports to remove scratches and cleaning blanks of both Pyrex and quartz (SIO-0051).
REF: Hodgman, C D — *Handbook of Chemistry and Physics*, 27th ed, Chemical Rubber Co., Cleveland, OH, 1943, [solution (1)]
SIO-0049b: Ibid.
SiO_2, soda-lime glass used for photo resist chrome glass masks. Developed solution (2) as an acid slurry solution to reduce the fine smear surface that can remain during optical polish of glass.

SIO-0052
ETCH NAME: Aqua regia
TYPE: Acid, cleaning
COMPOSITION:
3 .... HCl
1 .... HNO_3
TIME: 1—4 h
TEMP: RT

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DISCUSSION:

SiO_2, as quartzware. For general cleaning of laboratory glassware, epitaxy tubes, and parts quartzware. Immerse and soak parts until clean. Heavy wash with running DI water and dry under IR heat lamps.

REF: Zelinsky, T — personal communication, 1980

SIO-0053

ETCH NAME: Hydrochloric acid, vapor — TIME: 15 min

TYPE: Acid, pinhole — TEMP: Hot

COMPOSITION:

x .... HCl, vapor

DISCUSSION:

SiO_2, thin film RF sputtered. Authors say that dust in laboratory air will cause pinholes in an oxide film. Develop pinholes by soaking specimen in hot HCl vapors.

REF: Davidse, P D & Masiel, L I — *J Appl Phys*, 37,574(1966)

SIO-0054a-b

ETCH NAME: BHF — TIME:

TYPE: Acid, removal — TEMP: RT

COMPOSITION:

(1) 3 ml .... HF
15 g .... NH_4F
22 ml .... H_2O

(2) x .... HF, hot

DISCUSSION:

SiO_2, thin films thermally evaporated used as diffusion masks with KMER photo resist. The BHF solution (1) used for patterning with KMER. Hot HF used to strip oxide. A study of controlled etching in making thin film masks.

REF: Kelly, C E — *Electrochem Technol*, 2,358(1964)

SIO-0055

ETCH NAME: — TIME:

TYPE: Acid, patterning — TEMP:

COMPOSITION:

(1) 8% .... HF
84% .... HNO_3
8% .... HAc

(2) x .... BHF

DISCUSSION:

SiO_2, thin films deposited on silicon wafers for microcircuits using oxide isolation. Solution (1) was used to etch a groove through the oxide. After grooving all oxide was stripped in a BHF solution and surfaces re-oxidized.

REF: Schnable, G C et al — *J Electrochem Technol*, 4,57(1966)

SIO-0056

ETCH NAME: — TIME:

TYPE: Acid, cleaning — TEMP:

COMPOSITION:

x .... H_2SO_4
x .... x% CrO_3

DISCUSSION:

SiO_2 as (1) soda-lime glass and (2) fused silica microscope slides used as substrates for

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thin film metallization with chromium, copper, or nickel. The following cleaning procedure was used on the glass substrates before metallization:

1. Detergent clean: Liqui-nox:DIH_2O mixture with ultrasonic agitation and DI water rinse.
2. Acid clean: Use acid mixture shown (a standard "glass cleaner" solution) to remove organics.
3. Wash: DI water (deionized and distilled) — three times — in ultrasonic with rinsing to remove traces of acid.
4. Blow dry: He, single high pressure burst.

Metals were then evaporated: Ni, 2150 Å; Cu, 536 Å; and Cr, 1000 Å. Nickel on fused silica, others on soda-lime. As deposited, films were smooth with only an occasional pinhole. Thermal cycling developed pinholes/defects with crack networks. This was a study of failure mechanisms in thin metal films.
REF: Zito, R R — *Thin Solid Films,* 87,87(1982)
SIO-0067: Primak, W — *J Appl Phys,* 32,660(1961)

SiO_2, as vitreous silica glass used in a study of neutron induced damage and annealing. Cleaned in a standard glass cleaner, then in cool HCl, water wash, acetone rinse and dry.

SIO-0059
ETCH NAME:
TIME:
TYPE: Acid, removal/jet
TEMP: RT
COMPOSITION:
40 g .... $NH_4F.HF$
8 g .... NaF/L
DISCUSSION:
SiO_2, native oxide removal and etching of silicon as a jet etch solution. Produces flat bottomed pits in the silicon. Addition of $K_3F(CN)_6$ reduces pits size and formation.
REF: Schmidt, P F & Keipler, D A — *J Electrochem Soc,* 106,592(1959)

SIO-0061
ETCH NAME: Hydrofluoric acid
TIME:
TYPE: Acid, defect
TEMP:
COMPOSITION:
x .... HF, conc.
DISCUSSION:
SiO_2, thin film layers grown on silicon, (111) p-type, 6—12 Ω cm resistivity wafers. Wet thermal oxides grown 500—2000 Å thick at 1000°C, 1050°C, and 1100°C. Refractive index: n = 1.46. Silicon warps under the oxide coatings with severity depending upon thickness and deposition temperature. High oxidation temperature creates dislocations and warpage decreases with films under 0.5 μm thick.
REF: Lecni, H & Satch, S — *Jpn J Appl Phys,* 23,L743(1984)

SIO-0062
ETCH NAME:
TIME:
TYPE: Acid, removal
TEMP: RT
COMPOSITION:
5 g .... $NH_4F.HF$
5 ml .... HF
50 ml .... H_2O
50 ml .... glycerin

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DISCUSSION:

SiO_2, thin film oxidation of silicon, (111) n-type wafers at 1050°C as wet oxide, 2000—2500 Å thick. A study of both oxides and nitrides (both DC plasma deposited) vs. thermal oxidation and dislocation development at the silicon interface. Study included pre-diffusion of wafers with nickel to reduce dislocation development. Solution used as a slow removal etch of both oxides and nitrides. Wafers were cleaved ⟨110⟩ for observation of dislocation depth. Better than a 50% reduction in dislocation development was observed in wafers pre-diffused with nickel.

REF: Walker, P & Onidera, G — Spring Meet Phys Failure Conf, Battelle Memorial Institute, Columbus, OH, 1964

SIO-0063

ETCH NAME: Hydrofluoric acid — TIME:

TYPE: Acid, removal — TEMP:

COMPOSITION:

x .... HF, conc.

DISCUSSION:

SiO_2, thin film oxidation of silicon at 1200°C under 5×10^{-6} Torr and with added phosphorus (P_{H_2O}) vapor at 1180°C for PSG cover film. Used in a study of stress anisotropy of the films.

REF: Priest, J et al — *J Appl Phys*, 34,347(1963)

SIO-0064

ETCH NAME: Hydrofluoric acid — TIME:

TYPE: Acid, removal — TEMP:

COMPOSITION:

x .... HF, conc.

DISCUSSION:

SiO_2, grown as spherolitic pellets of alpha-cristobalite at the contact point of GaAs/quartz boat that were sandblasted. Alpha-cristobalite m.p. is 1713°C — it formed at less than 1250°C. GaAs, m.p. is 1238°C.

REF: Yamaguchi, M et al — *J Electrochem Soc*, 113,294(1966)

SIO-0065

ETCH NAME: Water — TIME:

TYPE: Acid, float-off — TEMP:

COMPOSITION:

x .... H_2O

DISCUSSION:

SiO_2, glass microscope slides used as substrates for epitaxy growth of ZnTe with cover aluminum metallization. The ZnTe was thermally evaporated from powder, and aluminum evaporation was 0.1 to 0.5 mm thick. Thin film was removed from glass by the float-off technique using water. Carbon replication made for TEM study.

REF: Patel, S M & Patel, N G — *Thin Solid Films*, 113,185(1984)

SIO-0066

ETCH NAME: Steam — TIME:

TYPE: Oxide, growth — TEMP: >500°C

COMPOSITION: — PRESSURE: >25 atms

x .... H_2O, steam

DISCUSSION:

SiO_2, thermally grown oxide on silicon p-n junction devices in steam between 500—900°C

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at pressures between 25—500 atm. Film growth appeared to be linear to about 50,000 Å thickness. At any given temperature there is a pressure above which the oxides are soluble in steam and fail to form. 500°C limit is 500 atm; 850°C limit is 150 atm. Cleaning of silicon prior to oxidation is critical. The following sequence was used:

1. Mechanically lap and polish etch silicon.
2. Soak in HNO_3, rinse in H_2O, soak in HF, rinse three times in H_2O.
3. Boil in HNO_3, rinse two times in H_2O and then in Hi-Q H_2O.

Note: Following HF step in (2), hold specimens under water in quartz beakers.

Recommend that oxide films be at least 3000 Å thick for maximum ambient sensitivity.
REF: Madden, T C & Gibson, W M — *Rev Sci Inst*, 34,50(1963)
SIO-0092: Madden, T C & Gibson, W B — *J Appl Phys*, 27,1418(1966)

SiO_2, thin film passivation on silicon p-n junction particle detectors. There can be bulk impurities in oxide with a p-type inversion layer at the SiO_2/Si interface which reduces with increased resistivity. Film is less impervious to water prior to phosphorus diffusion but softens after. A "ring" structure around active junctions reduces leakage from 30 to 0.6 μA at 400 V.

SIO-0069
ETCH NAME: TIME:
TYPE: Gas vapor, growth TEMP: Hot
COMPOSITION:
(1) x .... H_2O_2, vapor (2) x .... steam, O_2 (3) x .... steam, O_3
DISCUSSION:

SiO_2 and SiO_3, wet oxides used to "age" silicon devices using the three solutions shown. Silicon was etched in CP4 with 10 drops Br_2/50 ml solution. CP4 reactivity was varied with addition of acetic acid (HAc). See GE-0065c for additional use of CP4.
REF: Blankenship, J L & Borkowski, C J — *IRE Trans Nuc Sci*, NS-7,190(1960)

SIO-0070
ETCH NAME: TIME:
TYPE: Halogen, cleaning TEMP: RT
COMPOSITION:
3 mg .... I_2
100 ml .... MeOH
DISCUSSION:

Glass, as microscope slides and cover glasses used as physical contact masks on semiconductor wafers and as monitors for thickness measurement in study of metals reactions on metallized wafer surfaces. Glass was cleaned by (1) 1HF:1H_2O dip and water rinse; (2) 1HCl:1H_2O dip and water rinse, then (3) rinse in solution shown and N_2 blow dry. Also used on fused quartz substrate blanks prior to metallization and fabrication for microelectronic circuits. The iodine complexes and removes ionic contamination on surfaces at above 100°C, leaving a molecularly clean surface.
REF: Walker, P et al — material development, 1964/1980

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SIO-0071
ETCH NAME: BHF — TIME:
TYPE: Acid, removal — TEMP:
COMPOSITION:
1 .... HF
10 .... NH_4F
DISCUSSION:
SiO_2, thin films 160 nm thick. Films were nitrided at 1200°C in N_2 or NH_3 for 5 or 17 h. Resultant silicon oxynitride films were called "nitroxide thin films". Solution shown was used to determine etch rates of pure SiO_2 and the nitroxides at the two temperature levels. Refractive index of SiO_2, n = 1.46 and of nitroxide, n = 1.6—1.7. Dielectric constant of nitroxide, e = 4—5.
REF: Kato, I et al — *J Electron Mater,* 12,913(1984)
SIO-0072: Gordon, L — *J Appl Phys,* 34,1220(1963)
SiO_2, thin films grown on polycrystalline silicon called "polyoxide". Etch remove with BHF solutions.
SIO-0082: Mastroianni, S T — *Solid State Technol,* May 1984, 155
SiO_2, thin films characteristics evaluated.

SIO-0073a
ETCH NAME: Hydrofluoric acid, dilute — TIME:
TYPE: Acid, float-off — TEMP: RT
COMPOSITION:
x .... HF
x .... H_2O
DISCUSSION:
Corning glass #7059, used as substrates for epitaxy growth of amorphous $ZnGeAs_2$. Solution shown used to separate a-$ZnGeAs_2$ from the glass by the float-off technique for TEM study.
REF: Shah, S I & Greene, J E — *J Cryst Growth,* 68,537(1984)

SIO-0073b
ETCH NAME: — TIME:
TYPE: Solvent, cleaning sequence — TEMP:
COMPOSITION:
(1) x .... soap (2) x .... TCE (3) x .... acetone (4) x .... MeOH
x .... H_2O
DISCUSSION:
Corning glass #7059 used as substrates for a-$ZnGeAs_2$ epitaxy. Glass was cleaned by scrubbing with a detergent (1), then degreased successively with TCE, acetone, and MeOH rinses. Final blow dry with dry nitrogen jet.
REF: Ibid.

SIO-0074
ETCH NAME: Secco etch — TIME:
TYPE: Acid, preferential — TEMP:
COMPOSITION:
2 .... HF
1 .... 0.15 *M* $K_2Cr_2O_7$
DISCUSSION:
SiO_2, thin films grown on silicon, (100), n-type substrates used in a study of indium diffusion and oxidation reactions. Both dry O_2 and steam used at different temperatures and

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times. Secco etch used to develop stacking faults and other defects in both the oxide and silicon wafers at the SiO_2/Si interface.
REF: Cerofolini, G F et al — *J Cryst Growth*, 109,137(1983)

SIO-0075
ETCH NAME: Carbon black
TYPE: Metal, decoration
COMPOSITION:
x .... C, powder
TIME: N/A
TEMP: RT
DISCUSSION:
SiO_2, thermally oxidized thin films on p-type (100) silicon wafers used in a study of pinholes in oxides and phosphorus induced microdefects in silicon. Oxide was converted to PSG with P diffusion. Pinhole density was measured with both Metso etching and carbon black dusting of surface to enhance pinholes. Microdefects in silicon could be related to pinhole locations in the covering oxide.
REF: Blackstone, S et al — *J Electrochem Soc*, 130,667(1982)

SIO-0076
ETCH NAME: Steam
TYPE: Gas vapor, re-flow
COMPOSITION:
x .... H_2O, vapor
TIME:
TEMP: >100°C
PRESSURE:
DISCUSSION:
SiO_2, thin films on silicon wafers as doped BPSG. Steam used to re-flow oxide after deposition. With greater than 0.4% by weight concentration of boron, films blister during temperature annealing.
REF: Harris, T — *J Electrochem Soc*, 132,501(1985)

SIO-0077a
ETCH NAME: PAW (EDP)
TYPE: Acid, pinhole
COMPOSITION:
1 .... ethylenediamine
1 .... pyrocatechol
4 .... H_2O
TIME: 1—2 h
TEMP: 110—115°C
DISCUSSION:
SiO_2, thin films RF sputtered 200—700 nm thick on (100) silicon wafers and used in a study of pinhole density development. Etch ratio of Si:SiO_2 is about 1000:1 with the solution shown. Etching was done in a ventilated chemical sink to remove generated H_2 and under N_2 ambient to prevent oxidation of amine. After etching, remove SiO_2 and count etch pits in silicon for pinhole density. Method evaluated against Cu-decoration and RF plasma CF_4:O_2 etching. Plasma etching developed 10—15% less pinholes due to low conductance of pinholes and up to 50% less with Cu-decoration due to Cu coalescence in the presence of high pinhole density. Pinhole concentration density ranged from 10^5 down to about 6×10^3 as film thickness increased from 200 to 700 nm. PAW and RF plasma results were equivalent, but Cu-decoration was erratic. The plasma method is considered reliable and more easily applied.
REF: Meguro, T et al — *J Electrochem Soc*, 128,1379(1981)

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SIO-0077b
ETCH NAME: Copper
TYPE: Metal, decoration
COMPOSITION:
x .... $CuSO_4$
DISCUSSION:
SiO_2, thin films deposited on (100) silicon wafers used in a study of pinhole density. During etching a nonuniform electric field is applied to deposit copper at pinhole locations in the oxide, but will also precipitate randomly on the oxide film if the surface is contaminated. See SIO-0077a for additional discussion.
REF: Ibid.
TIME:
TEMP:

SIO-0078: Shannon, W J — *RCA Rev*, 31,431(1970)
Reference for copper solution and method applied in SIO-0077b.

SIO-0077c
ETCH NAME: RF plasma
TYPE: Ionized gas, pinholing
COMPOSITION:
x .... CF_4
x .... O_2 (10%)
DISCUSSION:
SiO_2, thin films deposited on (100) silicon wafers and used in a pinhole density study. Si:SiO_2 etch ratio was maximized with 10% O_2 at 1:130, though about 100 nm of SiO_2 had to be removed before pinhole etched points in silicon were of sufficient size for count evaluation. Density count was 10—15% less than the PAW method, but reliable and more easily applied. See SIO-0077a for further discussion.
REF: Ibid.
TIME:
TEMP:
GAS FLOW:
PRESSURE: 0.25 Torr
POWER: 50 W

SIO-0081: Kotani, H et al — *J Electrochem Soc*, 130,645(1983)
SiO_2, as PSG thin films used as interconnects on semiconductor devices, along with p-SiN. AlSi and AlSiCu thin films. Carrier gas was argon at 1.7 Pa pressure and 0.5W/cm^2 power. PSG etch rate was 60 Å/min.; p-SiN at 70 Å/min. At greater than 200°C, the AlSi etch rate was 100 Å/min.

SIO-0079
ETCH NAME:
TYPE: Colloid, replication
COMPOSITION:
x .... 4% collodion
x .... amyl acetate
DISCUSSION:
Si, (100) n-type wafers used in a study of island stage growth of oxidation. Wafers were cleaned prior to oxidation: (1) 10% HCl, RT for 10 min and DI water rinse; (2) chem/mech polish on pellon pad with 1% Br_2:MeOH; rinse in MeOH, then acetone and N_2 blow dry. After oxidation, surfaces were coated with solution shown, followed by water lift-off collodion for replica microscope study.
REF: Makky, W H & Wilmsen, G W— *J Electrochem Soc*, 130,659(1983)
TIME:
TEMP:

SIO-0080
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
x .... HF
TIME:
TEMP:

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DISCUSSION:

Si, (111) n-type 3—5 Ω cm resistivity wafers used in a study of SiO_2 properties as RF sputtered thin films. Wafers were cleaned: (1) TCE degrease; (2) acetone rinse; (3) $3H_2SO_4$:$1H_2O$, RT dip; (4) 1HF:$10H_2O$, RT dip and DI H_2O rinse. Oxide was etched in concentrated or dilute HF.

REF: Lels, M K — *J Electrochem Soc*, 130,658(1983)

SIO-0083

ETCH NAME: TIME:

TYPE: Mechanical, fracture TEMP: RT

COMPOSITION:

x .... fresh fracture

DISCUSSION:

SiO_2, glass specimens. A study of surface and internal structure of freshly fractured glass under an electron microscope. Fracture shown to be 50—300 Å deep with a granular surface structure. (*Note:* Common glass fracture is conchoidal with highly reflective surfaces.)

REF: Narez, M & Sella, C — *C R Acad Sci (Paris)*, 250,4325(1960)

SIO-0084

ETCH NAME: Hydrofluoric acid TIME:

TYPE: Acid, float-off TEMP:

COMPOSITION:

1 .... HF

10 .... H_2O

DISCUSSION:

Pyrex blanks used for co-deposition of CuPd thin films in a material study. Quartz blanks evaluated, but they develop a PdSi film. Solution shown used to clean blanks before metallization. Also used for film float-off and TEM study.

REF: van Langrveld, A D et al — *Thin Solid Films*, 109,193(1983)

SIO-0094: Lee, M K — *J Electrochem Soc*, 130,648(1983)

SiO_2, RF sputtered thin films on (111) silicon, n-type, 3—5 Ω cm resistivity, used in a properties study of deposited films. Prepare silicon wafers: (1) degrease in TCE plus acetone rinse; (2) acid clean with $3H_2SO_4$:$1H_2O$ and DI H_2O rinse; (3) dip in 1HF:$10H_2O$ rinse, prior to oxidation.

SIO-0095: Ligenza, J R — *J Electrochem Soc*, 109,73(1962)

SiO_2, thin films deposited on silicon wafers in a general study of oxidation.

SIO-0097: Ligenza, J R — *J Phys Chem*, 65,2011(1961)

SiO_2, thin films deposited on silicon wafers in a general study of oxidation.

SIO-0098: Ligenza, J R & Spitzer, W G — *J Phys Chem Solids*, 14,131(1960)

SiO_2, thin films deposited on silicon wafers in a general study of oxidation.

SIO-0099: Spitzer, W G & Ligenza, J R — *J Phys Chem Solids*, 17,196(1961)

SiO_2, thin films deposited on silicon wafers in a general study of oxidation.

SIO-0085

ETCH NAME: TIME:

TYPE: Oxide, adhesion coat TEMP:

COMPOSITION:

(1) x .... *HMDS (2) x .... TiO_2, ZrO_2, HfO_2

*Hexamethyldisilazane

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DISCUSSION:

SiO_2, thin films deposited on a variety of substrates/surfaces (glass, Si_3N_4, GaAs, Si, etc.). Highly polished surfaces show poor adhesion of photo resist lacquers. The materials shown above were deposited after the SiO_2 to improve adhesion. Spun on HMDS was not as effective as the oxides. For a TiO_2 film: bubble $TiCl_4$ in N_2/O_2 using a viton tube with glass nozzle at RT, then dry with He gas. 25 Å TiO_2 deposits in 5 sec at RT; 25 Å in 2—3 sec at 100°C. Photo resist was 1350J. (*Note:* These very thin oxide films produce a granular/island-like surface that improves photo resist adhesion.)

REF: Marinace, J C & McGibbon, R C — *J Electrochem Soc*, 129,2389(1982)

SIO-0086a

ETCH NAME: Hydrochloric acid — TIME:

TYPE: Acid, pinhole — TEMP: Hot

COMPOSITION:

x .... HCl, vapor

DISCUSSION:

SiO_2, thin films deposited on silicon substrates in a study of pinholes. HCl vapor will attack at pinhole locations, and etch down into the underlying silicon producing recognizable etched pits.

REF: Sullivan, M V — 1st Kodak Seminar on Microminiaturization, 1965, 30

SIO-0086b

ETCH NAME: EPD — TIME: 6 h

TYPE: Acid, pinhole — TEMP: 115°C

COMPOSITION:

17 ml .... ethylenediamine

3 g .... pyrocatechol

8 ml .... H_2O

DISCUSSION:

SiO_2, thin films deposited on silicon substrates in a study of pinholes. Solution used to develop pinholes in the SiO_2 coating.

REF: Ibid.

SIO-0087

ETCH NAME: — TIME:

TYPE: Acid, step-etch — TEMP:

COMPOSITION:

1 .... HF

10 .... NH_4HF

15 .... H_2O

DISCUSSION:

SiO_2, thin films RF sputter deposited in argon on (100) oriented silicon wafers in a study of argon pressure vs. film etch rates. Solution used to step-etch films for deposit thickness measurements.

REF: Ciachi, T & Serikawd, T — *J Electrochem Soc*, 131,2720(1984)

SIO-0088

ETCH NAME: RIE — TIME:

TYPE: Ionized gas, removal — TEMP:

COMPOSITION: — GAS FLOW: 4—45 SCCM

x .... CHF_3 — PRESSURE: 45—90 mT

POWER: 500—2500 W

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DISCUSSION:

SiO_2, thin films grown on (100) silicon wafers. Films grown by CVD at 950°C for 10 KÅ thickness. A study of etching SiO_2 films by RIE. Prior to RIE etching with gas shown, clean SiO_2 surfaces for 10 min in O_2.

REF: Light, R W & See, F C — *J Electrochem Soc*, 129,1152(1982)

SIO-0089
ETCH NAME: Boron
TIME:
TYPE: Metal, diffusion
TEMP: 1300 K
COMPOSITION:

x .... BSG

DISCUSSION:

BSG, as borosilicate glass on silicon. A thin sheet of this boron doped glass when wetted to a silicon wafer surface and heated to 1300 K will diffuse boron into the wafer as a p-type layer several microns deep with material under vacuum. (*Note:* Remove excess glass with HF.)

REF: Allen, F G et al — *J Appl Phys*, 31,979(1960)

SIO-0090: Tarn, W H & Walker, P — personal application, 1970

SiO_2 thin film layers about 2500 Å thick grown on (111) and (100) silicon wafers by wet oxidation for use as a diffusion mask. Patterns were opened in the silica layer by photolithography. With wafers in vacuum system liquid Boracine was bubbled with nitrogen gas as the carrier for pyrolitic diffusion of boron. Some "rosette" crystalline structures of borosilicate glass observed in the oxide thin films after diffusion.

SIO-0091a-c
ETCH NAME:
TIME:
TYPE: Solvent, cleaning
TEMP: Hot to boiling
COMPOSITION:

(1) x .... TCE (2) x .... acetone (3) x .... ISO/IPA

DISCUSSION:

SiO_xN:H and Si:H thin films, latter as cover film, deposited on glass, UV grade quartz, SnO_2, Al, and SST substrates. For good film adhesion, clean substrates: (1) TCE, boiling; (2) acetone, warm; (3) ISO, boiling before hydrogenated growth of films.

REF: Carasco, F et al — *J Appl Phys*, 57,5306(1985)

SIO-0093
ETCH NAME: Heat
TIME:
TYPE: Thermal, conversion
TEMP: 870°C
COMPOSITION:

x .... heat

DISCUSSION:

a-SiO_2, thin films used as a diffusion mask on silicon wafers can crystallize to cristobalite, SiO_2 at about 870°C in the presence of catalysts, as Fe_2O_3, CaO:KCl; NaCl:P_2O_5; Be_2O. Catalysts also affect c-SiO_2 similarly. Use of PBr_3 as diffusant at 170°C is possibly better due to rapid vaporization of bromine, and improves reverse leakage. (*Note:* The 670°C temperature is an inversion point between beta-quartz and alpha-tridymite.)

REF: Klerer, J — *Bell Tel Lab Rep*, 1964

SIO-0096: Silverman, S J & Singleton, J B — *J Electrochem Soc*, 105,891(1958)

SiO_2 as a residual PSG film after phosphorus diffusion can be removed with HF. Paint-on: 2 g $NiCO_3$:5 g P_2O_5:100 ml 2-methoxethanol — use camel hair brush. Diffuse at 1250°C, 16 h for 1.5 mil diffusion depth of phosphorus.

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SIO-0100
ETCH NAME: Glass
TYPE: Frit, passivation
COMPOSITION:
x .... SiO_2, powder
TIME:
TEMP: 800°C
DISCUSSION:
SiO_2, as finely powdered glass frit in a water slurry. A camel hair brush used to paint-on coat aluminum Sphere Alloy Zener diodes as final passivation after package stud mounting. Fired in air at 800°C to melt-form as glass covering. Time at temperature variable with thickness and/or agglomeration of frit. (*Note:* Agglomeration is a major problem in the application of frit slurries with incomplete melt resulting in unmelted frit, bubbles, and poor structuring with stress in the coating film.)
REF: Walker, P — personal development, 1963

SIO-0101
ETCH NAME: Potassium hydroxide
TYPE: Electrolytic, oxidizing
COMPOSITION:
x .... KOH
x .... H_2O
TIME:
TEMP:
ANODE:
CATHODE:
POWER:
DISCUSSION:
SiO_2, grown as a hydrated oxide on silicon wafers used in a study of oxidation rates.
REF: Glembocki, O J & Stahlbush, R E — *J Electrochem Soc,* 132,145(1985)

SIO-0102
ETCH NAME: Water
TYPE: Acid, oxidation
COMPOSITION:
x .... H_2O
TIME:
TEMP: Boiling
DISCUSSION:
SiO_2, grown as a hydrated oxide on silicon wafers as a surface cleaning step. After oxide growth, strip with HF or BHF, rinse in DI water, and dry.
REF: Heimann, R B — *J Vac Sci Technol,* B(1),108(1983)

SIO-0103
ETCH NAME: Nitric acid
TYPE: Acid, oxidation
COMPOSITION:
x .... HNO_3, conc.
TIME:
TEMP: Hot
DISCUSSION:
SiO_2, grown as a hydrated oxide on silicon wafers as a surface cleaning step. After oxidizing strip with HF or BHF solutions, rinse in DI water and dry.
REF: Yang, H T & Berry, W S — *J Vac Sci Technol,* B(2),206(1984)

SIO-0104
ETCH NAME: Potassium hydroxide
TYPE: Alkali, oxidation
COMPOSITION:
x .... 5—10% KOH
TIME: 5—10 min
TEMP: −10°C to RT
DISCUSSION:
SiO_2, grown as a hydrated oxide on silicon wafers as a surface cleaning step. Soak wafers in the solution shown below or up to RT, but not heated. After hydroxide treatment

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strip with HF or BHF, rinse in DI H_2O, then N_2 blow dry. This was part of a study of cleaning of silicon surfaces prior to device diffusion, alloying, and metallization.
REF: Walker, P et al — development, 1956—1968

SIO-0107
ETCH NAME: Plastic
TYPE: Organic, coating
COMPOSITION:
x .... plastic
TIME:
TEMP:
DISCUSSION:
SiO_2, drawn for fiber optics and laser applications. Fibers are coated with plastic as part of the drawing process to prevent structural damage from handling during further assembly.
REF: Watkins, L S — *Proc IEEE*, 70,626(1982)
SIO-0108: Pastor, A C — *J Cryst Growth*, 70,295(1984)
$NaNO_2$ grown as single crystal fibers. This initial development method can be applied to other materials.

WOP-0001
ETCH NAME: Hydrogen peroxide
TYPE: Acid, selective
COMPOSITION:
x .... H_2O_2
TIME:
TEMP:
DISCUSSION:
$W_2O_3(PO_4)_2$ as an amorphous glassy thin film. Material fabricated by mixing WO_3/H_3PO_4/100 ml H_2O, then drying 24 h at 110°C. Form melt in an electric furnace at 1200—1350°C using a platinum crucible for 2 h under an argon atmosphere. Fabricate thin films by pouring onto an aluminum plate at RT and quenching. Used in a study of glassy metallics.
REF: Kim, C U & Condrate, R A Jr — *J Phys Chem Solids*, 45,1213(1984)

LIST-0001
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
x .... HF
x .... HNO_3
TIME:
TEMP:
DISCUSSION:
$LiInS_2$, thin films on (111) silicon wafer substrates. Evaporated as both an amorphous glassy and a (100) single crystal thin film.
REF: Keriyama, K & Saitch, J — *J Solid Thin Films*, 111,331(1984)

GEO-0004
ETCH NAME: Ethyl alcohol
TYPE: Alcohol, cleaning
COMPOSITION:
x .... EOH
TIME:
TEMP: RT
DISCUSSION:
GeO glass. Powdered GeO was fired in an arc furnace from 1200 to 1600°C. Blanks cut from glass and polished with 400-grit SiC in petroleum lubricants. Wash in EOH and anneal at 230°C in vacuum 30 min.
REF: Magrunder, R H et al — *J Appl Phys*, 57,345(1985)

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PDNP-0001
ETCH NAME: Aqua regia — TIME:
TYPE: Acid, clean/removal — TEMP: RT
COMPOSITION:
3 .... HCl
1 .... HNO_3
DISCUSSION:
PdNiP, PtNiP, and PtCuP melt formed as metallic glasses in a materials study. As metastable single crystals: TiAl, TiNi, and ZrV. Fe_3C (cementite) with a high resistance of 1000 Ω at 25°C. Glass structure referred to as a dense random packing of spheres. Solution shown used as a general etch.
REF: Dumez, P — *J Vac Sci Technol,* B(1),218(1983)

GLA-0001
ETCH NAME: — TIME:
TYPE: Frequency, alteration — TEMP:
COMPOSITION:
x .... acoustics
DISCUSSION:
SiO_2 glass used in a study of the influence of frequency irradiation on OH^- concentration. Relaxation of the lattice structure may be frequency dependent? The Si-O-Si bond shows small change with lateral motion of oxygen, and the presence of OH^- radicals shows greater effect.
REF: Jager, R E — *J Am Ceram Soc,* 51,57(1968)

PHYSICAL PROPERTIES OF SILICON NITRIDE, Si_3N_4

Classification	Nitride
Atomic numbers	14 & 7
Atomic weight	140
Melting point (°C)	1900 (sublimes)
Boiling point (°C)	
Density (g/cm³)	3.18
Refractive index (n =)	2.00
Application limits (°C) in air	1400
inert atmosphere	1850
Specific heat (J/kg °C)	1050
Coefficient of linear thermal expansion ($\times 10^{-6}$ cm/cm/°C) 25—800°C — alpha/beta	2.9/2.3
Thermal conductivity (W/m °C) 1200°C	9.5
Electrical resistivity (ohms cm) 25°C	1013
480°C	1010
Dielectric constant (e =)	4—6
Hardness (Mohs — scratch)	6—7
Crystal structure (isometric — normal) alpha	(100) cube
(hexagonal — normal) beta	$(10\bar{1}0)$ prism
Color (solid)	Clear/greenish
Cleavage (imperfect)	(001)/(0001)
Fracture (solid)	Conchoidal

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SILICON NITRIDE, Si_3N_4

General: Does not occur as a natural compound. The atmosphere is approximately 75% nitrogen and 24% oxygen but, where oxygen is an active gas combining to form the silicate, oxide minerals, and many others, nitrogen is inert and does not form pure nitrides under normal atmospheric conditions. There are nitrates containing the NO_3^- radical which, as they are largely soluble in water, are found in dry, arid desert areas. Soda niter, $NaNO_3$ (Chile saltpeter) and niter, KNO_3 (saltpeter) are the two major minerals. There are important deposits in the dry, arid regions of Chile and western Bolivia, mainly as soda niter. Niter is common in the soil of Spain, Italy, Egypt, Arabia, Persia, and India. It is also found in the southwestern U.S. and with the Chilean soda niter deposits. They produce an alkaline earth that can be dissolved during rain, then redeposited under hot desert conditions. Such land areas also include the iodates and chlorides in the alkaline soil. Nitrate minerals are important as fertilizers as are the nitrites.

The nitridization process has been used in the heavy metals industry for many years. The metal or alloy part is thermally annealed at high temperature in a nitrogen gas atmosphere to convert the outer layer to a metallic nitride. Such surfaces have increased stability against atmospheric corrosion and show improved wear resistance.

The Rand Corporation developed the first silicon nitride for Solid State application, and Thompson-Ramo-Woolridge (TRW), Semiconductor Division developed the silicon oxy-nitrides in the early 1960s for application on semiconductor and similar devices. Silicon nitride is more stable and inert than silicon dioxide.

Technical Application: The pure silicon nitride, Si_3N_4, is difficult to etch, but forms an isomorphous series with silicon dioxide, SiO_2, such that the amount of oxygen in the compound can be varied from 0 to 100%. An oxynitride composition of $Si_3N_xO_y$ (5% O_2) has been found more compatible in wet chemical solution etch processing than the pure silicon nitride.

The compound has been deposited as an amorphous thin film by pyrolysis and CVD from gaseous sources; by DC or RF sputter from a solid Si_3N_4 target in argon or with a silicon bar in nitrogen. It also has been grown as a single crystal with a low temperature alpha phase (cubic) and a high temperature beta phase (hexagonal), the latter >1200°C. By laser annealing, the amorphous thin film can be converted to larger area crystallite structure. It also has been deposited as a hydrogenated colloidal structure, SiN:H, which may be as an oxynitride.

In Solid State processing the applications are similar to those of silicon dioxide: as a mask against diffusion, alloying, epitaxy, metallization, etching, and radiation. Also as a more stable passivation coating, as an active dielectric, such as for an MNOS diode or IC devices. It also has been used as an anti-reflective (AR) coating for solar cells, on glass masks, and lenses.

Silicon and germanium nitrides form an isomorphous series, and have been studied as various mixture combinations. Many other metal nitrides have been developed since the first silicon nitrides, and represent a major group of on-going experimental and application evaluations.

The combination of silicon oxide and nitride layers are used as either oxide/nitride or nitride/oxide. In the first case, the oxide acts as a pattern mask for etching the nitride; whereas in the second case, the oxide acts as an etch-stop mechanism. Both compounds have been doped with boron or phosphorus for special applications, to include use as dopant drive-in sources.

Etching: HF, BHF, hot H_3PO_4. $HF:HNO_3$, $HF:H_2O_2$. Dry chemical etching with ionized gases.

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SIN-0008a
ETCH NAME: BHF
TYPE: Acid, removal
TIME: 1—2 min
TEMP: RT
COMPOSITION:
50 ml .... HF
100 ml .... NH_4F, sat. sol.
100 ml .... H_2O
DISCUSSION:

Si_3N_4, thin film amorphous deposits, 1500—3000 Å thick, by DC sputter from a silicon bar in nitrogen. Wafer substrates were silicon, (111) n-type, 5—10 Ω cm resistivity used in an etch development study of silicon nitride and oxynitride in initial material development. The solution shown very slowly attacks pure silicon nitride, but rates are equivalent to silicon dioxide for silicon oxynitride (2—10% O_2). Photo resist evaluated as KMER with development of compatible etch solutions. The solution was used to thin nitrides, etch pattern photo resist devices, and for pinhole study.
REF: Walker, P & Hersch, N — initial development, 1962—1963
SIN-0022d: Ibid.

a-Si_3N_4:H thin films deposited by CVD on (100) silicon, n- or p-type. A BHF solution was used to etch pattern the hydrogenated nitride. Also used hot H_3PO_4 at 180°C, concentrated HF and dry chemical etching with CF_4/O_2. HF was most rapid.

SIN-0008b
ETCH NAME: BHF, modified
TYPE: Acid, removal
TIME: 1—10 min
TEMP: RT
COMPOSITION:
50 ml .... HF
50 g .... $NH_4F.HF$
100 ml .... H_2O
50 ml .... glycerin
DISCUSSION:

Si_3N_4, thin films. See discussion under SIN-0008a. Use of ammonium bifluoride as an unsaturated solution reduces crystallization of the saturated fluoride solution in solution due to evaporation during extended etching periods. The inclusion of glycerin produces a more uniform removal and a cleaner surface of both oxides and nitrides, and the solution shown is compatible with photo resist processing.
REF: Ibid.

SIN-0008c
ETCH NAME:
TYPE: Acid, removal
TIME: 10—20 min
TEMP: Boiling
RATE: 160 Å/min
COMPOSITION:
18 g .... NaOH
5 g .... $KHC_8H_4O_4$
100 ml .... H_2O
DISCUSSION:

Si and SiO_xN_y DC sputtered thin films on (111) silicon wafers, n-type, 5—10 Ω cm resistivity. Solution was very good on oxynitride (5% O_2). Rate can be reduced by using below boiling but is too slow for use at RT. It is compatible with photo resist but timing is critical as it will attack silicon preferentially at the boiling point.
REF: Ibid.

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SIN-0001
ETCH NAME: Phosphoric acid
TYPE: Acid, removal
COMPOSITION:
x .... H_3PO_4
DISCUSSION:

TIME:
TEMP: 80°C
RATE: 50 Å/min Si_3N_4
10 Å/min SiO_2

Si_3N_4, amorphous thin films deposited by CVD on silicon wafer substrates and on SiO_2 coated silicon substrates. Rates shown are as determined by author referenced who is the developer of this etchant for silicon nitride. Because of the etch rate disparity, SiO_2 can be used as an etch-stop and solution will not attack silicon.
REF: Kurtz, F et al — *J Electrochem Soc,* 113,1452(1966)
SIN-0008d: Ibid.

Si_3N_4 and $Si_3N_xO_{1-x}$, as amorphous thin films DC and RF sputtered on (111) silicon, n-type, 5—30 Ω cm resistivity wafers. Also applied PSG and BSG which showed an etch rate of about 25 Å/min. Work was in conjunction with KMER type photo resists.
SIN-0003: Williams, R K et al — *J Vac Sci Technol,* B(2),84(1984)

Si_3N_4, thin films deposited by both LPCVD and plasma CVD on silicon (100), p-type, 2—5 Ω cm resistivity substrates and then hydrogen irradiated to form: Si_3N_4:H thin films. Irradiation produced "swollen spots" in the nitride films that etched twice as fast as non-irradiated areas. (*Note:* The "swollen spots" are probably irradiation induced blisters.)
SIN-0022a: Fang, Y K et al — *J Electrochem Soc,* 132,1222(1985)

Si_3N_4:H, thin film doped with boron or phosphorus deposited on silicon, (100), n-type, 4—7 Ω cm resistivity substrates and used in a general etching study. H_3PO_4 was used at 80°C. Other etchants evaluated were HF, BHF, and CF_4/O_2(4%).

SIN-0004
ETCH NAME: Hydrofluoric acid, dilute
TYPE: Acid, removal
COMPOSITION:
1 .... HF
20 .... H_2O
DISCUSSION:

TIME:
TEMP: RT
RATE: 1000—2000 Å/min

Si_3N_4, thin films deposited on silicon substrates. This dilution of hydrofluoric acid can be used for controlled removal of thin films.
REF: Hersch, N — personal communication, 1965
SIN-0008f: Walker, P — personal application, 1965

Si_3N_4, thin films and silicon oxynitride thin films with varied oxygen content deposited by DC and RF sputter on n-type, 5—30 Ω cm silicon substrates in a general study of these thin films and their etching in conjunction with photo resist applications. Solution used as a dip etch for thinning and study of pinhole development.

SIN-0008e
ETCH NAME: Hydrofluoric acid
TYPE: Acid, pinholing
COMPOSITION:
x .... HF
DISCUSSION:

TIME:
TEMP: RT
RATE: 4000—5000 Å/min

Si_3N_4 and oxynitride thin films on silicon. Acid applied to thin surface with a wooden toothpick allowing surface tension to hold a droplet while etching a pit down through deposited nitride layer. Used to measure deposit thicknesses under standard microscope illumination using known surface color and recognizable color order steps in the etched side

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slope of etch-formed pit. This pitting method and a step-etch using HF hot vapors also were used in establishing a pure nitride color chart through third order red.
REF: Ibid.
SIN-0022c: Ibid.

a-Si_3N_4:H thin films grown by CVD as boron or phosphorus doped films on (100) silicon, n-type substrates. HF was the fastest removal solution of these evaluated. Also used H_3PO_4 at 180°C, BHF, and CF_4/O_2.

SIN-0008g
ETCH NAME: Hydrofluoric acid, vapor
TYPE: Acid, removal
COMPOSITION:
x .... HF, vapor
TIME: Dip
TEMP: Hot
DISCUSSION:

Si_3N_4 and oxynitride thin films on silicon. Hot hydrofluoric acid vapors and the pinholing technique (SIN-0008e) were used in establishing a color thickness chart based on a refractive index of n = 2.00 for Si_3N_4. A similar color chart for SiO_2 with n = 1.46 was made for comparative thickness values of these DC sputter deposited films (silicon bar in nitrogen).
REF: Ibid.

SIN-0005
ETCH NAME: Hydrofluoric acid, dilute
TYPE: Acid, removal
COMPOSITION:
1 .... HF
60 .... H_2O
TIME:
TEMP:
DISCUSSION:

Si_3N_4 thin films deposited by CVD on (100) silicon substrates from both N_2 and NH_3 sources. Both the solution shown and a 1:20 mixture were used in etch removal studies. Films grown in N_2 only were harder to etch remove.
REF: Chin, T Y et al — *J Electrochem Soc*, 13,2110(1984)

SIN-0006
ETCH NAME: BOE
TYPE: Acid, removal
COMPOSITION:
x .... HF
x .... NH_4F
x .... H_2O
TIME:
TEMP:
DISCUSSION:

Si_3N_4 thin films deposited by PECVD as a dielectric surface coating on ICs and solar cells. Solution used in studying the etch rates of deposits formed from SiH_4 + N_2 or NH_3. The latter gives a higher H^+ ion inclusion and a faster etch rate.
REF: Hess, D W — *J Vac Sci Technol*, A(2),243(1984)

SIN-0007
ETCH NAME:
TYPE: Acid, selective
COMPOSITION:
15 .... HF
10 .... H_3PO_4
60 .... EOH
TIME:
TEMP: RT
RATE: 100—150 Å/min

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DISCUSSION:

Si_3N_4, thin film amorphous deposits on silicon wafer substrates used for developing methods of etching patterns in silicon nitride. The solution shown is referenced to: Loic, H — U.S. Patent 3,867,28 (1975) with EOH replacing H_2O as shown in the patent. Use a drop of the etch solution on the specimen being spun at 2400 rpm on a photo resist type spinner. Solution applied at RT under a nitrogen atmosphere. Rinse in EOH or by bubbling HCl through EOH.

REF: Wurzbach, J A & Grunthaner, F J — *J Appl Phys*, 130,690(1983)

SIN-0008h

ETCH NAME:

TYPE: Acid, removal

COMPOSITION:

18 g .... NaOH
5 g .... $(NH_4)_2S_2O_8$
100 ml .... H_2O

TIME: 10—20 min
TEMP: Boiling
RATE: 160 Å/min

DISCUSSION:

Si_3N_4 and oxynitrides deposits on (111) silicon, n-type, 5—10 Ω cm resistivity wafers by DC sputter from a silicon bar in nitrogen. Solution developed for use with KMER photo resists in etch patterning devices. See comments under SIN-0008a.

REF: Ibid.

SIN-0008i

ETCH NAME: Sodium hydroxide

TYPE: Alkali, removal

COMPOSITION:

90 g .... NaOH
250 ml .... H_2O

TIME: 10—20 min
TEMP: Boiling
RATE: 180 Å/min

DISCUSSION:

Si_3N_4 and oxynitrides as DC sputtered thin film deposits on (111) silicon, n-type, 5—10 Ω cm resistivity wafers. Solution shown was developed for etching nitrides with KMER photo resists. Glycerin or ethylene glycol can be added for additional rate control. See SIN-0008a-h formats for additional development data.

REF: Ibid.

SIN-0008j

ETCH NAME:

TYPE: Halogen, removal

COMPOSITION:

"A" 5 g .... $NH_4F.HF$
50 ml .... H_2O

"B" 1 g .... *I_2
50 ml .... H_2O
50 ml .... glycerin

TIME: 30—45 min
TEMP: RT
RATE: 180 Å/min

*Pre-dissolve in 5 ml MeOH

Mix: 1 "A":1 "B" when ready for use.

DISCUSSION:

Si_3N_4 and oxynitrides grown as thin films by DC sputtering on (111) silicon wafers, n-type, 5—10 Ω cm resistivity using a silicon bar under nitrogen as silicon/nitrogen source. The solution can be used as a single solution, or combined as shown. This is an excellent solution for photo resist patterning as it can be re-used 4 h on 50 silicon wafers without

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degradation of solution or loss of P/R definition. Used with both KMER and KFER photo resists.
REF: Ibid.
SIN-0013a: Mann, J E & Walker, P — Electrochem Soc Meet, Dallas, Spring, 1967
$Si_3N_xO_y$ thin films. Reported development of silicon oxynitride as a surface protectant on silicon, to include etchants compatible with photo resist processing.

SIN-0014
ETCH NAME: BHF, modification
TYPE: Acid, removal
COMPOSITION:
15 ml .... HF
100 ml .... $NH_4F.HF$, sat. sol.
TIME:
TEMP: RT
RATE:
DISCUSSION:
Si_3N_4 and $Si_3N_xO_y$ thin films CVD grown from NH_3 with addition of NO_2 in increasing amounts to form oxynitrides. Pure SiO_2 also was grown, and in combination with the pure nitride as the two compounds are completely miscible forming an isomorphous series. The solution shown used in a general etching and properties study of both nitride and oxynitride to dioxide.
REF: Tombs, N C et al — *J Electrochem Soc*, 116,862(1969)

SIN-0015a
ETCH NAME: Glass cleaner
TYPE: Acid, cleaning
COMPOSITION:
35 ml .... $K_2Cr_2O_7$, sat. sol.
1000 ml .... H_2SO_4
TIME: Variable
TEMP: RT to hot
DISCUSSION:
Si_3N_4, SiO_2 and glass. Mixture shown is the standard glassware cleaning solution that has been in use for several years on glass and quartz. It can be used as a general surface cleaner on nitrides as well as oxides. If used hot, on a nitride surface, can convert some of the nitride to oxide. Solution used in a general study of etching nitrides.
REF: Walker, P — personal application, 1964

SIN-0015b
ETCH NAME: Aqua regia
TYPE: Acid, cleaning
COMPOSITION:
3 .... HCl
1 .... HNO_3
TIME: 1—5 min
TEMP: RT to boiling
DISCUSSION:
Si_3N_4, oxynitrides, SiO_2 as thin films or glass and quartzware. Aqua regia has long been a standard cleaner of glassware for removal of heavy metal contamination. After mixing, allow solution to sit in an open container until heavy bubble reaction diminishes, and solution turns a deep red/gold color. Never store in a closed container as reaction continues slowly and can cause explosive rupture due to pressure. Solution evaluated as a surface cleaner of thin film oxides and nitrides in a general etch development study.
REF: Ibid.

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SIN-0015c
ETCH NAME: Nitric acid
TYPE: Acid, cleaning
COMPOSITION:
x .... HNO_3
TIME: Variable
TEMP: RT to boiling
DISCUSSION:
Si_3N_4 and oxynitride thin films. Nitric acid has been used as a general glass cleaner with and without dilution for several years. If used hot on nitrides it can convert part of the surface to an oxide. Solution used in a general etching and cleaning study of nitrides.
REF: Ibid.

SIN-0015d
ETCH NAME: Chrome regia
TYPE: Acid, cleaning
COMPOSITION:
500 ml .... HCl
100 ml .... *10% CrO_3
TIME: 1—5 min
TEMP: RT
ASTM: #101

*13.4 g/1245 mil H_2O

DISCUSSION:
Si_3N_4, oxynitrides and SiO_2 thin film deposits by evaporation (CVD) or sputter (DC/RF). Solution can be used as a general oxide or nitride surface cleaning etch. With thin films on a silicon wafer surface it has also been used as a pinhole development solution as it attacks the silicon preferentially and enhances pinholes for microscopic observation. By etch back-thinning of the silicon wafer (HF:HNO_3 with nitride or oxide surface waxed with Apiezon W, black wax) pinholes can be observed in the nitride/oxide deposits by transmitted light.
REF: Ibid.
SIN-0027: ASTM E407-70
Reference for ASTM number of chrome regia.

SIN-0015e
ETCH NAME: Sodium hydroxide, dilute
TYPE: Alkali, cleaning
COMPOSITION:
1 g .... NaOH (KOH)
100 ml .... H_2O
TIME: 1—30 min
TEMP: RT
DISCUSSION:
Si_3N_4, oxynitrides and SiO_2 thin films. This is a general cleaning solution for glassware as the 10—15% concentration. The mixture shown was used on silicon oxynitride and dioxide thin film surfaces for light cleaning with only a cleaning effect on silicon wafer substrates. Using a 30% solution on a 2500 Å thick layer of silicon oxynitride (5% O_2), complete removal of the layer was done in about 2 h, at 80—90°C.
REF: Ibid.

SIN-0015f
ETCH NAME:
TYPE: Halogen, cleaning
COMPOSITION:
1 g .... I_2
500 ml .... MeOH
TIME: 30—90 sec
TEMP: RT

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DISCUSSION:

Si_3N_4 oxynitrides and SiO_2 DC/RF sputtered thin films on silicon wafers. Solution used as a general surface cleaner/conditioner on silicon wafers and both silicon oxides and nitrides prior to diffusion, epitaxy, metallization, or deposition. Soak in solution; DI water rinse, 10—15 sec; MeOH dip, 1—2 sec and either air dry or dry under an IR lamp. A small fraction of iodine will remain adsorbed on surfaces. Subsequent heating to above 100°C will allow iodine to complex ionic surface contamination with vaporization removal leaving a molecularly clean surface immediately prior to diffusion, epitaxy, etc.
REF: Ibid.

SIN-0021
ETCH NAME: Hydrofluoric acid, dilute
TYPE: Acid, removal
COMPOSITION:
1 .... HF
9 .... H_2O
TIME: Variable
TEMP: RT
DISCUSSION:

Si_3N_4 thin films. This is a general glassware cleaning solution that also has been used to clean thin film oxide and nitride surfaces as well as a native oxide removal solution of silicon wafers after etch polishing in HF:HNO_3 type solutions. Reference cited says that about 10 Å of oxide remains on a silicon surface etched in 1HF:5HNO_3.
REF: Wajda, E S et al — *IBM J*, 37,288(1961)

SIN-0025
ETCH NAME: Hydrofluoric acid
TYPE: Acid, removal
COMPOSITION:
x .... HF, conc.
TIME:
TEMP: RT
RATE: 75—100 Å/min
DISCUSSION:

Si_3N_4 deposited as pyrolytic thin films. Films used in a study of their preparation and properties.
REF: Doo, V Y et al — *J Electrochem Soc*, 113,1279(1966)

SIN-0009
ETCH NAME: BHF
TYPE: Acid, removal
COMPOSITION:
x .... BHF
TIME:
TEMP: RT
RATE: 4—6.1 Å/sec
DISCUSSION:

Si_3N_4, thin films, 100—1000 Å thick, grown by conversion of SiO_2 thermally grown on p-type silicon wafers, and with dry thermal oxidation at 900, 950, and 1000°C. Nitridization was done at 900 to 1100°C in N_2/NH_3 and AES measurements showed conversion of the films with N_2 pile-up at the dielectric/silicon interface. BHF mixture was not shown and rate variation depended upon thickness of the thin film, increasing with film thickness.
REF: Yoriume, Y — *J Vac Sci Technol*, B(1), 67(1983)

SIN-0023
ETCH NAME: BHF
TYPE: Acid, removal
COMPOSITION:
1 .... HF
10 .... NH_4F, sat. sol.
TIME:
TEMP: RT

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DISCUSSION:

N_xSiO_2 thin films. SiO_2 films 160 nm thick were deposited on silicon wafer substrates and then nitrided with N_2 or NH_3. Structure developed was termed: nitroxide as against a grown oxy-nitride. The SiO_2 films were nitrided at 1200°C for 5 and 17 h. SiO_2 etch rate was about 16 nm/sec; both nitroxides showed no initial etching for about 1.5 or 2.5 min for the 5 and 17 h growth periods, respectively, then etched at an approximate rate of 5—6 nm/sec. Refractive index varied from 1.46 (SiO_2) to about 1.6 (5 h nitroxide) to almost 1.7 (17 h nitroxide). Dielectric constant: e = 4.0 (SiO_2), stabilized at about 5.5 after 5 h nitroxide. AES depth profiles, C-V and VFB shown. Variation in refractive index of SiO_2 after N_2 and NH_3, and thickness vs. kV voltage applied shown.

REF: Kato, L et al — *J Electron Mater*, 13,913(1984)

SIN-0024: Ito, T et al — *J Electrochem Soc*, 127,2053(1978)

N_xSiO_2, thin films of SiO_2 nitrided at 900 to 1200°C in NH_3.

SIN-0022b

ETCH NAME:

TYPE: Ionized gas, removal

COMPOSITION:

x .... 96% CF_4

x .... 4% O_2

TIME:

TEMP:

GAS FLOW:

PRESSURE:

POWER:

DISCUSSION:

a-Si_3N_4:H thin films with different doping concentrations of B and P. Minimum etch rates with 2% B and 3% P doping increasing with higher concentrations. Doped films have a higher resistivity than undoped films: maximum resistivity ($\times 20^{10}$ Ω cm) — 6 for 3% P doped and 5% B doped. Breakdown strength (10^5 V/cm) — 9 for P doped; 8 for B doped. Pinhole density was obtained by copper decoration.

REF: Ibid.

SIN-0026: Shannon, W J — *RCA Rev*, 31,431(1970)

Reference for copper decoration technique used in SIN-0022b

SIN-0029: Kotani, H et al — *J Electrochem Soc*, 130,645(1983)

SiN, p-type thin films used as interconnects on semiconductor devices, along with AlSi, PSG, and AlSiCu thin films. Describes using CF_4/O_2 for multi-level etching. Used argon at 1.7 Pa pressure and 0.5 W/cm² power for dry chemical etching. p-SiN etch rate: 70 Å/min; PSG etch rate: 60 Å/min and AlSi etch at greater than 200°C temperature was 100 Å/min.

SIN-0030: Chayahara, A et al — *Jpn J Appl Phys*, 24,19(1983)

a-Si_3N_4:H thin films deposited in a study of the material development. Nitrogen can show three- or fourfold symmetry. Si:N formed using SiH_4:NH_3:H_2 in a microwave glow discharge with 250 W power and 1 Torr pressure; gas flows: SiH_4/H_2(10%) at 100—200 sccm; NH_3/N_2 at 0.5—5 SCCM.

SIN-0027

ETCH NAME: BHF

TYPE: Acid, stop etch

COMPOSITION:

1 .... HF

9 .... NH_4F(40%)

TIME: 10 sec

TEMP: RT

RATE: 0.01—0.02 μm/sec

DISCUSSION:

Si_3N_4, thin films RF plasma grown on silicon, (100), p-type, 2 Ω cm resistivity wafers with SiO_2 cap. Solution used as a stop-etch in removal of SiO_2 and then as a step-etch of the nitride. Sitting after several days the nitride rate increased from 0.02 to 0.03 μm/sec.

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SiO_2 etch rate changed from 0.01 to 2 μm/sec. Refractive index of pure SiO_2 shown as n = 1.462; for the pure nitride n = 2.00. Prior to nitride deposition wafers were cleaned: (1) H_2O_2 soak and DI water rinse; (2) H_2SO_4:H_2O_2 and DI water rinse; (3) HF dip and DI water rinse, and (4) BHF, RT, 60 sec.
REF: Reisman, A et al — *J Electron Mater*, 13,504(1984)

SIN-0028a
ETCH NAME: Carbon tetrafluoride
TYPE: Ionized gas, removal
COMPOSITION:
x .... CF_4

TIME:
TEMP:
GAS FLOW: 16 sccm
PRESSURE: 0.14 Torr
POWER:

DISCUSSION:
SiN_x and SiO_2 thin films deposited by RF plasma on silicon doped GaAs, (100) wafers. Photo resist windows were opened with BHF (1HF:5NH_4F), or by RIE.
REF: Blaauw, C et al — *J Electron Mater*, 13,251(1984)

SIN-0028b
ETCH NAME: BHF
TYPE: Acid, removal
COMPOSITION:
1 .... HF
5 .... NH_4F(40%)

TIME:
TEMP:

DISCUSSION:
SiN_x and SiO_2 thin films RF plasma and CVD deposited on GaAs, (100) silicon doped substrates. BHF used to remove nitride and oxide from photo resist developed windows. Also used RIE etching with CF_4.
REF: Ibid.

SIN-0010
ETCH NAME: BHF
TYPE: Acid, cleaning
COMPOSITION:
5 ml .... HF
20 g .... NH_4F
30 ml .... H_2O

TIME:
TEMP: Hot

DISCUSSION:
Si_3N_4, thin films deposited on (100) silicon wafers used in a study of nitride films. Pre-clean substrates in BHF — heating solution reduces H_2 density in film growth. Follow with 400°C bake-out prior to nitriding.
REF: Zhous, M S et al — *J Electron Mater*, 14,55(1985)

SIN-0031
ETCH NAME: Phosphoric acid
TYPE: Acid, removal/defect
COMPOSITION:
x .... H_3PO_4, vapor

TIME:
TEMP: Hot

DISCUSSION:
a-SiN:H thin films deposited on (100) silicon and germanium wafers, glass cover slides and fused silica blanks. Clean all substrates: (1) TCE degrease; (2) HF dip and (3) DI H_2O rinse. Deposit films by RF sputter of a silicon target in a gas mixture of air:N_2:H_2 at 175°C.

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Hot phosphoric acid vapors can be used for very slow removal and defect development in films. See SIN-0001.
REF: Martin, P M — *J Vac Sci Technol,* A2(2),330(1984)

SIN-0032
ETCH NAME: Hematite TIME:
TYPE: Compound, polish TEMP: RT
COMPOSITION:
(1) x .... Fe_2O_3, powder (2) x .... CH_3COCH_3 (acetone)
DISCUSSION:

Si_3N_4, as pressed powder blanks used in a study of Cr and Ti bonding. Nitride blanks were mechanically polished with red rouge, Fe_2O_3. Blanks were then annealed in a belt furnace for 2 h at 1000°C under N_2, and then oxidized for a structured layer of $SiO_2/SiNO_x/Si_3N_4$/blank . . . surfaces were rough, crystalline. Surfaces cleaned ultrasonically in acetone before metallization. Air anneal showed best adhesion for chromium, but poor for nitride. Presence of carbon produces some TiC with titanium.
REF: Orent, T W et al — *J Vac Sci Technol,* B1(3),844(1983)

SILICON VANADINIDE ETCHANTS

SIV-0001
ETCH NAME: TIME:
TYPE: Acid, removal/polish TEMP: RT
COMPOSITION:
x .... HF
x .... HNO_3
DISCUSSION:

SiV_2, thin films 500 Å thick deposited on (100) NaCl substrates. Follow with 300 Å a-Si — 1000 Å Ni — 500 Å SiO_2 cap layer. The thin film layered structure was water float-off removed by liquefying the NaCl/SiV_2 interface for TEM study. Solution shown is a general etchant for both silicon and vanadium.
REF: Chen, S H et al — *J Appl Phys,* 57,258(1985)

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PHYSICAL PROPERTIES OF SILVER, Ag

Classification	Transition metal
Atomic number	47
Atomic weight	107.87
Melting point (°C)	960.8
Boiling point (°C)	2210 (2212)
Density (g/cm^3)	10.5
Thermal conductance (cal/sec)(cm^2)(°C/cm) 0°C	0.999
Specific heat (cal/g) 25°C	0.056
Latent heat of fusion (cal/g)	25
Heat of vaporization (cal/g)	565
Atomic volume (W/D)	10.3
1st ionization energy (K-cal/g-mole)	17.5
1st ionization potential (eV)	7.574
Electron work function (eV)	4.52
Electronegativity (Pauling's)	1.9
Covalent radius (angstroms)	1.34
Ionic radius (angstroms)	1.26
Coefficient of linear thermal expansion ($\times 10^{-6}$ cm/cm/°C)	19.68
Electrical resistivity (micro ohms cm) 20°C	108.4
($\times 10^{-6}$ ohm cm) 20°C	1.59
Lattice constant (angstroms)	4.086
Thermionic work function (eV)	3.09—4.31
Elastic modulus (psi $\times 10^6$) 30°C	10.6
Tensile strength (psi — annealed)	25.000
Cross section (barns)	63
Vapor pressure (°C)	1865
Hardness (Mohs — scratch)	2.5—3
(Knoop — kgf mm^{-2})	60—90
Crystal structure (isometric — normal)	(100) cube, fcc
Color (solid)	Silver-white
Cleavage (imperfect)	(001)

SILVER CONTAINING ETCHANTS

Silver is classified in the Copper Group I-B of copper, silver and gold, in increasing order of atomic weight and density. They are the leading elements for heat and electrical conductivity with copper the most widely used due to its greater abundance. They also are known as the coinage metals, which now include nickel, and all have been mediums of exchange since earliest times. They are heavy metals due to their atomic structure, and are both malleable and ductile.

Though silver does not readily oxidize in air, surfaces are rapidly attacked by sulfur and sulfur compounds "tarnishing" as a black smut silver sulfide which can be easily removed with ammonia, NH_3 or ammonium hydroxide, NH_4OH.

In metal and metallic compound etching or cleaning solutions the most widely used silver compound is silver nitrate, $Ag(NO_3)_2$ as it is a highly active oxidizing agent and very

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soluble in water. Silver chloride, AgCl, on the other hand, is practically insoluble in water, but forms soluble complexes with the photographer's Hypo — sodium thiosulfate, $Na_2S_2O_3$ — which, when properly developed and "fixed" produces a black film of silver, and becomes a permanent black-and-clear photographic negative. The chloride, as a smoke, is also used as a water droplet nucleating agent in rain clouds and, in the laboratory, in the growth of snowflakes and single crystal ice crystals for scientific study.

As will be seen in the following list of solutions containing silver, the majority are used as preferential etchants to develop surface structure etch figures, some polishing solutions and as plating solutions to develop electrically active p-n junctions, such as in semiconductor device studies. Though other silver compounds are used, the majority are silver nitrate. Silver nitrate could be used in many other solutions on materials not shown in the following list, but copper nitrate, $Cu(NO_3)_2$ and several chromium compounds are more widely used as they too are extremely active oxidizing agents, and are lower in price. The following is a selected list from the Etchant Section:

Formula	Material	Use	Ref.
10 ml E etch:5 mg AgNO	CdTe	Preferential	CDTE-0006b
50 ml HNO_3:0.5 ml $AgNO_3$:50 ml H_2O	Cu	Preferential, macro-etch	CU-0011
1HNO_3:3H_2O + 1% $AgNO_3$	GaAs	Preferential	GAS-0045c
2HF:3HN(O_3:5H_2O: + $AgNO_3$ RC-1	GaAs	Dislocation	GAS-0048
37HF:13HNO_3:50H_2O:2$AgNO_3$	Ge	Preferential	GE-0037
4 ml HF:2 ml HNO_3:4 ml H_2O: 0.2 g $AgNO_3$ WAg Etch	Ge	Preferential	GE-0060c
x$AgNO_3$	Ge	Preferential (sphere)	GE-0195
40HF:20HNO_3:40H_2O:2$AgNO_3$	Ge	Preferential (sphere)	GE-0196
xAgI_2, vapor	Ice	Initiate growth	ICE-0007
1 ml HF:2 ml H_2O:8 mg $AgNO_3$: 1 g CrO_3 A/B Etch	InAs	Preferential	INAS-0005d
1 ml HF:2 ml H_2O:3 g CrO_3:8 mg $AgNO_3$	InP	Preferential	INP-0051
Silver glycol etch: A: 10 ml HF:400 ml HNO_3: 1 g $AgNO_3$:100 ml H_2O B: 200 ml H_2O:200 ml propylene glycol	Si	Preferential	SI-0041a
1HF:1HNO_3:x1% $AgNO_3$	Si	Polish	SI-0081
4 ml HF:2 ml HNO_3:4 ml H_2O: 200 mg $AgNO_3$ silver etch	Si	Dislocation	SI-0209
x$AgCN$	Ag	Electrolytic, polish	AG-0010
1AgF:10H_2O: + HF	Si	Junction, plate-out	SI-0280

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SILVER, Ag

General: Occurs in nature as a native element. It is found in fine single crystals but is more common as acicular, reticulated or aborescent shapes; also massive, in thin plates, grains and small scales. Like several native metal elements, it has no cleavage, breaking with a hackly fracture and is both ductile and malleable like native gold, iron and lead. Fresh surfaces show typical silver-white color but are often tarnished gray to black after reaction with air or sulfur compounds. It is widely distributed throughout the world, though not as common as gold and is usually found in the upper portion of silver bearing veins as an alternation product of underlying silver sulfide, arsenide and chloride minerals — the gangue mineral is cellular, rusty quartz. Native silver contains up to 10% gold and native "common" gold, up to 15% Ag.

Silver, gold and platinum are the three primary "precious metals", silver and gold being two of the seven metals known to ancient man. It has been used as a medium of monetary exchange since earliest times, and in the fabrication of jewelry and objets d'art. Present silver value runs between $6.00 to $8.00, against $350 to $450 for gold and $500 to $550 for platinum.

Though silver is the best electrical conductor, copper is more widely used due to its greater abundance. This is not to say that silver is not used for its electrical and heat dissipation characteristics: pins, pre-forms, wire and ribbon straps and bulk heat-sinks are used. There are many low temperature silver alloy solders and high temperature brazing alloys (1100 to 1600°F), the latter excellent for joining carbon steels, copper, brass, nickel, monel and several nickel/silver alloys. Solder alloy #63 (Pb:Sn + 2% Ag), m.p. 220°C, is widely used in the electronics industry.

Silver has wide use as an electroplated thin film on surfaces — metals, plastics, mylar, glass — though it was the original "silver mirror", both aluminum and nickel are now used to reduce cost.

Technical Application: Pure silver parts, such as electrical pins, and small pre-forms are used in many device assembly operations, as well as wire and ribbon as contact straps for electrical conduction. Pure silver is plated on many parts and evaporated in layered metallization thin film structures, such as Ag/Pd — Au/Ag — Ti/TiAg/Ag — Cu/Ag/Cu and others for structuring device contact pads and circuits. As wire it is used with wire bonders. There are a wide range of silver alloys used in device assemblies to include both low temperature solders and high temperature brazing alloys. Solder #63 has already been mentioned, and one of the widely used contact pastes is Ag-epoxy or a silver loaded polyimide that can be easily heat cured between 120 to 150°C with various time schedules. Large silver alloy base plates have been used in vacuum and epitaxy systems for heat transfer, though SST and copper are more widely used due to cost factors.

Electroplated and evaporated silver thin films have been studied for removal, defects, structure, photo doping and both natural and artificial single crystals have been studied for physical and morphological data to include single crystal spheres in oxidation experiments.

Etching: HNO_3, hot H_2SO_4 and cyanides. Mixed acids — aqua regia types — $HF:HNO_3$ and thermal etching. NH_3 and NH_4OH, tarnish removal.

SILVER ETCHANTS

AG-0001
ETCH NAME: Nitric acid
TYPE: Acid, preferential
COMPOSITION:
x .... HNO_3, conc.

TIME:
TEMP: 0°C to boiling

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DISCUSSION:

Ag, (111) wafers and other orientations as single crystal blanks. Solution used at RT to develop orientation etch figures.

REF: Farnsworth, H E — *Phys Rev*, 40,684(1932)

AG-0003: Dana, E S & Ford, W E — *A Textbook of Mineralogy*, 4th ed, John Wiley & Sons, New York, 1950, 403

Silver, natural crystals. Solution used to dissolve silver in pyrolytic and chemical study. Can be easily plated out of solution on a copper plate from nitric acid:water solutions in which silver is dissolved.

AG-0023: Walker, P — personal application, mineralogy, 1952

Ag, native single crystals. Use solution at 0°C, chilled in a water bath with chipped ice, as a momentary dip to clean surfaces and, by repeated dipping to develop orientation figures and defect structure.

AG-0002a

ETCH NAME: Nitric acid, dilute

TIME: 10—15 sec

TYPE: Acid, removal

TEMP: RT and 8°C

COMPOSITION:

1—8 .... HNO_3

1 .... H_2O

DISCUSSION:

Ag, thin films electroplated on brass. Used a 1:1 solution to strip silver from brass parts. Use of a rapid dip-water flush method as solution will attack brass. With sufficient solution volume to prevent heating, or, by chilling to 8°C with a bath of ice/acetone, minimum brass attack can be obtained. Remaining black smut on brass can be removed by rubbing with toweling soaked in water.

REF: Walker, P — personal application, 1968

AG-0002b

ETCH NAME: Potassium cyanide

TIME: 1—5 min

TYPE: Acid, cleaning

TEMP: RT

COMPOSITION:

10 g .... KCN/NaCN

100 ml .... H_2O

DISCUSSION:

Ag, as high purity pre-forms and silver alloy pre-forms used in silicon device assembly and on assembly pins of silver plated dumet or copper. Solution used to clean parts, and followed with heavy DI water washing. Clean parts immediately before alloy assembly.

REF: Ibid.

AG-0005

ETCH NAME:

TIME: 1—10 min

TYPE: Acid, removal/cleaning

TEMP: RT

COMPOSITION:

1 .... NH_4OH

1 .... H_2O_2

DISCUSSION:

Ag, thin film deposits. Described as a slow removal etch for silver. Will not attack silicon but, with presence of hydrogen peroxide, will attack gallium arsenide.

REF: MRC Metals Catalog, 1980

AG-0002c: Ibid.

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Ag, thin films electroplated on brass. Solution used to strip silver. Also used in etching of evaporated silver on (111) silicon wafers after photo resist patterning.
AG-0021: Nichols, D R — *Solid State Technol,* December 1979

Ag, thin film deposits in vacuum systems. Solution used to etch clean bell jars and internal fixtures. Can be used on steels, glass, ceramics and copper.

AG-0002d
ETCH NAME: Ammonium hydroxide, dilute
TYPE: Acid, cleaning
TIME: 1—10 min
TEMP: RT
COMPOSITION:
1 .... NH_4OH
1 .... H_2O
DISCUSSION:

Ag, high purity pre-forms and plated parts. Solution used to clean silver surfaces prior to alloy or assembly of parts in silicon device processing. (*Note:* Solution is easier and safer to use than the cyanides.)
REF: Ibid.

AG-0007
ETCH NAME:
TYPE: Acid, removal
TIME: 1—5 min
TEMP: RT
COMPOSITION:
1 .... HCl
1 .... HNO_3
1 .... H_2O
DISCUSSION:

Ag, 100 Å thick thin film of silver deposited on an a-SeGe glassy films. This layered structure was then annealed under a 200 W Hg UV lamp in a study of photo doping. Remaining residual silver after annealing was removed with the solution shown.
REF: Zebutsu, S — *Appl Phys Lett,* 39,969(1981)

AG-0008
ETCH NAME:
TYPE: Acid, polish
TIME: 3—5 sec
TEMP: RT
COMPOSITION:

(1) 1 .... *NaCN
1 .... H_2O_2 (30%)

(2) 1 .... *NaCN
1 .... H_2O_2 (20%)

*21.5 g/l

DISCUSSION:

Ag, (111) and (100) wafers used in a study of surface defects. Use solution (1) for (111) and (2) for (100) orientations. Etch 5 sec — rinse: then 3 sec — rinse. Rinse in (1) 37.5 g/l NaCN and (2) heavy water wash. Repeat this step-polishing and rinsing until surfaces are highly reflective, defect free and will hold a drop of pure water on the surfaces. Follow by Ar^+ ion sputter in UHV and 600°C anneal. (*Note:* 30% H_2O_2 is standard concentration.)
REF: Adzic, R R et al — *J Electrochem Soc,* 131,1730(1984)

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AG-0009a
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
x .... KCN

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

DISCUSSION:

Ag specimens used in a thermal etching study. Electropolish specimens in solution shown prior to thermal processing.
REF: Hondros, E D & Moore, A J W — *Acta Metall,* 8,751(1960)
AG-0004a: Hendrickson, A A & Machin, E S — *Acta Metall,* 3,69(1955)

Ag, (111) and (100) wafers used in a study of dislocations developed by thermal etching. Electropolish with 9% KCN at 125 A/in^2 power with rapid agitation. Wash 5 min in cold water, then alcohol rinse and blow dry with an argon gas jet. Thermal etch in 1 O_2:9 Ar for three time and temperature periods: (1) 600°C, 10 h; (2) 850°C, 10 h; and (3) 550°C, 16 h.

AG-0009b
ETCH NAME: Air/N_2/Vacuum
TYPE: Gas, preferential
COMPOSITION:
x .... air, N_2, or vacuum

TIME: 150 h
TEMP: 73°C
POWER: 31 A/cm^2 &
0.19 V

DISCUSSION:

Ag, specimens used in a thermal etching study. Acid saw cut wafers to minimize strain damage and electropolish (AG-0009a), before preferential etching. Results: (1) in air, etch patterns developed and reversed when power polarity was reversed; (2) in nitrogen, little or no structure developed and (3) in vacuum, large pits appeared in 5 h, and were larger and deeper at center (hottest area?).
REF: Ibid.

AG-0013
ETCH NAME: Air
TYPE: Gas, preferential
COMPOSITION:
x .... O_2

TIME:
TEMP:

DISCUSSION:

Ag, single crystal sphere used in a study of thermal oxidation of surfaces. Thickness of Ag_2O varies with crystallographic pole and great circle orientations of the sphere.
REF: Menzel, E & Menzel-Kopp, E — *Z Naturforsch,* 132,985(1958) (in German)

AG-0014
ETCH NAME: Heat
TYPE: Thermal, forming
COMPOSITION:
x .... heat

TIME:
TEMP:

DISCUSSION:

Ag, single crystal sphere formed from a single crystal wire. Wire was 1—2 mm diameter; spheres formed were 2—3 mm diameter. Gold and copper spheres also formed. The spheres were used for electrodes in electrocrystallization studies.
REF: Roe, D K & Gerisder, H — *J Electrochem Soc,* 110,310(1963)

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AG-0015b
ETCH NAME: Air
TYPE: Gas, oxidation
COMPOSITION:
(1) x .... air
(2) x .... HNO_3
x .... H_2O
TIME: 1 week
TEMP: 900°C

DISCUSSION:
Ag specimens. Oxidizing surfaces in (1) at time and temperature shown will both oxidize and thermally etch specimens. After treatment (1) remove oxide with acid solution shown in (2). Specimens were used in a study of the influence of surface structure on the tarnish reaction.
REF: Allpress, J C & Sanders, J V — *Phil Mag*, 10,827(1964)

AG-0010
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
x .... AgCN
TIME:
TEMP:
ANODE:
CATHODE:
POWER:

DISCUSSION:
Ag, single crystal specimens used in a study of thermal etching influence on surface energy. Electropolish material in solution shown prior to thermal treatment.
REF: Moore, A J W — *Acta Metall*, 6,292(1958)
AG-0011: Gilbertson, L & Fortner, O F — *Trans Electrochem Soc*, 81,199(1942)
Reference cited for electropolish solution shown in AG-0010.

AG-0012a
ETCH NAME:
TYPE: Electrolytic, saw
COMPOSITION:
35 g .... AgCN
37 g .... KCN
38 g .... K_2CO_3
1000 ml .... H_2O
TIME: Hours
TEMP: RT
ANODE:
CATHODE:
POWER:

DISCUSSION:
Ag, (001) wafers electrolytically cut with an acid-saw system using a cotton rug fiber as the cutting tool. Cutting time was 1″/120 h. Specimens were then hand-polished and dislocation etched in studying the material purity, dislocation density and strength.
REF: Hammar, R H et al — *Trans Met Soc AIME*, 239,1692(1967)

AG-0012b
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
(1) 15 ml .... H_2O_2
25 ml .... NH_4OH
(2) 0.5 ml .... HCl
5 g .... CrO_3
50 ml .... H_2O
TIME:
TEMP: RT

DISCUSSION:
Ag, (001) wafers. After acid-saw cutting (AG-0012a) specimens were alternately hand etch-polished in solution (1) and chemically polished in solution (2) until the desired surface finish was obtained.
REF: Ibid.

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AG-0012c
ETCH NAME:
TYPE: Acid, dislocation
COMPOSITION:
"A" 1 .... NH_4OH
1 .... H_2O_2
"B" 0.01 ml .... HCl
5 g .... CrO_3
25 ml .... H_2O
Mix: 1 part "A" to 0.16 parts "B".
DISCUSSION:
Ag, (001) wafers. Solution used to develop dislocations.
REF: Ibid.
TIME:
TEMP: RT

AG-0016
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
40 g .... Cr_2O_3
20 ml .... H_2SO_4
1000 ml .... H_2O
DISCUSSION:
Ag specimens used in a study of the reactions with this solution type mixture. After etching wash away red-brown coating that remains with water.
REF: Kurz, F — *Acta Metall,* 103,257(1956)
TIME: 1—2 min
TEMP: RT

AG-0017
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
67.5 g .... KCN
15 g .... KFe_2CN
15 g .... Rochelle salt
19.5 g .... H_3PO_4
2.5 g .... NH_3
DISCUSSION:
Ag specimens. Solution used to electropolish both gold and silver.
REF: Ruff, A W & Ives, L K — *Acta Metall,* 15,189(1967)
TIME:
TEMP:
ANODE: Ag
CATHODE: SS
POWER: 4 V

AG-0019
ETCH NAME:
TYPE: Acid, preferential
COMPOSITION:
x .... 5% KCN
x .... 5% $(NH_4)_2S_2O_8$
DISCUSSION:
Ag, single crystals used in a study of recrystallization by bending. Solution used as a micro-etch to develop structure.
REF: Semmel, J W Jr & Machin, E S — *Acta Metall,* 5,582(1957)
AG-0004b: Hendrickson, A A & Machin, E S — *Acta Metall,* 3,68(1955)
Ag, single crystal specimens. Solution used to develop structure.
TIME:
TEMP:

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AG-0022a
ETCH NAME: Tri-iodide
TYPE: Halogen, removal
COMPOSITION:
(1) 400 g .... KI
100 g .... I_2
400 ml .... H_2O
(2) 1 .... Tri-iodide
4 .... H_2O
TIME:
TEMP:
DISCUSSION:
Ag thin films. The solutions shown or other Tri-iodide composition etchants can be used to remove or pattern both silver and gold thin films.
REF: Brochure — *Application Data for Kodak Photosensitive Resists*, Eastman Kodak Co., Rochester, NY, 1960, 91

AG-0022b
ETCH NAME:
TYPE: Acid, removal (spray)
COMPOSITION:
35 g .... $Fe(NO_3)_2$
100 ml .... ethylene glycol
25 ml .... H_2O
TIME:
TEMP:
RATE: 3000 Å/min
DISCUSSION:
Ag thin films. Solution shown as a removal and patterning etchant. Removal rate is for application as a spray.
REF: Ibid.

