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MAIL STOP PROVISIONAL PATENT APPLICATION

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PROVISIONAL APPLICATION TRANSMITTAL
(37 CFR §1.51(2)(i))

Transmitted herewith for filing is:

1. **Type of Application:** PROVISIONAL (37 CFR §1.51(a)(2)(i)(A))
2. **Name and address of Inventor(s):** **VIJAY GUPTA; VASSILI A. KIREEV**

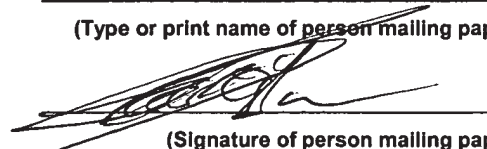
VIJAY GUPTA 15133 GREENLEAF STREET SHERMAN OAKS, CA 91403	VASSILI A. KIREEV 10920 WILSHIRE BOULEVARD, SUITE 1200 LOS ANGELES, CA90024-1406
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3. **Title of Invention:** GLASS-MODIFIED STRESS WAVES FOR ADHESION
MEASUREMENT OF ULTRA THIN FILMS AND NANO-ELECTRONICS
DEVICE FABRICATION
4. **Name, registration and telephone number of attorney:**
John P. O'Banion, Reg. No. 33,201
(916) 498-1010

CERTIFICATION UNDER 37 CFR 1.10

I hereby certify that this Provisional Application Transmittal and the documents referred to as enclosed therein are being deposited with the United States Postal Service on 05 MARCH 2004 in an envelope as "Express Mail Post Office to Addressee" Mailing Label Number EV352303977US addressed to the: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22213-1450.

John P. O'Banion

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5. **This invention was made by an agency of the U.S. Government or under contract with an agency of the U.S. Government**

☐ No.

☒ Yes.

Name of U.S. Government agency and the Government contract number:

ARMY RESEARCH OFFICE GRANT NO. DAAD19-00-1-0491

6. **Identification of Application Documents Enclosed:**

15 Pages of Specification

☐ Pages of Claims

11 Pages of Drawings

☐ Pages of Abstract

7. **Language**

☒ English

☐ non-English

☐ the attached translation is a verified translation. 37 CFR 1.52(d).

8. **Assignment**

☐ An assignment of the invention to: _____

☐ is attached. A separate "ASSIGNMENT COVER LETTER ACCOMPANYING NEW PATENT APPLICATION" is also attached.

☐ will follow.

9. **Fee Calculation (37 CFR 1.16)**

Small Entity Statement(s)

☒ Applicant qualifies as a small entity under 37 CFR 1.9 and 1.27.

Filing Fee Calculation

\$ 80.00

2

10. **Fee Payment Being Made At This Time**

☐ Not Enclosed

☐ No filing fee is to be paid at this time. (This and the surcharge required by 37 CFR 1.16(e) can/will be paid subsequently.)

☒ Enclosed

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☐ recording assignment (\$40.00; 37 CFR 1.21(h)) \$ _____

Total Fees Enclosed \$ 80.00

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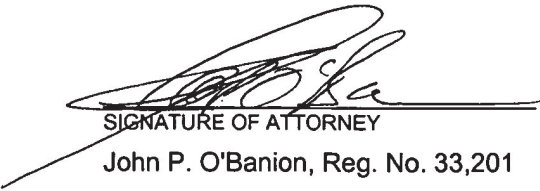
Please use the following correspondence address for all communications:

CUSTOMER NUMBER 8156

**John P. O'Banion, Reg. No. 33,201
O'BANION & RITCHEY LLP
400 Capitol Mall, Suite 1550
Sacramento, CA 95814
(916) 498-1010**

15. **Signature of Attorney**

Dated: 3/5/04


SIGNATURE OF ATTORNEY

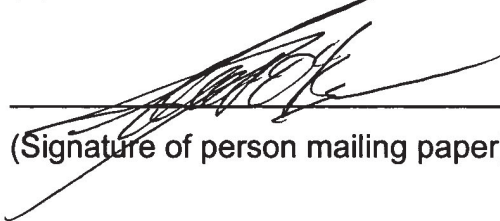
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TITLE OF THE INVENTION
**GLASS-MODIFIED STRESS WAVES FOR ADHESION MEASUREMENT OF ULTRA
THIN FILMS AND NANOELECTRONICS DEVICE FABRICATION**

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] Not Applicable

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH
OR DEVELOPMENT

[0002] This invention was made with Government support under Grant No. DAAD19-00-1-0491 awarded by the Army Research Office. The Government has certain rights in this invention.

INCORPORATION-BY-REFERENCE OF MATERIAL
SUBMITTED ON A COMPACT DISC

[0003] Not Applicable

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1. Title

Glass-Modified Stress Waves for Adhesion Measurement of Ultra Thin Films and Nanoelectronics Device Fabrication.

2a. Purpose

2a.1. Measure the tensile strength (adhesion) of very thin film interfaces (thickness less than 0.5 micrometers), and their multilayers deposited on engineering substrates.

2a.2. Separate and lift thin film lines or their complete structures from semiconductor and engineering substrates using glass-modified stress waves, and catch them on desired substrates for reconstructing structures. This can lead to a faster way of fabricating MEMS and nano-scale devices by bypassing the currently used wet-etching techniques. This method can provide an attractive alternative to create nanocircuitry.

2b. State of the Art Prior to the Invention-The Basic Laser Spallation Experiment

The state of the art is embodied in the United States Patent No. 5,438,402 incorporated herein by reference in its entirety.

The knowledge embodied in the above patent allows measurement of tensile strength of interfaces of films greater than 1 micrometer in thickness. The invention disclosed here will extend the capability of this tool to films below 0.5 μm in thickness. The basic procedure is outlined below.

In the laser spallation experiment (Fig. 1), a 3-nanoseconds (ns) long Nd:YAG laser pulse is made to impinge over a 3 mm-dia area on a 0.5 μm thick aluminum film that is sandwiched between the back surface of a substrate disc (12-25 mm diameter and 1-mm thick) and a 10 to 20 μm thick layer of SiO_2 . The melting-induced expansion of aluminum under confinement generates a compressive stress pulse (with 1 ns rise time) directed towards the test coating, which is deposited on the substrate's front surface. The compression stress pulse reflects into a tensile wave from the coating's free surface and leads to its spallation (complete removal) at a sufficiently high amplitude. The interface tensile stress is obtained by measuring the transient displacement history of the coating's free surface (induced during pulse reflection) by using an optical interferometer (Pronin and Gupta, 1993), a schematic of which is shown towards the right in Fig. 1. For a coating of density ρ and thickness h , the interface stress σ is calculated from the measured transient velocity $v(t)$ as

$$\sigma(h, t) = \frac{1}{2} \rho c \left[v\left(t + \frac{h}{c}\right) - v\left(t - \frac{h}{c}\right) \right] \quad (1)$$

where c is the longitudinal stress wave velocity in the film.

