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Characterization of mechanical and thermal properties of thin Cu foils and wires

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Abstract

There is an increasing necessity to record the deformation characteristics of microelements often consisting of freestanding foils and wires due to their increasing demand in engineering applications, where they are being used in the automotive industry, micromachines, special sensors, etc. The data required are either mechanical or thermal such as Young's moduli, stress–strain values, fatigue and thermal strain data. Due to the size effect these data cannot be taken from macrospecimens, thus special testing procedures were introduced. For the mechanical properties tensile tests were used for the freestanding foils and wires. Laseroptical sensors based on the speckle correlation method were applied to determine non-contacting strain values with high strain resolution. Equipped with a special air furnace mechanical data could be obtained up to 300 °C. For thermal strain measurements a laser speckle based dilatometer (LSBD) was designed. The following materials data are presented and discussed: Thermal expansion data, mechanical and fatigue properties of Cu foils (electrodeposited (ED), rolled and annealed) and wires with varying thickness. A pronounced thickness effect was detected. The applicability and limitations of the testing equipment used are discussed. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Laser speckle strain sensor; Size effect; Thin foils; Microwires; Tensile properties; Fatigue; Thermal expansion values

1. Introduction

To increase the reliability of microelectronic parts it is frequently required to determine the mechanical and thermal properties of the used materials in its practical dimensions (e.g. as thin foils and films, thin wires, membranes). This holds true for the development of special materials for conductor path, integrated circuits, special sensors, micromachines being used e.g. in the automotive and aircraft industry [1]. Due to thermal cycling of these microelectronic parts fracture may be induced. To reduce the fracture sensitivity an optimization and matching of various material properties such as coefficient of thermal expansion (CTE), Young's moduli, stress–strain values, creep, fatigue and fracture data are required.

However, due to the known “size effect”, these data cannot be taken from bulk material [1]. It is well known that mechanical testing of parts of microelectronic systems being small in all dimensions is a difficult task. Since the interpretation of the tensile test is simple, this testing technique was selected in this investigation although it is

known that specimen loading, handling and alignment require considerable experimental experience. For the determination of strain non-contacting laser speckle sensors based on the correlation technique [2,3] were applied, since strain measurements from cross-head movement are not accurate enough and conventional strain testing devices cannot be applied to specimens with small dimensions.

Based on the fact that most mechanical studies are related to thin film behavior (thickness in the range of less than a few micrometers, mostly attached to a substrate) [4–6] experimental data of freestanding foils are scarce. However, there exists a demand to establish criteria of the transition of the mechanical and thermal properties between films and bulk material. Thus, in this investigation the elastic, plastic and fracture properties of Cu foils in various conditions and thickness have been determined in continuation of previous work reported [7].

Because thin foils in microdevices are subjected to thermomechanical loading the effect of test temperatures up to 300 °C on the elasto-plastic behavior was investigated additionally [8].

Several data on the size effect of thin wires are presented in the literature mainly on the torsion stress–strain response [9,10], limited systematic data are presented on tensile properties of freestanding wires, thus a similar study was

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performed on the effect of the diameter on the tensile properties of heat treated microwires.

Freestanding thin metallic foils being used in microsystems are frequently subjected to fatigue loading caused by thermal fluctuation and mechanical vibration as discussed in a review paper concerning the fatigue crack growth properties of metallic foils by Hadrboletz et al. [11]. Most data listed in literature are reported on stress amplitude (S)–life (N = number of loading cycles) curves (S – N curves) of foils and wires and due to the fact that the results are contradictory [12,13] this study includes fatigue studies of foils and wires with different thickness, similar texture and a defined grain-size/thickness ratio.

Special fatigue testing techniques were selected based on the experience reported in [11].

For a better understanding of the deformation damaging processes (e.g. interaction of microstructural features (dislocations, grain boundaries, etc.) special scanning electron microscopy (SEM) techniques in combination with electron channeling contrast imaging (ECCI) are introduced [14].

Furthermore, thermal strain data of various Cu foils have been acquired using a special designed laser speckle based dilatometer (LSBD).

In the summary, the test techniques used are discussed and evaluated. The mechanical data determined should form a link between thin films and bulk material and should allow to form a basis for a quantification of the size effect.

2. Experimental procedures

2.1. Tensile testing

Tensile properties have been determined by use of a microtensile testing machine (load capacity between 10 and 500 N) equipped with a laser sensor. The entire device was mounted onto an optical table to avoid the influence of detrimental vibrations. The foils as well as the thin wires are glued to specially designed grips and have been aligned using a x – y die. The foil and wires have been used with a baselength between 20 and 40 mm. To record strain contactless an optical method using laser speckles (for a detailed description [2,3]) was used. For holding the test temperature constant a regulated stream of heated air was blown around the sample the temperature during the test had a maximum error of $\pm 5^\circ\text{C}$.

Laser speckles form when coherent light is reflected off a surface whose roughness exceeds a half wavelength. For practical purposes the surfaces of most materials are rough on this sub-micrometer scale of the wavelength of visible light (roughness about 100 nm and more), thus they yield visible speckles if illuminated. They are formed by the interference of de-phased but coherent wavelets emanating from different microscopic elements. Given constant illumination and observation geometry a particular subjective speckle pattern (which is characteristic for a certain

(imaged) surface element) moves in a well defined way when that surface element is displaced. This behavior permits to determine the relative displacement of surface elements of material specimens before and after loading by simply tracking the movement of the speckles associated with it. In the present study, a digital laser speckle correlation technique was used. The resulting displacement vector is derived from the location of the maximum of the digitally calculated two-dimensional cross-correlation estimate [2].

The optical arrangement to determine strain within a specimen is presented in Fig. 1 schematically.

In order to determine strain ε of the specimen two surface elements of defined distance l_0 are tracked. By determining the surface element displacements Δ_{surf} , taking their spatial difference Δl and dividing by a selectable baselength l_0 any strain value $\varepsilon = \Delta l / l_0 = (\Delta_{\text{surfII}} - \Delta_{\text{surfI}}) / l_0$ can be determined.

The technical arrangement of the laser set-up consists of an illuminating system and two displacement recording systems. The illumination consists of two collimated laser diodes with maximum power output of 15 mW and a wavelength of 680 nm providing beams each of 3 mm in diameter. The displacement recording is performed by two lenses (separated by the baselength l_0) with a selectable focal length and standard charge-coupled device (CCD) cameras feeding the signals of the images into a personal computer-based frame grabber, where the signal processing occurs.

For a selected baselength of 20 mm a strain resolution of 2×10^{-5} can be achieved. Since loading of the specimens may cause plastic deformation, phase transformation etc. distortions of the speckle patterns occur resulting in decorrelation effects. To overcome such effects a repetitive reinitialization of the image acquisition system is performed [2]. For low cycle fatigue (LCF) testing a similar optical arrangement using optical Fourier-filtering in combination with a line camera was applied suitable for measurement rates up to 100/s. The basic principle is being demonstrated in Fig. 2, a detailed description is presented in [15].