AG-0022c
ETCH NAME: Ferric nitrate
TYPE: Acid, removal
COMPOSITION:
x .... 55% $Fe(NO_3)_2.6H_2O$
TIME:
TEMP: 43—49°C
DISCUSSION:
Ag thin films. Solution shown as a removal and patterning etchant compatible with Kodak photo resists.
REF: Ibid.

AG-0025a
ETCH NAME:
TYPE: Acid, tarnishing
COMPOSITION:
(1) x .... HNO_3, conc.
+ .... dry air
(2) x .... HNO_3
x .... H_2O
(3) x .... NH_3
+ .... dry air
+ .... dry air
TIME: 5 & 10 min
TEMP: RT
DISCUSSION:
Ag specimen blanks 0.5 × 0.5 × 0.005 used in a study of silver tarnishing. All three solutions shown evaluated at the two times shown. Results showed a white matte surface. (*Note:* Sulfur compounds produce the normally observed grey to black tarnish. Some silver oxides will tarnish as white coating.)
REF: Reagor, B T & Sinclair, J D — *J Electrochem Soc*, 128,701(1981)

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AG-0025b
ETCH NAME: Ammonia — TIME: 5 & 10 min
TYPE: Acid, tarnishing — TEMP: RT
COMPOSITION:
x .... NH_3
+ .... dry air
DISCUSSION:
Ag specimen blanks used in a silver tarnish study. See AG-0025a for discussion.
REF: Ibid.

AG-0024
ETCH NAME: "Smog" — TIME:
TYPE: Gas, corrosion — TEMP:
COMPOSITION:
x .... Air (+ trace SO_2, H_2S, O_3, HCl and Cl_2, vapor)
+ .... dry air
DISCUSSION:
Ag specimens used in a corrosion study of surfaces in a dry "smog" type environment. Sulfur is the primary attacking element in the tarnishing of silver. Copper also was evaluated with addition of water vapor as controlled humidity (RH) atmosphere.
REF: Rice, D W et al — *J Electrochem Soc,* 128,275(1981)

AG-0026
ETCH NAME: Nitric acid — TIME:
TYPE: Acid, removal — TEMP:
COMPOSITION:
x .... 1 *M* HNO_3
+ .... light, collimated
DISCUSSION:
Ag thin film coatings on Si, Al_2O_3 and ZrO_2 substrates used in a study of surface coatings to withstand chemical attack. Silver is stable in air and water, but tarnishes in sulfur containing atmospheres. Also worked with Al, TiN, alpha-Si:H and PbS. Solution plus light used to remove silver plating.
REF: Martin, P J et al — *J Vac Sci Technol,* A2(2),341(1984)

SILVER ALLOYS, AgMx

General: Native silver occurs as an element in nature, but rarely as pure silver such as 99.99+%, for it contains trace elements of other metals such as copper, and sometimes platinum, antimony, bismuth, and mercury; the latter as the mineral amalgam, AgHg with varying amounts of both elements. The mineral called electrum (argeniferous gold), AuAg . . . Ag 18—36% . . . light yellow to yellow-white in color. Other natural silver alloys include dyscrasite, Ag_3Sb; stutzite, Ag_4Te; hessite, Ag_2Te, and there are other trinary tellurides and selenides. There are distinct mineral species of silver halogens, such as AgI, AgBr, and AgCl, and over 50 silver minerals. Argentite, Ag_2S, is a major ore found in veins associated with pure silver, other silver minerals, and/or copper and lead mines. Some pyrites, Fe_2S — a major ore of iron — are sometimes more valuable for their gold and silver content than for iron and sulfur.

Silver and gold are the two main coinage metals and, along with copper, were the three major metals of trade in ancient times, and there have been coins of silver and gold, as well as bronze, iron and stone in the past. The lead tin alloy #63 contains 2% silver as a low temperature solder; the silvaloys, BAg, are high temperature brazing alloys. The silver

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halides AgCl and AgBr, both natural and artificial materials, are used in photography, as the compounds turn black on exposure to light, and also are used as medical antiseptics. Silver alloys have wide use in the jewelry trade, as well as for tableware — knives, forks, spoons, cups, and plates. During the Spanish exploration of the Americas, the returning ships were known as the "Silver Plate" galleons, and silver is electrolytically plated as Ag, Ag/Pb, Ag/Ni, Ag/Cu, etc., for both decorative purposes and surface protection as silver plate is stable in air and water, though it will tarnish in the presence of sulfur to black Ag_2S (remove with ammonia solutions). The nitrate $AgNO_3$ is known as Lunar Caustic, and has medical applications, as well as being a primary etching constituent in many etches as already shown.

Technical Applications: Silver alloys have several applications in the Solid State industry other than as solder and brazing alloys. It is one thin film constituent in multi-layer metallization, such as for Ag/TiAg/Ti or Ag/Cu. As bulk parts: evaporator electrodes or vertical epitaxy system base plates, and used for electrical wiring, as is the best known conductor of electricity. Many small parts are silver alloy plated for electronic assemblies — wires, pins, plates and packages.

There are a number of single crystal metallic compound semiconductors, both binary and trinary systems, containing silver as tellurides, selenides and sulfides . . . these are under their separate sections in this book. Several alloys have been studied as both mixtures and single crystals, presented in this silver alloy section.

Etching: NH_3, NH_4OH, KCN and mixed acids. Varies with specific alloy.

SILVER ALLOY

Silver aluminum
Silver boron
Silver lead tin
Silver mercury
Silver tin
Silver titanium
Silver zinc

SILVER ALLOYS

SILVER ALUMINUM ETCHANTS

AGAL-0001
ETCH NAME: Nitric acid, dilute
TYPE: Acid, polish
COMPOSITION:
2 .... HNO_3
1 .... H_2O
DISCUSSION:
Ag_2Al, single crystal sphere grown and spark cut in a study of structure ordering. Solution used to etch polish the sphere.
REF: Neumann, J P — *Acta Metall*, 14,505(1966)

TIME:
TEMP:

AGAL-0002
ETCH NAME: Potassium cyanide
TYPE: Electrolytic, polish
COMPOSITION:
x .... 9% KCN

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

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DISCUSSION:

Ag_2Al, single crystal specimens used in a study of the effects of internal oxidation on plastic deformation. Specimens electropolished prior to deformation.
REF: Marcinkowski, M J & Wriedt, D F — *J Electrochem Soc*, 12,92(1964)

AGAL-0003a
ETCH NAME: A-2
TYPE: Electrolytic, polish
COMPOSITION:
x .... KCN
x .... H_2O
TIME: 1 min/8—10 sec
TEMP: RT
ANODE: Ag_2Al
CATHODE:
POWER: 1.5—2.5 A/cm²
DISCUSSION:

Ag_2Al, (0001) wafers used in a study of dislocation etch pits. Solution used to polish specimens prior to dislocation etching.
REF: George, J & Mote, J D — *J Appl Phys*, 36,1793(1963)

AGAL-0001b
ETCH NAME:
TYPE: Acid, dislocation
COMPOSITION:
5 .... HF
5 .... H_2SO_4
7 .... H_2O_2
TIME: 1 min
TEMP: RT
DISCUSSION:

Ag_2Al, (0001) wafers used in a study of dislocation etch pits. Solution used to develop pits: (1) etch in solution shown; (2) DI water rinse; (3) ethyl alcohol rinse and (4) dry with warm air. Hexagonal and pyramidal pits (dislocations) were observed that may develop a flat bottom with etch time. Dislocation density was $10^5/cm^2$.
REF: Ibid.

SILVER BORON ETCHANTS

AGB-0001
ETCH NAME: Ammonium hydroxide
TYPE: Acid, cleaning
COMPOSITION:
x .... NH_4OH, conc.
TIME:
TEMP: RT to hot
DISCUSSION:

AgB alloys with varying formulae used as high temperature brazing alloys. Surfaces can be cleaned prior to and after brazing with ammonia compounds.
REF: Pamphlet: *Silvaloy*, Silver Brazing Div., The American Platinum Works, NJ

SILVER LEAD TIN ETCHANTS

AGPS-0001a
ETCH NAME: Ammonium hydroxide
TYPE: Acid, cleaning
COMPOSITION:
x .... NH_4OH, conc.
TIME: 1—2 min
TEMP: RT
DISCUSSION:

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Pb:Sn #63 alloying 2% Ag used in device and package assembly of electronic devices. Solution used to clean surfaces prior to and after use.
REF: Walker, P — personal application, 1960

AGPS-0001b
ETCH NAME: Aqua regia, dilute — TIME: 10—30 sec
TYPE: Acid, removal/cleaning — TEMP: RT
COMPOSITION:
1 .... HCl
3 .... HNO_3
6 .... H_2O
DISCUSSION:
Pb:Sn + 2% Ag, solder used in device and package assembly of electronic devices. Solution used as a cleaning and light removal etch.
REF: Ibid.

AGPS-0001c
ETCH NAME: — TIME: 10—30 sec
TYPE: Acid, cleaning — TEMP: RT
COMPOSITION:
6 .... HAc
2 .... H_2O_2
2 .... H_2O
DISCUSSION:
Pb:Sn:Ag(2%), alloy #63 used in device and package assembly of electronic devices. Solution used to remove oxides after alloying and as a slightly preferential etch to observe alloy contact structure.
REF: Walker, P — personal development/application, 1959

AGPS-0001d
ETCH NAME: — TIME:
TYPE: Acid, removal — TEMP: RT
COMPOSITION:
1 .... HF
1 .... HNO_3
1 .... HAc
DISCUSSION:
Pb:Sn + Ag as various other alloys with or without silver. Solution used as a general removal and solubility etch in a study of 30 different alloy contacts for silicon.
REF: Ibid.

SILVER MERCURY ETCHANTS

AGHG-0001
ETCH NAME: Nitric acid — TIME:
TYPE: Acid, removal/polish — TEMP:
COMPOSITION:
(1) x .... HNO_3, conc.
(2) x .... HNO_3
x .... H_2O

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DISCUSSION:
Ag_2Hg, (110) single crystal surfaces of the natural mineral called Amalgam. Crystal was a dodecahedron, (110). Solutions used to etch clean surfaces.
REF: Walker, P — mineralogy study, 1953

SILVER TIN ETCHANTS

AGSN-0001
ETCH NAME: Potassium cyanide
TYPE: Electrolytic, polish
COMPOSITION:
6 g .... KCN
100 ml .... H_2O
TIME:
TEMP:
ANODE: Ag:Sn
CATHODE:
POWER:
DISCUSSION:
Ag:Sn(1%) single crystal alloys (also used 2, 4, 6 and 9% Sn) used in a study of dislocations and stacking fault energy in these alloys. Solution used to electropolish specimens.
REF: Ruff, A W & Ives, L K — *Acta Metall*, 15,189(1967)

SILVER TITANIUM ETCHANTS

AGTI-0001
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
1 .... HF
1 .... HNO_3
1 .... HAc
TIME: 30—60 sec
TEMP: RT
DISCUSSION:
Ti:TiAg:Ag thin films evaporated on silicon (111) devices for electrical contact. After photo resist patterning, solution used to remove excess metallization. Evaporated on silicon dioxide, SiO_2 thin films, then annealed in vacuum at 600°C for 15—30 min, converts to a conductive, glassy silicate. Solution also used to pattern the silicate.
REF: Walker, P — personal development/application, 1971

SILVER ZINC ETCHANTS

AGZN-0001
ETCH NAME: Nitric acid, dilute
TYPE: Acid, removal
COMPOSITION:
x .... HNO_3
x .... H_2O
TIME:
TEMP:
DISCUSSION:
Ag:Zn alloy specimens used in a study of self-diffusion in the material. After mechanically lapping surfaces, solution used to etch remove work damage.
REF: Cazarus, D & Tomizuka, C T — *Phys Rev*, 103,1155(1956)

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SILVER GERMANIUM PHOSPHIDE ETCHANTS

AGEP-0001
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
x .... HF
x .... HNO_3
DISCUSSION:
$Ag_6Ge_{10}P_{12}$, single crystal ingots grown by the Bridgman method as p-type semiconductor material. Band gap: 0.76 eV. Specimens were cut as parallelapipeds (001), (110) and (110) orientation, and mechanically lapped. Also grew $CuGe_2P_3$.
REF: McDonald, J E — *J Phys & Chem Solids*, 8,951(1985)

TIME:
TEMP:

PHYSICAL PROPERTIES OF SILVER BROMIDE, AgBr

Classification	Bromide
Atomic numbers	47 & 35
Atomic weight	187.8
Melting point (°C)	434
Boiling point (°C)	700
Density (g/cm^3)	5.8—6
Refractive index (n =)	2.25
Hardness (Mohs — scratch)	1—1.5
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Yellow/greenish
Cleavage (cubic — perfect)	(001)

SILVER BROMIDE, AgBr

General: Occurs in nature as the mineral bromyrite, AgBr, found in limited quantities both in Mexico and Chile and associated with other ores of silver, along with both silver chloride (cerargyrite) and the iodide (iodyrite). Primary use in industry is as a compound in photographic emulsions and in other chemical reactions.

Technical Application: Not used in Solid State device fabrication, other than in photographic film processing. It has been grown and studied as a single crystal for general morphology and defects.

Etching: KCN, NH_4OH and some sulfur compounds. Insoluble in water.

SILVER CADMIUM ETCHANTS

AGCD-0001
ETCH NAME: Nitric acid
TYPE: Acid, removal
COMPOSITION:
x .... HNO_3
x .... H_2O
DISCUSSION:
AgCd, single crystal specimens. A study of tracer diffusion as a chemical concentration gradient in the material. Both metals, individually, and as a compound single crystal can be etched in the solution mixture shown.
REF: Manning, J R — *Phys Rev*, 116,69(1959)

TIME:
TEMP: RT

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SILVER BROMIDE ETCHANTS

AGBR-0001
ETCH NAME: Emery
TYPE: Abrasive, polish
COMPOSITION:
x .... $Al_2O_3Fe_3O_4$
DISCUSSION:
AgBr, (110) wafers cut within $^1/_2°$ of plane. The ingot was grown by the Bridgman process with Cd added to pin dislocations. A (110) oriented cylinder was cut with a jeweler's saw, and surface of specimens was hand lapped and polished on emery paper.
TIME:
TEMP: RT
REF: Barber, R et al — *J Phys Chem Solids*, 46,107(1985)
AGBR-0002: Cain, L S — *J Phys Chem Solids*, 38,73(1977)
AgBr specimens. Similar study as in AGBR-0001

AGBR-0003
ETCH NAME: Sodium thiosulfate
TYPE: Acid, dislocation
COMPOSITION:
x .... 3—7 *N* NaS_2O_7
DISCUSSION:
AgBr, (100) and (111) wafers. Solution used to develop dislocations and dislocation loops in a general defect study of the material. Solution was not good on the (110) surfaces.
TIME:
TEMP:
REF: Bartlett, J T & Mitchell, J W — *Phil Mag*, 5,445(1960)

PHYSICAL PROPERTIES OF SILVER CHLORIDE, AgCl

Classification	Chloride
Atomic numbers	47 & 17
Atomic weight	143.34
Melting point (°C)	455
Boiling point (°C)	1550
Density (g/cm^3)	5.552
Refractive index (n =)	2.0611
Hardness (Mohs — scratch)	1—1.5
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Pearl-gray
Cleavage (solid)	None

SILVER CHLORIDE, AgCl

General: Occurs in nature as the mineral ceragyrite, AgCl, with the cubic structure similar to halite, NaCl (common salt), but has no cleavage. Common name of the mineral is "Horn silver". It is of secondary origin and usually found in the upper zones of silver vein deposits — quartz rock gangue — in association with silver bromides and iodides . . . pure native silver is often at the top of such veins due to hydrothermal and atmospheric alteration, though the chloride is insoluble in water. It is an ore of silver containing approximately 25% silver which can be easily separated by heating in the presence of carbon. In the presence of sunlight it turns brown-violet due to photoreaction by ultraviolet rays.

Both the natural mineral and artificial compound, along with AgBr and AgI, are the primary constituents in photographic emulsions in black and white photography . . . the silver, effectively, plating-out as the black oxide under intense UV light forming a permanent

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negative. As a finely divided particulate — smoke — the AgCl particles, like dust, are used to nucleate raindrops in storm clouds. When treated with salt of phosphorus and copper chips, it produces an intense azure-blue flame and, as a colloid, a brownish violet suspension.

Technical Application: Though not used in Solid State device processing, it is under study and development for its photoconductivity characteristics. Like seeding of rain clouds, AgCl smoke has been used in low temperature cryostats to grow snowflakes and single crystal ice. Both natural and artificially grown single crystals have been studied for physical and morphological data in addition to photoconductivity characteristics.

Etching: Soluble in NH_4OH and KCN.

SILVER CHLORIDE ETCHANTS

AGCL-0001
ETCH NAME: Potassium cyanide — TIME:
TYPE: Acid, polish — TEMP: RT
COMPOSITION:
x .... 3% KCN
DISCUSSION:
AgCl, (100) wafers used in a study of transient photoconductivity at low temperatures. Solution used on a glass plate to lap polish surfaces. Work was done in a dark room under red light.
REF: Van Heyningen, R S & Brown, F C — *Phys Rev*, 111,462(1958)
AGCL-0002: Brown, F C — *J Phys Chem Solids*, 4,206(1958)
AgCl, (100) wafers used in similar work as cited in AgCl-0001.

AGCL-0003
ETCH NAME: — TIME:
TYPE: Pressure, thinning — TEMP:
COMPOSITION:
x .... pressure
DISCUSSION:
AgCl specimens were thinned by applying physical pressure and followed by annealing for use in a structural study.
REF: Moser, F & Urbach, F — *Phys Rev*, 102,1519(1956)

AGCL-0004
ETCH NAME: Hydrochloric acid — TIME: 1 min
TYPE: Acid, cleaning — TEMP: RT
COMPOSITION:
x .... HCl, conc.
DISCUSSION:
AgCl, (100) wafers used in a photoconductivity study. Mechanically lap on a glass plate: (1) with coarse grit, use NH_4OH as the liquid carrier; (2) then fine grit using water as the carrier. After lapping, water rinse and follow with etch cleaning in the solution shown, with DI water rinse and dry.
REF: Gordon, A M — *Phys Rev*, 122,748(1961)

AGCL-0005
ETCH NAME: Nitric acid — TIME:
TYPE: Acid, cleaning — TEMP:
COMPOSITION:
x .... HNO_3, conc.

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DISCUSSION:

AgCl, (100) wafers used in a study of photoelectric behavior. Surfaces were cleaned as follows: (1) degrease in petroleum ether, (2) soak in nitric acid, and (3) wash in DI water.
REF: Wainfan, N — *Phys Rev*, 105,100(1957)

AGGL-0006
ETCH NAME: Ammonium hydroxide — TIME:
TYPE: Hydroxide, cleaning — TEMP:
COMPOSITION:

x .... NH_4OH

DISCUSSION:

AgCl, single crystal oriented bars cut with a jeweler's saw and then lapped down to size on a ground glass plate using a sodium thiosulfate solution. Wafers were cut on a lathe 1 cm diameter and .025" thick. Etch polish and clean in the solution shown, and rinse in DI water. HCl was used as an alternative etch polish solution.
REF: Wiegand, D A — *Phys Rev*, 113,52(1959)

AGCL-0007a
ETCH NAME: Hypo — TIME:
TYPE: Acid, polish/dislocation — TEMP: RT
COMPOSITION:

x .... 3 *N* $Na_2S_2O_5.5H_2O$

DISCUSSION:

AgCl, (100) oriented material cut as bars with the length in the c-axis direction ⟨001⟩. Bars used in a deformation study of dislocations induced by bending and annealing. Solution used to polish bars prior to anneal and bend. (*Note:* "Hypo" is the solvent used on AgCl in photography.)
REF: Sprackling, M T — *Phil Mag*, 9,739(1964)

AGCL-0007b
ETCH NAME: — TIME:
TYPE: Acid, preferential — TEMP: RT
COMPOSITION:

4 .... 3 *N* $Na_2S_2O_5.5H_2O$ ("hypo")
1 .... 0.88 *N* NH_4OH

DISCUSSION:

AgCl, (100) bars. Solution used to develop defects and dislocations in bars after annealing and bending. Also used 3 *N* "hypo" — AGCL-0007a.
REF: Ibid.

AGCL-0008: Jones, D A & Mitchell, J W — *Phil Mag*, 2,1047(1957)

AgCl single crystal specimens used in a study of dislocation etching and etching techniques. Also studied other silver halides.

SILVER EPOXY ETCHANTS

AGE-0001a
ETCH NAME: Nitric acid, dilute — TIME: 2—15 min
TYPE: Acid, removal — TEMP: RT or 50°C
COMPOSITION:

1 .... HNO_3
3 .... H_2O

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DISCUSSION:

Ag-Epoxy used for electrical attachment of GaAs FET dice to alumina substrate with gold circuit pads. Solution used to remove damaged FETs and Ag-Epoxy from alumina without affecting the gold circuits. This solution is called Schell etch (GAS-0045g) and is preferential on GaAs. After removal, wash alumina in DI H_2O, then bake in air oven at 125°C for $^1/_2$ h before re-using circuit.

REF: Walker, P — personal application, 1980—1985

AGEP-0001b

ETCH NAME:

TYPE: Gas, cleaning

TIME: 3—5 min

TEMP: RT

COMPOSITION:

x .... O_2, RF plasma

DISCUSSION:

Ag-Epoxy applied as a contact paste and cured at 120°C, 30 min in nitrogen. The epoxy can be "cleaned" in an RF plasma of oxygen developing a hard black surface coating of Ag_2O. So treated, the surface is very stable against chemical attack and atmospheres though, for some high reliability requirements, the black coating is undesirable. Remove by soak in HNO_3 or NH_4OH.

REF: Ibid.

PHYSICAL PROPERTIES OF SILVER IODIDE, AgI

Classification	Iodide
Atomic numbers	57 & 53
Atomic weight	234.8
Melting point (°C)	552 del.
Boiling point (°C)	
Density (g/cm^3)	5.5—5.7
Refractive index (n=)	2,182
Hardness (Mohs — scratch)	1.5—2
Crystal structure (hexagonal — hemimorphic)	(0001) platelets
(isometric — normal) >146°C	(100) cube
Color (solid)	Yellow
Cleavage (basal/cubic)	(0001)/(001)

SILVER IODIDE, AgI

General: Occurs in nature as the mineral iodyrite, AgI, associated with silver deposits as a minor compound.

Both silver iodide and bromide have major use in black and white photographic emulsions for conversion to the black oxide on film. The iodide has very low solubility in water and, as fine powder, it is used as a smoke to seed rain clouds and initiate precipitation. It is used in a similar manner to seed the growth of ice crystals in cold cryostats at LN_2 temperature in the study of ice formation both as snowflakes and single crystal ice.

Technical Application: In Solid State development silver iodide has been shown to have semiconducting properties as have several trinary and binary silver compounds, such as selenides and tellurides and their mixed compounds. Many such compounds have been grown as bulk single crystals or as thin films, but all are characteristically soft (H = 2—3) and can be tarnished in the presence of sulfur in the atmosphere or show oxide conversion under intense light conditions. When silver compounds are subjected to RF plasma with

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oxygen as a cleaning vehicle, surfaces will become coated with the black oxide, which can be removed NH_4OH.

Etching: KCN, NH_4OH, NH_3 and sulfur-containing compounds.

SILVER IODIDE ETCHANTS

AGI-0001
ETCH NAME:
TYPE: Acid, cleaning
TIME:
TEMP:
COMPOSITION:
x .... NH_4OH
DISCUSSION:

AgI, single crystals used to evaluate cohesive energy features of tetrahedral semiconductors. Forty materials studied.
REF: Aresti, A et al — *J Phys Chem Solids*, 45,361(1984)

AGI-0002
ETCH NAME: Silver iodide
TYPE: Compound, seeding
TIME:
TEMP: −195°C
COMPOSITION:
x .... AgI_2, powder
DISCUSSION:

AgI_2 powder used to seed growth of single crystal ice crystals in a cryostat at LN_2 temperature. See ICE-0007. (*Note:* Also used as a smoke to initiate precipitation from rain clouds.)
REF: Gonda, T & Koke, T — *J Cryst Growth*, 65,36(1983)

SILVER:GOLD ETCHANTS

AGAU-0001
ETCH NAME:
TYPE: Acid, preferential
TIME:
TEMP:
COMPOSITION:
x .... KCN
x .... $(NH_4)_2SO_4$
DISCUSSION:

Ag:Au, single crystal alloy ingots of various compositions grown in a study of twinning and defects after straining. Crystal compositions varied from 0, 10, 25, 50, 75, and 90% gold. Solution used as a metallographic structure development etch.
REF: Suzucki, H & Barrett, C S — *Acta Metall*, 6,156(1958)

AGAU-0002
ETCH NAME: Aqua regia, dilute
TYPE: Acid, step-etch/cleaning
TIME: Sec
TEMP: RT
COMPOSITION:
1 .... HCl
3 .... HNO_3
6 .... H_2O
DISCUSSION:

Ag:Au, evaporated thin films deposited on silicon (111) wafers as electrical contact

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pads. After alloy cycling solution used to clean surfaces and as a step-etch to observe alloy interface structure.
REF: Walker, P — personal application, 1973

SILVER MAGNESIUM ETCHANTS

AGMG-0001
ETCH NAME: TIME:
TYPE: Acid, cleaning TEMP:
COMPOSITION:
1 .... H_2SO_4
1 .... CrO_3
1 .... H_2O
DISCUSSION:
AgMg, single crystal specimens. Solution was used to clean surfaces before silver diffusion studies.
REF: Hagel, W C & Westbrook, J E — *Trans Met Soc AIME*, 221,951(1961)

PHYSICAL PROPERTIES OF SILVER SELENIDE, Ag_2Se

Classification	Selenide
Atomic numbers	47 & 34
Atomic weight	294.72
Melting point (°C)	880
Boiling point (°C)	
Density (g/cm^3)	8.0
Hardness (Mohs — scratch)	2—3
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Grey
Cleavage (cubic)	(001)

SILVER SELENIDE, Ag_2Se

General: Although silver selenide, Ag_2Se, does not occur in nature as a pure compound, there are minerals of this type containing one or more other elements: naumannite, $(Ag_2Pb)Se$; aguilarite, $Ag_2(S,Se)$; eucarite, Cu_2Se,Ag_2Se and crookesite, $(Cu,Tl,Ag)_2Se$. Where such minerals occur in sufficient quantity, they are mined for their silver and selenium content.

Technical Application: Silver selenide is a I—VI compound semiconductor and, although it has been studied, it has not been widely used due to its softness (H = 2.3) and solubility.

The compound has been grown as a single crystal for general morphological study and semiconductor characterization. There are a number of ternary and quaternary single crystals that have been grown and studied as a metallurgical, inter-related series: $AgSbSe_2$-$AgSbTe_2$-$AgBiSe_2$-PbSe-PbTe.

Etching: Mixed acids and alkalies. Soluble in alcohols.

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SILVER SELENIDE ETCHANTS

AGSE-0001a
ETCH NAME: TIME:
TYPE: Acid, polish TEMP: 50°C
COMPOSITION:
5 .... H_2SO_4
1 .... H_2O_2
DISCUSSION:
Ag_2Se, (100) wafers and other orientations. Solution used as a chemical polish etch and may show a degree of preferential attack.
REF: Sagar, A et al — *J Appl Phys*, 39,5336(1968)

AGSE-0001b
ETCH NAME: TIME:
TYPE: Acid, polish TEMP: 80°C
COMPOSITION:
2 .... KOH, sat. sol.
1 .... H_2O_2
2 .... ethylene glycol (EG)
DISCUSSION:
Ag_2Se, (100) wafers and other orientations. Solution shown as a polishing etch for this material.
REF: Ibid.
AGSE-0002: Gatos, H C Ed — *Properties of Elemental and Compound Semiconductors*, Interscience, New York, 1960, 69
$AgSbSe_2$, trinary compound semiconductors. Article covers trinary and quaternary compounds of silver selenides and tellurides with bismuth or lead additives.

AGSE-0003
ETCH NAME: BRM TIME:
TYPE: Halogen, polish TEMP: RT
COMPOSITION:
x .... x%Br_2
x .... MeOH
DISCUSSION:
Ag_2,Se (100) wafers used in a development study of new semiconducting compounds. Other compounds were $AgInTe_2$; Tl_2Se_3; SnSe; $SnSe_2$; TlSe; Bi_2Se_3; Li_3Bi; Bi_2Se_3; In_2Te_3; In_2Tl_3. Solution shown for reference.
REF: Mooser, E & Pearson, W B — *Phys Rev*, 101,492(1956)
AGSE-0004: Appel, J & Lantz, G — *Physica*, 20,1110(1954)
Additional reference on In_2Te_3.
AGSE-0005: Goodman, C H L & Douglas, R W — *Physica*, 20,1107(1954)
Additional reference on chalcopyrite, $AgInTe_2$.
AGSE-0006: Davidenko, V A — *J Phys (USSR)*, 4,170(1941)
Additional reference on compounds in AGSE-0003.

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SILVER GALLIUM SELENIDE ETCHANTS

AGGE-0001
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
(1) x .... NH_4OH (2) x .... HNO_3
DISCUSSION:
$AgGaSe_2$, single crystals used to evaluate cohesive energy features of tetrahedral semiconductors. Forty materials were studied.
REF: Aresti, A et al — *J Phys Chem Solids*, 45,361(1984)

SILVER INDIUM SELENIUM ETCHANTS

AGIE-0001
ETCH NAME: BRM TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
(1) x .... x% Br_2 (2) x .... HNO_3
x .... MeOH
DISCUSSION:
$AgInSe_2$, single crystals used to evaluate the cohesive energy features of tetrahedral semiconductors. Forty materials were studied.
REF: Aresti, A et al — *J Phys Chem Solids*, 45,361(1984)

SILVER TITANIUM SELENIDE ETCHANTS

AGTS-0001
ETCH NAME: BRM TIME:
TYPE: Halogen, polish TEMP:
COMPOSITION:
x .... x%Br_2
x .... MeOH
DISCUSSION:
AgTiSe, single crystal material grown in a study of optical and elastic properties. Structure is orthorhombic.
REF: Newman, P R — *J Appl Phys*, 54,1547(1983)

PHYSICAL PROPERTIES OF SILVER SULFIDE, AgS

Classification	Sulfide
Atomic numbers	47 & 16
Atomic weight	247.82
Melting point (°C)	825
Boiling point (°C)	
Density (g/cm^3)	7.2—7.36
Hardness (Mohs — scratch)	2.0—2.5
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Lead-grey/black
Cleavage (cubic)	(001)

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SILVER SULFIDE, AgS

General: Occurs in nature as the mineral argentite, AgS, common name: Silver Glance. It may be found in large deposits associated with other silver minerals, pyrite, galena, and nickel ores, as a primary deposit; with limonite, calcite, and quartz, as a deposit of secondary origin. It is a major ore of silver.

Other than as an ore of silver in industry, it is the compound source for silver plating baths, and used in black and white photography.

Technical Application: In Solid State processing, silver sulfide is used for silver plating of parts and packages. It is under evaluation for its optoelectric properties and as a trinary compound, $AgGaS_2$, has semiconducting properties.

Silver reacts readily with sulfur which is the black tarnish on silver plate can be due to sulfur fumes in the air. It is grown as a single crystal by the action of sulfur, sulfur dioxide or hydrogen sulfide on pure silver.

Etching: Soluble in acids and KCN.

SILVER SULFIDE ETCHANTS

AGS-0001a
ETCH NAME: Potassium cyanide
TYPE: Electrolytic, polish
COMPOSITION:
x .... KCN
x .... H_2O
DISCUSSION:
AgS, single crystal whiskers. Solution used to polish specimens.
REF: Drott, J — *Acta Metall,* 9,19(1961)

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

AGS-0001b
ETCH NAME: Nitric acid, dilute
TYPE: Acid, preferential
COMPOSITION:
x .... 0.1 *N* HNO_3
DISCUSSION:
AgS, single crystal whiskers. Solution used to develop dislocations in a study of crystal whisker structure.
REF: Ibid.

TIME:
TEMP:

SILVER GALLIUM SULFIDE ETCHANTS

AGAS-0001
ETCH NAME: Hydrochloric acid
TYPE:
COMPOSITION:
x .... HCl
DISCUSSION:
$AgGaS_2$, thin films as both polycrystalline and amorphous structures deposited by flash evaporation onto glass substrates and ITO film coated glass substrates. Material is a I—III—VI_2 compound semiconductor with optoelectronic and photoconducting capabilities. Structure is tetragonal chalcopyrite type.
REF: Camp, H V — *Thin Solid Films,* 111,17(1984)

TIME:
TEMP:

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PHYSICAL PROPERTIES OF SILVER TELLURIDE, Ag_2Te

Classification	Telluride
Atomic numbers	47 & 52
Atomic weight	343.37
Melting point (°C)	>300
Boiling point (°C)	
Density (g/cm³)	8.31—8.45 (hessite)
Hardness (Mohs — scratch)	2—2.5
Crystal structure (orthorhombic — normal)	(100) a-pinacoid
Color (solid)	Lead grey
Cleavage (traces)	(100)/(110)

SILVER TELLURIDE, Ag_2Te

General: Occurs as the mineral hessite, Ag_2Te, usually massive or fine grained. With increasing gold content it grades into the mineral petzite, $(Ag,Au)_2Te$, with G = 8.7—9.02. These are minor minerals, but industrially important for their silver and gold content, as well as tellurium.

Technical Application: Silver telluride is the base of a I—VI compound semiconductor as a trinary compound with antimony or iron, and has been evaluated for semiconducting characteristics. The trinary compounds are shown here.

Etching: Single acids of HNO_3, H_2O_2, and mixed acids HF:HNO_3 with salts.

SILVER ANTIMONY TELLURIDE ETCHANTS

SAT-0001
ETCH NAME: TIME:
TYPE: Acid, polish TEMP:
COMPOSITION:
1 .... HNO_3
1 .... HCl
2 .... $K_2S_2O_7$, sat. sol.
DISCUSSION:
$AgSbTe_2$, single crystal specimens. Solution is described as a metallographic polishing etching. Also can be used on $AgFeTe_2$.
REF: Wolfe, R et al — *J Appl Phys,* 31,1959(1960)

SAT-0002a
ETCH NAME: Nitric acid, dilute TIME: 10 sec
TYPE: Acid, preferential TEMP: RT
COMPOSITION:
x .... 55% HNO_3
DISCUSSION:
$AgSbTe_2$, single crystal specimens. Solution used to develop defect structure in this material. Authors refer this etch to Farnsworth (AG-0001) as a silver orientation etch.
REF: Armstrong, R W et al — *J Appl Phys,* 31,1954(1960)

SAT-0002b
ETCH NAME: Hydrogen peroxide TIME:
TYPE: Acid, dislocation TEMP:
COMPOSITION:
x .... H_2O_2

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DISCUSSION:

$AgSbTe_2$, single crystal specimens used in a structural study. Solution shown develops structure but not as well as SAT-0002a. Both CP4 and Dash Etch were evaluated, but were not as good. For CP4, see: SI-0047; for Dash Etch, SI-0046.

REF: Ibid.

SAT-0003: Gatos, H C Ed — *Properties of Elemental and Compound Semiconductors*, Interscience, New York, 1960, 69

$AgSbTe_2$, trinary compound semiconductor growth and study.

SILVER IRON TELLURIDE ETCHANTS

SIT-0001

ETCH NAME:

TIME:

TYPE: Acid, polish

TEMP:

COMPOSITION:

1 .... HNO_3

1 .... HCl

2 .... $K_2S_2O_7$, sat. sol.

DISCUSSION:

$AgFeTe_2$, single crystal specimens studied as a semiconductor compound. Solution used to polish specimens.

REF: Wernick, J H & Wolfe, R — *J Appl Phys*, 32,749(1961)

PHYSICAL PROPERTIES OF SILVER TELLURIDE, Ag_2Te

Classification	Telluride
Atomic numbers	47 & 52
Atomic weight	343.47
Melting point (°C)	955
Boiling point (°C)	
Density (g/cm^3)	8.3—8.45
Hardness (Mohs — scratch)	2.5—3
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Steel-grey
Cleavage (indistinct)	(001)

SILVER TELLURIDE, Ag_2Te

General: Occurs in nature as the mineral hessite, Ag_2Te, and the mineral stutzite, Ag_4Te. There are also some trinary compound telluride minerals, such as petzite $(Ag,Au)_2Te$; suvanite $(Ag,Au)Te_2$; krennerite $(Au,Ag)Te_2$ and muthmannite, (Ag,Au)Te, in addition to the complex mineral — tapalpite $3Ag_2(S,Te).Bi_2(S,Te)_3$. Silver forms over 50 sulfide minerals as well as silver halides of iodine, chlorine and bromine. Where there minerals are found in quantity, they are mined for their silver content and other elements. The sulfide, argentite (Ag_2S) is a major ore of silver. Primary use in industry is as ores of silver and tellurium.

Technical Application: Silver telluride is a I—VI compound semiconductor like silver selenide, Ag_2Se. Both compounds have been studied for their semiconductor characteristics and general morphology as single crystals. There are a number of ternary and quaternary single crystals that have been grown and studied, such as $AgSbSe_2$-$AgSbTe_2$-$AgBiSe_2$-PbTe-PbSe, as they form an inter-related metallurgical series.

Etching: Soluble in HNO_3 and KCN and mixed acids.

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SILVER TELLURIDE ETCHANTS

AGTE-0001
ETCH NAME: TIME:
TYPE: Acid, polish TEMP:
COMPOSITION:
2 .... H_2O_2
3 .... NH_4OH
DISCUSSION:
Ag_2Te, (100) wafers and other orientations. Solution shown as a general polish etch.
REF: Sagar, A et al — *J Appl Phys*, 39,5336(1968)
AGTE-0002: Gatos, H C, Ed — *Properties of Elemental and Compound Semiconductors*, Interscience, New York, 1960, 69
AgSbTe, trinary compound semiconductors.

SILVER GALLIUM TELLURIDE ETCHANTS

AGGT-0001
ETCH NAME: BRM TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... x%Br_2
x .... MeOH
DISCUSSION:
$AgGaTe_2$, single crystals used in evaluating cohesive energy features of tetrahedral semiconductors. Forty materials were studied. Solution shown for reference.
REF: Aresti, A et al — *J Phys Chem Solids*, 45,361(1984)

SILVER INDIUM TELLURIDE ETCHANTS

AGIT-0001
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... H_2O_2
x .... NH_4OH
DISCUSSION:
$AgInTe_2$, single crystals used to evaluate cohesive energy features of tetrahedral semiconductors. Forty materials were studied.
REF: Aresti, A et al — *J Phys Chem Solids*, 45,362(1984)

PHYSICAL PROPERTIES OF SODIUM, Na

Classification	Alkaline metal
Atomic number	11
Atomic weight	22.99
Melting point (°C)	97.8 (97.5)
Boiling point (°C)	892
Density (g/cm^3) solid/gas	0.97/1275
Thermal conductance (cal/sec)(cm^2)(°C/cm) 20°C	0.334
Specific heat (cal/g) 25°C	0.295
Heat of fusion (cal/g)	27.05
Heat of vaporization (k-cal/g-atom)	1.127

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Coefficient of linear thermal expansion ($\times 10^{-4}$ cm/cm/°C) 20°C	71
Atomic volume (W/D)	23.7
1st ionization energy (K-cal/g/mole)	119
1st ionization potential (eV)	5.138
Electronegativity (Pauling's)	0.9
Covalent radius (angstroms)	1.54
Ionic radius (angstroms)	0.97 (na^{+1})
Critical temperature (g/cm^3)	0.206
Electrical resistivity ($\times 10^{-6}$ ohm cm) 0°C	4.2
Electron work function (eV)	2.27
Cross section (barns)	0.53
Vapor pressure (°C)	701
Refractive index (n =)	4.22
Hardness (Mohs — scratch)	0.4
Crystal structure (isometric — normal)	(100) cube, bcc
Color (solid)	Silver white
(flame)	Intense yellow
Cleavage (cubic — poor)	(001)

SODIUM, Na

General: Sodium does not occur as a free element in nature, but there are over 150 known single crystal compounds that contain sodium and, if artificial compounds are included, there are over 300. Common salt, as the mineral halite, NaCl (sodium chloride) is the major compound in ocean (salt) water and is found as dried salt beds and domes from ancient buried seas. At Sperenberg, Germany there is a deposit 4000 ft thick; there are similar "salt domes" along the Gulf Coast of the U.S., and there are buried salt beds as farth north as the Great Lakes in the mid-continent. In Galicia, Poland there is a bed 50 miles long, 20 miles wide, and 1200 ft thick. The Dead Sea in Palestine, 47 miles long and 9 miles wide, contains 40 billion tons of salt in solution; there is the Great Salt Lake in Utah, and the Salton Sea in southern California. There are many dry salt flats throughout the southwestern U.S., as well is in other desert areas of the world.

Salt is one of the most important compounds in the chemical industry for it produces (1) sodium; (2) sodium hydroxide; (3) sodium carbonate (soda ash); (4) chlorine gas and (5) both hydrochloric and sulfuric acids are compounded from salt. In the metals industries, salt brine is used as a quenching medium of hot metals and alloys; as a molten flux for etching and as a metal flux for soldering. As soda ash (natron, $Na_2CO_3.nH_2O$, the natural compound is only found in solution) it was used by the Egyptians in mummification; used in the manufacture of soap, glass, and bleaching powder. The chemical symbol, Na, comes from mineral natron. In addition, salt has been a food preservative since ancient times and is essential in human and animal diet. The mineral borax, $Na_2B_4O_7.10H_2O$, is a source of both sodium, Na and boron, B, and is used for washing and cleaning as an antiseptic and preservative, as a solvent of metal oxides and as a flux in soldering and welding. The name comes from the Arabic buraq — which included niter (sodium carbonate) and natron of the Egyptians.

As sodium hydroxide, NaOH, it is a major cleaning and etching solution in various concentrations for most metals and metallic compounds in industrial processing; as a molten flux above 300°C it is both a cleaner and etchant on metals and alloys.

A major use of sodium metal is in the manufacture of sodium cyanide, NaCN, for electrolytic plating and in organic reactions; to compound tetraethyl lead for gasoline antiknock and in the making of dyes and perfumes, e.g., attar of roses. As liquid sodium, it is a coolant for high power aircraft engines. When housed in a metal tube, through which a

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quartz tube is inserted, it is a temperature stabilizing "heat pipe" in horizontal epitaxy reactors. The sodium lamp — sodium and mercury with a trace of neon as the activator, produces two intense yellow lines, characteristic of sodium . . . among other applications, such lamps are used in determining material surface flatness and step-height of thin films (D/Fx2945 Å = T) . . . in both cases the observer sees a brilliant yellow background with black lines.

Although pure sodium metal is shiny and silver in appearance, much like silver, it oxidizes rapidly in air to a white surface oxide. It is stored under toluene and, if the toluene is heated, sodium will liquefy and can then be poured into forms as a rod or wire with high electrical conductivity and heat dissipation.

Technical Application: Sodium is considered a contaminating species, much like oxygen, in many Solid State material processes, such as in silicon ingots. It is not used as elemental sodium in device fabrication and, where etchants containing sodium are used, heavy washing follows to insure complete removal of all traces of sodium prior to any high temperature processing.

As mentioned in the general section, liquid sodium is used as a heat pipe stabilization mechanism in epitaxy reactors and similar "pipes" are used as heat removal/transfer units in a variety of semiconductor and electronic hardware applications.

Many sodium compounds are used in etching formulations for the etching of semiconductors and metals, such as sodium hydroxide, NaOH; sodium bromide, NaBr; sodium carbonate, $NaCO_3$; sodium chromate, $NaCrO_4$ and dichromate, $Na_2Cr_2O_7$, as well as sodium cyanide, NaCN to mention only a few of the more prominent. They are used, alone, in various solution concentrations in water; as one constituent of a solution mixture, or in solid form as a molten flux etchant. The latter also is used as a growth flux for some metallic compounds, such as ferrites and garnets.

Etching: Deliquesces in cold water to hydroxides; can be explosive in hot water with release of hydrogen. Soluble in hot toluene.

SODIUM ETCHANTS

NA-0001
ETCH NAME: Nonyl
TYPE: Alcohol, polish
TIME: 30 sec
TEMP: RT
COMPOSITION:
x .... $CH_3(CH_2)_7CH_2OH$
DISCUSSION:

Na specimens cut with a razor blade under oil (also lithium and potassium). After cutting, put in heptane to remove oil, then into tetrahydrofuran to stop heptane reaction. Ten alcohols were evaluated for reactivity with sodium. Increasing molecular weight reduces reactivity. Nonyl develops grain boundaries with mostly (hkl) plane surfaces smooth and brilliant. MeOH, EOH, and 2-propanol are similar, but (hkl) surfaces vary from brilliant to dull with pitting. Other alcohols similar to MeOH and EOH with two producing only dull, pitted surfaces or an unspecified dark surface. See LI-0006 and K-0001 for additional discussion.
REF: Castellano, R N & Schmidt, P H — *J Electrochem Soc*, 118,653(1971)

NA-0002
ETCH NAME:
TYPE: Alcohol, polish
TIME:
TEMP:
COMPOSITION:
98% .... $C_6H_4(CH_3)_2$ (*o*-xylene)
2% .... isopropyl alcohol (IPA)
DISCUSSION:

Na specimens. Solution described as a polish etch for sodium.
REF: Bowers, R et al — Mater Sci Center Rep. #3, Cornell University, Ithaca, NY, 1961

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PHYSICAL PROPERTIES OF SODIUM BROMIDE, NaBr

Classification	Bromide
Atomic numbers	11 & 35
Atomic weight	103
Melting point (°C)	755
Boiling point (°C)	1300
Density (g/cm^3)	3.2
Hardness (Mohs — scratch)	2
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Colorless
Cleavage (cubic — poor)	(001)

SODIUM BROMIDE, NaBr

General: Does not occur as a natural solid compound due to solubility, but may be extracted from ocean water and some hot springs along with chlorides, iodides, and halite, NaCl, common salt. Halogen minerals in chemistry and Solid State are referred to as the halides or as ionic crystals, based on their weak Van Der Waal atomic bonding which can be readily altered by external factors: X-ray, infrared or ultraviolet light, and pressure, etc. The compound has had little use in general metal processing, but there are chemical and medical applications.

Technical Application: Sodium bromide has not been used in Solid State processing, although it has been grown as a single crystal for general morphology and electrical studies.

Etching: Water and alcohols, or acids.

SODIUM BROMIDE ETCHANTS

NABR-0001
ETCH NAME: Alcohol, dilute — TIME:
TYPE: Alcohol, cutting — TEMP:
COMPOSITION:
10% .... alcohol
DISCUSSION:
NaBr, single crystal specimens used in an electrical breakdown study. Specimens were cut using a string soaked in the solution shown. Surface polishing was done on a chamois cloth soaked in the solution.
REF: Fernandez, A et al — *J Appl Phys*, 37,36(1966)

NABR-0002
ETCH NAME: Sulfuric acid, dilute — TIME:
TYPE: Acid, removal — TEMP:
COMPOSITION:
x .... H_2SO_4
x .... H_2O
DISCUSSION:
NaBr, specimens used in a study of neutron-transfer reactions from nitrogen bombardment. Solution shown used as a general etch, and may include other acids.
REF: Halbert, M L et al — *Phys Rev*, 106,251(1957)

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PHYSICAL PROPERTIES OF SODIUM CHLORIDE, NaCl

Classification	Alkaline metal
Atomic numbers	11 & 17
Atomic weight	58
Melting point (°C)	800
Boiling point (°C)	1400
Density (g/cm^3)	2.1—2.3 (2.164 pure)
Refractive index (n =)	1.5442
Hardness (Mohs — scratch)	2.5
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Clear/tints
(flame)	Deep yellow
Cleavage (cubic — perfect)	(001)
Taste (atypical)	Saline

SODIUM CHLORIDE, NaCl

General: Occurs as the natural mineral halite, NaCl, called common salt or rock salt. As a solid, other than as the massive "rock salt", always forms in distinct cubes with perfect (100) cleavage — even when finely pulverized, it is in small cubes. Natural salt varies in color from colorless to white, yellowish, reddish, bluish and purplish, which has been related to the presence of colloidal sodium, inclusion of organic matter, etc. It is the major chemical compound in salt (ocean) water, and is found in some salt springs. As a dried mineral it occurs as irregular beds in sedimentary rocks to depths of greater than a mile from the evaporation of ancient shallow salt water seas. Salt is still obtained from the evaporation of ocean water — about 25% of world usage — with an additional 25% pumped directly to the industrial user as "brine" and 50% from salt beds.

Salt is one of the most important compounds in the chemical industry, not only as the chief source of sodium metal and chlorine (gas), but for a number of chemical compounds and processes. As sodium chloride it is used as an etchant and quenching medium in metal processing. Sodium is "metal of soda" with the name and symbol, Na, coming from the natural mineral natron, Na_2CO_3, and as sodium hydroxide, NaOH, it is a major etchant solution for metals, alloys, semiconductors and metallic compounds.

As salt, it is essential to mammillary diet — the body fluids are a salt electrolyte. (See Potassium chloride, KCl — natural mineral sylvite, as a dietary replacement.) Salt has long been a food preservative as salted fish, beef, vegetables, etc.

Technical Application: Sodium chloride is not used directly in Solid State device fabrication, but many compounds are used in etching and cleaning processes. Sodium, Na, is considered a contaminant as it will diffuse into the material during high temperature processing and can seriously affect device electrical parameters but, as brine, it is used as a "salt spray test" in high reliability evaluation of devices and assemblies. This is an accelerated test for corrosion effects that can occur in the natural environment during the operational life of metals, alloys and devices.

Sodium chloride is grown as single crystal boules of large size by controlled evaporation from saturated solutions and both natural and artificial crystals have been the subject of much study. As they are colorless, clear crystals, soft and easily deformed, they are of great interest for observing the basic mechanics of defect generation in materials that form in the isometric (cubic) system, normal class. Most semiconductors and metals used in general processing are in the isometric system. Sodium chloride crystals are of additional interest for the effects of defects and color generation by irradiation, infrared, or ultraviolet light that can be related to opaque materials.

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As cut or cleaved (100) substrates of NaCl are used for the evaporation of metal thin films or epitaxy growth of compounds with water float-off of the films for morphological study by TEM or SEM.

Etching: Soluble in water, alcohols, some esters; salts, including NaCl itself and several mixed acids.

SODIUM CHLORIDE ETCHANTS

NACL-0001
ETCH NAME: Water
TYPE: Acid, polish
COMPOSITION:
x .... H_2O
TIME:
TEMP: RT
DISCUSSION:

NaCl, (100), cleaved wafers used in an X-ray radiation study. Results were similar for specimens cleaved in air or under vacuum. Prepare wafers prior to irradiation by (1) chem/mech polish with water to remove cleavage steps or surface defects; (2) rinse in ethyl alcohol; (3) rinse in acetone.

REF: Lieder, N R — *Phys Rev*, 101,56(1956)

NACL-0002a: Greenler, R G & Hothwell, W S — *J Appl Phys*, 31,616(1960)

NaCl, (100) wafers used in a ductility study. Wafers were soaked in water as one part of the study.

NACL-0019: Vook, R W — *J Appl Phys*, 32,1557(1961)

NaCl, (100) cleaved wafers used for evaporation and study of Sn thin films. After cleaving, polish surfaces with chamois cloth dampened with water; follow with vacuum bake (UHV) at 375°C for 6 h; then seal in Pyrex ampoule and oven bake at 500°C. Kikuchi lines used to determine surface cleanliness.

NACL-0021: Rahman Kahn, M S — *Thin Solid Films*, 113,207(1984)

NaCl, (100) cleaved wafers or (110) and (111) wafers cut and used for growth of erbium hydride, ErH_2. Wafers were water polished. Deposits were poly-ErH_2 from RT to 298 K growth temperature. At 488 K, ErH_2 was (100) on all NaCl orientations. ErH_2 is cubic, fcc; heat in H_2 to convert to hexagonal, hcp, ErH_3.

NACL-0010a: In der Schmitten, W & Hassen, P — *J Appl Phys*, 32,1790(1961)

NaCl, (100) cleaved wafers used in a stress dislocation study. Prior to stressing, wafers were chem/mech polished on a silk cloth dampened with water; rinsed in ethyl alcohol: then rinsed in ether.

NACL-0031: Fjelivag, H et al — *J Phys Chem Solids*, 45,709(1984)

NaCl:Ni doped single crystal ingots grown by the Czochralski (CZ) method from a powdered mixture of NaCl:$NiCl_2$ using a vertical zone melting technique. A study of the controlled doping effects on this doped material. Crystals were transparent and pale pink in color.

NACL-0032: Walker, P et al — mineralogy study, 1953

NaCl, as natural single crystals of halite in cubic, (100) form. Surfaces were chem/mech polish/cleaned on a linen cloth saturated with water prior to pressurization for white and colored light irradiation studies. Also worked with natural crystals of sylvite, KCl, in comparative study of the two compounds.

NACL-0036: Eltoukhy, A H & Greene, J E — *J Appl Phys*, 50,505(1979)

NaCl, (100) cleaved substrates used for thin film growth of InGaSb. Both NaCl and BF_2, (111) substrates were cleaned: (1) TCE vapor degrease; (2) rinse in MeOH; (3) rinse in acetone. Glass substrates were detergent scrubbed and water rinsed. After epitaxy, thin films were etched thinned (AGSB-0001), then the float-off technique used to remove films for TEM study. Float-off (1) water for NaCl; (2) HF for glass; (3) dilute HCl for BF_2.

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NACL-0020b: Neogebauer, C A — *J Appl Phys*, 31,1096(1960)

NaCl, (100) cleaved wafers used as substrates for thin film gold evaporation. Remove films for TEM study with water float-off.

NACL-0005c: Ibid.

NaCl, (100) wafers used as substrates for epitaxy of germanium thin films. Use water float-off of film for TEM study.

NACL-0004a

ETCH NAME: Moran's etch — TIME: 30 sec

TYPE: Salt, dislocation — TEMP: RT

COMPOSITION:

3 g .... $HgCl_2$

1000 ml .... EOH

DISCUSSION:

NaCl, (100) wafers used in a dislocation development etching study. This was a preferred solution for general preferential and defect development.

REF: Moran, P R — *J Appl Phys*, 29,1768(1958)

NACL-0014: Webb, W W — *J Appl Phys*, 31,194(1960)

NaCl, single crystal whiskers studied for structure. Used Moran's etch and rinsed in CCl_4. The two types of pits that developed were (1) a triangle with flat bottom and not reproducible; and (2) a triangle with pointed bottom. The former is a surface damage pit; the latter a dislocation pit.

NACL-0015: Shaskol'skaya, H P & Sun-Jui, Fang — *Sov Phys-Cryst*, 4,74(1960)

NaCl, (100) specimens used in a structure study. Solution used at RT, 1—2 sec with rinse in ether. Etch figures and glide planes correspond to emergence of edge dislocations.

NACL-0011a: Machida, C A & Munir, Z A — *J Cryst Growth*, 68,664(1984)

NaCl, (100) cleaved wafers used in a study of thermal etch pits. After cleaving, store in dessicator with zeolite, $CaSO_4$ drying agent. Place wafers on quartz plate in vacuum, apply current parallel to (100) direction, and use heat lamp radiation outside of vacuum system to initiate thermal alteration. A 30 g $HgCl_2$:EOH solution was used to develop thermal etch pits. Pit density was on the order of $4 \times 10^6/cm^2$.

NACL-0017: Crawford, J A & Young, F W Jr — *J Appl Phys*, 31,1688(1960)

NaCl, (100) wafers studied for F-center development of gamma irradiation. Solution used at RT, 30 sec after irradiation to develop dislocations.

NACL-0010c: Ibid.

NaCl, (100) cleaved wafers used in a stress study. After stressing, a 1% $HgCl_2$:EOH solution was used to develop dislocations. See NACL-0010a-b.

NACL-0007f: Ibid.

NaCl, (100) wafers used in a dislocation study. Moran's etch used in addition to other solutions, and is similar to the "W" etch. See NACL-0024.

NACL-0008a

ETCH NAME: Water — TIME:

TYPE: Acid, forming — TEMP: RT

COMPOSITION:

(1) x .... H_2O (2) x .... NaCl, sat. sol.

DISCUSSION:

NaCl, single crystal specimens used to fabricate pointed filaments. Coat sections to be formed with glyptol. Form by (1) etch in water; (2) etch in NaCl saturated water solution; (3) rinse in ethyl alcohol. Method used for similar fabrication of other alkali halides.

REF: McNulty, J et al — *Rev Sci Instr*, 31,904(1960)

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NACL-0023
ETCH NAME: Water
TYPE: Acid, jet-forming
COMPOSITION:
x .... H_2O
TIME:
TEMP: RT
DISCUSSION:
NaCl, (100) wafers used as substrates for evaporation of gold thin films in a mechanical property study of gold. After metallization, a water jet was used to etch a 0.5 mm hole through the NaCl substrate back to expose the gold film on the opposite side without film removal.
REF: Carlin, A & Walker, W P — *J Appl Phys*, 31,2135(1960)

NACL-0024
ETCH NAME: Water
TYPE: Acid, float-off
COMPOSITION:
x .... H_2O
TIME:
TEMP: RT
DISCUSSION:
NaCl, (100) wafers used as substrates for thin film gold evaporation. Gold films removed for TEM study by liquefying the gold/NaCl interface with water as the solvent using the float-off technique. Also: SiO_2/A-Si/Ni/SiV_2 layer.
REF: Chen, S H et al — *J Appl Phys*, 57,258(1985)
NACL-0033:
NaCl, (100) wafers used as substrates for evaporated copper thin films. After deposition, the Cu/NaCl interface was liquefied with water for film float-off and TEM study. Mica, (0001) substrates also used with 10% HF for float-off.
NACL-0034: Nakhotkiu, N G et al — *Thin Solid Films*, 112,267(1984)
NaCl, (100) substrates used for growth of a-Ge thin films. Films removed by water float-off for TEM study.
NACL-0035: Pierrard, P et al — *Thin Solid Films*, 111,141(1984)
NaCl, (100) cleaved substrates used for growth of a-Ge thin films. Used de-salted water for float-off of films for TEM study. Heat from the TEM microscope did not crystallize the amorphous films. Laser application under TEM observation produced single crystal spherulitic structures.

NACL-0002b
ETCH NAME: Salt water
TYPE: Acid, polish
COMPOSITION:
x .... NaCl, sat. sol.
x .... H_2O
TIME:
TEMP:
DISCUSSION:
NaCl, (100) wafers used in a ductility study. Wafers were soaked in salt water as one step of surface preparation for the study.
REF: Ibid.
NACL-0008b: Ibid.
NaCl, single crystal specimens used to fabricate pointed filaments. Solution used as an etchant. See NACL-0008a.

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NACL-0002c
ETCH NAME: Ethylene glycol
TYPE: Ester, ductility effect
COMPOSITION:
x .... CH_2OHCH_2OH (EG)
TIME:
TEMP: RT
DISCUSSION:
NaCl, (100) wafers used in a ductility study. Wafers subjected to various liquids and atmospheres as H_2O, air and NaCl, saturate solution.
REF: Ibid.

NACL-0027
ETCH NAME: Chloroform
TYPE: Gas, cleaning
COMPOSITION:
x .... $CHCl_3$
TIME:
TEMP: RT
DISCUSSION:
NaCl, (100) wafers used as substrates for oriented epitaxy growth of $AuGa_2$ thin films. Prior to deposition, wafers were cleaned (1) in chloroform; (2) rinsed in acetone; and (3) rinsed in MeOH. Final drying in ultra high vacuum (UHV) immediately prior to metal compound growth.
REF: Richard, R D et al — *J Vac Sci Technol*, A(2),535(1984)

NACL-0003a
ETCH NAME: Ethyl alcohol
TYPE: Alcohol, polish
COMPOSITION:
x .... C_2H_5OH (EOH)
TIME:
TEMP:
DISCUSSION:
NaCl, (100) wafers. Solution used to polish wafers with rinse in carbon tetrachloride, CCl_4.
REF: Barr, L W & Morrison, J A — *J Appl Phys*, 31,617(1960)

NACL-0003c
ETCH NAME: Chlorine
TYPE: Gas, preferential
COMPOSITION:
x .... Cl_2, vapor
TIME: 48 h
TEMP: 700°C
PRESSURE: 500 mmHg
DISCUSSION:
NaCl, (100) cleaved wafers used in a dislocation study. Hot chlorine vapors developed square etch pits along cleavage steps on surfaces. Referred to as crystallite boundaries.
REF: Ibid.

NACL-0005b
ETCH NAME: Heat
TYPE: Thermal, float-off
COMPOSITION:
x .... heat
TIME:
TEMP: 800°C
DISCUSSION:
NaCl, (100) cleaved wafers used as substrates for growth studies of evaporated Au thin films. Very thin gold films were float-off removed by heat melt-away of the NaCl substrate.

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Thicker films sheared away by cleaving the substrate with residual salt remaining on gold removed by water washing.
REF: Ibid.

NACL-0029
ETCH NAME: Hydrochloric acid — TIME:
TYPE: Acid, cleaning — TEMP:
COMPOSITION:
x .... HCl, conc.
DISCUSSION:
NaCl, (100) wafers used in a study of laser induced irreversible absorption changes at 10.6 μm light frequency. Etch clean wafers in HCl, and rinse in isopropyl alcohol (IPA or ISO).
REF: Wu, S T & Bass, M — *J Appl Phys Lett*, 39,948(1981)

NACL-0020a
ETCH NAME: Ethyl alcohol, dilute — TIME:
TYPE: Alcohol, polish — TEMP: RT
COMPOSITION:
x .... C_2H_5OH (EOH)
x .... H_2O
DISCUSSION:
NaCl, (100) cleaved wafers used as substrates for thin film gold evaporation. Polish wafers in solution shown prior to metallization. Gold deposits as (100) oriented film. Remove film for TEM study with water float-off.
REF: Neogebauer, C A — *J Appl Phys*, 31,1096(1960)

NACL-0010b
ETCH NAME: — TIME: 5 min
TYPE: Alcohol, dislocation — TEMP: RT
COMPOSITION:
1 .... C_2H_5OH (EOH)
1 .... CH_3OH (MeOH)
DISCUSSION:
NaCl, (100) cleaved wafers used in a dislocation study of stressed specimens. Mechanically polish with water dampened silk cloth, rinse in EOH, then in ether. Stress wafer, then cross-section cleave on ⟨110⟩. Use solution shown to develop dislocation density and flow characteristics. Also used Moran's etch as a 1% $HgCl_2$:EOH mixture. See NACL-0004a.
REF: In der Schmitten, W & Hassen, P — *J Appl Phys*, 32,1790(1961)

NACL-0004b
ETCH NAME: Methyl alcohol — TIME:
TYPE: Alcohol, polish — TEMP: RT
COMPOSITION:
x .... CH_3OH (MeOH)
DISCUSSION:
NaCl, (100) cleaved wafers used in development of etching techniques. Solution used to polish surfaces prior to dislocation etching.
REF: Ibid.

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NACL-0007a
ETCH NAME: Propionic acid
TYPE: Acid, cleaning
COMPOSITION:
x .... CH_3CH_2COOH
DISCUSSION:
NaCl, (100) wafers used in a dislocation study. Wafers were cleaned (1) in solution shown, (2) rinsed in petroleum ether and air blown dried or (3) rinsed in pyridine and air blown dried.
REF: Barber, D J — *J Appl Phys*, 33,3140(1962)
TIME:
TEMP: RT

NACL-0005a
ETCH NAME: Hydrochloric acid
TYPE: Acid, polish
COMPOSITION:
70 .... HCl
30 .... H_2O
DISCUSSION:
NaCl, (100) wafers used as substrates for epitaxy growth of germanium thin films. Prior to epitaxy, polish wafers on pad soaked with solution shown using light pressure. Use water float-off of film for TEM study.
REF: Outlaw, R A & Hopson, P Jr — *J Appl Phys*, 55,1460(1984)
TIME:
TEMP: RT

NACL-0011b
ETCH NAME: Heat
TYPE: Thermal, dislocation
COMPOSITION:
x .... heat
DISCUSSION:
NaCl, (100) cleaved wafers used in a thermal etch pit study. Wafers placed on a quartz plate in vacuum, apply current parallel to (100), and use heat lamp radiation outside of vacuum chamber to initiate defects. A 30 g $HgCl_2$:EOH solution used to develop pits (NACL-0004a). Pit density was on the order of 4×10^6 cm^2.
REF: Ibid.
TIME:
TEMP: Approx. 800°C

NACL-0003b
ETCH NAME:
TYPE: Acid, dislocation
COMPOSITION:
3 g .... $Fe(NO_3)_2$
1000 ml .... C_2H_5OH (EOH)
DISCUSSION:
NaCl, (100) wafers used in a study of dislocations and defects. Prepare wafers by (1) polish dip in EOH; (2) rinse in CCl_4. Use solution shown to develop dislocations, and rinse in CCl_4.
REF: Ibid.
TIME: 1 min
TEMP: RT

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NACL-0007b
ETCH NAME: Barber etch — TIME:
TYPE: Acid, preferential — TEMP:
COMPOSITION:
1 .... HCl
50 .... HAc
1 .... $FeCl_3$, sat. sol.
1 .... H_2O
DISCUSSION:
NaCl, (100) wafers used in a dislocation study. Several etchants evaluated, and the one shown gave similar results to those of Moran's etch and the "W" etch.
REF: Ibid.

NACL-0007c
ETCH NAME: Cook's etch — TIME:
TYPE: Acid, preferential — TEMP:
COMPOSITION:
x .... CH_3CH_2COOH (propionic acid)
x .... CH_3COOH (HAc)
DISCUSSION:
NaCl, (100) wafers used in a dislocation study. Solution shown gave results similar to the "A" etch (NACL-0024).
REF: Ibid.
NACL-0028: Cook, J S — *J Appl Phys*, 32,2492(1961)
NaCl, (100) wafers used in a defect study. Reference for Cook's etch.

NACL-0007d
ETCH NAME: "W" etch — TIME:
TYPE: Acid, preferential — TEMP:
COMPOSITION:
x .... $HgCl_2$
x .... EOH
DISCUSSION:
NaCl, (100) wafers used in a dislocation study. Solution can be used to develop dislocations with varying amounts of $HgCl_2$. See NACL-0004a.
REF: Ibid.
NACL-0024: Gilman, J J & Johnston, W E — *Dislocations in Crystals*, John Wiley & Sons, New York, 1956
Reference for "A" and "W" etches for NaCl, and can be applied on other alkaline halides.

NACL-0007e
ETCH NAME: "A" etch — TIME:
TYPE: Acid, preferential — TEMP:
COMPOSITION:
x .... LiF
x .... H_2O
x .... EOH
DISCUSSION:
NaCl, (100) wafers used in a dislocation study. Results with this solution are similar to those of the Cook's etch. See NACL-0007c.
REF: Ibid.

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NACL-0022
ETCH NAME: Ferric chloride
TYPE: Salt preferential
COMPOSITION:
x .... x% $FeCl_3$
DISCUSSION:
NaCl, (100) wafers used in a dislocation development study of ionic crystals. Solution used as a dislocation etch on NaCl, KCl, and other alkali halides.
REF: Gilman, J J — *J Appl Phys*, 32,739(1961)

TIME:
TEMP:

NACL-0018
ETCH NAME:
TYPE: Acid, dislocation
COMPOSITION:
100 ml .... HAc
66 ml .... $FeCl_3$, sat. sol.
DISCUSSION:
NaCl, (100) cleaved wafers subjected to partial X-ray irradiation in a study of plastic flow. Solution used to develop dislocations after irradiation.
REF: Alvarez-Rivas, J L & Ahullo-Lopex, F — *Phil Mag*, 12,205(1965)

TIME:
TEMP:

NACL-0009
ETCH NAME:
TYPE: Acid, dislocation
COMPOSITION:
4 g .... $FeCl_3$
1000 ml .... HAc
DISCUSSION:
NaCl, (100) cleaved wafers used in a dislocation etch study. After etching in solution shown, rinse in acetone, and blow dry with warm air.
REF: Mendelson, S — *J Appl Phys*, 32,1578(1961)

TIME: 30 sec
TEMP: RT

NACL-0006
ETCH NAME: Freon
TYPE: Gas, cleaning
COMPOSITION:
x .... Freon, vapor
DISCUSSION:
NaCl, (100) blanks cleaved from natural "rock salt", and muscovite mica, (0001) blanks also cleaved. Blanks used as substrates for thin film gold and silver evaporation in a material structure study. Liquids were not used for substrate cleaning as they adsorb on mica, and attack NaCl. Blow surfaces clean with Freon, only.
REF: Chopea, K I — *J Appl Phys*, 37,2049(1960)

TIME:
TEMP:

NACL-0012
ETCH NAME: Air
TYPE: Gas, defect
COMPOSITION:
x .... air, moist
DISCUSSION:
NaCl, dendritic growth and development of defects. Dendrites of NaCl, KCl and NaCl:KCl mixture were formed by placing the salt solutions on a glass plate then rapidly cool in air

TIME:
TEMP:

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(LN_2?). Structure developed around dislocations, which may be due to etching action of moist air. Dislocation centers often contained dark spots — possibly due to impurities or other salts acting as nucleating agents.
REF: Forty, A J & Gibson, J C — *Acta Metall*, 6,137(1958)

NACL-0037
ETCH NAME: Nitrogen — TIME:
TYPE: Gas, embrittlement — TEMP:
COMPOSITION:
x .... N_2(O_2, O_3 or air)
DISCUSSION:
NaCl, (100) crystals subjected to the atmospheres shown used in a study of brittleness. Results showed (1) N_2 and O_2 had no effect on ductility; (2) crystals aged in air will be brittle in summer — ductile in winter; (3) subject a ductile crystal to O_3 (ozone) or O_2 and it will become brittle.
REF: Machin, E S & Murray, G T — *J Appl Phys*, 30,1731(1959)

NACL-0030
ETCH NAME: Gold — TIME:
TYPE: Metal, decoration — TEMP:
COMPOSITION:
x .... Au
DISCUSSION:
NaCl, (100) wafers. Evaporate thin film gold on surfaces, then over-coat with carbon thin film. The combined carbon/gold layer can be peeled away as the gold adheres well to the carbon. Beads of gold will remain on the NaCl surface along cleavage and slip steps. Method can be applied to other ionic crystals.
REF: Bassett, G A — *Phil Mag*, 3,1042(1958)
NACL-0038: Amelinck, S — *J Appl Phys*, 29,1110(1958)
NaCl, as single crystal whiskers. Gold evaporated, then diffused into material to delineate dislocations by gold decoration. Used on other alkaline halides.

PHYSICAL PROPERTIES OF SODIUM NITRITE, $NaNO_2$

Classification	Nitrite
Atomic number	11, 7 & 8
Atomic weight	69.01
Melting point (°C)	271
Boiling point (°C)	320
Density (g/cm^3)	2.168
Hardness (Mohs — scratch)	1.5—2
Crystal structure (hexagonal — rhombohedral)	(10$\bar{1}$1) rhomb
Color (solid)	Yellow
Cleavage (dodecadral)	(101)

SODIUM NITRITE, $NaNO_2$

General: Does not occur as a natural compound, but there are several natural minerals with the NO_3^- radical, such as soda niter, $NaNO_3$ and niter, KNO_3, both referred to as saltpeter, and soda niter as Chile saltpeter. There are other nitrates of calcium, magnesium, copper, cobalt as hydrates. Soda niter has major industrial use in the manufacture of nitric acid, HNO_3 and, along with ammonia, NH_3, as part of the initial reactants, one by-product

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is NO_2 which, in warm water, produces HNO_3 + NO. The yellow fuming (72%) and red fuming (74%) nitric acid colors are due to increasing concentration of NO_2 in the solutions. Nitrates/nitrites are important fertilizers; they also have medicinal applications.

Technical Application: Sodium nitrite is used as a chemical additive in some etching and cleaning solutions. It has been grown as a single crystal in the Solid State field and studied for ferroelectricity and other parameters. The material is hygroscopic and requires handling in a dry, inert atmosphere of nitrogen or argon.

Etching: Water, ammonia, alcohols.

SODIUM NITRITE ETCHANTS

NAN-0001
ETCH NAME: Ammonia
TYPE: Base, removal
COMPOSITION:
x .... NH_3
DISCUSSION:

TIME:
TEMP: RT

$NaNO_2$ single crystals grown in a study of ferroelectricity in the compound. Authors say the material was yellowish and transparent and cleaves easily along (101) planes. (*Note:* In that the material is hexagonal system, rhombohedral division, cleavage may have been $(10\bar{1}0)$ prismatic?)
REF: Sawada, S et al — *Phys Rev Lett,* 1,320(1958)

PHYSICAL PROPERTIES OF SPINEL, $MgAl_2O_4$

Classification	Oxide
Atomic numbers	12, 13, & 16
Atomic weight	142.3
Melting point (°C)	2135 (2105)
Boiling point (°C)	
Density (g/cm^3)	3.5—4.1
Refractive index (n=) natural	1.7155—2.00
(n=) artificial	1.723 (1.7202)
Dielectric constant (e=)	8.4
Thermal conductivity (cal/cm-sec-°C) 25°C	0.035
Coefficient of linear thermal expansion ($\times 10^{-4}$ cm/cm/°C) 25—800°C	7.45
Dissipation factor (tan)	10^{-3}—10^{-4}
Hardness (Mohs — scratch)	8
Crystal structure (isometric — normal)	(111) octahedron, fcc
Color (solid)	Red/green/black
Cleavage (octahedral — imperfect)	(111)

SPINEL, $MgAl_2O_4$

General: The natural mineral spinel, $MgAl_2O_4$ is classified into three categories by metal element inclusion as aluminum, iron or chromium spinel. Note that magnetite, Fe_3O_4 (magnetic iron ore) also is classed as a spinel. Chemically, they are oxygen-salts of the metals, as aluminates, ferrates, manganates. In industry the material is used as a high temperature ceramic, and the transparent colored varieties as gem stones: spinel ruby; Balas ruby; rubicelle (yellow/orange-red); and almandine (violet).

Technical Application: There has been some use in Solid State processing as a substrate, similar to silicon-on-sapphire (SOS), with thin film epitaxy growth in evaluation of structure

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and possible devices. Both natural and artificial spinels have been studied, the latter grown from molten fluxes, but the primary use is still as gem stones.

Etching: H_2SO_4, molten borax. H_2 gas.

SPINEL ETCHANTS

SPI-0001a
ETCH NAME: Sulfuric acid — TIME:
TYPE: Acid, polish — TEMP: RT
COMPOSITION:
x .... H_2SO_4, conc.
DISCUSSION:

$MgAl_2O_4$, (100) and (111) wafers used as substrates for silicon epitaxy growth for comparative data between silicon-on-sapphire (SOS) and silicon on spinel. Solution is a slow removal and polish etch for spinel. Material was artificially grown by a flux technique.
REF: Cullen, G W et al — *RCA Rev*, 70,355(1970)
SPI-0002: Dana, E S & Ford, W E — *A Textbook of Mineralogy*, 4th ed, John Wiley & Sons, New York, 1950, 488

$MgAl_2O_4$, natural crystals. Material is soluble with difficulty.

SPI-0002b
ETCH NAME: Borax — TIME:
TYPE: Molten flux, preferential — TEMP: 60—75°C
COMPOSITION:
x .... $Na_2B_4O_7.10H_2O$
DISCUSSION:

$MgAl_2O_4$, natural crystals. Slowly soluble in borax molten flux.
REF: Ibid.

SPI-0003
ETCH NAME: — TIME:
TYPE: Molten flux, decomposition — TEMP: Molten
COMPOSITION:
3 .... $NaBO_3$
1 .... $CaCO_3$
DISCUSSION:

$MgAl_2O_4$ single crystals were flux grown as clear octahedrons, (111). The eutectic flux shown is used to decompose the spinel — treat melt with HCl, then add excess EDTA and boil to complex the aluminum. Diamond saw cut wafers (111), (100) and (110) were mechanically polished with 30 μm boron carbide, then down to a final polish with 0.3 μm alumina. X-ray Laue photographs used to oriented crystals for cutting. A study of optical and dielectric properties.
REF: Wang, C C & Zanaucchi, P J — *J Electrochem Soc*, 118,586(1971)

SPI-0004
ETCH NAME: Hydrogen — TIME:
TYPE: Gas, preferential — TEMP: 1150°C
COMPOSITION:
x .... H_2
DISCUSSION:

$MgO.Al_2O_4$, (111) blanks used as substrates for epitaxy growth of (111) silicon thin films. Substrates were flame fusion, flux and CZ grown single crystals in a comparative

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study of flame fused sapphire, Al_2O_3 blanks used for SOS structures. Hole mobility used as measurement criterion with SEM study of 1.5 μm silicon epitaxy thick deposits. Substrates were vacuum fired to clean or silicon was deposited and removed — three cycles — with hole mobility improving and stabilizing after the third deposition/removal to remove impurities from the substrates. Spinel shows a better crystallographic match with silicon than does sapphire, e.g., cubic vs. hexagonal crystal systems.
REF: Nanasevit, H M & Simpson, W I — *J Electrochem Soc,* 118,644(1971)

SPI-0005
ETCH NAME: TIME:
TYPE: Solvents, cleaning TEMP:
COMPOSITION:
(1) x .... TCE (2) x .... H_2O (3) x .... MeOH
DISCUSSION:
$MgAl_2O_4$, (111) wafers cut from CZ grown single crystals ingots for use as substrates for epitaxy deposition of ZnSe, ZnTe, AnS, CdS, and CdSe oriented thin films. In all cases, thin film orientation was (111). Solutions shown were used in the order shown to clean spinel surfaces prior to epitaxy. Also evaluated Verneuil grown single crystal Al_2O_3 and flux grown BeO. Authors note results are comparable to those observed on GaAs epitaxy.
REF: Cullen, G W & Wang, C C — *J Electrochem Soc,* 118,640(1971)

PHYSICAL PROPERTIES OF STEEL, Fe_3C

Classification	Iron compound
Atomic numbers	26 & 6
Atomic weight	179.53
Melting point (°C)	1837
Boiling point (°C)	
Density (g/cm³)	7.4
Hardness (Mohs — scratch)	5—6
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Steel grey
Cleavage (cubic)	(001)

STEEL, Fe_3C

General: The physical properties shown are for iron carbide called cementite as an artificial compound as it does not occur as a natural mineral. The Fe_3C crystals are part of white cast iron structure with 2—4% carbon. If it is cooled slowly it becomes grey cast iron with separation of some of the cementite to iron and graphite. Native iron occurs in small quantity, and usually has traces of other metal elements such as nickel. Some meteoric iron contains $FeNi_3$, and there are over 250 iron containing minerals as oxides, silicates and phosphates, many of which are hydrates.

The Hittites of Asia Minor (now part of Turkey) are considered to be the the first to have smelted iron for use as weapons, circa 1350 B.C., although there are reports that iron tools may have been in use by the Egyptians as far back as 3500 B.C., about the time they developed soda-lime glass. This initial iron was wrought iron with low carbon content, as a fibrous structured, malleable mass of great toughness and tensile strength. It is still used for horseshoes, decorative fencing, and objets d'art.