Interface strengths in a variety of systems, including those between metal, ceramic, and polymeric coatings and engineering substrates, have been measured. It has been amply shown (Gupta et al., 1997; Yuan et al., 1995; Yuan and Gupta, 1995) that the laser spallation experiment allows a correlation between the interface tensile strength and the interface microstructure. This allows optimization of the coating deposition processes and a more robust prediction of service reliability. For reasons discussed next, the technique is generally limited to testing films with thickness greater than 1 μm .

The Need for Improvement in the State of the Art

Laser-generated stress wave profiles that are used to decohere films in the basic

technology discussed above are reproduced in Fig. 2. These measurements were made in a Si wafer. The rise and post-peak decay time do not change significantly with increasing stress pulse amplitudes obtained at increasing laser fluences. A typical stress pulse profile has a rise-time of 1-2 ns, and a gradual post-peak decay of about 16-20 ns. If the film is very thin compared to the length of the stress wave, then the tail end of the initial compression pulse is still at the interface while its front end has returned as a tensile wave after reflecting from the film's free surface. Because of destructive interference, the peak interfacial tensile stress is always less than the amplitude of the initial compression wave. The amplitude of the peak interface tensile stress decreases non-linearly with the film thickness, the rise-time and post-peak decay time of the stress pulse, and as expected, becomes zero in the limiting case of zero film thickness. Thus, for thin layers, the peak interfacial tensile stress can be much lower than the peak tensile stress inside the substrate. For intrinsically strong interfaces, such a condition usually leads to failure within the substrate before the desired interface can be separated. This is demonstrated through results presented in Table I, which summarizes interface tensile strength measurements using the basic laser spallation technique for a Cu/TaN bilayer system sputter-deposited on a Si wafer. Failure was observed inside the Si for the first three samples because of insufficient film thickness. Note the consistency in the peak tensile stress inside the Si for these samples, indicating a tensile strength of Si of 5 GPa. For these samples the Si/TaN interface tensile stress is rather low, and below its interfacial strength. As the film thickness was increased, the failure locus changed from the substrate to the interface. Consistently the peak tensile stress inside Si was lower than its strength measured in previous samples. The interface strength was found to be the same for the last two samples, indicating that if the interface chemistry is the same for films of varying thicknesses then the experiment measures the same fundamental strength. Thus, the interface strength can be measured in ultra thin film samples by simply increasing the film thickness or adding another layer on top to provide enough "room" for the reflecting compression pulse. This was demonstrated by Dijk, et al. (1998). **However, because of issues related to possible film structure and chemistry changes due to increased film thickness in several systems, the preferred way is always to test films in their initial low thickness state or in the same form as to be used in actual service devices and**

applications. Accordingly, the focus of research leading to this patent disclosure was to develop strategies to allow successful spallation of ultra thin films (thickness less than 0.5 μm) and consequently measure their adhesion.

Using a finite element-based simulation Yuan and Gupta (1993) showed that for a given coating thickness the post-peak decay time is the most critical in maximizing the interfacial tensile stress. Indeed, if the post-peak decay time is reduced to zero, or in other words, the wave profile has a “rarefaction shock” then theoretically, the interface tensile stress for a film of arbitrarily small thickness will equal the amplitude of the incoming compression pulse. As discussed next, this theoretical possibility has been realized experimentally using glasses, and is the subject of this invention disclosure.

2C. New Technology and Its Functionality

2C.1. Observations of Stress Pulses with Rarefaction Shocks in Glasses

Stress waves were measured in Pyrex, sodalime, quartz, and borosilicate glasses using the experimental procedure that is embodied in the US Patent 5,438,402. To define the entering stress wave profile in the glass specimen accurately, these tests were performed by bonding the glass slides of nominal 1-mm thickness to 0.7 mm-thick Si wafers using a 0.5 μm -thick optical grade EPOTECH 301 FL epoxy. The stress wave was generated inside the Si by exfoliating a waterglass-constrained Al film in a manner discussed above. For interferometric measurements, a 400Å layer of Al was deposited on the free surface of the glass.

Figure 3 shows a series of stress pulse profiles with increasing laser fluence, measured in a soda lime glass. At low stress amplitudes, the profiles are similar to that in Si and other materials, having a finite rise-time and gradual post-peak decay. However, as the stress pulse amplitude exceeds a certain threshold, the rise-time of the stress wave gets longer but the post-peak stress profile starts to decay rather quickly. Ultimately a profile is

attained with the post-peak stress dropping instantaneously (the drop time is within the resolution limit of our instrumentation), much like a "rarefaction shock." The modification in the stress wave profile due to glass compared with that due to Si can be appreciated by viewing Fig 3b.

Figure 4 shows the pulse profiles measured in Pyrex, soda lime, quartz, and borosilicate glasses. The magnitude of the above effect was found to vary from glass to glass, but all showed the formation of the rarefaction shock.

Previous investigators (e.g., see, Clifton et al., 1997) who used plate impact setups were unable to uncover the formation of rarefaction shocks in glasses since the pulse lengths in their work was larger than the specimen thickness. As suggested above, this effect develops due to finite propagation distance of the pulse into the material. This became possible in our work because of the short length (~ 0.1 mm) of the stress waves compared with the specimen thickness (~ 1 mm).

2C.2. Demonstration of the Invention

Application 1: Measurement of Tensile Strength of Thin Film Interfaces

To demonstrate the use of glass-modified stress waves, the sample sets summarized in Table II were prepared. All films were sputter deposited, with thickness indicated in the table. The stress waves were directly generated inside the glass. Accordingly, consistent with the laser spallation procedure, the free surface of glass was coated with a 0.5- μm -thick Al film and constrained from top using a 40-50 μm -thick layer of water glass. The TiN/glass, and the Ni/glass systems, which have interest in display technology, were chosen to demonstrate the applicability of the glass-modified waves in decohering very thin film interfaces.

In addition to the glass substrates, a Cu(1400nm)/TiN(70nm) bilayer system deposited on a Si wafer was considered. Previously, when the stress pulse was directly

generated inside the Si wafer, failure within Si was observed. This, as discussed above, resulted because of the very high strength of the interface. For these samples, the test-coating-free surface of Si was bonded using an UV-cured epoxy to Pyrex glass to allow loading due to glass-modified waves. Consistent with the spallation procedure, laser-absorbing Al layer was deposited on the backside of the glass substrate and constrained by a 40-50 mm-thick layer of waterglass from the top.

