High-temperature bulge-test setup for mechanical testing of free-standing thin films

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We developed a bulge-test setup which enables measurement of the elastic and plastic properties of free-standing thin film samples between room temperature and at least 300 °C. Mechanical stress is applied to the film by a differential gas pressure across the sample and the bulge height is measured by a scanning laser beam technique. To prevent sample oxidation the pressure cell containing the sample is mounted in a vacuum chamber. The correct operation of the setup is demonstrated by measurement of the thermal expansion of free-standing Al films. Creep experiments and tensile tests demonstrate measurement of the plastic deformation of these films at temperatures up to 200 °C. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1539901]

Mechanical properties of thin films are of interest for many advanced device applications.[1] Mechanical testing of thin films at room temperature is usually done by microtensile testing[2,3] or bulge testing,[4,5] while for testing at elevated temperatures the substrate-curvature method is often used.[6] The latter method is not suitable for free-standing films.

For a proper understanding of the physics of plastic deformation of thin metallic films there is an increasing need to measure the temperature dependence of the plastic properties of free-standing films. To this end we developed a new bulge-testing setup suitable for measurements up to at least 300 °C, using the scanning laser beam technique and the application of a differential gas pressure described earlier in detail for a room temperature setup in Ref. 7. Measurement at elevated temperatures imposes a number of nontrivial technical requirements. Among these are: a clamping method for the fragile samples (free-standing films on a supporting silicon frame) to a metal heating stage, a pressure seal for use at high temperatures, and prevention of oxidation of the thin film sample.

The aim of this Note is to describe our high-temperature bulge tester and to demonstrate its successful operation by showing measurements of the elastic and plastic properties of free-standing Al films for temperatures up to 200 °C.

To prevent the oxidation of the sample at high temperatures the pressure cell containing the sample is mounted in a vacuum chamber. Most parts are cylindrically symmetric and a schematic cross section of the setup is shown in Fig. 1. The pressure cell has been made from Ampcoloy 940 alloy for its good heat conductivity. The sample is mounted in the pressure cell using a stainless steel sample holder. The latter is held in position by a cylindrical stainless steel part, called bolt in Fig. 1, having thread on the outside which fits thread on the inside of the pressure cell. To ensure a uniform pressure in the pressure cell holes were drilled in the sample holder and the bolt.

The sealing between the pressure cell and the surrounding vacuum is done using two low-density graphite rings.[8] The advantage of using graphite seals is their high oxidation resistance and low seating load.

The cell is pressurized using a gas inflow, adjusted by a mass flow controller, and an exhaust valve. The valve is driven by a pressure control unit, controlled by a personal computer. The pressure across the sample is measured by a differential pressure gauge. The vacuum chamber (base pressure 1.10^{-5} mbar) is evacuated by a turbopump and a membrane roughing pump. To avoid the use of two different pump systems the gas outlet from the pressure cell is fed back into the vacuum chamber. As a result, during a measurement the pressure in the vacuum chamber is about 0.1 mbar. When oxidation-sensitive samples like Cu films are used, a reducing ambient can be created in the chamber by using forming gas ($N_2/10\%H_2$).

The optics for measuring the deflection of the membrane is somewhat different than used in the room temperature setup.[7] Here, because of the normal angle of incidence of the scanning laser beam, a mirror, and a beam splitter are applied.

The scanning laser beam method is suited for sample geometries with a well-defined shape of the deflected membrane. For rectangular windows with long edge $2b$ and short edge $2a$ and $b \geqslant 4a$, the bulge is known to have a parabolic shape.[4] In this case, it is straightforward to calculate the bulge height h in the center of the membrane from the deflection of the reflected laser beam when one ignores the presence of the beam splitter and the quartz window.[7] However, the quartz window and the beam-splitter cause a parallel displacement of the beam. To account for this effect we

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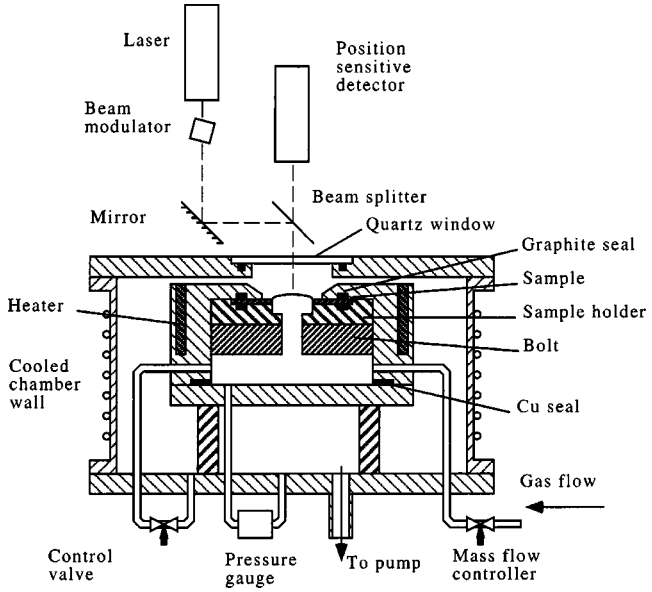