As the Iron Age progressed in the Mediterranean area with kiln firing of iron oxides with charcoal, the inclusion of carbon in the melt led to the first forms of steel. During the Middle Ages and medieval period of Europe, both Damascus and Toledo steels became the

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best forms of steel for swords and armor, even though large castings of bronze were still used for the making of cannon as late as the 16th century as hand iron/steel weapons were being developed.

Copper and bronze plate have been used on the hulls of wooden ships since ancient times, but it was not until the American Civil War that the first of the iron ships were built (the Monitor and the Merrimac), even though the Industrial Revolution in England was already using steam engines, rails and railroad cars, as well as iron and steel in building construction in the early 1800s.

Today there are hundreds of types of irons and steels graded by SAE numbers and other classifications. Two of the most widely used series are 302 and 304 steels in equipment construction, such as vacuum systems. The grades range from steels, magnetic steels, to stainless steel. Construction irons and steels included buildings, bridges, heavy duty equipment, vehicles, and "tin" cans. The latter is thin sheet steel plated with tin, the tin being highly inert to vegetable and meat acids.

As a very brief resume of iron and steel fabrication: Major iron ores are the iron oxides, such as hematite, Fe_2O_3; magnetite, Fe_3O_4; siderite, $FeO.xH_2O$ and the "iron pyrites" as a group — pyrite, FeS_2 as better than 80% of all iron ore mined in the world due to its abundance. The highest grade oxide ores come from Sweden as they are particularly low in other element contamination. Swedish iron is mostly magnetic iron from the original Achean Shield solidification of the earth's crust.

The iron ores are initially kiln fired and roasted in a vertical blast furnace to obtain "pig iron" (4% carbon; 3% silicon; phosphorus and sulfur. The phosphorus makes it brittle when cold; the sulfur, brittle when hot). Cast iron is then made from the initial pig (2—4% carbon) as white cast iron with a high cementite, Fe_3C content; and as grey cast iron with some cementite separation into iron and graphite. Wrought iron is made by removal of carbon by air blowing through the iron mass (Bessemer process).

Billets of ferro-silicon are obtained in the reduction of sand and other silicates, and there are starter billets of ferro-manganese, ferro-phosphorus, ferro-chromium, etc., which are then added to the pig, cast or wrought iron to produce the particular variety of iron and/or steel and stainless steel.

Silicon iron (15% Si) is brittle but very acid resistant and used as holding vessels, crucibles, piping and linings in acid processing equipment.

Manganese iron is tough. With 1—7% Mn used for machine tooling; with >7% Mn for heavy duty equipment, such as rock crushers or for safes.

Nickel steel has great tenacity and is a primary construction steel with 0.5% Ni, and for armor plate with 3—5% Ni.

Tungsten steel holds a temper at high temperatures, such that it is used for lathe cutting tools, knives, and scalpels. Both molybdenum and cobalt steels are similar.

Chromium steel with 12—15% Cr is corrosion resistant and used for tools, equipment and armor plate. With >18% Cr it is stainless steel, SST.

In construction — bridges and building girders, ship plate and so forth — the iron is coated with red lead to prevent rusting prior to further painting. There is on-going study of irons and steels as they are improved against atmospheric corrosion, fatigue failure, etc. Many of the etchant solutions in the ASTM 407-70 pamphlet were developed for metallographic study after such testing with mechanical lapping, polishing and preferential etching.

Many of the common iron and steel products of the past are being replaced by other materials — lighter weight metals, plastics and ceramics — but iron skillets, steel cookware, knives, forks, and spoons of steel alloys, and other such items are still in demand. The canning industry is a major user of thin sheet steel that is tin plated as "tin cans", and construction irons and steels for vehicles, buildings, bridges are still major users. In chemical processing and medicine, stainless steel beakers, forceps, and tweezers (acid resistant and nonmagnetic), as well as decorative items in the jewelry trade are still in wide use.

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Technical Application: Steels or irons are not directly used in the fabrication of Solid State devices themselves, but widely used in all processing and testing equipment, to include chemical processing or material handling with forceps, tweezers, SST beakers, and so forth. Device package construction may include steels, and special contact wires (dumet), and some magnetic steels. Much of the equipment and etchants used in Solid State originated in the iron and steel, rock and mineral, and glass industries.

Ferrites as resistor material are used as discrete devices in conjunction with circuit substrate assemblies in microwave device construction and are presented here, in the Ferrite section.

The amount of literature published on the study of irons and steels is second only to that of glass. There is still on-going study of steel alloys, some grown as single crystals. The etchants shown in the following section are only a brief cross-section of those available. Where the material is a steel, formats are numbered ST-xxxx; where an iron, as FE-xxxx, and both designations may be shown under the Steel or Iron sections in this book.

Etching: Varies with type steel. HNO_3:EOH, picric acid:EOH, $FeCl_3$, HCl.

STEEL ETCHANTS

ST-0001a
ETCH NAME: Al-7
TYPE: Acid, macro-etch
COMPOSITION:
50 ml .... HCl
50 ml .... H_2O
TIME: Dip
TEMP: 160—175°C
DISCUSSION:
Fe_3C steel and iron specimens. Solution used to develop micro-structure.
REF: Pamphlet — *A/B Metal Digest,* 22(3),14(1983) — Buehler Ltd

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ST-0001b
ETCH NAME: Kaling's reagent
TYPE: Acid, preferential
COMPOSITION:
33 ml .... HCl
33 ml .... EOH
33 ml .... H_2O
1.5 g .... $CuCl_3$
TIME: Swab
TEMP: RT
ASTM: #95
DISCUSSION:
SST 400 Series. Solution used as a structure development etch.
REF: Ibid.
ST-0017b: Pamphlet — *ASTM,* E407-70
Reference for ASTM number for Kaling's reagent shown as 40 ml HCl:80 ml EOH(MeOH):40 ml H_2O:2 g $CuCl_2$.

ST-0001c
ETCH NAME: Picral
TYPE: Acid, preferential
COMPOSITION:
10 g .... picric acid
100 ml .... EOH
TIME: Swab
TEMP: RT
ASTM: #76

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DISCUSSION:

Steels, high carbon and high alloy types. Solution used as a structure etch applied by swabbing the surface.

REF: Ibid.

ST-0017c: Pamphlet — *ASTM*, E407-70

Reference for ASTM number. Same solution as shown under ST-0001c, but MeOH can replace EOH.

ST-0001d

ETCH NAME: Potassium bisulfate

TYPE: Acid, preferential

TIME: Dip

TEMP: RT

COMPOSITION:

10 g .... potassium metabisulfate

90 ml .... H_2O

DISCUSSION:

Steel, alloys with untempered martensite. Solution used to develop micro- and macrostructure.

REF: Ibid.

ST-0001e

ETCH NAME: Vilella's reagent

TYPE: Acid, preferential

TIME: Swab

TEMP: RT

ASTM: #80

COMPOSITION:

5 ml .... HCl

1 g .... picric acid

100 ml .... EOH

DISCUSSION:

Steels, high carbon and alloys types. Solution used as a structure etch.

REF: Ibid.

ST-0017a: Pamphlet — *ASTM*, E407-70

Reference for ASTM number. Same solution as shown in ST-0001e, but EOH can be replaced with MeOH.

ST-0001f

ETCH NAME: Nital

TYPE: Acid, preferential

TIME: Swab

TEMP: RT

ASTM: #74

COMPOSITION:

2 ml .... HNO_3

98 ml .... EOH

DISCUSSION:

Steel, carbon type. Solution is a general cleaning and preferential etch to develop structure.

REF: Ibid.

ST-0017d: Pamphlet — *ASTM*, E407-70

Reference for ASTM number. Solution shown as 1—5 ml HNO_3:100 ml EOH or MeOH.

ST-0001g

ETCH NAME:

TYPE: Acid, preferential

TIME: 1—10 min

TEMP: RT

ASTM: #81

COMPOSITION:

2 g .... picric acid

1 g .... sodium tridecylbenzene sulfonate

100 ml .... H_2O

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DISCUSSION:
Steels, carbon and alloy types. Solution used to develop prior austenitic grain boundaries in martensite and bainite structures.
REF: Ibid.
ST-0017e: Pamphlet — *ASTM*, E407-70
Reference for ASTM number.

ST-0001h
ETCH NAME: Chromic acid
TYPE: Electrolytic, preferential
COMPOSITION:
10 g .... CrO_3
90 ml .... H_2O

TIME:
TEMP:
ANODE: Steel
CATHODE:
POWER: 3—5 V
ASTM: #18

DISCUSSION:
Steel, 300 series. Solution used to develop structure.
REF: Ibid.

ST-0001i
ETCH NAME:
TYPE: Electrolytic, preferential
COMPOSITION:
15 ml .... HCl
10 ml .... HAc
10 ml .... HNO_3
5 ml .... glycerin

TIME:
TEMP:
ANODE: Steel
CATHODE:
POWER: 3—5 V

DISCUSSION:
SST 300 series. Solution used as a structure etch.
REF: Ibid.

ST-0002a
ETCH NAME:
TYPE: Acid, preferential
COMPOSITION:
x .... CrO_3
x .... H_2O
x .... glycerin

TIME:
TEMP:

DISCUSSION:
SST 316. Solution used to develop corrosion fatigue cracks.
REF: Pamphlet — *A/B Metal Digest*, 22(2),17(1983) — Buehler Ltd

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ST-0013b
ETCH NAME: Persulfate etch
TYPE: Acid, contrast
COMPOSITION:
x .... $(NH_4)_2S_2O_8$
100 ml .... H_2O

TIME: Dip
TEMP: RT

DISCUSSION:
Steel alloys containing carbides. Immerse in solution until iron matrix darkens to develop carbide material by contrast.
REF: Ibid.

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ST-0002b
ETCH NAME: Oxalic acid, diluted
TYPE: Electrolytic, preferential
COMPOSITION:
x .... oxalic acid
x .... H_2O
DISCUSSION:
SST 304. Used to develop stress corrosion cracks.
REF: Ibid.
TIME:
TEMP:
ANODE:
CATHODE:
POWER:

ST-0003
ETCH NAME: Nitric acid, dilute
TYPE: Acid, cleaning
COMPOSITION:
x .... HNO_3
x .... H_2O
DISCUSSION:
SST substrates used for deposition of WC in a study of wear resistance, structure and adhesion. First, substrates were diamond-paste polished and then etched clean in dilute nitric acid. Follow with water rinse, then alcohol, then alcohol with ultrasonic agitation. WC deposited by RF sputter under acetylene with a tungsten sheet sputter target.
REF: Srivastava, P K et al — *J Electrochem Soc*, 131,1260(1984)
TIME:
TEMP:

ST-0004: Walker, P — personal application, 1970—1980.

SST 316 evaporation bell jars and vacuum tooling. Soak and scrub with a steel brush using a $1HNO_3$:$10H_2O$ or a 1HCl:$10H_2O$ solution for general cleaning and removal of excess evaporated metals build-up. Solutions used at RT up to 30 min scrubbing. Follow with heavy water flushing and toweling wipe-down if black smut is present, then final rinse in MeOH and air dry or dry under IR heat lamp.

ST-0005
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
x .... 5×10^3 *M* ferric perchlorate
x .... 0.75 *M* perchloric acid
*x .... lithium perchlorate

*Use to control ion strength at 1.0.

DISCUSSION:
Steel blanks. First, clean blanks in hot caustic (10—15% NaOH) and DI water wash thoroughly. This pre-cleaning step increases the etch removal rate by about 15%. This is a major article on the cleaning of steels.
REF: Maynard, R B — *RCA Rev*, 45,58(1984)
TIME:
TEMP:

ST-0006
ETCH NAME: Ferric chloride
TYPE: Acid, removal
COMPOSITION:
x .... $FeCl_3$ (30° Bé)
x .... *HCl

*Use to reduce concentration.
TIME: 2—6 min
TEMP: Various

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DISCUSSION:

Steel, low carbon blanks. First, pre-clean blanks by soaking in caustic (10—15% NaOH) with thorough water washing after soak. Start solution as shown with 30° Bé and increase concentration by boiling off water; reduce concentration by adding HCl.

REF: Maynard, R B et al — *RCA Rev*, 45,71(1984)

ST-0007a

ETCH NAME: TIME:

TYPE: Acid, removal TEMP:

COMPOSITION:

4 .... HCl

1 .... HNO_3

DISCUSSION:

Steel, high-speed blanks. Used as a general etch for steels. Increase nitric acid content to control rate. Also used on nickel and brass.

REF: Oberg, E & Jones, F D — *Machinery's Handbook*, 4th ed, The Industrial Press, New York, 1951

ST-0007b

ETCH NAME: TIME:

TYPE: Acid, removal/cleaning TEMP:

COMPOSITION:

1 .... HCl

2 .... HNO_3

DISCUSSION:

Steel, hard. Shown as a cleaning and removal solution for hard steel.

REF: Ibid.

ST-0007c

ETCH NAME: Nitric acid, dilute TIME:

TYPE: Acid, removal/cleaning TEMP:

COMPOSITION:

1 .... HNO_3

4 .... H_2O

DISCUSSION:

Steel, carbon. Shown as a removal and cleaning solution for carbon steels. Water content is varied with increase or decrease of carbon content. Added carbon increases steel hardness.

REF: Ibid.

ST-0008

ETCH NAME: TIME:

TYPE: Acid, cleaning for plating TEMP:

COMPOSITION:

(1) Degrease: toluene

(2) Mechanical lap/polish: 1/4/0 emery paper

(3) Electrolytic clean: SST part cathode: 1%$NaCO_3$. H_2O rinse

(4) Acid dip: 5% H_2SO_4, RT. H_2O rinse

(5) Acid polish: 30 sec, 30—35°C (acid not shown)

(6) Alkali clean: 10% NaCH, RT, 2—5 sec, H_2O rinse

(7) Acid dip: 5% H_2SO_4 with agitation to remove Fe(OH). H_2O rinse

(8) Into plating solution still wet

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DISCUSSION:

Steel, (100) single crystal specimens. Cleaning sequence shown used on specimens prior to tin plating in a morphology study of $FeSn_2$.

REF: Lunder, C & Murry, M V — *J Electrochem Soc*, 111,348(1964)

ST-0009a

ETCH NAME: Ammonium persulfate

TYPE: Acid, removal

TIME:

TEMP: 50—60°C

COMPOSITION:

x .... 10% ammonium persulfate

DISCUSSION:

Fe, specimens. Solution used as a general removal and surface cleaning etch.

REF: Berglund, T & Dearden, W H — *Metallographer's Handbook of Etching*, Pitman & Sons, London, 1931

ST-0009b

ETCH NAME: Sulfuric acid, dilute

TYPE: Acid, polish

TIME: 6—24 h

TEMP: Hot

COMPOSITION:

x .... 10—20% H_2SO_4

DISCUSSION:

Fe, specimens. Solution used as a polishing etch.

REF: Ibid.

ST-0009c

ETCH NAME: Nital

TYPE: Acid, preferential

TIME:

TEMP:

COMPOSITION:

x .... 10—15% HNO_3

x .... EOH or H_2O

DISCUSSION:

Fe specimens. As a general cleaning solution with some preferential attack. (*Note:* With EOH, solution is called Nital.)

REF: Ibid.

ST-0009d

ETCH NAME: Hydrochloric acid

TYPE: Acid, removal

TIME: 10—30 min

TEMP:

COMPOSITION:

1 .... HCl

1 .... H_2O

DISCUSSION:

Fe, specimens. Used as a cleaning and removal solution with some preferential attack.

REF: Ibid.

ST-0009e

ETCH NAME: Picral

TYPE: Acid, removal

TIME: 4—5 h

TEMP: RT

COMPOSITION:

x .... 3% picric acid

x .... EOH

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DISCUSSION:
Fe specimens. Use as a cleaning and removal solution with some preferential attack.
REF: Ibid.

ST-0010a
ETCH NAME: Nital
TYPE: Acid, preferential
COMPOSITION:
x .... 2% HNO_3
x .... EOH
DISCUSSION:
Fe, single crystal whiskers etched in this solution to observe dislocations.
REF: Coleman, R V — *J Appl Phys*, 29,1487(1958)

TIME:
TEMP:

ST-0010b
ETCH NAME: Picral
TYPE: Acid, preferential
COMPOSITION:
x .... picric acid
x .... EOH
DISCUSSION:
Fe, single crystal whiskers etched in this solution to observe dislocations.
REF: Ibid.

TIME:
TEMP:

ST-0011
ETCH NAME:
NAME: Electrolytic, polish
COMPOSITION:
15 .... H_2SO_4
63 .... H_3PO_4
10 .... CrO_3
12 .... H_2O
DISCUSSION:
Fe, polycrystalline discs used as substrates for deposition of carbon thin films. First, vacuum anneal at 82°C, 8 h at 10^{-6} Torr. Then electropolish in the solution shown. Rinse in DI H_2O; rinse in NaOH and final rinse in DI H_2O.
REF: Brown, D W — *J Vac Sci Technol*, 583(1985)

TIME:
TEMP: 50°C
ANODE:
CATHODE:
POWER:

ST-0012a
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
x .... HAc
x .... CrO_3
x .... H_2O
DISCUSSION:
Fe/Ni, martensitic transformation study of faulting and defects. Solution used to electropolish specimen.
REF: Reed, R P — *Acta Metall*, 14,1493(1966)

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

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ST-0012b
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
1 .... H_2O_2
2 .... H_3PO_4
DISCUSSION:
Fe/Ni, martensitic transformation study of faulting and defects. Solution used to polish surfaces.
REF: Ibid.

TIME:
TEMP: 100°C

ST-0013a
ETCH NAME:
TYPE: Acid, microetch
COMPOSITION:
100 ml .... C_2H_2OH
60 ml .... HCl
20 g .... $FeCl_3$
DISCUSSION:
Fe cast alloys. Used as a general microetch. Will develop concentrations of Steatite.
REF: *A/B Met Dig*, 21,12,23(1983) — Buehler Ltd

TIME: Swab
TEMP: RT

ST-0014
ETCH NAME: Water
TYPE: Acid, preferential
COMPOSITION:
x .... H_2O, distilled
DISCUSSION:
Fe, single crystal spheres $^3/_8$" in diameter with a (111), (110) and (100) flats cut on surfaces. A study of pitting by pure water on different orientations of iron. Pitting was lowest for high purity iron and the (110) orientation showed the most severe pitting.
REF: Kruger, J — *J Electrochem Soc*, 106,736(1959)

TIME: Variable
TEMP: RT

ST-0015
ETCH NAME:
TYPE: Acid, selective
COMPOSITION:
180 g .... NaOH
30 g .... $KMnO_4$
1000 ml .... H_2O
DISCUSSION:
Stellite specimens. Solution will selectively attack iron and cobalt in steel alloys.
REF: Reed, R D — *Electrochem Technol*, 2,192(1964)

TIME:
TEMP: 105°C

ST-0016
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
2 .... $HClO_4$
7 .... EOH
1 .... glycerin

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

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DISCUSSION:

Fe:C(1.5%):Ni(5%) alloy specimens used in a study of martensite growth. Solution is also slightly preferential and can be used as a macro-etch.

REF: Priestner, R & Glover, S G — *Acta Metall,* 5,537(1957)

ST-0018

ETCH NAME: Glyceregia

TYPE: Acid, cleaning

TIME: Wipe

TEMP: RT

ASTM: #87

COMPOSITION:

20—50 ml .... HCl
10 ml .... HNO_3
30 ml .... glycerin

DISCUSSION:

Steel and SST vacuum system parts. A plastic sponge wetted with solution used to wipe clean steel surfaces after using other etch solutions to remove metal evaporation contamination. Follow with water washing. ASTM E407-70 number is for original Glyceregia formulation.

REF: Menth, M — personal communication, 1980

ST-0001j: Ibid.

SST 316 specimens. Solution used as a micro- and macro-etch in the study of corrosion fatigue.

ST-0019a

ETCH NAME:

TYPE: Acid, removal

TIME: ("A") sec ("B") sec

TEMP: RT 5°C

COMPOSITION:

(A) 140 ml .... Picral*
12 ml .... 10% $FeCl_3$
4 ml .... Photoflo**

(B) 2.5 .... HF
40 .... H_2SO_4
58.5 .... H_2O

*xPicric acid:xEOH

**Eastman Kodak.

Mix 1:1 with liquid detergent.

DISCUSSION:

Steels, as coupons of different types used in a structure study. Solutions used in order for general removal and thinning. Solution ("A") is rapid; solution ("B") for final slow removal. Steel varieties evaluated were C, N, Ti, Nb, S, P, Si, Mn, Al, and Mo alloy materials.

REF: Campo, G O et al — *Metallography (MEIJAP),* 16,287(1983)

ST-0019b

ETCH NAME:

TYPE: Acid, thinning

TIME:

TEMP: RT

COMPOSITION:

5 .... HF
80 .... H_2O_2
15 .... H_2O

DISCUSSION:

Steel, blanks used in a structure study. Solution used to thin specimens for microscope study. See ST-0019a.

REF: Ibid.

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ST-0019c
ETCH NAME:
TYPE: Electrolytic, jet thin
COMPOSITION:
x .... $HClO_4$
x .... MeOH
TIME:
TEMP: RT
ANODE: Steel
CATHODE:
POWER: 20 V & 1.5 A/cm^2
DISCUSSION:
Steel blanks used in a structure study. Solution used as shown or with added 2% Nital ($xHNO_3$:xEOH). See ST-0019a, ST-0017d.
REF: Ibid.

ST-0019d
ETCH NAME: Nital
TYPE: Electrolytic, removal
COMPOSITION:
x .... 5% Nital*
TIME:
TEMP: RT
ANODE: Steel
CATHODE:
POWER: 10 V

*ASTM #74

DISCUSSION:
Steel, blanks used in a study of structure. Solution used as a general removal etch in thinning specimens for microscope study. See ST-0019a and ST-0017d.
REF: Ibid.

ST-0020
ETCH NAME:
TYPE: Alkali, cleaning
COMPOSITION:
x .... alkali detergent (pH 11)
TIME:
TEMP:
DISCUSSION:
Steel, 304 type blanks used in a material study. Prepare specimens: (1) vapor degrease in perchloroethylene, C_2Cl_4; (2) ultrasonic in alkali solution shown; (3) rinse in cold dmH_2O; (4) dry in air oven at 150°C. (*Note:* The mixed alkali solution could be KOH:Ivory soap:H_2O and adjust pH with NH_3 or NH_4OH.)
REF: Erlandsson, R — *J Vac Sci Technol*, 19,748(1981)

ST-0021
ETCH NAME: Trichloroethylene
TYPE: Solvent, cleaning
COMPOSITION:
x .... TCE
TIME: $^1/_2$—1 h
TEMP: RT to hot
DISCUSSION:
Steel, 304 type as used in oil vacuum pumps. Solvent soak interior of pump to remove vacuum oil, then scrub with rag-stock or fiber brush as needed. Rinse with MeOH and dry with compressed air line. Pump parts and associated system hardware TCE vapor degreased.
REF: Gomez, P & Walker, P — personal application, 1973—1975

PHYSICAL PROPERTIES OF STRONTIUM, Sr

Classification	Metal (alkaline)
Atomic number	38

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Atomic weight	87.63
Melting point (°C)	770
Boiling point (°C)	1384
Density (g/cm^3)	2.6
Thermal conductance (cal/sec)(cm^2)(°C/cm)	0.090
Specific heat (cal/g) 25°C	0.176
Heat of fusion (k-cal/g-atom)	2.1
Latent heat of fusion (cal/g)	25
Heat of vaporization (cal/g)	42.3
Atomic volume (W/D)	33.7
1st ionization energy (K-cal/g-mole)	131
1st ionization potential (eV)	5.692
Electronegativity (Pauling's)	1.0
Covalent radius (angstroms)	1.91
Ionic radius (angstroms)	1.12 (Sr^{+2})
Electrical resistivity (micro-ohms cm)	22.76
Lattice constant (angstroms)	6.05
Coefficient of linear thermal expansion ($\times 10^{-6}$ cm/cm/°C) 20°C	23
Electron work function (eV)	2.74
Cross section (barns)	1.3
Vapor pressure (°C)	1111
Hardness (Mohs — scratch)	1.8
Crystal structure (isometric — normal)	(100) cube, fcc
Color (solid)	Silver yellowish
(flame)	Purple-red
Cleavage (cubic)	(001)

STRONTIUM, Sr

General: Does not occur as a free element in nature. It is a relatively rare element and, though there are a dozen minerals containing strontium, only two are of major importance: strontinate, $SrCO_3$, and celestite, $SrSO_3$, the latter being the major source of commercial quantity ore. Both are of wide occurrence in rocks of all geologic ages commonly associated with limestone, gypsum, rock salt as veins and beds, and occasionally in volcanic rocks. The pure metal is silver-white with a slightly yellowish tinge, and properties are similar to those of calcium.

The metal has limited use in industry. It is used in sugar refining, and the nitrate in pyrotechnics for the deep purple-red flame color.

Technical Application: Strontium has had limited use in Solid State material processing. It is under development and evaluation as a fluoride surface coating with similar applications to those of oxides and nitrides, and SrF_2 has been used as a substrate for optical study of thin film metals. It is also a constituent in some artificial garnets and ferrites.

Etching: Deliquesces in water. Soluble in acids, alcohols, and ammonia.

STRONTIUM ETCHANTS

SR-0001
ETCH NAME: Ammonia
TYPE: Acid, removal/polish
COMPOSITION:
x .... NH_3

TIME:
TEMP: RT

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DISCUSSION:

Sr, (100) wafers and other orientations. Ammonia is a general removal and polishing etch for this material.

REF: Vossen, J L & Kern, W — *Thin Film Processes,* Academic Press, New York, 1978

SR-0002

ETCH NAME: Hydrochloric acid — TIME:

TYPE: Acid, removal — TEMP: RT to hot

COMPOSITION:

x .... HCl, conc.

DISCUSSION:

Sr material as the carbonate strontianite, $SrCO_3$ and sulfate celestite, $SrSO_4$ are both reduced by hydrochloric acid. Both are ores of strontium and used in preparing strontium nitrate for fireworks.

REF: Dana, E S & Ford, W E — *A Textbook of Mineralogy,* 4th ed, John Wiley & Sons, New York, 1950

PHYSICAL PROPERTIES OF STRONTIUM CHLORIDE, $SrCl_2$

Classification	Chloride
Atomic numbers	38 & 17
Atomic weight	158.54
Melting point (°C)	873
Boiling point (°C)	
Density (g/cm^3)	3.05
Hardness (Mohs — scratch)	1.5—2
Refractive index (n =)	1.7
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Colorless
Cleavage (cubic)	(001)

STRONTIUM CHLORIDE, $SrCl_2$

General: Does not occur in nature as the solid chloride due to solubility in water, but can be extracted from sea water and hot springs in certain locales. The two major minerals are strontianite, $SrCO_3$, and celestite, $SrSO_4$, which are found in minable quantity. There also are phosphates and silicates.

In ore extraction of strontium in industry, it is usually obtained as the chloride, and all of the compounds are used in pyrotechnics for their brilliant red flame color. The salts also have application in raw vegetable processing.

Technical Application: The material has been studied as a single crystal for paramagnetic S-states and other morphological data, but has found little use in Solid State processing, mainly due to solubility.

Etching: Water, alcohols, and acetic acid.

STRONTIUM CHLORIDE ETCHANTS

SRCL-0001

ETCH NAME: Vacuum — TIME:

TYPE: Vacuum, cleaning — TEMP: −196°C

COMPOSITION:

x .... vacuum

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DISCUSSION:

$SrCl_2$, (100) cut wafers used in a study of paramagnetic S-states. Wafers were cleaned for study in high vacuum at LN_2 temperatures. Authors noted the solubility as shown above under "Etching".

REF: Low, W & Rosenberger, U — *Phys Rev*, 116,621(1959)

PHYSICAL PROPERTIES OF STRONTIUM FLUORIDE, SrF_2

Classification	Fluoride
Atomic numbers	38 & 9
Atomic weight	123.67
Melting point (°C)	1190
Boiling point (°C)	
Density (g/cm^3)	
Ionic radius (angstroms)	1.27
Surface free energy (ergs cm^{-2}) 273°C	473 (111)
Hardness (Mohs — scratch)	1—3
(Knoop — kgf mm^{-2})	140
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Colorless
Cleavage (cubic)	(001)

STRONTIUM FLUORIDE, SrF_2

General: Does not occur as a natural compound, though calcium fluoride, CaF_2 (fluorite) has long been used as a soldering flux and for its optical properties. Like other strontium salts, it can be used in pyrotechnics for the brilliant red strontium flame color.

Technical Application: The material has not been used in Solid State device fabrication to date, though it has been used as a single crystal substrate for thin film metal studies. It has been evaporated as a polycrystalline and oriented thin film layer on GaAs and InP wafers in evaluations of possible optical applications in device development.

Etching: Slight solubility in water; soluble in hot HCl.

STRONTIUM FLUORIDE ETCHANTS

SRF-0001

ETCH NAME: Hydrochloric acid, dilute — TIME:

TYPE: Acid, removal — TEMP:

COMPOSITION:

1 .... HCl

10 .... H_2O

DISCUSSION:

SrF_2, (100) thin film deposited on GaAs substrates above 250°C is polycrystalline; deposited below 250°C is single crystal. Photo resist patterned as square openings and E-beam annealed. Fluorine absorbs and film discolors. The films can be "washed away" with the solution shown. CdF_2 processed in the same manner is washed away with water only (CDF-0004).

REF: Sullivan, P W — *J Vac Sci Technol*, B2,202(1984)

SRF-0002: Hodgman, C D — *Handbook of Chemistry and Physics*, 27th ed, Chemical Rubber Co, Cleveland, OH, 1943, 466

SrF_2, specimens. Bulk material is soluble in hot hydrochloric acid.

SRF-0003: Tu, C W — *J Vac Sci Technol*, B2,24(1984)

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SrF_2 and $Ba_xSr_{1-x}F_2$, (001) MBE grown on InP, (001) substrates and on InP, (110) substrates with thin film oriented (110). Also studied CaF_2 and BaF_2. All are cubic fluorides.
SRF-0004: Pulker, H K — *Thin Solid Films*, 89,191(1982)

SrF_2, (100) wafers used as substrates for metallization with aluminum, chromium and silver in a study of mechanical properties of optical films. Solution can be used to clean surfaces prior to metallization.

PHYSICAL PROPERTIES OF STRONTIUM OXIDE, SrO

Classification	Oxide
Atomic numbers	38 & 8
Atomic weight	103.63
Melting point (°C)	2430 (SrO)
Boiling point (°C)	
Density (g/cm³)	4.70 (SrO)
Hardness (Mohs — scratch)	6—7 (SrO)
Crystal structure (hexagonal — normal)	$(10\bar{1}0)$ prism (w/Ga)
Color (solid)	Yellow (w/Ga)
Cleavage (basal — distinct)	(0001) (w/Ga)

STRONTIUM GALLIUM OXIDE, $SrGa_{12}O_{19}$

General: Neither the pure nor gallium doped compound occurs in nature, though the mineral rinkolite contains Sr and other elements as a titanosilicate. Like other strontium compounds in industry, the oxide can be used for pyrotechnics for its brilliant red flame color.

Technical Application: The reference here is for a strontium gallium oxide grown as a single crystal in a materials study. It may have applications as a garnet-type material. See Garnet for similar strontium and gallium containing crystals.

Etching: Soluble in alcohols, acetic acid.

STRONTIUM GALLIUM OXIDE ETCHANTS

SGO-0001
ETCH NAME: Nitric acid
TYPE: Acid, removal
TIME:
TEMP: Hot
COMPOSITION:
20 .... HNO_3
80 .... H_2O
DISCUSSION:

$SrGa_{12}O_{19}$, (0001) cleaved wafers from crystals grown by the molten flux method as hexagonal yellow single crystals. Crystals were removed from the crucible by etching with the solution shown. Structure referred to as like magnetoplumbite, Space Group P63/mmc (hexagonal), which is an iron lead manganate.
REF: Haberey, F et al — *J Cryst Growth*, 61,284(1983)

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STRONTIUM TITANATE ETCHANTS

SRTO-0001
ETCH NAME: Alumina
TYPE: Abrasive, polish
COMPOSITION:
x .... c-Al_2O_3
DISCUSSION:
$SrTiO_3$ single crystal specimens used for development of optical memory devices. Polish specimens with colloidal alumina. See KTO-0001 (potassium tantalate) for further discussion.
REF: Yamaichi, E et al — *Jpn J Appl Phys*, 23,867(1984)
SRTO-0002: Chi, J — *J Phys Soc Jpn*, 40,1371(1976)
Reference for method of flux growth of crystals.

TIME:
TEMP:

SRTO-0003
ETCH NAME: Alumina
TYPE: Abrasive, polish
COMPOSITION:
x .... Al_2O_3
x .... H_2O
DISCUSSION:
$SrTiO_3$, single crystal specimens studied for optical transmission after heat treatment. Specimens were polished on a rotating wax lap with Linde A alumina in water.
REF: Gandy, H W — *Phys Rev*, 113,795(1959)

TIME:
TEMP:

PHYSICAL PROPERTIES OF STRONTIUM TUNGSTATE, $SrWO_4$

Classification	Tungstate
Atomic numbers	38, 74, & 8
Atomic weight	335.55
Melting point (°C)	200 del.
Boiling point (°C)	
Density (g/cm³)	6.187
Hardness (Mohs — scratch)	3—4
Crystal structure (tetragonal — pyrimidal)	(210) prism
Color (solid)	Colorless
Cleavage (prismatic)	(210)

STRONTIUM TUNSTATE, $SrWO_4$

General: Does not occur in natural as a tungstate compound, though there are carbonates, phosphates and oxides of strontium as mineral species. There is no industrial application at the present time.

Technical Application: The material has been grown in Solid State studies of tungstate compounds, but has not been developed as an electronic type device, to date.

Etching: Soluble in water and dilute acids with deliquescence.

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STRONTIUM TUNGSTATE ETCHANTS

SRWO-0001
ETCH NAME: Phosphoric acid
TYPE: Acid, polish
COMPOSITION:
x .... H_3PO_4
DISCUSSION:
$SrWO_4$ single crystal specimens were used in a study of Raman frequency shifts and temperature dependence. Material referred to as having the scheelite, $CaWO_4$, tetragonal system, pyrimidal class structure. Other tungstates studied were $CaWO_4$ and $BaWO_4$.
REF: Degreniers, S — *J Phys Chem Solids*, 415,1105(1984)

TIME:
TEMP: Hot

PHYSICAL PROPERTIES OF SULFUR, S

Classification	Nonmetal
Atomic number	16
Atomic weight	32
Melting point (°C)	119 (128, rhombic)
Boiling point (°C)	444.6
Density (g/cm³)	2.07
Gas conversion point (°C + O_2)	270 (to SO_2)
Thermal conductance (cal/sec)(cm²)(°C/cm)	0.0007
Specific heat (cal/g) 25°C	0.175
Latent of heat of fusion (cal/g)	9.3
Heat of vaporization (k-cal/g-atom)	3.01
Atomic volume (W/D)	15.5
1st ionization energy (K-cal/g-mole)	239
1st ionization potential (eV)	10.357
Heat of fusion (k-cal/g-atom)	0.34
Electronegativity (Pauling's)	2.5
Covalent radius (angstroms)	1.02
Ionic radius (angstroms)	0.37 (S^{+4})
Electrical conductance (micro-ohms^{-1})	10^{-23}
Critical temperature (°C) C.T.	1040
Critical pressure (atms) C.P.	116
Critical volume (ml/g)	2.48
Electrical resistivity ($\times 10^{-16}$ ohms cm) 20°C	2
Linear coefficient of thermal expansion ($\times 10^{-6}$ cm/cm/°C) 20°C	64.13
Cross section (barns)	0.51
Vapor pressure (°C)	327.2
Hardness (Mohs — scratch)	1.5—2.5
Refractive Index (n =)	1.96—2.25
Crystal structure (orthohombic — normal) alpha	(100) pinacoid
(monoclinic — normal) beta	(100) pinacoid
(monoclinic — normal) delta	(hk0) prism
Color (solid)	Brilliant, yellow
Cleavage (cubic/octahedral/dodecahedral)	(001)/(111)/(110)

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SULFUR, S

General: Wide occurrence as a native element and noted for its brilliant yellow color. It is polymorphous with three primary crystal structures, and also is found as natural amorphous sulfur. Alpha-sulfur is orthorhombic, the most common form as it is stable at standard pressure and temperature; beta-sulfur in monoclinic, occurs when alpha-sulfur is fused, then slowly converts back to the alpha variety with time. There is a second monoclinic form that occurs as the natural mineral rosickyite (delta-sulfur) yellow-brown in color, and mu-sulfur as a black, tar-like form, and there are two other known modifications.

Sulfur was one of the elements known to ancient man and called "brimstone" from the disagreeable odor when burned to sulfur dioxide, SO_2. It is an insecticide when burned, was made as a sulfur candle, and used to fumigate buildings, clothing and people. Several compounds are formulated as creams and salves for their medical curative powers, or used as dusting powders.

As sulfur combines with all elements other than gold and platinum, there are many major ore minerals that are reduced for both the metal and sulfur content. The major sulfides are the iron pyrites group, pyrite, FeS_2, as the the major source, which may also contain a high percentage of silver, Galena, PbS, also of wide occurrence as the major ore of lead. Sphalerite, ZnS, a source of zinc and sulfur and, as it has the atypical tetrahedral unit cell, is of importance crystallographically with regard to many binary compounds, such as the compound semiconductors. Cinnabar, HgS, which occurs in small pockets, the most major world deposit at Almaden, Ciuadad Real, Spain, and the coastal ranges of California in the U.S. It is a strategic war mineral (fulminate of mercury as detonators), and is the only ore of mercury.

As sulfates, there is barite, $BaSO_4$, called "heavy spar"; with gypsum, $CaSO_4.2H_2O$, and the unhydrated anhydrite, $CaSO_4$, most important as Plaster of Paris (named from the occurrence north of Paris, France).

Milk of sulfur (lac sulfuris) is an acidulated polysulfide as finely divided alpha-sulfur particles in water suspension, and has several major uses in chemistry and industrial processing.

The most important acid is sulfuric acid, H_2SO_4, and the amount produced is considered a measure of the industrial technology of a country as it is so widely used in so many chemical and industrial products. The acid is used in rayon manufacture, metallurgy, steel fabrication, in formulating paint pigments, in car and storage batteries, in the making of explosives, and as a catalyst in petroleum refining.

Petroleum is rated by sulfur content; high sulfur in the western U.S. and the Caribbean; low sulfur content in the eastern U.S. Middle Eastern oil, such as in Saudi Arabia, Russia, Balkans, the Crimea, and Southern Asia, all have variable sulfur content that requires extraction during refining. As an acid effluent from such processing, and other chemical processing, it is the major producer of acid rain, and is also a major element in smog. As hydrogen sulfide, H_2S, it is released by volcanic action, converting to sulfurous acid in contact with air.

Some other important compounds are sodium thiosulfite, $Na_2S_2O_3.5H_2O$, as photographic "hypo"; sodium hyposulfite, $Na_2S_2O_4$, used in processing indigo as a dye. The SO_4^- radical forms the "vitrols": copper, blue; iron, green; and zinc, white. Sulfuric acid was first obtained in the 15th century by heating green vitrol ($FeSO_4.7H_2O$) with sand, SiO_2. When mixed with ammonia, NH_3, it is ammonium sulfate as a fertilizer.

Sulfur forms several highly colored metal compounds such as HgS, PbS, CuS, FeS, NiS, CoS, Ag_2S, and Hg_2S, black; Bi_2S_3 and Sn_2, brown; CdS and As_2S_3, yellow; Sb_2S_3, orange; MnS, a pale pink and ZnS, white. The latter, as the mineral sphalerite, is yellow-brown to black.

Technical Application: Several of the compounds listed immediately above, such as CdS, show semiconducting and optical characteristics of use in Solid State device processing.

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Cadmium sulfide, CdS, is used by itself, as well as a coating on alpha-quartz radio frequency crystals for its additional piezoelectric properties.

Sulfuric acid is widely used as an etchant, itself, and as mixed with sodium dichromate, $Na_2Cr_2O_7$, the original "glass cleaner" solution. Ceric ammonium sulfate or nitrate is the primary compound of the "chrome etch" formulations used for pattern etching chrome glass masks used in photolithographic processing. The wide use of sulfur compounds and acids is shown in the following list as a quick-reference extracted from this Etchant Section for all metals and metallic compounds in this handbook.

Sulfur, as an element, has been the subject of study since ancient times in all of its polymorphous single crystal and other structure forms, both natural and artificial.

Etching: CS_2, toluene, benzene, ether, alcohols.

Formula	Material	Use	Ref.
$27H_2SO_4$:$3Na_2Cr_2O_7$:$7OH_2O$	Al	Cleaning	AL-0032
$1H_2SO_4$:$10H_2O$	Al	Electrolytic, anodizing	AL-0020b
25 ml H_2SO_4:70 ml H_3PO_4:5 ml HNO_3	Al	Polish	AL-0035
	Al:Cu	Polish	ALCU-0003
	Al:Si	Polish	ALSI-0002
$55H_2SO_4$:$35H_3PO_4$:$10HNO_3$ + H_2O	Al_2O_3	Native oxide removal	ALO-0014
xH_2SO_4 conc.	BaF_2	Removal, slow	BAF-0001
$30H_2SO_4$:$70H_2O$	BaF_2	Polish	BAF-0003
xH_2SO_4 conc.	BeO	Preferential	BEO-0001b
$1H_2SO_4$:$1H_2O$	BeO	Cleaning	BEO-0003
xH_2SO_4 conc.	Cr_3B_2	Removal	CRB-0001c
xH_2SO_4 conc.	Mo_2B_5	Removal	MOB-0001b
xH_2SO_4 conc.	Nb_3B_3	Removal	NBB-0001a
xHF:xH_2SO_4	TaB_2	Removal	TAB-0001b
$1H_2SO_4$:$10H_2O$	Brass	Removal	BRA-0002c
12 oz H_2SO_4:4 oz $NaCr_2O_3$:1 gal H_2O	Brass	Oxide removal	BRA-0008b
$1H_2SO_4$:$4H_2O$	Bronze	Cleaning	BRO-0003
xH_2SO_4:x0.3 *M* $KMnO_4$	CdS	Preferential	CDS-0006a
5 ml H_2SO_4:1 g Cr_2O_5:1250 ml H_2O	CdS	Preferential	CDS-0007b
xH_2SO_4:$xK_2Cr_2O_7$	CdTe	Polish	CDTE-0002
6 g NH_2SO_3H:100 ml H_2O	CaF_2	Dislocation	CAF-0002
xH_2SO_4, conc.	C	Reduction/removal	C-0002b
40 ml H_2SO_4:20 ml HNO_3:20 g $KClO_4$	C	Reduction/removal	C-0001d
$64H_3PO_4$:$15H_2SO_4$:$21H_2O$	Cr	Electrolytic polish	CR-0001c
xH_2SO_3	Co_9S_8	Preferential	COS-0001
$4H_2SO_4$:$2HNO_3$:2HF	Cb	Clean/polish	CB-0001
10 ml H_2SO_4:10 ml HF:10 ml H_2O + xH_2O_2	Cb	Preferential	CB-0002b
$6H_2SO_4$:$12CrO_3$:$82H_2O$	Cu	Cleaning	CU-0017
xH_2SO_4:$xCu(NO_2)_3$	Cu	Preferential	CU-0018
$3H_2SO_4$:$1HNO_3$:$1H_2O$ "Brite Dip"	Cu	Polish, rapid	CU-0028
2—5% $(NH_4)_2SO_4$	Cu_2O	Cleaning	CUO-0010
$6H_2SO_4$:$1H_2O_2$:$1H_2O$	$CuInSe_2$	Polish	CUIS-0003
$3H_2SO_4$:$1H_2O_2$:$1H_2O$	$CuInS_2$	Dislocation	CIS-0001c

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Formula	Material	Use	Ref.
$1H_2SO_4:1H_2O_2:8H_2O$	GaAs	Preferential	GAS-0064
$1H_2SO_4:1H_2O_2:50H_2O$	GaAs	Step-etch	GAS-0067
$18H_2SO_4:1H_2O_2:1H_2O$	GaAs	Damage removal	GAS-0068
$3H_2SO_4:1H_2O_2:1H_2O$	GaAs	Polish/clean	GAS-0069
	GaAs	Polish/clean/selective	GAS-0073
$4H_2SO_4:1H_2O_2:1H_2O$	GaAs	Isotropic (polish)	GAS-0006h
$1H_2SO_4:4H_2O_2:1HF$	GaAs	Polish/thinning	GAS-0088
$5H_2SO_4$:95 propylene glycol "SSA"	GaAs	Electrolytic, dislocation	GAS-0121
$2H_2SO_4:2HCl:1HNO_3:2H_2O$	GaP	Polish	GAP-0002
$3H_2SO_4:1H_2O_2:1H_2O$	GaP	Preferential, junction	GAP-0013
xH_2SO_4, conc.	Ge	Electrolytic, shaping	GE-0129d
125 ml 0.5 *M* $3K_2S_2O_3.H_2O$:4gI_2	Ge	Electrolytic, junction	GE-0039
175 ml 15%KOH:45 g $(NH_4)_2S_2O_8$	Ge	Preferential	GE-0054
1000 ml H_2SO_4:100 g $K_2Cr_2O_7$	Au	Electrolytic, polish	AU-0009
$2H_2SO_4:1H_2O$ or H_2SO_4 conc.	InSb	Removal	INSB-0002b
$1H_2SO_4:1H_2O_2:xH_2O$	InGaAs	Mesa forming	ING-0001
xH_2SO_4, conc. or $2H_2SO_4:1H_3PO_4$	InP	Removal	INP-0020c
$10H_2SO_4:1H_2O_2:1H_2O$	InP	Polish/selective	INP-0044
$2H_2SO_4$:1HBr(47%)	InP	Dislocation	INP-0051
$1H_2SO_4$:1HCl:$1H_2O$	Fe	Cleaning	FE-0108
$30H_2SO_4:30H_2O$:20Gly	LaB_6	Electrolytic, polish	LAB-0001
1KOH, sat. sol.:$1Na_2SO_3$ sat. sol:5 Gly	PbSnSe	Electrolytic, polish	PBTS-0001
$xPbSO_4$	$PbZrO_3$	Polish	PBZO-0001b
xH_2SO_4, conc.	$PbZrO_3$	Polish	PBZO-0001c
$xH_2SO_4:xH_2O$:x(acids?)	Mg	Removal	MG-0011
$1H_2SO_4:1H_2O$:5NH_4Cl, sat. sol.	MgO	Preferential	MGO-0008
$2H_2SO_4:1H_2O_2:200H_2O$	Mg_2Ge	Cleaning	MGGE-0001b
$xH_2SO_4:xKF$	Mo	Removal	MO-0001b
$xH_2SO_4:xNaCr_2O_7$	Mo	Cleaning	MO-0001d
$1H_2SO_4:1HNO_3:3H_2O$	Mo	Polish	MO-0007
870 ml H_2SO_4:30 ml H_2O	Mo	Electrolytic, thinning	MO-0012
2 15%NaOH:$1Na_2S_2O_8$, sat. sol.	HgTe	Dislocation	HGT-0002b
$10H_2SO_4:30HNO_3$:50HAc:$10H_2O$	Ni	Polish	NI-0001
$40H_2SO_4:60H_2O$	Ni	Electrolytic polish/ thin	NI-0013
xH_2SO_4, conc.	Nb	Removal	NB-0005a
$9H_2SO_4$:1HF	Nb	Electrolytic, polish	NB-0006
$xH_2SO_4:x$Gly	Pd	Electrolytic, polish	PD-0003
xH_2SO_4, conc.	Plastic	Removal, jet	PL-0003
$xH_2SO_4:xCrO_3$	Quartz	Cleaning	QTZ-0009
xH_2SO_4, conc.	Re	Removal	RE-0004e
$1H_2SO_4:1H_3PO_4$	Sapphire	Cleaning	SAP-0001
xH_2SO_4, conc.	Se	Removal	SE-0001
$xH_2SO_4:xHNO_3$	Se	Preferential	SE-0003a
$1H_2SO_4:10H_2O$	V_3Si	Electrolytic, polish	VSI-0001b
$1H_2SO_4:1H_2O_2$ "Caro's Etch"	Si	Cleaning	SI-0103
xH_2SO_4, conc.	SiO_2	Cleaning (soda lima)	SIO-0022

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Formula	Material	Use	Ref.
$40HF:15H_2SO_4$	SiO_2	Cleaning (Corning 7720)	SIO-0038a
1000 ml H_2SO_4:35 ml $Na_2Cr_2O_7$, sat. sol.	SiO_2	"Glass Cleaner"	SIO-0041b
1000 ml H_2SO_4:35 ml $K_2Cr_2O_7$, sat. sol.	Si_3N_4	Cleaning	SIN-0015
20 ml H_2SO_4:40 g Cr_2O_3:1000 ml H_2O	Ag	Polish	AG-0016
$xH_2SO_4:xH_2O$:x(acids?)	NaBr	Removal	NABR-0002
$1—2H_2SO_4:10H_2O$	Steel	Polish	FE-0009a
$5H_2SO_4:2HF:2HNO_3$	Ta	Preferential	TA-0004a
$61H_2SO_4:7HF:16HNO_3$	Ta	Polish	TA-0006
$xH_2SO_4:xK_2Cr_2O_7$, sat. sol.	Ta	Cleaning	TA-0003g
xH_2SO_4, conc.	Te	Polish	TE-0001a
xH_2SO_4, conc.	Tl	Removal	TL-0003
x9 *N* H_2SO_4:x 1 *N* H_3PO_4	Th	Electrolytic, polish	TH-0003
xH_2SO_4, conc.	ThO_2	Removal	THO-0001
$2H_2SO_4:10H_2O$	Ti	Removal	TI-0001f
$16H_2SO_4:3HF:1H_2O$	Ti	Electrolytic, polish	TI-00010
xH_2SO_4, conc.	TiO_2	Removal	TIO-0011
xH_2SO_4, conc. or $1H_2SO_4:xH_2O$	W	Removal	W-0001e
$1H_2SO_4:1H_3PO_4:1H_2O$	U	Electrolytic, polish	U-0002c
xH_2SO_4, conc.	UO_2	Removal	UO-0002b
xH_2SO_4, conc.	V	Removal	V-0002b
$1H_2SO_4$:6MeOH	V	Electrolytic, polish/ thin	V-0004
$5H_2SO_4$:9HAc	Zr	Electrolytic, thinning	ZR-0005b
xH_2SO_4, conc.	ZrO_2	Removal	ZRO-0001a

SULFUR, S

General: Sulfur is found as a native element, associated with sedimentary rock areas, and recognizable by its brilliant yellow color. It is found in extensive beds and sulfur domes, similar to and associated with salt domes, such as those along the Gulf Coast of the U.S. As it forms compounds with all elements other than gold and platinum, like oxygen, it is a major mineral and rock former as sulfides and sulfates.

Sulfuric acid, H_2SO_4, is the most important industrial acid, and the amount manufactured is a measure of a country's industrialization and technology. In metal processing the material is either desulfurized or can be sulfided under control conditions for specific material structure and use.

Technical Application: Pure sulfur is not used directly in Solid State processing, but there are several binary semiconductor compounds fabricated as electronic devices, such as cadmium sulfide, CdS, for both semiconducting and piezoelectric properties. Sulfuric acid is a major acid alone, or as a mixed acid in etching of all metals and metallic compounds in wet chemical etching (WCE) or as an electrolytic etching system. The latter is more prevalent in industrial metals than on semiconductors.

Both natural and artificial sulfur has been studied since ancient times in its several crystallographic single crystal or amorphous forms.

Etching: CS_2, hydrocarbon derivative solvents and alcohols.

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SULFUR ETCHANTS

S-0001
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... CS_2
x .... EOH
DISCUSSION:
S, (100) wafers as yellow alpha-sulfur single crystal and polycrystalline material. Solution used as a general etch for removal and polishing. (*Note:* Alpha-sulfur is classified in mineralogy as orthorhombic, though it is often referred to as "rhombic" sulfur in chemistry.)
REF: Walker, P — mineralogy studies, 1952

S-0002
ETCH NAME: Carbon disulfide TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... CS_2
DISCUSSION:
S, (001) wafers cleaved from single crystal of delta-sulfur (black sulfur) and also cut into strips with a knife. Black sulfur is orthorhombic at standard pressure and temperature; alters to isometric — normal cubic phase under 700 K bars pressure at RT. (*Note:* Black sulfur is a black, tar-like liquid at 230°C.)
REF: Okajima, M et al — *Jpn J Appl Phys*, 23,15(1983)

S-0003
ETCH NAME: Toluene TIME:
TYPE: Solvent, removal TEMP:
COMPOSITION:
x .... C_7H_8
DISCUSSION:
S specimens. Sulfur is soluble in this solvent, also benzene and slowly in alcohols.
REF: Hodgman, C D — *Handbook of Chemistry and Physics*, 27th ed, Chemical Rubber Co., Cleveland, OH, 1943, 468

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PHYSICAL PROPERTIES OF TALC, $H_2Mg_3(SiO_3)_4$

Classification	Silicate
Atomic numbers	1, 12, 14 & 8
Atomic weight	418
Melting point (°C)	>1500
Boiling Point (°C)	
Density (g/cm³)	2.7—2.8
Refractive index (n=)	1.539—1.589
Hardness (Mohs — scratch)	1—1.5
Crystal structure (orthorhombic — normal)	(100) pinacoid
(monoclinic — normal)	(100) pinacoid
Color (solid)	White/greenish
Cleavage (basal — perfect)	(001)

TALC, $H_2Mg_3(SiO_3)_4$

General: Occurs as a natural silicate compound found as small single crystals, but more commonly massive, granule to fibrous. Found in association with the rock serpentine and, in its asbestos form, may contain sharp silicate needles. The massive form is called soapstone and, in thin sheets can be flexible, but not elastic. Sheets show the six-rayed percussion figure common to micas. It contains about 4.8% water, about half appears to be in the crystal lattice. Entrapped water can be driven off at dull red heat with the remainder driven off rapidly above 900°C and, with water removed, fusibility index is about 9 . . . edges fuse with difficulty to a white enamel, which is not acted upon by acids.

In powder form it is a filler in paper, used as a dry lubricant, and as talcum powder it has had long use in cosmetics. It can be easily cut with a knife in the massive form of soapstone and, coupled with its inertness to chemical attack, has been used for washtubs and sinks, and is still in use as table tops in chemical laboratories. Soapstone can look like jade, is used as an ornamental stone or carved as objets d'art, but can be told from jade by its greasy appearance and softness.

Technical Application: As soapstone it is still found as table tops in chemical laboratories, and has been used as a mortar and pestle for grinding chemicals. As talcum powder, it is used with latex and rubber gloves for ease of hand insertion and for water absorption to some degree, but not recommended for Clean Room operation due to possible dusting contamination. Also, in powder form, it has been used as a packing vehicle to hold metal powders during pressure testing where it is nonreactive under the temperatures and pressures applied. Occasionally the powder has been used as a dry lubricant on equipment parts, similar to graphite powder applications.

Talc has been the subject of several studies, particularly with regard to its use as talcum powder. It has been found that specific mines have a higher percentage of silicate needles in the raw material that require removal before use as a facial powder. As an asbestos, it is being phased out as an insulating material due to possible carcinogenic properties, even though, as a natural compound, it is not a carcinogen.

Etching: H_2SO_4 (Rensselaerite, only).

TALC ETCHANTS

TALC-0001
ETCH NAME: Heat — TIME:
TYPE: Thermal, drying — TEMP: 150°C
COMPOSITION:
x .... heat

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DISCUSSION:

Talc, $H_2O.3MgO.4SiO_2$. Powdered talc used as a packing vehicle in a study of pressure-induced electronic transitions of La, U, and Th. Materials were handled under oil and washed with dry hexane, then the talc dried at 150°C before mixing and pressurizing. (*Note:* About half the water in talc is lost just below red heat; the remainder is driven off at about 900°C. Talc is not attacked by acids.)

REF: Vijayakumar, V — *J Phys Chem Solids*, 46,17(1985)

TALC-0002

ETCH NAME: TIME:

TYPE: Cleave, preferential TEMP:

COMPOSITION:

x .... cleave

DISCUSSION:

Talc, $H_2O.3MgO.4SiO_2$. Specimens cleaved (001) and studied for defects and dislocations under an electron microscope. Dislocations are present on the cleaved surface.

REF: Amelinck, S & Delavignette, P — *J Appl Phys*, 32,241(1961)

PHYSICAL PROPERTIES OF TANTALUM, Ta

Classification	Transition metal
Atomic number	73
Atomic weight	108.9
Melting point (°C)	2996
Boiling point (°C)	
Density (g/cm^3)	16.6
Thermal conductance (cal/sec)(cm^2)(/°C/cm)	0.13
Specific heat (cal/g) 20°C	0.036
Heat of fusion (k-cal/g-atom)	6.8
Heat of vaporization (k-cal/g-atom)	180
Atomic volume (W/D)	10.9
1st ionization energy (K-cal/g-mole)	138
Latent heat of fusion (cal/g)	41.5
Electronegativity (Pauling's)	1.5
Covalent radius (angstroms)	1.34
Ionic radius (angstroms)	0.68 (Ta^{+5})
Linear coefficient of expansion ($\times 10^{-6}$ cm/cm/°C)	6.5
Electrical resistivity (micro-ohms/cm)	13.5
Vapor pressure (mm $\times 10^{-11}$) 1727°C	9.5
Electrical work function (eV)	4.12
Magnetic susceptibility (cgs $\times 10^{-6}$)	0.93
Young's modulus (psi $\times 10^6$) 20°C	27
Tensile strength (psi $\times 10^3$) 20°C	30—70
Cross section (barns)	21
Hardness (Mohs — scratch)	6.0—6.5
Crystal structure (isometric — normal)	(100) cube, bcc
Color (solid)	Grey/silver (yellowish)
Cleavage (cubic — poor)	(001)

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TANTALUM, Ta

General: There are minor occurrences of native tantalum as water-washed grains associated with gold placer deposits, but very rare. The primary ore is as a tantalate in the mineral group classified as oxygen salts, of which the mineral columbite-tantalite is the most representative example. The general formula is MTa_2O_6 to MNb_2O_6/MCb_2O_6; where M = Fe or Mn with traces of Sn and W. (*Note:* The element name "Niobium" is now used in place of "Columbium".) Columbite-tantalite forms an isomorphous series, grading from pure tantalite to pure columbite depending upon locality. Like many of the rare metal elements the major occurrence is in pegmatites — the final extrusion of acid magmas which slow cool forming extremely large single crystals: both beryl and tourmaline crystals . . . 80 to 100 ft long and 4 to 5 ft in diameter.

The pure metal has high density and one of the highest of melting points, and is relatively inert to acids, though it will oxidize readily when heated. There are increasing applications for the pure metal as wire, rod, sheet, and bulk forms and, in nonoxidizing operations, often replaces platinum. Because of its high tenacity and acid resistance, it is used in the fabrication of special steels and similar alloys. Pressed powder and bulk material parts are particularly useful in the processing of halogens, such as bromine and iodine. A major commercial use is as the oxide Ta_2O_5 in the fabrication of tantalum capacitors and resistors used as discrete elements in electrical and electronic circuits. Although tungsten has largely replaced tantalum as a light filament, it is still used where vibration is a factor.

As carbides and borides tantalum forms refractory ceramic-type materials. Tantalum carbide, TaC, is used as a cutting wheel and, as a thin film coating on a steel blade to improve wear resistance and working life of the blade.

Technical Application: In Solid State device processing the pure metal is RF sputter evaporated in multilayer metallization structures and, as it is an oxygen "getter" similar to titanium, has been used for that purpose: as a rod in vacuum systems or as a thin film evaporation on the vacuum bell jar walls prior to subsequent metals being evaporated to reduce oxygen contamination during metallization structuring. In planar device and circuit fabrication, the metal is evaporated as a thin film and then oxidized to Ta_2O_5 for use as a capacitor. Evaporated in a nitrogen atmosphere, it forms tantalum nitride, TaN, and, like other metal nitrides and oxides is under development and application as a surface protective coating. It also is under development as a silicide: TaSi, Ta_2Si and $TaSi_2$ for use as a buffer layer in multilayer device heterostructures. And both the carbide and boride are under evaluation as surface protective coatings.

Tantalum has been grown as a single crystal for general morphological study and, in particular, studied for its oxidation and capacitance effects.

Etching: Insoluble in single acids other than alkalies. Mixed acids: $HF:HNO_3$.

TANTALUM ETCHANTS

TA-0001
ETCH NAME:
TYPE: Acid, cleaning
TIME: 2—10 sec
TEMP: RT
COMPOSITION:

1 .... HF	to	1 .... HF
1 .... HNO_3		1 .... HNO_3
2 .... H_2O		10 .... H_2O

DISCUSSION:

Poly-Ta rod, sheet, wire used as evaporation sources for tantalum metal and tantalum nitride thin films. Tantalum was evaporated, E-beam sputter evaporated and RF sputtered from a solid target. Material was etch cleaned in the solution shown; rinsed in DI water, then MeOH and N_2 dried prior to use as an evaporation source. Substrates were gallium

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arsenide, alumina and sapphire, as pure metal and tantalum nitride deposited thin films. Solution is extremely rapid at 2—3 sec for removal of 400 Å of Ta or TaN. Parts used in GaAs FET assembly.

REF: Walker, P — personal development, 1979—1984

TA-0019a: Walker, P & Moreland, M — personal application, 1981

Ta, thin films converted to Ta_2O_5 or as TaN. Solution used as a step-etch to establish thickness color order of both the oxide and nitride as deposited on (100) chromium doped gallium arsenide.

TA-0020: Klein, G P — *J Electrochem Soc,* 118,672(1971)

Ta, foil used as the base for Ta powder in the forming of tantalum oxide capacitors. Degrease and etch clean foil; paint-on a Ta powder slurry; sinter at 1900—2200°C in argon; anodize Ta film to Ta_2O_5; deposit manganese nitrate and convert to MnO_2 in hot gas. Describes a low-cost, mass fabrication method for fabrication of solid tantalum capacitors as "powder-on-foil" devices.

TA-0002

ETCH NAME: TIME:

TYPE: Acid, removal TEMP:

COMPOSITION:

1 .... HF

2 .... HNO_3

1 .... H_2O

DISCUSSION:

Ta material. Described as a general etch for tantalum metallographic specimens.

REF: *A/B Met Dig,* 21(2),23(1983), Buehler Ltd

TA-0011: Maissel, L I & Glang, H — *Handbook of Thin Film Technology,* McGraw-Hill, New York, 1970

Ta, thin films. Described as a general rapid etch for the material.

TA-0004a

ETCH NAME: TIME: Variable

TYPE: Acid, preferential TEMP: RT

COMPOSITION:

5 .... H_2SO_4

2 .... HNO_3

2 .... HF

DISCUSSION:

Ta, (111). Described as a general etch for tantalum. Pit size increases with time and shows good reproducibility. Used in studying dislocation density.

REF: Bakish, R — *Acta Metall,* 6,120(1958)

TA-0017: Dreiner, R & Schimmel, J — *J Electrochem Soc,* 111,452(1964)

Ta specimens used in a study of oxides films and effect on photoresponse. Solution used at RT, 15 sec to chemically polish. After polish, boil in water for 10 min to oxidize and anneal 30 min in vacuum at 2100°C to densify the oxide.

TA-0003e: Ibid.

Ta sheet. A general etch for tantalum.

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TA-0004b
ETCH NAME: BHF
TYPE: Acid, preferential
COMPOSITION:
1 .... HF
1 .... HN_4F(20%)
TIME:
TEMP:
DISCUSSION:
Ta, (111) and (100) wafers. Described as a general etch for tantalum. Best pit patterns were on the (112).
REF: Ibid.
TA-0003a: Kohl, W H — *Handbook of Materials & Techniques for Vacuum Devices*, Reinhold, New York, 1967, 304
Ta, poly sheet blanks used in an etch study of tantalum. Mixture was 10 ml HF:10 ml NH_4F(20%), 1 min at 50—60°C. Etch will develop grain structure and will not affect Ta_2S_5. The same solution, used boiling, turns Ta_2S_5 brown in color.

TA-0003b
ETCH NAME: Ammonium fluoride:A etch
TYPE: Acid, preferential
COMPOSITION:
x .... 20% NH_4F
TIME: 5—6 min
TEMP: 80°C
DISCUSSION:
Ta, poly sheet blanks used in an etch study of tantalum. Solution will develop grain structure but will not affect Ta_2S_5.
REF: Ibid.

TA-0003c
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
10 ml .... HNO_3
10 ml .... NH_4F(20%)
TIME:
TEMP: 60°C
DISCUSSION:
Ta, poly sheet blanks. The solution will etch tantalum and turn Ta_2S_5 black in color.
REF: Ibid.

TA-0003d
ETCH NAME:
TYPE: Acid, preferential
COMPOSITION:
20 ml .... H_2SO_4
10 ml .... NH_4F (20%)
TIME: 1—2 min
TEMP: 60°C
DISCUSSION:
Ta, poly sheet blanks. Solution will develop etched structures.
REF: Ibid.

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TA-0005a
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
x .... H_2SO_4
x .... H_2O_2
x .... H_2O
TIME:
TEMP:
DISCUSSION:
Ta material. Described as a general etch for tantalum. (*Note:* This is a major solution type for etching and cleaning semiconductor wafers. See the Caro etch formulations under Silicon.)
REF: Bergland, T & Dearden, W H — *Metallographer's Handbook of Etching*, Pitman & Sons, London, 1931

TA-0003i
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
90% .... H_2SO_4
TIME: 9—10 min
TEMP: 35—45°C
ANODE: Ta
CATHODE: C or Pt
POWER: 0.10 A/cm² — polish: 9 min
0.02 A/cm² — etch: 10 min
DISCUSSION:
Ta material. Mechanically polish before electropolish.
REF: Ibid.
TA-0009: Spitzig, A W & Mitchell, T E — *Acta Metall*, 11,1311(1966)
Ta specimens cut with a spark cutting wire from a single crystal in a study of dislocations due to tension deformation at 373 K. Solution used at RT to polish surfaces after spark cutting.
TA-0014: Gall, J F & Miller, H C — U.S. Patent 2,466,095 (1949)
Ta, specimens. Solution developed as an anodic etch for tantalum.

TA-0008b
ETCH NAME: Hydrofluoric acid
TYPE: Acid, cleaning
COMPOSITION:
1 .... HF
1 .... H_2O
TIME: 20 sec
TEMP: RT
DISCUSSION:
Ta specimens. Solution used to etch clean tantalum surfaces. The etch solution was blown off the specimen surfaces prior to water rinse to prevent oxidation.
REF: Ibid.

TA-0008a
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
1.5 .... HF
2 .... HNO_3
5 .... H_2SO_4
TIME:
TEMP:

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DISCUSSION:

Ta specimens used for oxidation and measurement of capacitance values. Polish etch in solution shown then clean surface by dip in $1HF:1H_2O$; blow off HF before DI water rinse to prevent surface oxidation. Capacitance measurements made with an electrolytic etch of 2% HNO_3 with Pt electrodes.

REF: Vermilyea, D A — *Acta Metall,* 6,166(1958)

TA-0010

ETCH NAME:

TIME:

TYPE: Acid, preferential

TEMP:

COMPOSITION:

7 .... HF

16 .... HNO_3

61 .... H_2SO_4

DISCUSSION:

Ta alloy specimens containing a Ta silicide surface coating. Use solution as a cleaning/polish etch prior to vapor phase deposition of the silicide. A study of the oxidation-resistance of the silicide coating.

REF: Lorenz, R H & Michael, A B — *J Electrochem Soc,* 108,885(1961)

TA-0003f

ETCH NAME: Potassium hydroxide

TIME:

TYPE: Alkali, removal

TEMP: RT or hot

COMPOSITION:

x .... x% KOH (NaOH)

DISCUSSION:

Ta specimens. Alkalies can be used as a general etch for tantalum. Solutions are slow etching at room temperature but fast when used hot.

REF: Ibid.

TA-0003g

ETCH NAME:

TIME:

TYPE: Acid, cleaning

TEMP: 110°C

COMPOSITION:

(1) x .... H_2SO_4
x .... $K_2Cr_2O_7$, sat. sol.

(2) x .... H_2SO_4
x .... Cr_2O_3

DISCUSSION:

Ta specimens. Both solutions are good surface cleaning etchants. (*Note:* Solution (1) is a form of the standard glass cleaning solution and (2) another variation.)

REF: Ibid.

TA-0003h

ETCH NAME:

TIME:

TYPE: Acid, polish

TEMP: RT

COMPOSITION:

(1) 35 g .... Al_2O_3, powder
20 ml .... HF

(2) 20 g .... NH_4F
100 ml .... H_2O

DISCUSSION:

Ta specimens. Both solutions used for acid lap polishing on a metallographic wheel with a felt pad.

REF: Ibid.

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TA-0006
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
1 .... HF
1 .... HNO_3
2 .... H_2SO_4
TIME:
TEMP:
DISCUSSION:
Ta specimens used for a metallographic study of the effect of oxidation at 750°C. Polish specimens with solution shown before oxidizing.
REF: Bakish, R — *J Electrochem Soc*, 105,70(1958)

TA-0011a
ETCH NAME:
TYPE: Acid, preferential
COMPOSITION:
2 .... HF
2 .... HNO_3
5 .... H_2SO_4
TIME:
TEMP:
DISCUSSION:
Ta, (100) wafers used in a study of the interaction of oxygen, nitrogen and hydrogen. Solution used as a metallographic etch after the following material treatments:
TaO_2 treatment: Shows oxygen embrittlement and fracture on (110). Has characteristic patterns that are not repetitive in location.
TaN treatment: Shows platelet dissolution with (110) cleavage associated with nitrided areas.
TaH treatment: Use heat to obtain cleavage structure or electrolytic etch (cathodic) in 1HF:10HNO_3 at RT and 0.5 A/in^{-2} power. (100) cleavage predominates over (110).
REF: Bakish, R — *J Electrochem Soc*, 105,574(1958)

TA-0012
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
10 .... HF
90 .... H_2SO_4
TIME:
TEMP:
ANODE:
CATHODE:
POWER:
DISCUSSION:
Ta specimens used in a study of the microtopography of oxide films. Electropolish specimens in solution shown before oxidizing.
REF: Pawel, R E et al — *J Electrochem Soc*, 107,956(1960)

TA-0013
ETCH NAME: Hydrofluoric acid
TYPE: Acid, cleaning
COMPOSITION:
x .... HF, conc.
TIME: 20—30 sec
TEMP: RT
DISCUSSION:
Ta, as high purity slugs used for a metal evaporation source on semiconductor wafers. Solution used to clean slugs prior to evaporation. Follow with water wash and N_2 blow dry.
REF: Walker, P & Menth, M — personal application, 1980

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TA-0015
ETCH NAME: TIME:
TYPE: Electrolytic, polish TEMP:
COMPOSITION: ANODE: Ta
x .... x% NH_4F CATHODE:
x .... formamide POWER:
DISCUSSION:
Ta specimens. Developed as an anodic polish solution for tantalum.
REF: Jenny, A L — U.S. Patent 2,742,416(1956)

TA-0016
ETCH NAME: TIME:
TYPE: Electrolytic, polish TEMP:
COMPOSITION: ANODE: Ta
x .... HF CATHODE:
x .... HCl POWER:
DISCUSSION:
Ta specimens. Developed as an anodic polish solution for tantalum.
REF: Gall, J F & Miller, H C — U.S. Patent 2,481,306(1949)
TA-0017: Kahan, G J — U.S. Patent 2,775,553(1956)

TA-0018
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
1 .... HF
1 .... HNO_3
DISCUSSION:
Ta thin films remaining in vacuum systems. Solution used to remove tantalum from bell jars and internal fixtures. Use on stainless steel; not on glass, ceramic or copper.
REF: Nichols, D R — *Solid State Technol*, December 1979
TA-0021: Teasdale, T S — *Phys Rev*, 99,1248(1955)
Ta specimens. Solutions of this mixture used for general removal.

TA-0011b
ETCH NAME: TIME:
TYPE: Alkali, removal TEMP:
COMPOSITION:
9 .... KOH (30%)
1 .... H_2O_2
DISCUSSION:
Ta thin films. Shown as a general removal etch.
REF: Ibid.
TA-0019b: Ibid.
Ta, thin films converted to Ta_2O_5 and TaN deposited on GaAs:Cr, (100) (SI) wafer substrates. Solution used as a step-etch in developing thickness color orders of the oxide and nitride.

TANTALUM ALLOYS, TaM_x

General: Does not occur as metal alloys in nature. The pure element is found in minor quantities associated with gold sands, and there are several tantalate minerals, chief among them the variable mixed mineral columbite-tantalite as an oxide containing iron.

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Industrially, tantalum and molybdenum are two of the high-temperature metals used as alloys in irons and steels to increase hardness and chemical resistance, and tantalum is used as a constituent in other metal alloys, such as for high temperature brazing above 800°C. The reference shown here is for a Ta:Mo studied for structure and mechanical properties.

Technical Application: Tantalum alloys have had little application in Solid State processing to date, although they can be used in brazing of metal packages.

Etching: Mixed acids HF:H_2SO_4, electrolytic.

TANTALUM:MOLYBDENUM ETCHANTS

TAMO-0001
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
x .... HF
x .... H_2SO_4
DISCUSSION:

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

TaMo as a single crystal alloy used in a structure and mechanical properties study. Solution shown used to polish specimens.
REF: Van Torne, L I & Thomas, G — *Acta Metall,* 14,621(1966)

TANTALUM HYDRIDE ETCHANTS

TAH-0001
ETCH NAME:
TYPE: Electrolytic, micro-etch
COMPOSITION:
1 .... HF
10 .... HNO_3
DISCUSSION:

TIME:
TEMP: RT
ANODE:
CATHODE: TaH
POWER: 0.5 A/in^2

TaH thin films grown on (100) Ta wafer substrates in a study of the interaction of oxygen, nitrogen and hydrogen. Solution used to develop defect structure. (100) cleavage predominates over (110). See TA-0011a for additional discussion.
REF: Bakish, R — *J Electrochem Soc,* 105,574(1958)

TAH-0002
ETCH NAME: Sulfuric acid
TYPE: Acid, removal
COMPOSITION:
x .... H_2O_4, conc.
DISCUSSION:

TIME:
TEMP:

TaH powder mixed in amyl acetate or cellulose nitrate and used as a binder in silicon wafer alloying. Apply to wafer surface and fire for 1—10 min at 600—900°C in an inert atmosphere (N_2 or Ar). Sulfuric acids will etch the hydrides. Other hydrides evaluated were NbH, TiH, VH, and ZrH.
REF: Sullivan, M V & Eigler, J H — *Acta Metall,* 103,218(1956)

PHYSICAL PROPERTIES OF TANTALUM CARBIDE, TaC

Classification	Carbide
Atomic numbers	73 & 6

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Atomic weight	192.9
Melting point (°C)	4150
Boiling point (°C)	5500
Density (g/cm^3)	14.48
Hardness (Mohs — scratch)	9
(Vickers (kg/mm^2)	1790
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Grey-white
Cleavage (cubic — imperfect)	(001)

TANTALUM CARBIDE, TaC

General: Does not occur as a natural compound, but is artificially fabricated as a high temperature ceramic. It is not as widely used as other carbides, such as SiC.

Technical Application: Has not been used in Solid State processing to any degree to date, but has been electrolytically deposited as a thin film on different substrates. TaC solid specimens also have been studied.

Etching: H_2SO_4, HF and other fluoride compounds, mixed salts with alkali.

TANTALUM CARBIDE ETCHANTS

TAC-0001
ETCH NAME: Murakama's reagent
TYPE: Acid, cleaning
COMPOSITION:
(1) 10 g .... $K_3Fe(CN)_6$
10 g .... KOH/NaOH
100 ml .... H_2O
(2) 10 g .... $K_3Fe(CN)_6$
2 g .... NaOH
100 ml .... H_2O
TIME:
TEMP:
ASTM: #98
DISCUSSION:
TaC, specimens. Solutions used to clean surfaces. Also used to clean tungsten carbide, WC and titanium carbide, TiC. Solution (2) is modified, with no number in the ASTM bulletin.
REF: Bull. ASTM E407-70, 1947

TAC-0002
ETCH NAME: Sulfuric acid
TYPE: Acid, removal
COMPOSITION:
(1) x .... H_2SO_4, conc.
(2) x .... HF, conc.
TIME:
TEMP:
DISCUSSION:
TaC, specimens. Both solutions can be used as slow removal etchants.
REF: *A Dictionary of Carbide Terms*, Adams Carbide Corp., Kenilworth, NJ, 07033

TAC-0003a
ETCH NAME: Sulfamic acid
TYPE: Acid, removal
COMPOSITION:
x .... 20% H_2NSO_3H
TIME: 1 min
TEMP: RT
DISCUSSION:
TaC, electrolytically deposited thin films on different substrates. Solution used for general removal of the material.
REF: Hockman A J — *J Electrochem Soc*, 130,221(1983)

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TAC-0003b
ETCH NAME: BHF
TYPE: Acid, removal
COMPOSITION:
x .... HF
x .... NH_4F (40%)
TIME: 4 sec
TEMP: RT
DISCUSSION:
TaC, electrolytically deposited thin films on different substrates. A BHF solution used to remove material more rapidly than with sulfamic acid.
REF: Ibid.

PHYSICAL PROPERTIES OF TANTALUM NITRIDE, TaN

Classification	Nitride
Atomic numbers	73 & 7
Atomic weight	195
Melting point (°C)	3360
Boiling point (°C)	
Density (g/cm^3)	
Hardness (Mohs — scratch)	8—9
Crystal structure (hexagonal — normal)	(1010) prism
Color (solid)	Colorless
Cleavage (basal)	(0001)

TANTALUM NITRIDE, TaN

General: Does not occur as a natural compound. The most important minerals are pure tantalite of the isomorphous columbite-tantalite series. There are several other tantalum-bearing minerals as oxides, but they are of minor importance as ores. Tantalum metal parts are nitrided in industry to obtain a thin surface coating to improve the physical characteristics of the material.

Technical Application: All references shown here are as thin film deposits on Solid State materials for relatively chemically inert surface coatings with possible optical characteristic applications. It also is deposited in conjunction with metals for use as a resistor when oxidized to Ta_2O_5.

Etching: Rapid attack with HF:HNO_3 mixtures; slow with alkalies and aqua regia.

TANTALUM NITRIDE ETCHANTS

TAN-0001
ETCH NAME: Aqua regia
TYPE: Acid, polish
COMPOSITION:
3 .... HCl
1 .... HNO_3
TIME:
TEMP:
DISCUSSION:
TaN, thin film deposits. Aqua regia is a slow removal and polishing etch on tantalum nitride.
REF: Weast, R C et al — *Handbook of Chemistry and Physics*, 65th ed, CRC Press, Boca Raton, FL, 1985, 154

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TAN-0002
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
50 ml .... HF
100 ml .... HNO_3
50 ml .... H_2O
TIME: 5—15 sec
TEMP: RT
ASTM: #161 (1:5:10)
DISCUSSION:

TaN thin films. Developed from the ASTM #161 solution which is for tantalum as an etch for tantalum nitride. TaN/Au and TaN/TiW/Au or TaN/Mo/Au deposited on alumina, beryllia, and quartz substrates used for microwave circuit fabrication with GaAs FETS. After AZ-1350J photo resist patterning of metal/nitride deposited substrates. Solution used to remove the TaN.

REF: Walker, P — personal development, 1983
TAN-0007; ASTM E107-70

Reference for ASTM #161 solution used to etch tantalum.

TAN-0003
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
1 .... HF
2 .... HNO_3
1 .... H_2O
TIME:
TEMP:
DISCUSSION:

TaN thin films. Described as a very rapid etch. It will etch both TaN and Ta (TA-0002). Slow reaction with addition of water.