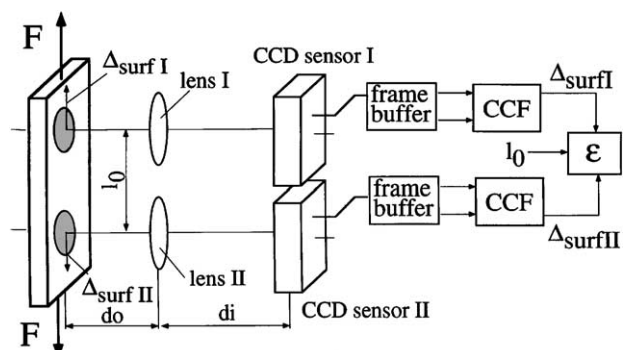


Fig. 1. The optical arrangement of the laser speckle correlation system.

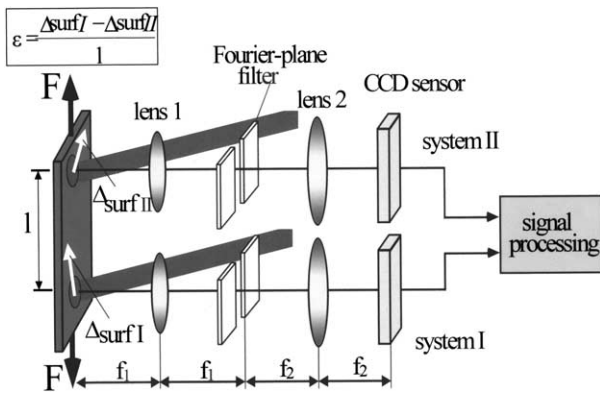


Fig. 2. Typical four- f arrangement of a cross-correlation based strain sensor.

2.2. Fatigue testing

For LCF and high cycle fatigue (HCF) testing of free-standing thin foils and wires two test facilities have been introduced: To obtain data on the cyclic stress–strain response of thin foils and LCF data, rarely found in literature, a specially adapted servohydraulic testing machine (type: Shimadzu with a loading capacity of 500 N) was used. The specimen geometry was 60 mm × 10 mm. Hysteresis (stress–strain) loops were measured using with the

described laser sensors. The plastic strain was deduced from the width of the hysteresis loops. The loading applied was tension–tension and the used testing frequencies up to 10 Hz.

For HCF studies of thin wires a resonance system operating at 20 kHz being described in [11] was used and the wires were glued onto a bar shaped holder of high strength Al alloy containing a small notch. Thus, the freestanding microwires were subjected to tension–compression loading with zero mean stress. Details, the calibration of the system and the determination of strain is described in [11]. The tested specimens were investigated with the ECCI technique in a scanning electron microscope to reveal the global dislocation arrangement.

2.3. Thermal strain measurements

For measurements of the thermal strain of thin foils a LSBDD was designed and adapted to a closed specimen chamber see Fig. 3. In this chamber of a proportional integral differential (PID) temperature controlled furnace the samples are heated up to 300 °C. Thermocouples were used to determine the specimen temperature with an error of ±0.5 °C in thermal equilibrium. With the laser speckle correlation technique, as described before (Fig. 1), the strain of the sample is measured in steps of 20 °C after reaching

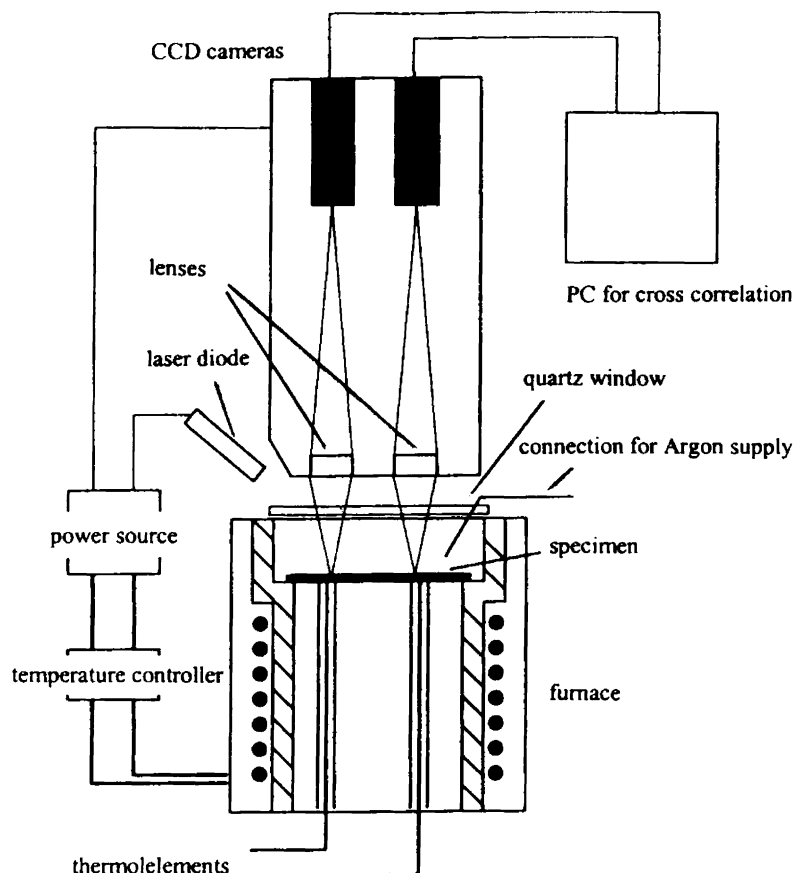


Fig. 3. Set-up of a LSBDD.

Table 1
Microstructural parameters and some mechanical properties of copper foils and wires

Material	Thickness (μm)	Heat treatment	Grain size (μm)	Young's modulus (GPa)	R_m (MPa)	A (%)	Texture
Cu foil	50	600 °C/2 h	45	90	161	15	Cube + twin texture
Cu foil	125		40	95	184	27	
Cu foil	250		45	105	199	36	
Cu wire	50	600 °C/2 h	25–50		165	12	Fibre
Cu wire	122	900 °C/2 h	60–122		186	16	
Cu foil,ED	35	180 °C/30 min	0.3	105	–	–	random
Cu foil, rolled	35	180 °C/30min	4.5	100	–	–	$\langle 1\ 0\ 0 \rangle$ and $\langle 1\ 1\ 1 \rangle$

temperature equilibrium for 5 min. The sample chamber can be floated with argon gas to avoid surface oxidation of the samples, which is especially important beyond 200 °C. With this set-up, thermal strain data of thin foils can be measured with a reproducibility of 1.5% between different runs on one sample.