Table II shows the measured strengths in thin film systems. The failures were observed at the interfaces of interest, with specific strength values for each shown in the table. This clearly demonstrates the potential of the new technology in measuring the tensile strength of very thin film interfaces.

Figure 5a shows the failure inside the Si in a Cu(1400nm)/TiN(70nm)/Si system when no glass was used. Figure 5b shows a high magnification view of failure when the new technology of glass-modified waves were used. The arrow shows the failure to be at the Cu/TiN interface with no fracture inside the Si. An interface tensile strength value of 2.62 GPa was calculated. This is a rather high value, which was not possible to attain using the basic spallation setup. This is quantitatively shown in Fig. 6 (a) and (b). Figure 6(a) shows the measured free surface velocity profiles in the borosilicate glass and Si substrates corresponding to the failures shown in Fig. 5. The interface tensile stress history corresponding to each profile is shown in Fig. 6 (b). The dramatic increase in the interface tensile stress due to the glass-modified wave is evident.

Application 2: MEMS and Nanoelectronics Device Fabrication

A related application that could become significant in the nanotechnology area is the use of these special waves in manufacturing nanoscale circuits and devices, which are structures composed of nanowires on electronic substrates (Si, Ge, etc.). Nanocircuitry-based devices should lead to significantly faster than those possible with the current micron-scale-technology. The photolithography technology used today has essentially

reached its limit in terms of the smallest feature size that can be manufactured. New ideas for nanowire fabrication include, E-beam etching (which is extremely slow), and anisotropic-lattice-mismatch-based film deposition ideas. In the latter approach, the materials for the film and the substrate are chosen such that there is a substantial lattice mismatch in one direction, and almost none in another. The large mismatch constrains film growth, while encouraging film growth in the zero-lattice-mismatch-direction. This procedure is thus constrained to few selected film/substrate combinations.

How will the New Waves help?

A schematic of the conceptual idea is shown in Fig. 7. The nanowires deposited on specialized substrates are separated using the new waves. As discussed above, separation of such thin wires is only possible using the glass-modified waves. The separated wires are then “caught” on desired electronic substrates having either a very thin layer of adhesive or a self-assembled molecular layer. Circuits can then be constructed by repeated use of this procedure. There are no restrictions on the type of substrate used.

This has so far been demonstrated on a multilayer plastic substrate whose cross-section is shown in the inset of Fig. 8. The focused view on the top layer shows lead bumps that are used for electrical contacts allowing the Si device to communicate with the substrate circuitry. The laser was impinged onto the undersurface of the plastic substrate to launch the stress wave towards the top. The stress wave separated the bumps and these separated bumps were caught on a flexible plastic tape. The success of this procedure is demonstrated in Figs. 8 and 9, which show low and high magnification micrographs of the separated lead bumps on the tape. It was interesting to find that as the laser energy was increased, separation below the bump areas was also accomplished. Thus, in such cases, the bumps along with their underlying film were pulled free from the substrate and caught on the plastic tape. The dark region in Fig. 8 is the line that was below the bumps, which now appears on the very top of the flexible tape substrate. Figure 9 shows focused view of the separated bumps in their entirety.

One application of the glass-modified waves is to make high performance circuits on flexible substrates such as plastics. This has applications in manufacturing of low cost RFID tags, TFT-based high performance computers, sensor systems. This idea was disclosed as part of a DARPA proposal in May 2003 (details available in the proposal). The basic idea is to fabricate a very high quality polysilicon or a single crystal Si layer (over a finite area) on a glass or a quartz substrate using already well-established process variables (Fig. 10). Similarly, as shown in Fig. 11, already fabricated high performance circuits can be generated first on a Si or a glass platform. Next, the entire polysilicon surface will be spun coated with the polymer film-the "recipient substrate." This will result in a glass/Si/polymer sandwich. Instead of spin coating the polymer substrate an already formed polymer foil could also be placed in closed vicinity of the Si platform or circuits as shown in Fig. 11. A high amplitude compressive stress wave will be generated on the backside of the glass substrate by focusing a Nd:YAG laser pulse onto a 0.5 mm-4 mm-dia area that has a water-glass-constrained Al film. The stress wave will directly detach the Si/glass interface to completely transfer the entire Si film or Si platforms, or circuits onto the plastic substrate.

A variation of this idea will be not to use the Al or the constraining waterglass layer on the backside. In that case the laser will go through the glass and directly interact with the Si surface to launch a stress wave. This wave will then pry off the Si/glass interface to transfer the Si film or any circuits to the polymer substrate on top. Thus, the mechanism of pattern transfer remains the same in both approaches.

2D. Why would somebody pay for this Invention?

Films and coatings have widespread use in different industries. Examples include, thermal barrier coatings for engines; tribological coatings in cutting tools, seals, and joints; polymeric layers in paint assemblies; fiber coatings in composites; electrical, magnetic and optical multilayers in electronic devices; metal and ceramic films in MEMS-based mechanical and clinical devices, among others. Adhesion of films is a prerequisite to carryout their intended functions. Thus, the central goal in all these applications is to avoid film delamination and coating failures by *maximizing* adhesion, and to predict long-term reliability of the coated components. These issues are usually addressed by seeking a fundamental understanding of the adhesion between different layers as a function of process (film deposition and surface variables) and service (moisture and temperature) variables. This is accomplished using adhesion metrology tools, one of which is the basic laser spallation experiment developed by Gupta and coworkers (Gupta et al., 1992; Yuan and Gupta, 1993; Yuan et al., 1993, Yuan and Gupta, 1995; Yuan et al., 1995). This technique, as discussed above, is limited in testing films with thickness greater than 1 micrometer. Similarly strengths of very strong interfaces cannot be measured.

An experimental effort to measure adhesion of ultra-thin layers is rather timely as films with thickness below 0.5 micrometers are now the focus of research in the microelectronics industry for developing ultrahigh density devices using nanotechnology. Also, adhesion at similar length scales will become important in the material optimization of MEMS-based mechanical and clinical devices when they are mass-produced.

When mechanical failures become a problem, this technique will allow selection of different material systems that exhibit improved adhesion.

Any technology that assists or greatly improves construction of nanoelectronic and MEMS-based devices can become significant in realizing the potential of nanotechnology and biotechnology.

References - the following publications are incorporated herein by reference in their entirety.¹

Barker L.M., Hollenbach R. E., 1970. Shock-wave studies of PMMA, fused silica, and sapphire. *Journal of Applied Physics* 41 (10), 4208-4226.

