

MECHANICAL PROPERTIES OF MEMS STRUCTURES

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ABSTRACT

Mechanical characterization of MEMS materials is increasingly important in view of improving reliability and assessing the life time of new devices. In this paper a number of testing methods are described. These methods include tensile, torsion and fatigue testing of specially designed microstructures, as well as wave propagation methods based on an optical pump probe setup to test thin films. Difficulties arise from manufacturing and handling of small structures and the determination of its geometrical dimensions, which directly affect the accuracy of material parameters extracted from the experiments. In addition the measurement of the mechanical parameters like small forces and torques or strains on small specimens or with ps time resolution pose challenges. This paper focuses on size effects in copper foils of thickness between 10 and 250 microns as determined from tensile testing and probing of inhomogeneities caused e.g. by diffusion at interfaces in thin films.

1. INTRODUCTION

With increasing use of miniaturized systems a strong need arises for the mechanical characterization of the materials used. In particular for polycrystalline materials it is often not possible to use properties obtained from macroscopic experiments, as the microscopic elements have particular microstructures, which are related to the special manufacturing processes used. Therefore, specimens need to be tested in their “small” configurations in view of measuring the relevant design parameters.

In this paper an overview of a set of measurement techniques is given, which complement the well known nanoindentation technique: Mechanical wave propagation techniques based on laser generated ultrasound for determining elastic properties, and tensile, torsion and fatigue tests with high crack resolution on specially manufactured microscopic specimens. These techniques are applied to probe material property gradients in thin film structures and to investigate size effects in thin rolled copper foils, respectively.

2. THIN FILM TESTING USING AN OPTICAL PUMP PROBE SETUP

When wave propagation techniques are to be used to determine elastic properties, very high frequencies need to be generated because of the small size of the specimens: The wavelength needs to be small compared to the relevant geometrical parameters. Such high frequency waves can be generated and measured in a contactless manner using a pump probe set-up with an ultra short pulse laser (Thomsen [1] and Fig. 1). In a project called *Nanosonic* an improved version of this set-up is used to characterize thin films, their adhesion to the substrate and material property gradients (Vollmann [2] and Fig. 2).

The laser acoustic method works in the thermoelastic region which means that there is no ablation and therefore the technique is nondestructive. A short laser pulse (800 nm, 70 fs), the

pump pulse, is absorbed at the metallic thin film surface and is initiating an elastic pulse which propagates into the film. Echoes, occurring at discontinuities of the acoustic impedances, are heading back to the surface and are causing a slight temporary modification of the optical reflectivity. The optical reflectivity at the surface is scanned versus the relative time shift to the initial pump pulse with a probe pulse, which is created by a partly reflecting mirror and which follows a different path in order to allow the control of the time shift between the excitational and the detecting pulse. The experiment is repeated constantly at a repetition rate of 81 MHz while the time shift is changed. The method was first presented by Thomsen [1].

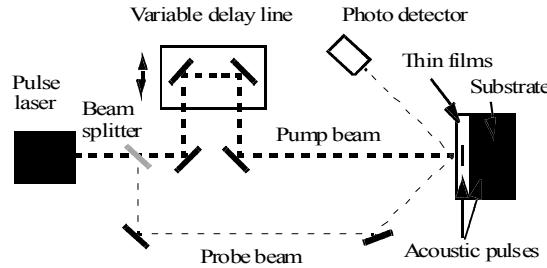


Fig. 1: Short-pulse-laser-acoustic setup (simplified).

A detailed analysis of the light-matter interaction for the excitation as well as for the photo acoustic detection is given by Profunser [3]. The main challenge lies in the reduction of optical and electronic crosstalk between the excitational and the detecting signal path. Therefore two modulation frequencies, cross polarization, and balanced photo detection is used. A detailed description of the improved set-up and of the signal processing is given by Vollmann [2].