3. Results and discussion

All materials used in this investigation have been produced industrially by rolling or electrodeposition (ED). The effect of thickness of selected rolled and annealed Cu foils (99.95%) (thickness varied between 50, 125 and 250 μm) on

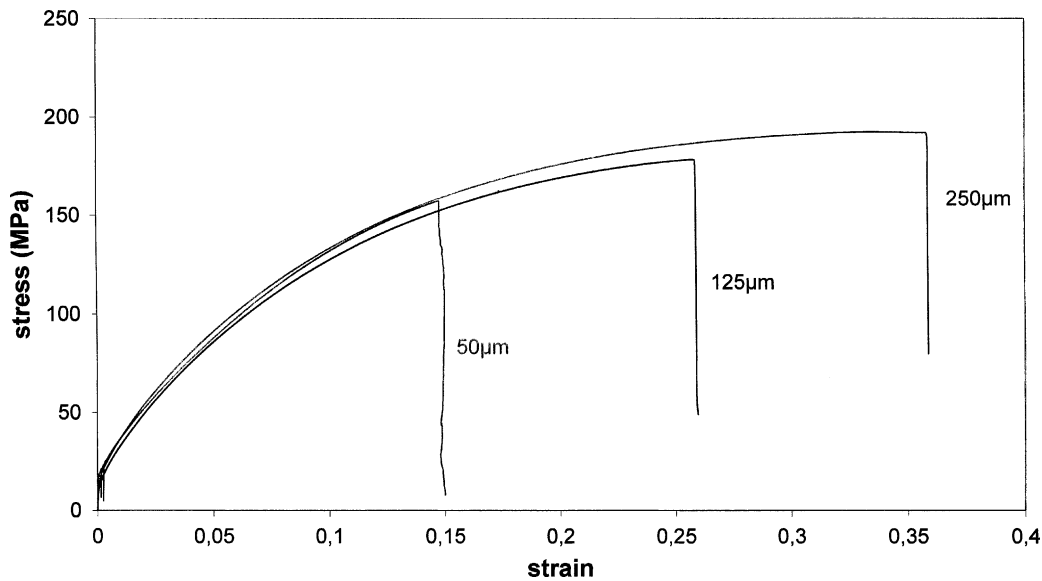


Fig. 4. The effect of foil thickness on stress–strain curves of Cu.

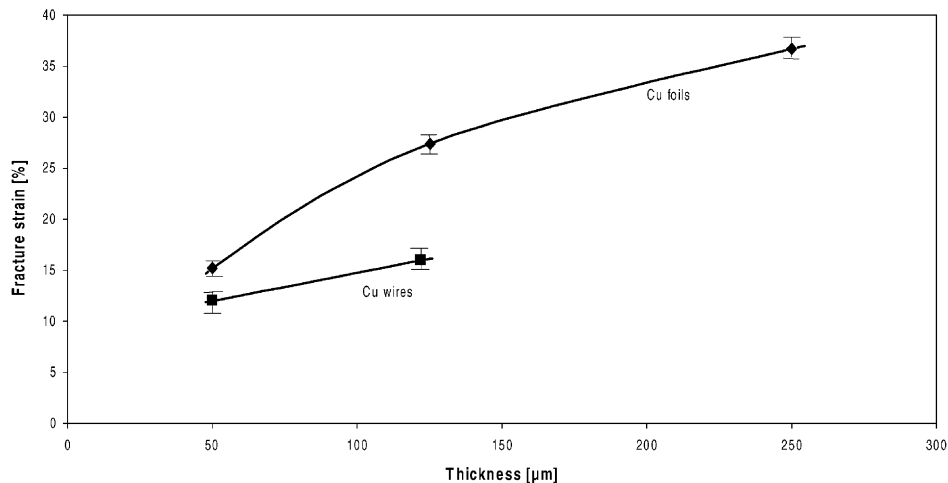


Fig. 5. Fracture strain of Cu foils and wires as function of thickness.

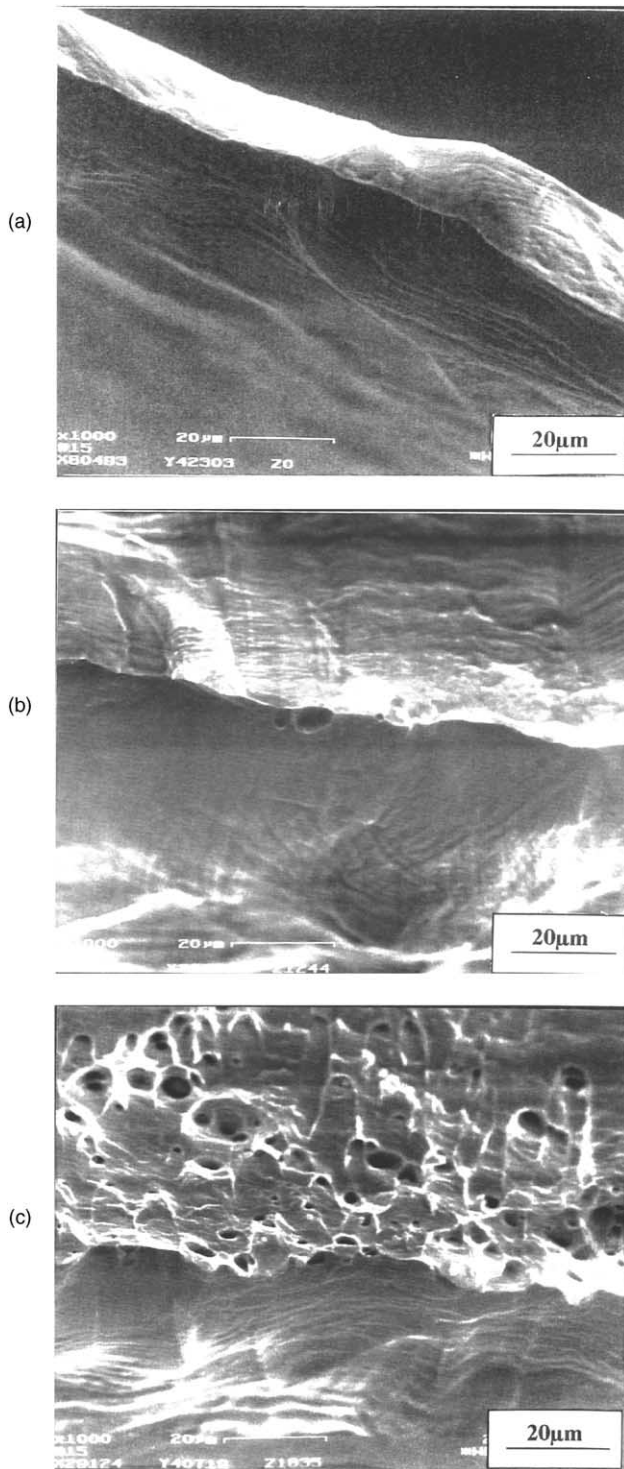


Fig. 6. SEM-fractographs of recrystallized Cu foils tensile tested; (a) 50 μm ; (b) 125 μm ; (c) 250 μm .

the mechanical response was investigated. For a detailed investigation using a laser speckle strain sensor for uniaxial testing see [7]. The heat treatment, microstructural information and basic mechanical data (Young's modulus, ultimate tensile strength (R_m) and total strain (A)) are given in Table 1.