REF: Bulletin 1116R/Rev-HPD/VB/TM/0883/1K, MRC

TAN-0004
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
9 .... 30% KOH
1 .... H_2O_2
TIME:
TEMP:
Note: Add H_2O_2 when solution is hot.
DISCUSSION:

TaN thin films. Described as a general etch for tantalum nitride. Solution will attack most photo resists. **CAUTION**: When adding any cold solution to a hot solution reaction may splatter.

REF: Grossman, S & Herman, D S — *J Electrochem Soc,* 116,674(1969)
TAN-0005a: Kodak Data Book, 1966, 91
TAN-0006: Electron. Div. Abstract, 23,1966

TAN-0005b
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
(1) 92 g .... $K_3Fe(CN)_6$
20 g .... KOH
300 ml .... H_2O
TIME:
TEMP:
(2) 200 g .... $K_3Fe(CN)_6$
20 g .... NaOH
3—3.5 g .... sodium oxalate
1000 ml .... H_2O

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DISCUSSION:
TaN thin films. Solutions will etch tantalum and molybdenum and their nitrides.
REF: Ibid.

TAN-0008
ETCH NAME: TIME:
TYPE: Gas, conversion TEMP: RT to 750°C
COMPOSITION:
x .... N_2
x .... Ar
DISCUSSION:
TaN thin films deposited 3000 Å thick on substrates at the following temperatures: 20, 250, 500, and 750°C used in a composition study of the DC sputtered films. Ta was sputtered in an N_2/Ar atmosphere. Films showed the following structures: (1) $TaN_{0\,05}$ as beta; Ta_2N as gamma; TaN_2 as alpha; and TaN as epsilon. No etch shown.
REF: Reichelt, K et al — *J Appl Phys*, 49,5284(1978)

PHYSICAL PROPERTIES OF TANTALUM OXIDE, Ta_2O_5

Classification	Oxide
Atomic numbers	73 & 8
Atomic weight	441.76
Melting point (°C)	1470, del.
Boiling point (°C)	
Density (g/cm³)	8.7 approx.
Hardness (Mohs — scratch)	7—8
Crystal structure (hexagonal — rhombohedral)	$(10\bar{1}1)$ rhomb
Color (solid)	Clear/greyish
Cleavage (basal)	(0001)

TANTALUM OXIDE, Ta_2O_5

General: Does not occur as a natural compound, though it does occur as a tantalate oxide with iron and manganese as an isomorphous series with niobium (columbium). The pure tantalite as near Ta_2O_6. The pentoxide form, above, is the most widely used form, though their are other oxides. It has been used industrially in the fabrication of capacitors, both as a bulk material and a thin film evaporation on ceramics, mica, and mylar.

Technical Application: In Solid State development, several of the oxides have been deposited as amorphous thin film capacitor elements in semiconductor circuit fabrication. They are under development as surface passivation coatings similar to those of silicon dioxide and nitride. In fabrication, tantalum metal is first evaporated, then oxidized by controlled heating as a separate process step. The nitride also has been similarly converted to the oxide.

The oxide has use as a general surface coating or for fabrication of discrete capacitor elements of a semiconductor circuit.

Etching: HF; mixed HF:HNO_3; and alkalies, variable with type of oxide.

TANTALUM DIOXIDE ETCHANTS

TAO-0001a
ETCH NAME: TIME:
TYPE: TEMP:
COMPOSITION:

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DISCUSSION:
TaO_2. Material is a dull grey powder and insoluble in acids.
REF: Weast, R C et al — *Handbook of Chemistry and Physics*, 65th ed, CRC Press, Boca Raton, FL, 1984, B154

TAO-0002
ETCH NAME:
TYPE: Alkali, removal
TIME:
TEMP:
COMPOSITION:
9 .... KOH (30%)
1 .... H_2O_2
DISCUSSION:
Ta_2O_3, thin films. Solution shown as a removal etch for Ta, Ta_2O_3 and TaN.
REF: Miasel, L I & Glang, R — *Handbook of Thin Film Technology*, McGraw-Hill, New York, 1970

TANTALUM PENTOXIDE ETCHANTS

TAO-0003a
ETCH NAME: Hydrofluoric acid
TYPE: Acid, removal
TIME:
TEMP:
COMPOSITION:
x .... HF
DISCUSSION:
Ta_2O_5. Material has rhombohedral structure and is colorless. Can be etched in HF and fused $KHBO_4$.
REF: Weast, R D et al — *Handbook of Chemistry and Physics*, 65th ed, CRC Press, Boca Raton, FL, 1984, B154

TAO-0003b
ETCH NAME: Potassium borate
TYPE: Salt, molten flux, removal
TIME:
TEMP: <150°C
COMPOSITION:
x .... $KHBO_4$, fused
DISCUSSION:
Ta_2O_5 specimens. As a molten flux, solution will etch this oxide.
REF: Ibid.

TAO-0004
ETCH NAME: Air
TYPE: Gas, stabilizing
TIME: Minutes
TEMP: 250°C
COMPOSITION:
x .... air
DISCUSSION:
Ta_2O_5 thin films RF magnetron sputter deposited on GaAs (100) wafers, sapphire and alumina substrates for use as capacitors in microelectronic circuits. After deposition material was heat treated on a hot plate in air to stabilize capacitance values.
REF: Marich, L A & Walker, P — personal application, 1980—1985

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PHYSICAL PROPERTIES OF TANTALUM SELENIDE, $TaSe_2$

Classification	Selenide
Atomic numbers	73 & 34
Atomic weight	338.80
Melting point (°C)	2000
Boiling point (°C)	
Density (g/cm³)	
Hardness (Mohs — scratch)	6—7
Crystal structure (isometric — normal?)	(100) cube
Color (solid)	Grey-black
Cleavage (cubic)	(001)

TANTALUM SELENIDE, $TaSe_2$

General: Does not occur as a natural compound, and there has been no use in industry other than in studies as a metal dichalogenide.

Technical Application: No present application in Solid State processing. It has been grown as a single crystal by the Vapor Transport (VT) method in an evaluation of dichalcogenide transition metal compounds. There is possible application of the material as a basic V—VI compound semiconductor with other metals for a ternary compound semiconductor.

Etching: Mixed acids ($HF:HNO_3$).

TANTALUM SELENIDE, $TaSe_2$

TASE-0001
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... HF
x .... HNO_3
DISCUSSION:

$TaSe_2$ single crystal specimens grown by the iodine Vapor Transport (VT) method. Specimens used in a study of transition metal dichalcogenides. TaS_2 also grown and evaluated.

REF: Naito, M & Tanaka, S — *J Phys Soc Jpn*, 4,1216(1984)

PHYSICAL PROPERTIES OF TANTALUM SULFIDE, TaS_2

Classification	Sulfide
Atomic numbers	73 & 16
Atomic weight	400
Melting point (°C)	1300 (del)
Boiling point (°C)	
Density (g/cm³)	
Hardness (Mohs — scratch)	4—5
Crystal structure (isometric — normal?)	(100) cube
Color (solid)	Grey black
Cleavage (cubic)	(001)

TANTALUM SULFIDE, TaS_2

General: Does not occur as a natural compound, even though there are a number of other metal sulfides — galena, PbS, being of major importance. Not used to any extent in

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the metal industries, other than when tantalum metal is sulfurized with a thin surface film which, in part, may be in crystalline form.

Technical Application: The compound has not been used in Solid State processing to date, though it has been grown and studied as a transition metal dichalcogenide. It is a possible basic V—VI compound semiconductor and, with other metals, a trinary compound type.

Etching: Mixed acids ($HF:HNO_3$).

TANTALUM SULFIDE ETCHANTS

TAS-0001
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... HF
x .... HNO_3
DISCUSSION:

TaS_2 single crystals were grown by the iodine Vapor Transport (VT) method. Specimens used in a study of transition metal dichalcogenides. $TaSe_2$ also grown and evaluated.
REF: Naito, M & Tanaka, S — *J Phys Soc Jpn*, 4,1216(1984)

TANTALUM TUNGSTEN ETCHANTS

TAW-0001
ETCH NAME: Ethylene chloride TIME:
TYPE: Solvent, removal TEMP:
COMPOSITION:
x .... CH_2Cl_2
DISCUSSION:

TaW thin films deposited or glass cover slides. Latex spheres 0.09 μm in diameter adsorbed on glass prior to 30 nm thick metal deposition. After metallization, ultrasonic (sonication) agitation used to remove spheres using the solution shown. Leaves an array of 0.09—0.1 μm "light" transmission holes through the TaW film as a method of controlled forming of pinholes.
REF: Fischer, Ch — *J Vac Sci Technol*, B3(1),386(1985)

PHYSICAL PROPERTIES OF TELLURIUM, Te

Classification	Semi-metal
Atomic number	52
Atomic weight	127.6
Melting point (°C)	449.5
Boiling point (°C)	989.8
Density (g/cm^3)	6.24
Specific heat (cal/g) 25°C	0.047
Latent heat of fusion (cal/g)	32
Heat of vaporization (k-cal/g-atom)	11.9
Heat of fusion (k-cal/F-atom)	4.27
Atomic volume (W/D)	20.5
Covalent radius (angstroms)	1.36
Ionic radius (angstroms)	0.70 (Te^{+4})
1st ionization potential (eV)	9.01

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1st ionization energy (K-cal/g-mole)	208
Electrical resistivity (ohms cm) 22°C	0.43 // c-axis
Electronegativity (Pauling's)	2.1
Electron work function (eV)	4.72
Electrical conductance (micro-ohms^{-1})	10—6
Modulus of elasticity (psi $\times 10^6$)	6
Poissons ratio	0.33
Vapor pressure (°C)	838
Cross section (barns)	4.3
Activation energy (eV)	0.34
Hall mobility (cm^2/V sec 0—320°C)	1800—2500
Specific resistance (ohm-cm)	0.35
Refractive index (n =)	2—3.5
Dielectric constant (e =)	5.0
Optical activation energy (eV)	0.37
Hardness (Mohs — scratch)	2—2.5
Crystal structure (hexagonal — rhombohedral)	$(10\bar{1}1)$, rhomb
Color (solid)	Tin-white
Cleavage (prismatic — perfect)	$(10\bar{1}0)$

TELLURIUM, Te

General: Occurs as a native element but in minor quantities. It has been found as fine prismatic crystals but is more commonly columnar to massive. It is completely volatile in a bunsen burner flame and produces a deep red solution in warm sulfuric acid. The mineral tellurite, TeO_2, is of greater occurrence from which the metal is obtained by reduction. It is a semi-metal similar to sulfur and is found as a minor constituent in several minerals, to include gold. As an alloy with iron or copper it produces high electrical resistance materials, and with less than $^1/_{10}$ of 1% alloyed with lead greatly increases both strength and hardness. As a coloring agent in glass it produces a deep blue to brown coloration. If the vapors are breathed it imparts a very offensive odor called "telluride breath".

Technical Applications: Tellurium is used as a doping element in some compound semiconductors. It is a growth element in several semiconductor compounds such as cadmium telluride, CdTe; zinc telluride, ZnTe; lead telluride, PbTe and trinary compounds containing tin or mercury. As a single crystal tellurium has been grown and studied for general physical data and morphology.

Etching: H_2SO_4, aqua regia and HF:HNO_3 mixtures.

TELLURIUM ETCHANTS

TE-0001a
ETCH NAME: Sulfuric acid
TYPE: Acid, dislocation
COMPOSITION:
x .... H_2SO_4, conc.
TIME:
TEMP: 150°C
RATE: 11 μm/min
DISCUSSION:
Te, $(10\bar{1}0)$ wafers used in a dislocation development study. Etch is very temperature dependent. Used at room temperature, dislocation pits may be asymmetric in the $\langle 10\bar{2}0\rangle$ direction. Also used as a polish etch at elevated temperature.
REF: Blackmore, J S et al — *Phys Rev*, 117,687(1960)

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TE-0001b
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
1 .... HF
1 .... HNO_3
1 .... CH_3COOH (HAc)
TIME:
TEMP: RT or 0°C
RATE: 5 μm/min at RT
DISCUSSION:
Te, $(10\bar{1}0)$ wafers used in a dislocation development study. Solution used as a polishing etch. It is a rapid etch at both RT and 0°C.
REF: Ibid.

TE-0001c
ETCH NAME:
TYPE: Acid, preferential
COMPOSITION:
1 .... HCl
1 .... CrO_3
3 .... H_2O
TIME:
TEMP: RT
RATE: 4 μm/min
DISCUSSION:
Te, $(10\bar{1}0)$ wafers used in a dislocation study. Polish wafers in H_2SO_4 (TE-0001a) then use solution shown to develop dislocation etch pits. Pits are sharply defined in a smooth, bright background surface.
REF: Ibid.
TE-0004: Naukkarienen, K & Toomi, T C — *J Appl Phys*, 40,3054(1969)
Te, single crystal wafers used in an X-ray topographic study of defects. Solution mixture used for defects but development not shown.
TE-0007: Blackmore, J S et al — *J Appl Phys*, 31,2226(1960)
Te, (1010) wafers used in a dislocation study. Solution used as a polish etch. Produces a bright surface with an etch rate of 4 μm/min.

TE-0002
ETCH NAME: Aqua regia
TYPE: Acid, removal/polish
COMPOSITION:
3 .... HCl
1 .... HNO_3
TIME:
TEMP:
DISCUSSION:
Te, $(10\bar{1}0)$, cleaved wafers used in a study of optical properties. Agitate during etching to prevent pitting. Solution used to remove 7—15 μm of a surface conductance layer.
REF: Caldwell, R S — Special Rep Purdue University, January 1958

TE-0003
ETCH NAME: CP4, modified
TYPE: Acid, saw
COMPOSITION:
3 .... HF
5 .... HNO_3
3 .... HAc
TIME:
TEMP: RT
DISCUSSION:
Te, (0001) wafers acid saw cut from the ingot. A 0.025 platinum wire, wetted with the

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solution shown, was used as the cutting tool. The ingot was also cleaved to obtain rectangular specimens. (*Note:* CP4 without bromine is called CP4A.)
REF: Vis, V A — *J Appl Phys,* 35,361(1964)

TE-0005
ETCH NAME: CP4
TYPE: Acid, removal
COMPOSITION:
30 ml .... HF
50 ml .... HNO_3
30 ml .... HAc
0.5 ml .... Br_2
TIME: 10 sec
TEMP: RT
DISCUSSION:
Te, (0001) cleaved wafers, used in measuring the galvanmagnetic effects. Etch was used to remove the conducting layer from cleaved surfaces (about 15 μm). (*Note:* Original CP4 called for 0.6 ml Br_2.)
REF: Roth, H — *J Phys Chem Solids,* 8,525(1959)

TE-0006
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
3 .... HF
5 .... HNO_3
6 .... HAc
TIME: 30 sec
TEMP: RT
RATE: 7 mil/min
DISCUSSION:
Te, (0001) wafers used in a study of dislocations. Solution rate was 7 mil/min and used to remove damaged surface.
REF: Lovell, L C et al — *Acta Metall,* 6,716(1958)

TE-0008
ETCH NAME: Aqua regia, dilute
TYPE: Acid, polish
COMPOSITION:
3 .... HCl
1 .... HNO_3
1 .... H_2O
TIME: 4—5 min
TEMP: RT
RATE: 30—40 μm/4—5 min
DISCUSSION:
Te, (0001) wafers used in a study of annealing behavior of extrinsic tellurium. Solution used in the etch preparation of Hall samples.
REF: Skadron, P & Johnson, V A — *J Appl Phys,* 37,1912(1966)

TE-0009a
ETCH NAME:
TYPE: Acid, cleaning
COMPOSITION:
(1) 2 .... HNO_3
4 .... H_2O
1 .... CrO_3
(2) x .... 30% HCl
(3) x .... H_2SO_4
TIME: 2 h
TEMP: RT
DISCUSSION:
Te, (0001) and $(12\bar{1}0)$ wafers cut from ingot; $(10\bar{1}0)$ cleaved in air or in LN_2. Wafers

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used as substrates for deposition of antimony. Polish etch tellurium in solution (1) with rotation during etch period. Follow with 30% HCl, RT, 2 min to remove oxides. In vacuum, thermal faceting observed above 145°C. After Sb growth, cleave wafers ⟨110⟩. Growth appears columnar, bladed. Used hot H_2SO_4 (3) as a dislocation etch.
REF: Shih, I et al — *J Cryst*, 9,523(1984)

TE-0010
ETCH NAME: Tetraborane
TYPE: Salt, removal
COMPOSITION:
x .... x% $Na_2B_4H_{10}$

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

DISCUSSION:
Te, material used for epitaxy growth of HgCdTe that was then used in a chemical etching and oxidation study. Clean tellurium and remove native oxide in the solution shown.
REF: Aspenes, D E & Arwin, H — *J Vac Sci Technol*, A(2),1309(1984)

TE-0011
ETCH NAME: Alumina
TYPE: Abrasive, polish
COMPOSITION:
x .... Linde A and B

TIME:
TEMP: RT

DISCUSSION:
Te, (0001) specimens used in a study of the effect of pressure on infrared absorption. Because tellurium is soft and brittle initial coarse lapping was limited to not greater than 400 grit. A pitch lap (1 beeswax:1 resin) used for first stage-polish using aluminas shown. Final lap used canvas cloth attached to an optical flat. Also polished silicon and germanium with the pitch lap method.
REF: Waters, W P — personal communication, 1957

TE-0012a
ETCH NAME:
TYPE: Acid, saw
COMPOSITION:
x .... HCl
x .... CrO_3
x .... H_2O

TIME:
TEMP:

DISCUSSION:
Te, (0001) and $(10\bar{1}0)$ wafers used in a study of infrared absorption in intrinsic tellurium. Solution used as an acid saw for cutting specimens.
REF: Ades, S & Champness, C H — *J Appl Phys*, 49,4543(1978)

TE-0012b
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
10 ml .... HNO_3
5 g .... CrO_3
20 ml .... H_2O

TIME:
TEMP:

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DISCUSSION:
Te, (0001) and (10$\bar{1}$0) wafers used in a study of infrared adsorption in intrinsic tellurium. Solution used to chemical polish surfaces.
REF: Ibid.

TE-0012c
ETCH NAME: Hydrochloric acid
TYPE: Acid, oxide removal
COMPOSITION:
x .... HCl
TIME:
TEMP: RT
DISCUSSION:
Te, (0001) and (10$\bar{1}$0) wafers used in a study of infrared optical adsorption of intrinsic tellurium. After chemical etch polish, solution shown used to remove residual native oxide from previous etch solutions.
REF: Ades, S & Champness, C H — *J Appl Phys*, 49,4543(1978)

PHYSICAL PROPERTIES OF TELLURIUM DIOXIDE, TeO_2

Classification	Oxide
Atomic numbers	52 & 8
Atomic weight	159.61
Melting point (°C)	800
Boiling point (°C)	
Density (g/cm^3)	5.9
Refractive index (n=)	2.00—2.35
Hardness (Mohs — scratch)	2
Crystal structure (orthorhombic — normal)	(100) a-pinacoid
Color (solid)	Yellow-white
Cleavage (prismatic — perfect)	(010)

TELLURIUM DIOXIDE, TeO_2

General: Occurs in nature as the mineral tellurite, TeO_2, largely as an oxidation product on other tellurides. A black, amorphous monoxide, TeO, appears on the more stable dioxide, and there is a bright orange colored trioxide, TeO_3, form. There is no use of the oxides in the metal industries, but both the pure metal and oxides are a blue to brown coloring agent in glass.

Technical Application: The oxides have had no use in Solid State material processing to date, although the metal element is used in the growth of both binary and trinary compound semiconductors of cadmium, lead and zinc. It has been grown as a thin film on CdTe, and referred to as a native oxide with stabilization time in air.

Etching: HCl, HNO_3, alkalies (TeO_2); HCl, and dilute acids (TeO); HCl and hot alkalies (TeO_3).

TELLURIUM DIOXIDE ETCHANTS

TEO-0001
ETCH NAME: Tetraborane
TYPE: Salt, removal
COMPOSITION:
x .... $Na_2B_4H_{10}$
TIME:
TEMP:
ANODE:
CATHODE:
POWER:

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DISCUSSION:

TeO_2, crystalline native oxide or TeO, amorphous native oxide (black). Solution used to remove native oxide and etch clean tellurium used in the epitaxy growth of HgCdTe thin films. Material was then studied for chemical etching and oxidation characteristics.

REF: Aspenes, D E & Arwin, H — *J Vac Sci Technol*, A(2),1409(1984)

TEO-0002

ETCH NAME: Hydrochloric acid — TIME: 2 min

TYPE: Acid, oxide removal — TEMP: RT

COMPOSITION:

x .... 30% HCl

DISCUSSION:

Te, (0001) and other wafer orientations. Solution used to remove residual oxide remaining on surfaces after chemical etch polishing. (See TE-0009c and TE-0012c).

REF: Shi, I et al — *J Cryst*, 9,523(1984)

TEO-0003: Ades, S & Champness, C H — *J Appl Phys*, 49,4543(1978)

Te, (0001) and $(10\bar{1}0)$ wafers. Solution used to remove residual oxide after chemical etch polishing. (See TE-0012c.)

TEO-0004

ETCH NAME: BRM — TIME:

TYPE: Halogen, removal — TEMP: RT

COMPOSITION:

x .... 5% Br_2

x .... MeOH

DISCUSSION:

TeO_2, grown as a stable native oxide on CdTe (110) wafers. Mechanically lap wafers with 32,000-mesh abrasive, then chem/mech polish with BRM solution. Specimens allowed to sit in air under light for 30 days for a 600 Å thick TeO_2 stable oxide. Remove oxide with HCl or HNO_3.

REF: Ponce, F A et al — *J Appl Phys Lett*, 39,951(1981)

TEO-0005

ETCH NAME: — TIME:

TYPE: Acid, removal/polish — TEMP: RT

COMPOSITION:

(1) x .... HCl, conc. (2) 1 .... HCl

10 .... H_2O

DISCUSSION:

TeO_2, as natural single crystals used in a general study of natural mineral oxides. Solution (1) used to etch surfaces; solution (2) to develop structure.

REF: Walker, P — mineralogy study, 1953

PHYSICAL PROPERTIES OF TERBIUM, Tb

Classification	Lanthanide
Atomic number	65
Atomic weight	159
Melting point (°C)	1356
Boiling point (°C)	2800 (2530)
Density (g/cm^3)	8.27

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Specific heat (cal/g) 25°C	0.044
Heat of fusion (k-cal/g-atom)	3.9
Latent heat of fusion (cal/g)	12.71
Heat of vaporization (k-cal/g-atom)	70
Atomic volume (W/D)	19.2
1st ionization energy (K-cal/g-mole)	155
1st ionization potential (eV)	6.74
Electronegativity (Pauling's)	1.2
Covalent radius (angstroms)	1.59
Ionic radius (angstroms)	0.93 (Tb^{+3})
Electrical resistivity (micro-cm) 298 K	114.5
Compressibility ($cm^2/kg \times 10^{-6}$)	2.45
Cross section (barns)	44
Magnetic moment (Bohr magnetons)	9.7
Magnetic susceptibility ($\times 10^{-6}$ emu/mole)	193,000
Transformation temperature (°C)	1287
Hardness (Mohs — scratch)	4—5
Crystal structure (hexagonal — normal)	$(10\bar{1}0)$ prism, hcp
Color (solid)	Grey
Cleavage (basal)	(0001)

TERBIUM, Tb

General: Does not occur as a native element. It is a rare earth member of the lanthanide, yttrium group. Found as a minor constituent in yttrium and gadolinium minerals: the mineral gadolinite is a mixed silicate with iron, yttrium, etc. There is no current use in industry due to scarcity.

Technical Application: Only limited use to date, in Solid State processing. It is a constituent in some ferrites and garnets. Most of the rare earth elements have been grown as single crystals for general morphology and defect study.

Etching: Heat used to develop defects.

TERBIUM ETCHANTS

TB-0001
ETCH NAME: Heat — TIME:
TYPE: Thermal, preferential — TEMP:
COMPOSITION:
x .... heat
DISCUSSION:
Tb, single crystal specimens. Describes a method of growing rare earth single crystal. Thermal annealing after growth used to develop defects, low angle grain boundaries and other structure. Other metals studied were Dy, Gd, Er, Ho, Tr, Th, and Y.
REF: Nigh, H E — *J Appl Phys,* 34,3323(1963)

PHYSICAL PROPERTIES OF THALLIUM, Tl

Classification	Metal
Atomic number	81
Atomic weight	204.37
Melting point (°C)	303
Boiling point (°C)	1457

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Density (g/cm^3)	11.85
Thermal conductivity (cal/sec)(cm^2)(°C/cm)	0.093
Specific heat (cal/g) 20°C	0.031
Heat of fusion (cal/g)	5.04
Heat of vaporization (cal/g)	190
Atomic volume (W/D)	17.2
1st ionization energy (K-cal/g-mole)	141
1st ionization potential (eV)	6.106
Electronegativity (Pauling's)	1.8
Covalent radius (angstroms)	1.48
Ionic radius (angstroms)	1.47 (Tl^{+1})
Coefficient of linear thermal expansion ($\times 10^{-6}$ cm/cm/°C)	28
Electrical resistivity (micro-ohms-cm)	18
Electron work function (eV)	3.95
Tensile strength (psi)	1300
Cross section (barns)	3.3
Vapor pressure (°C)	1196
Hardness (Mohs — scratch)	1—2
(Brinell — mm/kg)	2
Crystal structure (hexagonal — normal)	($10\bar{1}0$) prism, hcp
(tetragonal — normal)	(100) a-pinacoid
Color (solid)	Blue-white
(spectrum)	Brilliant green
Cleavage (basal)	(0001)

THALLIUM, Tl

General: Does not occur as a native element. There are three known single crystal minerals containing thallium in small quantities: vrbalite, a sulfide; hutchinsonite and lorandite, arsenides. All three minerals are closely associated with the arsenic sulfides realgar and orpiment — the former usually orange-red; the latter brilliant yellow. The thallium minerals are all brilliant red to red/black. Thallium is found as a trace element in many other minerals, notably, sphalerite, ZnS, and is more abundant than gallium and indium, the other two metals of the aluminum family of elements. It has a brilliant green spectrum which is used in scientific study.

Though it is reasonably abundant, the metal has little or no use in industry, but thallium salts are poisonous and have been used for killing rodents. In 1939, Iln, in Moscow, showed that the nitrate $TlNO_3$, when fed to sheep in small doses, caused them to molt like birds.

Technical Application: To date, the metal has had little or no use in Solid State device fabrication, in part, due to the highly poisonous nature of its salts though it has been evaluated as a silicide and may have dopant properties for compound semiconductors and other materials.

Thallium has been grown as a single crystal for physical data and morphological study to include diffusion reactions with metals such as gold and silver.

Etching: HNO_3, H_2SO_4, HCl and mixed acids HF:HNO_3, HF:H_2O_2.

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THALLIUM ETCHANTS

TL-0001
ETCH NAME: TIME:
TYPE: Acid, cleaning TEMP:
COMPOSITION:
1 .... 10% KCN
1 .... 10% $(NH_4)_2S_2O_8$
DISCUSSION:
Tl, (0001) wafers cut from single crystals used in a study of gold and silver diffusion. Solution used to clean surfaces.
REF: Meier, B W et al — *Acta Metall*, 16,1398(1968)

TL-0002
ETCH NAME: Nitric acid TIME:
TYPE: Acid, removal TEMP: RT
COMPOSITION:
x .... HNO_3
DISCUSSION:
Tl, polycrystalline specimens, Nitric acid is a slow, general etch for thallium, as is sulfuric acid. Hydrochloric acid is very slow.
REF: Anthony, T R et al — *J Appl Phys*, 39,1391(1968)

TL-0003a
ETCH NAME: Sulfuric acid TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... H_2SO_4, conc.
DISCUSSION:
Tl, specimens. Shown as an etch for thallium.
REF: Hodgman, C D — *Handbook of Chemistry and Physics*, 27th ed, Chemical Rubber Co., Cleveland, OH, 1943, 474

TL-0003b
ETCH NAME: Hydrochloric acid TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... HCl
DISCUSSION:
Ti, specimens. Shown as a slow etch on thallium.
REF: Ibid.

PHYSICAL PROPERTIES OF THALLIUM SELENIDE, Tl_2Se

Classification	Selenide
Atomic numbers	81 & 34
Atomic weight	487.74
Melting point (°C)	340
Boiling point (°C)	
Density (g/cm^3)	
Hardness (Mohs — scratch)	4—5

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Crystal structure (isometric — normal?)	(100) cube
Color (solid)	Grey
Cleavage (cubic)	(001)

THALLIUM SELENIDE, Tl_2Se

General: Does not occur as a natural compound. There are three listed thallium minerals, all as mixtures of arsenic and sulfur with lead or antimony. There has been no industrial use as the selenide.

Technical Application: The compound as Tl_2Se_3 has been grown in Solid State development in a study of possible new semiconducting compounds, but is not presently used for fabrication as a device.

Etching: Halogens.

THALLIUM SELENIDE ETCHANTS

TLSE-0001
ETCH NAME: BRM — TIME:
TYPE: Halogen, polish — TEMP: RT
COMPOSITION:
x .... x% Br_2
x .... MeOH
DISCUSSION:
Tl_2Se_3 and TlSe single crystals were grown in a study of new semiconducting compounds. Other compounds were Ag_2Se, Li_3Bi, SnSe, $SnSe_2$, In_2Tl_3, $AgInTe_2$, In_2Te_3, and Bi_2Te_3.
REF: Mooser, E & Pearson, W B — *Phys Rev*, 101,492(1956)

THALLIUM BISMUTH TELLURIDE ETCHANTS

TLBT-00011
ETCH NAME: Aqua regia — TIME:
TYPE: Acid, removal/polish — TEMP:
COMPOSITION:
3 .... HCl
1 .... HNO_3
DISCUSSION:
$TlBiTe_2$, grown as a single crystal n-type, degenerate semiconductor compound in a study of superconductivity. Also as TlBeTe and TlSnTe. Two general formulas shown: $T^{I}B^{II}C^{III}$ and $A^{III}B^{V}C_2^{V}$. Solution shown as a general etchant for tellurium and thallium compounds (TE-0022).
REF: Rogers, T S et al — *J Cryst Growth*, 61,400(1983).

PHYSICAL PROPERTIES OF THORIUM, Th

Classification	Actinide
Atomic number	90
Atomic weight	232.1
Melting point (°C)	1750
Boiling point (°C)	3850
Density (g/cm^3)	11.7
Specific heat (cal/g) 25°C	0.034
Thermal conductance (cal/sec)(cm^2)(°C/cm)	0.093

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Heat of fusion (k-cal/g-atom)	4.6
Latent heat of fusion (cal/g)	19.82
Heat of vaporization (k-cal/g-mole)	140
Atomic volume (W/D)	19.9
Electronegativity (Pauling's)	1.3
Covalent radius (angstroms)	1.65
Ionic radius (angstroms)	1.02 (Th^{+4})
Electrical resistivity (micro-em)	14
Electronwork function (eV)	3.51
Coefficient of thermal linear expansion ($\times 10^{-6}$ cm/cm/°C) 20°C	12.5
Poisson ratio	0.27
Young's modulus (psi $\times 10^6$)	10.5
Shear modulus (psi $\times 10^6$)	4
Cross section (barns)	7.4
Half-life ($\times 10^{10}$ years)	7.4
1st ionization potential (eV)	5.7
Hardness (Mohs — scratch)	4—5
Crystal structure (isometric — normal)	(100) cube, fcc
Color (solid)	Grey
Cleavage (cubic)	(001)

THORIUM, Th

General: Does not occur in nature as a metal element. The chief ore of thorium, as thorium oxide, ThO_2, is from the mineral monazite — a phosphate of cerium metals but with sufficient oxygen and silicon to form both thorium oxide and thorium silicate — and the mineral thorite, $ThSiO_4$ (like zircon in form and structure). The mineral thorotungstate is a mixture of tungsten and thorium oxides but is of rare occurrence and the formula has not been established.

Industrial use includes fabrication of incandescent gas mantels using thorium oxide.

Technical Application: No application in semiconductor processing, though thorium has been studied as a single crystal and polycrystalline element.

Etching: HCl, H_2SO_4 and aqua regia; slowly in HNO_3.

THORIUM ETCHANTS

TH-0001
ETCH NAME: TIME:
TYPE: Acid, cleaning TEMP:
COMPOSITION:
x .... HNO_3
x .... H_2SiF_6
DISCUSSION:
Th specimens. Solution used to etch clean surfaces. Produces a high metallic luster surface that is retained indefinitely even in air.
REF: Wallace, D C — *Phys Rev*, 120,84(1960)

TH-0002
ETCH NAME: Aqua regia TIME:
TYPE: Acid, polish TEMP:
COMPOSITION:
3 .... HCl
1 .... HNO_3

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DISCUSSION:
Th specimens. Referred to as a slow removal and polishing etch for thorium.
REF:

TH-0003
ETCH NAME:
TYPE: Electrolytic, polish/removal
COMPOSITION:
x .... 9 *N* H_2SO_4
x .... 11 *N* H_3PO_4

TIME:
TEMP: 130°F
ANODE: Th
CATHODE:
POWER: 150 A/ft²
1500 A/ft²

DISCUSSION:
Th specimens prepared for electroplating. High power used for removal; low power used for polishing. A thorium phosphate film on thorium surfaces will reduce the effect of oxidation and corrosion.
REF: Beach, J G & Schaer, G R — *J Electrochem Soc*, 106,392(1959)

TH-0004
ETCH NAME: Nitric acid
TYPE: Acid, removal
COMPOSITION:
x .... HNO_3, conc.

TIME:
TEMP:

DISCUSSION:
Th specimens. Solution shown as a general removal etch.
REF: Teasdale, T S — *Phys Rev*, 99,1248(1955)

PHYSICAL PROPERTIES OF THORIUM DIOXIDE, ThO_2

Classification	Oxide
Atomic numbers	90 & 8
Atomic weight	264
Melting point (°C)	1845
Boiling point (°C)	3220
Density (g/cm³)	9.5—9.9
Application limit (°C)	2700
Specific heat (°C) 25—1000°C	290
Coefficient of linear thermal expansion ($\times 10^{-6}$/cm/cm/°C) 25—800°C	9.5
Thermal conductivity (W/m °C) 1000°C	3
Electrical resistivity (ohm/cm $\times 10^4$) 500°C	2.6
(ohm/cm $\times 10^4$) 1200°C	1.5
Hardness (Mohs — scratch)	7
Index of refraction (n =)	2.20
Crystal structure (isometric — normal)	(100) cube
Color (solid)	White
Cleavage (cubic)	(001)

THORIUM DIOXIDE, ThO_2 (Thoria)

General: Occurs in nature as a native oxide, not as a singular mineral, but mixed with other rare earth elements such as cerium. The major source is the mineral monazite, a complex phosphate with sufficient oxygen and silicon to form both thorium oxide and thorium

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silicate. It is the most widely used compound of thorium as it produces a brilliant white light in the presence of oxygen without burning of itself. The other major ore is thorite, $ThSiO_4$.

Prior to 1900 the Welsbach gas mantel (99% ThO_2) was a major source of illumination before the advent of the light bulb. Camp lanterns using white gas under pressure as the ignition source are still manufactured. It is considered a high refractory oxide and, with other similar metallic oxides, is used as a ceramic material in the fabrication of high temperature ceramics for furnace bricks and similar applications.

Technical Application: No major use in the electrical or electronic industries other than as a mixed ceramic compound. It has been grown as a single crystal for physical data and morphology studies.

Etching: Soluble in hot H_2SO_4 and fluorine compounds/acids.

THORIUM DIOXIDE ETCHANTS

THO-0001
ETCH NAME: Sulfuric acid
TYPE: Acid, removal
TIME:
TEMP: Hot
COMPOSITION:
x .... H_2SO_4, conc.
DISCUSSION:
ThO_2, specimens. A general, slow etch for thoria.
REF: Hodgman, C D — *Handbook of Chemistry and Physics*, 27th ed, Chemical Rubber Co., Cleveland, OH, 1943, 477

THO-0002
ETCH NAME: Ammonium fluoride
TYPE: Acid, preferential
TIME: 5 min
TEMP: Boiling
COMPOSITION:
x .... x% NH_4F
DISCUSSION:
ThO_2, (111) wafers used in a study of slip and fracture. An Instron Hardness Tester with a heated tip was used to indent surfaces to produce fracture. The solution shown developed triangle etch pits on (111) surfaces.
REF: Edington, J W & Klein, W — *J Appl Phys*, 27,3906(1966)

PHYSICAL PROPERTIES OF THULIUM, Tm

Classification	Lanthanide
Atomic number	69
Atomic weight	169
Melting point (°C)	1545
Boiling point (°C)	1727
Density (g/cm^3)	9.33
Specific heat (cal/g) 25°C	0.038
Heat of fusion (k-cal/g-atom)	4.4
Latent heat of fusion (cal/g)	26.04
Heat of vaporization (k-cal/g-mole)	59
Atomic volume (W/D)	18.1
Electronegativity (Pauling's)	1.2
Covalent radius (angstroms)	1.56

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Ionic radius (angstroms)	0.87 (Tm^{+3})
Electrical resistivity (micro-ohms-cm) 298 K	67.6
Compressibility ($cm^2/kg \times 10^{-6}$)	2.47
Cross section (barns)	118 (125)
Magnetic moment (Bohr magneton)	7.62
Magnetic susceptibility ($\times 10^{-6}$ emu/mole)	26,000
1st ionization potential (eV)	5.61
Hardness (Mohs — scratch)	4—5
Crystal structure (hexagonal — normal)	$(10\bar{1}0)$ prism, hcp
Color (solid)	Grey
Cleavage (basal)	(0001)

THULIUM, Tm

General: Does not occur as a native element. It is a rare earth element in the yttrium group of lanthanides, and occurs in yttrium and gadolinium minerals: gadolinite, a silicate of iron and rare earths is the major ore. It has had no use in industry to date, due to scarcity.

Technical Application: The element has not been used in Solid State material processing to date, but single crystals of thulium and other rare earths have been studied for general morphology and defects.

Etching: Heat used to develop defects.

THULIUM ETCHANTS

TM-0001
ETCH NAME: Heat
TYPE: Thermal, preferential
COMPOSITION:
x .... heat
DISCUSSION:

Tm, single crystal specimens. Material developed in a growth study. After crystal growth, thermal annealing was used to develop defects, low angle grain boundaries and other structure. Additional elements grown were dysprosium, holmium, gadolinium, erbium, thulium, terbium and yttrium.

TIME:
TEMP:

REF: Nigh, H E — *J Appl Phys*, 34,3323(1963)

PHYSICAL PROPERTIES OF TIN, Sn

Classification	Metal
Atomic number	50
Atomic weight	118.69
Melting point (°C)	232
Boiling point (°C)	2270
Density (g/cm^3)	7.298
Thermal conductance (cal/sec)(cm^2)(°C/cm)	0.16
Specific heat (cal/g) 20°C	0.053
Latent heat of fusion (cal/g)	
Specific heat (cal/g) 20°C	0.054
Latent heat of vaporization (cal/g)	520
Atomic volume (W/D)	16.3
1st ionization energy (K-cal/g-mole)	169
1st ionization potential (eV)	7.442

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Electronegativity (Pauling's)	1.8
Covalent radius (angstroms)	1.41
Ionic radius (angstroms)	0.71 (Sn^{+4})
Linear coefficient of thermal expansion ($\times 10^{-6}$ cm/cm/°C) 20°C (0°C)	23 (19.9)
Electrical resistivity (micro-ohms) 20°C (0°C)	11.5 (11)
Vapor pressure (nm $\times 10^{-6}$) 1000 K	7.4
Magnetic susceptibility (cgs $\times 10^{-6}$) 18°C	0.027
Tensile strength (psi) 15°C	2100
Hardness (Mohs — scratch)	2—3
(Brinell — mm/kg)	3.9
Crystal structure (tetragonal — normal) alpha	(110) prism, 1st order
(hexagonal — normal) beta	$(10\bar{1}0)$ prism
Color (solid)	Tin-white
Cleavage (basal)	(001)/(0001)

TIN, Sn

General: Though tin does occur as a native element it is extremely rare, limited in quantity, and is often found in rounded grains associated with gold placer deposits. The minerals cassiterite and stannite, both SnO_2, are the major tin ores. In the presence of soda, usually sodium carbonate, Na_2CO_3, on charcoal, using a hot flame (Bunsen burner) the oxide is easily reduced to metallic tin. Tin was one of the seven metals known to ancient man, and the smelting of copper with tin lead to the Bronze Age. The name tin is Anglo-Saxon (German/Norse origin), and the symbol, Sn, comes from the Latin *stannum*.

Needless to say, it has been a major industrial metal since the beginning of man's metal culture. Today it is said that the U.S. alone uses enough tin every year to form a sheet, 10 ft wide, around the earth's equator. Its primary use is for "tin cans", actually a tin coating on steel, as tin does not tarnish when exposed to moist air and is not affected by organic acids in foods. Tin plating is done by dipping the part in a bath of molten tin using thin sheets of low-carbon steel. The iron is protected against corrosion as long as the plating remains intact. Tin can be recovered by heating scrap plated parts, as the tin m.p. is only 232°C, or by chlorinating to $SnCl_4$ and/or sodium hydroxide, NaOH to form Na_2SnO_2. The chloride, $SnO_2.2H_2O$, is used in electrolytic tin plating.

There are three crystal forms of tin: (1) grey tin, below about 18°C; (2) beta-tin, stable between 18—170°C and (3) delta-tin, stable above 161°C. The grey- or alpha-tin is known as "tin pest" as it can cause tin parts to crumble into powder even in cold weather. The beta- and delta-tin forms are the stable white-tins of industry.

Though there are a number of tin-lead solders, solder #62, m.p. 185°C, and #63, m.p. 220°C (with 2% silver) are two of the best known and generally used. They are commercially available in solid bars and wire form, but are widely used as rosin-core soldering wire . . . the rosin acting as a wetting flux to form a smooth, pure tin solder joint. And many parts, such as device lead wires, are pre-tinned by dipping in a molten tin bath before being soldered into a circuit board which, in turn, has been pre-soldered — heat is applied, the solder/tin flows — and forms the solder joint. There are dozens of soldering irons with replaceable tips of various sizes and geometries available for the heat source as hand-held irons or in automatic equipment. With PCB construction — plasticized linen boards with laminated copper — holes for discrete parts are drilled, the copper lacquered for pattern etching of the required electrical/electronic circuit to include mounting pads for individual devices if required. Parts can be individually inserted and soldered or the complete assembly passed through a belt furnace to solder all parts in a single heat cycle. And devices that fail are easily de-soldered, removed, and replaced. High reliability PCBs commonly have the copper circuits overplated with gold to reduce copper corrosion. Such "boards" are common in

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much of the current electrical and electronic equipment, such as automatic profiled cutting machines in the metals industries, computers, etc. . . . anything requiring an electronic-type circuit for operation, and used as an inserted board. If a part fails, the board is pulled and replaced.

In ancient times, bronze coins were used, but with the fall of Carthage cutting off the Mediterranean supply of tin, tin was replaced with lead. The study of ancient artifacts uses this tin-lead content for dating purposes. Today, of course, most coinage is silver/copper/nickel or gold and gold alloys, though stone, copper, iron, and aluminum all have been used in the past.

When a bar of crystalline tin is bent, the crystallites rub against each other producing the "tin cry". It is the only metal known that produces such a recognizable sound to the human ear.

Technical Application: In the Solid State semiconductor industry tin is used as a dopant species in some compound semiconductors and is grown as both binary and trinary semiconductor materials: lead tin telluride, PbSnTe, lead tin selinide, PbSnSe, and tin telluride, SnTe as examples. It is also used in fabricating the Josephson Junction device, Nb_3Sn, and is under study and development as an oxide, nitride and silicide. Its use as lead-tin solders, particularly in PCB construction, has already been mentioned and is representative of the wide use of tin in the electrical and electronic industries.

Needless to say, it has been widely studied as tin plating, both molten baths and electrolytic films, for literally hundreds of years. Both natural single crystals and artificially grown boules have been studied for physical and morphological data. As cut bars and spheres it is under study for its superconducting properties.

Etching: HNO_3, H_2SO_4, alkalies, aqua regia, and mixed acids. Deliquesces in HCl.

TIN ETCHANTS

SN-0001a
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
(A) x .... 5% HCl
x .... isopropyl alcohol
(B) x .... 10% fluoboric acid
x .... isopropyl alcohol
TIME: (A) 10 min
(B) 30 sec
TEMP: RT RT
DISCUSSION:

Sn, electroplated thin film. Solutions used in studying vapor phase fusion of pure electroplated tin.

REF: Bander, T L — *Solid State Technol*, March 1983, 141

SN-0002a
ETCH NAME:
TYPE: Acid, defect
COMPOSITION:
100 ml .... HCl
100 ml .... HN_4NO_3
500 ml .... H_2O
x .... 5×10^{-5} *M* $CuSO_4 \times 5H_2O$
TIME:
TEMP:
DISCUSSION:

Sn, (010) wafers used in an etch study. Solution will produce hillocks as dislocations. Dislocations move on (010) in both ⟨100⟩ and ⟨110⟩ directions.

REF: Ojima, K et al — *Jpn J Appl Phys*, 22,46(1984)

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SN-0001b
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
(A) x .... 5% HCl
100 ml .... H_2O
(B) x .... 10% Na_2S
10 ml .... H_2O
TIME: (A) 10 min
(B) 30—40 sec
TEMP: RT RT
DISCUSSION:
Sn, electroplated thin film. Solutions used in studying vapor phase fusion of pure electroplated tin.
REF: Ibid.

SN-0002b
ETCH NAME:
TYPE: Acid polish
COMPOSITION:
1 .... HNO_3
1 .... HAc
4 .... glycerin
TIME:
TEMP:
DISCUSSION:
Sn, white-tin single crystal ingots and wafers. Solution used to polish surfaces before defect etching. (**CAUTION** — Glycerin and nitric acid is a precursor mix for nitroglycerin.)
REF: Ibid.

SN-0003
ETCH NAME: Hydrochloric acid
TYPE: Acid, cleaning
COMPOSITION:
x .... HCl
TIME:
TEMP:
DISCUSSION:
Sn, shot. Clean with hydrochloric acid and vacuum melt to form high purity tin ingots. Material used as an epitaxy growth source for InSn/InP thin films.
REF: Chin, B H — *J Electrochem Soc*, 131,1372(1984)
SN-0012: Rutherford, R J — *Proc Am Soc Test Mater*, 24,739(1924)
Sn specimens. Solution used as a general removal and cleaning etch. Also used on Pb and Bi.

SN-0004
ETCH NAME: Nitric acid, dilute
TYPE: Electrolytic, saw
COMPOSITION:
1 .... HNO_3
1 .... H_2O
TIME:
TEMP:
ANODE: Sn
CATHODE:
POWER: 30 mA
DISCUSSION:
Sn, single crystal ingots cut into wafers. Use a plastic Saran thread as a cutting tool. Pass thread through the solution, then across the specimen. Also used to cut zinc ingots.
REF: Sternheim, G — *Rev Sci Instr*, 26,1206(1955)

SN-0005a
ETCH NAME:
TYPE: Acid, preferential
COMPOSITION:
x .... HCl
TIME:
TEMP:

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DISCUSSION:
Sn, single crystal sphere etched to finite crystal form.
REF: Yamamoto, M & Watanabe, J — *Met Abstr*, 8,493(1958)

SN-0005b
ETCH NAME: Aqua regia
TYPE: Acid, preferential
COMPOSITION:
3 .... HCl
1 .... HNO_3
TIME:
TEMP:
DISCUSSION:
Sn, (100) single crystal tetragonal (alpha) form used in a general study of etching.
REF: Ibid.

SN-0006a
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
20 .... $HClO_4$
10 .... butyl cellusolve
70 .... EOH
TIME:
TEMP: 15—20°C
ANODE: Sn
CATHODE: SS (2)
POWER: 12 V
DISCUSSION:
Sn, (001) and (111) — tetragonal alpha-tin single crystals — prepared for TEM study. Solution used to polish and etch thin specimens. The specimen edges were insulated with microstop, and one side was lacquered against etching. Another polish etch used the same constituents in a ratio of 5:10:85 at 10°C and 30 V. Solutions should be stirred slowly during the etch period. Wash in DI water, then EOH. Remove lacquer with acetone.
REF: Fourie, J T et al — *J Appl Phys*, 31,1136(1960)

SN-0006b
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
5 .... $HClO_4$
10 .... butyl cellusolve
85 .... EOH
TIME:
TEMP: 10°C
ANODE: Sn
CATHODE:
POWER: 30 V
DISCUSSION:
Sn, (001) and (111) wafers. Solution used as a final polish etch after an initial rough reduction etch. See SN-0006a.
REF: Ibid.

SN-0007
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
20 .... $HClO_4$
70 .... HAc
TIME:
TEMP:
ANODE:
CATHODE:
POWER:
DISCUSSION:
Sn, single crystal wires used in a study of paramagnetic effects in superconductors — resistance transition in tin. Solution used to polish specimens. Rinse in DI water, then alcohol.
REF: Meissner, H — *Phys Rev*, 109,668(1958)

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SN-0008
ETCH NAME: Neon
TYPE: Ionized gas, preferential
COMPOSITION:
x .... Ne^+ ions

TIME:
TEMP:
GAS FLOW:
PRESSURE:
POWER:

DISCUSSION:
Sn, single crystal specimens. Neon ion bombardment used to develop structure and orientation figures. Other metals studied were Al, Bi, Cd, Co, Cu, Mg, and Zn.
REF: Yurasova, V E — *Kristallografiya*, 2,770(1957)

SN-0009
ETCH NAME:
TYPE:
COMPOSITION:

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

DISCUSSION:
Sn, single crystal sphere machined on a lathe, to 1" +/− .0004 diameter. The amorphous layer left from cutting removed by electropolish. Sphere used in a study of magnetic moments in a superconducting sphere. Etch not shown.
REF: Teasdale, T S — *Phys Rev*, 99,1248(1955)
SN-0010: Jacquet, P A — *Int Tin R&D Council Bull*, 90,(1949)
Reference for electropolish etch on tin sphere in SN-0009.
SN-0011: Steinberg, F & Teghtsoonian, E — *Acta Metall*, 5,455(1957)
Tin, specimens used in a study of grain boundaries. Specimens flame cut prior to lapping, polishing and preferential etching.

SN-0014
ETCH NAME: Nitric acid
TYPE: Acid, cutting
COMPOSITION:
(1) 1 .... HNO_3
1 .... H_2O
(2) 200 ml .... HCl
50 g .... $FeCl_3$
250 ml .... H_2O

TIME: 30 min
TEMP: RT

DISCUSSION:
Sn, specimen, $^1/_4$" wafer in diameter. Terylene thread soaked in solution (1) used to cut both tin and zinc. Solution (2) used for aluminum, brass and copper. (*Note:* Excellent review of metal growth methods.)
REF: Honeycombe, R W K — *Met Rev*, 4,1(1959)

SN-0015
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
4 oz .... $NaNO_3$
x .... $Na_3PO_4.H_2O$
x .... H_2O (1/liter?)

TIME: 1—3 min
TEMP: 200—212°F
ANODE: Steel
CATHODE: Sn
POWER: 20—15 A/ft² &
2—6 V

DISCUSSION:
Sn specimens. Solution used to electropolish. Dip in HCl and NaCl to final clean after etching.
REF: ASTE Committee — *Tool Engineer's Handbook*, McGraw-Hill, New York, 1949

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PHYSICAL PROPERTIES OF TIN DIOXIDE, SnO_2 (Cassiderite)

Classification	Oxide
Atomic numbers	50 & 16
Atomic weight	150.7
Melting point (°C)	1127, decomposes
Boiling point (°C)	
Density (g/cm^3)	6.95
Refractive index (n =)	1.997—2.093
Hardness (Mohs — scratch)	6.7
Crystal structure (tetragonal — normal) alpha	(110) prism
(monoclinic — normal) beta	(100) pinacoid
Cleavage (basal)	(001)

TIN DIOXIDE, SnO_2

General: Occurs in nature as the minerals cassiterite and stannite, both SnO_2, and the two primary ores of tin. England was called the Cassiderides by the Romans (the "Tin" Isles) for the tin mines in Cornwall that the ancient world used in the making of bronze, Cu:Sn (30%) — the Bronze Age — dating back to around 6000 B.C., even before the rise of the Roman Empire, circa 550 B.C. It is closely associated with fluorine and boron minerals which indicates a pneumatolytic origin and is found in granite, quartz or pegmatitic rocks as veins and stringers and, in the Malay Penninsula and several South Pacific islands, in sand and stream gravels — a major source of tin, and one economic reason for the Japanese invasion of the area during World War II — fortunately for the Allies, major tin mines, similar to the worked-out mines in Cornwall, were discovered in Bolivia about the same time. The Bolivian deposits represent about 80% of the world tin reserves at the present time.

There is little direct use in industry as a working compound. The hydrated oxide, $SnO_2.xH_2O$, is called alpha-stannic acid or simply stannic acid (H_2SnO_3). See the general discussion under tin for major use of the element.

Technical Application: No major use in the electronics or electrical industries, at present. It is under evaluation and study as a surface protective thin film coating similar to that of silicon dioxide as it is quite inert to most acids. It has been grown and studied as a single crystal for physical and morphological data.

Etching: Very difficult to etch as it is inert in acids and deliquesces in alkalies. Some hot mixed acids can be used. Reacts with chromium and zinc in solutions.

SNO-0001a
ETCH NAME: TIME:
TYPE: Acid, removal TEMP: 90°C
COMPOSITION:
1 g .... Cr (add as needed)
20 ml .... HBr
20 ml .... H_2O
DISCUSSION:
SnO_2, thin films grown on silicon substrates coated with SiO_2:SnO_2/SiO_2/Si. This solution is better for removal repeatability than cathodic etching.
REF: Hsu, Y S & Ghandhi, S K — *J Electrochem Soc*, 127,1592(1980)

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SNO-0001b
ETCH NAME: Hydrochloric acid, dilute
TYPE: Electrolytic, removal
COMPOSITION:
1 .... HCl
5 .... H_2O
DISCUSSION:
TIME:
TEMP:
ANODE: Pt
CATHODE: Sn
POWER:

SnO_2, thin films deposited on SiO_2 coated silicon wafer substrates as: $SnO_2/SiO_2/Si$. Electrolytic removal is not as controllable and effective as the solution shown in SNO-0001a.
REF: Ibid.

SNO-0002
ETCH NAME: Hydrogen bromide, dilute
TYPE: Halogen, removal
COMPOSITION:
(1) 1 .... HBr
1 .... H_2O
(2) 1 .... HBr
1 .... H_2O
x .... Cr, pcs
DISCUSSION:
TIME:
TEMP: 110°C

SnO_2, thin films deposited on 1 mm glass slides or SiO_2 coated silicon wafers. After depositing SnO_2, overcoat with 1500 Å SiO_2. Use photo resist techniques to pattern the cap SiO_2 as a mask, removing SiO_2 with HF, then remove SnO_2 with solutions shown.
REF: Hau, Y-S & Ghandhi, S K — *J Appl Phys,* 126,1434(1979)

SNO-0003
ETCH NAME:
TYPE:
COMPOSITION:
DISCUSSION:
TIME:
TEMP:

SnO_2, thin films deposited by CVD in a study of material physical parameters. Refractive index of deposits is n = 1.860—1.953. No etch shown.
REF: Melsheimer, J & Ziegler, D — *Thin Solid Films,* 109,71(1983)

SNO-0004
ETCH NAME:
TYPE:
COMPOSITION:
DISCUSSION:
TIME:
TEMP:

SnO_2, (110) single crystal wafers as-grown. Mechanically polish down to 1000 Å thick. Material used in a study of the ultraviolet absorption edge of the material. (*Note:* At the time of this work the authors said there was no known etch for SnO_2.)
REF: Summitt, R et al — *J Phys Chem Solids,* 25,1465(1965)

SNO-0005
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
3000 ml .... 6 *N* HCl
20 g .... Cr, pcs
x .... Zn, pcs
TIME: 1—4 min
TEMP: 90°C

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DISCUSSION:
SnO_2 specimens. Shown as an etch for tin oxide. Add zinc during etching.
REF: Simon, P W — U.S. Patent 4,009,061 1977 (Burroughs Corp)

PHYSICAL PROPERTIES OF TIN SELENIDE, SnSe

Classification	Selenide
Atomic numbers	50 & 34
Atomic weight	197.66
Melting point (°C)	861
Boiling point (°C)	
Density (g/cm^3)	8.18
Hardness (Mohs — scratch)	6—7
Crystal structure (hexagonal — normal)	$(10\bar{1}0)$ prism
Color (solid)	Grey/black
Cleavage (basal)	(0001)

TIN SELENIDE, SnSe

General: Does not occur as a natural compound. There are less than a dozen tin minerals, including native tin in small quantities, as sulfides and oxides. The most important ore is cassiderite, SnO_2. Metallic tin has been an important metal element in industry since ancient times, but the selenide has had no applications to date.

Technical Application: Both SnSe and $SnSe_2$ have been grown as single crystals in a study of new semiconducting compounds, but there have been no devices fabricated in Solid State processing other than for evaluation of properties and characteristics.

Etching: HCl, HNO_3, alkalies, and aqua regia. Mixed acids and halogens.

TIN SELENIDE ETCHANTS

SNSE-0001
ETCH NAME: TIME:
TYPE: Halogen, polish TEMP: RT
COMPOSITION:
x .... x% Br_2
x .... MeOH
DISCUSSION:
SnSe and $SnSe_2$ single crystal ingots grown in a study of new semiconducting compounds. Other compounds studied were Ag_2Se, Li_3Bi, Tl_2Se_3, In_2Tl_3, $AgInTe_2$, In_2Te_3, and Bi_2Se_3.
REF: Mooser, E & Pearson, W B — *Phys Rev*, 101,492(1956)

PHYSICAL PROPERTIES OF TIN TELLURIDE, SnTe

Classification	Telluride
Atomic numbers	50 & 52
Atomic weight	246.31
Melting point (°C)	780
Boiling point (°C)	
Density (g/cm^3)	6.47
Hardness (Mohs — scratch)	6—7
Crystal structure (hexagonal — normal)	$(10\bar{1}0)$ prism

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Color (solid)	Grey/black
Cleavage (basal)	(0001)

TIN TELLURIDE, SnTe

General: Does not occur as a natural compound. There are only some four tin tellurite or tellurite minerals as hydrates, and the dioxide tellurite, TeO_2. Metallic tin has had wide use in industry since ancient times, but there has been no use, to date, of the telluride.

Technical Application: The telluride has been grown as a single crystal in general studies of Solid State materials. It also has been deposited as a thin film for general morphology study of the compound.

Etching: HCl, HNO_3, and mixed acids.

TIN TELLURIDE ETCHANTS

SNTE-0001
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
1 .... HNO_3
11 .... HCl
20 .... $K_2Cr_2O_7$, sat. sol.
TIME: Minutes
TEMP: RT
DISCUSSION:

SnTe, (100) wafers used in developing polish etching for this material. Stir the solution during the etch period. If a stain remains on the surface, dip etch in 20% NaOH and rinse in DI water. Surfaces show ripples and some pitting.
REF: Lorenz, M R — *J Electrochem Soc*, 112,240(1965)

SNTE-0002
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
100 cm^3 .... H_3PO_4
1 g .... NaOH
2 g .... agar-agar
TIME:
TEMP:
ANODE: SnTe
CATHODE:
POWER: 6 V
DISCUSSION:

SnTe, (111) ingot cut axially on the (111) and studied in the vicinity of the metallic compound SnTe. Both Sn and Te turn brown in color in this polish solution and SnTe compound shows no color. Also called stannous telluride.
REF: Umoda, J et al — *Jpn J Appl Phys*, 1,277(1962)

SNTE-0003
ETCH NAME: Nitric acid dilute
TYPE: Acid, float-Off
COMPOSITION:
x .... HNO_3
x .... H_2O
TIME:
TEMP: RT
DISCUSSION:

SnTe, thin films grown on muscovite mica (0001) substrates. After growth films were removed by the float-off technique for TEM structure study in the solution shown. See Mica, MI-0001.
REF: Davenere, A et al — *J Cryst Growth*, 70,452(1984)

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SNTE-0004
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... HF
x .... HNO_3
x .... HAc
DISCUSSION:

SnTe, amorphous thin films evaporated on glass substrates at near LN_2 temperature in a T_c study. Irreversible resistivity transformation is at about 10 K. T_c values decrease with film thickness. Material source for thin film was 80 mesh powder from SnTe Bridgman grown ingots. Also deposited thin films of GeTe. (*Note:* Solution mixtures shown are general removal etchants.)
REF: Fuki, K et al — *Jpn J Appl Phys*, 23,1141(1984)

PHYSICAL PROPERTIES OF TITANIUM, Ti

Classification	Transition metal
Atomic number	22
Atomic weight	47.90
Melting point (°C)	1668
Boiling point (°C)	3260
Density (g/cm^3)	4.5
Thermal conductance (cal/sec)(cm^2)(°C/cm)	9.41
Specific heat (cal/g) 25°C	0.1386
Heat of fusion (k-cal/g-atom)	3.7
Latent heat of fusion (cal/g)	104.5
Heat of vaporization (k-cal/g-atom)	106.5
Atomic volume (W/D)	10.6
1st ionization energy (K-cal/g-mole)	158
1st ionization potential (eV)	6.83
Electronegativity (Pauling's)	1.5
Covalent radius (angstroms)	1.32
Ionic radius (angstroms)	0.94 (Ti^{+2})
Coefficient of linear thermal expansion ($\times 10^{-6}$ cm/cm/°C)	8.5
Electrical resistivity (micro-ohm-cm)	47.8
Modulus of elasticity (psi $\times 10^6$)	14.7 (15.5)
Magnetic susceptibility (emu/g)	3.17
Poisson ratio	0.41
Cross section (barns)	6.1
Tensile strength (psi $\times 10^3$)	34—95
Elongation (% inch/inch 2 inches)	24—54
Hardness (Mohs — scratch)	2
(Vicker's — mm/kg)	80—100
Crystal structure (hexagonal — normal)	$(10\bar{1}0)$ prism
Color (solid)	Grey-silver
Cleavage (basal)	(0001)

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TITANIUM, Ti

General: Does not occur in nature as a native element. The most prominent ores are rutile, TiO_2, titanite, $CaTiSiO_5$ and ilmenite, $FeTiO_3$, though there are over 40 single crystal minerals containing titanium. It is widely distributed in igneous rocks, mostly as the oxide. Ordinary rutile is commonly brownish-red in various shades, but not black. There are several ferriferrous varieties containing up to 30% ferrous titanate, all black in color. The minerals brookite and octahedrite, both also TiO_2, differ in their melting temperatures, and all three oxide crystals have been grown artificially. The metal is part of the tin group and is the most inactive metal of the group, so much so, that the chloride hydrolizes in air. The metal will burn in air and is the only known metal that will burn in nitrogen.

Industrially, titanium is considered one of the light metals and, being physically inert to most acids at room temperature, is finding increasing application in the aircraft and space vehicle industries. It is a major alloy element in steels to increase strength. The chloride, $TiCl_4$, with water and moisture in the air is sufficient to cause fog and will hydrolyze as TiO_2 as white smoke which is used in sky-writing and for smoke screens in the military.

Technical Application: The metal is used as an evaporated thin film in contact metallization structures on many metals as the initial evaporant as it has excellent "tie-down" properties due to its affinity for oxygen: Ti:Au, Ti:TiAg:Ag as examples. As TiW (10% Ti), known as Ti-tungsten, it is used as a contact metallization (Au:TiW) and a buffer layer in heterostructure semiconductor devices. Because of its high affinity for oxygen, it is the primary "gettering" metal used as rods in ion pump vacuum systems to remove air and other gases to achieve high vacuum and ultra-high vacuum levels.

It is used to deposit surface thin films of TiO_2 and TiN as surface protectants similar to silicon dioxide, and under development as a silicide: TiSi, Ti_2Si, and $TiSi_2$ for use as a buffer layer in semiconductor heterostructures.

As an electrode, both pure metal and alloys, it is used as such in electrolytic etching and plating solutions. The bulk metal is used in device package construction of semiconductor and similar devices and, as wire mesh, is sometimes used to hold parts in cleaning and etching solutions. Loops of wire or slugs are the evaporation sources.

As pure metal sheets and alloys it is still under study, even more so as thin film Ti, TiO_2, TiN and the silicides. Titanium has been grown as a single crystal for physical and morphological data, and oxidation is used for structural study.

Etching: Soluble in most hot, dilute acids and alkalies, but not in HNO_3 or aqua regia (RT). Several mixed acids.

TITANIUM ETCHANTS

TI-0001a
ETCH NAME: Hydrochloric acid, dilute
TYPE: Acid, cleaning
COMPOSITION:
x .... 20% HCl
TIME:
TEMP: Hot
RATE: 50 mil/year (mpy)
DISCUSSION:

Ti, sheet and alloy specimens. Solution can be used to clean surfaces. Hot solutions of greater than 20% concentration will etch titanium.

REF: Bulletin ADV 215-10M-11-59, Crucible Steel Company of America, Pittsburgh, PA

TI-0004b: Ibid.

Ti specimens. Dilute solutions used at RT show very slow attack.

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TI-0001b
ETCH NAME: Trichloroacetic acid
TYPE: Acid, removal
COMPOSITION:
x .... $CCl_3COOC_2H_5$
DISCUSSION:
Ti, sheet and alloy specimens. Titanium is generally resistant to mono- and dichloroacetic acids, but is attacked rapidly with trichloroacetic acid.
REF: Ibid.
TIME:
TEMP:

TI-0001c
ETCH NAME: Formic acid
TYPE: Acid, removal
COMPOSITION:
x .... 25% HCOOH
DISCUSSION:
Ti, sheet and alloy specimens. Boiling solutions above 25% concentration will etch titanium.
REF: Ibid.
TIME:
TEMP: Boiling

TI-0001d
ETCH NAME: Phosphoric acid
TYPE: Acid, removal
COMPOSITION:
x .... 20% H_3PO_4
DISCUSSION:
Ti, sheet and alloy specimens. Hot phosphoric acid with greater than 30% concentration will attack titanium.
REF: Ibid.
TI-0004: Kohl, W H — *Handbook of Materials and Techniques for Vacuum Devices*, Reinhold, New York, 1967
Ti specimens etched at room temperatures with concentrated (85%) solution.
TIME:
TEMP: Hot

TI-0001e
ETCH NAME: Hydrofluoric acid
TYPE: Acid, removal
COMPOSITION:
x .... HF
DISCUSSION:
Ti, sheet and alloy specimens. Titanium is rapidly attacked by hydrofluoric acid and other fluorine compounds.
REF: Ibid.
TI-0004e: Ibid.
Ti specimens. A 1% HF solution, RT, will attack titanium rapidly.
TIME:
TEMP:

TI-0001f
ETCH NAME: Sulfuric acid
TYPE: Acid, removal
COMPOSITION:
x .... 20% H_2SO_4
TIME:
TEMP: Hot
RATE: 500 mil/year (mpy)

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DISCUSSION:

Ti, sheet and alloy specimens. Hot sulfuric acid with greater than 20% concentration will slowly attack and clean titanium.

REF: Ibid.

TI-0004d: Ibid.

Ti specimens. Used as a concentrated solution at 145°C or as a 10% solution at 158°C. Both solutions will clean and etch titanium.

TI-0002

ETCH NAME: Hydrofluoric acid, dilute — TIME:

TYPE: Acid, cleaning — TEMP:

COMPOSITION:

1 .... HF

1 .... H_2O

DISCUSSION:

Ti, rod (electrode). First, degrease in TCE, then MeOH and then acetone. Use solution shown to etch/clean the electrode prior to electrodeposition of CdSe.

REF: Tomkiewicz, N — *J Electrochem Soc*, 192,2016(1982)

TI-0003

ETCH NAME: — TIME:

TYPE: Acid, removal — TEMP:

COMPOSITION:

x .... HF

x .... HCl

x .... H_2O

DISCUSSION:

Ti, sheet specimens used as substrates for deposition of thin film $CdSe_{1-x}Te_x$. Solution etch/cleans and slightly roughens surfaces for better film adhesion.

REF: Russak, M A & Creter, C J — *J Electrochem Soc*, 131,556(1984)

TI-0004c

ETCH NAME: Hydrochloric acid — TIME:

TYPE: Acid, removal — TEMP: RT & hot

COMPOSITION:

x .... HCl, conc.

DISCUSSION:

Ti specimens. Concentrated hydrochloric acid will attack titanium rapidly at both room temperature or when used hot.

REF: Ibid.

TI-0004f

ETCH NAME: Potassium hydroxide — TIME:

TYPE: Alkali, removal — TEMP: Hot & RT

COMPOSITION:

x .... % KOH

DISCUSSION:

Ti specimens. Concentrated potassium and sodium hydroxide solutions will attack titanium.

REF: Ibid.

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TI-0005
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
1 .... HF
5 .... HNO_3
4 .... H_2O
DISCUSSION:
Ti specimens used in an oxide study. First: lap to a 1 μm finish with diamond paste. Second: degrease with hexane. Use solution shown to etch/clean surfaces to remove native oxide (about 20 Å thick). Oxidation showed the following results:

1. Heat on hot plate in air = gold color TiO_2
2. 600°C in muffle furnace = gold color TiO_2
3. Heat with flame = grey-colored oxide

(*Note:* The gold colored thin films are being used as a hard colored surface coating as a replacement for gold on many products.)

REF: Rolison, D E & Murry, R W — *J Electrochem Soc,* 131,336(1984)

TI-0006
ETCH NAME: TIME:
TYPE: Acid, removal TEMP: 32°C
COMPOSITION: RATE: 18 μm/min
1 .... HF
4 .... HNO_3
5 .... H_2O
DISCUSSION:
Ti thin films. Solution used to remove thin films at time and rate shown. See TIO-0008 for anodic oxidation of specimens.
REF: DeBerry, D W & Viehbeck, A — *J Electrochem Soc,* 130,248(1983)

TI-0007
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
1 .... HF
1 .... HNO_3
50 .... H_2O
DISCUSSION:
Ti specimens and thin films. Shown as a general etch for removing metallic titanium.
REF: Brochure: *Metals Catalog,* 1035-AMD-CHG/ID/RCG/0977/30M SSS-MRC

TI-0008
ETCH NAME: TIME: 2—3 sec
TYPE: Acid, removal TEMP: RT
COMPOSITION:
10 ml .... HF
20 ml .... HNO_3
70 ml .... H_2O

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DISCUSSION:

Ti, thin films sputtered on 2 μ" and 4 μ" surface finish alumina substrates as a AuTi (400 Å:100 μ") metallization in fabricating microwave circuits with a gold cap. Used photo resist techniques to open circuit pattern. Etch remove gold with tri-iodide solution (AU-0007). Use solution shown to remove titanium. Removal is extremely rapid but without undercutting of gold. Line definition down to one micron.

REF: Walker, P — personal application, 1984

TI-0024a: Brochure: *Applications Data for Kodak Photosensitive Resists,* Eastman Kodak Co., Rochester, NY, 1960, 91

Ti thin films. Solution shown as a removal and patterning etchant compatible with Kodak photo resists. Temperature used was 32°C with an 18 Å/minute removal rate.

TI-0004f

ETCH NAME:

TYPE: Acid, removal

COMPOSITION:

1 .... HF
1 .... H_2O_2
20 .... H_2O

TIME:

TEMP:

DISCUSSION:

Ti specimens. Shown as an etch for titanium.

REF: Ibid.

TI-0010

ETCH NAME:

TYPE: Electrolytic, polish

COMPOSITION:

3 .... HF
16 .... H_2SO_4
1 .... H_2O

TIME:

TEMP:

ANODE:

CATHODE:

POWER:

DISCUSSION:

Ti specimens used in a study of oxidation. Electropolish with this solution and DI H_2O rinse before oxidation.

REF: Mizushima, W — *J Electrochem Soc,* 108,b25(1961)

TI-0011a

ETCH NAME: Hydrofluoric acid, dil.

TYPE: Acid, removal

COMPOSITION:

10 ml .... HF
90 ml .... H_2O

TIME:

TEMP: 32°C

RATE: 12 Å/min

DISCUSSION:

Ti specimens and thin films. Used as a general etch for titanium.

REF: Missel, L I & Glang, R — *Handbook of Thin Film Technology,* McGraw-Hill, New York, 1970, 7

TI-0021a: Nichols, D R — *Solid State Technol,* December 1979

Ti thin film evaporation left on bell jars and internal fixtures of vacuum systems. Solution used to clean systems. Use on stainless steel and copper, but not recommended for glass or ceramic.

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TI-0011b
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
10 ml .... HF
20 ml .... HNO_3
70 ml .... H_2O
DISCUSSION:
Ti specimens and thin films. Used as a general etch for titanium.
REF: Ibid.

TI-0001f
ETCH NAME: Oxalic acid TIME:
TYPE: Acid, removal TEMP: Hot
COMPOSITION:
x .... $COOHCOOH.2H_2O$
DISCUSSION:
Ti specimens. Solution will etch titanium in all concentrations.
REF: Ibid.

TI-0012a
ETCH NAME: TIME:
TYPE: Halogen, removal TEMP:
COMPOSITION:
x .... Br_2
x .... ethyl acetate
DISCUSSION:
Ti, thin film deposit was etch removed with the solution shown.
REF: Abu-Yaron, A — *Thin Solid Films,* 112,349(1984)
TI-0022: Douglass, D L & Van Landuyt, J — *Acta Metall,* 14,491(1966)
Ti specimens used in a structure and morphology study of titanium oxidation. Solution was 10% Br_2 at 75°C. Will remove titanium but not titanium oxide. Authors say that using solution above 650°C will initiate growth of TiO_2 at points on surface followed by coalescence as an oxide thin film.

TI-0012b
ETCH NAME: TIME:
TYPE: Halogen, removal TEMP:
COMPOSITION:
x .... I_2
x .... MeOH
DISCUSSION:
Ti thin film deposit was etch removed with the solution shown. (*Note:* Iodine can be replaced with bromine.)
REF: Ibid.

TI-0013
ETCH NAME: TIME:
TYPE: Electrolytic, polish TEMP:
COMPOSITION: ANODE:
1 .... HF CATHODE:
1 .... MeOH POWER:

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DISCUSSION:

Ti specimens used for electroplating. Solution shown used to polish surfaces prior to plating. (*Note:* A major article on plating of transition metals, including both etchants and plating solutions.)

REF: Saubestre, E B — *J Electrochem Soc*, 106,305(1959)

TI-0014a: Stanley, C L & Brenner, A — *Proc Am Electroplat Soc*, 43,123(1956)

Reference used by Saubestre for the solution shown in TI-0013.

TI-0023

ETCH NAME: Tin chloride — TIME:

TYPE: Salt, polish — TEMP:

COMPOSITION:

x .... x% $SnCl_2$

DISCUSSION:

Ti specimens. Use as an immersion etch for this material.

REF: Beukman, F & Tucker, W — U.S. Patent 2,801,213, (1957)

TI-0015

ETCH NAME: — TIME:

TYPE: Electrolytic, polish — TEMP:

COMPOSITION: — ANODE:

x .... HF — CATHODE:

x .... HNO_3 — POWER:

x .... H_2SO_4

x .... $FeSO_4$

x .... $Al_2(SO_4)_3$

DISCUSSION:

Ti specimens. During etching period switch: cathode-anode-cathode.

REF: Dalley, J J — *Aviat Week*, 66,79(1957), May 13

TI-0016

ETCH NAME: — TIME:

TYPE: Acid, polish — TEMP:

COMPOSITION:

x .... HF

x .... HBF_4

DISCUSSION:

Ti, specimens. Use solution by immersion as a general polishing etch.

REF: Slomin, G W & Christensen, M P — U.S. Patent 2,798,843 (1957)

TI-0017

ETCH NAME: — TIME:

TYPE: Electrolytic, polish — TEMP:

COMPOSITION: — ANODE: Ti

x .... HF — CATHODE:

x .... ethylene glycol — POWER:

DISCUSSION:

Ti specimens. Solution used as a general anodic etch for titanium.

REF: Colner, W H et al — *J Electrochem Soc*, 110,486(1953)

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TI-0018
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
x .... x% $AlCl_3$
x .... ether
DISCUSSION:
Ti specimens. Solution used as a general anodic etch for titanium.
TIME:
TEMP:
ANODE: Ti
CATHODE:
POWER:
REF: Couch, D E & Brenner, A — *J Electrochem Soc*, 99,234(1952)

TI-0019a
ETCH NAME:
TYPE: Acid, removal/polish
COMPOSITION:
x .... HF
x .... $CuSO_4$
DISCUSSION:
Ti specimens. Solution used as an immersion etch on this material.
TIME:
TEMP:
REF: *Met Finish*, 55,46(1951)

TI-0019b
ETCH NAME:
TYPE: Acid, removal/polish
COMPOSITION:
x .... HF
x .... $Na_2Cr_2O_7$
DISCUSSION:
Ti specimens. Solution used as an immersion etch on this material.
TIME:
TEMP:
REF: Ibid.

TI-0020
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
x .... H_2SO_4
x .... H_3PO_4
DISCUSSION:
Ti specimens. Solution can be used as a mixed acid solution, or acids can be used alone as general anodic polishing etchants.
TIME:
TEMP:
ANODE: Ti
CATHODE:
POWER:
REF: Richards, R — *Corrosion*, 4,400(1950)

TI-0021b
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
1 .... NH_4OH
2 .... H_2O_2
DISCUSSION:
Ti thin film evaporation in vacuum systems. Solution used to remove and clean bell
TIME:
TEMP:

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jars and internal fixtures of titanium. Can be used on stainless steel, glass, copper, and ceramics.
REF: Nichols, D R — *Solid State Technol,* December 1979

TI-0014b
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
x .... HF
x .... CH_3COOH (HOAc)
TIME:
TEMP:
ANODE: Ti
CATHODE:
POWER:
DISCUSSION:
Ti specimens. Start etching by immersion, then apply AC current. (*Note:* The chemical acronym HOAc = HAc = GAA = glacial acetic acid.)
REF: Stanley, C L & Brenner, A — *Proc Am Electropl Soc,* 43,123(1956)

TI-0024
ETCH NAME: Heat
TYPE: Thermal, preferential
COMPOSITION:
x .... heat
TIME:
TEMP: Elevated
DISCUSSION:
Ti specimens used in a thermal etching study.
REF: Evans, P — *Acta Metall,* 5,342(1957)

TI-0026
ETCH NAME:
TYPE: Halogen, removal
COMPOSITION:
(1) x .... x% Br_2
x .... ethyl acetate
(2) x .... x% I_2
x .... MeOH
TIME:
TEMP: Hot
DISCUSSION:
Ti evaporated as thin films. Either solution can be used for etch removal. (*Note:* Both solutions can be used on other metals and metallic compounds as general removal, polishing or preferential etches.)
REF: Abu-Yaron, A — *Thin Solid Films,* 112,349(1984)

TITANIUM ALLOYS: TiM_x

General: Titanium alloys do not occur in nature but are artificially compounded, not only as titanium alloys, themselves, but as an additive to irons, steels, copper, tungsten, etc. as a strengthening and hardening agent.

Technical Application: The most widely used alloy in Solid State processing is as Ti-tungsten, TiW(10%Ti), although other concentrations of titanium have been evaluated. The material is used as a multilayer metallization with gold as contact pads in device fabrication, such as for gallium arsenide, indium phosphide and silicon diodes, FETs, etc.

The TiW compound presented in its own section under tungsten. The only reference, here, is one as a AuTi thin film.

Etching: HF:HNO_3. See TiW and W for additional solutions.