Clifton R. J, 1993. Analysis of failure wave in glasses. *Applied Mechanics Review* 46 (12) Part 1, 540-546.

Clifton R. J., Mello M., Brar N. S., 1998. Effect of shear on failure waves in soda lime glass. *Shock Compression of Condensed Matter- 1997. The American Institute of Physics*, 521-524.

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Mittal K., 1987. Selected bibliography on adhesion measurement of films and coatings. *Journal of Adhesion Science and Technology* 1 (3), 247-259.

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Pronin A. N., Gupta V., 1993. Interferometry on diffuse surfaces in high-velocity measurements. *Review of Scientific Instruments* 64 (8), 2233-2236.

Raiser G. F., Wise J. L., Clifton R. J., Grady D. E., Cox D. E., 1994. Plate impact response of ceramics and glasses. *Journal of Applied Physics* 75 (8) 3862-3869.

Yuan J., Gupta V., 1995. The effect of microstructures and chemistry on tensile strength of Nb/Sapphire interfaces, with and without the interlayers of Cr and Sb. *Acta Metallurgica et Materialia* 43 (2), 781-794.

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Yuan J., Gupta V., 1993. Measurement of interface strength by the modified laser spallation experiment. Part I: Experimental technique and modeling the spallation process. *Journal of Applied Physics* 74 (4), 2388-2404.

Yuan J., Gupta V., 1993. Measurement of interface strength by the modified laser spallation experiment. Part II: Application to metal-ceramic interfaces. *Journal of Applied Physics* 74 (4), 2388-2404.

Yuan J., Gupta V., Pronin A. N., 1993. Measurement of interface strength by the modified laser spallation experiment. Part III. Experimental optimization of the stress pulse. *Journal of Applied Physics* 74 (4), 2405-2410.

Table I. Effect of Film Thickness on the Failure Mode in the Laser Spallation Experiment.

Configuration	Peak Interface Tensile Stress (MPa)	Peak Tensile Stress in the Substrate (MPa)	Failure Locus
Cu[100nm]/TaN[20nm]/Si	38±4	5100	Si Failure
Cu[500nm]/TaN[20nm]/Si	260±18	4900	Si Failure
Cu[1μm]/TaN[20nm]/Si	280±29	5200	Si Failure
Cu[5μm]/TaN[20nm]/Si	1380±110	4700	Interface Failure
Cu[10μm]/TaN[20nm]/Si	1370±140	4200	Interface Failure

Table II. Interface Tensile Strengths Measured Using Glass-Modified Stress Waves.

Sample No	Multilayer Configuration	Peak Interface Tensile Stress (MPa)	Peak Tensile Stress in Substrate (MPa)	Failure Locus
1	Cu(1400nm)/TiN(70nm)/Si	451	5340	Silicon failure
2	Cu(1400nm)/TiN(70nm)/Si/ Borosilicate glass	2620±210		Cu/TiN interface
3	Mo[400nm]/Borosilicate glass	577±47	2624	Mo/Borosilicate glass interface
4	Mo[1000nm]/Borosilicate glass	245±26	1050	Borosilicate glass Failure
5	Mo[400nm]/Borosilicate glass	577±33	2624	Mo/Borosilicate glass interface
6	Mo[400nm]/Pyrex	135±11	1125	Mo/Pyrex interface
7	Mo[400nm]/Quartz	99±8	1110	Mo/Quartz interface
8	Mo[1000nm]/Borosilicate glass	223±24	980	Borosilicate glass failure
9	Mo[1000nm]/Pyrex	53±6	470	Borosilicate glass failure
10	Mo[1000nm]/Quartz	48±4	460	Borosilicate glass failure
11	TiN [320nm]/Borosilicate Glass	164±11		TiN/Borosilicate glass interface
12	Ni [350 nm]/Borosilicate Glass	102±13		Ni /Borosilicate glass interface
13	Cr[400nm]/Borosilicate glass	197±16	2100	Cr/Borosilicate glass interface
14	Mo[1000nm]/Cr[400nm]/ Borosilicate glass	210±19	980	Cr/Borosilicate glass interface

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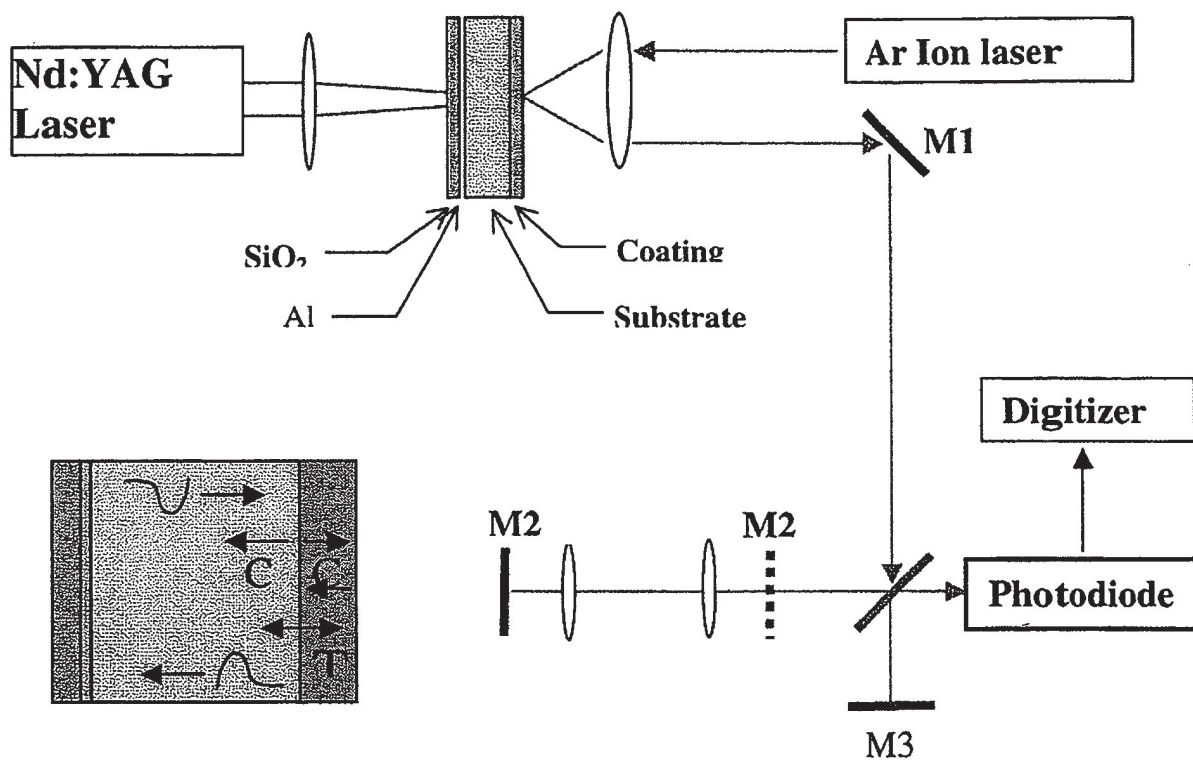


Figure 1. A schematic of the Laser Spallation Setup along with that of an interferometer used to record the free surface velocities of the film.