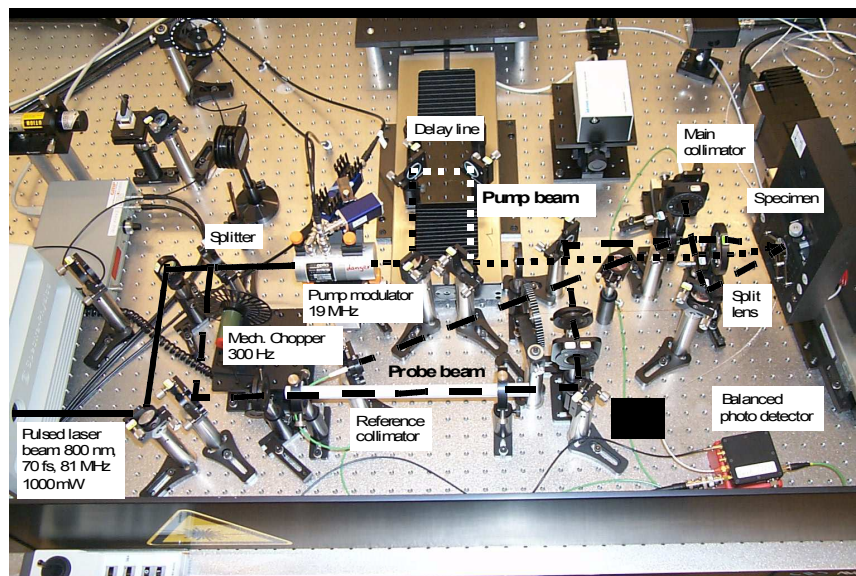


Fig. 2: Experimental set-up as used for the photo acoustic thin film profiling at the ETH Zurich.

The diagram of Fig. 3 shows two independently measured reflectivity curves compared with a simulation (top). The thicknesses of the multilayer are: 60 nm Al / 30 nm Au / 60 nm Al on a Al_2O_3 substrate.

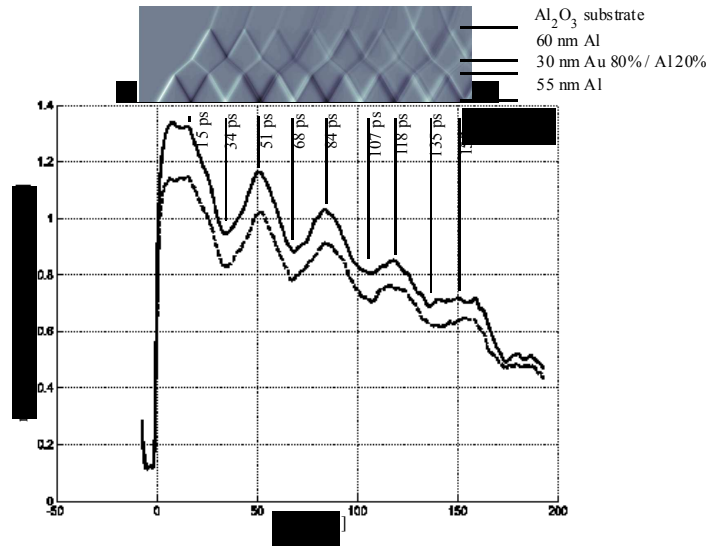


Fig. 3: Two photo acoustic measurements of a 60 nm Al / 30 nm Au / 60 nm Al multilayer on a Al_2O_3 substrate compared with a one dimensional numerical simulation.

Each curve represents an average of 50 individually measured curves. Two effects are governing the shape of a reflectivity change curve versus the time shift between the pump pulse and the probe pulse, which are presented in Fig. 3: The dominant effect is the initial jump of the reflectivity change caused by the local heating at the surface (time = 0 ps in Fig. 3). Proportionally to the heat conduction into the surrounding area, the reflectivity change decays nearly exponentially. This is due to the fact, that the heating pump pulse is much shorter than the thermal relaxation time. Superimposed to this thermally caused effect, one can see the periodic alternation of the optical reflectivity change which is caused by the stress pulse echoes reaching the surface. The strain pulse, created by a pump laser pulse for the parameters used in the photo acoustic set-up, is calculated according to Profunser [3]. In order to demonstrate the dependence of the acoustic reflection/transmission ratio on the wavelength/'smooth'-acoustic-interface-thickness ratio experimentally, the thickness of the 'soft acoustic interface' needs to be varied. This has been realized by the exposure of a series of equally manufactured specimen to different temperature treatments, which leads to different grades of thermally induced intermetallic diffusion.