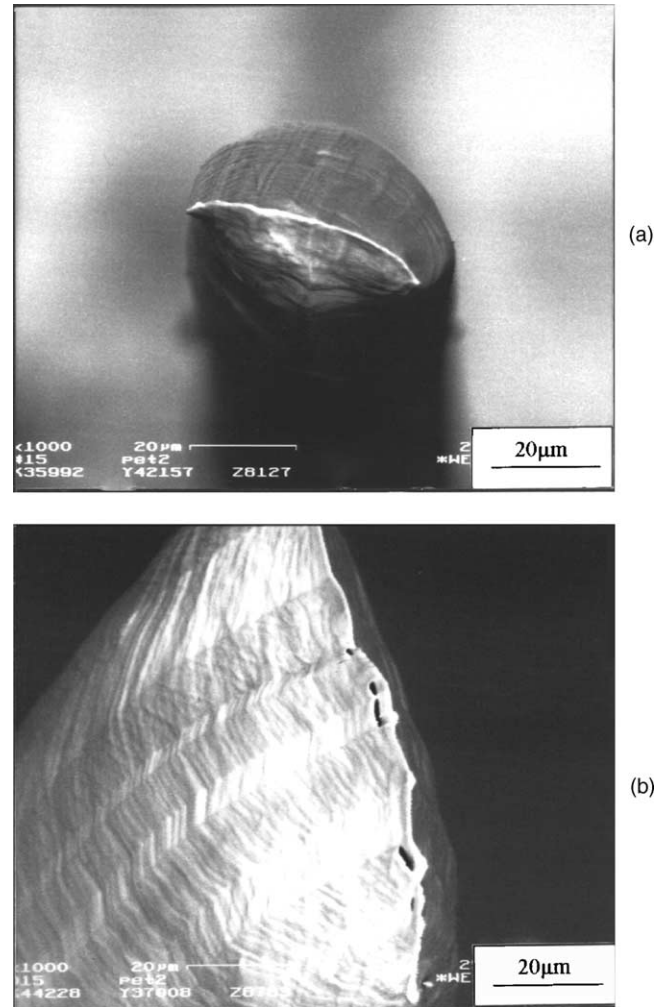


Fig. 7. SEM fractographs of recrystallized Cu wires tensile tested: (a) $d = 50 \mu\text{m}$; (b) $d = 122 \mu\text{m}$.

Special emphasis was placed on the heat treatment to obtain a single grain layer in the thinnest samples and a gradual transition to the case of several grains across the foil thickness for the thicker samples in order to detect the characteristic differences of the mechanical performance in those cases. In addition, the heat treatment was also selected to obtain a similar cube texture within all foils as reported in Table 1.

To study the effect of test temperature on the yield strength special foils with thickness of 35 μm and different conditions were selected. The mechanical behavior of an electrodeposited (ED) Cu foil with a very fine grain size of 300 nm was compared with a rolled and heat treated material with a grain size of 4.5 μm . For a detailed description of these materials and testing see [8].

The stress strain curves of the annealed Cu foils with varying thickness obtained with the non-contacting laser-optical strain sensor are presented in Fig. 4. An evaluation of the yield strength, strain hardening coefficient and fracture strain showed a pronounced effect of thickness on the

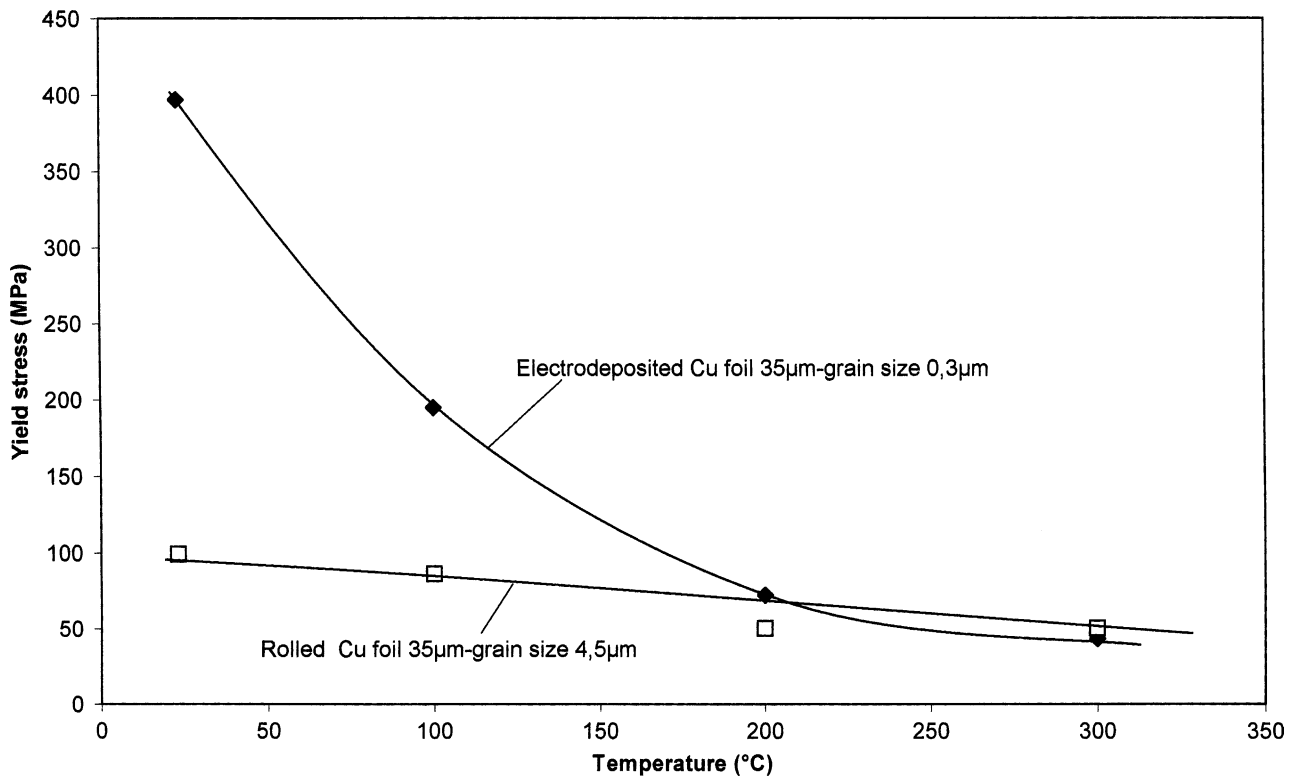


Fig. 8. Effect of temperature on the yield stress of 35 μm thick Cu foils in different conditions and with various grain sizes.