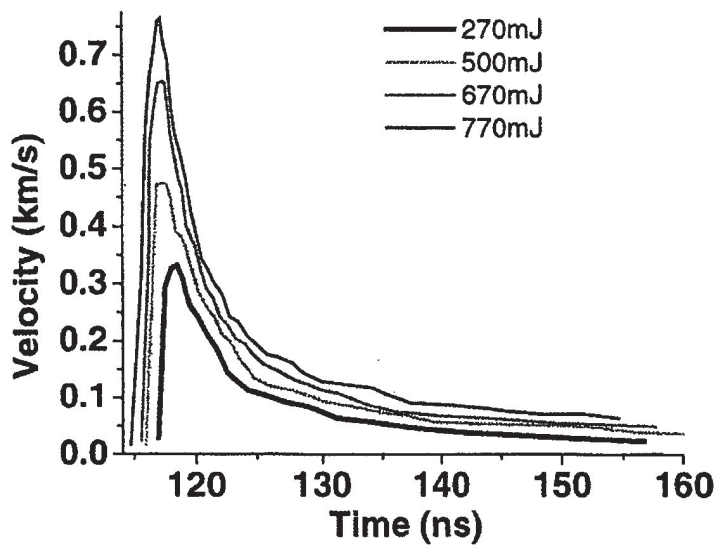


Figure 2. Measured stress pulse profiles in Si at increasing laser fluence.

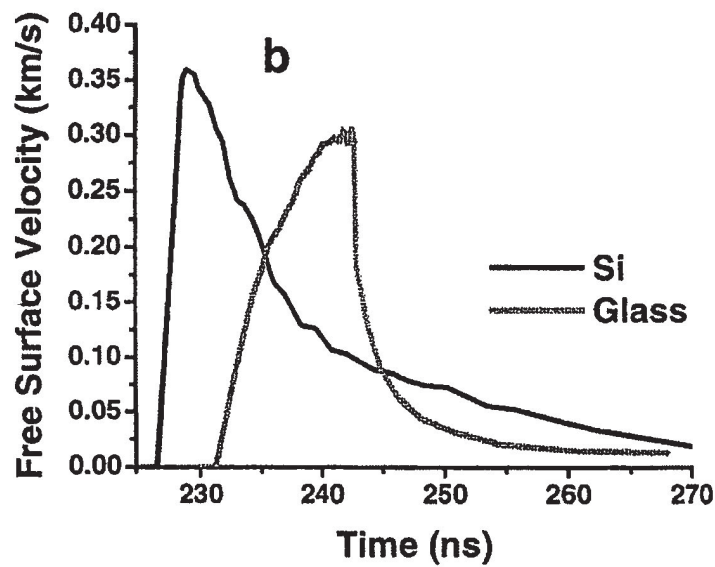
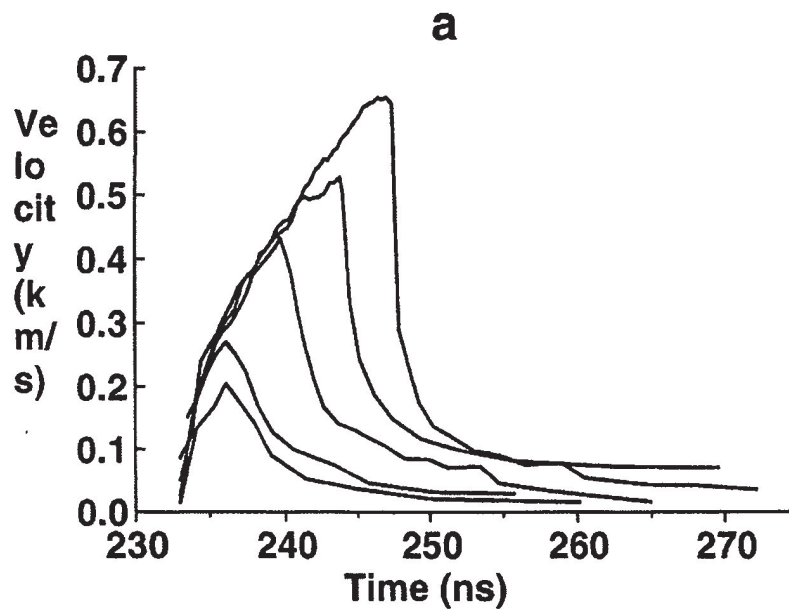


Figure 3 (a) Stress wave profiles in a soda lime glass under increasing laser fluence. (b). Comparison of one of the profiles in (a) with that measured in Si.

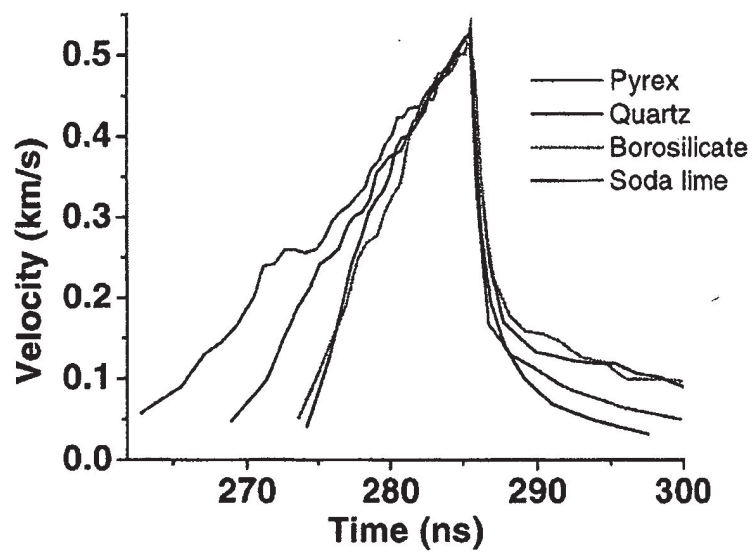


Figure 4. Measured stress wave profiles in different types of glasses.