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TITANIUM ALLOY ETCHANTS

TITANIUM GOLD

TIAU-0001
ETCH NAME: TIME:
TYPE: Acid, thinning TEMP:
COMPOSITION:
1 .... HF
10 .... HNO_3
DISCUSSION:

AuTi thin films and SiO_2 both used as etching masks on silicon wafers containing epitaxy Si layers in the fabrication of photo diodes. Solution shown used to both etch photo resist patterned AuTi films and to thin silicon.
REF: Ataman, A — *J Appl Phys*, 49,5324(1978)

PHYSICAL PROPERTIES OF TITANIUM CARBIDE, TiC

Classification	Carbide
Atomic numbers	22 & 6
Atomic weight	60
Melting point (°C)	3140
Boiling point (°C)	4300
Application limits (°C)	1500
Density (g/cm^3)	6.5
Specific heat (mean)(J/kg °C)	1050
Linear coefficient of thermal expansion ($\times 10^{-6}$ cm/cm/°C)	6.9
Thermal conductivity (W/m °C) 1100°C	40
Electrical resistivity (ohms/cm)	9
Hardness (Mohs — scratch)	9+
(Knoop — kg/mm_2)	2470
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Grey-white
Cleavage (cubic)	(001)

TITANIUM CARBIDE, TiC

General: Does not occur in nature as a compound, though there are over 40 titanium containing minerals: ilmenite, $FeTiO_2$, rutile, TiO_2 and titanite, $CaTiSiO_5$ all important ores of titanium.

Titanium metal is a major alloying element for hardening steel, and is used as an electrode in electrolytic solutions. As the carbide or boride, it is considered one of the high temperature ceramic materials, and has been used as a thin film coating on cutting tools. The oxide, rutile, TiO_2, has long been used as a paint pigment as "titanium white".

Technical Application: The carbide has had little use in Solid State processing, to date, though it has been studied as a grown single crystal and as a thin film, both amorphous and single crystal, for both physical and electrical characteristics. Doped with a p-n junction, like silicon carbide, SiC, it will probably show semiconducting properties for application as a high temperature diode . . . see Silicon Carbide, and Cubic Boron Nitride, BN.

Etching: HNO_3 and aqua regia type solutions; electrolytic with mixed acids.

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TITANIUM CARBIDE ETCHANTS

TIC-0001
ETCH NAME: Argon
TYPE: Gas, cleaning
COMPOSITION:
x .... Ar
TIME:
TEMP: 100°C
DISCUSSION:
TiC, (100) wafers. After mechanical polishing, final clean surfaces in argon gas at 1000°C.
REF: Fukunda, S et al — *J Vac Sci Technol,* A2,50(1984)

TIC-0002
ETCH NAME:
TYPE: Electrolytic, preferential
COMPOSITION:
1 .... HF
1 .... HNO_3
6 .... H_2O
TIME: 1 sec
TEMP: RT
ANODE: TiC
CATHODE:
POWER: 3 V
DISCUSSION:
TiC, (001) cleaved wafers used in a study of Mondrian precipitation patterns. The compound has the cubic NaCl structure. Solution developed square pits with pointed bottoms as dislocations. The flat bottom pits are surface etch pits.
REF: Williams, W S — *J Appl Phys,* 32,552(1961)
TIC-0003b: Ibid.
TiC, (100) wafers. See discussion under TIC-0002.

TIC-0004
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
x .... HF
x .... HNO_3
x .... HAc
TIME:
TEMP:
ANODE: TiC
CATHODE:
POWER:
DISCUSSION:
TiC, (100) thin films as oriented deposits and used in a study of plastic deformation. Prior to deformation, specimens were electropolished in the solution shown.
REF: Hollax, G E & Smallman, R E —, *J Appl Phys,* 37,818(1966)
TIC-0009: Lee, C W et al — *J Vac Sci Technol,* 21,43(1982)
TiC thin films CVD deposited on WC substrates. At 1000°C deposition temperature, orientation was (111) grains with equal axes; at 1100°C grains were elongated with (110) orientation.

TIC-0005
ETCH NAME: Murakama's reagent
TYPE: Acid, cleaning
COMPOSITION:
10 g .... $K_3Fe(CN)_6$
10 g .... KOH (NaOH)
100 ml .... H_2O
TIME:
TEMP:
ASTM #98

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DISCUSSION:

TiC specimens. Used as a cleaning etch on titanium carbide surfaces. Also used on tungsten carbide, WC and tantalum carbide, TaC.

REF: Pamphlet: ASTM E407-70 (1972)

TIC-0006

ETCH NAME: Nitric acid
TYPE: Electrolytic, polish
COMPOSITION:
x .... HNO_3

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

DISCUSSION:

TiC single crystal ingot grown by the Float Zone (FZ) method under high-pressure helium. Spark erosion specimens cut into square prisms 0.2 × 0.2 mm, and weld a Ta contact wire. Electropolish into a tip point of 0.1 μm shape. Specimens used in a study of field emission properties of TiC.

REF: Fujii, K et al — *J Appl Phys*, 57,1723(1985)

TIC-0007

ETCH NAME:
TYPE:
COMPOSITION:

TIME:
TEMP:

DISCUSSION:

TiC, (001) cleave wafers used in a study of electrical conductivity and thermoelectric effects. The (001) cleavage is very prominent. No etch shown.

REF: Hollander, L E Jr — *J Appl Phys*, 32,997(1961)

TIC-0008a

ETCH NAME: Diamond
TYPE: Element, polish
COMPOSITION:
x .... diamond paste

TIME:
TEMP: RT

DISCUSSION:

TiC, (001) cleaved wafers used in a study of elastic constants. TiB_2 also studied. Both materials were also cut with a diamond saw and polished with diamond paste. Both materials appear to be harder than diamond. (*Note:* See properties section for Knoop hardness values . . . they are measurably less hard than diamond at H = 7000 Knoop.)

REF: Gilman J J & Roberts, B W — *J Appl Phys*, 32,1405(1961)

PHYSICAL PROPERTIES OF TITANIUM DIOXIDE, TiO_2

Classification	Oxide
Atomic numbers	22 & 8
Atomic weight	80
Melting point (°C)	1640
Boiling point (°C)	
Density (g/cm³)	4.18 (5.2)
Dielectric constant (e =) (100 KC) 26°C	190 // c-axis
	85 ⊥ c-axis
	85—100 pressed powder
Magnetic susceptibility ($\times 10^{-6}$ emu/g)	0.067

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Electrical conductivity (eV)	1.5—1.8
Electrical resistivity (ohms cm $\times 10^{13}$)	1
Transmission range (microns)	0.42—6.0
Optical gap width (eV)	3
Thermal activation energy (eV)	1.5
Debye temperature (K)	1184
Coefficient of linear thermal expansion	9.94 // c-axis
($\times 10^{-6}$ cm/cm/°C)	7.19 $\perp$ c-axis
Energy gap (Ev)	3.1
Refractive index (n=)	2.6—2.9
Hardness (Mohs — scratch)	6—6.5
(Vickers — kg/mm^2)	659—900
Crystal structure (tetragonal — normal)	(110) prism, 1st order
Color (solid)	Clear-yellowish
Cleavage (basal — distinct)	(001)

TITANIUM DIOXIDE, TiO_2

General: There are three natural minerals: rutile, octahedrite and brookite. Brookite is orthorhombic, the others are tetragonal with rutile having the higher melting point. Natural minerals are commonly highly twinned, prismatic with surface striations and, as they contain some iron, vary in brown coloration to near-black. The colorless varieties have a refractive index equivalent or higher than that of diamond, with faint yellow tint, called mock topaz; artificially grown clear, called titania, and used as a replacement for diamond in jewelry. The three minerals are the major ores of titanium.

Other than as titanium ores, the primary use in industry is as a high grade white pigment with superior opacity and stability for paints.

Technical Application: In Solid State operations TiO_2 is under development as a thin film surface coating on semiconductors and other metals as a replacement for silicon dioxide, and has been deposited as TiO, TiO_2, and Ti_2O_3 for a protective surface coating and, as TiO_2, as a thin film capacitor in planar circuit construction.

For use as a capacitor in general industrial applications it is either deposited as an amorphous thin film or grown, cut and used with single crystal orientation or processed in pressed powder form. The (001) orientation gives the highest dielectric constant as a single crystal element.

Both the natural crystals and artificially grown crystals have been the subject of wide study for morphology, electrical parameters, etc. As doped crystals, e.g., iron, etc., for semiconducting properties and used as filters, lenses, prisms.

Etching: Soluble in H_2SO_4 and alkalies, but insoluble in other single acids.

TITANIUM DIOXIDE ETCHANTS

TIO-0011a
ETCH NAME: Sulfuric acid
TYPE: Acid, removal
TIME: x minutes
TEMP: Hot to boiling
COMPOSITION:
x .... H_2SO_4
DISCUSSION:

TiO_2 as natural minerals. Rutile — colorless to brown/black. Octahedrite — brown to black. Brookite — brown to black. All of these natural forms of TiO_2 are soluble in sulfuric acid, but insoluble in other acids.

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REF: Weast, R C et al — *Handbook of Chemistry and Physics*, 65th ed, CRC Press, Boca Raton, FL, 1984, B154

TIO-0001a: Walker, P — personal application, 1980—1985

TiO_x, thin film residue from titanium sputter metallization on alumina and quartz substrates used for fabricating microwave circuits. Solution used in a cleaning sequence to remove a final grey fraction of TiO_x from substrates. Soak substrates until they are returned to their normal white color.

TIO-0007b: Ibid.

TiO_2 as natural rutile crystal specimens (110) oriented. Acid shown used boiling, 30 min to develop etch pits.

TIO-0003
ETCH NAME: Hydrochloric acid
TYPE: Acid, cleaning
COMPOSITION:
x .... HCl
TIME: 12 h
TEMP: RT
DISCUSSION:

TiO_2, natural rutile specimens. After soak in HCl, water rinse for 1 h. Cleaned surfaces prior to a magnetic susceptibility study. Rutile surfaces pick up ferro-magnetic dust probably from atmosphere.

REF: Senttle, F E — *Phys Rev*, 120,821(1960)

TIO-0004a
ETCH NAME: Aqua regia
TYPE: Acid, cleaning
COMPOSITION:
3 .... HCl
1 .... HNO_3
TIME:
TEMP: Boiling
DISCUSSION:

TiO_2, single crystal natural rutile crystals used to study electric charge transfer through specimens. Use solution to polish and clean surfaces; wash in warm x% KOH, rinse in DI water, then vapor clean in steam and dry at 125°C.

REF: Srivastava, K G — *Phys Rev*, 119,520(1960)

TIO-0005a
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
2 .... HF
25 .... NH_4F
5 .... H_2O
TIME:
TEMP: RT
RATE: 50 Å/sec
DISCUSSION:

TiO_2, thin film deposited on GaAs,(100) substrates by chemical plating with 3.5% Ti-isoperoxide or Ti-ethylhexoxide in isopropyl alcohol for 1 min. Hold over water (95% RH) for about 1 week — film growth between 500 and 2000 Å. Etch in the solution shown under nitrogen, rinse in IPA and MeOH under nitrogen. RF sputter etch with Ar^+ ions produces craters in film. Liquid etching, as shown, may produce some pits.

REF: Bertrand, P A & Fleischauer, P D — *Thin Solid Films*, 103,167(1983)

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TIO-0006
ETCH NAME:
TYPE: Acid, cleaning
COMPOSITION:
x .... H_2SO_4
x .... x% $K_2Cr_2O_7$
TIME:
TEMP:
DISCUSSION:
TiO_2, (001) basal oriented wafers used in a study of piezoresistivity in the oxide semiconductor rutile. After cleaning in solution shown, indium solder was used for contact purposes.
REF: Hollander, L E Jr et al — *Phys Rev*, 117,1469(1960)

TIO-0007a
ETCH NAME: Lithium hydroxide
TYPE: Salt flux, decoration
COMPOSITION:
x .... LiOH
TIME: Hours
TEMP: 450°C
DISCUSSION:
TiO_2, (110) natural single crystal rutile specimens. Molten flux of lithium salts used to decorate dislocations. Molten KNO_3, at 300°C, xx hours used to remove residual lithium after decorating.
REF: Johnson, O W — *J Appl Phys*, 35,3048(1964)

TIO-0008
ETCH NAME: Potassium chloride
TYPE: Electrolytic, oxidation
COMPOSITION:
x .... 1 *M* KCl
x .... *HCl
TIME:
TEMP:
ANODE: Ti
CATHODE: Ti (specimen)
POWER: 50 mV/sec @ 0.6—3 V

*for pH 3.45

DISCUSSION:
Ti thin films. A 10 nm thick anodic oxide was grown on specimens surfaces using the solution shown. See TI-0006 for titanium etch used prior to oxidation.
REF: DeBerry, D W & Viehbeck, A — *J Electrochem Soc*, 130,248(1983)

TIO-0004b
ETCH NAME: Potassium hydroxide
TYPE: Alkali, cleaning
COMPOSITION:
x .... x% KOH
TIME:
TEMP: Warm
DISCUSSION:
TiO_2 as natural single crystal rutile used in a study of electric charge transfer through bulk specimens. Solution used as a wash after etching with aqua regia. Follow KOH with heavy water wash, then steam clean and dry at 125°C.
REF: Ibid.

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TIO-0004c
ETCH NAME: Steam
TYPE: Gas, cleaning
COMPOSITION:
x .... steam
TIME:
TEMP: 1000°C (?)
DISCUSSION:
TiO_2 as natural single crystal rutile used to study electric charge transfer through specimens. After etching in aqua regia; wash in warm KOH and heavy DI water rinse, final cleaning done with steam, then dried at 125°C.
REF: Ibid.

TIO-0005b
ETCH NAME: Argon
TYPE: Ionized gas, cleaning/removal
COMPOSITION:
x .... Ar^+ ions
TIME:
TEMP:
GAS FLOW:
PRESSURE:
POWER:
DISCUSSION:
TiO_2 thin films deposited on GaAs (100) substrates in a film study. Film was chemically deposited and treated under high humidity. Both acid wet etching and argon RF sputtering used to remove the film. Ar^+ ions produced craters in the thin film.
REF: Ibid.

TIO-0009
ETCH NAME: BHF
TYPE: Acid, removal
COMPOSITION:
x .... HF
x .... NH_4F (40%)
TIME:
TEMP:
DISCUSSION:
TiO_2 thin films deposited on (111), n-type silicon substrates in a study of anti-reflective (AR) coatings for solar cells. $TiCl_4$ at 130—250°C applied by spray onto a rotating silicon substrate at about 16 μm/min with higher temperature increasing film hardness. Under 136°C there is only a milky film that can be wiped off with a cloth. Refractive index at 130°C was n = 2.13; at 250°C, n = 2.4. At 180°C film is a-TiO_2, and above is mc-TiO_2 (amorphous to microcrystalline). TiO_2 powder formed at spray nozzle can create pin holes in films; pin holes were 1—2 μm in diameter.
REF: Young, K S & Lam, Y W — *Thin Solid Films*, 109,169(1983)

TIO-0010
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
1 .... HF
5 .... HNO_3
4 .... H_2O
TIME:
TEMP:
DISCUSSION:
TiO_2, as a native oxide on titanium substrates being prepared for an oxidation study. Solution shown used as a native oxide removal etch, though it is a general etchant for titanium metal.
REF: Rolison, D E & Murry, R W — *J Electrochem Soc*, 131,336(1984)

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TITANIUM MONOXIDE ETCHANTS

TIO-0011b
ETCH NAME: Sulfuric acid, dilute
TYPE: Acid, removal
COMPOSITION:
x .... H_2SO_4
x .... H_2O
TIME:
TEMP:
DISCUSSION:

TiO, thin film deposits. Color is yellow to black, probably amorphous in structure. Material is slowly soluble in dilute sulfuric acid.

REF: Weast, R C et al — *Handbook of Chemistry and Physics*, 65th ed, CRC Press, Boca Raton, FL, 1984, B154

TIO-0001b: Walker, P — personal application, 1980—1985

TiO_x thin film residue from titanium sputter metallization on alumina and quartz substrates used for fabricating microwave circuits. Solution used in a cleaning sequence to remove the final grey fraction of a TiO_x remaining on substrates.

TITANIUM TRIOXIDE ETCHANTS

TIO-0011c
ETCH NAME: Sulfuric acid, dilute
TYPE: Acid, removal
COMPOSITION:
x .... H_2SO_4
x .... H_2O
TIME:
TEMP:
DISCUSSION:

Ti_2O_3 specimens and thin films. Color is velvet-black. All titanium oxides are slowly soluble in concentrated or dilute sulfuric acid. The trioxide is insoluble in HCl or HNO_3.

REF: Weast, R C et al — *Handbook of Chemistry and Physics*, 65th ed, CRC Press, Boca Raton, FL, 1984, B154

TIO-0001c: Ibid.

TiO_x as a thin film residue from titanium sputter metallization on alumina and quartz substrates used for fabricating microwave circuits. Solution used in a cleaning sequence to remove a final grey fraction of TiO_x from substrates.

PHYSICAL PROPERTIES OF TITANIUM NITRIDE, TiN

Classification	Nitride
Atomic numbers	22 & 7
Atomic weight	61.9
Melting point (°C)	3220
Boiling point (°C)	
Density (g/cm³)	5.3
Hardness (Mohs — scratch)	5—7
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Bronze-red/gold
Cleavage (cubic)	(001)

TITANIUM NITRIDE, TiN

General: Does not occur as a natural compound. The primary mineral is rutile, TiO_2.

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Titanium metal has been surface nitrided in industry, but there has been little other use of the nitride in metal processing. Based on its thin film color as bronze-red, it can be used as a surface coating replacement for gold in the decoration of clay products as a glaze.

Technical Application: Primary development in Solid State processing has been as a deposited thin film on silicon and other wafer materials as a dielectric type coating, and for wear resistance on metal tools.

Etching: Mixed acids HF:HNO_3; fluorine compounds; H_2O_2:HF. DCE with CF_4.

TITANIUM NITRIDE ETCHANTS

TIN-0003b
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
1 .... HF
27.5 .... HNO_3
10 .... H_2O
TIME:
TEMP: RT
DISCUSSION:
TiN thin films deposited on poly-Si. Solution used to pattern etch poly-Si with TiN acting as a patterning etch mask. Attack of TiN is 4× slower than on poly-Si.
REF: Ibid.

TIN-0003c
ETCH NAME: RF plasma
TYPE: Ionized gas, removal
COMPOSITION:
x .... CF_4
TIME:
TEMP: RT
ANODE: TiN
CATHODE:
PRESSURE: 58 mTorr
POWER: 200 W
DISCUSSION:
TiN thin films deposited on poly-Si. TiN etch rate was 240 Å/min. Used as a general removal etch for TiN or for selective removal and structuring.
REF: Ibid.

TIN-0004
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
10 ml .... HF
45 ml .... HNO_3
45 ml .... H_2O_2
TIME: Seconds
TEMP: RT
DISCUSSION:
TiN thin films deposited on Ti, (0001) substrates using NH_3 as nitrogen source. The film was deep gold in color. Also deposited HfN and ZrN.
REF: Dawson, P T — *J Vac Sci Technol*, 21,36(1982)

TIN-0006
ETCH NAME: BHF
TYPE: Acid, removal
COMPOSITION:
1 .... HF
5 .... NH_4F (40%)
TIME:
TEMP: Hot

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DISCUSSION:

TiN thin films. The films can be used as: (1) high temperature single surface absorbers; (2) silicon diffusion barriers; (3) a gold colored replacement for gold as a surface decorative coating; (4) a wear resistant coating on tools. Solution shown is a general removal etchant for TiN.

REF: Martin, P L et al — *J Vac Sci Technol*, A2(2),341(1984)

TIN-0001

ETCH NAME:

TIME:

TYPE: Acid, removal

TEMP: 60°C

COMPOSITION:

2 g .... EDTA (tetrasodium salt)
60 ml .... H_2O_2
120 ml .... H_2O

DISCUSSION:

TiN thin films deposited on silicon wafers as part of a metallization structure: Ni/TiN/Al and used in the fabrication of diodes. The nitride acts as a blocking layer. After nickel removal, the solution shown was used to remove TiN.

REF: Martin, P L et al — *J Electron Mater*, 13,309(1984)

TIN-0005

ETCH NAME:

TIME:

TYPE: Acid, removal

TEMP: 60°C

COMPOSITION:

2.8 g .... EDTA (tetrasodium salt)
60 ml .... H_2O_2
120 ml .... H_2O
5 ml .... NH_4OH

DISCUSSION:

TiN thin films deposited on (100) silicon wafers as part of a diode metallization structure: Ni/TiN/Al similar to TIN-0001, above. Remove nickel with: 10 ml HF:10 ml HNO_3:1000 ml H_2O. Remove aluminum with: 80 ml H_2PO_4:5 ml HNO:5 ml HAc; 10 ml H_2O, at 45°C. Remove TiN in solution shown.

REF: Finetti, N et al — *J Electron Mater*, 13,327(1984)

TIN-0003a

ETCH NAME: BHF

TIME:

TYPE: Acid, removal

TEMP: 0°C

COMPOSITION:

RATE: 2.5 g/sec & 1 g/sec

134 ml .... HF
452 g .... NH_4F
625 ml .... H_2O

DISCUSSION:

TiN thin films deposited on poly-Si epitaxy layers. Heated in air at 500°C will convert TiN to semi-insulating (SI), TiO_2. Heated in an inert atmosphere, will remain as TiN to above 900°C. Solution used to remove residual SiO_2 prior to TiN deposition.

REF: Whittmer, M et al — *J Appl Phys*, 54,1423(1983)

TITANIUM:TUNGSTEN, TiW

General: Does not occur as a native compound. There are a dozen tungsten single crystal minerals — the tungstates — of which, wolframite, $(Fe,Mn)WO_4$ and scheelite, $CaWO_4$ are primary ores of the metal. Wolframite varies from the mineral: ferberite, $FeWO_4$ to hubnerite,

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$MnWO_4$ and, depending upon the iron content, may be magnetic. Other metal:tungstates are cuprotungstate, $CuWO_4$; stolzite, $PbWO_4$, as well as the oxide, tungstite, WO_3 and sulfide, tungstenite, WS_2 and other minerals contain some molybdenum.

Pure tungsten and tungsten alloys are of major use in industry as they have the highest melting points of any known elements, high electrical efficiency and are inert to most single acids and alkalies. Mixtures of titanium and tungsten — called "Ti-tungsten" — are artificial alloys and are being used in semiconductor fabrication.

Technical Application: Titanium:tungsten alloys are in use and under development as amorphous and polycrystalline thin film RF sputter deposites on semiconductor wafers as a blocking layer against crossover diffusion and reaction of other metals in multilayer structures. Although several alloy concentrations of titanium in tungsten have been evaluated, the most widely used is 10% titanium: TiW:(10%Ti) — commonly known as Ti-tungsten.

Due to the high melting point of tungsten, deposition of thin films is by RF sputtering in argon from a solid target of Ti-tungsten as an amorphous coating a-TiW. By controlled conditions of deposition and annealing, it is possible to produce colloidal structure, c-TiW and even single crystal — monoclinic — (001) basal structure like wolframite.

Etching: H_2O_2, HNO_3. Alkalies and ionized gases. See Tungsten

TI-TUNGSTEN ETCHANTS

TIW-0001
ETCH NAME: Hydrogen peroxide
TYPE: Acid, removal
COMPOSITION:
x .... H_2O_2, conc. (30%)
TIME: To 1 h
TEMP: RT
RATE: 20—30 μ"/sec
DISCUSSION:

TiW thin films. Described as a general etch for Ti-Tungsten. Sputter metallization from a TiW (10% Ti) target as RF or DC sputter in an argon atmosphere. (*Note:* Can also be sputtered with O_2 or N_2 in argon as a glassy passivation surface coating of TiO_2 or TiN.)
REF: Bulletin 1116B/Rev-HPD/VB/TM/0883/1K, MRC
TIW-0002: Walker, P et al — personal application, 1980—1986

TiW, thin films RF sputter deposited in argon on alumina and quartz blanks for fabricating microwave circuits. Metallization is Au:TiW (400 Å:100 μ"). Used tri-iodide solution to remove gold and hydrogen peroxide to remove TiW. In cleaning substrates for complete removal of metallization: (1) soak 5 min to remove bulk of TiW in solution shown; (2) if grey film remains, soak in HF and then H_2SO_4; (3) if an orange film remains (a hydrated WO_x) soak in hot 20—30% KOH.

TIW-0003
ETCH NAME: RIE
TYPE: Ionized gas, removal/pattern
COMPOSITION:
x .... CCl_4
x .... O_2 (40—50%)
TIME:
TEMP:
GAS FLOW:
PRESSURE:
POWER:
DISCUSSION:

TiW, thin films sputter deposited on silicon substrates in a study of etch removal from the following multilayer structures: SiO_2/TiW/Si; Al/TiW/Si; Si_3N_4/TiW/Si and AlCu/TiW/Si. After thin film formation, wafers were photo resist patterned using standard techniques: (1) P/R pattern and etch remove SiO_2 and Si_3N_4 with BHF; (2) with P/R pattern, deposit Al or AlCu and removal with acetone as standard lift-off technique, and (3) RIE gas etch remove

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TiW. This is a method of selective etching called subtractive etching as removal etch rate is minimal on all components other than TiW.
REF: Schaible, P M & Schwartz, G C — *J Electrochem Soc*, 132,730(1985)

TIW-0004
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
1 .... NH_4OH
2 .... H_2O_2
DISCUSSION:

TiW (1%Ti) thin films. This solution is recommended for use in cleaning vacuum systems for titanium/tungsten removal from stainless steel, glass, copper and ceramics. It also can be used as an etch solution of TiW thin films.
REF: Nichols, D R — *Solid State Technol*, December 1979
TIW-0005: Nowicki, R S et al — *Thin Solid Films*, 53,195(1978)

Ti_3W_7 thin films sputter deposited on an aluminum film evaporated on (111) silicon wafers used in a study of film morphology and, with a gold cover evaporation, cross-over diffusion reactions. Microstructure analysis was done with Au/TiW films deposited on graphite. Pinhole density was evaluated with TiW deposited on glass substrates using a microscope with transmitted light.

TIW-0006
ETCH NAME: Aqua regia TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
3 .... HCl
1 .... HNO_3
DISCUSSION:

$Ti_3W_7Si_2$, thin films on silicon wafers, (111), (110) and (100) oriented. A composite target of the Ti/W was RF sputtered on the silicon wafers and annealed at 350°C to form the silicide and was unaffected in temperatures from 675 to 900°C. Once formed, the films were unaffected by hot H_2O_2, HF or CP4 and dissolved only slowly in aqua regia.
REF: Harris, J M et al — *J Electrochem Soc*, 123,120(1976)

PHYSICAL PROPERTIES OF TUNGSTEN, W

Classification	Transition metal
Atomic number	74
Atomic weight	183.85
Melting point (°C)	3387 (3370)
Boiling point (°C)	5927 (5900)
Density (g/cm^3)	19.3 (same as gold)
Thermal conductance (cal/sec)(cm^2)(°C/cm)	0.40
Specific heat (cal/g) 25°C	0.032
Latent heat of fusion (cal/g)	44
Heat of fusion (k-cal/g-atom)	8.05
Heat of vaporization (cal/g)	197
Atomic volume (W/D)	9.53
1st ionization energy (K-cal/g-mole)	184
1st ionization potential (eV)	7.98

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Electronegativity (Pauling's)	1.7
Covalent radius (angstroms)	1.30
Ionic radius (angstroms)	0.62 (W^{+6})
Electrical resistivity (micro-ohms-cm) 25°C	5.6
Tensile strength (psi)	500,000
Thermal expansion (ppm/°C)	4.6
Vapor pressure (°C)	5168
Electron work function (eV)	4.5
Hardness (Mohs — scratch)	6—6.5
Crystal structure (isometric — normal) alpha	(100) cube bcc
(isometric — normal) beta	(100) cube fcc
Color (solid)	Grey-black
Cleavage (cubic — poor)	(001)

TUNGSTEN, W

General: Does not occur as a native element. There are two groups of tungsten oxides: (1) wolframite group, monoclinic, as Fe, Mn, and $PbWO_4$ and (2) scheelite group, tetragonal, as Ca, Pb and $CuWO_4$. Wolframite, $(Fe,Mn)WO_4$ and scheelite, $CaWO_4$ are the two primary ores of tungsten. As the pure metal, tungsten is hard, brittle, nonmagnetic and oxidizes readily when heated in air.

Tungsten is of major importance in industry. Because of its high electrical efficiency it is the primary filament in light bulbs and is a major alloy element in irons and steels for increased hardness and wear resistance armor plate, projectile casings and high speed cutting tools in addition to furnace linings and high temperature crucibles.

Technical Application: Although pure tungsten is not used in Solid State device fabrication it is used as a block or disc heat sink for mounting purposes as it has a very close thermal coefficient of expansion match with silicon and has been used both as single crystal and pressed powder parts. As a crystalline wire it is probably the most widely used test probe of all metals for DC electrical testing of semiconductor devices in wafer or discrete device form.

As an iron or steel alloy it has many uses in processing equipment. Titanium tungsten (10% Ti) — commonly called Ti-tungsten — is finding more and more use as the initial evaporation layer on semiconductor wafers and circuit substrates. It has been evaluated with other concentrations of titanium, but the 10% Ti has become a general industrial standard. Ti-tungsten is commonly RF sputter deposited from a solid target and followed by copper, gold or other metal layers.

It is under development and evaluation as a silicide: WSi, W_2Si, and WSi_2 for use as a buffer layer in semiconductor heterostructures. It also is fabricated as the carbide, WC, and boride, WB and may have application as a thin film amorphous surface coating as either of these compounds.

Although most studies have been with crystalline tungsten, it has been grown as a single crystal and as a deposited amorphous thin film for general evaluation.

Etching: H_2O_2, HF, alkalies and mixed acids.

W-0001a

ETCH NAME: Sodium hydroxide — TIME: 15 min

TYPE: Alkali, cleaning — TEMP: Boiling

COMPOSITION:

x .... 20% NaOH

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DISCUSSION:

W, specimens and parts. Used for general cleaning of tungsten surfaces.

REF: Kohl, W H — *Handbook of Materials and Techniques for Vacuum Devices*, Reinhold, New York, 1967

W-0002a Walker, P — personal application, 1957—1980

W, filaments and boats used for metal evaporation. Clean in boiling solution 5—15 min and heavily water wash with IR heat lamp or N_2 gas drying. Before commercial parts were on the market and each laboratory wound their own coils or made their own boats (mid-1950s), parts were placed in vacuum attached to electrodes and fired to red heat, before being loaded with metal for evaporation. Pump to 1×10^{-6} Torr, heat to 800—1000°C, 2—5 min. Also used HF soak and water wash.

W0003a: Booklet: Tungsten, Schwartzkopf Development Corp, 395/1/71

W, specimens. Alkali, alone, will not etch tungsten but can be used as a cleaning solution. With addition of oxidizers (KNO_3 or PbO_2) becomes a rapid removal etch for tungsten.

W-0003b

ETCH NAME: Hydrofluoric acid — TIME: 5—15 min

TYPE: Acid, removal — TEMP: RT

COMPOSITION:

x .... HF or 1 .... HF
1 .... H_2O

DISCUSSION:

W specimens. Both concentrated and dilute solutions of HF will slowly etch tungsten.

REF: Ibid.

W-0002b: Ibid.

W, coils and boats used for metal evaporation. (See W-0002a)

W-0004: Stacy, W T et al — *J Electrochem Soc*, 132,444(1985)

W, thin film deposits. Substrates were silicon, (111), p-type 7—21 Ω cm resistivity: bare silicon or with SiO_2 and with aluminum, thin films. Use photo resist techniques to open patterns. Selectively deposit tungsten from WF_6 by LPCVD. Clean surfaces before W deposition with either HF or DCE $CF_4 + O_2$. HF gives good results; the ionized gas etching is erratic.

W-0005

ETCH NAME: Hydrogen peroxide — TIME:

TYPE: Acid, removal — TEMP:

COMPOSITION:

x .... H_2O_2

DISCUSSION:

W, thin films deposited by LPCVD. Substrates were bare silicon wafers, with SiO_2 or with $TaSi_2$ thin films. Photo resist techniques were used to pattern and open windows through the SiO_2 and the $TaSi_2$ before selective deposition of tungsten. Solution used in step-etching for tungsten thickness measurement. (*Note:* Good discussion of stress and adhesion of tungsten.)

REF: Green, M L & Levy, R A — *J Electrochem Soc*, 132,1243(1985)

W-0036c: Ibid.

W single crystal wafers of various orientations used in a study of etched surface properties. 30% H_2O_2 used boiling for 30 min.

W-0030c: Ibid.

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W, specimens from vapor grown crystal used in a study of macro-structure. Solution was 10%, boiling for 20 sec for polish and removal. (*Note:* 30% H_2O_2 is standard concentration.)
W-0002c: Ibid.

W, thin films deposited as TiW(10% Ti) on alumina and quartz substrates used in fabricating microwave circuits. Sputter deposition was: Au:TiW (400 Å:100 μ"). After removing gold pattern with tri-iodide, tungsten was removed with this solution at RT, with 5 min soak and DI water wash. If there is a remaining grey fraction, soak in HF; if the fraction is an orange gel, soak in 20—30% KOH, hot — both are oxides of tungsten.
W-0001b: Ibid.

W, specimens. Used 5% H_2O_2 boiling and shows the solution as a slow etch.

W-0006
ETCH NAME:
TIME:
TYPE: Acid, polish
TEMP:
COMPOSITION:
x .... 25% $CuSO_4$
x .... NH_4OH
DISCUSSION:
W, specimens used in an etch study. Solution used to polish tungsten specimens.
REF: Wolff, V E — *Acta Metall*, 6,556(1958)

W-0001c
ETCH NAME: Nitric acid
TIME:
TYPE: Acid, removal
TEMP: Warm
COMPOSITION:
x .... HNO_3
DISCUSSION:
W, specimens. Very slight attack with formation of yellow/orange colored WO_3.
REF: Ibid.

W-0001d
ETCH NAME: Aqua regia
TIME:
TYPE: Acid, removal
TEMP: Hot
COMPOSITION:
3 .... HCl
1 .... HNO_3
DISCUSSION:
W, specimens used in an etch study. Little or no attack at room temperature. Slow attack used warm to hot.
REF: Ibid.

W-0001e
ETCH NAME: Sulfuric acid
TIME:
TYPE: Acid, removal
TEMP: RT & hot
COMPOSITION:
(1) x .... H_2SO_4, conc.
(2) x .... H_2SO_4
x .... H_2O

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DISCUSSION:
W specimens. Both concentrated and dilute solutions show slow attack used hot or at room temperature.
REF: Ibid.

W-0001f
ETCH NAME: Hydrochloric acid
TYPE: Acid, removal
COMPOSITION:
(1) x .... HCl
(2) x .... HCl
x .... H_2O
TIME:
TEMP:
DISCUSSION:
W specimens. Both concentrated and dilute solutions show slow attack used hot or at room temperature.
REF: Ibid.

W-0002d
ETCH NAME:
TYPE: Acid, cleaning
COMPOSITION:
3 .... HF
2 .... HNO_3
1 .... HAc
TIME: 3—5 min
TEMP: RT
RATE: 0.1 mil/5 min
DISCUSSION:
W discs of pressed powder and cut sheet used as heat sinks in SCR fabrication. Solution used to clean discs before silicon alloy.
REF: Ibid.

W-0001g
ETCH NAME:
TYPE: Acid, cleaning
COMPOSITION:
5 .... HNO_3
3 .... H_2SO_4
2 .... H_2O
TIME:
TEMP:
DISCUSSION:
W specimens. Solution used to clean specimens. Rinse in chromic acid:water solution.
REF: Ibid.

W-0001h
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
1 .... HNO_3
4 .... HF
TIME:
TEMP: Hot
DISCUSSION:
W specimens. Solution shows very rapid attack.
REF: Ibid.

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W-0007
ETCH NAME:
TYPE: Acid, cleaning
COMPOSITION:
33 .... HCOOH (formic acid)
33 .... H_2O
33 .... H_2O_2
TIME: 4—5 min
TEMP: 60—70°C
RATE: 0.1 mil/5 min
DISCUSSION:
W, specimens. Used in a study of surface cleaning.
REF: *Bell Tel Systems Tech Publ* #3143, 1958, 61

W-0002e
ETCH NAME:
TYPE: Acid, cleaning
COMPOSITION:
10 g .... $2NH_4NO_3.Cd(NO_3)_3.4H_2O$
50 ml .... HNO_3
50 ml .... H_2O
TIME: 20—30 sec
TEMP: RT
DISCUSSION:
W, wire 0.015—0.025 diameter. In working with chrome etch solutions it was shown that this mixture will etch clean tungsten wire.
REF: Ibid.

W-00011
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
305 g .... $K_3Fe(CN)_6$
44.5 g .... NaOH
1000 ml .... H_2O
TIME:
TEMP:
DISCUSSION:
W specimens. Solution is a very rapid etch.
W-0029: Gulbransen, E A & Andrews, K F — *J Electrochem Soc,* 107,619(1960)
W, specimens used in a study of the kinetics of oxidation of pure tungsten from 500 to 1300°C. Solution mixture was not shown but was used as a "scale" removal etch, e.g., removal of native oxide and other surface contamination. Follow with cleaning in petroleum ether and then ethyl alcohol.
REF: Ibid.

W-0003b
ETCH NAME:
TYPE: Acid, clean or electropolish
COMPOSITION:
80 g .... $KClO_3$
10 g .... NaOH
40 g .... KCO_3
1000 ml .... H_2O
TIME:
TEMP:
ANODE: W
CATHODE: SS screen
POWER: 0.05—0.5 A/cm^3
DISCUSSION:
W specimens. Use as a pickling solution without current to clean tungsten. As an electrolytic etch will produce a bright etch polish finish.
REF: Ibid.

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W-0008
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

DISCUSSION:
W, poly-W ribbon. Electropolishing solution not shown. Specimens used in a study of oxide formation with and without sulfur diffusion. Clean in vacuum at 2500 K for 10 h. Before each evaporation flash clean at 2500 K, 5—10 sec.
REF: Ishikawa, K & Tanemura, M — *Jpn J Appl Phys*, 25,850(1984)

W-0030a
ETCH NAME: Sodium hydroxide
TYPE: Electrolytic, polish
COMPOSITION:
x .... 0.1% NaOH

TIME: 1 h
TEMP: RT
ANODE: W
CATHODE:
POWER: 50 mA/cm^2 & 10 V

DISCUSSION:
W, specimens from vapor grown single crystals used in a study of sub-crystal macro-structure. Solution used to electropolish specimens. After polish, etch in 1 10%$K_2Fe(CN)_6$:3% NaOH, RT, 1 min; then in boiling 10% H_2O_2, 20 sec and again electrolytic using 1% NaOH 10—20 sec at 1V. This etch sequence will develop macro-structure.
REF: Rieck, G D & Bruning, H A M — *Acta Metall*, 8,97(1960)

W-0030b
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
1 .... 10% $K_2Fe(CN)_6$
1 .... 3% NaOH

TIME: 1 min
TEMP: RT

DISCUSSION:
W, specimens cut from vapor grown crystals used in a study of macro-structure. Solution used following electropolish. See W-0030a.
REF: Ibid.

W-0009
ETCH NAME: Xenon fluoride
TYPE: Ionized gas, removal
COMPOSITION:
(1) x .... XeF_2^+
(2) x .... XeF_2^+
x .... Ar^+

TIME:
TEMP:
GAS FLOW:
PRESSURE:
POWER:

DISCUSSION:
W, (111) wafers as deposited thin film. Etch product is mainly WF_6 (gas) during ion etching. Addition of argon enhances etch rate.
REF: Winters, H F — *J Vac Sci Technol*, B3,9(1985)

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W-0010
ETCH NAME: Carbon tetrafluoride
TYPE: Ionized gas, removal
COMPOSITION:
(1) .... CF_4^+
(2) .... SF_6^+
DISCUSSION:
W, thin films deposited on borosilicate glass to 0.4 μm thick. Reaction chamber 7" diameter with SS electrodes. Place specimens on bottom electrode heated to 60°C.
REF: Tang, C C & Hess, D W — *J Electrochem Soc*, 131,115(1984)

TIME:
TEMP:
GAS FLOW:
PRESSURE:
POWER:

W-0011
ETCH NAME:
TYPE: Ionized gas, removal
COMPOSITION:
x .... $CBrF_3$
DISCUSSION:
W, thin film evaporated on silicon, (100) substrates with a nickel mask evaporated over tungsten. Also chromium then on silicon, then tungsten. Chrome acts as an etch-stop in RIE etching with this gas.
REF: Schattsnberg, M L — *J Vac Sci Technol*, B3,12(1985)

TIME:
TEMP:
GAS FLOW:
PRESSURE:
POWER:

W-0012
ETCH NAME: Sodium hydroxide
TYPE: Electrolytic, polish
COMPOSITION:
x .... 5—20% NaOH
DISCUSSION:
W, wires 0.010 to 0.060 diameter. The 5% mixture is used as a general cleaning solution. The 15—20% mixture is used to form probe points on 0.060 wires with a dip-and-pull method: about 10 dips with slow pull-out produces a tapered, fine point. Second method is to immerse until a stub-tip is formed. Produces a fine polished surface.
REF: Lucky, H — personal communication, 1979
W-0013: Everson, K M et al — *J Appl Phys*, 57,956(1985)
W, whisker, 25 μm diameter electrolytically pointed. Either a 25° sharp point or a 40—50° blunt point. A poly-Ni substrate was mechanically polished then nickel evaporated in forming a W-NiO-W antenna as a MIM diode.
W-0014: Carroll, R W — *J Appl Phys*, 39,2339(1968)
W, wire electropolished in the solution shown.

TIME:
TEMP: RT
ANODE: W
CATHODE: SS screen
POWER: 20—30 mA/cm^3 &
12—15 V

W-0015
ETCH NAME: Acetone
TYPE: Keytone, cleaning
COMPOSITION:
x .... CH_3COCH_3
DISCUSSION:
W specimens. Used to degrease and clean surfaces.
REF: Bronnes, R T — *J Appl Phys*, 36,1445(1963)

TIME:
TEMP:

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W-0016
ETCH NAME: TIME:
TYPE: Alcohol, cleaning TEMP:
COMPOSITION:
(1) x .... petroleum ether (2) x .... EOH
DISCUSSION:
W specimens. Used to degrease and clean surfaces in an oxidation study.
REF: Gulbransen, J & Andrews, R C — *J Appl Phys*, 36,1540(1963)

W-0017a
ETCH NAME: Sodium hydroxide, dilute TIME:
TYPE: Electrolytic polish TEMP:
COMPOSITION: ANODE:
x .... 2% NaOH CATHODE:
POWER:
DISCUSSION:
W, (001) wafers used in a dislocation density study. Electropolish surfaces with solution shown. Develop etch pits in a solution of $xCuSO_4$:xNH_3 on freshly polished surfaces.
REF: Hull, D et al — *Phil Mag*, 12,1021(1965)
W-0024: Bewkitt, K M et al — *Phil Mag*, 12,841(1965)
W, (001) wafers structure study under a field ion microscope (FIM). Specimens were electropolished and thinned; concentration not shown.
W-0027: Ryan, H F & Suiter, J — *Acta Metall*, 14,847(1966)
W, single crystal specimens. Electropolish in sodium hydroxide prior to dislocation etching to observed grain boundaries.
W-0028a: Shukovsky, H B et al — *Acta Metall*, 14,821(1966)
W, single crystals. A study of low temperature resistivity of lattice defects in deformed specimens. A 2% NaOH solution was used at 6 V; 5% solution at 8 V to electropolish before dislocation etching.

W-0017b
ETCH NAME: TIME:
TYPE: Acid, preferential TEMP:
COMPOSITION:
x .... NH_3
x .... $CuSO_4$
DISCUSSION:
W, (001) wafers used in a dislocation study. After electropolishing, develop dislocations with the etch shown using freshly polished surfaces.
REF: Ibid.

W-0018a
ETCH NAME: Potassium ferricyanide TIME:
TYPE: Acid, polish TEMP:
COMPOSITION:
x .... $K_3Fe(CN)_6$
x .... H_2O

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DISCUSSION:

W specimens used for electroplating. Immerse specimens in solution to polish. (*Note:* A major article on plating transition metals; gives both etch solutions and plating solutions.)

REF: Saubestre, E B — *J Electrochem Soc,* 106,305(1959)

W-0019: Keilholtz, G W & Bergin, M J — U.S. Patent 2,566,615(1951)

Solution used by Saubestre (W-0018) referenced to this patent.

W-0018b

ETCH NAME: TIME:

TYPE: Acid, polish TEMP:

COMPOSITION:

x .... H_3PO_4

x .... $HClO_4$

DISCUSSION:

W specimens used for electroplating. Immerse specimens in solution shown for polishing prior to plating.

REF: Ibid.

W-0020: Schoeler, W R & Powell, A R — *Analysis of Minerals and Ores of the Rare Earth Elements,* Chas. Griffin & Co., London, 1940, 207

This reference given for solution shown above: W-0018b.

W-0018c

ETCH NAME: TIME:

TYPE: Acid, polish TEMP:

COMPOSITION:

x .... H_2O_2

x .... HOOCCOOH (oxalic acid)

DISCUSSION:

W specimens used for electroplating. See W-0018a for further discussion. Use solution by immersion.

REF: Ibid.

W-0021: Gusev, S & Ulkumov, T — *Zh Anal Khim,* 3,373(1948)

Reference for etch in W-0018c.

W-0018d

ETCH NAME: TIME:

TYPE: Acid, polish TEMP:

COMPOSITION:

x .... HF

x .... HNO_3

DISCUSSION:

W, specimens used for electroplating. Use solution by immersion. See W-0018a for further discussion.

REF: Ibid.

W-0022: Ribbons, D — *Metallurgica,* 55,257(1957)

Reference for solution used above in W-0018d.

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W-0018e
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
x .... $K_3Fe(CN)_6$
x .... H_2O
TIME:
TEMP:
DISCUSSION:
W, specimens used for electroplating. Immerse specimens in solution. See W-0018a for further discussion.
REF: Ibid.
W-0023: Faust, C L & Beach, J C — *Plating*, 43,1134(1950)
Reference for solution shown above in W-0018e.

W-0025
ETCH NAME:
TYPE: Acid, thinning
COMPOSITION:
20 .... HF
20 .... HNO_3
15 .... CH_3COOH (HAc)
TIME:
TEMP:
DISCUSSION:
W, (001) wafers and other orientations. Wafers used in a neutron damage study. Solution used to etch thin specimens for microscope study after irradiation.
REF: Lacefield, K et al — *Phil Mag*, 13,1079(1966)

W-0026
ETCH NAME: Argon
TYPE: Ionized gas, entrapment
COMPOSITION:
x .... Ar^+ ion
TIME:
TEMP:
GAS FLOW:
PRESSURE:
POWER:
DISCUSSION:
W, thin films deposited by sputtering in an argon atmosphere. Argon and other gases can be trapped in the film during growth as an ionic species. The films were used in a study of low energy inert gas ion sputtering of metals.
REF: Sinha, M K — *J Appl Phys*, 39,2150(1968)

W-0031
ETCH NAME: Oxygen
TYPE: Gas, forming
COMPOSITION:
x .... O_2
+ .... heat
TIME:
TEMP:
DISCUSSION:
W, specimens as wire. Wires were heated in oxygen to form points.
REF: Muller, E W — *Z Phys*, 108,668(1938)
W-0036b: Ibid.
W single crystal wafers of various orientations used in a study of etched surface properties. Specimens oxidized at 2600 K in O_2 at 5×10^{-5} Torr.

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W-0034a
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
1 .... HF
1 .... HNO_3
DISCUSSION:
W, thin film evaporation in vacuum systems. Solution used to clean vacuum bell jars and internal fixtures. Solution can be used on stainless steel but can damage copper, glass, and ceramic.
REF: Nichols, D R — *Solid State Technol,* December 1979

W-0035
ETCH NAME: TIME: 1 min
TYPE: Acid, step-etch TEMP: RT
COMPOSITION:
5 g .... $K_3Fe(CN)_6$
100 ml .... H_2O
DISCUSSION:
W, thin films deposited by LPCVD on poly-Si epitaxy grown on SiO_2 coated silicon, (100) p-type wafers. Solution used to step-etch tungsten for stylus depth measurements.
REF: Tsao, K Y & Busta, H H — *J Electrochem Soc,* 131,2702(1984)

W-0034b
ETCH NAME: TIME:
TYPE: Acid, removal TEMP: RT
COMPOSITION:
1 .... NH_4OH
2 .... H_2O_2
DISCUSSION:
W, thin films. Solution is recommended for cleaning vacuum systems for the removal of titanium and tungsten from stainless steel, glass, copper and ceramics. It can also be used for etching tungsten and ti-tungsten thin films.
REF: Nichols, D R — *Solid State Technol,* December 1979

W-0036a
ETCH NAME: TIME: 2 min
TYPE: Acid, removal TEMP: RT
COMPOSITION:
1 .... 30% HF
1 .... 70% HNO_3
DISCUSSION:
W single crystal wafers of various orientations used in a study of etched surface properties.
REF: Hughes, F L et al — *Phys Rev,* 113,1023(1959)

PHYSICAL PROPERTIES OF TUNGSTEN CARBIDE, WC

Classification	Carbide
Atomic numbers	74 & 6
Atomic weight	196

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Melting point (°C)	2780 (2777)
Boiling point (°C)	6000
Application limits (°C)	1500
Density (g/cm³)	14.3 (15.7)
Specific heat (mean)(J/kg °C) 25—1000°C	300
Linear coefficient of thermal expansion ($\times 10^{-6}$ cm/cm/°C) 25—800°C	6.3
Thermal conductivity (W/m °C) 1100°C)	43.3
Electrical resistivity (ohm cm) 1000°C	15
Hardness (Mohs — scratch)	9+
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Grey
Cleavage (cubic — poor)	(001)

TUNGSTEN CARBIDE, WC

General: Does not occur as a natural compound. Tungsten occurs mainly as an oxide, and there is one sulfide as minerals. Sheelite, $CaWO_4$, and wolframite, $(Fe,Mn)WO_4$ are the chief ore minerals. The carbide is artificially produced as one of the high temperature ceramics with uses similar to those of silicon carbide, though not as widely used. Several carbides have been used as surface coatings on metal cutting tools to improve wear resistance and tool working life.

Technical Application: There has been no major use in Solid State processing, to date, though the material has been studied as a single crystal. It has been deposited as a columnar thin film crystalline structure. There are possible applications as a dielectric type surface coating on electronic devices, and as a passivation material.

Etching: Acids and alkalies for cleaning. Heat for structure.

WC-0001
ETCH NAME: Murakami's reagent — TIME: 5 min
TYPE: Acid, cleaning — TEMP:
COMPOSITION: — ASTM #98

10 g .... $K_3Fe(CN)_6$
10 g .... KOH (NaOH)
100 ml .... H_2O

DISCUSSION:

WC specimens. Used as a cleaning etch for tungsten carbide surfaces. Can also be used on titanium carbide, TiC and tantalum carbide, TaC. See FE-0003 for WC deposition on steel.

REF: Pamphlet: ASTM E407-70 (1947) #98

WC-0002
ETCH NAME: Heat — TIME:
TYPE: Thermal, preferential — TEMP:
COMPOSITION:

x .... heat

DISCUSSION:

WC, single crystal specimens. Specimens were polished with diamond paste and subjected to thermal annealing to observe structure and slip.

REF: Takahashi, T & Freise, E J — *Phil Mag*, 12,12(1965)

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WC-0003
ETCH NAME: TIME:
TYPE: TEMP:
COMPOSITION:
DISCUSSION:

WC, thin films RF magnetron sputter deposited on SST substrates in acetylene gas with a tungsten target. The steel substrates were (1) polished with diamond-grit in oil paste; (2) cleaned in dilute HNO_3; (3) alcohol ultrasonic rinsed and then Ar^+ ion sputtered in vacuum for 10 min before WC growth. WC growth was columnar.
REF: Srivastava, P K et al — *J Vac Sci Technol,* A(2),1261(1984)

PHYSICAL PROPERTIES OF TUNGSTEN SELENIDE, WSe_2

Classification	Selenide
Atomic numbers	74 & 34
Atomic weight	341.84
Melting point (°C)	1500 est.
Boiling point (°C)	
Density (g/cm³)	12 est.
Hardness (Mohs — scratch)	5—6
Crystal structure (hexagonal — normal)	$(10\bar{1}0)$ prism
Color (solid)	Grey
Cleavage (basal)	(0001)

TUNGSTEN SELENIDE, WSe_2

General: Does not occur in nature as a compound, although there are other metallic selenide minerals. There has been no use of the selenide in industry at the present time.

Technical Application: There is no major use of the compound in Solid State processing, though it has been grown as a single crystal in general material studies with possible applications as a photo-anode.

Etching: Mixed acids HF:HNO_3, and halogens.

TUNGSTEN SELENIDE ETCHANTS

WSE-0001a
ETCH NAME: Carbon tetrachloride TIME:
TYPE: Solvent, cleaning TEMP:
COMPOSITION:
x .... CCl_4
DISCUSSION:

WSe_2, single crystal specimens studied for use as photo-anodes. After polishing in a BRM solution, carbon tetrachloride was used to wash remove residual bromine from surfaces. (*Note:* BRM solutions are x% Br_2:MeOH.)
REF: Baglio, J A et al — *J Electrochem Soc,* 129,1461(1982)

WSE-0001b
ETCH NAME: BRM TIME:
TYPE: Halogen, polish TEMP:
COMPOSITION:
x .... x% Br_2
x .... MeOH

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DISCUSSION:

WSe_2, single crystal specimens used in a study for photo-anodes. After etch wash cleaning (WSE-0001a), polish and thin single crystals in a BRM solution. Thin specimens can be peeled from the bulk material. Also evaluated: MoS_2, WS_2 and $MoSe_2$.
REF: Ibid.

PHYSICAL PROPERTIES OF TUNGSTEN SULFIDE, WS_2

Classification	Sulfide
Atomic numbers	74 & 16
Atomic weight	248.04
Melting point (°C)	1000 est.
Boiling point (°C)	
Density (g/cm³)	7.5 (7.4)
Hardness (Mohs — scratch)	2.5
Crystal structure (orthorhombic — normal)	(100) pinacoid
Color (solid)	Lead-grey
Cleavage (basal)	(001)

TUNGSTEN SULFIDE, WS_2

General: Occurs as the natural mineral tungsenite, WS_2, associated with other tungsten ores as an earthy deposit, has not been observed as a natural single crystal, but as a colloidal structure. Not used in industry as a natural mineral, but tungsten metal has been processed through the sulfurization process to introduce sulfur into the metal.

Technical Application: Tungsten sulfide has been grown as a single crystal in Solid State development with applications as a photo-anode, and is under evaluation with other sulfides and selenides.

Etching: Mixed acids HF:HNO_3; halogens.

TUNGSTEN SULFIDE ETCHANTS

WS-0001
ETCH NAME: BRM
TYPE: Halogen, polish
COMPOSITION:
x .... x% Br_2
x .... MeOH
TIME:
TEMP: RT

DISCUSSION:

WS_2, single crystal specimens used in a study for photo-anodes. After etch polishing in solution shown, wash in CCl_4 to remove residual bromine. Thin single crystal wafer units can be peeled from the bulk ingot. Other materials evaluated were WSe_2, MoS, and $MoSe_2$.
REF: Raglio, J A et al — *J Electrochem Soc*, 129,1461(1982)

TUNGSTEN:RHENIUM ETCHANTS

WRH-0001
ETCH NAME: Sodium hydroxide
TYPE: Electrolytic, polish
COMPOSITION:
x .... 0.75% NaOH
TIME:
TEMP:
ANODE:
CATHODE:
POWER:

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DISCUSSION:

WRh(2%) and (6%), (100) wafers cut from single crystal ingots. Solution used to electropolish specimens in the study of surface work function.

REF: Abey, A E — *J Appl Phys*, 39,120(1968)

PHYSICAL PROPERTIES OF TUNGSTEN TRIOXIDE, WO_3

Classification	Oxide
Atomic numbers	74 & 8
Atomic weight	231.92
Melting point (°C)	1473
Boiling point (°C)	
Density (g/cm^3)	7.16
Refractive index (n =)	2.00—2.26
Hardness (Mohs — scratch)	6—7
Crystal structure (orthorhombic — normal)	(100) pinacoid
Color (solid)	Bright yellow
Cleavage (basal — perfect)	(001)

TUNGSTEN TRIOXIDE, WO_3

General: Occurs in nature as the mineral tungstite, WO_3 or as the hydrated colloid mineral keymacite, $WO_3.H_2O$. In either case, occurrence is rare and usually associated with wolframite, $(Fe,Mn)WO_4$, as a surface coating, occasionally as an earthy mass. Due to its brilliant yellow color it is called tungstic ocher, occurring naturally as a fine, yellow powder.

Not used industrially as natural occurring compound due to scarcity, but as an artificial compound has been used as a coloring pigment in glass, as a surface glaze, or in coloring ceramics. When a light bulb filament burns out, traces of either the yellow trioxide or brown dioxide are often seen.

Technical Application: Not used in Solid State device fabrication at present, although it may have application as a thin film oxide coating similar to that of other metal oxides, such as silicon dioxide, SiO_2 and aluminum oxide, Al_2O_3. It has been observed as a by-product reaction in the sputter deposition of Ti-tungsten, TiW(10%Ti) on glass and quartz substrates as a yellow/orange gel remaining after Ti-tungsten removal.

Tungsten oxides, WO_x have been deposited as thin films on both tin oxide, SnO_2 and indium tin oxide, $InSnO_2$ (ITO). On glass, WO_3 has been converted to single crystal by annealing.

Etching: Soluble in HF and hot alkalies.

Note: Tungsten dioxide, WO_2, is an artificial compound with isometric-normal structure as a cube, (100).

TUNGSTEN OXIDE ETCHANTS

WO-0001a

ETCH NAME: Potassium hydroxide — TIME:

TYPE: Alkali, removal — TEMP: 60°C

COMPOSITION:

x .... 20—30% KOH (NaOH)

DISCUSSION:

WO_2. This oxide is cubic in structure and insoluble in acids. Color: brown. In removing sputtered thin films of TiW(10%Ti) from alumina and quartz substrates a grey/brownish

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film may remain — WO_x/TiO_x. Sequence etching in concentrated HF, then 30% H_2O_2 and then KOH will remove both oxides.
REF: Weast, R C et al — *Handbook of Chemistry and Physics*, 65th ed, CRC Press, Boca Raton, FL, 1984, B154
WO-0002b: Ibid.

WO_x thin films as TiW(10%Ti) sputtered on alumina and quartz substrates. See discussion above.

WO-00016
ETCH NAME: Potassium hydroxide
TYPE: Alkali, removal
COMPOSITION:
x .... 30% KOH
TIME: 2—10 min
TEMP: Hot (60°C)
DISCUSSION:

WO_3. This oxide is hexagonal system, rhombohedral division in structure. Color is yellow to yellow-orange. Thin film TiW(10% Ti) Rf sputtered on alumina and quartz substrates. TiW removed by soaking in H_2O_2 but can leave a yellow-orange gel — hydrated WO_3. Remove gel by soaking in hot KOH.
REF: Weast, R C et al — *Handbook of Chemistry and Physics*, 65th ed, CRC Press, Boca Raton, FL, 1984, B145
WO-0002c: Ibid.

WO_3 hydrated film left on alumina and quartz substrates after stripping TiW (10% Ti) RF sputtered deposits. See discussion above.

WO-0003
ETCH NAME: Sulfuric acid
TYPE: Acid, removal
COMPOSITION:
x .... 7.8 *N*—13.3 *N* H_2SO_4
TIME:
TEMP:
DISCUSSION:

WO_3 thin film deposited on 2100 Å SnO_2 on a glass substrate. Deposit is amorphous. Heat treating in argon at greater than 350°C will convert to single crystal. Use sulfuric acid to etch remove.
REF: Viennet, R & Randin, J F — *J Electrochem Soc*, 129,2349(1982)

WO-0007
ETCH NAME:
TYPE: Oxide, growth
COMPOSITION:
(1) x .... Ar
(2) xx .... Ar
x .... 0—1% O_2
TIME:
TEMP: RT to 100°C
GAS FLOW:
PRESSURE: 3×10^{-1} Torr
POWER: 100 W
RATE: 30—60 Å/min to 3000 Å
DISCUSSION:

Li_xWO_3 (blue) and Li_xWO_3 (clear) sputter deposited in gases (1) and (2), respectively, as an A or B film. During growth of B film, the A film bleaches to an A′ film. Deposited as a B/A film on an ITO coated quartz substrate, then Au evaporated with electrical contact between Au and ITO to measure device response time with an He/Ne laser. Layer B operates as an electrolyte supplying layer A with Li^+ ions for electrochromic action. Bias alters A-A′ color change from blue (+) to clear (−) . . . "bleached". Optical density vs. response time measured with a voltagram. Very high absorption of infrared light in B film, sharply

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reduced with addition of oxygen. Pressed powder target: Li_2CO_3:WO_3 used for RF sputter depositing.
REF: Kitabatake, M et al — *J Electrochem Soc*, 132,433(1985)

WO-0004
ETCH NAME:
TYPE: Electrolytic, removal
COMPOSITION:
x .... H_2SO_4

TIME:
TEMP:
ANODE:
CATHODE:
POWER: 1.5 V

DISCUSSION:
WO_x (x = 3), thin film RF magnetron sputtered, RF plated or evaporated onto ITO ($InSnO_2$). Under electrical bias with a Pt counter-electrode the WO_x turns blue-black.
REF: Yoshimuria, T — *J Appl Phys*, 57,911(1985)

WO-0001c
ETCH NAME:
TYPE:
COMPOSITION:

TIME:
TEMP:

DISCUSSION:
W_2O_5. Color is violet-black. Insoluble in acids.
REF: Weast, R C et al — *Handbook of Chemistry and Physics*, 65th ed, CRC Press, Boca Raton, FL, 1984, B145

WO-0005
ETCH NAME: Sodium hydroxide
TYPE: Alkali, removal
COMPOSITION:
x .... 0.01—0.04 *N* NaOH

TIME:
TEMP: RT
RATE: 1000°A/minute

DISCUSSION:
a-WO_3 thin films 499—8500 Å thick, evaporated from a tungsten boat on In_2O_3 coated glass substrates. Films irradiated with sodium silicate or aluminosilicate. Etch patterned with solution shown for use as a high-contrast inorganic ion resist material.
REF: Koshida, N & Tomita, O — *Jpn J Appl Phys*, 24,92(1985)

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PHYSICAL PROPERTIES OF URANIUM, U

Classification	Actinide
Atomic number	92
Atomic weight	238.03
Melting point (°C)	1132
Boiling point (°C)	3818
Density (g/cm^3)	19.04 (19.07)
Thermal conductance (cal/sec)(cm^2)(°C/cm)	0.064
Specific heat (cal/g) 25°C	0.028
Latent heat of fusion (cal/g)	19.75
Heat of fusion (k-cal/g-atom)	2.7
Heat of vaporization (k-cal/g-atom)	110
Atomic radius (W/D)	12.5
Electronegativity (Pauling's)	1.7
Covalent radius (angstroms)	1.42
Ionic radius (angstroms)	0.97 (U^{+4})
Electrical conductance (micro $ohms^{-1}$)	0.034
Magnetic susceptibility (emu/g $\times 10^{-6}$) 15°C	1.72
Thermal expansion (ppm/°C)	7.14
Half life ($\times 10^9$ years)	4.51
Gross section (barns)	7.6
Electronwork function (eV)	3.63
Hardness (Mohs — scratch)	3—4
(Brinell — kgf/mm^2)	187
Crystal structure (orthorhombic — normal)	(100) a-pinacoid
Color (solid)	Grey-greenish
Cleavage (basal)	(001)

URANIUM, U

General: Does not occur as a native metal. There are over 50 uranium-containing minerals as oxides, vanadinates, or phosphates, etc. The two primary ores are carnotite (brilliant yellow and orange color) and uraninite (pitchblende, and coal-black color), often intimately mixed together. All uranium minerals are radioactive with a slow disintegration series ending in heavy lead, Pb. The discovery of radium in pitchblende (it is one million times more active than uranium) by the Curies' — pure radium by Madame Curie in 1910 — established the field of radioactivity.

Though most people think of uranium as the atomic bomb, it has far more important scientific applications. The ratio of helium gas in uranium ore, which shows a constant rate of decay — 1 cc in 16,000,000 years — is used for radioactive dating as the radium clock). It indicates that the earth is at least 1.5 billion years old. Radium is luminescent, subject to light and decay, it glows in the dark, and was used on clock and watch faces for many years before the true danger of radioactivity was established.

Industrially, today the most important application of uranium and its associated decay series is for nuclear energy power production, and there are power plants in every industrial nation throughout the world. In medicine, radiation therapy is a primary tool in the cure of cancer and, in the food industry, it is under development for food preservation. Uranium is used as a glass pigment for its luminosity and yellow color.

Technical Application: The particle emissions — alpha, beta, and gamma rays from radioactive materials — are of importance in scientific study. Semiconductor alpha radiation-counter devices have been tested using alpha particles with subsequent device use in space probes to evaluate the radiation level in the Van Allen belt. Lead pellets are alpha radiation

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activated with cobalt-60 for such testing purposes, and polonium metal strips are used in static eliminators: a camel-hair brush with a Po strip or a unit blowing cold/hot air across the strip with an operational half-life of approximately 1 year. Such air eliminates the build-up of a static charge on high frequency microwave devices, such as GaAs field effect transistors (FETs). Static elimination systems are built into air ducts or within Clean Rooms to prevent dust accumulation, as dust can develop a static charge which is device destructive. Note that on a very dry day, when humidity is around 10% RH, just walking past a static-sensitive device can be enough to short-out and destroy such a device if not properly grounded.

Uranium pressed pellets have been the subject of much study, and it has been grown as a single crystal for morphological data.

Etching: Soluble in single acids and mixed acids.

U-0001a
ETCH NAME: Nitric acid
TYPE: Acid, cleaning
TIME:
TEMP:
COMPOSITION:
x .... HNO_3
DISCUSSION:
U specimens. The following sequence was used to clean uranium surfaces: (1) dip in HNO_3, (2) rinse in DI H_2O, and (3) rinse in acetone.
REF: Robb, W L — *J Electrochem Soc*, 108,126(1959)

U-0002a
ETCH NAME:
TYPE: Acid, polish
TIME: 5 min
TEMP: RT
COMPOSITION:
x .... HNO_3
x .... CrO_3
x .... H_2O
x ... Al_2O_3, abrasive
DISCUSSION:
U specimens. Used as a lapping/polishing acid slurry to etch and polish uranium material.
REF: Cochran, F L & Wallace, W P — *Research & Development*, October 1966, 126

U-0002b
ETCH NAME:
TYPE: Acid, polish
TIME: Minutes
TEMP: RT
COMPOSITION:
x .... HF
x .... HNO_3
x .... H_2O
DISCUSSION:
U specimens. Apply as drops on the surface at a rate of approximately 15 drops/min to polish.
REF: Ibid.

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U-0002c
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
1 .... H_2SO_4
DISCUSSION:
U specimens. Used as an electropolish etch.
REF: Ibid.

TIME:
TEMP:
ANODE: U
CATHODE:
POWER: 500 mA/cm²

U-0002d
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
130 ml .... H_2SO_4
50 ml .... H_2O_2
60 ml .... H_2O
2 mg .... Na_2SiF_6
DISCUSSION:
U specimens. Used as a polish etch.
REF: *Ibid.*

TIME: 30 sec
TEMP: RT

U-0002e
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
300 ml .... HAc
30 ml .... H_2O
25 g .... CrO_3
DISCUSSION:
U specimens. Used as an electropolish etch.
REF: Ibid.

TIME: 1 min
TEMP: RT
ANODE: U
CATHODE:
POWER: 200 mA/cm²

U-0003a
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
133 ml .... HAc
25 g .... CrO_2
7 ml .... H_2O
DISCUSSION:
Alpha-U specimens. Solution used to electropolish specimens.
REF: Hudson, B — *Phil Mag*, 9,949(1964)

TIME:
TEMP: 10°C
ANODE: U
CATHODE: SST
POWER: 0.4 A/cm² &
35—40 V

U-0003b
ETCH NAME:
TYPE: Electrolytic, cleaning
COMPOSITION:
75 ml .... H_2SO_4
18 ml .... glycerin
7 ml .... H_2O

TIME: 20 sec
TEMP: RT
ANODE: U
CATHODE: Ni
POWER: 9.15 mA/cm² &
6—10 V

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DISCUSSION:
Alpha-U specimens. Solution used as an electrolytic cleaning system.
REF: Ibid.

U-0001b
ETCH NAME: Iodine
TYPE: Halogen, cleaning
COMPOSITION:
x .... I_2, vapor
TIME:
TEMP: Red heat
DISCUSSION:
U specimens. Dip and coat surfaces with iodine solution that will produce UI_4. In vacuum system, bring specimen up to red heat to vaporize UI_4 and clean surface. Used prior to depositing zirconium on uranium by the iodine process. (*Note:* Iodine surface treatment is used on diamonds as a surface conditioner against etching. This vapor treatment can be used on any material surface as a complexing agent . . . heat above 100°C . . . to remove ionic contamination (F^+, Cl^+, O^+, etc.) from previous etching for a micromolecular clean surface.)
REF: Ibid.

PHYSICAL PROPERTIES OF URANIUM CARBIDE, UC

Classification	Carbide
Atomic numbers	92 & 6
Atomic weight	251 (263)
Melting point (°C)	2260
Boiling point (°C)	4100
Density (g/cm^3)	11.28
Hardness (Mohs — scratch)	9+
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Grey
Cleavage (cubic)	(001)

URANIUM CARBIDE, UC

General: Does not occur as a compound in nature. All carbides are artificially grown, other than a single occurrence of silicon carbide, SiC associated with a meteor strike at Cañon Diablo, Arizona. There has been no industrial use of the carbide. Silicon carbide (carborundum), in particular, is far more useful as a major lapping and polishing abrasive.

Technical Application: No use in Solid State processing to date. The carbide has been grown as a single crystal for general morphological studies along with the nitride and sulfide. Other metal uraninides also are shown here.

Etching: Single and mixed acids.

URANIUM CARBIDE ETCHANTS

UC-0001
ETCH NAME:
TYPE: Electrolytic, jet thin
COMPOSITION:
4 .... H_3PO_4
1 .... MeOH
TIME:
TEMP:
ANODE:
CATHODE:
POWER:

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DISCUSSION:

UC, (001) wafers cleaved from single crystal ingots and used in a study of slip with observation under an electron microscope (EM). Solution used to thin UC for microscope observation. Solution also used for uranium nitride and uranium sulfide.

REF: Solo, M J & Van Der Walt, C M — *Acta Metall*, 16,501(1968)

UC-0002

ETCH NAME: Nitric acid
TYPE: Acid, removal
COMPOSITION:
x .... HNO_3

TIME:
TEMP:

DISCUSSION:

UC_2 specimens. Material is soluble in acids.

REF: Hodgman, C D — *Handbook of Chemistry and Physics*, 27th ed, Chemical Rubber Co., Cleveland, OH, 1943, 482

UC-0003

ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
x .... H_3PO_4
x .... MeOH

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

DISCUSSION:

UC single crystal wafers cleaved (100) used in a study of uranium dicarbide precipitates. Solution shown used as a wet chemical etch (WCE) to reduce size of initial specimens. Solution as an electrolytic jet polish with added MeOH to reduce viscosity: 200 VDC and 150—200 mA/cm^2 used for general removal; 85 V then to 45 V for x-min to initial polish with final etching done with 1-sec on-off switching. Remove from etch bath under MeOH spray, rotating nozzle to prevent pitting. Store under MeOH. No surface alteration for about 1 h. Electron microscope used for study.

REF: Eyre, B L & Sole, M J — *Phil Mag*, 9,545(1964)

URANIUM CESIUM ETCHANTS

UCE-0001

ETCH NAME: Kalling's etch
TYPE: Acid, polish
COMPOSITION:
100 ml .... HCl
5 g .... $CuCl_3$
100 ml .... EOH

TIME: 10—60 sec
TEMP: RT

DISCUSSION:

UCe_2 arc melted in forming and used in a study of metallic compounds. Solution used as a general removal and polishing etchant. Other uranium compounds studied were UPt_3, UNi_2, and UCo_2. See Cerium Platinide: CEPT-0001 for other compounds studied.

REF: Slepowronsky, M et al — *J Cryst Growth*, 65,293(1983)

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URANIUM COBALT ETCHANTS

UCO-0001
ETCH NAME: Kalling's etch
TYPE: Acid, polish
COMPOSITION:
100 ml .... HCl
5 g .... $CuCl_3$
100 ml .... EOH
TIME: 10—60 sec
TEMP: RT
DISCUSSION:
UCo_2 arc melted in forming and used in a study of metallic compounds. Solution used as a general removal and polishing etchant. Other uranium compounds studied were UCe_2, UNi_2, and UPt_3. See Cesium Platinide: CEPT-0001 for other compounds studied.
REF: Slepowronsky, M et al — *J Cryst Growth*, 65,293(1983)

URANIUM NICKELIDE ETCHANTS

UNI-0001
ETCH NAME: Kalling's etch
TYPE: Acid, polish
COMPOSITION:
100 ml .... HCl
5 g .... $CuCl_3$
100 ml .... EOH
TIME: 10—60 sec
TEMP: RT
DISCUSSION:
UNi_2 arc melted in fabrication and used in a study of metallic compounds. Solution used as a general removal and polishing etchant. Other uranium compounds studied were UCe_2, UCo_2, and UPt_3. See Cesium Platinide: CEPT-0001 for other compounds studied.
REF: Slepowronsky, M et al — *J Cryst Growth*, 65,293(1983)

PHYSICAL PROPERTIES OF URANIUM DIOXIDE, UO_2

Classification	Metal oxide
Atomic numbers	92 & 8
Atomic weight	271
Melting point (°C)	2176
Boiling point (°C)	
Density (g/cm^3)	10.9
Hardness (Mohs — scratch)	6—7
Crystal structure (orthorhombic — normal)	(100) prism
Color (solid)	Brown/black
Cleavage (basal — distinct)	(001)

URANIUM DIOXIDE, UO_2

General: Occurs in nature as a hydrate oxide as the minerals ianthinite, $UO_2.7H_2O$, which is orthorhombic, violet black color to yellow at edges, and becquerelite, $UO_3.2H_2O$, also orthorhombic, but brownish-yellow in color. Both have distinctive (001) cleavage. There is little use in industry other than in processing uranium ores, and as a fluorescent yellow coloring agent in glass.

Technical Application: There is no use in Solid State device processing, although the

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dehydrated oxide has been grown as a single crystal for general structural and dislocation study.

Etching: HNO_3, H_2SO_4. Electrolytic H_3PO_4.

URANIUM DIOXIDE ETCHANTS

UO-0001
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
67 ml .... H_2SO_4
310 ml .... H_3PO_4
120 ml .... H_2O
78 g .... CrO_3
TIME:
TEMP:
ANODE: UO_2
CATHODE:
POWER:
DISCUSSION:
UO_2 specimens. Solution used to electropolish and to thin specimens.
REF: Whapham, A D & Wheldon, B E — *Phil Mag*, 12,1179(1965)

UO-0002a
ETCH NAME: Nitric acid
TYPE: Acid, removal
COMPOSITION:
x .... HNO_3
TIME:
TEMP:
DISCUSSION:
UO_2 specimens. Shown as an etch for this material, and can be used diluted with water.
REF: Hodgman, C D — *Handbook of Chemistry and Physics*, 27th ed, Chemical Rubber Co., Cleveland, OH, 1943, 482

UO-0002b
ETCH NAME: Sulfuric acid
TYPE: Acid, removal
COMPOSITION:
x .... H_2SO_4 conc.
TIME:
TEMP:
DISCUSSION:
UO_2 specimens. Shown as an etch for this material.
REF: Ibid.

PHYSICAL PROPERTIES OF URANIUM NITRIDE, U_3N_4

Classification	Nitride
Atomic numbers	92 & 7
Atomic weight	775
Melting point (°C)	>800
Boiling point (°C)	
Density (g/cm^3)	10.09
Hardness (Mohs — scratch)	6—7
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Brown/black
Cleavage (cubic)	(001)

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URANIUM NITRIDE, U_3N_4

General: Does not occur as a native metallic compound, although there are many other metal nitrates and nitrites. There is no use of the nitride in industry at the present time.

Technical Application: There is no use for the nitride in Solid State material processing, to date, although it has been grown as a single crystal for general study as a compound.

Etching: HNO_3. Electrolytic H_3PO_4.

URANIUM NITRIDE ETCHANTS

UN-0001
ETCH NAME:
TYPE: Electrolytic, jet thin
COMPOSITION:
4 .... H_3PO_4
1 .... MeOH
TIME:
TEMP:
ANODE:
CATHODE:
POWER:
DISCUSSION:
UN, (001) wafers cleaved from single crystal ingots used in a study of oxidation and deformation. Solution used to jet thin specimens for electron microscope (EM) study.
REF: Solo, M J & Van Der Walt, C M — *Acta Metall*, 16,501(1968)

UN-0002
ETCH NAME: Nitric acid
TYPE: Acid, removal
COMPOSITION:
x .... HNO_3
TIME:
TEMP:
DISCUSSION:
U_3N_4 specimens. Material is soluble in nitric acid.
REF: Hodgman, C D — *Handbook of Chemistry and Physics*, 27th ed, Chemical Rubber Co., Cleveland, OH, 1943, 482

URANIUM PLATINIDE ETCHANTS

UPT-0001
ETCH NAME: Kalling's etch
TYPE: Acid, polish
COMPOSITION:
100 ml .... HCl
5 g .... $CuCl_3$
100 ml .... EOH
TIME: 10—60 sec
TEMP: RT
DISCUSSION:
UPt_3 arc melted in fabrication and used in a study of metallic compounds. Solution used as a general removal and polishing etchant. Other uranium compounds studied were UCe_2, UCo_2, and UNi_2. See Cesium Platinide: CFPT-0001 for other compounds studied.
REF: Slepowronsky, M et al — *J Cryst Growth*, 65,293(1983)

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PHYSICAL PROPERTIES OF URANIUM DISULFIDE, US_2

Classification	Sulfide
Atomic numbers	92 & 16
Atomic weight	302.19
Melting point (°C)	1100
Boiling point (°C)	
Density (g/cm^3)	
Hardness (Mohs — scratch)	3—4
Crystal structure (tetragonal — normal)	(100) prism
Color (solid)	Grey/black
Cleavage (basal)	(001)

URANIUM SULFIDE, US

General: Does not occur in nature as a sulfide, although there are two sulfate minerals, johannite and zippeite, both as hydrates. The sulfide has had no use in industry, although uranium can be sulfurized, much like nitriding.

Technical Application: There has been no use in Solid State processing, although the sulfide has been grown as a single crystal for general study.

Etching: HCl, HNO_3. Electrolytic H_3PO_4.

URANIUM SULFIDE ETCHANTS

US-0001
ETCH NAME:
TYPE: Electrolytic, jet thin
COMPOSITION:
4 .... H_3PO_4
1 .... MeOH
TIME:
TEMP:
ANODE:
CATHODE:
POWER:
DISCUSSION:

US, (001) wafers cleaved from single crystal ingot and used in a study of slip with an electron microscope (EM). Compressed air was used for the acid jet action to thin specimens. Also used on uranium nitride and uranium carbide.

REF: Soto, M J & Van Der Walt, C M — *Acta Metall,* 16,501(1968)

US-0002
ETCH NAME: Hydrochloric acid
TYPE: Acid, removal
COMPOSITION:
x .... HCl, conc.
TIME:
TEMP:
DISCUSSION:

US_2 specimens. Solution shown as a general etch for this material.