fracture strain only as exhibited in Fig. 5, which is qualitatively in agreement with previous measurements [7]. In comparison to bulk material with a fracture strain of 50%, the fracture strain of foils are generally smaller. It may be hypothesized that the observed size effect is attributed to the transition (thickness in the order of 150 μm) of plane

stress to plane strain conditions due to the constraint of the strain component in the direction of foil thickness. This may result in a reduced activation of gliding systems. This reasoning is supported by the appearance of typical fracture topographs changing from a knife edge rupture type to a typical ductile fracture morphology showing voids and

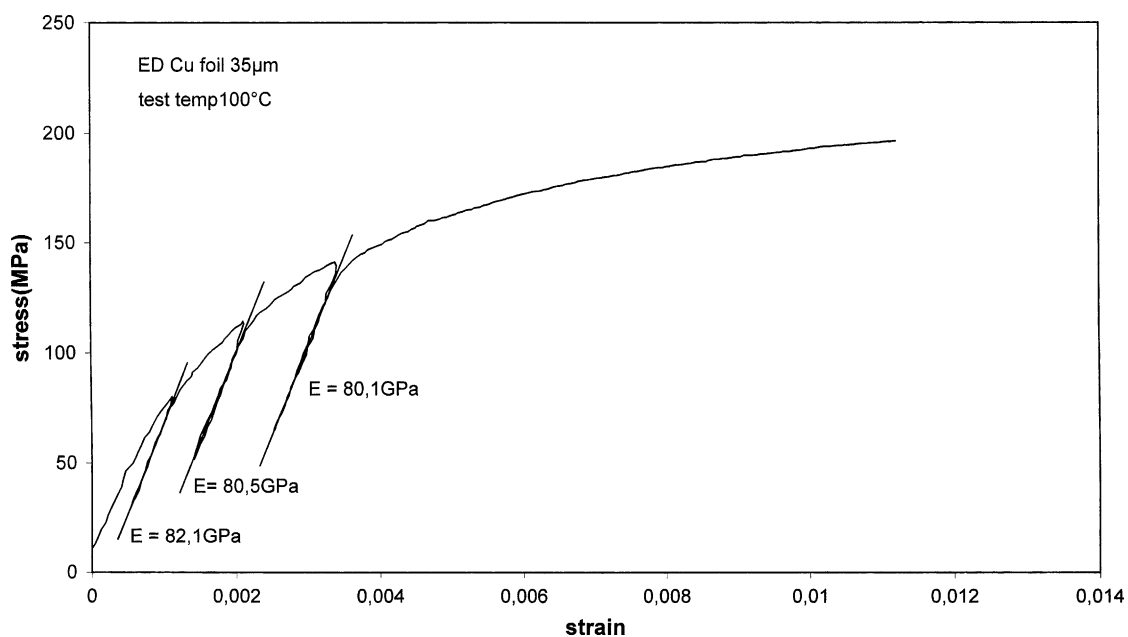


Fig. 9. Loading–unloading procedure for the determination of the elastic modulus of a 35 μm thick Cu foil.

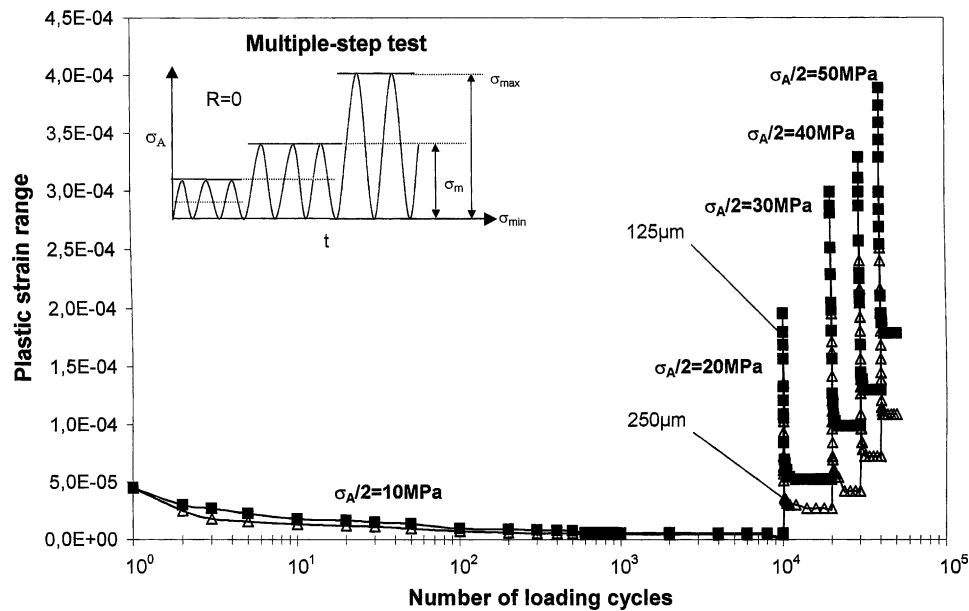


Fig. 10. Multiple step test of two Cu foils with different thickness (loading conditions: $R = 0$).

dimples typical for thicker foils with numerous gliding systems as shown in Fig. 6. However, the interaction of grain sizes to foil thickness have also to be considered.

A similar study was performed with thin wires with diameters ranging from 50 to 122 μm . The microstructure was of the bamboo-structure type with about one grain across the diameter. The texture was mainly a fibre texture as reported in literature. A discussion on the texture evaluation of thin wires and foils are given elsewhere [16].

The evaluated data of fracture strain, are also presented in Fig. 5. The elasto-plastic response of the microwires is similar to that of the foils. However, it may be hypothesized that the transition thickness (plane stress to plane strain) for microwires is different to the foils. The difference in the stress conditions may result in the different slip activity. The SEM fractographs Fig. 7 of the tensile fractured wires show a typical knife edge rupture indicating slip bands and twins.