FIG. 1. Schematic cross section of the high-temperature bulge-test setup. The sample and sample holder are mounted in a pressure chamber. The pressure chamber is mounted in a vacuum chamber. Bulging of the sample is measured using a scanning laser beam, which is guided into the vacuum chamber using a mirror, a beam splitter and a quartz vacuum window. The deflection of the beam is detected by a position sensitive detector.

take advantage of the fact that all optical components are mounted on a vertically suspended optical board, which can be aligned using an *XYZ* translation stage. A translation in the *z* direction allows measurement of the deflection of the beam as function of the sample-detector distance enabling the determination of the parallel displacement of the beam. The stress σ_x and strain ε_x in the film are calculated using the gas pressure and/or the bulge height.[7]

The pressure cell is heated using eight 125 W resistive heaters in parallel, positioned symmetrically in the wall of the pressure cell. The temperature is measured by type K thermocouples, located close to the sample. The measured temperature stability is better than ± 0.5 K over 24 h. Radiation shields around the pressure chamber ensure good temperature uniformity across the sample and the pressure cell. The setup has been tested at temperatures up to 300 °C. Temperatures above about 100 °C require water cooling of the walls and the bottom of the vacuum chamber.

To check the correct operation of the setup we measured the thermal expansion of free-standing Al films supported in silicon frames. Films with submicron thickness were made using sputter deposition and standard bulk micromachining techniques.[9] The dimensions of the membrane are $2a = 4$ mm and $2b = 16$ mm. After anneal at 500 °C the free-standing Al film is initially under a high, equibiaxial tensile stress. A measurement of this initial stress is carried out by ramping the gas pressure to a set point of typically 1 kPa and back. From a plot of σ_x vs ε_x we can determine $M \equiv E/(1-\nu^2)$ and the initial equibiaxial stress in the film σ_0 ($\sigma_x = M\varepsilon_x + \sigma_0$, see Ref. 7). E is Young's modulus, and ν Poisson's ratio.

A number of measurements at different temperatures are shown in Fig. 2. The slope of the parallel lines is $M = 65 \pm 4$ GPa. This value is smaller than expected on the basis of

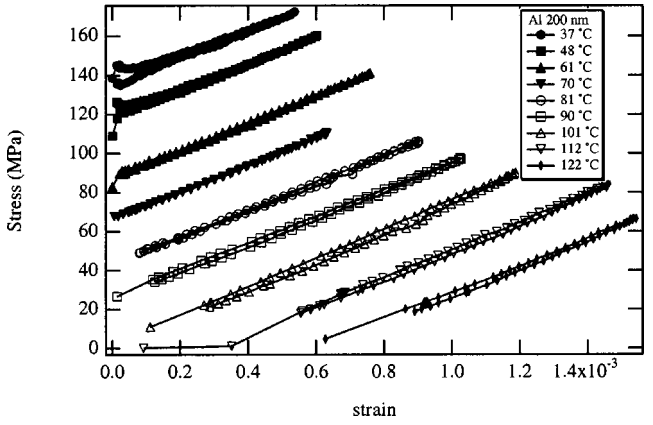

FIG. 2. Elastic stress (σ_x) vs strain (ε_x) for a 200 nm Al film measured at different temperatures. The initial film stress is given by the intercept with the $\varepsilon_x = 0$ axis. The slope of the lines is equal to $E/(1-\nu^2)$.

bulk values for E and ν. Taking for bulk Al $E = 70$ GPa and $\nu = 0.345$, we calculate $M_{\text{bulk}} = 79$ GPa. The reason for this discrepancy is that for experiments on Al films that involve a nonequibiaxial stress state, as is the case in bulge testing on rectangular membranes, anelastic effects occur. Ignoring these effects, which are most probably due to grain boundary sliding, leads to apparently smaller values of E.[10] σ_0 decreases with temperature due to the difference in coefficient of thermal expansion between the Al and the surrounding Si window. In Fig. 3 we show the initial stress σ_0 as a function of temperature. A linear fit of σ_0 versus temperature yields a slope $\partial\sigma_0/\partial T = (2.15 \pm 0.10)$ MPa/K. This value is in excellent agreement with the value of (2.2 ± 0.1) MPa/K which is obtained when we multiply the biaxial modulus Y for $\langle 111 \rangle$ textured Al, $Y = 108$ GPa, with the difference in coefficient of thermal expansion of Al (24 ppm/K) and Si (3.3 ppm/K). The value used for $Y\,[=E/(1-\nu)]$ does not include anelasticity and is close to the bulk value. This is correct because thermal expansion does not induce anelastic effects, only the magnitude of the equibiaxial stress state is changed. The agreement for $\partial\sigma_0/\partial T$ indicates directly that the strain measurement system works properly and that changes in the sample temperature are measured correctly.