REF: Hodgman, C D — *Handbook of Chemistry and Physics,* 27th ed, Chemical Rubber Co., Cleveland, OH, 1943, 482

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URANIUM TITANIUM ETCHANTS

UTI-0001
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
60 ml .... $HClO_4$
350 ml .... butyl cellusolve
350 ml .... MeOH

TIME: 10 sec
TEMP: 8°C
ANODE:
CATHODE:
POWER: 50 V

DISCUSSION:
UTi, single crystal specimens used in a diffusion study. Solution shown used as a general removal and polish etch.
REF: Adda, Y & Philibert, J — *Acta Metall,* 8,700(1960) (in French)

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PHYSICAL PROPERTIES OF VANADIUM, V

Classification	Transition metal
Atomic number	23
Atomic weight	50.942
Melting point (°C)	1750 (1900)
Boiling point (°C)	3000
Density (g/cm^3)	6.11
Thermal conductance (cal/sec)(cm^2)(°C/cm)100°C	0.074
Specific heat (cal/g) 20°C	0.120
Heat of fusion (k-cal/g-atom)	4.2
Latent heat of fusion (cal/g)	82.5
Heat of vaporization (k-cal/g-atom)	106
Atomic volume (W/D)	8.35
1st ionization energy (K-cal/g-mole)	156
1st ionization potential (eV)	6.74
Electronegativity (Pauling's)	1.6
Covalent radius (angstroms)	1.22
Ionic radius (angstroms)	0.59 (V^{+5})
Coefficient of linear thermal expansion ($\times 10^{-6}$ cm/cm/°C) 25°C	9.7
Electrical resistivity (micro-ohms cm)	24.8
Modulus of elasticity (psi $\times 10^6$)	18—19
Cross section (barns)	4.9
Magnetic susceptibility (ergs $\times 10^{-6}$)	1.4
Poisson ratio	0.36
Shear modulus (psi $\times 10^6$)	6.73
Refractive index (n =)	3.03
Hardness (Mohs — scratch)	4
Crystal structure (isometric — normal)	(100) cube, bcc
Color (solid)	Silver white
Cleavage (cubic — indistinct)	(001)

VANADIUM, V

General: Does not occur as a native element, but is found as a minor constituent in several minerals with the most important ore being vanadinite, $(PbCl)Pb_4(VO_4)_3$. The mineral patronite is a complex vanadium sulfate containing large quantities of vanadium and is one of the few normally amorphous minerals. As pure vanadium it is a high temperature, infusible metal.

As an alloy in steels it greatly increases toughness, elasticity, and tensile strength and as an oxide coloring agent — blue, yellow, and red, depending upon the oxide — is widely used in the glass and ceramic industries. The oxide also is used in the preparation of sulfuric acid.

Technical Application: Not presently used in Solid State device fabrication although as a silicide it is under evaluation as a buffer layer in heterostructure device like other metal silicides.

It has been studied in both crystalline and single crystal form. Also as a carbide, VC and boride, VB and as other metallic compounds.

Etching: HNO_3, H_2SO_4, HF, and aqua regia but insoluble in HCl and alkalies.

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VANADIUM ETCHANTS

V-0001
ETCH NAME: TIME:
TYPE: Acid, removal/polish TEMP:
COMPOSITION:
(1) 1 .... HNO_3 (2) 1 .... HF
1 .... H_2O 1 .... HNO_3
DISCUSSION:
V specimens. Both solutions are general removal and polishing etches.
REF:

V-0002a
ETCH NAME: Aqua regia TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
3 .... HCl
1 .... HNO_3
DISCUSSION:
V specimens. Shown as a general etch for vanadium.
REF: Hodgman, C D — *Handbook of Chemistry and Physics*, 27th ed, Chemical Rubber Co, Cleveland, OH, 1943, 484

V-0002b
ETCH NAME: Sulfuric acid TIME:
TYPE: Acid, remove TEMP:
COMPOSITION:
x .... H_2SO_4
DISCUSSION:
V, specimens. Shown as a general etch for vanadium.
REF: Ibid.

V-0002c
ETCH NAME: Hydrofluoric acid TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... HF
DISCUSSION:
V, specimens. Shown as a general etch for vanadium.
REF: Ibid.

V-0003
ETCH NAME: Nitric acid TIME:
TYPE: Acid, sizing TEMP: RT
COMPOSITION:
x .... HNO_3
DISCUSSION:
V, poly-wire used in a study of the effects of pressure on the mobility of interstitial oxygen and nitrogen in vanadium. The wires were reduced in size from 0.020 to 0.015 diameter by etching in nitric acid.
REF: Tichelaar, G W — *Phys Rev*, 121,748(1961)

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V-0004
ETCH NAME:
TYPE: Electrolytic, polish/thin
COMPOSITION:
1 .... H_2SO_4
6 .... MeOH
TIME:
TEMP:
ANODE:
CATHODE:
POWER: 10 V
DISCUSSION:
V specimens. Specimens neutron irradiation and annealed at 1300—1400°C. Solution used to etch polish and thin specimens for microscope study.
REF: Rau, R C & Ladd, R L — *J Appl Phys*, 40,2899(1969)

V-0005
ETCH NAME: Heat
TYPE: Thermal, forming
COMPOSITION:
x .... heat
TIME:
TEMP:
DISCUSSION:
V, single crystal and polycrystalline spheres formed by splattering using an arc melter in an He atmosphere. Other materials studied were Be, Fe, Zr, NiCu, and NiAl. Sphere sizes ranged from 0.1 to 1.0 mm diameter and 5 to 50% were single crystal.
REF: Ray, A E & Smith, J F — *Acta Metall*, 11,310(1958)

V-0006
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
x .... Na_2CO_3
x .... $NaHCO_3$
TIME:
TEMP:
ANODE: V
CATHODE:
POWER:
DISCUSSION:
V specimens. Used solution as an anodic polish etch for this material.
REF: Brown, C M — U.S. Patent 2,803,596 (1957)

V-0007
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
1 .... HF
10 .... HNO_3
TIME:
TEMP:
DISCUSSION:
V, thin films evaporated on silicon substrates. Solution used to remove vanadium from silicon surfaces. A study of vanadium growth on silicon.
REF: Miller, K J et al — *J Electrochem Soc*, 113,902(1966)

VANADIUM ALUMINUM ETCHANTS

VAL-0001
ETCH NAME: Nitric acid
TYPE: Acid, removal/clean
COMPOSITION:
x .... HNO_3, conc.
TIME:
TEMP:
DISCUSSION:
VAl_3, deposited on silicon, (001) substrates in an evaluation of vanadium and aluminum

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reactions. Authors say that if there is more than 15% O_2 in the vanadium an Al_2O_3 (?) layer forms between the V/Al interface as a blocking layer. Vanadium was evaporated on various aluminum layer combinations as thin films: Al, Al/Cr; Al/Ti, and Al/TiN.
REF: Finstad, T G et al — *Thin Solid Films*, 114,271(1985)

VANADIUM BROMIDE ETCHANTS

VBR-0001
ETCH NAME: Ethyl alcohol
TYPE: Alcohol, removal/polish
COMPOSITION:
x .... EOH (MeOH)
TIME:
TEMP:
DISCUSSION:
VBr_2, single crystals. Solution shown used as a general cleaning, removal and polishing etchant on the material. Specimens used in a general properties study, and particularly for antiferromagnetic characteristics.
REF: Ailshi, M et al — *J Phys Soc Jpn*, 4,1214(1984)

PHYSICAL PROPERTIES OF VANADIUM CARBIDE, VC

Classification	Carbide
Atomic numbers	23 & 6
Atomic weight	62.96
Melting point (°C)	2810
Boiling point (°C)	3900
Density (g/cm^3)	5.4
Lattice constant (angstroms)	4.16
Hardness (Mohs — scratch)	8—9
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Grey-white
Cleavage (cubic — perfect)	(001)

VANADIUM CARBIDE, VC

General: Does not occur as a native carbide in nature. Industrially, it is a high temperature ceramic material, but has had little use other than for study of characteristics.

Technical Application: It has been grown as single crystal ingots, and as both single crystal and polycrystalline filaments for general study of the material.

Etching: HNO_3. Electrolytic mixed acids.

VANADIUM CARBIDE ETCHANTS

VC-0001
ETCH NAME:
TYPE: Electrolytic, preferential
COMPOSITION:
1 .... HF
1 .... HNO_3
10 .... H_2O
TIME: 20—40 sec
TEMP: RT
ANODE:
CATHODE: VC
POWER: 2—3 V
DISCUSSION:
VC, (100) single crystal ingots of various compositions. Wafers cleaved from ingots.

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Solution used in a study of structure and dislocations. Large, flat-bottomed pits appear first, and are considered to be associated with carbon impurity. Small pits that then appear are dislocations.
REF: Hou, Y — *J Cryst Growth,* 68,733(1984)

VC-0002a
ETCH NAME: Nitric acid
TYPE: Acid, removal
TIME:
TEMP:
COMPOSITION:
x .... HNO_3
DISCUSSION:
VC, (100) single crystal and polycrystalline specimens. Shown as a general etch for this material.
REF: Hodgman, C D — *Handbook of Chemistry and Physics,* 27th ed, Chemical Rubber Co, Cleveland, OH, 1943, 484

VC-0002b
ETCH NAME: Potassium nitrate
TYPE: Molten flux, preferential
TIME:
TEMP: >400°C
COMPOSITION
x .... KNO_3
DISCUSSION:
VC specimens. Shown as an etch for this material.
REF: Ibid.

VANADIUM GALLIUM ETCHANTS

VGA-0001
ETCH NAME: Nitric acid, dilute
TYPE: Acid, removal/preferential
TIME:
TEMP:
COMPOSITION:
x .... HNO_3
x .... H_2O
DISCUSSION:
VGa, single crystal filaments grown in a copper flux system. Solution used to etch clean filaments in a morphological study.
REF:

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PHYSICAL PROPERTIES OF WATER, H_2O

Classification	Solvent
Atomic numbers	1 & 8
Atomic weight	18.02
Melting point (°C)	0
Boiling point (°C)	100
Density (g/cm^3) 4°C (liquid)	1.00
(g/cm^3) 0°C (solid)	0.9168
Heat of vaporization (cal)	540
Heat of fusion — ice (cal)	76
Hardness (Mohs — scratch — ice)	1—1.5
Crystal structure (hexagonal, normal — ice) alpha	$(10\bar{1}0)$ platelets
(isometric — normal — ice) beta	(100)
Color (solid)	Bluish
(liquid)	Colorless
Cleavage (basal/cubic — solid)	(0001/001)

WATER, H_2O

General: There are two primary forms of water in nature: (1) salt water and (2) fresh water, but there are over 100 varieties depending upon mineral content, source location, treatment, and so forth. Fresh water is also classified by soft or hard, again, based on mineral content. The oceans cover approximately 85% of the land surface of the world, are salt water, mainly sodium chloride, NaCl, but contain many other dissolved salts with types and concentrations varying with locale and water depth.

Fresh water is land water — lakes, rivers, streams — but it is not "pure" water, as it contains dissolved minerals and organic matter: much of the mineral content is essential to human and animal diet and health. Water also collects underground in aquifer layers — usually sandstone, limestone, or granites — returning to the surface as springs or, when pumped, as well water.

In many industrial areas the underground "water table" is maintained and used as part of the economic water supply of a region. Many of the major rivers in the world have been dammed to form reservoirs as both a water supply and a source of hydroelectric power production — hundreds of small streams have "spill" dams producing mechanical power by use of a water-wheel . . . also used for lifting water in irrigation.

The normal evaporation cycle from the oceans produces naturally distilled water vapor — clouds — then rain with about 80% of such fresh water as snow and ice frozen in the Arctic and Antarctic ice caps.

Many major industries, such as steel plants and other metal processing plants, paper mills, and nuclear reactors are located on rivers or along seacoasts for the high volume source of water needed, primarily, as washing or cooling water in operation. Several of the quartz crystal processing plants are located east of the Continental Divide (Rocky Mountains) in the U.S. to take advantage of the high purity ground water coming off of the Divide.

"Pure" water, either demineralized or distilled, is manmade on site by many companies, particularly those involved in metal processing as well as for medical purposes. Distilled water is available as "bottled" water for car batteries or clothing irons. Water "stills" are nothing more than large boilers with the steam bled off to recondense as distilled water, and it may be single, double, or triple distilled: ion-exchange positive and negative resin beds may be included in the distillation process with, say, the triple distilled — highest purity form — processed through an all quartz boiling system. All distilled or demineralized water is unstable, in a sense, as it will immediately begin absorbing gases, dust and other

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contamination when exposed to air, reverting to normal, natural mineralized fresh water. Distilled water is also an excellent medium for algae growth, such that distilled water lines require on-going maintenance cleaning — once a week in highly critical surface cleaning operations — and should include submicron filters in-line at the user point, as in a laboratory chemical sink. Inert PVC piping is preferred — not copper piping with rosin-core solder joints . . . and there should not be any stub-off lines where water can collect and stagnate. In closed recirculation systems (recirc systems), common practice is to include a glass tube section in-line that can be flooded with UV light to kill algae.

In chemistry, water is not only called "the staff of life" with regard to human and animal consumption, but is considered the universal solvent as, with sufficient time and/or temperature/pressure conditions, it will dissolve and take all materials into solution, or it will form an environmentally stable oxide such as silica, SiO_2 or alumina, Al_2O_3 as these are the two most stable natural minerals under geologic conditions.

Recently, a new form of water has been discovered; polywater. It is a high density chain of polymer water molecules that can be formed in capillary tubes of about 10 mm diameter. It boils at 150°C and freezes at −40°C into a glass-like substance that does not resemble ice.

Many of the natural minerals contain water in their chemical formula as the hydrated minerals, such as gypsum, $CaSO_4.2H_2O$. With water removed it is the mineral anhydrite, $CaSO_4$, better known as Plaster of Paris . . . used in medicine for immobilizing broken bones and in dentistry as a mold in making false teeth; it is the plasterboard of construction used for holding small stones for cutting and polishing. When dry, powdered calcium sulfate is mixed with water, it sets as a hard white form of gypsum that is relatively inert to further alteration.

The precious and semi-precious gem stone opal, $SiO_2.NH_2O$, has water both in its physical crystal structure and as entrapped liquid. It is a colloid, highly stress fractured, and the entrapped water is what produces the splay of colors. Opals should be moistened occasionally or kept in a humid atmosphere to maintain their "color" — otherwise they will slowly dry out and, eventually, can even commence to crumble and powder. There are many other natural minerals containing water in their formulas like opal and gypsum.

Heavy water, D_2O — deuterium — is best known for its use in developing the first atomic bombs. It does occur naturally in moist air as a very small constituent and has been used as a tracer element in drinking water to determine the length of time water is retained in the body before elimination, approximately 30 d.

Waste disposal is of major concern today, particularly in industrial areas as, in many cities, chemical wastes enter the sewage system or are dumped into rivers, streams, and lakes directly from the user plant. Such contamination, to include "acid rain", has polluted our lakes, waterways, and many coastal areas killing fish and altering the ecology of plant and sea life. Most acids can be readily neutralized with bases, such as ammonia, NH_3 and ammonium hydroxide, NH_4OH added to the acid/water waste, but many chemicals require special filtration and conversion to insoluble salts — chromium acids are particularly difficult to remove and can severely contaminate the underground water table. The same applies to alcohols and solvents: these are disposed of separately, not into sewage drains along with the acids — the mixture can be explosive with certain combinations — and cannot be neutralized by water. Trichloroethylene (TCE) is a major industrial cleaning solvent, is carcinogenic and, in many areas where it has been used, has contaminated the underground water table, springs, and wells. Waste disposal and its neutralization with water, acids, and bacteria is a major industrial business by itself. The waste water can be reconstituted as fresh, human potable water, or for reuse in agriculture and industry. See Ice for additional discussion.

Technical Application: Commercial water — sometimes referred to as "water with the rocks removed" — is used for general cleaning of parts where maximum chemical purity

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is not required. It also is the water used as the cooling liquid in any heat generating operation to absorb and carry the heat away with continual replacement of cooler water. A system may be water-jacketed; water pipes coiled around the heater vessel; as a bath into which hot parts are quenched. In the latter case, both fresh and salt water are used as a quenching medium: certain types of irons, steels and other metal alloys are structurally formed in this manner. Semiconductor metals and compounds have been quenched for both device fabrication and in structure studies at RT, hot (40—80°C) or chilled with ice (4—10°C).

Many laboratory chemical processing sinks contain both commercial and distilled water faucets with the DI water used for finally cleaning and mixing of etchants. As many etchants are exothermic, a beaker of the etch solution is placed in a water bath — there are commercial systems available for batch etching using recirc water systems — and small ultrasonic generators often use water baths in which the etch beaker is placed for ultrasonically generated bubble stirring action. The larger degreaser systems using cleaning solvents are both water jacketed at the top to reduce hot solvent evaporation loss, but may use ultrasonic vibration cleaning in the hot solvent tank section.

There are a number of "cold solutions" using water, snow, and ice as part of the chemical mixture to establish a specific cold temperature: a bath of ice and acetone, as an example, is often used for slight preferential etching and tuning of a semiconductor p-n junction device with the etch solution at about 8°C. Water and dry ice, solid CO_2, in a bubbler are used to remove water vapor from processing gases by freezing out the water as the gas is bubbled through — argon gas in particular.

Boiling water is used to form a porous hydrated oxide on metal and compound surfaces, which is then removed — usually with HF — as a surface cleaning process, or the oxide remains as surface passivation. Thermal oxidation, as water vapor in an inert carrier gas, such as nitrogen, is carried into a furnace at 1000°C and was the initial method used to produce SiO_2 protective thin films on silicon wafers, the process called wet thermal oxidation. Dry thermal oxidation uses oxygen, O_2 gas in a nitrogen, N_2 or argon, Ar inert gas carrier. The SILOX system uses an $SiH_4/N_2/O_2$ gas mixture for deposition of a silicon dioxide, SiO_2 layer on any metal or metallic compound between 350—500°C. The original pyrolytic deposition of an oxide from liquid chemical solutions passed through a furnace between 400—800°C is now called chemical vapor deposition (CVD) and there are several variations with their own acronyms: LPCVD, low pressure CVD, HPCVD, high pressure CVD, as only two examples.

Steam oxidation, as hot water vapor under pressure measured in atmospheres (atm) or as pounds-per-square-inch (psi) is now used for silicon dioxide growth on silicon wafers, and can be used to deposit other oxides on other metals, such as aluminum, nickel, copper, chromium, etc. Steam also is used for general surface cleaning . . . from a Solid State wafer or electronic part . . . to building walls in cities . . . to clothing in the garment industry. Needless to say, steam under pressure is a major mechanical operator, and was fundamental to the Industrial Revolution that began in England in the early 19th century: railroad engines, steam ships, factories, and home heating. The Romans were building steam-heated homes prior to 1 A.D. in Italy as well as in England. Today, volcanic steam from Mt. Vesuvius in northern Italy is tapped for steam power, and is being developed as an alternate source of electric power generation from subsurface hydrothermal areas throughout the world . . . northern California, as one example.

Even though water is considered neutral, with a pH 7 on the Sorenson Scale — it can act as an "acid" solvent on many soluble minerals, such as single crystal halite, NaCl or sylvite, KCl. These compounds, as well as others, are used as substrates for thin film metal and compound epitaxy growth in the study of growth and crystal structure of the thin films. Water is used to etch polish the surfaces of these substrates prior to deposition; it also is used to remove the subsequently deposited thin film using the "float-off" technique by

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liquefying the substrate/thin film interface in order to release the thin film for study under a transmission electron microscope (TEM).

Water is not only used as the cooling medium in many cutting operations, to cool both the cutting blade and parts being cut, it is used as the cutting tool, itself, as a water jet under pressure or as high pressure steam.

In the growth of ice crystals (snowflakes) as well as single crystal ice, a cryogenic stage or cryostat for the water vapor chilled with either LN_2, dry ice, CO_2 or LHe, and may be as a cryogenic microscope stage for observing ice growth as it occurs. This may include nucleation with silver chloride, $AgCl_2$, as is done with seeding of rain clouds. And water also has been grown as salt water crystals from natural salt waters or artificial variations of chemical brine mixtures. Cold temperature studies have been in progress in Greenland and on both the Arctic and Antarctic ice caps for many years — core samples of ice have been cut, sectioned, then melted to obtain foraminifera and radiolaria as diatom fossil exoskeletons, as well as for water samples, to establish environmental conditions and age correlations of geologic periods in the past. There are similar studies with annual lake ice.

Several cold solutions have been developed using specific chemicals, alone, or in combination as eutectic mixtures for a specific temperature level below room temperature. Including the cryogenic liquid gases, temperatures range to several degrees below zero. These cold solutions are mainly used to establish a particular temperature for an etching solution, for chilling or quenching metals, and occasionally used as the direct etchant, themselves. The following collection is referred to here as Cold Etchants and is presented as a collective reference.

Etching: As an etching medium, alone, on soluble materials. The primary liquid carrier for mixing or chilling of other etchants.

WATER ETCHANTS

WATR-0001
ETCH NAME: Ice
TIME:
TYPE: Acid, solvent/storage
TEMP: 0°C
COMPOSITION:
x .... H_2O (solid)
DISCUSSION:

H_2O in its frozen solid form, used alone, has some special applications and major areas of study. It will freeze and preserve both organic and inorganic matter. Ice cores 2—4″ in diameter are cut from glacial ice in the Antarctic, then sections melted to obtain ancient sea life exoskeletons, to evaluate alkalinity, oxygen content, etc., in determining earth sea life and atmospheric conditions to as much as one million years in the past. In metal studies, hot liquified metal droplets are splashed onto an ice surface or pressed between two blocks of ice to observe quick-frozen material structure. It has been used as a slow etchant on water soluble compounds, an ice cube being rubbed across the surface to polish, or develop preferential etched figures and structure.

Ice caves in mountain areas have been used for food storage since ancient times, the Eskimos still use ice to freeze their food for preservation and, there are records from Asia Minor describing the transportation of ice by camel caravan from the mountains to the desert courts of Mesopotamia, circa 3500 B.C. The Arabs in those ancient times mixed chipped ice with fruit juices — sharbat — today's sherbets or the French sorbets — also called "ices".

In geology, ice expansion and contraction in rock crevasses is a primary method of reducing solid rock to soil. There are still ice houses making solid blocks of ice. See the Ice section for further details.

REF:

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WATR-0002
ETCH NAME: Water
TYPE: Acid, solvent
COMPOSITION:
x .... H_2O
DISCUSSION:
TIME:
TEMP: 4—99°C

H_2O in liquid form from above the freezing point to below the boiling point. As already discussed in the General and Technical Application sections, water is considered the "universal solvent" in chemistry. It is the primary quenching medium following etching of all materials, unless they are water soluble compounds. On soluble compounds it acts like an acid, and is used for surface polishing, preferential etching, structure forming, etc. As a quenching medium, it is used chilled, at RT, or warm to near boiling. For general quenching, washing, or rinsing it is most often used at RT or warm. It is also used as a cleaning spray, and water is tumbled to aerate the liquid, such as for drinking water to increase the oxygen content. Water vapor also is used for controlled humidity in air conditioners. In industrial systems as 50 to 100 ton units, home use systems are smaller, such as the Swamp Coolers where water is sprayed across fan-generated air.

Hot water, up to and including boiling water, is used to form a hydrated oxide on metal surfaces by immersion, often then stripped with HF, as a surface cleaning step. Occasionally, hot water is used as the rinsing medium after etching, or as the quenching medium in hot metal fabrication.
REF:

WATR-0003
ETCH NAME: Steam
TYPE: Acid, solvent
COMPOSITION:
x .... H_2O, hot vapor
DISCUSSION:
TIME:
TEMP: Above 100°C

H_2O above the boiling point as hot vapor at standard atmosphere and pressure (1 atm = 760 mmHg pressure) or under pressure as xx atm or psi, as pressurized steam. At standard atmosphere and pressure steam is used to drip clean or lightly oxidize surfaces. Under pressure, as in an autoclave to either clean metal parts (medicine) or to oxidize a metal, such as silicon for steam oxide growth at 1000°C and 10—150 psi.

Steam jets are used for metal cutting, for drilling, for mechanical motion operations of valves, etc., and steam is used in hydroelectric power generation. Natural steam from volcanoes has long been used for heating and cooking; there are steam irons for clothing, and steam pressing as part of dry cleaning, or used for compacting of materials. Steam boilers have been in use as a driving mechanism for railroad engines and steam ships, or in factory and mining for operation of steam cylinders or valves for over 150 years. Steam heating of housing, both natural from volcanoes and from man-generated boilers, has been in use since before the time of the Roman Empire, circa 400 B.C.

Steam is still used in cooking. A double-boiler, with heated water in the lower section, is used for cooking steamed vegetables, meat, poultry, or fish and shellfish.

Distilled water, DI H_2O, is made by boiling water, passing the hot steam into a second collecting vessel, and reliquefying as high purity water. This removes all minerals and organic compounds from the water with the exception of small amounts of chlorine and sulfur oxides. The water can be further purified by passing through positive and negative ion exchange resin beds, and single-distilled water is the major source of DI water used in industry. It can be single-, double-, or triple-distilled. It also can be as demineralized, dmH_2O, only. Distilled water is flat in taste and not used for drinking or cooking, although it can be so used, but no longer contains the essential minerals needed for human and animal

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consumption. It is used in both steam ironing and in vehicle batteries. Most chemical laboratory sinks have both industrial and DI water taps, with the DI water used in final rinsing of critical parts. DI water lines required on-going cleaning maintenance with potassium permanganate or hydrogen peroxide solutions to prevent algae build-up in the lines, and water recirculating systems, recirc systems, use an in-line glass section with ultraviolet light passing through the water to kill algae growth as distilled water is an excellent medium for such growth.

In nature, the evaporation of ocean salt water into the atmosphere as clouds produces naturally distilled DI water which, as rain, is still high purity until it reaches the ground and remineralizes. It does pick up some minerals from the atmosphere, is potable water — soft water — and is still collected from rainstorms in cisterns in arid countries and areas. Hard water contains calcium and magnesium sulfates and other salts that reduce foaming action of soaps, the compounds can build up in steam boilers and lines, requiring descaling and removal. Such boiler cleaning and conversion of hard water to soft water is a major industry, as is the disposal of industrial and home generated waste waters. Most sewage plants include steam boilers as part of the plant operation in addition to the aerobic and anaerobic bacterial action, and addition of chemical reducing agents. It is worth noting that such treated water is potable, and can be reused as agricultural or industrial water.
REF:

COLD ETCHANTS

Note: The following solutions are used to obtain a specific temperature below room temperature (RT) (22.2°C/72°F) for the purpose of chilling another solution or material more than as a direct cleaning or etching medium.

COLD-0001
ETCH NAME: Ice water
TYPE: Solute, chilling
COMPOSITION:
x .... H_2O, liquid
x .... H_2O, solid
TIME:
TEMP: 4°C, nominal
DISCUSSION:

H_2O, as a mixture of ice and fresh water (may be salt water). Used as a water bath to cool an etching solution to prevent excessive exothermic heating, only, to obtain preferential etching action, or for cleaning action, only. As a heat removal medium against exothermic reaction of an etchant such chilling is used to control etch action with removal of heat using heat exchanger coils which, themselves, may be additionally chilled. As a quenching medium to obtain specific structure of irons, steels, copper, etc., it can be either chilled fresh or salt water. Similar quenching of Solid State materials is done mainly to establish electronic functions of the device being fabricated. As a final washing medium — static, flowing, or spray — cold water can be used following etching or use of cleaning solutions on materials. Ice water solutions will maintain a temperature of about 4°C as long as ice is still present in the liquid, but can vary from just under 22°C (RT) to a cold ice slush if chipped or shaved ice is used. Note that most etch solutions, as they are chilled, become progressively more preferential in attack, eventually accentuating only cracks and other major damage artifacts on a material surface.
REF:

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COLD-0002
ETCH NAME: Ice/acetone
TYPE: Solute, chilling
COMPOSITION:
x .... ice, chunk
x .... acetone (ace)
TIME:
TEMP: 80°C nominal
DISCUSSION:

H_2O, as solid ice in the ketone acetone, CH_3COCH_3. Used as an etchant chilling medium for reactive application of a solution for general surface cleaning with minimum attack removal of material. HF:HNO_3 etchants with or without HAc or H_2O become progressively more preferential below RT. In processing silicon aluminum alloy transistors, as an example, used to fine-tune the p-n junction for optimum electrical characterization with slight preferential and surface cleaning action. Note that acetone alone is the primary solvent for photo resist lacquers using the life-off technique to remove excess metallization form photolithographically patterned semiconductor wafers and similarly structured materials. This ice/acetone mixture also is used for the removal of water vapor (moisture) from gases, such as for high purity argon gas from cylinders where the argon is used to back-fill a quartz diffusion tubes used for gallium diffusion of silicon controlled rectifiers, SCRs. Any trace water moisture in the argon can produce gallium oxides which are detrimental in SCR fabrication.
REF:

COLD-0003
ETCH NAME: Acetone
TYPE: Ketone, cleaning
COMPOSITION:
x .... $(CH_3)_2CO$ (Ace)
TIME:
TEMP: −95°C
DISCUSSION:

Acetone, used as a spray from a stainless steel can pressurized with nitrogen gas. It is the standard solvent for removal of photo resist lacquers and surface cleaning of parts in photolithographic processing. **CAUTION:** Can condense water droplets from a humid atmosphere as beads on an otherwise clean surface. It is a clear liquid with a highly pungent, slightly sweet odor and, when mixed with bromine, Br_2, becomes the lachrymator . . . tear gas . . . as a bromoacetone.

Acetone also is used as a soaking liquid for removal of photo resists, usually at room temperature. If used hot, it should be used in sufficient volume to prevent possible explosive ignition for, like many solvents, its hot vapor pressure ignition point is relatively low.
REF:

COLD-0004
ETCH NAME: Nitrogen, liquid
TYPE: Gas, freezing
COMPOSITION:
x .... LN_2
TIME:
TEMP: −196°C (nominal)
DISCUSSION:

Nitrogen gas as a cryogenic liquid obtain by the fractionation of air. A major use of LN_2 in industry is chilling the cold traps of vacuum evaporator systems. In a hot diffusion pump system, the cold trap is located immediately below the gate valve that opens into the bell jar chamber, and immediately above the diffusion pump, preferably, as a vertical stack. The trap is kept filled with LN_2 during an evaporation cycle to, initially, pump-down and remove air, other gas and water vapor from the bell jar, and then to help maintain a gas-free vacuum in the operating chamber. The LN_2 primary function is to remove water vapor

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by freeze-out to prevent reverse diffusion back up into the chamber while the system is operating, particularly with the main gate valve opened for high vacuum pumping by the hot oil diffusion pump. It also prevents back-diffusion of the rising hot oil in the diffusion pump from entering above the gate valve, helping to recondense the oil, which returns to the bottom of the diffusion pump by draining down the internal pump wall.

In other vacuum systems, such as those used for RF sputtering the initial roughing pump uses LN_2, the diffusion pump is a cryopump operating with LHe, such that all hot oils are eliminated. These vacuum systems are optimum oil-free operations.

LN_2 has been used as a quenching medium similar to ice water (COLD-0001) in the metals industries. There is a "0°C Etch" used in Solid State material etching that is commonly chilled with LN_2 (See Gallium Arsenide). LN_2 storage tanks are maintained at many industrial facilities as a source of the liquid or for conversion to nitrogen gas from the LN_2 source through heat exchangers with both LN_2 and gas piped to user points in buildings. The gaseous nitrogen is often used for equipment air-valve operation rather than having to run in separate air lines, as LN_2 is already at the location. LN_2 can be drawn off at the outside tank or from an internal line for small quantity use from polystyrene dewars. Larger metal dewars are rated from 50 to 200 l volume, the larger types on wheels. Note that the best piping method for bringing in liquid nitrogen with minimum gas-off loss is in double-walled steel pipes with a light vacuum drawn on the annular space . . . the same construction used in the storage tanks and 2000 gallon tanker trailers used for transportation of all cryogenic gases.

Ion pump vacuum systems, titanium rod getters in the ion pump replacing the hot oil diffusion pump, have from one to six pumping cylinders filled with charcoal or a Dri-Rite-type compound, with the dewars attached around the cylinders and kept filled with LN_2 during the evaporation cycles. At operation end, the cylinders are allowed to automatically out-gas themselves; whereas the gettering-charcoal of a cryopump system requires pump bake-out on at least a weekly cycle to remove collected gases.

As a chemically inert, clear, freezing liquid at −196°C, LN_2 should be handled with the caution due any dangerous chemical. A rose, for example, dipped into liquid nitrogen for 10 sec, then removed still frozen, will shatter like glass when struck, and the same thing can happen to a human finger. Where the cold liquid is off-gassing to nitrogen gas, the area should be well ventilated. Too much nitrogen in a work area causes sleepiness, and can cause death if all oxygen in the air is replaced by pure nitrogen. Operators required to enter and clean LN_2 storage tanks or mobile trailers allow them to come up-to-air before entering, wear air-packs, or work with an outside source pumping air into the tanks and trailers.

LN_2 has been used to freeze mercury spills. It is the "cold" portion of hot/cold high reliability test units used in evaluating electronic parts, and has been used in thin film adhesion testing: specimen from a hot plate to quenching in LN_2. LN_2 cryostats are used in laboratory growth and study of single crystal ice, snowflakes, and both natural and artificially compounded salt water brines.

REF:

COLD-0005

ETCH NAME: Helium, liquid

TYPE: Gas, freezing

TIME:

TEMP: −269°C (nominal)

COMPOSITION:

x .... LHe

DISCUSSION:

Helium is a colorless, odorless, inert cryogenic liquid. As a liquid, helium reaches the coldest temperature known to man, to within 1°A, but cannot be solidified. The cryopump units designed for vacuum systems use a combination of LHe (to 2.4°A) in the pump and fine chunk graphite as the absorber. These are called "oil-free" pumping systems when coupled with an LN_2 pump unit replacing the standard mechanical oil pump used in pulling

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the initial vacuum to 1 μm or 1×10^{-3} Torr. After 50 to 100 evaporation cycles, the cryopumps require regeneration by bake-out for removal of absorbed gases in the graphite, with installation of new graphite on a yearly basis. LHe can be used as a chilling liquid for etching, but LN_2 is more widely used due to cost factors.

LHe has been used as a source for helium gas like LN_2 (COLD-0004). It is occasionally used as an inert furnace gas and for drying . . . helium blow-off, rather than nitrogen blow-off in processing Solid State parts.

REF:

COLD-0006

ETCH NAME: Carbon dioxide

TIME:

TYPE: Gas, chilling

TEMP: −85°C (nominal)

COMPOSITION:

x .... CO_2, solid (dry ice)

DISCUSSION:

CO_2, as a white crystalline solid called dry ice. Supplied commercially from the liquifaction and solidification of the gaseous carbon dioxide similar to the process for liquifaction of air and separation of cryogenic nitrogen and oxygen. Dry ice is one of the few compounds that sublimes directly from the solid to the gas without passing through a liquid state. It must be kept below its cryogenic temperature as a solid or else it converts to gas and dissipates into the atmosphere; not like water that can be frozen as ice, liquified as water, gassed as steam, then recondense as water and re-frozen as ice with little or no loss of the original material volume.

Small quantities of dry ice can be fabricated from pressurized CO_2 gas cylinders. When CO_2 gas is released rapidly from a pressurized container it will freeze as a solid if confined during release. Attach a hinged steel unit on the cylinder release port, open the cylinder valve, and a small solid cake of CO_2 (about 3″ diameter, 1″ thick) will be formed.

A major application is in shipping of materials in the frozen state. Both epoxy and polyimide resins are shipped in polystyrene containers packed with pea-sized chunks of dry ice. The dry ice slowly gasses-off, but is good for about 7 to 8-days as a temporary freezing medium. Commercially it is supplied as one foot square/one inch thick blocks separated by brown wrapping paper and in a cardboard box. At room temperature a block will last nearly 48 h. Where dry ice is used on a daily basis, it is delivered every 2 days at minimum, as very few laboratories have cold storage facilities below −100°C available.

Dry ice also is used as a source of carbon dioxide gas in metal processing, and in the preparation of fertilizers. As a furnace gas it has been used for both its oxygen and carbon content, oxidation or carbonation, respectively. As a gas mixture in Solid State processing in this regard, such as for a carbon thin film with annealing conversions to a diamond-like compound (DLC).

As the gas, it is produced naturally by the fermentation process (wines and beers); added to water as seltzer water; and added to many soft drinks, such as colas, root beer, etc. as carbonated beverages. It occurs in some natural springs and has been directly bottled as carbonated water.

REF:

COLD-0007

ETCH NAME: Dry ice/acetone

TIME:

TYPE: Gas, chilling

TEMP: −80 to −100°C

COMPOSITION:

x .... CO_2, solid

x .... CH_3COCH_3 (acetone, Ace)

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DISCUSSION:

CO_2, as dry ice in liquid acetone. This cold liquid mixture is used to remove water moisture from process gases, such as argon. Argon has an affinity for moisture adsorption even in a pressurized cylinder, such that it is bubbled through a Pyrex or quartz bubbler containing a dry ice/acetone mixture to freeze-out and remove water moisture before use. This method has been used where argon has been an inert back-fill gas for closed tube gallium diffusion of silicon wafers in making silicon controlled rectifiers, SCRs.

This mixture also has been used as a general chilling medium for etching solutions, such as the "0°C" etch (See Gallium Arsenide section).

REF:

COLD-0008

ETCH NAME: Oxygen, liquid — TIME:

TYPE: Gas, reactive — TEMP: −183°C (nominal)

COMPOSITION:

x .... LOX

DISCUSSION:

O_2, as a colorless, reactive cryogenic liquid. Obtained by the liquifaction and fractionation of air for both LN_2 and LOX. Delivered in 2000 gallon trailers to on site storage tanks like LN_2, mainly as a source of gas. Until the advent of Solid State semiconductor processing in the early 1950s the primary users were medical facilities and the metal industries. Today, as LOX, it is a primary fuel for aircraft, rockets/missiles, and space vehicles. **CAUTION:** LOX is instantly explosive in the presence of grease and oil.

As oxygen gas it is mainly used as an oxidizer in furnace processing, such as an O_2/N_2 mixture for growth of thin film SiO_2 on silicon wafers as dry thermal oxidation at 1000°C. As a 5% O_2/CF_4 gas mixture it is a dry chemical etching (DCE) system for structuring or selective etching of a wide number of Solid State materials in device construction.

REF:

COLD-0008a

ETCH NAME: Air, liquid — TIME:

TYPE: Gas, reactive — TEMP: −175 to −180°C

COMPOSITION:

x .... L_{air}

DISCUSSION:

Air, as a colorless, odorless cryogenic liquid. Manufactured by repeated pressurizing to liquify, then used as a cryogenic source of air or fractionated for LOX, LN_2, and other cryogenic rare gases, such as neon, krypton, xenon, and others by additional pressurizing, chilling and fractionation.

It can be used industrially as a very clean, high quality source of air, though its primary use is as a source of LN_2 and LOX.

Liquid air is used to bottle gas under pressure as a cylinder gas . . . Al cylinder, 3600 psi . . . where high purity air is required in processing, such as for an oxidizing agent.

REF:

COLD-0009

ETCH NAME: Hydrogen, liquid — TIME:

TYPE: Gas, reactive — TEMP: −253°C (nominal)

COMPOSITION:

x .... LH_2

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DISCUSSION:

H_2, as a clear, odorless cryogenic liquid. The gas is pressure liquified in a process similar to that used for air, nitrogen and oxygen. It is supplied to industry in 2000 gallon tanker trailers, as is LOX and LN_2, but normally used from the trailer parked on site rather than being transferred to a storage tank.

Its major use is as a source of hydrogen gas for the reducing atmosphere in furnace processing of materials. Also as a source of high purity hydrogen in pressurized cylinders (Al cylinders, 3600 psi) as pure dry hydrogen in a single cylinder or as a "tank farm", such as an A21 interconnected series of cylinders that are transported as a single unit for installation on site. Such a farm with an LN_2 tank source available are used to mix forming gas (FG) (85% N_2/15% H_2). As an Al pressurized cylinder or small lecture bottle gas mixtures and are available as doping gases in Solid State material processing, such as SiH_4, AsH_4, PH_4.

As LH_2 it is still used as rocket/missile, aircraft, or space vehicle fuel in combination with of LOX. **CAUTION:** Both LH_2 and H_2 are explosive and flammable in concentration and in the presence of air. Most hydrogen gas reduction furnaces have at least one hydrogen burn-off outlet with an automatic trigger spark to insure ignition.

REF:

COLD-0010

ETCH NAME: Chlorine

TIME:

TYPE: Gas, reactive

TEMP: 0°C @ 6 atm

COMPOSITION:

x .... LCl_2

DISCUSSION:

Cl_2, as a pale greenish/yellow cryogenic liquid. Chlorine was the first gas to be liquified, as it requires minimum chilling and pressurizing as compared to other cryogenic gases, as shown above. When mixed with water and ice it forms a crystalline greenish-yellow hydrate, $Cl_2.8H_2O$ that is unstable, though chlorinated water has many industrial uses, to include water treatment as drinking water, as a swimming pool cleaner, and in industrial chlorination processing of metals and alloys.

LCl_2 is industrially supplied in both railroad and truck trailers, similar to LN_2 and LOX, with railroad cars holding 8000 to 10,000 gallons. The primary use is as a gas in the making of chemical compounds, to include hydrochloric acid, HCl, and in the compounding of fertilizers. Chlorine is a very active gas, second only to fluorine, and will combine with all elements other than gold and platinum. Sodium chloride — the mineral halite, NaCl — is common table salt, and the major source of chlorine. The mineral sylvite, KCl, also is used as a salt, more bitter than table salt, but used as a dietary replacement as it is not retained by the body. Both are sea salts, ocean brines, and obtained by evaporation of salt water, or mined from buried salt domes, such as those along the Gulf Coast of the U.S. and the tremendous buried salt beds in Germany that are 100 miles long, and as much as a mile thick, derived from the evaporation of shallow ancient seas.

As a cryogenic liquid it has little direct use in Solid State material processing, but is a source for the compounding of many chemicals used as gaseous doping elements, such as PCl_3 and $POCl_3$ (pockle) widely used as a phosphorus dopant for silicon. It is supplied in pressurized cylinders of Al size (9" diameter, 5 ft high) for use as a chlorinating gas in metal processing. **CAUTION:** LCl_2 is a highly corrosive liquid and gas. Also, as a gas, the fumes attack the wet membranes — mouth, nose, throat — rapidly and are unbreathable forming a dense white cloud, primarily as HCl vapors.

REF:

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COLD-0011
ETCH NAME: Ammonia
TYPE: Gas, chilling
TIME:
TEMP: −34°C (nominal)
COMPOSITION:
x .... NH_3
DISCUSSION:

NH_3, as a crystalline chemical, cryogenic liquid or gas. It can be liquified by the combination of hydrogen and nitrogen gases in the presence of iron at 550°C under pressure. It is shipped as a liquid, can then be reduced to nitrogen and hydrogen as a major industrial hydrogen source, but a better portion is used in the compounding of fertilizers. It has application in many cleaning and etching solutions: Windex, as a cleaner and, medically, it is "smelling salts". It was one of the first refrigeration liquids, is still so used, though the more inert Freons are a more modern replacement. Both ammonia, NH_3, and ammonium hydroxide, NH_4OH, are basic solutions on the Sorenson pH Scale and used to neutralize acid sumps in waste disposal.

In Solid State material processing both ammonia compounds are used as cleaning solutions, and NH_3 gas is a source for growing metal nitrides, such as Si_3N_4.
REF:

COLD-0012
ETCH NAME: Freon
TYPE: Solvent, cleaning
TIME:
TEMP: −17 to −50°C, variable
COMPOSITION:
x .... *Freon

* Freon is the trademark of the Dupont Company and there are several variations of artificial compounds, several of which have a boiling point below 0°C.

DISCUSSION:

Freon, as gas or liquid. As liquids they also are compounded with other solvents, such as acetone and other azeotrope chemicals for specific material cleaning applications. Vapor degreasers use hot liquid Freon (Freon-TA) for cleaning purposes, and the cold-capable freons are used as a replacement for ammonia, NH_3, in refrigeration. As a liquid under pressure in a spray can, applications are similar to those for acetone (COLD-0003), for the cleaning of surfaces in Solid State material processing. They are excellent cleaning solvents, as they evaporate without leaving any residue. As a cold spray, they have been used to chill specimens surfaces under electron microscopes to prevent the heat from electron impingement altering the surface of a material bulk under observation.
REF:

COLD-0013
ETCH NAME: Salt/snow
TYPE: Salt, chilling
TIME:
TEMP: (1) 0°C (2) −0.4°C
COMPOSITION:
(1) 1 .... NaCl
3 .... snow
(2) 1 .... NaCl
1 .... snow
DISCUSSION:

NaCl/snow solution. Both solutions can be used to chill etchants to about 0°C. Also have been used for temporary packing and shipping of fish and other perishable foodstuffs. Salt water with ice or snow added will be about 4°C at the near-freezing point. As pure,

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fresh water (snow and ice) freezes at about 0°C, salt is spread on roads in the winter time to help in the melting and removal of road snow and ice. See section on ICE.
REF:

COLD-0014
ETCH NAME: Salt/snow
TYPE: Salt, chilling
TIME:
TEMP: (1) −27°C
(2) −44°C

COMPOSITION:

(1)	(2)
3 .... $CaCl_3$	2 .... $CaCl_3$
2 .... snow	1 .... snow

DISCUSSION:
$CaCl_3$/snow solution. The solutions shown can be used alone or to chill other solutions to the specific temperatures shown. Used in metal processes, chemical compounding and medicine.
REF:

COLD-0015
ETCH NAME: KOH/snow
TYPE: Alkali, chilling
TIME:
TEMP: −35°C
COMPOSITION:
4 .... KOH
3 .... snow
DISCUSSION:
KOH/snow solution. Used in chemical and metal processing for the specific temperature chilling point. It can be used as an extremely slow surface preferential damage etchant.
REF:

COLD-0016
ETCH NAME: Ammonium nitrate
TYPE: Salt, chilling
TIME:
TEMP: +3°C
COMPOSITION:
1 .... NH_4NO_3
1 .... H_2O
DISCUSSION:
NH_4NO_3 solution. Used in the metal industries as a "cool" quenching medium of irons, steels, etc. In Solid State material processing for material cleaning and slow preferential etching of surfaces with minimum removal as for fine-tuning of electrical characteristics (See COLD-0002).
REF:

COLD-0017
ETCH NAME:
TYPE: Salt, chilling
TIME:
TEMP: (1) +10°C
(2) −11°C

COMPOSITION:

(1)	(2)
5 .... NH_4Cl	1 .... NH_4Cl
5 .... KNO_3	1 .... KNO_3
16 .... H_2O	1 .... H_2O

DISCUSSION:
NH_4Cl/KNO_3 solutions. By varying the amount of constituents between the limits shown,

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a complete range of stable temperatures can be obtained. Both solutions used in general processing of metals and chemicals.
REF:

COLD-0018
ETCH NAME: HCl/ice
TYPE: Acid, chilling/etching
TIME:
TEMP: −86°C, eutectic
COMPOSITION:
1 .... HCl
1 .... ice
1 .... H_2O
DISCUSSION:
HCl acid mixed as a cold temperature solution in the proportions shown is a stable eutectic solution at −86°C. It has application in chemical passivation of metal surfaces as a chlorinated surface.
REF:

COLD-0019
ETCH NAME: KOH/ice
TYPE: Alkali, chilling
TIME:
TEMP: −65°C, eutectic
COMPOSITION:
1 .... KOH
1 .... ice
1 .... H_2O
DISCUSSION:
KOH/ice as a solution with water is a stable eutectic mixture. Like HCl/ice (COLD-0018) used as a metal surface passivating system for an oxidized surface.
REF:

COLD-0020
ETCH NAME: Nitric acid/ice
TYPE: Acid, chilling
TIME:
TEMP: −43°C, eutectic
COMPOSITION:
1 .... HNO_3
1 .... ice
1 .... H_2O
DISCUSSION:
HNO_3/ice as a solution with water is a stable eutectic mixture. Like COLD-0018 and COLD-0019 this solution can be used as an oxidation pacification system of metal surfaces in general processing.
REF:

COLD-0021
ETCH NAME: Salt/ice
TYPE: Salt, chilling
TIME:
TEMP: −55°C, eutectic
COMPOSITION:
1 .... $CaCl_3$
1 .... ice
1 .... H_2O

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DISCUSSION:

$CaCl_3$/ice in a water solution is a stable eutectic mixture. A general purpose solution for processing materials at the temperature shown.

REF:

COLD-0022

ETCH NAME: Salt/ice

TIME:

TYPE: Salt, chilling

TEMP: −21.13°C, eutectic

COMPOSITION:

1 .... NaCl

1 .... ice

1 .... H_2O

DISCUSSION:

NaCl/ice in a water solution is a stable eutectic mixture. Used as a quenching medium in metal processing for the specific temperature shown.

REF:

COLD-0023

ETCH NAME: MeOH/ice

TIME:

TYPE: Alcohol, chilling

TEMP: 8 to 10°C

COMPOSITION:

x .... MeOH

x .... ice

DISCUSSION:

MeOH/ice as a liquid solution of methanol with similar applications to that of ice/acetone (COLD-0002). See Ice/acetone for additional discussion.

REF:

COLD-0028

ETCH NAME: Ethyl alcohol

TIME:

TYPE: Alcohol, chilling

TEMP: −117 to +78°C

COMPOSITION:

x .... C_2H_5OH (EOH or ethanol)

x .... H_2O

DISCUSSION:

Ethanol (drinking alcohol) is completely miscible in water. As absolute alcohol it is 100% EOH. As a constant boiling mixture: 95% EOH:5% H_2O, and the most common maximum concentration. Denatured alcohol (industrial alcohol) has 5% methanol or ethylene glycol added to make it unfit for human consumption. As drinking alcohol the concentration is measured by proof/gallon: Absolute = 200 proof; 95% EOH = 195 proof. Absolute is primarily used in medicine as a disinfectant and as a skin chilling medium soaked on a cotton ball, etc., and occasionally used in industry for material surface cleaning in a similar manner or as a liquid rinsing medium like methanol after water quenching following etching, though denatured alcohol is more commonly used. Also used as a spray for chilling and cleaning surfaces. Like methanol, it too has been an additive to etchant solutions. See SI-0092c; SI-0041c (EOH, MeOH, glycols, glycerin). Some water- and alcohol-soluble compounds are etch polished in EOH.

REF:

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COLD-0029
ETCH NAME: Isopropyl alcohol
TYPE: Alcohol, chilling
COMPOSITION:
x .... $CH_3CHOHCH_3$ (IPA, ISO)
TIME:
TEMP: −85 to +82°C
DISCUSSION:
Propyl alcohol (isopropanol) is completely miscible with water and has similar uses to those of ethanol and methanol, but is not as widely used as those two alcohols. Often called propanol only and occasionally used mixed with acids in etch solutions. There are several other alcohols used, such as butyl, etc., for general processing of metals and metallic compounds, but to even a lesser extent than IPA.
REF:

COLD-0024
ETCH NAME: MeOH/dry ice
TYPE: Alcohol, chilling
COMPOSITION:
x .... CO_2 (dry ice)
x .... MeOH
TIME:
TEMP: −65°C or 209 K
DISCUSSION:
Dry ice/methanol used as an etch in a study of the dislocation slip system on (0001) zinc surfaces. Also used LN_2 (77 K or −196°C) at room temperature in air (298 K or 25°C).
REF: Billello, J C et al — *J Appl Phys*, 54,1821(1983)

COLD-0025
ETCH NAME: Poly water
TYPE: Acid, cleaning(?)
COMPOSITION:
x .... H_2O
TIME:
TEMP: −40°C
DISCUSSION:
H_2O, as poly water, is a high density form of water with tightly packed strings of connected water molecules. It has been made by pouring DI water into 10 mm diameter tubes, then allowing the tube to sit over DI water for high humidity action until the poly forms in the tube. It boils at +150°C and freezes at −40°C to a clear, plastic-like glassy material.
REF: Eisenberg, D & Kadsmann, W — *The Structure and Properties of Water*, Oxford University Press, London, 1969

COLD-0026
ETCH NAME: Ethylene glycol
TYPE: Glycol, chilling
COMPOSITION:
x .... CH_2OHCH_2OH (EG)
x .... H_2O
TIME:
TEMP: −17 to +197°C
DISCUSSION:
Ethylene glycol is completely miscible with water and is mixed as "anti-freeze" used in car radiators along with anti-rust compounds, as it can both prevent water from freezing or boiling. Also used in etchant solutions like glycerin for viscosity control, as a wetting agent, and can improve planarity of an etched surface to include improved surface cleaning action. See SI-0092h; SI-0099.
REF:

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COLD-0027
ETCH NAME: Methyl alcohol
TYPE: Alcohol, chilling
COMPOSITION:
x .... CH_3OH (MeOH)
x .... H_2O
DISCUSSION:
TIME:
TEMP: −97 to +65°C

Methanol (wood alcohol) is completely miscible with water, and also has been used as an anti-freeze, e.g., Zerone. As it has good affinity for water it is often used as a final rinse following water quenching of an etchant solution with air dry or nitrogen blow-off drying. Occasionally used as a constituent of an etchant, and will reduce viscosity. See Nital solutions under Iron and Steel where it is used as a replacement for ethanol. See SI-0092i.
REF:

WATR-0004
ETCH NAME: Air + water moisture
TYPE: Acid, reactive
COMPOSITION:
x .... air, moist
DISCUSSION:
TIME:
TEMP: 4—99°C

H_2O as liquefied droplets in air which is measured as relative humidity (RH) for a 0 to 100% moisture content. In the atmosphere, high pressure areas are low in moisture content (''hot'' air); whereas low pressure areas are high in moisture content (''cool'' air), and also referred to as ''dry'' or ''wet''. A 30″ water meter also is used in measuring moisture content and, as applied to high and low pressure areas, above 30, is high pressure, below 30, low pressure. In vacuum systems, such meters are used during the initial roughing pump-down cycle with 30″ roughly equivalent to 1 μm (10^{-3} Torr) vacuum, at which point the system is switched to high pressure pumping, e.g., 10^{-4} to 10^{-12} Torr.

Both the oxygen and water moisture content in air is responsible for the oxidation of all materials, from metals and rocks to the decay by-products of vegetation and animal remains. Light, dry air can be cold, such as over the Arctic ice packs, or light, dry, and hot, as in desert areas. In winter time, heavy, wet, and cold and, with a wind, the wind-chill factor can drop air temperature below −40°C with severe freezing effects. Conversely, hot dry winds can produce dangerous dehydration in humans and animals.

Both temperature and moisture content in the air in buildings is controlled by air-conditioning and humidifier systems. A ''swamp cooler'', widely used in homes during hot weather, blows hot air over water to both increase the humidity as well as reduce temperature. The larger commercial air-conditioners are rated for 50 or 100 ton capacity, and the amount of air-conditioning needed is established by the cubic volume of building area to be controlled. A single 50-ton unit can easily maintain temperature and humidity in a 100 ft^2 single story building under most atmospheric conditions. In Solid State material process areas, the ''Yellow Room'', used in handling photo resist application, is critical: nominally maintained at 70—72°F and 40% RH. In general work areas, the humidity can vary between, say, 50 to 60%. In the processing of semiconductor wafers, for example, a silicon wafer will automatically oxidize when exposed to air/moisture producing a passivating oxide surface of about 20 to 30 Å thickness — referred to as a ''native oxide''. Such oxides are usually stripped with HF immediately prior to processing steps, such as metallization, diffusion, epitaxy, etc., as the oxide can produce unwanted resistive layers in a device structure. Most metals passivate themselves automatically when exposed to air with the thickness of the oxide varying up to 50 Å thick depending upon the metal involved. Operating Clean Rooms are constructed similar to Yellow Rooms for temperature and humidity control, and both

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type rooms include a particulate dust size control with use of static arrestors . . . 1 μm sized dust particles or less . . . preferred at less than 10% per air foot volume.

Moist air is used in furnace oxidation of materials, and many metallic compounds have been studied under controlled high and low humidity conditions.

Along seacoasts both moisture and salt content in air is a consideration. Metal coupons are evaluated for corrosion in such environments as a standard test; moist air is used in other studies, to include smog mixtures.

REF:

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PHYSICAL PROPERTIES OF XENON, Xe

Classification	Rare gas
Atomic number	54
Atomic weight	131.30
Melting point (°C)	−111.9
Boiling point (°C)	−107.1
Density (g/cm³) 20°C	5.895
Thermal conductivity (10^4 cal/cm²cm/°C/sec) 20°C	1.24
1st ionization potential (eV)	12.127
Neutron cross section (barns)	24
Vapor pressure (°C)	−132.8
Critical temperature (C.T.) 0°C	+16.6
Hardness (Mohs — scratch) solid	1—2
Crystal structure (isometric — normal) solid	(100) cube, fcc
Color (solid)	Clear/bluish
Cleavage (cubic) solid	(001)

XENON, Xe

General: Like other rare gases, xenon occurs as a minor elemental gas in the atmosphere at about 0.0018%. It is obtained as a cryogenic liquid, LXe, by the fractionation of liquid air, L_{air}. It is chemically inert, in group 0 of the Periodic Table, but when electrically activated under pressure produces intense white light that can go from full intensity to extinction in about a millionth of a second. By synchronizing xenon flashes on a spinning part, such as a vehicle fan belt, the belt appears to stand still and can be studied while in motion. This is the stroboscope, and similar units are used as the "flash bulb" of photography; with chemical solutions, such as in plating out copper from $CuSO_4$ solutions; along with argon, neon, and mercury, xenon bulbs are used for high intensity white light for microscopes: and it is one of the white light constituents in neon signs.

Technical Application: Both the stroboscope and xenon microscope light bulbs are used in Solid State processing. Though seldom used due to scarcity, like argon, nitrogen, and helium, xenon can be used as an inert furnace atmosphere gas or as a blow-off gas for drying parts. It has been grown as a single crystal in vacuum under pressure and cryogenic temperature conditions for morphological study.

Etching: Vapor pressure variation under vacuum.

XENON ETCHANTS

XE-0001
ETCH NAME: Pressure
TYPE: Pressure, preferential
TIME:
TEMP: −112°C
COMPOSITION:

x .... pressure, vapor

DISCUSSION:

Xe single crystals grown in vacuum under pressure and cryogenic temperature (LN_2, −196°C). Crystal surfaces can be preferentially etched by varying the vapor pressure in the vacuum system.

REF: Schwentner, N et al — *Rare Gas Solids*, Vol 3, Academic Press, New York, 1970

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PHYSICAL PROPERTIES OF YTTERBIUM, Yb

Classification	Lanthanide
Atomic number	70
Atomic weight	173.04
Melting point (°C)	824
Boiling point (°C)	1427
Density (g/cm^3)	6.98
Latent heat of fusion (cal/g)	12.71
Heat of fusion (k-cal/g-atom)	1.8
Specific heat (cal/g) 25°C	0.035
Heat of vaporization (k-cal/g-atom)	38
Atomic volume (W/D)	24.8
1st ionization potential (eV)	6.2
1st ionization energy (K-cal/g-mole)	143
Electronegativity (Pauling's)	1.1
Electrical resistivity (micro-ohms) 298 K	25.1
($\times 10^{-4}$ ohm cm) 25°C	28
Compressibility ($cm^2/kg \times 10^{-6}$)	7.39
Neutron cross section (barns)	36 (37)
Magnetic moment (Bohr magneton)	0.41
Tensile strength (psi)	10,400
Yield strength (psi)	9,500
Magnetic susceptibility ($\times 10^{-6}$ emu/mole)	71
Transformation temperature (°C)	792
Standard electrode potential (volts)	+2.1
Coefficient of lineal thermal expansion ($\times 10^{-4}$ cm/cm/°C) 125°C	25
Ionic radius (angstroms)	0.86 (Yb^{+3})
Hardness (Mohs — scratch)	2—3
Crystal structure (isometric — normal)	(100) cube fcc
Color (solid)	Grey
Cleavage (cubic) solid	(001)

YTTERBIUM, Yb

General: Does not occur in nature as a native element, but is extracted from minerals of the Yttrium Group of rare earths, such as gadolinite, $Be_3FeY_2Si_{10}$, and others. Like most rare earth elements its chemical reactivity is similar to alkalies, and it will deliquesce in water with evolution of hydrogen. It is commercially available in powder, foil, and sheet, as well as single crystal forms. There are no major industrial applications, but there are possible chemistry and medical applications.

Technical Application: The metal is not used in Solid State processing at present, but there may be applications like other rare earth elements in fabricating garnets and similar materials. It has been grown and studied as a single crystal.

Etching: Water, dilute and mixed acids.

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YTTERBIUM ETCHANTS

YB-0001
ETCH NAME: Water
TYPE: Acid, removal
COMPOSITION:
x .... H_2O
DISCUSSION:
Yb as foil or single crystal specimens. Water can be used to dissolve the metal. Acids for etch patterning.
REF: Foster, W & Alyea, H N — *An Introduction to General Chemistry*, 3rd ed, D Van Nostrand, New York, 1947, 663

TIME:
TEMP: RT to hot

PHYSICAL PROPERTIES OF YTTRIUM, Y

Classification	Transition metal
Atomic number	39
Atomic weight	88.9
Melting point (°C)	1509 (1490)
Boiling point (°C)	2927
Density (g/cm^3)	4.472
Thermal conductance (cal/sec)(cm^2)(°C/cm)	0.035
Specific heat (cal/g) 25°C	0.071
Latent heat of fusion (cal/g)	46
Heat of fusion (k-cal/g-atom)	2.7
Heat of vaporization (k-cal/g-atom)	93
Atomic volume (W/D)	19.8
1st ionization energy (K-cal/g-mole)	152
1st ionization potential (eV)	6.6
Electronegativity (Pauling's)	1.3
Covalent radius (angstroms)	1.62
Ionic radius (angstroms)	0.92 (Y^{+3})
Electrical resistivity (micro-ohms cm) 298 K	59.6
Compressibility ($cm^2/kg \times 10^{-6}$)	2.68
Magnetic moment (Bohr magnetons)	0.67
Tensile strength (psi)	22,000
Yield strength (psi)	9,700
Magnetic susceptibility (emu/mole $\times 10^{-6}$)	191
Transformation temperature (°C)	1479
Cross section (barns)	1.3
Hardness (Mohs — scratch)	4—5
Crystal structure (hexagonal — normal)	$(10\bar{1}0)$ prism
Color (solid)	Grey-black
Cleavage (basal)	(0001)

YTTRIUM, Y

General: Does not occur in nature as a native element but there are some 30 minerals that contain yttrium and other rare earth metals. Many of these are niobates and tantalates but carbonates, silicates, and phosphates are also common. Xenotime, YPO_4 is one of the better known phosphates. As yttria, Y_2O_3, it has been known for almost 200 years and was named after the town of Ytterby, Sweden. "Yttrium earths" is a general term applied to

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minerals containing both the Lanthanide (Cerium group) and Actinide (Thorium group) of rare earth elements even though yttrium is not a member of either group. As the pure metal it is grey/black metallic in appearance with a higher melting point than silicon though it is best known in the oxide form as it is more stable than the metal, e.g., yttrium oxidizes readily in air and boiling water.

No major usage as the pure metal in industry other than as a minor constituent in some metal alloys and, as the oxide, is used in the glass and ceramics industries as a coloring compound.

Technical Application: None in general Solid State semiconductor type device fabrication, but a major component in artificially grown single crystal garnets: Yttrium iron garnet, YIG and Yttrium aluminum garnet, YAG, two of the better known laser materials. The electromagnetic properties of certain garnets, YIG as an example, are the basis of the bubble memory device that is finding increasing application in the computer field. Yttrium has been grown as a single crystal for general study. Additional yttrium compounds are shown here.

Etching: Very soluble in dilute acids and hot alkalies.

Y-0001
ETCH NAME: Potassium hydroxide
TYPE: Alkali, removal
TIME:
TEMP: Hot
COMPOSITION:
x .... x% KOH
DISCUSSION:
Y specimens. Shown as a general etch for yttrium.
REF: Hodgman, C D — *Handbook of Chemistry and Physics*, 27th ed, Chemical Rubber Co, Cleveland, OH, 1943, 484

Y-0002
ETCH NAME: Nitric acid
TYPE: Acid, macro-etch
TIME:
TEMP:
COMPOSITION:
x .... HNO_3
DISCUSSION:
Y single crystal specimens. Solution used to develop macro-structure in ytterium.
REF: Carlson, O N et al — *J Electrochem Soc*, 107,541(1960)

YXM-0001
ETCH NAME: Kalling's etch
TYPE: Acid polish
TIME: 10—60 sec
TEMP: RT
COMPOSITION:
100 ml .... HCl
100 ml .... EOH
5 g .... $CuCl_2$
DISCUSSION:
$Y_2(CoM)_{17}$, single crystal ingots. M = Al, Fe, or $(FeCu)_x$. Cut and mechanically polish wafers, then chemical polish with solution shown. Used in a morphological study of these materials.
REF: Slepowronsky, M et al — *J Cryst Growth*, 65,293(1983)

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YITTRIUM ZIRCONATE ETCHANTS

YZR-0001
ETCH NAME: Phosphoric acid
TYPE: Acid, polish
COMPOSITION:
x .... c-SiO_2
x .... H_3PO_4 (pH: 3—4)
DISCUSSION:
$(Y_2O_3)_m(ZrO_2)_{1-m}$, (100) wafers used as substrates for silicon epitaxy. First, mechanical lap with alumina, then chem/mech lap with the solution shown. Degrease: (1) TCE, (2) acetone rinse, (3) MeOH rinse, and (4) N_2, blow dry. Used as a substrate for epitaxy CVD growth of silicon.
REF: Liu, A L & Golecki, I — *J Electrochem Soc*, 132,234(1985)

TIME:
TEMP:

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PHYSICAL PROPERTIES OF ZINC, Zn

Classification	Transition metal
Atomic number	30
Atomic weight	65.37
Melting point (°C)	419.5
Boiling point (°C)	907
Density (g/cm^3)	7.133
Thermal conductance ($cal/sec)(cm^2)(°C/cm$)	0.27
Specific heat (cal/g) 20°C	0.0195
Latent heat of fusion (k-cal/g)	24.09
Heat of fusion (k-cal/g-atom)	1.76
Heat of vaporization (cal/g)	27.43
Atomic volume (W/D)	9.2
1st ionization energy (K-cal/g-mole)	216
1st ionization potential (eV)	9.391
Electronegativity (Pauling's)	1.6
Covalent radius (angstroms)	1.25
Ionic radius (angstroms)	0.74 (Zn^{+2})
Linear coefficient of thermal expansion ($\times 10^{-6}$ cm/cm/°C)	49.7
Electrical resistivity (micro-ohms-cm) 20°C	5.92
Cross section (barns)	1.10
Vapor pressure (°C)	736
Refractive index (n=)	
Hardness (Mohs — scratch)	2.5
Crystal structure (hexagonal — normal) alpha	$(10\bar{1}0)$ prism
(monoclinic — normal) beta	(100) a-pinacoid
Color (solid)	Bluish-white
Cleavage (basal)	(0001)/(001)

ZINC, Zn

General: Probably does not occur in nature as a native element. Three of the major zinc ores are calamine, a hydrated silicate of zinc; smithsonite $ZnCO_3$, the carbonate form; and sphalerite, ZnS — common name zincblende. The latter mineral is a compound semiconductor but even more important as it has the atypical tetrahedral unit cell crystallographic structure of most binary compound semiconductors. Zinc ores have been used for years in making brass, CuZn (30%), yet it was not recognized as a separate metal element until 1746 (Marggraf). As the pure metal, zinc is bluish-white, malleable at or above 100°C and a fair conductor of electricity. At red heat it will burn in air to the oxide although it is extremely stable under normal temperature conditions.

Zinc has major industrial applications as an alloy with copper, iron, and other metals. Although bronze, CuSn, and brass, CuZn have been used since antiquity — the Bronze Age — brass is more workable and useful as it is less brittle. The process known as galvanizing is a surface plating process and, along with tinning, galvanized zinc coatings are applied to iron and other metals for corrosion resistance to include direct dipping in molten zinc. Zinc is used as the negative electrode (cathode) in many electrolytic plating baths and as cathode plates in DC batteries. An added note on bronze and brass — there are well over 100 alloys and today the term brass is more common whether the alloy contains zinc or tin or a mixture of the two with copper. As the oxide, like titanium oxide, it is a high quality white pigment in paints; zinc chromates are used as brilliant yellow coloring

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agents in paints, glass, and ceramics. Metallic zinc also is used in the preparation of some organic compounds.

Technical Application: Zinc is considered a "poisoning" element in much Solid State material device processing as it can degrade electrical parameters and, due to its ability to diffuse through solid metals when subject to thermal processing, can embrittle alloy contacts. Regardless, brass is widely used in device assemblies and packaging with a copper plating of sufficient thickness to buffer or block solid-solid diffusion of zinc.

Zinc is used in the growth of several compound semiconductors, the most well known being zinc sulfide, ZnS (the natural mineral sphalerite), but also include zinc arsenide, ZnAs; zinc selenide, ZnSe; and zinc telluride, ZnTe. It also has been studied as a zinc tungstate and a silicon zinc phosphide single crystal.

It has been widely studied and grown as a single crystal and as single crystal platelets with (0001) surface orientation and it can be readily cleaved in this orientation.

Etching: Readily soluble in both acids and alkalies.

ZINC ETCHANTS

ZN-0001a
ETCH NAME: Hydrochloric acid, dilute — TIME:
TYPE: Acid, removal — TEMP:
COMPOSITION:
1 .... HCl
1 .... H_2O
DISCUSSION:
Zn specimens. Solution used as a general removal etch for zinc.
REF: Rezek, J & Craig, G B — *Trans Metall Soc AIME,* 221,715(1961)

ZN-0002
ETCH NAME: Hydrochloric acid, dilute — TIME:
TYPE: Acid, cleaning — TEMP:
COMPOSITION:
x .... 5% HCl
x .... H_2O
DISCUSSION:
Zn, pellets used as a zinc source for LPE growth of ZnSe from a Zn-Ga solute. Clean zinc in dilute HCl before use. Also studied sulfur doped ZnSe as ZnSSe.
REF: Fujitz, S et al — *J Appl Phys,* 50,1079(1979)

ZN-0001b
ETCH NAME: Nitric acid, dilute — TIME:
TYPE: Acid, saw — TEMP:
COMPOSITION:
1 .... HNO_3
1 .... H_2O
DISCUSSION:
Zn, (0001) wafers cut from single crystal zinc ingots using an acid saw technique. The solution shown used as the acid vehicle for cutting.
REF: Ibid.

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ZN-0003
ETCH NAME: TIME:
TYPE: Acid, cleaning TEMP:
COMPOSITION:
x .... HNO_3
x .... HAc
DISCUSSION:
Zn, (0001) platelets. Fabricated by pressing a 4 9s pure zinc 0.4 mm sheet between two glass plates with aluminum spacers. Heat to 693 K in argon. Most platelets were (0001) basal. Solution used to etch clean and remove surface contamination.
REF: Takama, T et al — *Jpn J Appl Phys*, 23,11(1984)

ZN-0004
ETCH NAME: TIME: 2 min
TYPE: Acid, polish TEMP: RT
COMPOSITION:
1 .... HNO_3
1 .... H_2O_2
1 .... EOH
DISCUSSION:
Zn, (0001) wafers and ingots. Solution used to polish specimens. Mix fresh when ready for use.
REF: Gilman, J & DeCarlo, V J — *Trans AIME*, 206,511(1956)
ZN-0001c: Rezek, J & Craig, G B — *Trans Metall Soc AIME*, 221,715(1961)
Zn specimens. Used in studying striation effects of critical residual shear stress on single crystal zinc. Solution used as a polish etch.
ZN-0005: Rosenbaum, H S & Saffren, M M — *J Appl Phys*, 32,1866(1961)
Zn, (0001) cleaved wafers using a razor and hammer for cleaving, also called the anvil and hammer method. Solution used to etch polish surfaces before etching for dislocations. Cleaving was done in LN_2.
ZN-0027a: DeCarlo, V J & Gilman, J J — *J Met*, 8,144(1956)
Zn specimens. Development work on chemical etching of pure zinc and cadmium metals. Solution used as a general polish etch.
ZN-0027b: DeCarlo V J & Gilman, J J — *J Met Abstr*, 8,84(1985)
Zn specimens. Work similar to that on ZN-0027a.
ZN-0027c: DeCarlo, V J & Gilman, J J — *Trans Am Inst Min Met Eng*, 206,511(1956)
Zn specimens. Work similar to ZN-0027a-b.

ZN-0006a
ETCH NAME: TIME: 1 min
TYPE: Acid, polish TEMP: RT
COMPOSITION:
32 g .... Cr_2O_3
4 g .... $Na_2SO_4.10\ H_2O$
2 g .... $Cu(NO_3)_2$
100 ml .... H_2O
DISCUSSION:
Zn, single crystals alloyed with copper or aluminum. Wafers were cut by an acid saw technique from as-grown platelets. Solution used as a polish etch. The Zn/Cu material had a bluish stain after etching. Remove with 5 ml HNO_3:20 ml H_2O_2:50 ml EOH, RT, 5 sec.
REF: Sinha, P P & Beck, P A — *J Appl Phys*, 33,625(1962)
ZN-0007: Sinha, P P & Beck, P A — *J Appl Phys*, 32,1222(1961)

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Zn, single crystal specimens used in a bending/polygonization study. #ZN-solution used as a polish etch.

ZN-0006b
ETCH NAME: TIME: 5 sec
TYPE: Acid, polish TEMP: RT
COMPOSITION:
5 ml .... HNO_3
20 ml .... H_2O_2
50 ml .... EOH
DISCUSSION:
Zn alloy single crystal platelets cut by an acid saw technique. Zn/Cu alloy has bluish stain after initial polish etch (Zn-0006a). The solution shown will remove the stain and produce a highly polished surface.
REF: Ibid.

ZN-0007a
ETCH NAME: TIME:
TYPE: Acid, preferential TEMP:
COMPOSITION:
16 g .... CrO_3
5 g .... $NaSO_4$
100 ml .... H_2O
DISCUSSION:
Zn, (0001) wafers were dislocation etched in this solution. Solution is good unless cadmium is present in the zinc.
REF: Gilman, J J — *J Metal*, 8,998(1956)

ZN-0025
ETCH NAME: TIME:
TYPE: Acid, defect TEMP:
COMPOSITION:
x .... alcohol
DISCUSSION:
Zn, (0001) freshly cleaved surface used in a study of dislocation development. Authors say that dislocations can be developed on freshly cleaved surfaces with alcohols, water and weak acid etchants.
REF: Regel, U R & Stepanova, V — *Kristallografiya*, 4,226(1959)

ZN-0006c
ETCH NAME: TIME: 10 sec
TYPE: Acid, preferential TEMP:
COMPOSITION:
10 ml .... NHZ_4OH
50 ml .... H_2O
2 g .... $HN_4(NO_3)_2$
DISCUSSION:
Zn, single crystal with 0.07 and 0.02% aluminum in a study of defects created by bending. After etching, rinse in DI water, then MeOH and dry with a stream of dry air.
REF: Ibid.

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ZN-007b
ETCH NAME:
TYPE: Acid, dislocation
COMPOSITION:
4 ml .... HNO_3
100 ml .... H_2O
TIME: 15 sec
TEMP: RT
DISCUSSION:
Zn, single crystal specimens used in a bending/polygonization study. After etching, rinse in DI water, then MeOH and blow dry with air. Etch time is critical — insufficient time, and no pits develop; excess etch time and pits are too large and overlap.
REF: Ibid.

ZN-0023a
ETCH NAME: Cadmium
TYPE: Metal, decoration
COMPOSITION:
x .... Cd
TIME:
TEMP: >321°C
DISCUSSION:
Zn, (0001) single crystal wafers used in a study of dislocations and impurity boundaries. Metallic cadmium diffused into specimens to decorate dislocations.
REF: Damiano, V V & Tint, G S — *Acta Metall,* 9,177(1961)

ZN-0023b
ETCH NAME:
TYPE: Acid, preferential
COMPOSITION:
32 g .... CrO_3
6 g .... Na_2SO_4
100 ml .... H_2O
TIME:
TEMP:
DISCUSSION:
Zn, (0001) wafers used in a dislocation study. Solution used as a dislocation etch and referred to Gilman: ZN-0007a.
REF: Ibid.

ZN-0024
ETCH NAME:
TYPE: Acid, cutting
COMPOSITION:
(1) 200 ml .... HCl
50 g .... $FeCl_3$
250 ml .... H_2O
(2) 1 .... HNO_3
1 .... H_2O
TIME: 30 min
TEMP: RT
DISCUSSION:
Zn, specimens $^3/_8$" thick. Solutions used with terylene thread to cut material. Zinc and tin were cut with solution (2); aluminum, brass, and copper with solution (1). (*Note:* Excellent review of metal growth methods.)
REF: Honeycombe, R W K — *Rev Met,* 4,1(1959)

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ZN-0008a
ETCH NAME: P-1 TIME: 5—6 sec
TYPE: Acid, preferential TEMP: RT
COMPOSITION:
160 g .... CrO_3
20 g .... Na_2SO_4
500 ml .... H_2O
DISCUSSION:
Zn, (0001) wafers and cylinders with prism surfaces $(12\bar{1}0)$ and $(10\bar{1}0)$ used in an etching and etch development study. Wafers were cleaved (0001) at LN_2 temperature (cool slowly to prevent deformation). Other wafers and cylinders were acid saw cut with 8 *N* HNO_3 and an SS wire. After cleaving or cutting, lap polish wafers on a lucite plate with 8 *N* HNO_3 liquid carrier. During etching in solution shown, agitate specimens and wash heavily in DI water to ensure removal of CrO_3 as it produces very rapid corrosion. Air blow dry.
REF: Brandt, R C et al — *J Appl Phys*, 34,587(1963)

ZN-0008b
ETCH NAME: P-2 TIME: 2—3 sec
TYPE: Acid, preferential TEMP: RT
COMPOSITION:
1 .... 16 *N* HNO_3
1 .... H_2O_2
z .... MeOH
DISCUSSION:
Zn, (0001) wafers and cylinders. Agitate specimens during etching; rinse in tap water, then running DI water and air blow dry. See ZN-0008a for additional disscussion.
REF: Ibid.

ZN-0008c
ETCH NAME: P-3 TIME: 2—3 sec
TYPE: Acid, preferential TEMP: RT
COMPOSITION:
160 g .... CrO_3
500 ml .... H_2O
DISCUSSION:
Zn, (0001) wafers and cylinders. Agitate specimens during etching; rinse heavily in tap water and then running DI water to remove residual chromic acid. Air blow dry. See ZN-0008a for additional discussion.
REF: Ibid.

ZN-0008d
ETCH NAME: E TIME: 5—6 sec
TYPE: Acid, preferential TEMP: RT
COMPOSITION:
(1) 1 g .... $Hg(NO_3)$
1 ml .... 16 *N* HNO_3
500 ml .... H_2O
(2) 1 .... (1)
2 .... H_2O
DISCUSSION:
Zn, (0001) wafers. Agitate specimen during etching; rinse in tap water, then running DI water and air blow dry. Solutions produce small hillocks and pips and size varies with

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etch time. Solution is preferential to planes perpendicular to (0001), the $\langle 10\bar{1}0 \rangle$ prism plane directions. See ZN-0008a for additional discussion.
REF: Ibid.

ZN-0008e
ETCH NAME: Nitric acid
TYPE: Acid, preferential
COMPOSITION:
x .... 8 *N* HNO_3
x .... H_2S
Note: Saturate nitric acid with H_2S
TIME:
TEMP: RT
DISCUSSION:
Zn, cylinder with $(10\bar{1}0)$ prism orientation. Develops dark bands within 15° of $(12\bar{1}0)$ but none on $(10\bar{1}0)$. All wafers and cylinders (ZN-0008a thru ZN-0008e) were held under vacuum for 12 h and then re-etched — showed a reduced etch removal/polish rate. Sub-boundary pile-up at dislocations, caused by deformation.
REF: Ibid.