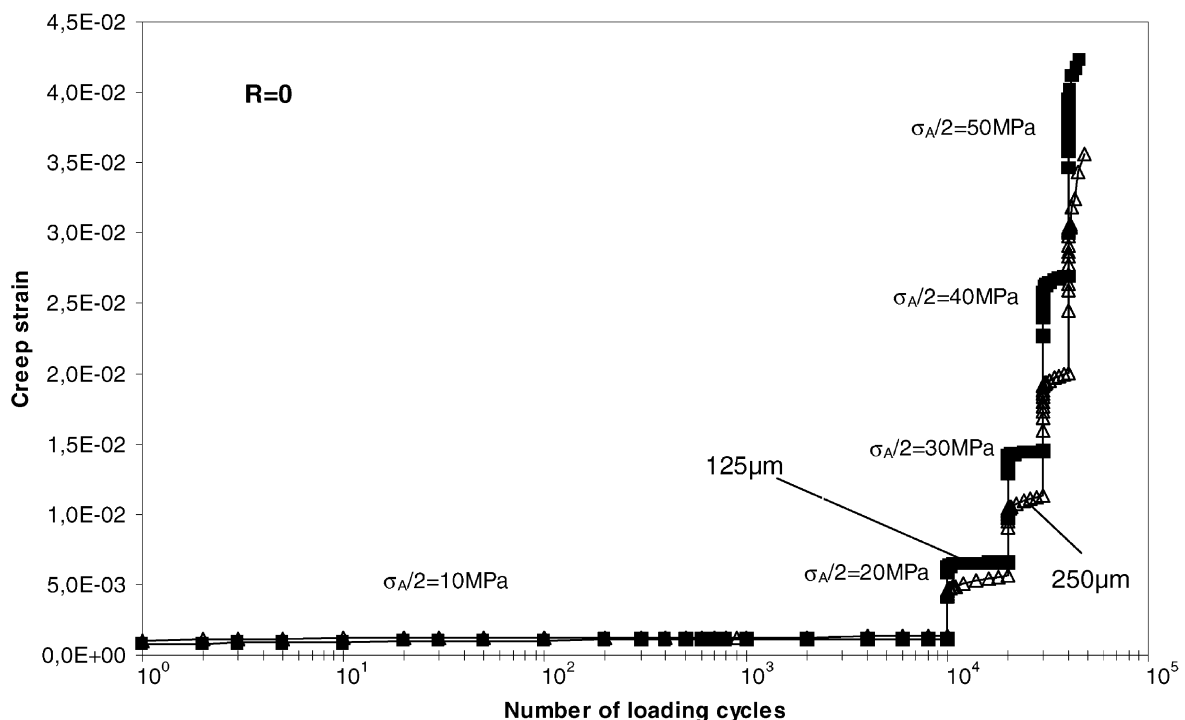


Fig. 11. Creep strain of two Cu foils with different thickness during the multiple step test (loading condition: $R = 0$).

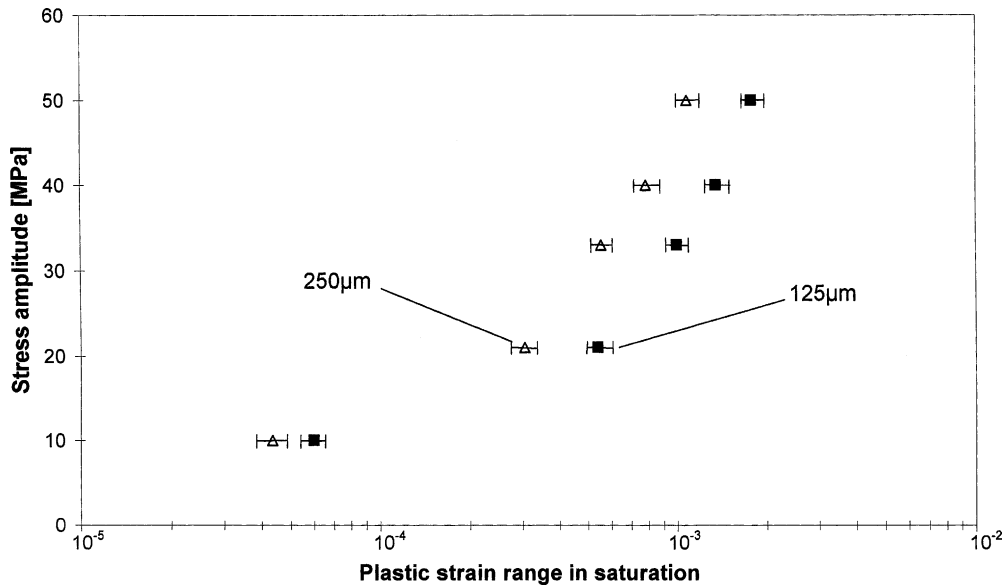


Fig. 12. CSS curves of recrystallized Cu foils with different thickness at $R = 0$.

The effect of test temperature indicated a strong effect on the yield stress for the ED foil compared to the rolled foil as depicted in Fig. 8. An explanation is given on the basis of changes in the microstructure, mainly resulting in grain growth of the ED foils as discussed in detail in [8,17].

Due to the well known high amount of vacancies in ED materials [20], the formation of vacancy clusters and numerous small voids at grain boundaries formed during testing the strong reduction of yield stress may be explained. In contrast, the rolled foils indicate only minor changes of the yield stress as expected.

Because of the high strain resolution the laser speckle technique is especially suited for the determination of the Young's modulus (E) of thin foils and wires, however, great

care has to be taken on the alignment and the design of the gripping systems. The values are determined by a loading–unloading technique as shown for a 35 μm thick ED foil tested at 100 $^{\circ}\text{C}$, see Fig. 8. Almost parallel lines in the stress–strain diagram were obtained showing a high degree of strain resolution, resulting in a Young's modulus value of 80 GPa with an error of measurement smaller than 5% (Fig. 9).

3.1. Fatigue properties

To obtain a deeper insight into the fatigue mechanism of thin foils cyclic stress strain (CSS) curves were determined as function of foil thickness. It is well known that the CSS