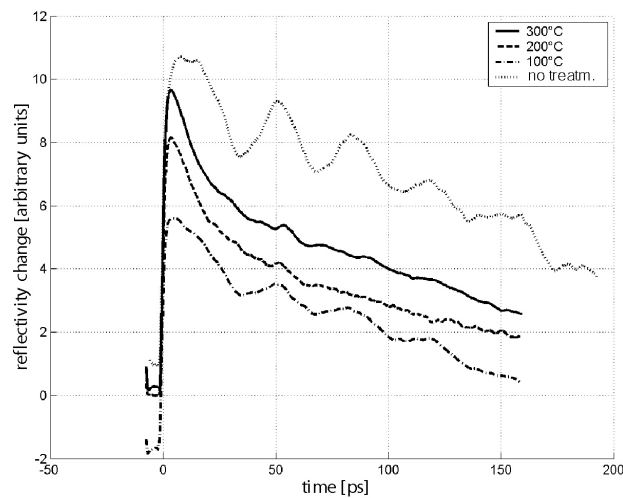


Fig. 4: Photo acoustic measurements of four 60 nm Al / 30 nm Au / 60 nm Al multi layers on a Al_2O_3 substrate which were exposed to different temperatures in order to 'smoothen' the acoustic interfaces by thermally induced intermetallic diffusion.

Fig. 4 shows the photo acoustic measurements of three standard specimens which were heated up to 100° C / 200° C / 300° C, in comparison with the measurement of the untreated specimen previously shown in Fig. 3. The measured reflectivity change curves of the thermally treated specimen clearly indicate that the broadening of the diffusion zone i.e. the broadening of the ‘soft acoustic interface’ suppresses the acoustic contrast. One can also see that the echo which occurs at the Al/Al₂O₃ substrate interface remains detectable even after the thermal treatment of 300° C.

Thus the photo acoustic method is used for nondestructive in-depth-profiling achieving a resolution of few nanometers.

3. TESTING OF MINIATURIZED SPECIMENS

In this section several methods for the mechanical testing of small specimens of MEMS materials are described. Hereby, the samples used are not thin films on a substrate but structures which consist entirely of MEMS materials. They are fabricated in the same way as MEMS structures.

3.1 Specimens

The specimens used were of single crystal silicon, rolled copper foil or various NiFe LIGA (lithography, galvanic, abformung) alloys, where the first two mentioned are patterned by lithography and wet etching. For better handling the specimens include two relatively large (e.g. 4 mm x 5 mm) end plates connected by the actual testing region (cross-section of e.g. 20 µm x 30 µm, length of 500 µm). An example is given in Fig. 5.

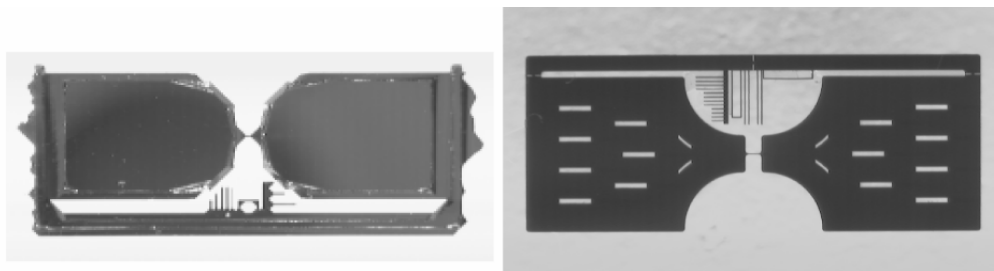


Fig. 5: Microstructure specimens (left: Silicon, right: metallic LIGA) used in order to determine material parameters. The small testing region between the large plates has a length of about 300 µm. The additional small structures are used for detecting intrinsic stresses.