ZN-0009
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
x .... 17% CrO_3
TIME: 20 sec
TEMP: RT
ANODE: Zn
CATHODE:
POWER: 8 V/cm²
DISCUSSION:
Zn, (0001) specimens used in a surface etching study of 8 KeV argon ion bombardment. Polish surfaces in this solution before bombardment. Also studied gold and aluminum, which were polished in other solutions.
REF: Cunningham, R L et al — *J Appl Phys*, 31,839(1960)

ZN-0010
ETCH NAME: Neon
TYPE: Ionized gas, preferential
COMPOSITION:
x .... Ne
TIME:
TEMP:
GAS FLOW:
PRESSURE:
POWER:
DISCUSSION:
Zinc specimens. Ne^+ ion bombardment used to develop etch figures on metals. Other metals evaluated were Al, Bi, Cd, Co, Mg, Cu, and Sn.
REF: Yurasova, V E — *Kristallagrafia*, 2,770(1957)

ZN-0011
ETCH NAME:
TYPE: Acid, polish/thin
COMPOSITION:
(1) 160 g .... CrO_3
20 g .... Na_2SO_4
500 ml .... H_2O
(2) x .... 10% HNO_3
TIME:
TEMP:

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DISCUSSION:

Zn, (0001) wafers. Wafers were deformed and studied for dislocation development. Solutions used to polish surfaces or etch thin.

REF: Kratochvil, P — *Phil Mag*, 13,267(1966)

ZN-0012

ETCH NAME:

TIME: (1) 5—10 sec
(2) 30—60 sec

TYPE: Acid, polish

TEMP: RT 20°C

COMPOSITION:

(1) 75 ml .... HNO_3, fuming
25 ml .... H_2O

(2) 70 ml .... HAc
30 ml .... HNO_3, fuming

DISCUSSION:

Zn specimens. Etch polish with (1) and follow with washing in (2). This same etch-clean system was used on cadmium. (*Note:* Red or yellow fuming nitric not shown. Usually refers to red fuming, 74% concentration.)

REF: Tegart, W J McG — *The Electrolytic and Chemical Etching of Metals*, Pergamon Press, London, 1956

ZN-0013

ETCH NAME: Nitric acid, dilute

TIME:

TYPE: Acid, cutting

TEMP:

COMPOSITION:

ANODE: Zn

1 .... HNO_3

CATHODE:

1 .... H_2O

POWER: 30 mA/cm^2

DISCUSSION:

Zn, single crystal specimens. Cutting done by passing a plastic thread (Saran) through solution and across specimen. Also used on tin.

REF: Sternhein, G — *Rev Sci Instr*, 26,1206(1955)

ZN-0014: Williams, W R et al — *Acta Metall*, 5,435(1957)

Zn, specimens acid saw cut in an X-ray study of plastic deformation in single crystal zinc.

ZN-0015

ETCH NAME: Hydrochloric acid, dilute

TIME:

TYPE: Electrolytic, forming/defects

TEMP:

COMPOSITION:

ANODE: Zn

1 .... HCl

CATHODE:

1 .... H_2O

POWER:

DISCUSSION:

Zn specimens. After electrolytic forming of specimens as hemispheres, solution used to develop grain boundaries.

REF: Cochran, J F & Mapother, D E — *Phys Rev*, 121,1688(1961)

ZN-0016

ETCH NAME: Nitric acid

TIME: 2—5 min

TYPE: Acid, preferential

TEMP: Boiling

COMPOSITION:

x .... HNO_3, conc.

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DISCUSSION:

Zn, (0001) wafers used in an etch pit study. Pits were different between the positive (0001) and negative (0001) surfaces.

REF: Czyak, S J & Reynolds, D C — *J Appl Phys*, 29,1190(1958)

ZN-0017a

ETCH NAME:

TYPE: Acid, preferential

TIME: 2—5 min

TEMP: RT

COMPOSITION:

1—5 .... 2% NaOH

1 .... H_2O_2

DISCUSSION:

Zn, diffused into GaAs wafers in a study of the precipitation of zinc in gallium arsenide. Solution produced sharply triangular pits on (111) surfaces. Also used $1HNO_3$:$2H_2O$ to develop dislocation pits.

REF: Black, J F & Jungbluth, E D — *J Electrochem Soc*, 114,181(1967)

ZN-0007c

ETCH NAME: Water

TYPE: Acid, defect

TIME:

TEMP: Hot to cold

COMPOSITION:

x .... H_2O

DISCUSSION:

Zn, (0001) cleaved wafers. By cycling the specimens from hot to cold water, a six-rayed star was developed. (*Note:* Similar to the percussion figure on mica, and the asterism of the corundum star sapphire)

REF: Ibid.

ZN-0007d

ETCH NAME:

TYPE: Electrolytic, polish

TIME:

TEMP:

ANODE:

CATHODE:

POWER:

COMPOSITION:

33% .... H_3PO_4

67% .... C_2H_5OH

DISCUSSION:

Zn, (0001) cleaved wafers. Solution used to polish specimens prior to preferential etching for defects.

REF: Ibid.

ZN-0018: Regal, V R & Stepanova, V M — *Sov Phys-Cryst*, 4,204(1960)

Zn, single crystal specimens. Specimens were subject to strain in a defect structure study. Authors say that no dislocations are observed if the specimens are electropolished after being strained.

ZN-0019: Damiano, V V & Herman, E M — *J Appl Phys*, 38,2740(1967)

Zn, (0001) wafers. After electropolish and preferential etching of specimens, loops and spirals were observed with both growth and movement with extended etch time.

ZN-0020: Yoo, M H & Wei, C T — *J Appl Phys*, 38,2974(1967)

Zn, (0001) wafers. After electropolishing, specimens were indented and etching produced twinning in the ⟨0001⟩ direction.

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ZN-0021
ETCH NAME: Nitric acid
TYPE: Acid, cutting
COMPOSITION:
x .... HNO_3, conc.
TIME:
TEMP:
DISCUSSION:
Zn, single crystal wafers acid-saw cut using a braided SST wire and nitric acid. Used fly-wheel to trim-cut, then lap on emery paper and polish with alumina wheel. Specimens used in a study of elastic constants from 4.2 to 77.6 K.
REF: Garland, C W & Dalven, R — *Phys Rev*, 111,1232(1958)
ZN-0026b: Kiritani, M et al — *Jpn J Appl Phys*, 2,595(1963)
Zn specimens were acid saw cut as rods with nitric acid.

ZN-0022
ETCH NAME:
TYPE: Acid, junction stain
COMPOSITION:
x .... HF
x .... H_2O_2
x .... H_2O
TIME:
TEMP:
DISCUSSION:
Zn, diffused into InSb, (100), n-type wafers to form p-n junctions. Wafers cleaved ⟨110⟩ and solution used to stain develop junction.
REF: Nishitani, K — *J Electron Mater*, 12,124(1983)

ZN-0025a
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
5 oz .... $Ni_3PO_4.H_2O$
1.2 oz .... $NaCO_3$
TIME: $^1/_2$—2 min
TEMP: 160—180°F
ANODE:
CATHODE:
POWER: 6 V
DISCUSSION:
Zn Specimens. Solution used to electropolish followed by cathodic dip in 220 g CrO_3/L at 212°F. See Zn-0008c.
REF: ASTM Committee — *Tool Engineer's Handbook*, McGraw-Hill, New York, 1949

ZN-0025b
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
4 oz .... NaCN
1 oz .... $NaCrO_4$
2 oz .... NaOH
1000 ml .... H_2O
TIME: 15—35 sec
TEMP:
ANODE:
CATHODE:
POWER: 20—40 A/ft² & 2 V
DISCUSSION:
Zn specimens. Solution used to electropolish specimens with current reversal during etch period, and with or without and added wetting agent. (*Note:* Igpal is one wetting agent.)
REF: Ibid.

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ZN-0026a
ETCH NAME: Chromic acid
TYPE: Electrolytic, polish
COMPOSITION:
x .... 20% CrO_3

TIME:
TEMP:
ANODE:
CATHODE:
POWER:

DISCUSSION:
Zn specimens acid saw cut as rods with HNO_3. Solution shown used both electrolytically to polish or as a wet chemical etch — see P-3 etch. ZN-0008c for use as a preferential etch. This was a study of impurity substructure in zinc crystals.
REF: Kiritani, S et al — *Jpn J Appl Phys*, 2,595(1963)

ZN-0028
ETCH NAME:
TYPE: Mechanical, defect
COMPOSITION:
x .... LN_2

TIME:
TEMP: −196°C

DISCUSSION:
Zn specimens cleaved under LN_2. Studied the as-cleaved surfaces for tilt boundary motion relative to static and dynamic sheer stress.
REF: Veerland, T Jr — *Acta Metall*, 9,112(1961)

ZN-0029
ETCH NAME: Argon
TYPE: Ionized gas, defect
COMPOSITION:
x .... Ar^+ ion

TIME:
TEMP:
GAS FLOW:
PRESSURE:
POWER:

DISCUSSION:
Zn and Cu specimens. Use of positive cathode sputtering as ion bombardment for micro-relief and surface erosion. Dislocation etch pits in both materials, and slip traces on deformed zinc. There can be re-deposition of metal in the close-packed crystal plane directions.
REF: Yurasova, V E — *Zhtekh Fiz*, 28,1966(1958)

ZINC ALLOYS, ZNM

General: Zinc does not occur as a metallic alloy in nature, though there are over 50 known minerals classified as zinc-minerals as oxides, sulfides, silicates, and carbonates, in addition to phosphates and arsenates. Sphalerite, ZnS; smithsonite, $ZnCO_3$; willemite, $ZnSiO_4$ and zincite, ZnO are representative and major ores of zinc. It is associated with copper, iron, lead, and manganese deposits.

Metallic zinc is considered one of the "heavy" metals of industry, and is associated with copper, iron, steel, and lead in manufacture.

The best known artificial zinc alloy is brass, CuZn, and its associate, bronze, CuSn. There are several hundred brass, bronze, and brass/bronze mixtures designed for specific applications with the term "Brass" being the more common. A few of the widely used brass alloys are

Formula	Description
1Sn:28Zn:71Cu	Admiralty brass. Developed for the British Admiralty
70Cu:30Zn	Cartridge brass. Original brass composition

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65Cu:1Pb:33Zn	Clock brass. Easily machined for watches, engravings
61.5Cu:3Pb:35.5Zn	Free-cutting. Similar to Yellow, Cartridge or Spring
65Cu:30Zn	Yellow brass. Most widely used general brass

There are hundreds of zinc containing alloy products which, in quantity, rank fourth behind iron/steel, copper, and aluminum, not only for industrial applications, but in the jewelry trade and as objets d'art. This includes powdered brass in plastic as a spray coating replacement for gold, similar to the use of aluminum as a replacement for silver.

Technical Application: Zinc alloys, usually as some form of brass, have many applications in the processing of parts and equipment associated with the fabrication of Solid State devices and assemblies, such as contact pins, heat sinks, test blocks, package parts, tube housing in addition to screws, shim stock masks, springs, and similar items to name only a few such products. Zinc can be an unwanted element in the fabricating of certain semiconductor devices as it can affect electrical characteristics due to its ability to diffuse under thermal or electrical load, yet it is a p-type dopant element in some compound semiconductors, and is a primary constituent in others, such as zinc selinide, ZnSe and zinc telluride, ZnTe. The two latter compounds are semiconductors even though they could be classified as zinc alloys.

Zinc alloys have been the subject of growth studies and general morphological study, to include single crystallite evaluations. Both ZnSe and ZnTe have been studied as single crystals and are listed under their own headings.

Etching: Varies with alloy. Soluble in most acids and alkalies.

ZINC ALLOYS

ZINC TIN ETCHANTS

ZNSN-0001
ETCH NAME: Nital
TYPE: Acid, preferential
COMPOSITION:
10 ml .... HNO_3
1000 ml .... EOH
TIME:
TEMP: RT
ASTM: #74
DISCUSSION:

Zn:Sn, alloys processed in a study of solidification in lamellar eutectic systems. Evaluation specimens were quenched and others used as as-grown controls. A 10% Nital solution was used to develop structure. Also studied PbSn and CdZn.

REF: Tiller, W A & Mrdjenovich, R M — *J Appl Phys*, 34,3639(1963)

ZNSN-0002: ASTM E407-70 1947

Reference for ASTM Number 74. Nital shown as 1—5:HNO_3:100 ml EOH/MeOH.

ZINC-CADMIUM ETCHANTS

ZNCD-0001
ETCH NAME: Nital
TYPE: Acid, preferential
COMPOSITION:
10 ml .... HNO_3
100 ml .... EOH
TIME:
TEMP: RT
ASTM: #74
DISCUSSION:

Zn:Cd, alloys processed in a study of solidification in lamellar eutectic systems. Eval-

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uation specimens were quenched and others as-grown for control. A 10% Nital solution was used to develop structure. Also studied PbSn and ZnSn.
REF: Tiller, W A & Mrdjenovich, R M — *J Appl Phys*, 34,3639(1963)
ZNCD-0002: ASTM E407-70
Reference for ASTM Number 74. Nital shown as 1—5 ml HNO_3:100 ml EOH/MeOH.

ZINC MERCURY ETCHANTS

ZNMG-0001
ETCH NAME: Acid, cut — TIME:
COMPOSITION: — TEMP:
1 .... HNO_3
1 .... H_2O_2
1 .... alcohol
DISCUSSION:
ZnHg specimens wire saw cut using the solution shown in a study of crack initiation in this compound. After cutting wash in water, dry in alcohol and then in ether. (*Note:* This is an amalgam. See section on Amalgam.)
REF: Westwood, A R C — *Phil Mag*, 9,199(1964)

SILVER ZINC ETCHANTS (See Silver Alloys)

AGZN-0002
ETCH NAME: — TIME:
TYPE: Acid, cleaning — TEMP:
COMPOSITION:
1 .... 10% KCN
1 .... 10% $(NH_4)_2SO_4$
DISCUSSION:
AgZn specimens as alpha structure used in a study of temperature dependence of magnetic susceptibility. Solution used to clean surfaces.
REF: Myers, L & Weiner, D — *Phys Rev*, 108,1426(1957)

PHYSICAL PROPERTIES OF ZINC ARSENIDE, Zn_3As_2

Classification	Arsenide
Atomic numbers	30 & 33
Atomic weight (°C)	346
Melting point (°C)	1015
Boiling point (°C)	
Density (g/cm³)	4—5
Refractive index (n=)	1.66—1.72
Hardness (Mohs — scratch)	2.5—3
Crystal structure (tetragonal — normal) Zn_3As_2	(100) prism, 2nd order
(monoclinic — normal) $ZnAs_2$	(100) a-pinacoid
Color (solid)	Grey metal
Cleavage (b-pinacoid — perfect)	(010)

ZINC ARSENIDE, Zn_3As_2

General: The pure binary alloy does not occur as a natural compound, but there are several arsenate minerals, such as barthite, chlorophoenivite, holdenite, and kottigite (re-

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fractive index shown, above) as hydrates containing zinc with manganese or copper. They are all minor in occurrence and associated with other copper, cobalt, and zinc vein ores. As arsenates or an arsenide there has been little or no use in industry. The compounds can be used as coloring pigments in glass and ceramic manufacture.

Technical Application: Little use in Solid State material fabrication to date, although the single crystals have been evaluated as binary- and trinary-type semiconductors of tetrahedral unit cell type.

Etching: HNO_3, H_2SO_4 and alkalies.

ZINC ARSENIDE ETCHANTS

ZNAS-0001
ETCH NAME: Heat — TIME:
TYPE: Thermal, preferential — TEMP:
COMPOSITION:
x .... heat
DISCUSSION:
Zn_3As_2, single crystal sphere used in a study of lattice parameters. Heating of sphere develops crystallographic facets.
REF: Cole, H et al — *IBM R&D*, 1,90(1957)

ZINC TIN ARSENIDE ETCHANTS

ZSAS-0001
ETCH NAME: Nitric acid — TIME:
TYPE: Acid, removal — TEMP:
COMPOSITION:
(1) x .... HNO_3 (2) x .... x% KOH (NaOH)
DISCUSSION:
$ZnSnAs_2$, single crystals used in an evaluation of cohesive energy features of tetrahedral semiconductors. Forty materials were studied.
REF: Aresti, A et al — *J Phys Chem Solids*, 45,361(1984)

PHYSICAL PROPERTIES OF ZINC FLUORIDE, ZnF_2

Classification	Fluoride
Atomic numbers	30 & 9
Atomic weight	103.38
Melting point (°C)	872
Boiling point (°C)	
Density (g/cm³)	4.84 nom.
Hardness (Mohs — scratch)	2—3
Crystal structure (monoclinic — normal)	(100) a-pinacoid
Color (solid)	Yellow/brown
Cleavage (basal)	(001)

ZINC FLUORIDE, ZnF_2

General: Does not occur as a natural compound. Like other fluorides, such as fluorite, CaF_2, it can be used as a solder flux in metal alloy assembly, but zinc compounds have wider use as the pure metal or as the oxide. Pure zinc is dip plated as galvanized surface coatings on iron as it stabilizes readily with a greyish zinc oxide; as heated zinc dust called

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sheradized iron. It has numerous uses as an alloy: brass and bronze, German silver (Zn:Ni:Cu), etc. Zinc dust can ignite explosively in air.

Technical Application: Zinc fluoride has no major use in Solid State processing, although it has shown semiconducting properties as a doped fluoride, $ZnSiF_2$. It has been grown and studied as the pure single crystal ZnF_2 and as the trinary compound.

Etching: Water, hot acids, and NH_4OH.

ZINC SILICON FLUORIDE ETCHANTS

ZSIF-0001
ETCH NAME: TIME:
TYPE: Acid, removal TEMP: Hot
COMPOSITION:
x .... NH_4OH
DISCUSSION:

$ZnSiF_2$, single crystals used to evaluate cohesive energy features of tetrahedral semiconductors. Forty materials studied.

REF: Aresti, A et al — *J Phys Chem Solids*, 40,361(1980)

PHYSICAL PROPERTIES OF ZINC OXIDE, ZnO

Classification	Oxide
Atomic numbers	30 & 8
Atomic weight	81.38
Melting point (°C)	>1800
Boiling point (°C)	
Density (g/cm^3)	5.47—5.7
Refractive index (n=)	2.01—2.03
Hardness (Mohs — scratch)	4—4.5
Crystal structure (hexagonal — hemimorphic)	xtls rare
Color (solid)	Deep red
Cleavage (basal — perfect)	(0001)

ZINC OXIDE, ZnO

General: Occurs as the natural mineral zincite, ZnO, known as red oxide of zinc and an ore of zinc. It is found in limited areas, but can be in massive deposits. For an oxide it is very heavy, and can be characterized by its deep red color and an orange-yellow streak, e.g., most oxides produce a white streak when drawn across an alumina blank. It often occurs as an artificial furnace by-product in the reduction of other zinc ores, notably, the sulfides.

Other than as an ore of zinc in industry, a primary use is as a high quality pigment in paints with addition applications in glass and ceramics.

Technical Application: The oxide is not used in Solid State processing as a semiconductor material, although it exhibits the polar characteristics of binary semiconductors on the basal (0001) surfaces.

There are possible applications for its electroluminescent properties, and has been grown as an artificial single crystal for general structural study as both the artificial compound and natural single crystal. It has also been developed as a thin film structure by the oxidation of zinc selenide, ZnSe, wafer surfaces.

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Etching: The artificial compound is soluble in mineral acids, acetic acid, and a sodium hydroxide. The natural mineral is soluble in most single acids, alkalies and ammonium chloride. Both are soluble in mixed acids.

ZINC OXIDE ETCHANTS

ZNO-0001a
ETCH NAME: Nitric acid, dilute — TIME: 90 sec
TYPE: Acid, preferential — TEMP: RT
COMPOSITION:
x .... 20% HNO_3
DISCUSSION:
ZnO, (0001) wafers from natural zincite, and artificially grown from a PbF_2 molten flux and platelets from furnace residue. Solution produces hexagonal etch pits on the (0001) surface. The $(000\bar{1})$ surface is rough and etches more rapidly.
REF: Marino, A N & Hanneman, R E — *J Appl Phys,* 34,384(1963)

ZNO-0002
ETCH NAME: Perchloric acid, dilute — TIME:
TYPE: Acid, preferential — TEMP:
COMPOSITION:
x .... $HClO_4$
x .... H_2O
DISCUSSION:
ZnO, (0001) as platelets and grains. Solution will develop twinning structure. Also used to etch remove platelets and grains from the growth matrix.
REF: Santhanam, A T et al — *J Appl Phys,* 50,853(1979)

ZNO-0003
ETCH NAME: Phosphoric acid — TIME: 30—40 min
TYPE: Acid, polish — TEMP: RT
COMPOSITION:
x .... H_3PO_4
DISCUSSION:
ZnO, (0001) wafers. After cutting and mechanical lap polish this solution was used as a finish etch.
REF: Lagowski, J et al — *J Appl Phys,* 48,3566(1977)

ZNO-0004
ETCH NAME: CP4 — TIME:
TYPE: Acid, preferential — TEMP:
COMPOSITION:
3 ml ... HF
5 ml .... HN_3O_3
3 ml .... CH3COOH (HAc)
x .... Br_2
x = 10 drops/50 ml of solution
DISCUSSION:
ZnO, thin film deposit formed by oxidation of ZnSe. Etch will polish the (0001)Zn surface but is only rough on the $(000\bar{1})$O surface which etches more rapidly.
REF: Iwanaga, H — *Jpn J Appl Phys,* 22,1098(1983)

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ZNO-0001b
ETCH NAME: TIME:
TYPE: Acid, preferential TEMP:
COMPOSITION:
6 .... HNO_3
6 .... HAc
1 .... H_2O
DISCUSSION:
ZnO, (0001) and $(10\bar{1}0)$ wafers. Etch use to develop polar etch figures. Solution will produce triangular etch pits on the $(10\bar{1}0)$.
REF: Ibid.

ZNO-0006a
ETCH NAME: Nitric acid, dilute TIME: 90 sec
TYPE: Acid, preferential TEMP: RT
COMPOSITION:
x .... 20% HNO_3
DISCUSSION:
ZnO, single crystal wafers cleaved (0001). Etch is more rapid on (0001)A surface than $(000\bar{1})$B. The (0001)A surface develops hexagonal pits with flat bottoms; the $(000\bar{1})$B surface shows a blocky, angled textured surface similar to structure observed on germanium.
REF: Horn, F N — *J Appl Phys,* 32,900(1961)

ZNO-0006b
ETCH NAME: TIME:
TYPE: Acid, preferential TEMP:
COMPOSITION:
6 .... HNO_3, fuming
6 .... HAc
1 .... H_2O
DISCUSSION:
ZnO, $(10\bar{1}0)$ prism cut wafers. Solution produces polar etch figures. (*Note:* White fuming (standard) = 70%; yellow fuming = 72%; red fuming = 74%.)
REF: Ibid.

ZNO-0005
ETCH NAME: TIME:
TYPE: Acid, polish TEMP:
COMPOSITION:
10 .... HF
45 .... HNO_3
45 .... H_2O
DISCUSSION:
ZnO, single crystal specimens used in a study of deformation.
REF: Soo, P & Higgins, G T — *Acta Metall,* 16,177(1968)

ZNO-0007
ETCH NAME: Sodium hydroxide, dilute TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... 6 *N* NaOH
x .... ? (acids?)

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DISCUSSION:

ZnO specimens used in a study of neutron-transfer reactions from nitrogen ion bombardment. Solution used as a general removal and surface cleaning etch.
REF: Halbert, M L et al — *Phys Rev*, 106,251(1957)

ZNO-0009
ETCH NAME: Heat
TYPE: Thermal, treatment
TIME: 2 h
TEMP: 600°C
COMPOSITION:

x .... heat

DISCUSSION:

ZnO, single crystal material was diamond saw cut as wafers, and mechanically polished with 1 μm diamond paste. Annealed as shown in study of electroluminescence.
REF: Pike, G E et al — *J Appl Phys*, 57,5512(1985)

PHYSICAL PROPERTIES OF ZINC PHOSPHIDE, Zn_3P_2

Classification	Phosphide
Atomic numbers	30 & 15
Atomic weight	258.18
Melting point (°C)	>420
Boiling point (°C)	1100
Density (g/cm³)	4.55
Hardness (Mohs — scratch)	2—3
Crystal structure (isometric — normal)	(100) cube
(tetragonal — normal)	(100) prism, 2nd order
Color (solid)	Grey/black
Cleavage (cubic)	(001)

ZINC PHOSPHIDE, Zn_3P_2

General: Does not occur as the pure phosphide in nature. The mineral sencerite, $Zn_3(PO_4)_2.Zn(OH)_2.3H_2O$, is representative of one of several phosphates. As a binary phosphide, the artificial material has been used as an additive to iron, steel, copper, brass, and bronze, such as for phosphor bronze or other special alloys.

Technical Application: The material has been evaluated as a possible II—V binary compound semiconductor, and a trinary compound with silicon. It also has been grown as a polycrystalline thin film, all in general material studies.

Etching: H_2SO_4, HNO_3, and mixed acids. Can be violent in dilute acids.

ZINC PHOSPHIDE ETCHANTS

ZNP-0001
ETCH NAME:
TYPE: Acid, removal
TIME:
TEMP: RT
COMPOSITION:

1 .... HF
1 .... HNO_3

DISCUSSION:

Zn_3P_2, grown as single crystal and polycrystalline thin films in a study of the material. Silver used as a p-type dopant. Single crystal was tetragonal with lattice constants: a =

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8.05 and c = 11.45. Poly-type was cubic with a = 5.82. (*Note:* Solution shown is referenced from $ZnSiP_2$.)
REF: Chu, T L — *J Electrochem Soc,* 128,2063(1981)

ZINC SILICON PHOSPHIDE ETCHANTS

ZNSP-0001
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
1 .... HF
1 .... HNO_3
TIME: 1 h
TEMP: RT
DISCUSSION:
$ZnSiP_2$, (100) single crystal wafers. Etch cleaned in the solution shown in a material growth study. After etching bake at 1000°C.
REF: Nisida, H et al — *Jpn J Appl Phys,* 22(2),272(1983).

ZINC TIN PHOSPHIDE ETCHANTS

ZNTP-0001
ETCH NAME:
TYPE:
COMPOSITION:
x .... HF
x .... HNO_3
TIME:
TEMP:
DISCUSSION:
$ZnSnP_2$, thin films grown on GaAs, (100) substrates by LPE. Growth was perpendicular to (001) but 2°-off toward ⟨110⟩.
REF: Davis, G A et al — *J Cryst Growth,* 69,141(1984)

PHYSICAL PROPERTIES OF ZINC SELENIDE, ZnSe

Classification	Selenide
Atomic numbers	30 & 34
Atomic weight	144.34
Melting point (°C)	>1000
Boiling point (°C)	
Density (g/cm^3)	5.42
Refractive index (n =)	2.89
Hardness (Mohs — scratch)	5—6
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Grey/yellow
Cleavage (cubic — perfect)	(001)

ZINC SELENIDE, ZnSe

General: Does not occur as a natural compound, but has been grown as an artificial metallic single crystal. There are zinc oxides, arsenates, phosphates, and sulfates, but no selenides, although there are other metal selenides and tellurides in nature. There is no use of the zinc selenide in industry.

Technical Application: Zinc selenide is a II—VI type semiconductor material under eval-

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uation as a polar binary compound for its electroluminescent and general etching characteristics. It has been grown both as a single crystal and as a thin film.

Etching: Mixed acids and alkalies.

ZINC SELENIDE ETCHANTS

ZNSE-0002c
ETCH NAME: Sodium hydroxide — TIME:
TYPE: Alkali, cleaning — TEMP: Boiling
COMPOSITION:
x .... 25% NaOH
DISCUSSION:
ZnSe:S (111) (SI) wafers used as substrates for epitaxy growth of ZnSe. Solution used for surface cleaning prior to and after epitaxy.
REF: Ibid.

ZNSE-0005
ETCH NAME: Sodium hydroxide — TIME:
TYPE: Alkali, polish — TEMP: Hot
COMPOSITION:
x .... x & NaOH
DISCUSSION:
ZnSe, (100) wafers used in a study of the Gunn Effect. Specimens were polished in a hot sodium hydroxide solution (concentration not shown).
REF: Ludwig, G W & Aven, M — *J Appl Phys*, 38,5326(1967)

ZNSE-0002b
ETCH NAME: — TIME: 1 min
TYPE: Acid, cleaning — TEMP: 95°C
COMPOSITION:
40 .... H_2SO_4
60 .... $K_3Cr_2O_7$, sat. sol.
DISCUSSION:
ZnSe:S, (111) (SI) wafers used as substrates for epitaxy growth of ZnSe. After mechanical polishing, etch clean in the solution shown; then etch in boiling 25% NaOH. After epitaxy, clean surface with a light etch in 35% NaOH. (*Note:* Solution shown is similar to the standard glass cleaner etch.)
REF: Ibid.
ZNSE-0007b: Ibid.
ZnSe, single crystal wafers of various orientations used in an etch development study. Solution also used on CdS and ZnS.

ZNSE-00007a
ETCH NAME: — TIME: $2^1/_2$ min
TYPE: Acid, preferential — TEMP: RT
COMPOSITION:
x .... H_2SO_4
x .... 0.3 *M* $KMnO_4$

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DISCUSSION:
ZnSe, single crystal wafers of various orientations used in an etch development study. Solution used to develop dislocations and defects. Also used on CdS and ZnS.
REF: Rowe, J E & Forman, R A — *J Appl Phys*, 39,1917(1968)

ZNSE-0001
ETCH NAME: BRM, vapor
TYPE: Halogen, preferential
TIME:
TEMP: Hot
COMPOSITION:
(1) x .... Br_2
x .... MeOH
(2) x .... Br_2
x .... CS_2
DISCUSSION:
ZnSe, (111) wafers. Both solutions used to preferential etch specimens.
REF: Sethi, B R et al — *J Appl Phys*, 50,353(1979)
ZNSE-0009: Sethi, B R et al — *J Appl Phys*, 39,533(1968)
ZnSe, (111) wafers. Specimens used in a defect structure study.

ZNSE-0002a
ETCH NAME: Sodium hydroxide
TYPE: Alkali, preferential
TIME:
TEMP: Boiling
COMPOSITION:
x .... 50% NaOH
DISCUSSION:
ZnSe, single crystal platelets grown in a Zn-Ga molten flux. Solution used to etch remove specimens from the flux and to etch clean platelets after removal.
REF: Fujita, S et al — *J Appl Phys*, 50,1079(1979)

ZNSE-0003
ETCH NAME: Sodium hydroxide
TYPE: Alkali, dislocation
TIME: 3 min
TEMP: 110°C
COMPOSITION:
x .... 30% NaOH
DISCUSSION:
ZnSe, (110) wafers cleaved from (111) oriented ingots grown by the Bridgman process. Solution used to develop dislocations on cleaved surfaces. Two types of triangle pits observed: (1) Type I: isosceles triangles and (2) Type II: equilateral triangle, normal.
REF: Iwanaga, H et al — *J Cryst Growth*, 67,97(1984)

ZNSE-0004
ETCH NAME: Sodium hydroxide
TYPE: Alkali, preferential
TIME: 2 min
TEMP: 90°C
COMPOSITION:
x .... 14 *N* NaOH
DISCUSSION:
ZnSe, (100) wafers etched in this solution during a study of the electrical properties of the material.
REF: Satch, S et al — *Jpn J Appl Phys*, 22,1167(1983)

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ZNSE-0006
ETCH NAME: Hydrochloric acid — TIME:
TYPE: Acid, preferential — TEMP: RT
COMPOSITION:
x .... HCl
DISCUSSION:
ZnSe, (100) wafers used in a materials study. There is a 3C to 2H transformation at 1425°C to (00.1) hexagonal structure. Solution used to develop twinning and slip associated with ⟨111⟩ directions.
REF: Kikuuma I & Furkoghi, M — *J Cryst Growth*, 71,136(1985)

ZNSE-0008
ETCH NAME: — TIME:
TYPE: Acid, polish/clean — TEMP:
COMPOSITION:
5 .... H_2SO_4
1 .... H_2O_2
1 .... H_2O
DISCUSSION:
ZnSe, thin films epitaxially grown on GaAs:Cr, (100) (SI) substrates. Solution shown used to polish and clean substrates prior to epitaxy, follow with MeOH rinse and vacuum clean at 550°C in H_2, 10 min. Substrates at 250—350°C during epitaxy. ZnSe films showed hillocks in ⟨011⟩ direction.
REF: Fujitas, R et al — *J Cryst Growth*, 71,169(1985)

ZNSE-0009
ETCH NAME: — TIME: 1
TYPE: Acid, polish — TEMP: (1) 95°C (2) RT (3) 90°C
COMPOSITION:
(1) 2 .... H_2SO_4 (2) x .... CS_2 (3) x .... 14 *N* NaOH
3 .... $K_2Cr_2O_7$
DISCUSSION:
ZnSe, single crystal wafers, undoped or chlorine doped used in a study of photoluminescence and general properties. Etch clean and polish specimens in the solutions above and in the order shown.
REF: Satch, S & Igaki, K — *Jpn J Appl Phys*, 22,68(1984)

PHYSICAL PROPERTIES OF ZINC SULFIDE, ZnS

Classification	Sulfide
Atomic number	30 & 16
Atomic weight	97.4
Melting point (°C)	>800
Boiling point (°C)	
Density (g/cm³)	3.9—4.1
Refractive index (n =)	2.4—2.43
Optical energy gap (eV)	3.38
Hardness (Mohs — scratch)	3.5—4
Crystal structure (isometric — tetrahedral)	(111) tetrahedron (311) dodecahedron

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Color (solid)	Yellow/brown to black
Cleavage (dodecahedral — perfect)	(110)

ZINC SULFIDE, ZnS

General: Occurs in nature as the mineral sphalerite, ZnS (zincblende, blende, or black jack), as a major ore of zinc, and is closely associated with galena, PbS. Sphalerite is the stable form below 1020°C and is isometric system-tetrahedral class; whereas the mineral wurtzite, ZnS, hexagonal-hemimorphic, stable above 1020°C, and the oxide form (zincite, ZnO) has a similar structure to that of wurtzite. The Sphalerite Group of minerals, all with the same crystallographic structure, includes five mercury compounds: HgS, (Hg,Zn)S, HgSe, Hg(SSe), and HgTe. Several have also been artificially grown as semiconductor compounds as has been sphalerite. Sphalerite is the major ore of zinc and can contain valuable amounts of gold and silver.

Technical Application: Zinc sulfide is a II—VI compound semiconductor and is used in the fabrication of electroluminescent diodes and laser diodes both as a single crystal wafer and a deposited single crystal thin film. Like cadmium sulfide, CdS, it can be alternately and sequentially deposited as ZnS/SZn/ZnS.

In addition to its semiconductor properties it is of major interest in semiconductor development for the isometric-tetrahedral crystallographic structure as it has the atypical tetrahedron unit cell of many of the binary compound semiconductors, such as gallium arsenide, indium phosphide, and others. Both artificially grown and natural single crystals have been the subject of much study for general morphology, structure and defects.

Etching: Easily etched in HCl, H_2O_2 and mixed acids.

ZINC SULFIDE ETCHANTS

ZNS-0001a
ETCH NAME:
TYPE: Acid, preferential
TIME: 10 min
TEMP: RT or 95°C
COMPOSITION:
x .... 0.5 *M* $K_3Cr_2O_7$
x .... 16 *N* H_2SO_4
DISCUSSION:

ZnS, (111) wafers used in a dislocation and defect study and etch solution development. The material was both natural sphalerite and synthetic ZnS. The natural mineral developed triangular pits with a background of shallow triangular etch figures on the (111)Zn surfaces. The (111)S surfaces were irregular. Used on synthetic ZnS at RT does not develop the same etch patterns. Used on synthetic ZnS for 10 min at 95°C, produced a highly polished surface with some triangular etch pits on the (111)Zn and shallow, disc-like pits on the $(\overline{1}\overline{1}\overline{1})$S surface.

REF: Warekois, E P et al — *J Appl Phys*, 33,691(1962)

ZNS-0009b: Ibid.

ZnS, single crystal wafers used in an etch development study. Results were similar to ZNS-0009a. Solution used on CdS and ZnSe.

ZNS-0010: Scranton, R A — *J Appl Phys*, 50,842(1979)

ZnS, $(\overline{1}\overline{1}\overline{1})$ wafers. Solution used at 95°C, 10 min. Produces a highly polished surface with dislocations on (111)S surfaces; shallow disc-like structure on (111)Zn. Similar results obtained on CdS (CDS-0001b).

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ZNS-0002a
ETCH NAME: Potassium chromate
TYPE: Acid, dislocation
COMPOSITION:
x .... 0.5 *M* $K_2Cr_2O_7$
TIME: 10 min
TEMP: 95°C
DISCUSSION:
ZnS, (111) wafers. Solution will develop dislocation pits on the (111)A surface. Can be used on other orientations.
REF: Harper, C A — *Handbook of Materials and Processes for Electronics*, McGraw-Hill, New York, 1970, 7-5L

ZNS-0003
ETCH NAME:
TYPE: Acid, defect
COMPOSITION:
x .... H_2O_2
TIME: 10—60 min
TEMP: 60—80°C
DISCUSSION:
ZnS, (111) cleaved wafers, both artificial and natural crystals. Also phosphors of ZnS containing: (1) Cu, Al; (2) Cu, Al, Pb; (3) Cu, Cl-Mn, and (4) Cu, Cl. Hydrogen peroxide concentration was varied from $7^1/_2$ to 30%. Striations and some cleavage steps observed without etching, and etching will develop etch pits on both (111)A and $(\bar{1}\bar{1}\bar{1})$B surfaces. Used in a study of defects in electroluminescent ZnS.
REF: Goldberg, P — *J Appl Phys*, 32,1520(1961)
ZNS-0004: Froelich, H — *J Electrochem Soc*, 100,496(1953)
Reference for ZnS:Cu, Al phosphor.
ZNS-0005: Homer, H H et al — *J Electrochem Soc*, 100,566(1953)
Reference for ZnS:Cu,Cl-Pb and ZnS:Cu,Cl-Mn phosphors.

ZNS-0006
ETCH NAME: Nitric acid
TYPE: Acid, preferential
COMPOSITION:
x .... HNO_3
TIME: 2—5 min
TEMP: Boiling
DISCUSSION:
ZnS, (001) wafers used in an etch pit study. Pit structures are different between (001)A and $(00\bar{1})$B surfaces.
REF: Czyzak, S J & Reynolds, D C — *Bull Am Phys Soc*, 5,190(1960)

ZNS-0001b
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
1 .... HF
1 .... HNO_3
TIME: 8—10 sec
TEMP: RT
DISCUSSION:
ZnS, (100) wafers. The (100)A surface produces shallow disc-like pits. The $(\bar{1}00)$B shows dislocation pits.
REF: Ibid.

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ZNS-0002b
ETCH NAME:
TYPE: Acid, preferential
COMPOSITION:
1 .... HCl
1 .... HNO_3
DISCUSSION:
ZnS, (111) wafers. The (111)A surface develops triangular etch pits and etch figures. The $(\overline{1}\overline{1}\overline{1})$B shows some structure but is rough. Both surfaces are coated with a residual sulfur film.
REF: Ibid.
TIME:
TEMP:

ZNS-0007
ETCH NAME: Nitric acid
TYPE: Acid, removal
COMPOSITION:
x .... 6 *N* HNO_3
DISCUSSION:
Zn phosphors. Solution used as a general removal etch in studying the physical characteristics of the following phosphors: ZnS:CuCl:Pb; ZnS:Cu:Al; Zn(S,Se)Cu:Br, and ZnS:Cu:Cl:Mn.
REF: Goldberg, P & Faria, S — *J Electrochem Soc*, 107,521(1960)
TIME:
TEMP: 60—80°C

ZNS-0008
ETCH NAME: Hydrochloric acid
TYPE: Acid, removal
COMPOSITION:
x .... HCl, conc.
DISCUSSION:
Zn phosphors. Solution used for general removal in a study of the following phosphor: (ZnCd)(S,Se):Cu:Cl. It was observed that ball milling increased brightness and that the hydrochloric acid increased central grain luminescence.
REF: Remheller, A K — *J Electrochem Soc*, 107,8(1960)
TIME:
TEMP:

ZNS-0009a
ETCH NAME:
TYPE: Acid, preferential
COMPOSITION:
x .... H_2SO_4
x .... 0.3 *M* $KMnO_4$
DISCUSSION:
ZnS, single crystal wafers used in an etch development study. Solution also used on CdS and ZnSe.
REF: Rowe, J E & Forman, R A — *J Appl Phys*, 39,1917(1968)
TIME:
TEMP:

ZNS-0011
ETCH NAME: Potassium cyanide
TYPE: Acid, cleaning
COMPOSITION:
x .... 4% KCN
TIME:
TEMP:

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DISCUSSION:

ZnS, (0001) hexagonal wafers. A study of paramagnetic resonance absorption by manganese activation. Solution used to clean surfaces. (*Note:* Sphalerite is isometric system, tetragonal class under normal conditions.)

REF: Keller, S P et al — *Phys Rev*, 110,850(1958)

PHYSICAL PROPERTIES OF ZINC TELLURIDE, ZnTe

Classification	Telluride
Atomic number	30 & 52
Atomic weight	193
Melting point (°C)	1240
Boiling point (°C)	
Density (g/cm^3)	6.34
Refractive index (n =)	3.6
Hardness (Mohs — scratch)	6—7
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Grey/reddish
Cleavage (cubic — perfect)	(001)

ZINC TELLURIDE, ZnTe

General: Does not occur as a natural compound. There are over 50 known zinc containing minerals of which sphalerite, ZnS, willemite, Zn_2SiO_4 and smithsonite, $ZnCO_3$, are major ores, in addition to zincite, ZnO and Franklinite, (Fe,ZnMn) $O(Fe,Mn)_2O_3$. The telluride has had no use in the metal industry, to date.

Technical Application: Zinc telluride is a II—VI compound semiconductor with the usual negative and positive (111) orientation polarity of these binary compounds.

It has been grown by both the Bridgman and Czochralski methods as single crystals for evaluation and general study. As thin films it has been grown by CVD and from powder on glass substrates. Fabricated devices include both diodes and transitors.

Etching: Acid, alkalies, and mixed acids.

ZINC TELLURIDE ETCHANTS

ZNTE-0001a
ETCH NAME: Sodium hydroxide — TIME: 3 min
TYPE: Alkali, dislocation — TEMP: 110°C
COMPOSITION:

x .... 30% NaOH

DISCUSSION:

ZnTe,(110) wafers and ingots were both etched in this solution. Produced isosceles triangles on (110) faces — called Type III pits.

See ZNSE-0003

REF: Iawanaga, H et al — *J Cryst Growth*, 67,97(1984)

ZTE-0002a
ETCH NAME: Warekois' reagent — TIME: 2 min
TYPE: Acid, preferential — TEMP: RT
COMPOSITION:

3 .... HF
2 .... H_2O_2
1 .... H_2O

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DISCUSSION:

ZnTe, (111) wafers used in an etch development and dislocation study. The (111)Zn surface produced triangular etch pits. The ($\overline{111}$)Te surface was coated with a Te film but there were no observable etch figures.

REF: Warekois, E P et al — *J Appl Phys,* 39,690(1962)

ZTE-0001b: Iwanaga, H et al — *J Cryst Growth,* 67,97(1984)

ZnTe,(110) wafers and ingot specimens. Refers to using this solution.

ZTE-0003

ETCH NAME: Water — TIME:

TYPE: Acid, float-off — TEMP: RT

COMPOSITION:

x .... H_2O

DISCUSSION:

ZnTe, thin films deposited from ZnTe powder onto glass substrates at 550 K and 10^{-4} Pa for 120 nm thickness, then overcoated with 0.1—0.5 nm aluminum. For TEM study the thin film was float-off removed from the glass substrate by soaking in water. After removal, carbon replicas were made for morphological study.

REF: Patel, S M & Patel, N G — *Thin Solid Films,* 113,185(1984)

ZNTE-0004

ETCH NAME: Sodium hydroxide — TIME:

TYPE: Alkali, preferential — TEMP:

COMPOSITION:

x .... 20 *M* NaOH

DISCUSSION:

ZnTe, thin films homoepitaxially deposited on GaAs, InAs, CdTe, and GaSb substrates in atmospheres of H_2; H_2:I_2, and Ar. Solution used in a morphological study of the thin films grown under the different atmosphere conditions shown.

REF: Nishio, M — *Jpn J Appl Phys,* 22,1101(1983)

ZNTE-0005a

ETCH NAME: Nitric acid, dilute — TIME: 3 min

TYPE: Acid, preferential — TEMP: RT

COMPOSITION:

x .... 25% HNO_3

DISCUSSION:

ZnTe, (111) wafers used in a dislocation and defect study. The (111)Zn surfaces turns blackish in color with no observable etch figures. The ($\overline{111}$)Te surface develops triangular etch pits.

REF: Dillon, J A Jr — *J Appl Phys,* 33,668(1962)

ZNTE-0005b

ETCH NAME: Aqua regia — TIME: 1 min

TYPE: Acid, preferential — TEMP: RT

COMPOSITION:

3 .... HCl

1 .... HNO_3

DISCUSSION:

ZnTe, thin film deposits. Aqua regia produces similar etch patterns on both (111)Zn and ($\overline{111}$)Te surfaces. The author points out that ZnTe films should not be overexposed to aqua regia vapors or a Te film will form on surfaces.

REF: Ibid.

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ZNTE-0002b
ETCH NAME: TIME:
TYPE: Acid, preferential TEMP:
COMPOSITION:
1 .... HNO_3
1 .... HCl
DISCUSSION:
ZnTe, (111) wafers. The (111)Zn surface shows fine, deep triangular etch pits in arrays. The $(\overline{1}\overline{1}\overline{1})$Te produces a flat polished surface with shallow triangular etch figures and with extended etching time the surface polish improves.
REF: Ibid.

ZNTE-0008
ETCH NAME: Nitric acid TIME:
TYPE: Acid, cleaning TEMP:
COMPOSITION:
x .... HNO_3, conc.
DISCUSSION:
ZnTe, specimens cut as bars 0.04 × 0.03 × 0.250. Etch cleaned in concentrated nitric acid before high temperature conductivity studies in zinc vapors.
REF: Thomas, D G & Sadowski, E A — *J Phys Chem Solids*, 25,395(1964)

ZNTE-0009
ETCH NAME: TIME:
TYPE: Alkali, dislocation TEMP: Hot
COMPOSITION:
x .... x% NaOH
DISCUSSION:
ZnTe, (111) wafers used as substrates for epitaxy growth of CdS to form heterojunctions. Solution used to develop structure on ZnTe wafers. The (111)A developed triangular pits; the $(\overline{1}\overline{1}\overline{1})$B showed only erratic structure.
REF: Aven, M & Garwacki, W — *J Electrochem Soc*, 110,401(1963)

ZNTE-0002c
ETCH NAME: TIME: 8—10 sec
TYPE: Acid, polish TEMP: RT
COMPOSITION:
4 .... HNO_3
3 .... HCl
DISCUSSION:
ZnTe, (111) wafers. Solution will polish both surfaces. The Te film remaining can be removed with concentrated HCl.
REF: Ibid.

ZNTE-0002d
ETCH NAME: Hydrochloric acid TIME:
TYPE: Acid, removal TEMP: RT
COMPOSITION:
x .... HCl, conc.

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DISCUSSION:

ZnTe, (111) wafers. Solution used to remove Te films remaining on surfaces after etching in other solutions.

REF: Ibid.

ZNTE-0007

ETCH NAME: Thermal etch — TIME:

TYPE: Heat, preferential — TEMP: 600°C

COMPOSITION:

x .... Ar(?)

DISCUSSION:

ZnTe, (111) wafers. Wafers were heated in an inert atmosphere. Heating develops etch pits and decorates dislocations. Dislocations precipitate at random Te spots, and heating can cause super-saturation of Zn with Te segregates. Slow cooling will allow re-indiffusion of Te.

REF: Lynch, R T et al — *J Appl Phys*, 34,706(1963)

ZNTE-0010

ETCH NAME: — TIME:

TYPE: Acid, removal/polish — TEMP:

COMPOSITION:

4 .... HF

3 .... HNO_3

DISCUSSION:

ZnTe, (111) wafers. Shown as a general removal and polish etch for this material. Etch solution leaves a dark Te film on surfaces that can be removed with a dip in HCl, then DI water wash.

REF: Sagar, A et al — *J Appl Phys*, 39,5336(1968)

ZNTE-0002e: Ibid.

ZnTe, (111) wafers. Solution used as a general polish etch.

PHYSICAL PROPERTIES OF ZINC TUNGSTITE, ZnW

Classification	Tungstite
Atomic number	30 & 74
Atomic weight	249.2
Melting point (°C)	
Boiling point (°C)	
Density (g/cm^3)	
Hardness (Mohs — scratch)	7—8
Crystal structure (isometric — normal)	(100) cube
Color (solid)	Grey
Cleavage (cubic)	(001)

ZINC TUNGSTITE, ZnW

General: Does not occur as a natural metallic compound. There are tungstate minerals with the WO_4^- radical, and one sulfate. There is some use in industry as a high temperature alloy.

Technical Application: Zinc tungstite has been grown as a single crystal for defect and crystallographic study.

Etching: Acids and alkalies.

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ZINC TUNGSTITE ETCHANTS

ZNW-0001a
ETCH NAME: Phosphoric acid
TYPE: Acid, polish
COMPOSITION:
x .... H_3PO_4

TIME: 15 min
TEMP: 400—450°C

DISCUSSION:
ZnW, (001) cleaved wafers. Mechanically polish: (1) with 305 SiC abrasive; (2) $5^1/_2$-grit diamond in kerosene and (3) Linde B alumina in KOH. Then etch polish in the solution shown. Material used in a study of growth, defects, and crystallography.
REF: O'Hara, S — *J Appl Phys*, 35,1312(1964)

ZNW-0001b
ETCH NAME:
TYPE: Alkali, preferential
COMPOSITION:
(1) 1 .... KOH
1 .... NaOH
(2) x .... 4 *M* KOH/NaOH

TIME: 4—7 min
TEMP: Boiling

DISCUSSION:
ZnW, (001) cleaved wafers. Solution used to develop dislocation etch pits. Pits were the normal square outline with a common directional orientation in ⟨110⟩.
REF: Ibid.

PHYSICAL PROPERTIES OF ZIRCONIUM, Zr

Classification	Transition metal
Atomic number	40
Atomic weight	91.22
Melting point (°C)	1852
Boiling point (°C)	3580 (3578)
Density (g/cm^3)	6.5 (6.44)
Thermal conductance (cal/sec)(cm^2)(°C/cm)	0.0505
Specific heat (cal/g) 25°C	0.066
Latent heat of fusion (cal/g)	60.3
Heat of fusion (k-cal/g-atom)	3.74
Heat of vaporization (k-cal/g-atom)	120
Atomic volume (W/D)	14.1
1st ionization energy (K-cal/g-mole)	160
1st ionization potential (eV)	6.95
Electronegativity (Pauling's)	1.4
Covalent radius (angstroms)	1.45
Ionic radius (angstroms)	0.79 (Zr^{+4})
Coefficient of linear thermal expansion ($\times 10^{-6}$ cm/cm/°C)	5.78 (5.85)
Electrical resistivity (micro-ohms-cm)	41.4
Electron work function (eV)	4.33
Compressibility (cm^2/kg $\times$ 10^{-6})	11.77
Magnetic susceptibility (emu/g-atom $\times$ 10^6)	119
Poisson ratio	0.34
Young's modulus (kg/cm^2 $\times$ 10^{-4})	0.939

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Grueneisen constant	128,900
Hall coefficient (V-cm/A-Os × 10^{12})	0.18
Cross section (barns)	0.18
Yield strength (1000 psi)	35—45
Hardness (Mohs — scratch)	2—3
(Vicker's — kg/mm^2)	110
(Rockwell, B) annealed	75—85
Crystal structure (hexagonal — normal) alpha <865°C	(10$\bar{1}$0) prism, hcp
(isometric — normal) beta >865°C	(100) cube, fcc
Color (solid)	Silver-white/grey
Cleavage (basal/cubic)	(0001)/(001)

ZIRCONIUM, Zr

General: Does not occur as a native element. The major ore is zircon, $ZrSiO_4$, which is found widely as an accessory mineral in igneous rocks, particularly from acid magmas with large crystals occurring in pegmatites. As the variety hyacinth, it is orange-reddish to brown and transparent, and a gem stone. The artificial zircon as a blue stone is called the gem stone starlite. Both the natural and artificial clear and transparent varieties are the gem stone called zirconia, often a substitute for diamond and, when tinted yellow, a substitute for gem quality topaz. As a metallic element it is in the titanium family and its oxides can be either basic or acidic.

In industry the pure metal has some use as a metal alloy although, as ferro-zirconium, it is of more importance as a desulfurizer or deoxidizer in steels. It has wider use as an oxide, such as for gas mantles, as an opacifier in paints like titanium and zinc oxides, as a filler in lacquers, and as an insulator. The oxide also is used as an abrasive for lapping and polishing.

Technical Application: None as the pure metal in Solid State processing, although the oxide has been used as a lapping abrasive.

Zirconium has been grown as a single crystal for general studies though much of the work has been with crystalline blanks, rods and other forms . . . defect, oxidation, copper alloying, deformation, and superconducting studies. It has also been grown as a thin film amorphous and single crystal nitride and is under evaluation as a silicide for use as a buffer layer in semiconductor heterostructures. It has been fabricated as a carbide, ZrC and boride, ZrB for use as an insulator.

Etching: HF, aqua regia slowly in other single acids. Acid mixtures.

ZIRCONIUM ETCHANTS

ZR-0001a
ETCH NAME:
TYPE: Acid, removal
TIME: 1 min
TEMP: RT
COMPOSITION:
8 .... HF
50 .... HNO_3
50 .... H_2O
DISCUSSION:

Zr specimens. Solution used to clean and polish specimens in a corrosion and oxidation study. Anodic oxide grown in H_3BO_3 as a saturated solution, RT, 500 V, and a 30 A°/v^{-1} rate.

REF: Misch, R D & Gunzel, F H Jr — *J Electrochem Soc,* 106,14(1959)

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ZR-0001b
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
1 .... HF
1 .... H_2O_2
20 .... H_2O
DISCUSSION:
Zr specimens. See discussion under ZR-0001a.
REF: Ibid.

ZR-0002
ETCH NAME: TIME: x sec
TYPE: Acid, removal/cleaning TEMP: RT
COMPOSITION:
10 ml .... HF
45 ml .... HNO_3
45 ml .... H_2O_2
DISCUSSION:
Zr, polycrystalline sheet. Discs were spark cut from sheet material and mechanically polished with 1 μm diamond paste. Solution used to etch clean surfaces. Also used on ZrN (ZRN-0001).
REF: Dawson, P T — *J Vac Sci Technol*, 21,36(1982)
ZR-0003b: Ibid.
Zr and Zr alloy specimens used in a study of oxidation. Solution used as polishing etch.

ZR-0003a
ETCH NAME: TIME:
TYPE: Acid, slurry polish TEMP: RT
COMPOSITION:
1 ml .... HF
0.5 ml .... HNO_3
98.5 ml .... H_2O
\+ .... Linde A abrasive
DISCUSSION:
Zr specimens. Solution used as an acid/slurry polish lap.
REF: Ports, N A et al — *J Electrochem Soc*, 107,506(1960)

ZR-0004a
ETCH NAME: TIME: 30 sec
TYPE: Acid, cleaning TEMP: RT
COMPOSITION:
x .... HF
x .... HNO_3
DISCUSSION:
Zr, polycrystalline rod etch cleaned in this solution prior to Cu:Sn(12% Sn) plating with Sn aiding in the formation of an Nb_3Sn coating that can then be diffused to form ZnNb(1% Nb). Zr rod electropolished prior to Cu:Sn plating. Specimens used in a superconductivity study.
REF: Bussiere, J F & Suenasa, M A — *J Appl Phys*, 47,707(1976)

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ZR-0004b
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
1 .... HF
4 .... HNO_3
2 .... H_2O
TIME: 15—60 sec
TEMP: RT
ANODE: Zr
CATHODE:
POWER: 10 V
DISCUSSION:
Zr, polycrystalline rod. Electropolishing in this solution will produce a satin finish.
REF: Ibid.

ZR-0005a
ETCH NAME:
TYPE: Acid, thinning
COMPOSITION:
5% .... $HClO_4$
95% .... H_2O
TIME:
TEMP:
ANODE:
CATHODE:
POWER: 50 V
DISCUSSION:
Zr, poly sheet used as substrates for copper evaporation and microstructure study after laser annealing. Sheet mechanically polished with 0.25 μm diamond paste. Degrease: boiling TCE; rinse acetone, then EOH. With parts in vacuum, Ar^+ ion sputter etch prior to copper evaporation. Use laser in air to form a 5000 Å deep layer of alloyed Zr/Cu. Etch thin for TEM study with the solution shown, then argon ion mill to remove oxide.
REF: Denbroeder, F J A et al — *Thin Solid Films,* 111,43(1984)

ZR-0005b
ETCH NAME:
TYPE: Electrolytic, thinning
COMPOSITION:
x .... HCl_3O_4
x .... CH_3COOH (HAc)
x .... ethylene glycol (EG)
TIME:
TEMP:
ANODE:
CATHODE:
POWER: 50 V
DISCUSSION:
Poly-Zr sheet used as substrates for copper evaporation. Mechanically polish sheet with $^1/_4$ μm diamond paste. Degrease in boiling TCE, then rinse in acetone, then MeOH. Ar^+ ion sputter etch in vacuum system before copper deposition. Use a laser in air to produce a Cu/Zn alloy, 5000 Å deep. Etch thin and/or remove zirconium substrate with the electrolytic etch shown. Argon ion milling used to remove residual oxygen left on surface from etching. Alloyed thin film used in TEM study.
REF: Ibid.

ZR-0009
ETCH NAME:
TYPE: Acid, removal
COMPOSITION:
50 ml .... HF
50 ml .... HNO_3
5 ml .... H_2O
TIME: 1 min
TEMP: RT

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DISCUSSION:

Zr specimens used in a study of the dissolution of oxide films. Anodize zirconium in 1% KOH at RT, $^1/_2$ h at 0—35 V with a Pt anode. Use etch shown to remove grown oxide.
REF: Misch, R D — *Acta Metall,* 5,178(1957)

ZR-0011
ETCH NAME:
TYPE: Acid, polish
COMPOSITION:
5 .... HF
50 .... HNO_3
45 .... H_2O
TIME: 2 min
TEMP: RT
DISCUSSION:

Zr specimens used in a study of oxygen diffusion and its relationship to oxidation and corrosion. Polish with solution shown before oxidizing. Form oxide by anodic oxidation in a 1% KOH solution with Pt wire cathode in 15 min steps from 5 to 95 V power. The oxide color changes with voltage used. (*Note:* Color change is due to variation of oxide thickness.)
REF: Pemsler, J P — *J Electrochem Soc,* 105,315(1958)

ZR-0006
ETCH NAME:
TYPE: Electrolytic, polish
COMPOSITION:
5 .... $HClO_4$
3 .... HAc
2 .... ethylene glycol (EG)
TIME:
TEMP: 5—10°C
ANODE: Zr
CATHODE:
POWER: 0.5—0.7 A/cm² @ 20 V
DISCUSSION:

Zr, (0001) and (10$\bar{1}$0) wafers used in a dislocation study. Solution shown used to electropolish surfaces. Use same solution for dislocation development at 5—8 V and 0.2—0.5 sec.
REF: Mills, D & Craig, G B — *J Electrochem Technol,* 4,300(1966)
ZR-0010a: Rappeport, E J — *Acta Metall,* 7,132(1959)

Zr specimens used in a study of room temperature deformation. An acetic acid:perchloric acid solution used to electropolish.

ZR-0007
ETCH NAME: Aqua regia
TYPE: Acid, removal
COMPOSITION:
3 .... HCl
1 .... HNO_3
TIME:
TEMP:
DISCUSSION:

Zr specimens. Shown as an etch for zirconium.
REF: Hodgman, C D — *Handbook of Chemistry and Physics,* 27th ed, Chemical Rubber Co., Cleveland, OH, 1943, 490

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ZR-0008
ETCH NAME: TIME: 1 min
TYPE: Acid, macro-etch TEMP: Hot
COMPOSITION:
8 ml .... HF
50 ml .... HNO_3
50 ml .... H_2O, tap (hot)
DISCUSSION:
Zr, polycrystalline blanks used in a study of anodic film growth of $ZrO_x.nH_2O$ and its effect on grain orientation. Anodize surfaces with boiling nitric acid. Use solution shown to develop microstructure.
REF: Misch, R D & Fisher, E S — *Acta Metall,* 4,222(1956)
ZR-0001c: Misch, R D & Gunzel, F H Jr — *J Electrochem Soc,* 106,15(1959)
Zr, polycrystalline blanks oxidized and studied for electrical resistance and corrosion. Use etch at RT, 1 min. Anodize in saturated H_3BO_3 with a Pt gauze cathode.

ZR-0021
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
1 .... HF
1 .... HNO_3
50 .... H_2O
DISCUSSION:
Zr, thin film evaporation metal in vacuum systems. Solution used to clean bell jars and internal fixtures. Can be used on steels, ceramics, and glass. Do not overetch glass as solution contains HF.
REF: Nichols, D R — *Solid State Technol,* December 1979.

ZR-0016
ETCH NAME: BHF TIME:
TYPE: Acid, polish TEMP:
COMPOSITION:
x .... HF
x .... NH_4F
DISCUSSION:
Zr, specimens used for electroplating. Polish specimens by immersion. (*Note:* A major article on electroplating of transition metals. Presents both polish etchants and plating solutions.)
REF: Saubestre, E B — *J Electrochem Soc,* 106,106(1959)
ZR-0010: Faust, C L & Beach, J G — *Plating,* 43,1134(1950)
Reference for solution shown in ZR-0009.
ZR-0017: Schickner, W C et al — *J Electrochem Soc,* 100,289(1953)
Zr specimens. Used as a general immersion polishing etch on zirconium.

ZR-0015
ETCH NAME: Heat TIME:
TYPE: Thermal, forming TEMP:
COMPOSITION:
x .... heat
DISCUSSION:
Zr, single crystal and polycrystalline spheres formed by splatter using an arc melter

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under an He atmosphere with flakes and chips in a water cooled copper pot. Other materials were Be, V, Fe, NiCu, and NiAl. Size was from 0.1 to 1.0 mm with 5 to 50% single crystal.
REF: Ray, A E & Smith, J F — *Acta Metall,* 11,310(1958)

ZR-0020
ETCH NAME: TIME: 30 sec
TYPE: Acid, polish TEMP: RT
COMPOSITION:
1 .... HF
10 .... HNO_3
DISCUSSION:
Zr, specimens used in a study of oxygen gradients in the oxidation of metals. Specimens were first polished with 4/0 emery, then give a cleaning polish in the solution shown.
REF: Pemsler, J P — *J Electrochem Soc,* 111,381(1964)

ZIRCONIUM ALLOYS

General: Does not occur as a metallic alloy. The most important industrial alloy is ferrozirconium used to desulfurize and deoxidize irons and steels.

Technical Application: Some single crystal zirconium alloys have been studied for their electronic characteristics, general morphology and structure.

Etching: HF:HNO_3 mixed acids ± H_2O_3, HAc, etc.

ZIRCONIUM NICKEL ETCHANTS

ZRNI-0001
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... HF
x .... HNO_3
DISCUSSION:
Zr_2Ni single crystal specimen used in a study of electronic phase transitions. Solution will etch material. Also studied NiTi.
REF: Lee, R N & Withers, R — *J Appl Phys,* 49,5488(1978)

ZIRCONIUM:VANADINITE ETCHANTS

ZRV-0001
ETCH NAME: TIME:
TYPE: Acid, removal TEMP:
COMPOSITION:
x .... HF
x .... HNO_3
DISCUSSION:
ZrV single crystal metallic alloy grown in a general study of these materials. Other materials studied were TiAl, TiNi, and Fe_3C. Referred to as metastable alloys. Metallic glasses studied were PdNiP, PtNiP, and PtCuP.
REF:

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PHYSICAL PROPERTIES OF ZIRCONIUM DIOXIDE, ZrO_2

Classification	Ceramic
Atomic numbers	40 & 8
Atomic weight	123
Melting point (°C)	2700
Boiling point (°C)	
Application limits (°C)	2400
Density (g/cm³)	5.5—6.0
Specific heat (°C) 25—1000°C	590
Coefficient thermal linear of expansion ($\times 10^{-6}$ cm/cm/°C) 25—800°C	7.5
Thermal conductivity (W/m °C) 1315°C	3
Electrical resistivity ($\times 10^6$ ohms cm) 385°C	1
($\times 10^2$ ohms cm) 1200°C	3.6
Refractive index (n=)	2.13—2.20
Hardness (Mohs — scratch)	6.5
Crystal structure (monoclinic — normal)	(100) a-pinacoid
(tetragonal — normal) >1000°C	(100) 2nd order prism
Color (solid)	Colorless/tinted
Cleavage (basal)	(001)

ZIRCONIUM DIOXIDE, ZrO_2

General: Occurs in nature as the minor mineral baddeleyite, ZrO_2. Usually found as small pebbles and grain associated with diamond sands, e.g., Brazil, where they are called favas (beans) and vary between pure ZrO_2 and pure TiO_2. The most important ore mineral is zircon, $ZrSiO_4$, and there are several other silicate minerals. It is a trace constituent in titanium minerals, which may contain hafnium oxide. The oxide is called zirconia and is the name for clear gem stone quality zircon, either natural or artificial. The natural gem stone most prized is a fine, transparent yellow similar to quartz citron.

As the oxide, there are several industrial applications, chiefly in the ceramics industry as a high temperature refractory dielectric, alone, or mixed with alumina, silica, or titania. Added to silica it produces a "hard" type glass that is tough and, added as a white powder, it has similar opacifier characteristics in enamels as a replacement for SnO_2. Zirconium also acts like a getter in vacuum systems for removing gases similar to titanium.

Technical Application: Under development and evaluation as a thin film dielectric surface coating on semiconductor wafers and other metallic compounds with similar applications to those of silicon dioxide or silicon nitride. As zirconium has a low neutron cross-section, both the metal and the oxide have application in nuclear reactor areas similar to those of lead.

Both the natural mineral and artificially grown single crystals, as well as thin films, have been the subject of general morphological, defect and electrical studies.

Etching: Soluble in HF, H_2SO_4, and mixed acids.

ZIRCONIUM OXIDE ETCHANTS

ZRO-0001a
ETCH NAME: Sulfuric acid
TYPE: Acid, removal
COMPOSITION:
x .... H_2SO_4

TIME:
TEMP: 60°C

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DISCUSSION:

ZrO_2, single crystal specimens. Material is monoclinic with a reversible inversion point at 1000°C to tetragonal. Solution used as a general etch for the material.

REF: Brochure: Magnesium Electron Inc (MEI), Farmington, NJ

ZRO-0004: Ben-Dor, L et al — *J Electron Mater*, 13,263(1984)

ZrO_2, thin film deposited by MOCVD on silicon, (111), degenerate n-type wafers. Prior to epitaxy growth, wafers were cleaned in (1) H_2SO_4:H_2O_2 and (2) HF:H_2O.

ZRO-0001b

ETCH NAME: Hydrofluoric acid — TIME:

TYPE: Acid, removal — TEMP:

COMPOSITION:

x .... HF

DISCUSSION:

ZrO_2, single crystal specimens. Solution used as a general etch.

REF: Ibid.

ZRO-0002

ETCH NAME: — TIME: 5 min

TYPE: Electrolytic, polish — TEMP: 10°C

COMPOSITION: — ANODE: ZrO_2

1 .... $HClO_4$ — CATHODE:

9 .... CH_3COOH(HAc) — POWER: 20 V

DISCUSSION:

ZrO, thin film deposits used in a study of gamma radiation effects on electrical parameters and corrosion in nuclear reactors. Solution was used to polish surfaces prior to radiation.

REF: Harrop, P J & Wanklyn, J N — *Br J Appl Phys*, 16,155(1965)

ZRO-0003

ETCH NAME: — TIME: (1)1 min

TYPE: Acid, polish/cleaning — (2)10—180 h

COMPOSITION: — TEMP: RT 44°C

(1) 4 .... HF — (2) x .... H_2SO_4

30 .... HNO_3

DISCUSSION:

ZrO_2, specimens with low hafnium content used in a study of corrosion kinetics in sulfuric acid solutions. Etch polish in solution (1) then soak in solution (2). Several concentrations of H_2SO_4 were evaluated.

REF: Smith, T — *J Electrochem Soc*, 107,82(1960)

PHYSICAL PROPERTIES OF ZIRCONIUM NITRIDE, ZrN

Classification	Nitride
Atomic numbers	40 & 7
Atomic weight	105
Melting point (°C)	>2800
Boiling point (°C)	
Density (g/cm^3)	
Hardness (Mohs — scratch)	6—7
Crystal structure (isometric — normal)	(100) cube

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Color (solid)	Brownish
Cleavage (cubic — perfect)	(001)

ZIRCONIUM NITRIDE, ZnN

General: Does not occur as a native compound. Metallic zirconium has been surface nitrided in industry for a thin skin more stable against corrosion.

Technical Application: Zirconium nitride is under evaluation in Solid State processing as a surface coating with similar characteristics and applications as those of silicon and aluminum nitrides, though it is not as stable against atmospheric effects of moisture in air.

Etching: HF:HNO_3 and other mixed acids.

ZIRCONIUM NITRIDE ETCHANTS

ZRN-0001
ETCH NAME:
ETCH: Acid, cleaning
TIME: Seconds
TEMP: RT
COMPOSITION:
10 ml .... HF
45 ml .... HNO_3
45 ml .... H_2O_2
DISCUSSION:
ZrN, thin films grown with NH_3 as the nitrogen source. Films were non-uniform in color. A dark grey center area (high O_2), and a bronze outer area (O_2 + C). Ar^+ ion sputtering of film removes the O_2 and C. Solution shown used to clean the thin films.
REF: Dawson, P T — *J Vac Sci Technol,* 21,36(1982)

ZRN-0002
ETCH NAME: Heat
TYPE: Thermal, cleaning
TIME:
TEMP: 1300°C
COMPOSITION:
x .... heat
DISCUSSION:
ZrN, (100) wafers cut from a polycrystalline Zr rod nitrided at 2800°C. This treatment forms large crystallites of cubic, ZrN (NaCl type). After zone anneal crystallite growth, specimens require flash cleaning with heat every 3 h to keep surfaces clean.
REF: Callenas, A et al — *J Physics Rev B,* 30,635(1984)

ZIRCONIUM PALLADIUM ETCHANTS

ZRPD-0001
ETCH NAME: Aqua regia
TYPE: Acid, removal/polish
TIME:
TEMP: RT
COMPOSITION:
3 .... HCl
1 .... HNO_3
DISCUSSION:
a-Zr_2Pd, thin films deposited as amorphous structure in a study of metal-metaloid alloys. Material is cubic, bcc, a-Zr_3Rh was cubic (E93 space group).
REF: Cantrall J S et al — *J Appl Phys,* 57,544(1985)

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ZIRCONIUM RHENIUM ETCHANTS

ZRRH-0001
ETCH NAME: Aqua regia
TYPE: Acid, removal/polish
COMPOSITION:
3 .... HCl
1 .... HNO_3
DISCUSSION:
a-Zr_3Rh, amorphous thin films deposited in a study of these metal-metaloid alloys. Crystals are cubic (E93 space group). Also deposited a-Zr_2Pd, which is cubic, bcc.
REF: Cantrall, J S et al — *J Appl Phys*, 57,544(1985)

TIME:
TEMP: RT

PHYSICAL PROPERTIES OF ZIRCONIUM SULFIDE, ZrS_2

Classification	Sulfide
Atomic numbers	40 & 16
Atomic weight	155.34
Melting point (°C)	>800
Boiling point (°C)	
Density (g/cm³)	3.87
Hardness (Mohs — scratch)	4—5
Crystal structure (hexagonal — normal)	$(10\bar{1}0)$ prism
Color (solid)	Steel-grey
Cleavage (basal-distinct)	(0001)

ZIRCONIUM SULFIDE, ZrS_2

General: Does not occur as a natural compound. There are about a dozen zirconium minerals; zircon, $ZiSiO_4$, is the most widespread and important as an ore.

The sulfide has no direct application in the metal industry, though the metal is added to irons and steels as a desulfurizer, e.g., gettering sulfur as ZrS_2. Natural zircon is an opalizer in enamels, and the fine yellow variety is a precious gem stone.

Technical Application: Zirconium sulfide is a IV—VI compound semiconductor with hexagonal structure similar to that of MoS_2 with distinctive basal (0001) cleavage like CdI and CdS. It has layered octahedral symmetry whereas MoS_2 has trigonal prismatic symmetry.

It is under evaluation as a semiconductor and, along with $ZrSe_2$, HfS_2, and $HfSe_2$, as a possible cathode storage battery.

Etching: Soluble in hot HBr.

ZIRCONIUM SULFIDE ETCHANTS

ZRS-0001
ETCH NAME: Hydrogen bromide
TYPE: Halogen, removal
COMPOSITION:
x .... x% HBr
DISCUSSION:
ZrS_2, single crystal specimens. Solution shown will etch the material.
REF:

TIME:
TEMP:

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A0-001 APPENDIX A: CRYSTALLOGRAPHY

Crystallography is the study of inorganic single crystal structures that have an internal ordered atomic lattice. The method was developed in geology for the classification of natural minerals, but is applicable to any solid material. In Solid State material processing of semiconductors and similar devices an understanding of the single crystal structure is vital in device design and fabrication, as well as in the development of new metallic compounds.

All single crystals can be classified into one of six crystal systems, and one of 32 classes within the systems. Note in the list below that the hexagonal system has two divisions, the rhombohedral division sometimes being classified as a seventh system. The six systems and their axial relationships are shown below in reducing order of complexity:

SYSTEM	AXIAL RELATIONSHIP
Isometric (cubic) system	Three mutually perpendicular axes of equal length. As a, b, c; x, y, z; x_1, x_2, x_3
Tetragonal system	Three mutually perpendicular axes. Two horizontal of equal length; one vertical, longer or shorter. As a, b, c or x, y, z
Hexagonal system	
Hexagonal division	Three horizontal axes at 60°; vertical axis at right angle. As x_1, x_2, x_3, z
Rhombohedral division	As above but with trigonal symmetry rather than vertical symmetry around the z-axis
Orthorhombic system	Three mutually perpendicular axes of unequal length. As x, y, z (a, b, c). y-axis longest and held parallel to observer (macro-axis); x-axis shortest and held facing observer (brachy-axis); z-axis vertical
Monoclinic system	Three axes of unequal length. Two at right angles; one oblique. As x, y, z. x-axis held facing observer and is oblique axis; y-axis as longest and held horizontal; z-axis shortest and held vertical
Triclinic system	Three axes of unequal length with all intersections oblique. As x, y, z (a, b, c). x-axis is shortest (brachy-axis) held downward toward observed; y-axis longer (macro-axis); z-axis held vertical. Angle between b/c = alpha; a/c = beta; a/b = delta, any can be less or greater than 90°

Fortunately, the majority of metal elements and metallic compounds form in the isometric system, normal/tetrahedral/pyritohedral class . . . most in the normal class. Although this is the most complex class of all systems, it is the simplest to understand due to axial relationships. As shown, the system may be referred to as the "cubic" system, based on the cube, (100) form that occurs only in the isometric system-normal class. Tin is an exception, as normal white-tin it is tetragonal.

There are two important compounds used as circuit substrates in Solid State assemblies that occur in the hexagonal system. Sapphire, Al_2O_3, in the hexagonal division — normal class, and alpha-quartz, SiO_2, in the rhombohedral division — trapezohedral class. The latter as left- or right-hand face rotated natural crystals often with penetration twinning. Single crystal quartz blanks are used as circuit substrates for high frequency microwave devices (silicon and gallium arsenide), and are the primary (quartz) radiofrequency crystals.

The atomic structure of several natural minerals are used as the atypical structure of many artificially compounded and grown single crystals, as binary, trinary, and quaternary

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crystals. Sphalerite, ZnS, for most of the binary compound semiconductors based on a tetrahedral unit cell; pyrite, chalcopyrite, and others for more complex trinary and quaternary atomic structures.

Other structural notations are space lattice forms of which there are 15 in number related to the normal class of each system. Number I is the simple cubic lattice with eight atoms at cube corners; II body-centered cubic lattice (bcc) with an additional atom at cube center; III face-centered cubic lattice (fcc) with additional atoms in the center of each cube face. The other systems have similar fcc lattices, and the hexagonal can have a hexagonal close-packed (hcp) natural crystal lattice. Several metals grown artificially under pressure may show similar close-packed lattices, such as the tetragonal (tcp) and hexagonal (hcp) high pressure structure of silicon.

A third system of notation is based on the crystal symmetry of the 32 classes as related to their systems. They are usually shown in increasing complexity from triclinic, monoclinic/orthorhombic, trigonal, tetragonal, hexagonal to cubic (isometric). Notation is a combination of letters, numbers, and other symbols, such as X3, X/m4/m, Xm6mm, etc. The complete list of notations is not shown here as the Miller Indices notations are more widely used in general processing of Solid State materials, and the space group notations used in crystallographic studies. See Appendix D for Miller Indices.

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APPENDIX B: MAJOR ELEMENTS AND RADICALS

The following lists all major metal elements with their group classification according to the Periodic Table of Elements for the relationship of compound semiconductors, such as III—V gallium arsenide, IV—VI germanium selenide, etc. Primary valence electron configuration also shown with some of the more important negative radical units, such as the sulfate SO_4^-. Note that only one silicate radical is shown; there are half a dozen other important radicals as primary rock and mineral-forming silicates.