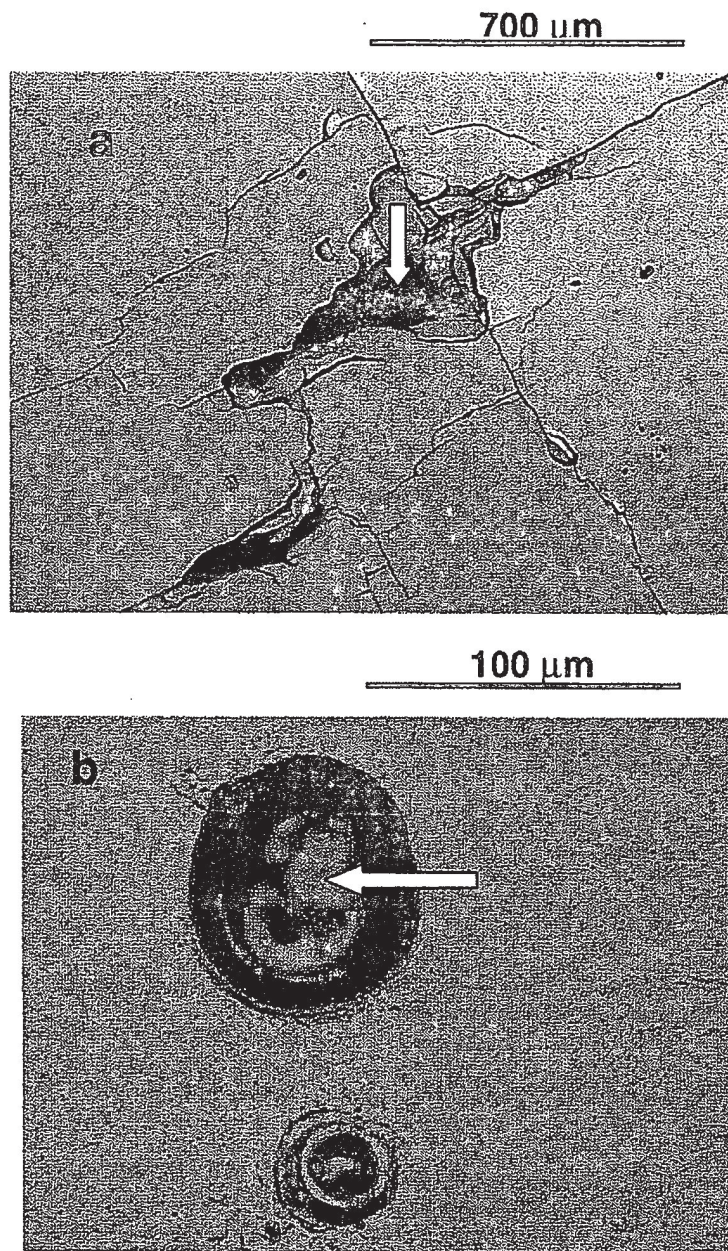


Figure 5. Failure in a Cu(1400nm)/TiN(70nm)/Si system. (a) With no glass attached to the Si, the failure occurred cohesively inside the Si. (b) With glass-modified wave, failure was observed at the interface failure as shown by the arrow. The failure was determined to be specifically at the Cu/TiN interface.

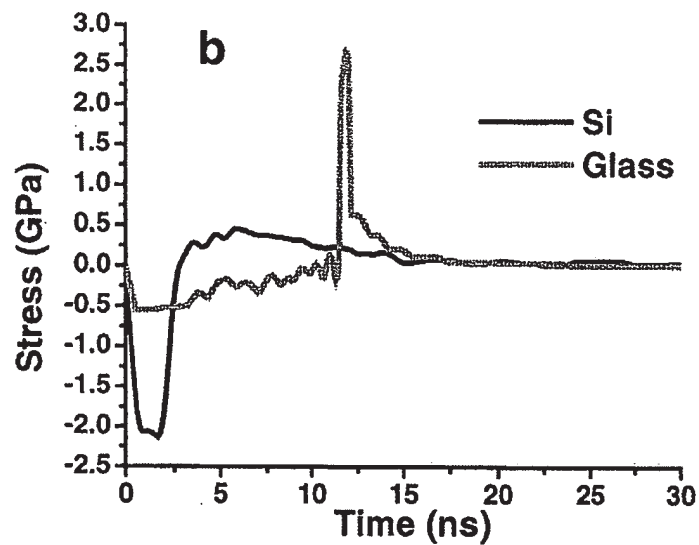
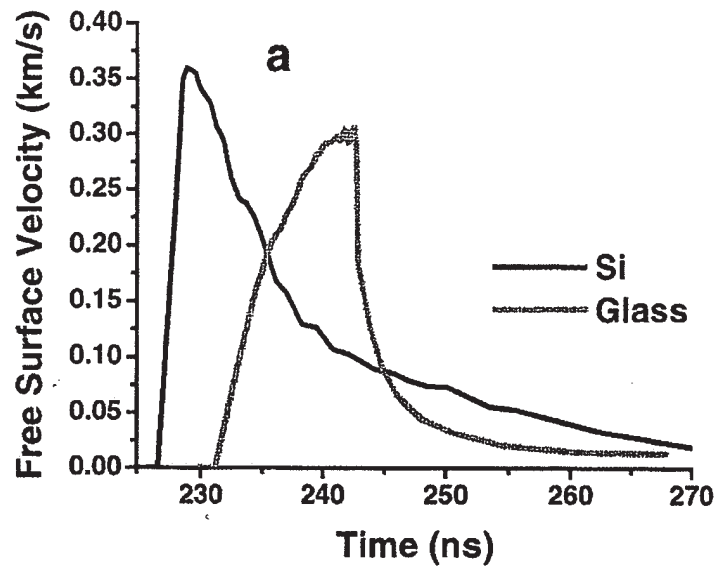


Figure 6. Free surface velocity profiles (a) and stress history at the Cu/TiN interface with and without the borosilicate-glass-modified waves.