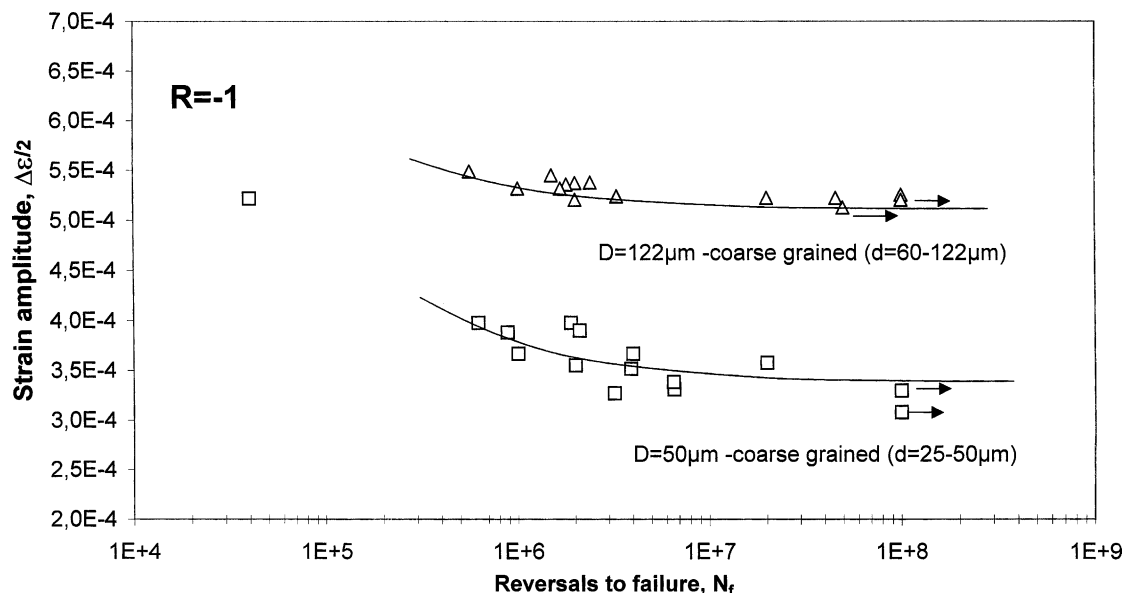


Fig. 13. S – N curves of Cu wires with different diameter. Test conditions: $R = -1$, 20 kHz.

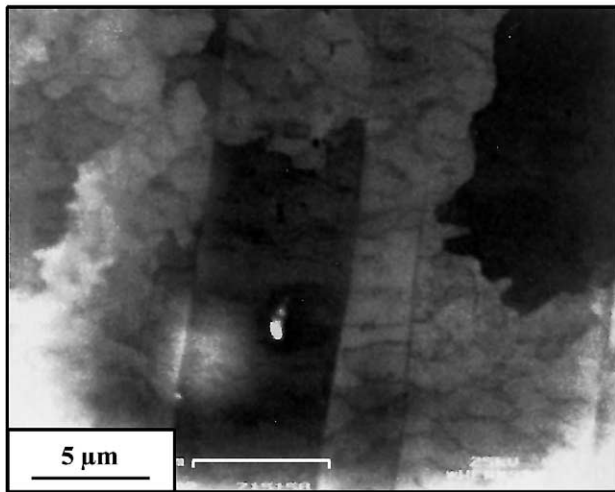
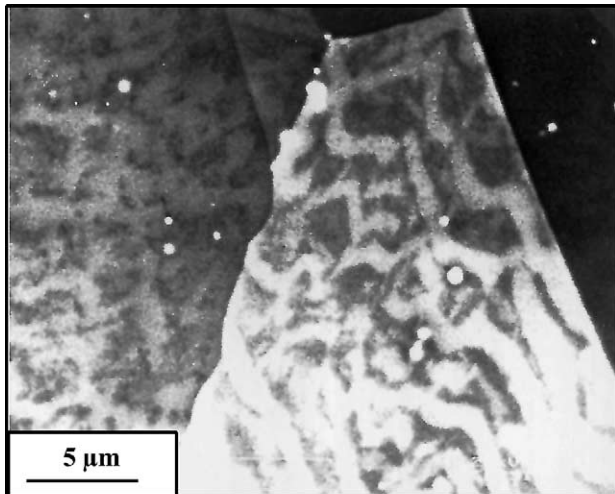


Fig. 14. Typical dislocation arrangement in a fatigued, recrystallized Cu wire (122 μm) obtained with the ECCI technique: $R = -1$, $N = 7.7 \times 10^5$, $\varepsilon_f = 5.4 \times 10^{-4}$.

curve reflects the dependence of the stress amplitude on the plastic strain amplitude in the stabilized state (for details see [18]). Using a multiple step test as shown in Fig. 10, (the specimens are loaded with different stress amplitudes and are cycled until saturation in plastic strain is observed. These values are taken for the determination of the CSS curve. With increasing amplitudes the cyclic hardening of two rolled and heat-treated Cu foils with thickness of 250 and 125 μm , respectively, were investigated under tension–tension loading (parameter $R = 0$) and a testing frequency of 0.05 Hz. Figs. 10 and 11 indicate both the changes of plastic strain and creep strain during the multiple step test. As can be seen if only fatigue hardening occurs no creep can be observed this changes drastically if significant changes (for $\sigma_A/2 > 20 \text{ MPa}$) in plastic strain occurs. As well the plastic strain as creep strain increases with smaller foil thickness.

The CSS curves for both foils are presented in Fig. 12. It may be deduced that the thinner foil shows a higher degree

of plastic strain and creep strain. As discussed in detail [16], the results indicate that fatigue loading of thinner foils results in less dislocation activity due to the difference in the stress state, however the ratio grain size/thickness have to be taken into account.

Fig. 13 represents the S – N data after symmetrical tension–compression loading (parameter $R = -1$) of thin wires with different diameters and a similar grain size-to-diameter ratio. The results indicate a reduced fatigue life for the thinner wires possibly due to a reduction of gliding systems.

A study of the global dislocation structure of the fatigued wire is shown in Fig. 14, indicating typical fatigue induced structures (ranging from veins to cells, depending on the grain orientation) for symmetrical loading.

3.2. Thermal strain data

For testing the measurement reliability of the LSB, the system was calibrated against the US National Institute of Standards (NIST) standard reference material (SRM) 736 (6 mm bulk copper), Fig. 15. For strain measurements up to 200 $^{\circ}\text{C}$ very good agreement between the measured values and the reference data could be achieved.

Slightly larger deviations are observed in the region above 250 $^{\circ}\text{C}$ due to foil oxidation. This problem can be avoided, when the runs are carried out under argon atmosphere.

The strain data were fitted with a fourth-order polynomial to determine the temperature dependence of the coefficient of thermal expansion (CTE). Fig. 16 shows the CTE behaviour of two 35 μm copper foils, one ED and one rolled copper foil (Table 1), compared to the CTE reference data of the NIST standard. The grain size for bulk material is not available but it should be at least an order of magnitude higher than the grain size of the foils [8]. It can be seen, that the CTE for foils is initially higher than that of bulk copper at room temperature. The deviation between bulk and foil material becomes especially pronounced with increasing temperatures. A rapid rise of CTE above a critical temperature somewhere between 100 and 150 $^{\circ}\text{C}$ can be observed. This remarkable aspect of temperature was also ascertained for other foil types. However, both Young's modulus (E) and CTE trends deviate for the foil and bulk material, the deviation is especially pronounced for CTE, and is attributable in part to the grain boundary contribution and a possible microcrack formation at voids due to thermal loading. This contribution increases with decreasing grain size [8]. A detailed interpretation will be given elsewhere [17].