3.2 Torsion Testing

Relatively little work has been done on torsional testing. In Schiltges [4] a torsional testing set-up is described: The torque is determined from the rotation of a bar, which is supported with a calibrated torsional spring. The rotation of the bar is measured with a two point optical fiber interferometer. The specimens are attached with one plate to the bar and with the other plate to a rotational stage. The actual rotation angle is measured with a linear diode detector. The torque resolution is about 0.05 µNm. While the silicon specimens show a brittle behavior in a torsion test, the LIGA Ni and NiFe specimens show large deformations including plastic behavior.

3.3 Fatigue Testing

When fatigue testing is concerned, a resonance technique turns out to be the most suitable (Connally [5], Schlums [6]). It is based on the fact that upon crack propagation the resonance frequency of a structure in bending decreases in a monotonous way with increasing crack

length. By using a phase-locked feedback loop, the resonance frequency can be tracked, such that frequency vs. time curves result. Using simple fracture mechanics, the resonance frequency of a beam can be related to the crack length, using a compliance matrix based on stress intensity factors. The specimens used are similar to the ones shown in Fig. 5 with the difference that the testing region contains a notch. They are vibrated in their plane using a stacked piezo element. The vibrations are measured with a laser optic displacement sensor in order to close the feedback loop.

3.4 Tensile Testing

In order to determine critical stresses for microstructures, specimens with a design similar to Fig. 5 were subjected to a tensile test. A tensile test apparatus was designed for testing small specimens: The force acting on the specimen is determined using a high resolution balance, while the strain is determined using a video camera and a least square template matching algorithm (LSM) which yields a super resolution in the specimen extension around 10nm under an optical microscope (Mazza [7] and Mazza [8]). The LSM algorithm matches the six parameters that characterize a homogeneous deformation by adaptive least squares correlation. The LSM results agree very well with results of vibration measurements and the Young's modulus typically used for Silicon in the <100> direction (Hull [9]). Critical stresses as computed from force and cross-sectional area in the middle of the testing region amounted to 586 MPa with a standard deviation of $\pm 3\%$. Of course all the specimens broke at their ends because of stress singularities. In fact, with the anisotropic etching a nearly perfect notch was produced there. As fracture starts at the atomic level, such a tensile test can therefore be used as a window towards molecular dynamics. Continuum mechanics can be matched with atomistic theories.

4. SIZE EFFECTS IN TENSILE TESTING OF THIN COPPER FOILS

The influence of the size of a specimen on its mechanical behavior is an object of current research. An overview of possible size effects is given in Arzt [10]. Many size effects are explained based on a theory developed by Fleck [11] who explained size effects in copper wires in torsion using a strain gradient plasticity theory.

While elastic properties do not seem to vary with the size of the specimen (Namazu [12] and Sharpe [13]), for other quantities large discrepancies might arise. When testing Ni specimens made with the LIGA technology it was found, that the ultimate strength was considerably higher than for macroscopic specimens (Mazza [8]). Klein [14] reported a decrease of the fracture strain with smaller sample size and a more complicated influence of the size on the ultimate tensile strength when testing copper wires and stripes. The thickness of the tested specimens varied between 9 and 200 μm .