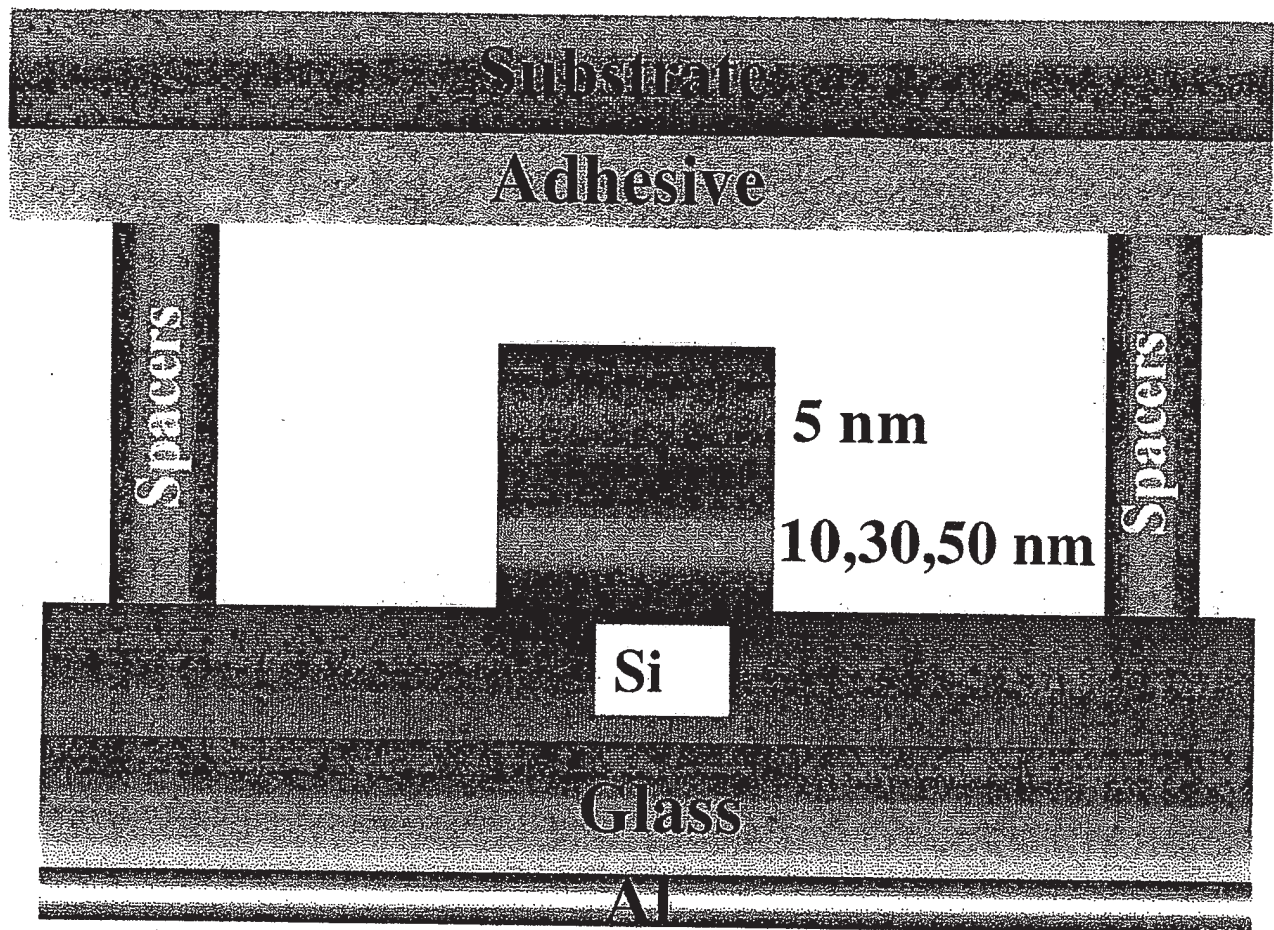


Fig. 7

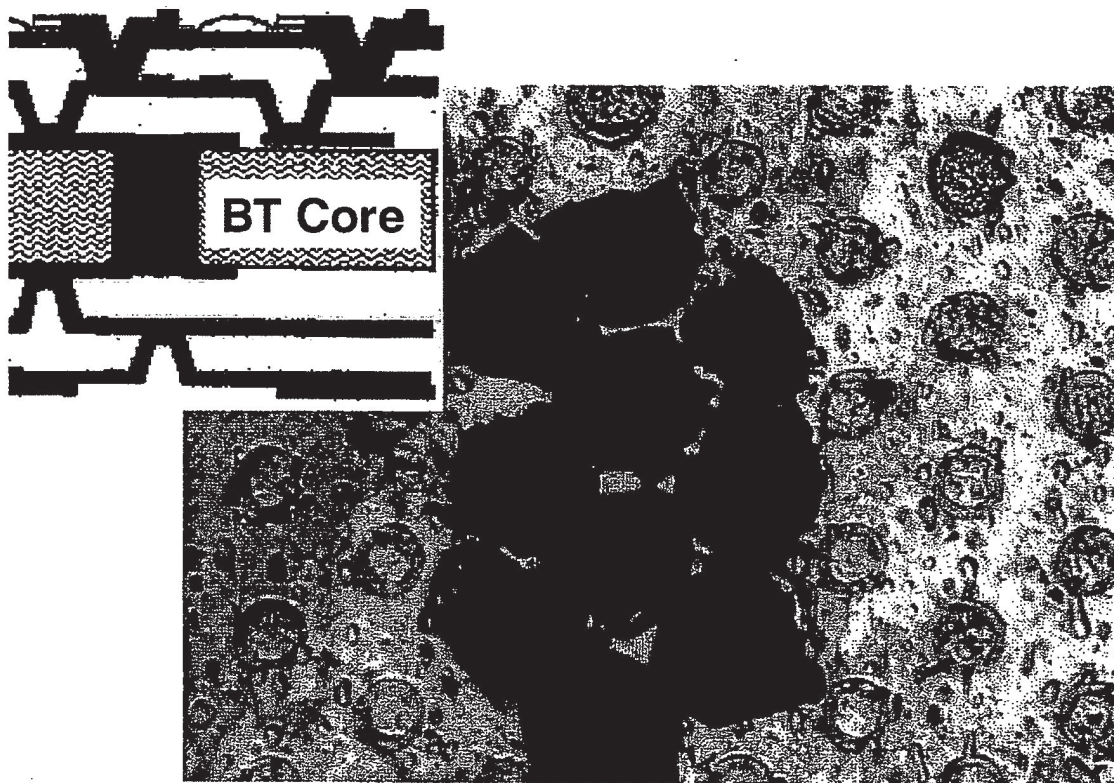


Fig. 8

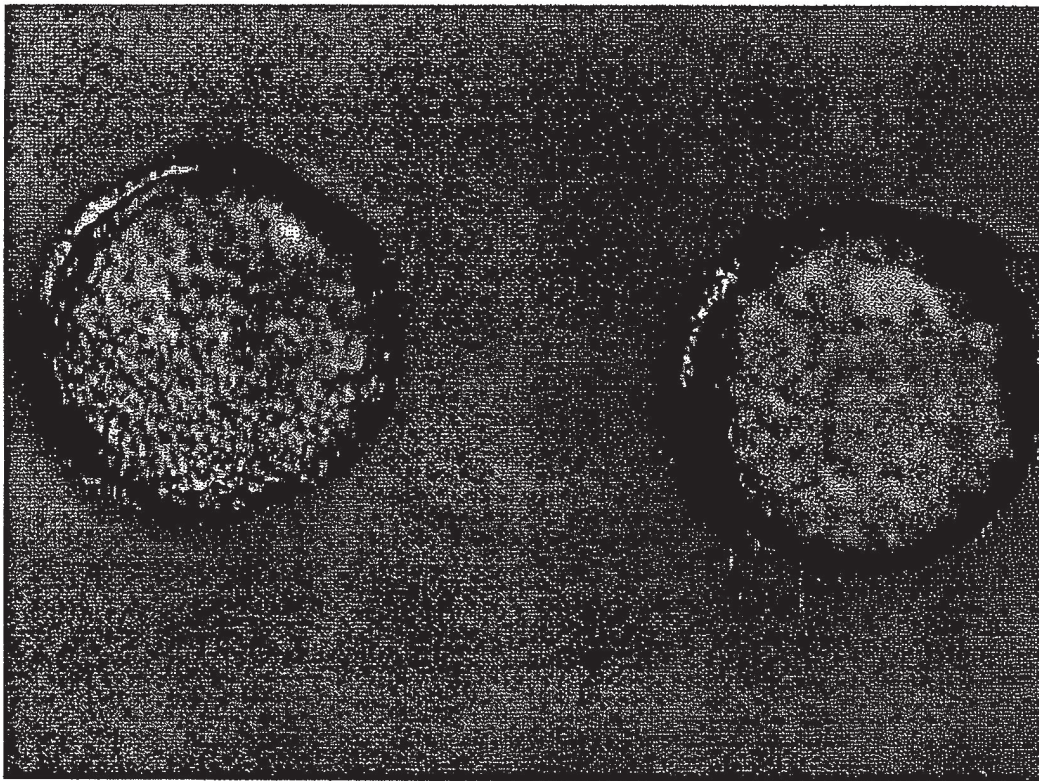


Fig. 9