A consequence of increasing the measurement tempera-

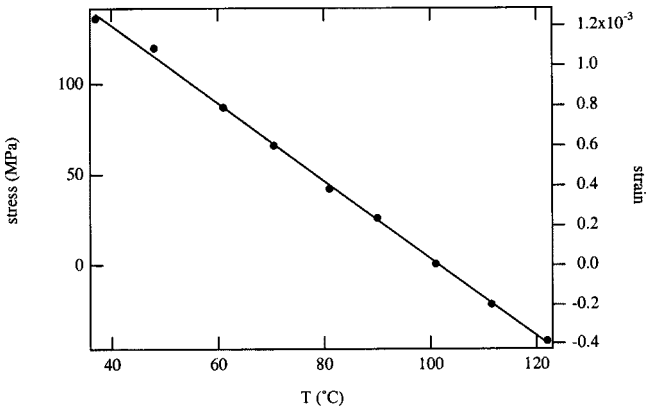

FIG. 3. Temperature dependence of the initial film stress. The line is a linear fit with a slope of (2.15 ± 0.10) MPa/K. The corresponding strain is shown on the right axis.

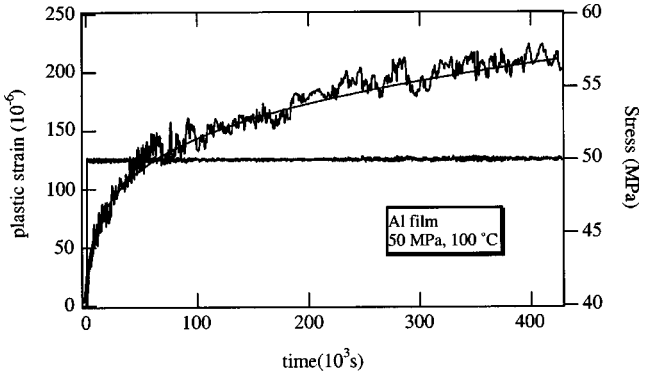

FIG. 4. Creep measurement on a 500 nm Al film at a test temperature of 100 °C. The applied stress was kept constant at 50 MPa. The solid line is a guide to the eye.

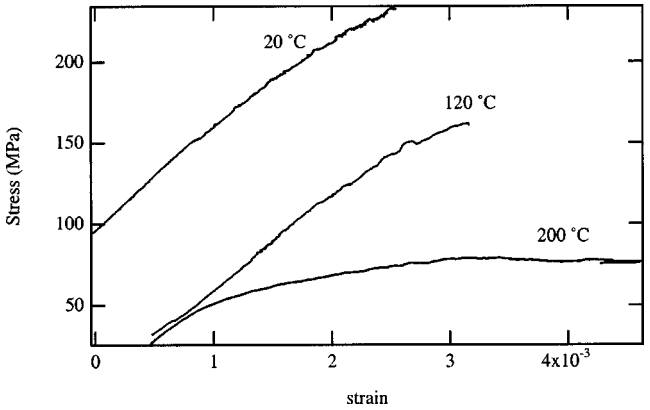

FIG. 5. Tensile tests on 200 nm Al films at 20, 120, and 200 °C. The strain rate was kept constant at 2.10^{-6}/s.

ture is that films initially flat at room temperature become slack at some elevated temperature. This represents a problem for bulge testing methods using an interferometric technique.[11] With those techniques it is not possible to determine the zero pressure bulge height. With the scanning laser method the zero-pressure bulge height is calculated from the zero-pressure membrane curvature.[7] Figure 2 shows that our Al film becomes slack at ~100 °C. The measurements at 112 and 122 °C clearly show the presence of an initial strain in the film. The thermally induced initial strain provides a practical upper limit for the measurement temperature. For the films described here the limit was 200 °C. This is due to the facts that the maximum total strain that can be measured is about 1%, and that a finite stress is required to remove the slack in the film.

Various tests, like constant-strain rate tensile tests, stress relaxation measurements, creep measurements, and dynamic measurements[10] can be performed, using a feedback loop with appropriate software to control and adjust the gas pressure. In Fig. 4 a creep measurement at 100 °C is shown. A constant stress of 50 MPa is applied on a 500 nm Al sample, while the strain is recorded as a function of time. A characteristic transient creep curve is obtained. The stress is kept constant within 0.5 MPa, while strain rates down to about 1.10^{-10}/s can be measured. It is clear that the relatively small creep strains that are often observed in thin films[12] can be readily measured.

Tensile tests were performed on thin film Al samples at a temperature up to 200 °C. In Fig. 5 we show the results of tensile tests of a 200 nm Al film at a constant applied strain rate of 2×10^{-6}/s, at different temperatures. The tests are run until fracture of the film. The measurements at 120 and 200 °C start at a stress of 35 MPa which is the applied stress required to obtain a taut film. It can be seen that the ductility of the film increases with increasing temperature.

The quality of the data is strong support for the idea that our setup is a very suitable tool to study plastic deformation in thin films with essentially the same techniques as used for studies of bulk material (e.g., creep, tensile test, etc.).

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