Although it is known that the microstructure of a polycrystalline sample affects its mechanical behavior to a large extent this fact was often neglected in many studies concerning size effects in small structures. Here, results of a study are presented which tries to close this gap for thin rolled copper foils: Thin rolled copper foils of varying thickness (10 – 250 μm) were tested in tension in order to minimize effects of external strain gradients. Larger specimens (copper foils thicker than 20 μm) are tested with a commercial tensile test machine (Zwick 1445) with a 10 N and 200 N load cell and a specially designed clamping apparatus to allow clamping free of initial tension. Smaller samples are tested with the apparatus explained in the previous section. The specimens were made of 99.9% pure copper foils (Goodfellow) and had ratios of thickness to width to gauge length of 1 to 20 to 200, which was kept constant when downscaling (further details in Villain [15]). The specimens were manufactured by

photolithography and wet etching and had the longitudinal axis in the rolling direction. The strain rate was in the order of 10^{-4} 1/s - 10^{-5} 1/s. Additionally, a detailed characterization of the microstructure was performed prior and after the test including X-ray diffraction, hardness tests and metallography.

4.1 Microstructure

The microstructure of the foils has a strong (100)[001] cube texture and weak deformation texture resulting from the rolling process. Fig. 6 shows a sketch of the grain structure: It is dominated by large grains which have approximately a cylindrical shape with a flat ellipse as a cross section normal to the rolling direction where the major diameter (D) is in the range of a few microns and is approximately 5 times the minor diameter. The length of these grains (L) is 5-10 times the major diameter (D). Furthermore, there are some small grains which are situated in between the larger grains.

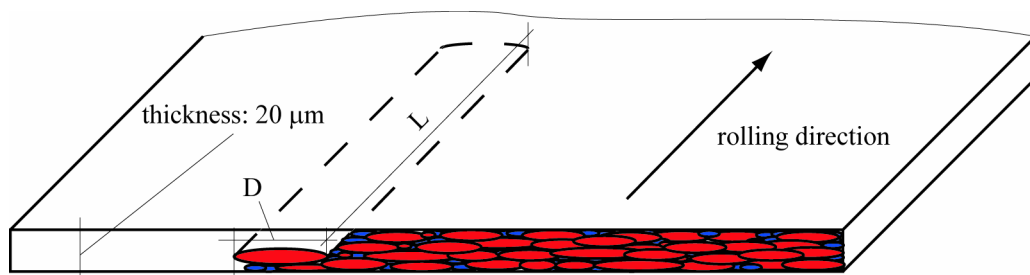


Fig. 6: Sketch of the grain structure of the copper foils: Large cylindrical grains (diameter D and length L) are accompanied by small grains.

4.2 Results of Tensile Tests

In Fig. 7 on the left the results of a tensile test for a 10 and 34 μm thick foil are presented. The 34 μm thick foil shows a typical stress strain relationship for ductile materials, whereas the 10 μm thick foil has a stress strain behavior which is typical for brittle materials (nearly only elastic part). The right graph of Fig. 7 displays the relationship between foil thickness and fracture strain (circle, left vertical axis) and ultimate strength (dot, right vertical axis). The results of the tensile tests indicate a moderate increase of the tensile strength (except for the 10 μm foil) and a strong decrease of the fracture strain with decreasing thickness of the copper foils. In terms of fracture strain, when going from 250 μm to 10 μm thickness, the fracture strain is reduced dramatically from 20% to 0.2-0.5% (scatter of fracture strain values). For the interpretation of the strain results it has to be stressed that the strain measured is the average strain of the whole sample in tensile direction ("macroscopic strain"). Fig. 8 shows a comparison of the tensile results of copper foils and wires from the literature (Klein [14]) with results from this study. Thinner specimens show a lower fracture strain for both wires and foils. The situation is more complicated for the tensile strength. The results suggest that when both dimensions of the cross-section are scaled (wires of Klein [14] and foils of this study) the tensile strength increases with decreasing size. When only one dimension is scaled (stripes in Klein [14]) the trend is opposite.