4. Summary and conclusions

The laser based non-contacting strain sensor is due to the simple optical arrangement easily adaptable to various testing machines and characterized by high strain resolution in the order of 2×10^{-5} . This system allows to determine the

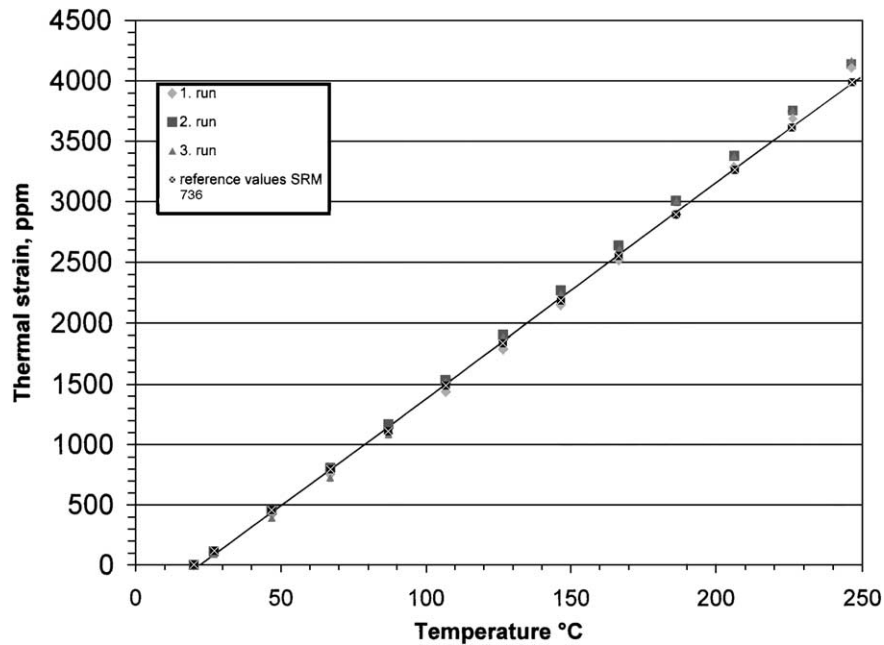


Fig. 15. Reproducibility test for three successive runs on NIST SRM 736. The solid line indicates a linear fit to the reference values of the copper standard. An average deviation between the different runs of about 1.5% could be achieved.

thermal and mechanical strain of thin foils films and wires of most materials, without any special surface preparation. Elastic properties such as Young's modulus—necessary for the characterization of thin foils—have been successfully measured with high accuracy using a microtensile machine in combination with the laser based strain sensor. Since the baselength is at present relatively large, structures with minimum size of about 15 mm at least in one dimension are required. Investigations can be performed over a wide

range of testing temperatures corresponding to real test conditions of the microelectronic devices.

A new LSBSD for determination of thermal strain values of thin foils is presented. This system is based on a non-contacting laser optical sensor using the laser speckle correlation technique without detecting any interference pattern. This method achieves sufficient degree of accuracy and precision for succeeding CTE evaluation in the temperature range from room temperature up to 300 °C and thermal

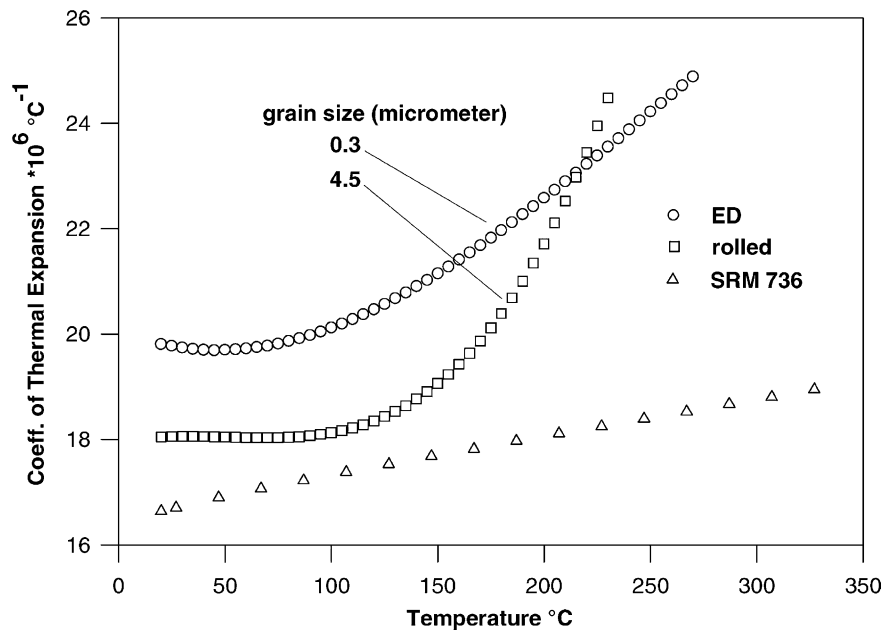


Fig. 16. Effect of temperature on CTE for two 35 μm copper foils, with different grain sizes, for comparison the CTE for SRM NIST SRM 736 (bulk material) is shown.

strain data of thin foils can be evaluated with a reproducibility of 1.5%.

Various applications in materials science like CTE evaluation of metals, polymers, ceramic components and multilayer-structures as well as detection of phase transitions will be possible with the advantage of a simple optical arrangement and low cost compared to other detection systems.

It is known, that detailed knowledge of the temperature dependence of CTE and Young's modulus values are necessary for reasons like finite element analysis of printed wire boards and determination of multilayer warpage [8,19].

A strong size effect could be detected especially concerning the fracture strain of thin metallic foils and wires. The change in stress state should be responsible and thus influencing the dislocation activity. This could explain for thinner foils their reduced plasticity, however, for a similar texture also the effect of grain size/thickness ratio have to be considered.

The data obtained from the tension test allow to design microcomponents subjected to complicated stress–strain states.

The pronounced influence of test temperature on the elasto-plastic response of foils should allow to optimize the selection of the foil material for printed wire boards and may be explained on the basis of grain growth and the special microstructure of ED foils.

Using a newly designed test set-up LCF and HCF fatigue data of free-standing thin metallic foils and wires under symmetrical and unsymmetrical loading conditions have been obtained. Also a strong size effect was detected, which can be explained with the corresponding stress state. The ratio of grain size-to-foil thickness for values in the order of unity should influence fracture and fatigue life, additionally.

In addition, the ECCI technique is feasible to record the global dislocation arrangement of thin structures without sophisticated preparation. This should lead to a better understanding in order to optimize the fatigue properties of the used materials.

The fatigue data obtained allow to establish microfracture criteria which are necessary to design reliable microelectronic components. This method can also be applied for the study of the fatigue behavior of multilayered structures.

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