Figure 10.

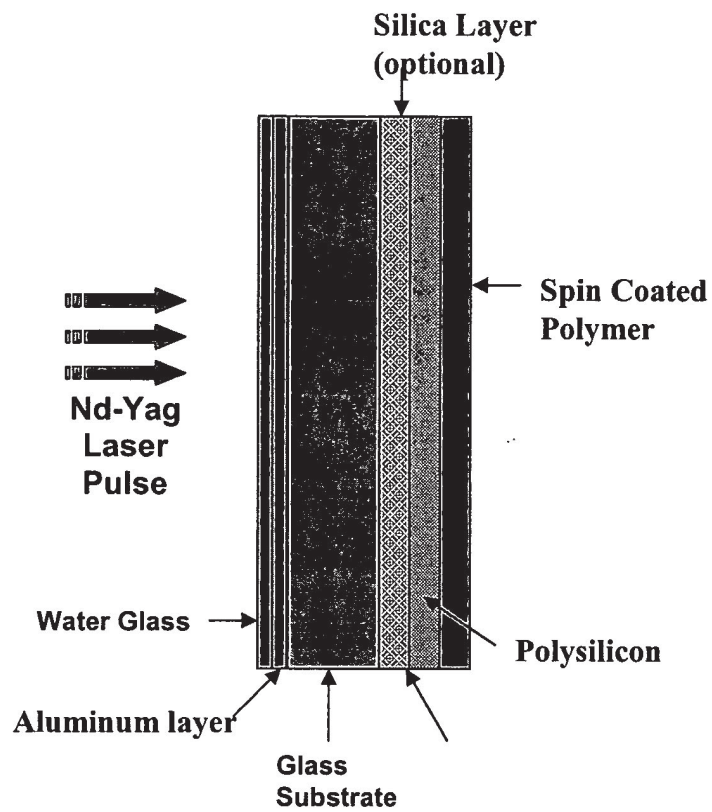
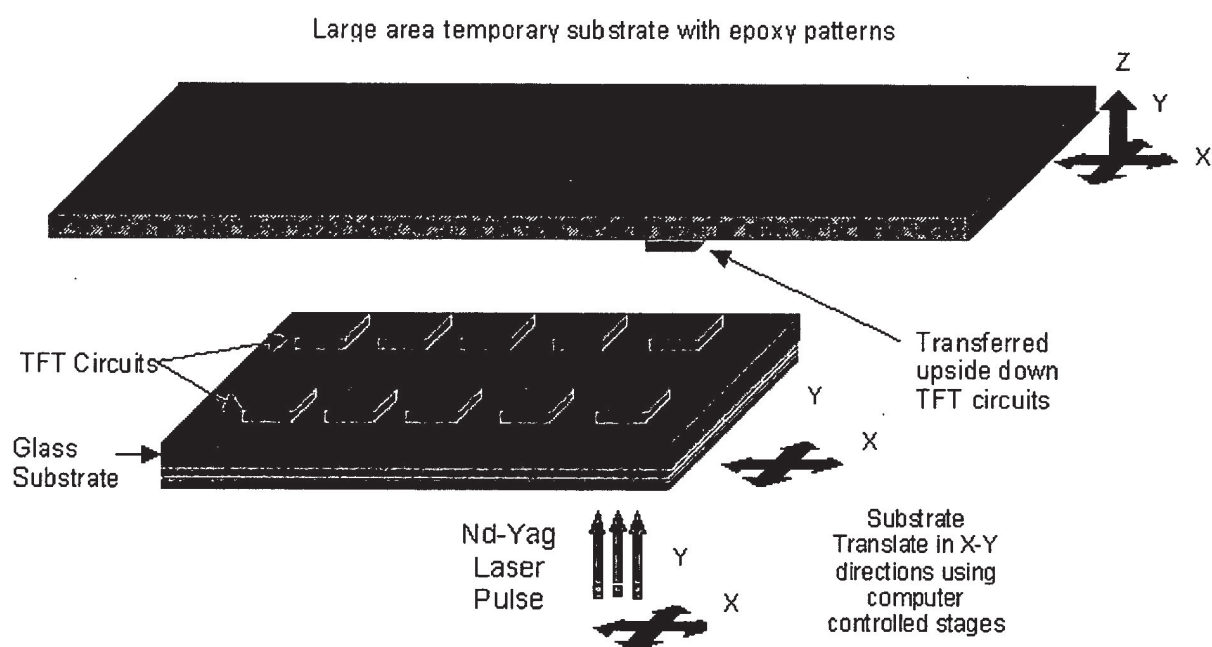


Figure 11.



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