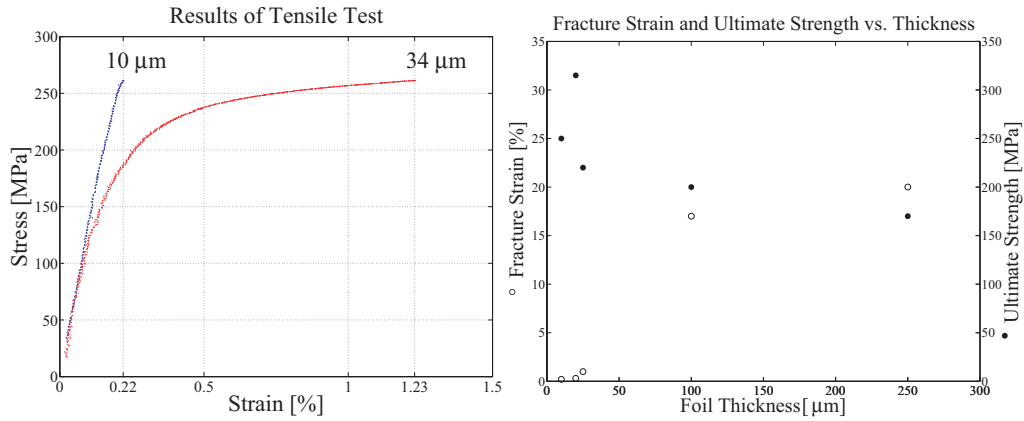


Fig. 7: Tensile test results (strain rate $2.5 \cdot 10^{-5}$ 1/s): Stress-strain curve for a 10 and 34 μm thick foil (left image) and fracture strain (circle, left vertical axis) and ultimate strength (dot, right vertical axis) vs. foil thickness (right image).

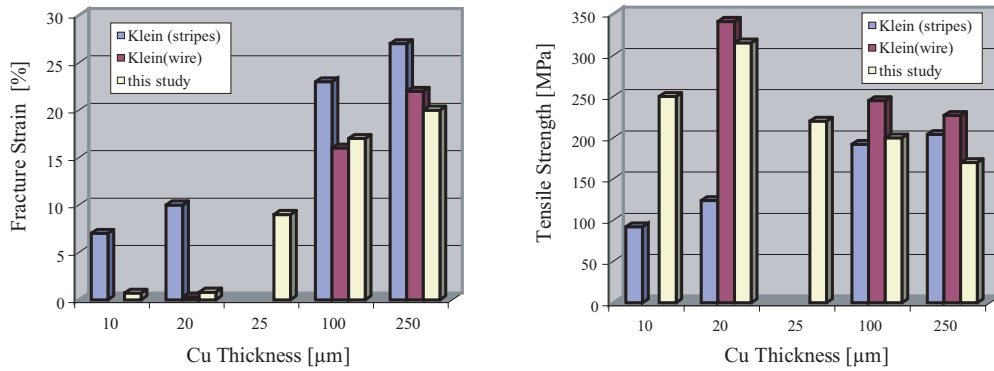


Fig. 8: Comparison of the tensile test results of copper foils and wires from Klein [14] and this study.

The reason for the strong decrease in fracture strain is unclear at the moment. Considerable efforts are currently made to characterize the specimens used in terms of surface roughness, grain shapes, orientations, size, dislocation densities, etc. Because of the small size this turns out to be quite difficult though. Furthermore, the effect of additional heat treatments is studied. First tests on annealed samples (300°C for 2h) indicate a strong increase in the fracture strain and decrease of the ultimate tensile strength which is expected as the amount of strain hardened material is reduced due to recrystallization. Nevertheless, thinner foils still display a smaller fracture strain than thicker foils.

5. CONCLUSIONS

In addition to the well-known nanoindentation technique a number of other methods for material testing of micromaterials exist, which have received increasing attention. They offer insight into many interesting phenomena like size effects (bending, microtensile and torsion tests) or probing of continuous interfaces (pump probe pulsed laser set-up). Together with elaborate manufacturing possibilities as known from MEMS and IC fabrication, they offer possibilities of probing material behaviour in the nm range. With this it is hoped that an improved understanding of processes important for the life time and reliability of MEMS components will be possible in the near future.

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