Elements	Group	Valence	Radical	Valence
Aluminum, Al	III-A	3	Hydroxyl, OH	1
Antimony, Sb	V-A	3,5	Molybdate, MoO_4	1
			Nitrate, NO_3	1
Arsenic, As	V-A	3,5	Silicate, SiO_4	1,2
			Sulfate, SO_4	2
Barium, Ba	II-A	2	Phosphate, PO_4	3
Beryllium, Be	II-A	2	Carbonate, CO_3	2
Bismuth, Bi	V-A	3,5	Bicarbonate, HCO_3	1
Boron, B	III-A	3	Tungstate, WO_4	1
Bromine, Br	VII-A	1,3,5,7		
Calcium, Ca	II-A	2		
Carbon, C	IV-A	2,3,4		
Chlorine, Cl_2	VII-A	1,3,5,7		
Chromium, Cr	VI-B	2,3,6		
Cobalt, Co	VIII	2		
Copper, Cu	I-B	1,2		
Fluorine, F_2	VII-A	1		
Gallium, Ga	III-A	3		
Germanium, Ge	IV-A	4		
Gold, Au	I-B	1,3		
Hydrogen	I-A	1		
Iodine, I_2	VII-A	1,3,5,7		
Iron, Fe	VIII	2,3		
Lead, Pb	IV-A	2,4		
Lithium, Li	I-A	1		
Magnesium, Mg	II-A	2		
Manganese, Mn	VII-B	2,3,4,6,7		
Mercury, Hg	II-B	1,2		
Nitrogen, N_2	V-A	3,5		
Oxygen, O_2	VI-A	2		
Phosphorus, P	V-A	3,5		
Platinum, Pt	VIII	2,4		
Potassium, K	I-A	1		
Selenium, Se	VI-A	4,6,−2		
Silicon, Si	IV-A	4		
Silver, Ag	I-B	1		
Sodium, Na	I-A	1		
Sulfur, S	VI-A	2,4,6		

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Elements	Group	Valence
Tantalum, Ta	V-B	3,4,5
Titanium, Ti	IV-B	3,4
Tin, Sn	IV-A	2,4
Uranium, U	Actinide	4,6
Vanadium, V	V-B	3,4,5
Yttrium, Y	III-B	3
Zinc, Zn	II-B	2
Zirconium, Zr	IV-B	4

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APPENDIX C: METALS AND METALLIC COMPOUNDS WITH REFERENCE ACRONYMS

Material	Acronym
A	
Air, N_2/O_2	AIR-
Aluminum, Al	AL-
Aluminum alloys, AlMx	
Aluminum:beryllium, Al:Be	ALBE-
Aluminum:cerium, Al:Ce	ALCE-
Aluminum:copper, Al:Cu	ALCU-
Aluminum:gold, Au:Au	ALAU-
Aluminum:silicon, Al:Si	ALSI-
Aluminum:silver, Al:Ag	ALAG-
Aluminum:zinc, Al:Zn	ALZN-
Aluminum:zinc:copper, Al:Zn:Au	AZCU-
Aluminum antimonide, AlSb	ALSB-
Aluminum arsenide, AlAs	ALAS-
Aluminum nitride, AlN	ALN-
Aluminum oxide, Al_2O_3	ALO-
Aluminum phosphate, AlPO4	ALPH-
Aluminum phosphide, AlP	ALP-
Amalgam, $HgAg_x/ZnHg_x$	HGAG-/HGZN-
Antimony, Sb	SB-
Argon, Ar	AR-
Arsenic, As	AS-
B	
Barium, Ba	BA-
Barium fluoride, BaF_2	BAF-
Barium titanate, Ba_2TiO_3	BAT-
Beryllium, Be	BE-
Beryllium alloys, BeMx	
Beryllium:aluminum, Be:Al	BEAL-
Beryllium:copper, Be:Cu	BECU-
Beryllium oxide, BeO	BEO-
Bismuth, Bi	BI-
Bismuth alloys, BiM_x	
Bismuth:antimony, BiSb	BISB-
Bismuth:cadmium, BiCd	BICD-
Bismuth:tin, BiSn	BISN-
Bismuth germanate, $Bi_{14}Ge_3O_{12}$	BGO-
Bismuth selenide, Bi_2Se_3	BISE-
Bismuth silicate, $Bi_{12}SiO_{20}$	BSO-
Bismuth telluride, Bi_2Te_3	BITE-
Bismuth trioxide, Bi_2O_3	BIO-
Blister	BLIS-
Bonding	BB-
Boron, B	B-

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Borides, M_xB_y	
Cerium boride, CeB_6	CEB-
Chromium boride, Cr_3B_2	CRB-
Cobalt boride, CoB	COB-
Iron boride, FeB	FEB-
Lanthanum boride, LaB_6	LAB-
Molybdenum, Mo_2B_3	MOB-
Nickel boride, NiB	NIB-
Niobium boride, Nb_3B_3	NBB-
Rare earth borides, R_3B_2	REB-
Silicon boride, SiB_6	SIB-
Titanium boride, TiB_2	TIB-
Tungsten boride, WB_2	WB-
Uranium boride, UB_4	UB-
Vanadium boride, VB_2	VB-
Zirconium boride, ZrB	ZRB-
Boron carbide, B_4C	BC-
Boron nitride, BN	BN-
Boron phosphide, BP	BP-
Boron telluride, B_2Te_3	BTE-
Boron trifluoride, BF_3	BTF-
Brass, Cu:Zn	BRA-
Bromine, Br_2	BR-
Bronze, Cu:Sn	BRO-

C

Cadmium, Cd	CD-
Cadmium antimonide, CdSb	CDSB-
Cadmium arsenide, Cd_2As_2	CDAS-
Cadmium fluoride, CdF_2	CDF-
Cadmium indium selenide, $CdIn_2Se_4$	CISE-
Cadmium indium telluride, $CdIn_2Te_4$	CITE-
Cadmium iodide, CdI_2	CDI-
Cadmium molybdate, $CdMoO_3$	CDMO-
Cadmium oxide, CdO	CDO-
Cadmium phosphide, CdP_2	CDP-
Cadmium silicon arsenide, $CdSiAs_2$	CSA-
Cadmium selenide, CdSe	CDSE-
Cadmium sulfide, CdS	CDS-
Cadmium telluride, CdTe	CDTE-
Cadmium mercuric telluride, CdTe.HgTe	CDHT-
Calcium, Ca	CA-
Calcium carbonate, $CaCO_3$	CAC-
Calcite, $CaCO_3$	CAC-
Calcium fluoride, CaF_2	CAF-
Calcium molybdate, $CaMoO_2$	CAMO-
Calcium nitride, CaN	CAN-
Calcium silicon fluoride, $CaSiF_2$	CASF-
Calcium tin fluoride, $CaSnF_2$	CATF-
Calcium tungstate, $CaWO_4$	CAW-
Californium, Cf	CF-

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Carbon, C	C-
Graphite, C	GR-
Carbon dioxide, CO_2	COD-
Ceramics, general	CERA-
Cerium, Ce	CE-
Cerium dioxide, CeO_2	CEO-
Cermets, general	CMET-
Cesium, Cs	CS-
Cesium bromide, CsBr	CSBR-
Cesium chloride, CeCl	CECL-
Cesium iodide, CsI	CSI-
Cesium platinide, CePt	CEPT-
Carbides, M_xC	
Boron carbide, B_4C	BC-
Iron carbide, Fe_3C	FEC-
Molybdenum carbide, Mo_2C	MOC-
Niobium carbide, NbC	NBC-
Silicon carbide, SiC	SIC-
Tantalum carbide, TaC	TAC-
Titanium carbide, TiC	TIC-
Tungsten carbide, WC	WC-
Uranium carbide, UC	UC-
Vanadium carbide, VC	VC-
Chlorine, Cl_2	CL-
Chlorine containing etchants	
Chromium, Cr	CR-
Chromium containing etchants	
Chromium alloys, CrM_x	
Chromium titanium, Cr:Ti	CRTI-
Chromium trioxide, Cr_2O_3	CRO-
Cleaning, general	CLE-
Cobalt, Co	CO-
Cobalt oxide, CoO	COO-
Cobalt sulfide, Co_9S_8	COS-
Colemanite, $Ca_2B_6O_{11}.5H_2O$	COL-
Columbium, Cb	CB-
Copper, Cu	CU-
Copper alloys, CMx	
Copper antimonide, Cu_2Sb	CUSB-
Copper:beryllium, Cu:Be	CUBE-
Copper cerium, $CeCu_6$	CUCE-
Copper lanthanum, $LaCu_6$	CULA-
Copper:gallium, Cu:Ga	CUGA-
Copper:germanium, Cu:Ge	CUGE-
Copper gold, Cu_2Au	CUAU-
Copper nickel, CuNi	CUNI-
Copper palladium, CuPd	CUPD-
Copper bromide, $CuBr_2$	CUBR-
Copper chloride, $CuCl_2$	CUCL-
Copper iodide, CuI	CUI-
Copper oxide, Cu_2O	CUO
Cuprite, Cu_2O	CUO-

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Copper phosphide, Cu_3P	CUP-
Copper germanium phosphide, $CuGe_2P_3$	CUGS-
Copper selenide, Cu_2Se	CUSE-
Copper indium selenide, $CuInSe_2$	CISE-
Copper sulfide, CuS	CUS-
Copper gallium sulfide, $CuGaS_2$	CUGS-
Copper indium sulfide, $CuInS_2$	CIS-
Copper phospho-sulfuric iodide, CuPSI	CPS-
Copper telluride, Cu_4Te_3	CUTE-
Copper gallium telluride, $CuGaTe_2$	CGTE-
Copper indium telluride, $CuInTe_2$	CUIT-
Corundum, Al_2O_3	COR-
Aluminum oxide, Al_2O_3	ALO-
Cronstedite, $4FeO.2Fe_2O_3.3SiO_2.4H_2O$	CRON-
Cristobalite, SiO_2	CRIS-

D

Deuterium D	DEU- (TID-; SCD-)
Diamond, C	D-
Dielectric, M_xO or M_xN	DIE-
Dysprosium, Dy	DY-

E

Electrides, $C_xH_yO_zM$	ETD
Epoxy, general	EPAG- (EPAL-, EPAL-)
Erbium, Er	ER-
Erbium dihydride, ErH_2	ERH-
Etching, general	
Dry chemical etching, DCE	DCE-
Electrolytic etching, EE	EE-
Gas etching, GE	GE-
Molten flux etching, MFE	MFE-
Pressure etching, PRE	PRE-
Thermal etching, THE	THE-
Wet chemical etching, WCE	WCE-
Vacuum etching, VE	VE-
Europium, Eu	EU-
Europium oxide, Eu_2O_3	EUO-
Europium sulfide, EuS	EUS-

F

Ferric oxides, FeOx	FEO-
Limonite, $2Fe_2O_3.3H_2O$	LIM
Hematite, Fe_2O_3	HEM-
Magnetite, Fe_3O_4	MAGT-
Ferrites, $FeM_xM_yO_z$	FER-
Fluorapatite, $(CaF)Ca_4(PO_4)_3$	FAP-
Fluorine, F_2	F-
Fluorine-containing etchants	

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Fluorite, CaF_2	CAF-
Fresnoite, $Ba_2Si_2TiO_8$	FRE-

G

Gadolinium, Gd	GD-
Gadolinium nitride, GdN_{12}	GDN-
Gadolinium terbium iron, GdTbFe	GDTF-
Gallium, Ga	GA-
Gallium alloys, GaM_x	
Gold gallium, Au_2Ga	GAAU-
Gallium antimonide, GaSb	GASB-
Gallium arsenide, GaAs	GAS
Gallium aluminum arsenide, (Ga,Al)As	GALA-
Gallium arsenide phosphide, GaAsP	GASP-
Gallium iron oxide, $GaFeO_3$	GIO-
Gallium nitride, GaN	GAN-
Gallium oxide, Ga_2O_3	GAO-
Gallium phosphate, $GaPO_4$	GAPH-
Gallium phosphide, GaP	GAP-
Gallium selenide, GaSe	GASE-
Garnets, $M_3M_2(SiO_4)_3$	
Boron germanium, $B_4G_e3O_{12}$	BGG-
Calcium aluminum germanium, $Ca_3Al_2Ge_3O_{12}$	CAGG-
Europium scandium iron, $Eu_3Sc_2Fe_3O_{12}$	ESG-
Gadolinium gallium, $Gd_3Ga_5O_{12}$	GGG-
Gadolinium selenium gallium, $Gd_3Se_{1\ 8}Ga_{3\ 2}O_{12}$	GSGG-
Magnesium zinc yttrium, $Mn_xZn_{1-x}Y_zO_{12}$	MZYG-
Natural garnets, $Fe_2Al_2Si_3O_{12}$	GAR-
Strontium gallium, $SrCa_{12}O_{19}$	SGG-
Yttrium aluminum, $Y_3Al_5O_{12}$	YAG-
Yttrium gallium, $Y_2Ga_7O_{12}$	YGG-
Yttrium iron, $Y_3Fe_5O_{12}$	YIG-
Germanides, M_xGe	
Copper germanide, CuGe	CUGE-
Holmium copper germanide, $HoCu_2Ge_2$	HOCG-
Indium germanide, InGe	INGE-
Iron germanide, Fe_3Ge_2	FEGE-
Magnesium germanide, Mg_2Ge	MGGE-
Molybdenum germanide, Mo_3Ge	MOGE-
Niobium germanide, Ng_3Ge	NBGE-
Terbium copper germanide, $TbCu_2Ge_2$	TBCG-
Vanadium germanide, V_3Ge	VGE-
Germanium, Ge	GE-
Germanium alloys, M_xGe	
Gold:germanium, Au:Ge	AUGE-
Tin:germanium, Sn:Ge	SNGE-
Germanium arsenide, GeAs	GEAS
Germanium oxide, GeO_2	GEO-
Germanium nitride, Ge_3N_4	GEN-
Germanium selenide, Ge_xSe_{1-x}	GESE-
Germanium silicon, GeSi	GESI-

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Germanium sulfide, GeS	GES-
Germanium telluride, GeTe	GETE-
Glass, SiO_2 (under Silicon Dioxide)	GLA-
Gold, Au	AU-
Gold alloys, AuM_x	
Gold aluminum, Au_2Al	AUAL-
Gold bismuth, Au_2Bi	AUBI-
Gold cadmium, Au:Cd	AUCD-
Gold gallium, $AuGa_2$	AUGA-
Gold germanium, AuGe(13%)	AUGE-
Gold indium, Au:In	AUIN-
Gold mercury, $AuHg_x$ (amalgam)	AUHG-
Gold silver, $AuAg_x$ (electrum)	AUAG-
Gold tin, AuSn(20%)	AUSN-
Gold zinc, Au:Zn	AUZN-
Gold boron, AuB(2—5%)	AUB-
Gold copper, Cu_2Au	CUAU-
Gold palladium, Pd:Au	AUPD-
Gold phosphorus, Au:P(2—5%)	AUP-
Graphite, C	GR-
Carbon, C	C-
Diamond, C	D-

H

Hafnium, Hf	HF-
Hafnium alloys, HfM_x	
Hafnium tin, Hf_5Sn_3	HFSN-
Hafnium nitride, HfN	HFN-
Helium, He	HE-
Hematite, Fe_2O_3	HEM-
Holmium, Ho	HO-
Holmium alloys, HoM_x	
Holmium cobalt, Ho:Co	HOCO-
Holmium nitride, HoN_{12}	HON-
Hydrogen, H_2	H-
Hydrides, M_xH	HYDR-
Chromium hydride, CrH	CRH-
Germanium hydride, Ge:H	GEH-
Niobium hydride, Nb:H	NBH-
Silicon hydride, Si:H	SIH-
Silicon nitride hydride, SiN:H	SINH-
Silicon oxynitride hydride, SiON:H	SIONH-
Silicon carbide hydride, SiC:H	SICH-
Tantalum hydride, Ta:H	TAH-
Titanium hydride, Ti:H	TIH-
Vanadium hydride, V:H	VH-
Zirconium hydride, Zr:H	ZRH-
Hydroxides, KOH/NaOH	HYD-
Hydrocarbon, CH_4	HC-

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I

Ice, H_2O	ICE-
Indium, In	IN-
Indium alloys, InM_x	
Indium bismuth, In:Bi	INBI-
Indium tin, In:Sn	INSN-
Indium antimonide, InSb	INSB-
Indium arsenide, InAs	INAS-
Indium arsenide phosphide, $InAs_xP_{x-1}$	IASP-
Indium bismuthide, In_3Bi_3	INBI-
Indium gallium antimonide, InGaSb	IGSB-
Indium gallium arsenide, InGaAs	IGAS-
Indium gallium arsenide phosphide, InGaAsP	IGAP-
Indium oxide, In_2O_3	INO-
Indium phosphide, InP	INP-
Indium selenide, InSe	INSE-
Indium telluride, In_2Te_3	INTE-
Indium thallinide, In_2Tl_3	INTL-
Indium tin oxide, $InSnO_2$	ITO
Indium, rare earths, RE_In_3	INRE-
Iodine, I_2	I-
Iridium, Ir	IR-
Iridium vanadinide, IrV	IRV-
Iron, Fe	FE-
Iron alloys, FeM_x	
Iron beryllium, Fe:Be	FEBE-
Iron carbon, Fe_3C	FEC-
Iron chromium cobalt, Fe:Al:Cr:Co	KA-
Iron germanium, Fe_3Ge_2	FEGE-
Iron manganese, Fe:Mn	FEMN-
Iron nickel, $FeNi_3$	FENI-
Iron phosphide, FeP_2	FEP-
Iron silicon, Fe:Si	FESI-
Iron oxide, Fe_xO_y	FEO-
Ferrites, $FeM_xM_yO_z$	FER-
Iron palladium, FePd	FFPD-
Iron phosphide, FeP_2	FEP-
Iron sulfide, FeS_2	FES-
Pyrite, FeS_2	PYR-
Iron titanate, Fe_3TiO_4	FETI-

J

K

Kanthal, 72Fe:6Al:22Cr:1Co	KA-
Kovar, $Fe_xNi_yCo_z$	KO-
Krypton, Kr	KR-

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L

Lanthanum, La	LA-
Lanthanum boride, LaB6	LAB
Lanthanum bromide, $LaBr_3$	LABR-
Lanthanum nitride, Lan_{12}	LAN-
Lanthanum strontium cobaltite, $LaSrCoO_3$	LACO-
Lanthanum strontium ferrite, $LaSrFeO_3$	LAFE-
Lead, Pb	PB-
Lead alloys, PbM_x	
Lead silver, Pb:Ag	PBAG-
Lead tin, Pb:Sn	PBSN-
Lead titanium, Pb:Ti	PBTI-
Lead iodide, PbI_2	PBI-
Lead monoxide, PbO	PBO-
Lead molybdate, $PbMo_4$	PBM-
Lead selenide, PbSe	PBSE-
Lead sulfide, PbS	PBS-
Galena, PbS	PBS-
Lead tin selenide, PbSnSe	PBTS-
Lead telluride, PbTe	PBTE-
Lead germanium telluride, PbGeTe	PBGT-
Lead tin telluride, PbSnTe	PBTT-
Lead tungstate, $PbWO_4$	PBW-
Lead zirconate, $PbZrO_3$	PBZR-
Lithium, Li	LI-
Lithium bismuthide, Li_3Bi	LIBI-
Lithium bromide, LiBr	LIBR-
Lithium chloride, LiCl	LICL-
Lithium fluoride, LiF	LIF-
Lithium sulfide, Li_2S	LIS-
Lithium indium sulfide, $LiInS_2$	LIIS-
Lithium niobate, $LiNbO_3$	LINB-
Lithium nitride, Li_3N	LIN-
Lithium tantalate, $LiTaO_3$	LITA-
Lucite	LUC-

M

Magnesium, Mg	MG-
Magnesium fluoride, MgF_2	MGF-
Magnesium germanide, Mg_2Ge	MGFE-
Magnesium nitride, Mg_3N_2	MGN-
Magnesium oxide, MgO	MGO-
Magnesium selenide, MgSe	MGSE-
Magnesium silicide, Mg_2Si	MGSI-
Magnesium sulfide, MgS	MGS-
Magnesium telluride, MgTe	MGTE-
Magnetite, Fe_3O_4	MAGT-
Manganese, Mn	MN-
Manganese dioxide, MnO_2	MNO-
Manganese ferrite, $MnFe_2$	MNFE-

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Manganese stannide Mn_2Sn	MGSN-
Manganese sulfide, MnS_2	MNS-
Manganese telluride, $MNTe_2$	MNTE-
Mercury, Hg	HG-
Mercury alloys, HgMx	
Amalgam, $HgAg_x$	HGAG-
Amalgam, $HgZn_x$	HGZN-
Mercuric iodide, HgI_2	HGI-
Mercuric oxide, HgO	HGO-
Mercuric selenide, HgSe	HGSE-
Mercuric telluride, HgTe	HGTE-
Mercuric cadmium telluride, HgCdTe	HGCT-
Metal, general	MET-
Micas, $(OH)_2Al_2K(AlSi_3O_{10})$	MI-
Molybdates, $M_2Mo_3O_8$	FEMO-
Molybdenum, Mo	MO-
Molybdenum carbide, Mo_2C	MOC-
Molybdenum germanide, Mo_3Ge	MOGE-
Molybdenum nitride, MoN	MON-
Molybdenum oxide, MoO_3	MOO-
Molybdenum selenide, $MoSe_2$	MOSE-
Molybdenum selenide sulfide, $MoSeS_2$	MOSS-
Molybdenum sulfide, MoS_2	MOS-
Molybdenum telluride, $MoTe_2$	MOTE-
Mounting materials	
Apiezons	APZW-
Beeswax	BEEW-
Dielectrics	CER-; GLA-
Epoxy/polyimide	EPOX-; POLY-
Lacquers	PR-; VAR-; SHE-
Metals	IN; BRZ-
Paraffin	PARA-
Pitch	PICH-
Plastics	CRY-; LUC-; MYL-; BAK; DEK; PUT-; GLY-; SIL-
Resins	RES-
Vacuum	VAC-
Waxes	SWAX-; CAR-; PARA;
Wood	WOO-

N

Neodymium, Nd	ND-
Neon, Ne	NE-
Neptunium, Np	NP-
Nickel, Ni	NI-
Nickel alloys, NiMx	
Nichrome Ni:Cr	NICR-
Nickel aluminum, Ni:Al	NIAL-
Nickel boride, Ni_2B	NIB-
Nickel cobalt, Ni:Co	NICO-
Nickel:copper, Ni:Cu	NICU-

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Nickel iron, $FeNi_3$	FENI-
Nickel manganese, Ni:Mn	NIMN-
Nickel titanium, Ni:Ti	NITI-
Nickel zirconium, Zr_2Ni	NIZR-
Permalloy, Ni:Fe:Mg	PERM-
Nickelides, (rare earths) $ReNi_{12}$	RENI-
Nickel iodide, NiI	NII-
Nickel oxide, NiO	NIO-
Nitrogen, N_2	N-
Niobium, Nb	NB-
Niobium alloys, NbM_x	
Niobium aluminum, $NbAl_y$	NBAL-
Niobium zinc, Nb:Zr(25%)	NBZR-
Niobium boride, Nb_3B_3	NBB-
Niobium carbide, NbC	NBC-
Niobium germanide, Nb_3Ge	NBGE-
Niobium hydride, Nb:H	NBH-
Niobium nitride, NbN	NBN-
Niobium oxide, Nb_2O_5	NBO-
Niobium selenide, Nb_3Se	NBSE-
Niobium stannide, Nb_3Sn	NBSN-
Niobium telluride, Nb_3Te	NBTI-
Nitrides, M_xN_y	
Aluminum nitride, AlN	AlN-
Boron nitride, BN	BN
Calcium nitride, Ca_2N_3	CAN-
Cerium nitride, CeN_{12}	CEN-
Dysprosium nitride, DyN_{12}	DYN-
Erbium nitride, ErN_{12}	ERN-
Gadolinium nitride, GdN_{12}	GDN-
Gallium nitride, GaN	GAN
Germanium nitride, Ge_3N_4	GEN-
Hafnium nitride, HfN	HFN-
Holmium nitride, HoN_{12}	HON-
Iron nitride, Fe_xN_y	FEN-
Lanthanum nitride, LaN_{12}	LAN-
Lithium nitride, Li_3N	LIN-
Magnesium nitride, Mg_3N_4	MGN-
Molybdenum nitride, MoN	MON-
Neodymium nitride, NdN_{12}	NDN-
Niobium nitride, NbN	NBN
Praseodymium nitride, PrN_{12}	PRN-
Rare earth nitrides, REN_{12}	REN-
Samarium nitride, SmN_{12}	SMN-
Silicon nitride, Si_3N_4	SIN
Tantalum nitride, TaN	TAN
Terbium nitride, TbN_{12}	TBN-
Titanium nitride, TiN	TIN-
Uranium nitride, UN	UN
Yttrium nitride, YN_{12}	YN-
Zirconium nitride, ZrN	ZRN

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O

Organic semiconductors	OTGS-
Electrides, $C_xO_yH_zM$	ETD-
Osmium, Os	OS-
Osmium sulfide, OsS_2	OSS-
Osmium telluride, $OsTe_2$	OSTE-
Oxygen, O_2	O-

P

Palladium, Pd	PD-
Palladium alloys, PdM_x	
Palladium gold, Pd:Au	PDAU-
Palladium silver, Pd:Ag	PDAG-
Palladium hydride, Pd:H	PDH-
Palladium nickel phosphorus, PdNiP	PDNP-
Phosphorus, P	P-
Phosphorus containing etchants	
Phosphorus alloys, PM_x	
Gold phosphorus, Au:P	AUP-
Iron phosphorus, Fe:P	FEP-
Phosphorous pentoxide, P_2O_5	PO-
Photo resist	AZ-; PMMA-; COP-
Mounting materials (lacquers)	PR-; LAC-
Plastic	PL-; IUC-; POLY-; PCB-
Mounting materials (plastics)	CRY-; LUC-; MYL-; BAK-; DEK-; PUT-; GLY-; SIL-
Platinum, Pt	PT-
Platinum alloys, PtM_x	
Platinum:gold, Pt:Au	PTAU-
Platinum:palladium, Pt:Pd	PTPD-
Platinum:rhodium, Pt:Rh	PTRH
Platinum antimonide, $PtSb_2$	PTSB-
Platinum arsenide, $PtAs_2$	PTAS-
Platinum hydride, Pt:H	PTH-
Platinum oxide, PtO	PTO-
Platinum phosphide, PtP_2	PTP-
Plutonium, Pu	PU-
Polyvinylidene fluoride, PVF_2	PVF-
Potash alum, $KAl(SO_4)_2.12H_2O$	ALUM-
Potassium, K	K-
Potassium bromide, KBr	KBR-
Potassium chloride, KCl	KCL-
Potassium iodide, KI	KI-
Potassium manganese ferrite, $KMnFe_3$	KMI-
Potassium tantalate, $KTaO_3$	KTO-
Praseodymium, Pr	PR-
Praseodymium nitride, PrN_{12}	PRN-

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Q

Quartz, SiO_2	QTZ-
Coestite, SiO_2/SiO_4	COE-
Cristobalite, SiO_2	CRIS-
Opal, $SiO_2.nH_2O$	OPAL-
Silicon dioxide, SiO_2	SIO-
Stitsovite, $SiO_2/Si3O_4$	STI-
Tridymite, SiO_2	TRI-

R

Radium, Ra	RA-
Radon, Rn	RN
Rare earths, $RENi_{12}$	RENI-
Rhenium, Re	RE-
Rhodium, Rh	RH-
Rhodium alloys, RhM_x	
Platinum:rhodium, Pt:Rh	PTRH-
Rhodium silicide, RhSi	RHSI-
Rochelle salt, $NaKO_4H_4O_6.4H_2O$	RS-
Rubidium, Rb	RB-
Rubidium bromide, RbBr	RBBR-
Rubidium iodide, RbI	RBI-
Ruthenium, Ru	RU-
Ruthenium selenide $RuSe_2$	RUSE-
Ruthenium sulfide, RuS_2	RUS-
Ruthenium telluride, $RuTe_2$	RUTE-

S

Samarium, Sm	SM
Samarium bromide, $SmBr_3$	SMBR-
Samarium cobalt, $SmCo_5$	SMCO-
Samarium nitride, SmN_{12}	SMN-
Sapphire, Al_2O_3	SAPP-
Aluminum oxide, Al_2O_3	ALO-
Scandium, Sc	SC-
Selenium, Se	SE-
Silicides, M_xSi_y	
Boron silicide, B_4Si	BSI-
Cesium silicide, Ce Si_2	CESI-
Chromium silicide, $CrSi_2$	CRSI-
Cobalt silicide, $CoSi_2$	COSI-
Erbium silicide, ErSi	ERSI-
Hafnium silicide, HfSi	HFSI-
Iridium silicide, IrSi	IRSI-
Iron silicide, FeSi	FESI-
Iron tungsten silicide, FeWSi	IWSI-
Manganese silicide, MnSi	MNSI-
Molybdenum silicide, MoSi	MOSI-
Nickel silicide, NiSi	NISI-

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Niobium silicide, NbSi	NBSI-
Osmium silicide, OsSi	OSSI
Palladium silicide, PdSi	PDSI-
Platinum silicide, PtSi	PTSI
Praseodymium cobalt silicide, $PrCo_2Si_2$	PRCS-
Rhenium silicide, ReSi	RESI-
Rhodium silicide, RhSi	RHSI-
Tantalum silicide, TaSi	TASI-
Tellurium silicide, TeSi	TESI-
Terbium cobalt silicide, $TbCo_2Si_2$	TBCS-
Titanium silicide, TiSi	TISI-
Titanium/tungsten silicide, TiWSi	TWSI-
Tungsten silicide, WSi	WSI-
Vanadium silicide, VSi	VSI-
Zirconium silicide, ZrSi	ZRSI-
Silicon, Si	SI-
Silicon carbide, SiC	SIC-
Silicon alloys, MSi_x	
Aluminum silicon, Al:Si	ALSI-
Iron silicon, Fe:Si	FESI-
Silicon germanium, Si_xGe_y	SIGE-
Silicon tin, SiSn	SNSI-
Silicon dioxide, SiO_2	SIO-
Glass, SiO_2M_x	GLA-
Silicon monoxide, SiO	SIO-
Silicon nitride, Si_3N_4	SIN-
Silicon vanadinide, SiV_2	SIV-
Silver, Ag	AG-
Silver containing etchants	
Silver alloys, Ag_xM_y	
Silver aluminum, Ag_2Al	AGAL-
Silver amalgam, Ag_xHg	AGHG-
Silver gold, Ag_xAu_y	AGAU-
Silver lead/tin, Alloy #63	AGLT-
Silver tin, AgSn	AGSN-
Silver titanium, AgTi	AGTI-
Silver zinc, AgZn	AGZN-
Silver bromide, AgBr	AGBR-
Silver cadmium, AgCd	AGCD-
Silver chloride, AgCl	AGCL-
Silver mercury, Ag_2Hg	AGHG-
Silver germanium phosphide, $Ag_6Ge_{10}P_{12}$	AGEP-
Silver iodide, AgI	AGI-
Silver magnesium, AgMg	AGMG-
Silver sulfide, AgS	AGS-
Silver gallium sulfide, $AgGaS_2$	SGA-
Silver telluride, Ag_2Te	AGTE-
Silver antimony telluride, $AgSbTe_2$	SAT-
Silver gallium telluride, $AgGaTe_2$	AGGT-
Silver indium telluride, $AgInTe_2$	AGIT-
Silver iron telluride, $AgFeTe_2$	SIT-
Silver selenide, Ag_2Se	AGSE-

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Silver gallium selenide, $AgGaSe_2$	AGGE-
Silver indium selenide, $AgInAg_2$	AGIE-
Silver titanium selenide, AgTiSe	AGTS-
Silver epoxy	AGEP-
Sodium, Na	NA-
Sodium bromide, NaBr	NABR-
Sodium chloride, NaCl	NACL
Sodium niobate, $NaNbO_3$	NNO-
Sodium nitrite, $NaNO_3$	NAN-
Spinel, $MgAl_2O_4$	SPI-
Steel, Fe_3CM_x	ST-
Iron, Fe	FE-
Ferrite, general	FER-
Strontium, Sr	SR-
Strontium chloride, $SrCl_2$	SRCL-
Strontium fluoride, SrF_2	SRF-
Strontium gallium oxide, $SrGa_{12}O_{19}$	SGO
Strontium titanate, $SrTiO_3$	SRTO
Strontium tungstate, $SrWO_4$	SRWO-
Sulfur, S	S-
Sulfur containing etchants	

T

Talc, $H_2Mg_3(SiO_3)_4$	TALC-
Tantalum, Ta	TA-
Tantalum carbide, TaC	TAC-
Tantalum hydride, Ta:H	TAH-
Tantalum molybdenide, TaMo	TAMO-
Tantalum nitride, TaN	TAN-
Tantalum oxide, Ta_2O_5	TAO-
Tantalum selenide, $TaSe_2$	TASE-
Tantalum silicide, TaSI	TASI-
Tantalum sulfide, TaS_2	TAS-
Tantalum tungsten, TaW	TAW-
Tellurium silicide, TeSi	TESI-
Tellurides, $M_xM_yTe_z$	
Bismuth telluride, Bi_2Te_3	BITE-
Boron telluride, B_2Te_3	BTE-
Cadmium telluride, CdTe	CDTE-
Cadmium mercuric telluride, CdTe.HgTe	CDHT-
Copper indium telluride, $CuInTe_2$	CUIT
Germanium telluride, GeTe	GETE
Indium telluride, In_2Te_3	INTE
Lead telluride, PbTe	PBTE-
Lead germanium telluride, PbGeTe	PBGT-
Lead tin telluride, PbSnTe	PBTT-
Manganese telluride, $MnTe_2$	MNTE-
Mercuric telluride, HgTe	HGTE-
Mercuric cadmium telluride, HgCdTe	HGCT-
Molybdenum telluride, $MoTe_2$	MOTE-
Niobium telluride, Nb_3Te_4	NBTE-

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Osmium telluride, $OsTe_2$	OSTE-
Ruthenium telluride, $RuTe_2$	RUTE-
Silver telluride, Ag_2Te	AGTE-
Ferrous silver telluride, $AgFeTe_2$	FAGT-
Tin telluride, SnTe	SNTE-
Zinc telluride, ZnTe	ZNTE-
Tellurium, Te	TE-
Tellurium dioxide, TeO_2	TEO-
Terbium, Tb	TB-
Terbium nitride, TbN_{12}	TBN-
Thallium, Tl	TL-
Thallium beryllium telluride, TlBeTe	TLBET-
Thallium bismuth telluride, TlBiTe	TLBE-
Thallium tin telluride, TlSnTe	TLST-
Indium thallinide, In_2Tl_3	INTL-
Thallium selenide, Tl_2Se_3	TLSE-
Tin, Sn	SN-
Tin alloys, SnM_x	
Lead tin, 60Pb:40Sn #62	PBSN-
Tin dioxide, SnO_2	SNO-
Tin selenide, $SnSe_2$	SNSE-
Tin telluride, SnTe	SNTE-
Titanium, Ti	TI-
Titanium alloys, TiM_x	
Titanium gold, TiAun	TIAU-
Titanium aluminum, TiAl	TIAL-
Titanium carbide, TiC	TIC-
Titanium dioxide, TiO_2	TIO-
Titanium monoxide, TiO	TIO-
Titanium trioxide, Ti_2O_3	TIO-
Rutile, TiO_2	TIO-
Titanium nickel, TiNi	TINI-
Titanium nitride, TiN	TIN-
Titanium silicide, TiSi	TISI-
Titanium tungsten, TiW	TIW-
Titanium tungsten silicide, TiWSi	TWSI-
Tridymite, SiO_2	TRI-
Silicon dioxide, SiO_2	SIO-
Tritium, T	TRIT- (under Deuterium)
Tungsten, W	W
Tungsten oxides, WO_x	
Tungsten dioxide, WO_2	WO-
Tungsten pentoxide, W_2O_5	WO-
Tungsten trioxide, WO_3	WO-
Tungsten boride, WB_2	WB-
Tungsten carbide, WC	WC-
Tungsten rhenium, WRh	WRH-
Tungsten selenide, WSe_2	WSE-
Tungsten sulfide, WS	WS-
Tungsten titanium, TiW	TIW-

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U

Uranium, U	U-
Uranium boride, UB_4	UB-
Uranium carbide, UC	UC-
Uranium cesium, UCe_2	UCE-
Uranium cobalt, UCo_2	UCO-
Uranium dioxide, UO_2	UO-
Uranium nickel, UNi_2	UNI-
Uranium nitride, U_3N_4	UN-
Uranium platinum, UPt_3	UPT-
Uranium sulfide, US	US-
Uranium titanium, UTi	UTI-

V

Vanadium, V	V-
Vanadium aluminum, VAl_3	VAL-
Vanadium boride, VB_2	VB-
Vanadium bromide, VBr_2	VBR
Vanadium carbide, VC	VC-
Vanadium gallium, V_3Ga	VGA-
Vanadium germanium, V_3Ge	VGE-
Vanadium silicide, VSi	VSI-

W

Water, H_2O	WAT-
Cold etchants, general	COLD-
Water etchants	WATR-

X

Xenon, Xe	XE-

Y

Yttrium, Y	Y-
Yttrium cobalt:Mx, $Y_2(CoM)_{17}$	YCM
Yttrium garnets (see Garnets)	
Yttrium zirconate, $(Y_2O_3)_m(SrO_2)_{1-m}$	YZR

Z

Zinc, Zn	ZN-
Zinc alloys, ZnMx	
Zinc cadmium, ZnCd	ZNCD-
Zinc copper, Cu:Zn (brass)	BRAZ-
Zinc copper tin, Zn:Cu:Sn (bronze)	BRO-
Zinc gold, Au:Zn	AUZN-
Zinc mercury, ZnHg (amalgam)	ZNHG-
Zinc nickel, NiZn	ZNNI-

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Zinc niobium, ZnNb	NBZN-
Zinc silver, ZnAg	ZNAG-
Zinc tin, ZnSn	ZNSN-
Zinc arsenide, Zn_3As_2	ZNAS-
Zinc oxide, ZnO	ZNO-
Zinc phosphide, Zn_3P_2	ZNP-
Zinc selenide, ZnSe	ZNSE-
Zinc silicon fluoride, $ZnSiF_2$	ZSIF
Zinc silicon phosphide, $ZnSiP_2$	ZNP-
Zinc tin arsenide, $ZnSnAs_2$	ZNSA-
Zinc sulfide, ZnS	ZNS-
Sphalerite, ZnS	ZNS-
Zinc telluride, ZnTe	ZNTE-
Zinc tin phosphide, $ZnSnP_2$	ZTP-
Zinc tungsten, ZnW	ZNW-
Zirconium, Zr	ZR-
Zirconium boride, ZrB	ZRB-
Zirconium hydride, Zr:H	ZRH-
Zirconium oxide, ZrO_2	ZRO-
Zirconium nitride, ZrN	ZRN-
Zirconium palladium, Zr_2Pd	ZRPD-
Zirconium rhenium, Zr_3Rh	ZRRH-
Zirconium silicide, ZrSi	ZRSI-
Zirconium sulfide, ZrS_2	ZRS-
Zirconium vanadium, ZrV	ZRV-

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APPENDIX D: MILLER INDICES

In describing the external faces of any single crystal, the Miller Indices notations are most commonly used, and are directly related to the crystallographic axes. The numbers are derived from the reciprocals of fractional plane intercepts on axes using the smallest integer. As shown below, an x, y, z coordinate system is used for all crystal systems other than the hexagonal, which is x_1, x_2, x_3, z. Any given inorganic metal or metallic compound will always form with the same physical atomic distances within its ordered lattice, such that they will always give the same measurement when measured by X-ray and Laue photography. Atom-to-atom distances range between 1 to 5 Å and vary with every element or combination of elements, such that each mineral, compound or metal is always singularly distinctive.

In general processing of materials knowledge of specific atom distances is not essential, but the orientation of a single crystal face and the direction of bulk internal planes can be critical to a device construction and operation, such that an understanding of Miller Indices is a necessary working tool.

The Miller Indices are used for general notation of such atom locations where an external face crosses each axis, such that a (111) face is a triangle as it cuts all three axes. Where a face cuts only one axis and is parallel to the other two it is square and a (100) face . . . infinitely parallel to the other two axes, shown as a "Ø" rather than using the infinity symbol. In the isometric system, normal class, two forms as the octahedron, (111) and cube, (100) are the two most widely used surface planes in a semiconductor wafer processing. The dodecahedron, (110) planes are important for the directional orientation of bulk ⟨110⟩ planes in etching grooves and channels, the ⟨0$\bar{1}$0⟩ being preferred. Note that where a face cuts a negative axis, a negative sign appears above the Miller number.

Where the hexagonal system, normal class with three horizontal x-axes are involved, the 1st Order Prism is (10$\bar{1}$0), and the 2nd Order Prism, (11$\bar{2}$0), and in the rhombohedral division as a negative or positive rhombohedron, (101$\bar{1}$) or (10$\bar{1}$1), respectively.

Specific closure symbols are used in conjunction with the Miller Indices numbers as: (xxx) or (xxxx) = a specific face plane, such as the positive octahedron (111). ⟨xxxx⟩ or ⟨xxxx⟩ = a specific planar direction, such as ⟨110⟩. {xxx} or {xxxx} = all planes of a given type, such as {111}, all eight planes of the octahedron, (111). Note that the parenthesis is used when referring to a specific crystal form, such as the octahedron, cube, trisoctahedron, etc.

In addition to closed numbers, letters are used as general number face location symbols, such as (hk0), (hhl), (hkl), (hk0), such as the general notation for a textrahexahedron, which can be as (210), (310), (320), and a common form of a single crystal sphere of silicon when preferentially etched to finite crystal form.

Miller Indices are shown as a unit distance along an axis from a theoretical crystal center, and each axis has a positive and negative direction from center. This means that a (122) surface plane cuts the x-axis at unity, and the y- and z-axis at twice unity . . . which will produce a (122) isosceles shaped triangle surface face (facet) or, as a basic closed crystal form it is the trisoctahedron, (122). If the y- and z-axis cut their respective axes at half the x-axis unity distance, the face also will be an isosceles triangle, but the closed form will be trapezohedron, (211).

In establishing a plane face, each axial length is measured from crystal center to where the face (facet) crosses each axis. Form a fraction, then clear the fraction to obtain the specific face numbers as solid integers.

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Measure: x, y, z	Form fraction: x, y, z	Clear: x, y, z
1 2 2	1 2 2 — — — 1 1 1	1 2 2 = (122)
Measure: x, y, z	Form fraction: x, y, z	Clear: x, y, z
1 1/2 1/2	1 1/2 1/2 — — — 2 2 2	2 1 1 = (211)
Measure: x, y, z	Form fraction: x, y, z	Clear: x, y, z
1 1 0	1 1 0 — — — 1 1 0	1 1 0 = (110)
Measure: x, y, z	Form fraction: x, y, z	Clear: x, y, z
1 1/3 0	1 1/3 0 — — — 3 3 3	3 1 0 = (310)

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APPENDIX E: NATURAL MINERALS

ACTINIUM

*Actinium, Ac
Note: Radioactive series (gas)

ALABAMINE

*Alabamine, Ab
Hydrides, only to date

ALUMINUM

*Aluminum, Al
Cryolite, Na_3AlF_6
Corundum, Al_2O_3
Spinel, $MgO.Al_2O_3$
Bauxite, $Al_2O_3.2H_2O$
Gibbsite, $Al_2O_3.3H_2O$
Topaz, $(Al(F,OH))2SiO_4$
Kaolins, $H_4Al_2Si_2O_9$
Montmorillonite, $H_3Al_2Si_3O_{12}.nH_2O$
Turquoise, $CuO.3Al_2O_3.2P_2O_5.9H_2O$
Soda alum, $NaAl(SO_4)_2.12H_2O$
Potash alum, $KA1(SIO_4)_2.12H_2O$
Alunite, $K2Al_6(OH)_{12}.(SO_4)_4$

ANTIMONY

Native antimony, Sb
Stibnite, Sb_2S_3

ARGON

Native argon, Ar
Air, N_2/O_2 + about 1% Ar
Note: Inert gas, no compounds.

ARSENIC

Native arsenic, As
Realgar, AsS
Orpiment, As_2S_3
Arsenopyrite, FeAsS

BARIUM

*Barium, Ba
Barite, $BaSO_4$
Witherite, $BaCO_3$
Barylite, $Be_2BaSi_2O_7$
Benitoite, $BaTiSi_3O_9$

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BERYLLIUM

*Beryllium, Be
Beryl, $Be_3Al_2(SiO_3)_6$
Crysoberyl, $BeAl_2O_4$
Phenacite, Be_2SiO_4
Gadolinite, $Be_2FeY_2Si_2O_{10}$

BISMUTH

Native bismuth, Bi
Bismuthinite, Bi_2S_3
Guanajuatite Bi_2Se_3
Tetradymite, $Bi_2(Te,S)_3$

BORON

*Boron, B
Sassolite, $B(OH)_3$
Datolite, $HCaBSiO_5$
Axinite, Ca,Al borosilicate
Tourmaline, complex borosilicate
Lunebergite, $2MgO.Be_2O_3.P_2O_5.8H_2O$
Boric acid, H_3BO_3

BROMINE

*Bromine, Br
Hydrogen bromide, HBr (sea water extract)

CADMIUM

*Cadmium, Cd
Greenockite, CdS
Otavite, Cd carbonate

CALCIUM

*Calcium, Ca
Fluorite, CaF_2
Calcite, $CaCO_3$
Dolomite, $CaCo_3.MgCO_3$
Argonite, $CaCO_3$
Oligoclase, $CaAl_2Si_2O_8$
Amphibole group, Ca,Mg, etc., silicates
Titanite, $CaTiSiO_4$
Pyrochlore, Ca, Ce niobate
Alatite, Ca_3 (CaF) $(PO_4)_3$
Apatite, Ca4 (CaF) $(PO_4)_3$
Scheelite, $CaWO_4$
Limestone (chalk), $CaCO_3$

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Gypsum, $CaSO_4.2H_2O$
Anhydrite, $CaSO_4$

CARBON

*Carbon, C
Graphite, C
Diamond, C

CERIUM

*Cerium, Ce
Pyrochlore, Ca,Ce niobate
Monazite, $(Ce,La,Di)PO_4$
Yttrotantalate, Fe,Ca,Y,Fr,Ce tantalate

CESIUM

*Cesium, Cs
Pollucite, $2Cs_2O.2Al_2O_3.9SiO_2.H_2O$
Rhodizite, Al, K,Cs borate

CHROMIUM

*Chromium, Cr
Chromite, $FeO.Cr_2O_3$
Crocoite, $PbCrO_4$
Uvarovite, $Ca_2Cr_2\ (SiO_4)_3$

COBALT

*Cobalt, Co
Smaltite, $CoAs_2$
Cobaltite, CoAsS

COLUMBIUM

*Columbium, Cb
see Niobium

COPPER

*Copper, Cu
Horsfordite, Cu_6Sb
Mohawkite, Cu_3As
Richardite, Cu_4Te_3
Berzelianite, Cu_2Se
Eucairite, $Cu_2Se.Ag_2Se$
Chalcocite, Cu_2S
Covellite, CuS
Bornite, Cu_5FeS_4
Chalcopyrite, $CuFeS_2$

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Tetrahedrite, $3Cu_2S.Sb_2S_3$
Tennanite, $2Cu_2S.As_2S_3$
Cuprite, Cu_2O
Malachite, $CuCo_3.Cu(OH)_2$
Azurite, $CuCo_3.Cu(OH)_2$
Note: Over 300 copper minerals.

CHLORINE

*Chlorine, Cl
Halite, NaCl
Sylvite, KCl
Hydrogen chloride, HCl (volcanic)

DYSPROSIUM

*Dysprosium, Dy
Monazite, $(Ca,La,Di)PO_4$

ERBIUM

*Erbium, Eu
Fergusonite, Y,Er,Ce,U niobate

EUROPIUM

*Europium, Eu
Monazite, $(Ce,La,Di)PO_4$

FLUORINE

*Fluorine, F_2
Fluorite, CaF_2
Cryolite, Na_3AlF_6
Note: Over 100 fluorine minerals.

GADOLINIUM

*Gadolinium, Gd
Gadolinite, $Be_2FeY_2Si_2O_5$

GALLIUM

*Gallium, Ga
Bauxite, Al,Fe(OH) + trace Ga
Sphalerite, ZnS + trace Ga

GERMANIUM

*Germanium, Ge

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Germanite, $Cu_3(Fe,Ge)S_4$
Tennanite, $6(Zn,Fe)S.Sb_2S_3$
Freibergite: +3 − 30 Ag
Schwartzite: +6 − 17 Hg
Malinowskite: +13 − 16 Pb
Tetrahedrite, like Tennanite
Pyrite, FeS_2
Argyrodite, $4AgS.GeS_2$

GOLD

*Gold, Au
Petzite, $(Ag,Au)_2Te$
Sylvanite, $(Au,Ag)Te_2$
Krennerite, $(Au,Ag)Te_2$
Calverite, $AuTe_3$
Muthmannite (Ag,Au)Te
Magyagite, Au,Pb,sulfo-telluride

HAFNIUM

*Hafnium, Hf
Zircon, $ZrSiO_4$ + trace Hf
Note: Trace in most zirconium minerals.

HELIUM

Helium, He
Helium, He (gas well)

HYDROGEN

Hydrogen, H_2
Water, H_2O
Note: Over 2000 minerals contain hydrogen in their formulae.

HOLMIUM

*Holmium, Ho
Gadolinite, $Be_2FeY_2Si_2O_{10}$
Note: In other rare earth minerals.

ILLINIUM

*Illinium, Il
Monazite,$(Ce,La,Di)PO_4$

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INDIUM

*Indium, In

IODINE

*Iodine, I_2
Kelp, seaweed + iodine
Iodyrite, AgI
Miersite, 4AgI.CuI
Soda niter, $NaNO_3/KNO_3$ (saltpeters)

IRIDIUM

*Iridium, Ir
Iridosmine, Ir/Os mixture
Newyanskite, + 40 Ir
Siserskite, + 30 Ir

IRON

Iron, Fe
Awaruite, $FeNi_2$
Josephinite, $FeNi_3$
Pyrrhotite, FeS
Chalcovrite, $CuFeS_2$
Pyrite, FeS_2
Marcasite, FeS_2
Arsenopyrite, FeAsS
Hematite, Fe_2O_3
Ilmenite, $FeTiO_3$
Magnetite, Fe_3O_4
Franklinite, $(Fe,Zn,Mn)O.(Fe,Mn)_2O_3$
Chromite, $FeO.Cr2O_3$
Goethite, $Fe_2O_3.H_2O$
Limonite, $2Fe_2O_3.3H_2O$
Siderite, $FeCP_3$
Wolframite, $(FeMn)WO_4$
Note: Over 250 iron-bearing minerals.

KRYPTON

Krypton, Kr
Air, N_2/O_2 + trace krypton

LANTHANUM

*Lanthanum, La
Monazite, $(Ca,La,Di)PO_4$
Cerite, hydrated silicate of Ce,La, etc.

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LEAD

Native lead, Pb
Galena, PbS
Massicot, PbO
Plumboferrite, $PbO_2Fe_2O_3$
Cerussite, $PbCO_3$
Anglesite, $PbSO_4$
Sphalerite, ZnS + trace In
Pyrite, FeS_2 + trace In
Siderite, FeO.(OH) + trace In
Wulfenite, $PbMnO_4$
Note: Over 150 lead minerals.

LITHIUM

*Lithium, Li
Spodumene, $LiAl(SiO_2)_5$
Cookeite, Li Mica
Lepitdolite, Li Mica
Triphylite, $Li_3(Fe,Mn)PO_4$
Amblygonite, $LiAl(F,OH)PO_4$

LUTECIUM

*Lutecium, Lu
Note: Trace in Y minerals.

MAGNESIUM

*Magnesium, Mg
Magnesite, $MgCO_3$
Pyroxine group, Ca,Mg silicates
Amphibole group, Ca,Mg silicates
Pyrope, $Mg_3Al_2(SiO_4)_3$
Biotite, Mg,Fe silicate (mica)
Phlogophite, Mg (mica)
Penninite, $H_3Mg_5Al_2Si_3O_{18}$
Serpetine, $H_4Mg_3Si_2O_9$
Talc, $H_2Mg_3(SiO_3)_4$
Roselite, $(Ca,Co.Mg)_3As_2O_8.2H_2O$
Note: Over 150 magnesium minerals.

MANGANESE

*Manganese, Mn
Hauerite, MnS_2
Pyrolucite, MnO_2
Manganite, $Mn_2O_3.H_2O$

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Rhonochlosite, $MnCO_3$
Rhodonite, $MnSiO_3$
Spessartite, $Mn_3Al_2(SiO_4)_3$
Psilomelane, MnO_2
Note: Over 150 manganese minerals.

MASURIUM

*Masurium, Ma
Gadolinite, $Be_2FeY_2Si_2O_5$
Fergusonite, $(Nb,Ta)O_4$

MERCURY

Native mercury, Hg
Cinnabar, HgS
Amalgam, (Ag,Hg) variable
Calomel, HgCl
Coloradoite, HgTe
Onofrite, Hg(S,Se)
Tietmannite, HgSe
Montroydite, HgO

MOLYBDENUM

Molybdenum, Mo
Molybdenite, MoS_2
Molybdite, MoO_3
Wulfenite, $PbMoO_4$

NEODYMIUM

*Neodymium, Nd
Monazite, $(Ca,La,Di)PO_4$
Note: "Didymium" is two minerals as neodymium and praseodymium.

NEON
Neon, Ne
Air, N_2/O_2 + Ne trace

NICKEL

*Nickel, Ni
Awaruite, $FeNi_2$
Josephinite, $FeNi_2$
Millerite, NiS
Niccolite, NiAs
Wolfachite, Ni(As,Sb)S
Morenosite, $NiSO_4.7H_2O$

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NIOBIUM

*Niobium, Nb
Columbite-tantalite, $(Fe,Mn)\ (Nb,Ta)_2O_6$
Tapolite, $(Fe,Mn)\ (Nb,Ta)_2O_6$
Stibnotantalite, $(SbO)_2(Ta,Nb)_2O_6$
Samarskite, $(Fe,Ca)\ (Nb,Ta)_6O_2$

NITROGEN

*Nitrogen, N_2
Air, N_2/O_2 (24%)

OSMIUM

*Osmium, Os
Iridosmine, Ir/Os mixture

OXYGEN

Oxygen, O_2
Air, N_2/O_2 (24%)

OZONE

*Ozone, O_3
Air, $N_2/O_2 + O_3$

PALLADIUM

Native palladium, Pd
Platinum, Pt + trace Pd

PHOSPHORUS

*Phosphorus, P
Phosphates, metal(s) + PO_4
Xenotime, YPO_4
Monazite, $(Ce,La,Di) + PO_4$
Apatite, $(Ca,F)Ca_4(PO_4)_3$

PLATINUM

Native platinum, Pt
Sperrylite, $PtAs_2$
Cooperite, $Pt(As,S)_2$
Note: May be magnetic (+iron)?

POLONIUM

Polonium, Po

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Radium F is polonium
Note: Radioactive decay series.

POTASSIUM

*Potassium, K
Sylvite, KCl
Orthoclase, $KAlSi_3O_8$
Niter, KNO_3

PRASEODYMIUM

*Praseodymium, Pr
Monazite, (Ca,La,Di)PO_4

PROTOACTINIUM

*Protoactinium, Pa
Note: Radioactive series (gas) "parent" of actinium

RADIUM

Radium, Ra
Uraninite, complex uranate
Carnotite, $K_2O.2U_2O.V_2O_5.nH_2O$
Note: A radioactive decay product of uranium.

RADON

Radon, Rn
Air, N_2/O_2 + trace radon
Radon (as gas from earth faults)
Note: Active decay product of uranium.

RHENIUM

*Rhenium, Re
Wolframite, (Fe,Mn)WO_4
Tantalite, (Fe,Mn(Nb,Ta))$2O_6$

RHODIUM

*Rhodium, Rh
Platinum, Pt + trace Rh
Iridosmine, Ir/Os complex + Rh

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RUBIDIUM

Rubidium, Rb
Lepidolite, $(OH,F)_2KLiAl_2Si_3O_{10}$(mica)#
Pollux, $H_2O.2Cs_2O.2Al_2O_3.9SiO_2$(Elba)
Note: Castor and pollux; not defined minerals.

RUTHENIUM

*Ruthenium, Ru
Iridosmine, Ir/Os complex + Ru
Platinum, Pt + trace Ru

SAMARIUM

*Samarium, Sm
Fergusonite, $(Nb,Ta)O_4$ + Sm

SELENIUM

*Selenium, Se
Pyrite, FeS_2 + trace Se + Ge

SILICON

*Silicon, Si
Quartz, SiO_2 and varieties
Sand, SiO_2 (grains)
Silicates, general
Note: Over 60% of all minerals are silicates.

SILVER

Native silver, Ag
Amalgam, (Ag,Hg)
Argentite, Ag_2S
Hessite, Ag_2Se
Naumannite, $(Ab_2Pb)Se$
Stutzite, Ag_4Te
Petzite, (Ag,Au)2Te
Eucairite, $Cu_2Se.Ag_2Se$
Krennerite, $Au,AgTe_2$
Smithite, $Ag_2S.As_2S_3$
Auracvrite, $3Ag_2S.Sb_2S_3$
Ceragyrite, AgCl
Bromyrite, AgBr
Embolite, Ag(Br,Cl)
Miersite, 4AgI.CuI
Iodyrite, AgI

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SODIUM

*Sodium, Na
Halite, NaCl
Albite, $NaAlSi_3O_8$
Oligoclase, $Na/CaAlSi_3O_8$
Jadeite, $NaAl(SiO_3)_2$
Nephelite, $NaAlSiO_4$
Sodalite, $2Na_2Al_2Si_2O_8.2NaCl$
Lazurite, $3Na_2Al_2S_2O_8.2Na_2S$
Heulandite, $(Ca,Na_2)O.Al_2O_3.6SiO_2.5H_2O$
Borax, $Na_2B_4O_4.10H_2O$
Note: Over 150 sodium minerals.

STRONTIUM

*Strontium, Sr
Strontianite, $SrCO_3$
Celestite, $SrSO_4$

SULFUR

Native sulfur, S
Galena, PbS
Pyrite, FeS_2
Note: Over 300 minerals ores are reduced for sulfur content.

TANTALUM

Native tantalum, Ta(rare)
Columbite-tantalite, $(Fe,Mn)(Nb,Ta)_2O_6$
Fergusonite, $(Nb,Ta)O_4$
Yttrotantalite, $(Ta,Nb)4O_{15}.4H_2O$
Samarskite, $(Nb,Ta)_6O_2$
Note: Over 20 $Ta/Nb/UO_2$ minerals.

TELLURIUM

Native tellurium, Te#(rare)
Tellurite, TeO_2
Melonite, $NiTe_3$
Hessite, Ag_2Te
Note: A dozen Te minerals.

TERBIUM

*Terbium, Tb

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Monazite, $(Ca,La,Li)PO_4$
Gadoliniate, $Be_2FeY_2Si_2O_5$

THALLIUM

*Thallium, Tl
Hutchinsonite, $PbS\ (Tl,Ag)_2S.2As_2S_3$
Vbraite, $Tl_2S_3(As,Sb)_2S_3$
Lorandite, $Tl_2S.As_2S_3$
Pyrite, FeS_2 + trace Tl,Se,Ge

THORIUM

*Thorium, Th
Thorite, $ThSiO_4$
Monazite, $(Ce,La,Di)PO_4$ with ThO_3
Pyrochlore, $Nb_2O_6.(Ti,Th)O_3$
Note: Primary ore as ThO_3.

THULLIUM

*Thullium, Tm
Monazite, $(Ce,La,Di)PO_4$ + Tm
Note: Other rare earth minerals.

TIN

Native tin, Sn
Stannite, $Cu_2S.FeS.SnS_2$
Cassiterite, SnO_2
Teallite, $PbSnS_2$

TITANIUM

*Titanium, Ti
Rutile, TiO_2
Titanite, $CaTiSiO_5$
Perovskite, $CaTiO_3$
Polycrease, niobate-titanates
Brannerite, $(UO,TiO,UO_2)TiO_3$
Pyrochlore, $Nb_2O_6.(Ti,Th)O_5$
Note: Over 50 titanium minerals.

TUNGSTEN

*Tungsten, W
Wolframite, $(Fe,Mn)WO_4$
Tungstite, WO_3
Tungstenite, WO_2

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Scheelite, $CaWO_4$
Stolzite, $PbWO_4$

URANIUM

*Uranium, U
Carnotite, $K_2O.2U_2O.V_2O_5.3H_2O$
Uraninite, Uranate + Pb, Th, Zr, La, Ce; N_2, He, Ar gases
Note: Over 50 uranium minerals.

VANADIUM

*Vanadium, V
Carnotite, $K_2O.2U_2O,V_2O_5.3H_2O$
Note: Over 30 vanadium minerals.

VIRGINIUM

*Virginium, Va
Pollucite, $2OS_2O,2Al_2O.9SiO_2.H_2O$
Lepidolite, $(OF,F)_2KLiAl_2Si_3O_{10}$(mica)

WATER

Native water, H_2O
Salt water, oceans, and seas
Fresh water, on land (surface/aquifers)

XENON

Xenon, Xe
Air, N_2/O_2 + trace Xe

YTTERBIUM

*Ytterbium, Yb
Monazite, $(Ce,La,Di)PO_4$ + Yb
Note: Other rare earth minerals.

YTTRIUM

*Yttrium, Y
Gadolinite, $Be_2FeY_2Si_2O_{10}$
Fergusonite, $(Nb,Ta)O_2$ + Y
Xenotime, YPO_4
Note: Also niobate-tantalates/silicates.

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ZINC

Native Zinc, Zn
Sphalerite, ZnS
Wurtzite, ZnS
Zincite, ZnO
Franklinite, $(Fe,Zn,Mn)O.(Fe,Mn)_2O_3$
Smithsonite, $ZnCO_3$
Willemite, $Zn2SiO_4$
Calamine, H_2ZnSiO_5
Zinkosite, $ZnSO_4$

ZIRCONIUM

*Zirconium, Zr
Zircon, $ZrSiO_4$
Baddeleyite, ZrO_2
Note: Over ten zirconium minerals.

Note: Where item is starred (*), item is extracted from an ore, not a natural mineral compound.

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APPENDIX F: COMMON CHEMICALS

Chemical	Common name or acronym
Acetic acid (glacial), CH_3COOH	HAc(HOAc)
Acetone, CH_3COCH_3	Ace
Acetylene, C_2H_2	
Alum, potassium, $K_2Al_3(SO_4)_4.24H_2O$	Potash
Ammonia, NH_3	
Ammonium bifluoride, $NH_4F.HF$	
Ammonium chloride, NH_4Cl	
Ammonium fluoride, NH_4F	
Ammonium hydroxide, NH_4OH	
Ammonium nitrate, NH_4NO_3	
Ammonium sulfate, $(NH_4)_2SO_4$	
Antimony trichloride, $SbCl_3$	
Antimony trisulfide, Sb_2S_3	
Arsenic trioxide, As_2O_3	
Arsine, AsH_4	
Barium hydroxide, $Ba(OH)_2$	
Barium oxide, BaO	
Beet sugar, $C_6H_{12}O_6$	Glucose
Benzene, C_6H_6	
Bismuth chloride, $BiCl_4$	
Bismuth trioxide, Bi_2O_3	
Bleaching powder, CaCl(OCl)	Bleach
Borax, $Na_2B_4O_7.10H_2O$	
Boric acid, $B(OH)_3$	
Calcium carbonate, $CaCO_3$	
Calcium fluoride, CaF_2	
Calcium hydroxide, $Ca(OH)_2$	
Calcium oxide, CaO	Lime
Cane sugar, $C_{12}H_{22}O_{11}$	Sucrose
Carbolic acid, C_6H_5OH	
Carbon disulfide, CS_2	
Carbon tetrachloride, CCl_4	
Chloroform, $CHCl_2$	
Copper chloride, $CuCl_2$	
Copper nitrate, $Cu(NO_3)_2$	
Copper oxide, CuO	
Copper sulfate, $CuSO_4$	
Diborane, B_2H_6	
Ethane, C_2H_6	
Ethyl alcohol, C_2H_5OH	Ethanol, EOH
Ethyl ether, $C_2H_5OC_2H_6$	Ether
Ethylenediamine, $NH_2(CH_2)NH_2$	ED
Ethylene glycol, CH_2OHCH_2OH	EG
Ethylene, C_2H_4	
Ferric chloride, $FeCl_3$	
Ferric oxide, Fe_2O_3	Red ocher (rouge)
Ferric oxide, FeO(OH)	Yellow/tan rouge

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Ferrous oxide, Fe_3O_4	Black rouge
Ferrous sulfide, FeS	
Formic acid, HCOOH	
Fruit sugar, $C_6H_{12}O_6$	Fructose
Glucose, $C_6H_{12}O_6$	
Hydrochloric acid, HCl (95%)	Muriatic acid
Hydrofluoric acid, HF (49%)	HF
Hydrogen bromide, HBr	
Hydrogen peroxide, H_2O_2 (30%)	Peroxide
Hydrogen sulfide, H_2S	
Hypochlorous acid, $HClO_4$	
Iodic acid, HIO_3	
Iodoform, CHI_3	
Lactic acid, $CH_3CHOHCOOH$	
Lactose, $C_{12}H_{22}O_{11}$	Mother's milk
Lead chloride, $PbCl_3$	
Lead chromate, $PbCrO_4$	
Lead nitrate, $Pb(NO_3)_2$	
Lead oxide, PbO	Litharge
Lime, CaO	
Litharge, PbO	
Lithium chloride, LiCl	
Magnesia, MgO	
Magnesium hydroxide, $Mg(OH)_2$	Milk of magnesia
Maltose, $C_{12}H_{22}O_{11}$	Malt
Manganese dioxide, MnO_2	
Mercuric chloride, $HgCl_2$	
Mercurous nitrate, $Hg_2(NO_3)_2$	
Nitric acid, HNO_3 (70%)	White fuming
Nitric acid, HNO_3 (72%)	Yellow fuming
Nitric acid, HNO_3 (74%)	Red fuming
Methane, CH_4	
Methyl alcohol, CH_2OH	Methanol, MeOH
Methyl ether, CH_3OCH_3	
Methyl ethyl ketone	MEK
Oxalic acid, HOOC.COOH	
Perchloroethylene, C_2Cl_4	Perk, PCE
Phosphine, PH_3	
Phosphorous pentoxide, P_2O_5	
Plaster of Paris, $CaSO_4$ ($+H_2O$)	
Potassium bromide, KBr	
Potassium chlorate, $KClO_3$	
Potassium chloride, KCl	
Potassium cyanide, KCN	
Potassium dichromate, $K_2Cr_2O_7$	
Potassium ferricyanide, $K_3Fe(CN)_6$	
Potassium ferrocyanide, $K_4Fe(CN)_6$	
Potassium hydroxide, KOH	
Potassium iodide, KI	
Potassium nitrate, KNO_3	
Potassium nitrite, KNO_2	
Potassium permanganate, $KMnO_4$	

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Potassium sulfate, K_2SO_4	
Propane, C_3H_6	
Propionic acid, CH_3CH_2COOH	
(Iso)propyl alcohol, C_3H_7OH	ISO, ISO_4
Pyrocatechol, $C_6H_4(OH)_2$	P
Quinone, $C_6H_4O_2$	
Silver bromide, KBr	
Silver chloride, AgCl	
Silver iodide, AgI	
Silver nitrate, $AgNO_3$	
Soap, $3C_{17}H_{35}O$	
Sodium bicarbonate, $NaHCO_3$	
Sodium carbonate, Na_2CO_3	
Sodium chloride, NaCl	
Sodium hydroxide, NaOH	
Sodium iodide, NaI	
Sodium nitrate, $NaNO_3$	
Sodium sulfate, Na_2SO_4	
Sodium thiosulfate, $Na_2S_2O_3$	
Stannic chloride, $SnCl_4$	
Stannous chloride, $SnCl_2$	
Stearic acid, $C_{17}H_35COOH$	
Strontium chloride, $SrCl_3$	
Strontium nitrate, $Sr(NO_3)_2$	
Sucrose, $C_{12}H_{22}O_{11}$	Cane sugar
Sulfamic acid, NH_2SO_3H	
Sulfur dioxide, SO_2	
Sulfur trioxide, SO_3	
Sulfuric acid, H_2SO_4 (95%)	
Toluene, $C_6HC_5H_3$	
Turpentine, $C_{10}H_{16}$	
Xylene, $C_6H_4(CH_3)_2$	
Zinc chloride, $ZnCl_2$	
Zinc oxide, ZnO	
Zinc sulfate, $ZnSO_4$	
Zirconium dichloride, $ZrCl_2$	
Zirconium iodide, ZrI_4	

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APPENDIX G: HARDNESS

The ability of a material to withstand abrasion is a measure of its hardness. It is a relative figure of merit, and in processing material it is a handling factor. It is related to other materials characteristics, such as being brittle or soft, fracture potential, cleavage, sectility, in addition to physical structure being massive, fibrous, or granular. As a single crystal the crystallographic face orientation is hardness related to the direction of internal bulk planes which, in turn, are related to cleavage planes with their relative surfaces hardness.

In geologic study of crystallography and mineralogy the Mohs Hardness Scale was developed in the late 1800s as one measurement characteristic, and is still in use on all inorganic materials. There are other scales, such as the Mohs-Woodell, Knoop, Brinell, Vickers, Rockwell, and Shore. The latter is used for rubber and plastics. The Mohs scale uses specific natural minerals in a scale of 1 to 10 as a scratch test . . . a mineral of known hardness is drawn across the surface of the unknown sample. If it scratches readily, it is softer than the testing mineral; if unscratched, harder. A very faint scratch line represents a near equal hardness value. The Mohs Hardness Scale below is shown in comparison to other equivalent scales.

Mineral	Formula	Mohs	Mohs-Woodell	Knoop
Talc	$Mg_3Si_4O_{10}(OH)_2$	1	1	—
Gypsum	$CaSO_4.2H_2O$	2	2	(32)
Calcite	$CaCO_3$	3	3	135
Fluorite	CaF_2	4	4	163
Apatite	$Ca_5F(PO_4)_3$	5	5	430
Feldspar	$KAlSi_3O_8$	6	6	560
Quartz	SiO_2	7	7	820
Topaz	Al_2SiO_4	8	8	1340
Corundum	Al_2O_3	9	9	2100
Diamond	C	10	42.5	7000

The Mohs-Woodell used abrasive lap removal rather than a single scratch to determine hardness, such that it shows the wide difference between corundum and diamond in the Mohs Scale. The Knoop Scale uses a Tukon Hardness Tester with controlled pressure of a shaped diamond tip for measurement in kg/mm^2. As can be seen for measurements above H = 3, it is more definitive. Today it is being used in the measurement of micro-hardness on thin films.

As several artificial compounds are being used in both device construction or as lap and polish abrasives, some comparative values are shown in the following list.

Mineral	Mohs	Knoop
Corundum, Al_2O_3	9	2100
Tungsten carbide, WC	9+	1880
Tantalum carbide, TaC	9+	2000
Zirconium carbide, ZrC	9+	2100
Beryllium carbide, BeC	9+	2410
Titanium carbide, TiC	9+	2470
Silicon carbide, SiC	9+	2480
Aluminum boride, AlB	9+	2500
Titanium boride, TiB	9+	2720

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Cubic boron nitride, B_4N	9+	4600
Diamond, C	10	7000

The construction metals and alloys, such as irons, steels, copper and aluminum are measured by Rockwell A—G scales (U.S.A.); Brinell and Vickers (Great Britain and Europe), and all use a steel ball of different diameters as the indention tool. They are now used worldwide and comparative tables are available.

Most of the Solid State materials, semiconductors in particular, are all about Mohs H = 7, with the compound semiconductor being more brittle. Circuit substrates are all relatively hard. Alumina (H = 9) and beryllia (H = 8) mostly as pressed powders. Sapphire (single crystal alumina), or as fused ruby. Quartz as either fused or single crystal, and fused glass or silica (H = 6 to 7).

In fabrication of multilayer thin films or single surface coatings, micro-hardness of thin films becomes an important relationship, particularly, with regard to adhesion and processing conditions or reactions between layers. The measurement of micro-hardness for these layers is difficult due to their extreme thinness, such that they do not give a true bulk hardness and are directly affected by the hardness of the underlying substrate. Both optical reflection and electrical potentials have been used, and the Knoop hardness test and Rockwell D test have been converted for such measurement.

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APPENDIX H: COMMON ACRONYMS AND ABBREVIATIONS USED THROUGHOUT THE TEXT

I. Chemical

- Ace = Acetone
- AGW = Alcohol/Glycol/Water
- BC = Butyl Cellusolve
- BHF = Buffered Hydrofluoric Acid
- BRM = Bromine:Methyl Alcohol Note: BRM is used extensively in chem/mech lapping process
- CRY = Cryogenic (Gas) Liquid
  - LCl_2 = chlorine
  - LH_2 = hydrogen
  - LHe = helium
  - LN_2 = nitrogen
  - LOX = oxygen
  - LAR = argon
  - L_{air} = air
- DCE = Dry Chemical Etching (ionized gas)
- EA = Ethylene Acetate
- ED = Ethylenediamine
- EDP = ED:Pyrocatachol
- EG = Ethylene Glycol
- EOH = Ethyl Alcohol
- FG = Forming Gas (85% N_2:15% H_2)
- Gly = Glycerin
- Gly = Glycol
- HAc = (Glacial) acetic acid
- HOAc = (Glacial) acetic acid
- GAA = (Glacial) acetic acid
- IPA = Isopropyl Alcohol
- ISO = Isopropyl Alcohol
- KEY = Ketone (ref: acetone)
- MEK = Methyl Ethyl Ketone
- MeOH = Methyl Alcohol
- PCE = Perchloroethylene
- Perk = Perchloroethylene
- PG = Propylene Glycol
- P/R = Photo Resist (lacquer)
  - SH- = Shipley
  - AZ- = Horscht
  - COP- = Similar to AZ-types
  - KMER = KM series no longer used
  - PMMA = Designed for electron lithography
- TCA = Trichloroethane
- TCE = Trichloroethylene

II. Crystal, physics

- G(SG) = Specific Gravity (geology)
- g/cm^3 = Density (SG) (chemistry)

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H	=	Hardness (Mohs — geology)
		Brinell Hardness — (metals)
		Knoop Hardness (metals/materials)
		Rockwell Hardness (metals)
		Shore Hardness (rubber/plastic)
		Vickers Hardness (metals)
n	=	Refractive Index (Isometric System)
bcc	=	body-centered cubic
fcc	=	face-centered cubic
hcp	=	hexagonal close-packed
tcp	=	tetragonal close-packed
α, β, δ	=	tetragonal and other axes

III. Crystal, planes

(111)/(100)	=	Specific plane (Miller Indices) (xxx) parentheses
{110}	=	All planes of this type {xxx} brackets
⟨221⟩	=	Plane directions ⟨xxx⟩ hachures
$(10\bar{1}0)$	=	Hexagonal System (4-axes). May be as: (10.0)
$(\bar{1}11)$	=	Negative over-script "$\bar{1}$" denotes negative crystal axis.

IV. Crystal Structure

c	=	colloidal (c-Si)
a	=	amorphous (a-Ge)
c	=	crystalline (c-Si)
poly	=	polycrystalline (poly-Si) = crystalline
mc	=	microcrystalline (mc-Si)
mu	=	microcrystalline (mu-Si) (Greek letter "mu" = μ)
Mc	=	macrocrystalline (Mc-Si)
i/DLC	=	Diamond-Like Carbon (i-C/DLC)
xtl	=	single crystal
sxtl	=	single crystal
bixtl	=	bicrystal
r	=	ribbon crystal (dendritic)
GB	=	grain boundary

V. Etching

WCE	=	Wet Chemical Etching (WF = Wet Format, e.g., liquids, etc.)
EE	=	Electrolytic Etching (EF = Electrolytic Format)
DCE	=	Dry Chemical Etching (DF = Dry Format, e.g., ionized gas)

VI. Process/Equipment

A. Equipment

(1) Microscopes

AES	=	Auger Electron Microscope
FDX	=	Energy Dispersive X-ray
ESCA	=	Electron Spectroscopy for Chemical Analysis
FIM	=	Field Ion Microscope
HEED	=	High Energy Electron Diffraction
LEED	=	Low Energy Electron Diffraction
PLM	=	Polarized Infrared Microscope
SAM	=	Scanning Auger Microscope
SIMS	=	Secondary Ion-Mass Spectroscopy

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SLAM = Scanning Laser Acoustic
UPS = Ultraviolet Photo-Electron Spectroscopy
XPA = X-ray Photo-Electron Spectroscopy
*SEM = Scanning Electron Microscope
*TEM = Transmission Electron Microscope

*Widely used in general material processing as diagnostic defect failure tool, with EDX unit.

(2) Chemical Vapor Deposition

CVD	=	Chemical Vapor Deposition
APCVD	=	Atmospheric Pressure CVD
HOMOCVD	=	Homogeneous CVD
HPCVD	=	High Pressure CVD
LPOMCVD	=	Low Pressure OMCVD
OMCVD	=	Organo-metallic CVD
PECVD	=	Plasma Enhanced CVD
VHPCVD	=	Very High Pressure CVD
HMCVD	=	Horizontal Magnetic CVD
VMCVD	=	Vertical Magnetic CVD

(3) Epitaxy Growth (Epi)

HEP	=	Horizontal Epitaxy
HPE	=	Horizontal Phase Epitaxy
HWE	=	Hot-Wall Epitaxy
LPE	=	Liquid Phase Epitaxy
CCLPE	=	Current Controlled LPE
L-SPE	=	Lateral Solid Phase Epi
VEP	=	Vertical Epitaxy
VPE	=	Vapor Phase Epitaxy
V-SPE	=	Vertical Solid Phase Epi
*MBE	=	Molecular Beam Epitaxy

*Most advanced and versatile system in present technology.

(4) Growth Systems, general

*CZ	=	Czochralski (pulled xtl)
FZ	=	Float Zone (solid xtl)
BM	=	Bridgman Method
**HB	=	Horizontal Bridgman
VB	=	Vertical Bridgman
EFG	=	Edge Defined Film Fed Growth (ribbon xtl) + other acronyms by developers
VM	=	Verneiul Method (hot droplet)
LEVCZ	=	Levitation CZ (development for space application)
LEC	=	Liquid Encapsulated CZ
MFG(FS)	=	Molten Flux Growth or Fused Salt (*Note:* xtls may be contaminated by flux)
HEM	=	Heater Enhance Method (poly CZ type)

Note: Term "ingot" = boule in sxtl growth, and more widely used.
* 2″ diameter now standard; 6″ available CZ/FZ/HB methods the most widely us then sliced as wafers.
** recognized by half-moon shape of cut wafers

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(5) Vapor Transport Deposition

VT	=	Vapor Transport
CSVT	=	Close-Spaced VT

(6) Element Doping/Deposition

ALY	=	Alloy into material (Al into silicon is Square Law).
DIF	=	Diffuse element into material (B, Sb, As, etc., is Gaussian Diffusion Law). Called: Graded Junction
I^2	=	Ion Implantation (Si^+ ionized particle at eV/MeV energy levels. Also Gaussian)
EVAP/M	=	Metal evaporation + thermal
DEC/M		drive-in (also used to metal decorate defects/decoration)
EVAP/Ox	=	Oxide deposit with doping element as glass (ASG, BSG, PSG, BPSG, etc., and may be a nitride as Final coat or for thermal drive-in)
SSDIF	=	Solid-Solid Diffusion (may be Solid Phase Epi, SPE)
P-ON	=	Paint on compound + thermal drive-in. (Gaussian)
CONV	=	Evaporate metal + thermal conversion, e.g., Silicides. (MoSi, Mo_2Si, $MoSi_2$, etc.)
OX	=	Oxidation (Wet, Dry, Steam or SILOX System). Also electrolytic
W/Mo	=	Std light filaments, white
SILOX	=	Oxidation from SiH_4:O_2/N_2 300—500°C
RF/DC	=	RF/DC Plasma deposition of oxides, nitrides, metals and compounds under vacuum
V-MET	=	Metal(s) evaporation under vacuum (metallization). With RF/DC Plasma as metallization or thin film compound deposition
RF-MAG	=	RF magnetron deposition. Magnet enhances deposition rate/opn
EB/E-Beam	=	Electron Beam metallization (260° bent beam now common)
PD	=	Pyrolitic Deposition (See: CVD)

(7) Etching Systems/Methods

IM	=	Ion Milling (pattern ion gas etch of thin films)
EBL	=	Electron Beam Lithography (Ref: P/R with PMMA)
MFE	=	Molten Flux Etching
PE	=	Plasma Etching
PL	=	Photolithography
RIE	=	Reactive Ion Etching
IE	=	Ion Etching (nonreactive)
PR	=	Photo Resist

(8) Lamps/Lights

Ar	=	Argon, white
Cd	=	Cadmium, yellow
Co	=	Cobalt, blue
Cr	=	Chromium, yellow
Fe	=	Iron, yellow-green
IR	=	Infrared (below VL)
K	=	Potassium, bright yellow
Kr	=	Krypton, yellow-green
Na	=	Sodium, common yellow
Ne	=	Neon, orange
Sr	=	Strontium, deep red

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UV	=	Ultraviolet, (above VL)
VL	=	Visible light spectrum, white
W/Mo	=	Std. light filaments, white
Xe	=	Xenon, intense white

VII. Water, H_2O

Recirc	=	Recirculating water
DI	=	Distilled
dd	=	double distilled (2d) (2DI)
ddd	=	triple distilled (3d) (3DI)
Hi-Q	=	DI + ion exchange
HQ	=	High Quality
dm	=	demineralized
di	=	deionized

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