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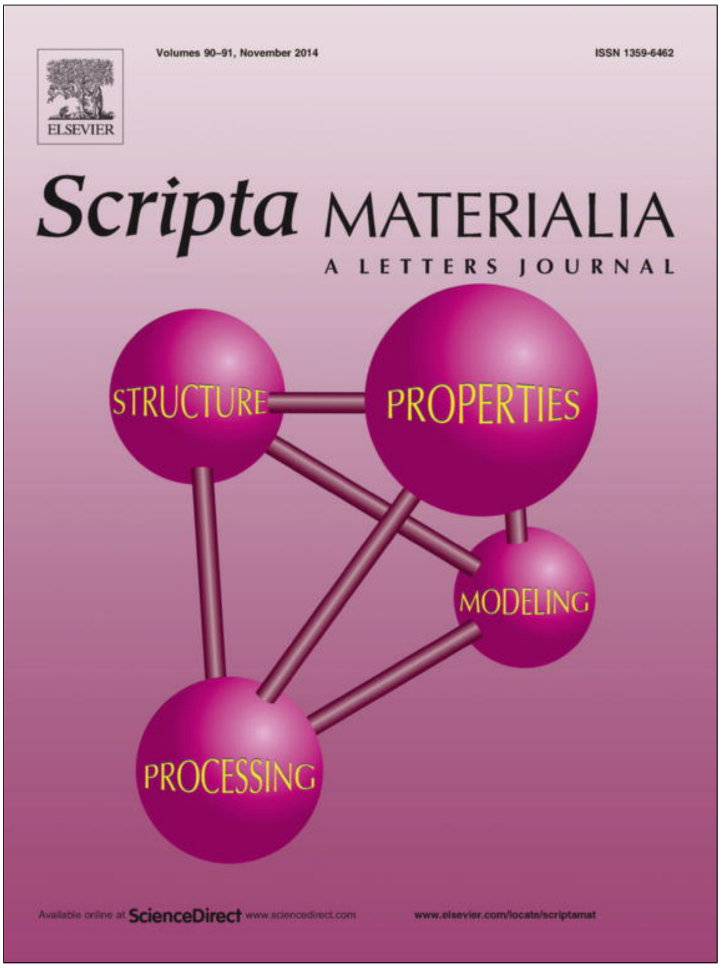
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A large strain rate effect in thin free-standing Al films

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We present tensile tests of thin aluminum films under quasi-static, medium and high strain rates. A large strain rate effect is revealed, as the ultimate tensile strength increases by more than 400% compared to quasi-static tests. An analysis of the kinetic relation for plastic flow shows that all commonly used kinetic laws cannot explain our results. Instead, we suggest an newly elaborated kinetic law that is in good agreement with the results over the entire range of strain rates.

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Thin metallic films are commonly employed in micro/nanoelectromechanical systems (MEMS/NEMS) and are frequently subjected to various mechanical constraints [1,2]. A main characteristic of thin films is that the specimen dimensions become comparable to the characteristic length scales that govern the mechanical behavior. Therefore, specimens at the micrometer and sub-micrometer scales often exhibit a mechanical behavior that may be different from that of bulk specimens, which is referred to as the "size effect" [3].

MEMS and NEMS devices may be subjected to a wide range of operating velocities and frequencies, depending on their use, environmental conditions and method of actuation. A device can be used as a transducer for long-term measurements, which implies quasi-static loads, or alternatively as a switch [4–6], which mostly dictates high rates and dynamic loads. As a rule, the resonance frequency of mechanical devices increases as their size decreases. In accordance, smaller devices are subjected to higher strain rates during vibrations or other dynamic motion. In particular, vibrating metallic nanowires, which are of great interest for resonator applications (see e.g. Refs. [7,8]), are subjected to very high strain rates on the order of 10^3–10^6 s^{-1} during their operation. Hence, to design devices that may last and perform well under various loading regimes, the investigation of the mechanical properties of thin films at various and high strain rates is crucial.

Strain rate sensitivity is also a fundamental problem in materials mechanics. At the microscopic scale, plastic deformation occurs through the motion of crystal defects (usually dislocations). Quasi-static tests usually demonstrate a critical stress, called the yield stress, below which dislocations are pinned and above which accumulated dislocation motions result in significant plasticity. Under higher strain rates, dislocations are forced to move at some velocity to produce an accumulated plastic strain rate equal to the strain rate applied in the test. From the physical point of view, there is some fundamental kinetic relation between the force acting on a dislocation and its velocity. As a result, a larger stress is required to produce a higher plastic strain rate. Thus, material characteristics such as the yield and ultimate stress values may depend on the strain rate.

The study of strain rate effects is hampered by limitations of the existing experimental methods. Experimental methods for testing bulk specimens (with typical dimensions above the millimeter scale) allow the application of strain rates either below $\sim 1\ s^{-1}$ using large-scale tensile instruments or above $\sim 500\ s^{-1}$ using impact techniques. Thus, there is a range of strain rates spanning roughly two orders of magnitude, from macroscopic specimens to microscopic specimens such as thin films, over which it is difficult to obtain reliable mechanical characteristics. Many common engineering

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applications, including automotive crash and low-velocity impact testing, lead to strain rates in this range.

In principle, the tensile testing of thin free-standing films has the ability to bridge the above-mentioned gap of strain rates and allows testing at a variable rate from the quasi-static regime up to approximately 1000 s^{-1}. The strain rate in tensile tests is given by $\dot{\varepsilon} = v/L$, where v is the cross-head velocity and L is the specimen length. Commercial small-scale actuators can provide adjustable velocities of up to ~0.1 m s^{-1}. Thus, for a specimen length of ~100 μm, a strain rate of up to 10^3 s^{-1} can be obtained in principle.

The main problems associated with tensile testing of free-standing films at high strain rates come from the short duration of the overall test. The latter can be estimated by $\Delta t = \varepsilon_f / \dot{\varepsilon}$, where ε_f is the strain at failure. Thin metallic films tend to be less ductile than bulk materials and usually exhibit an ε_f of approximately 5% [9,10]. Thus, the overall test duration at a strain rate of 1000 s^{-1} is expected to be 50 μs. This short duration imposes a severe requirement for the bandwidth and the sampling rate, which should be much larger than $1/\Delta t$ for all measuring devices.

Recently, we presented a novel apparatus and a method for the tensile testing of free-standing thin films under adjustable strain rates [11] from the quasi-static regime to approximately 500 s^{-1}. To provide this capability, a unique displacement measurement method [12] that provides a resolution of 25 nm, a bandwidth above 1 MHz and a sensing range above 1 mm was implemented. In addition, a microdevice that meets several strict requirements was implemented and a testing methodology was developed [11]. However, because of rapture of the thin films during their preparation process, Ref. [11] demonstrates the capabilities of the apparatus and method using microdevices without thin-film specimens. Thus, while the apparatus and method have been demonstrated to function properly, there has been no report on high strain rate tensile tests of thin free-standing films.

In this paper, we apply the above-mentioned experimental system and methodology to study the mechanical response of thin Al films under various strain rates from 0.001 to 170 s^{-1}. We report an unusually large strain rate effect, which results in a greater than fourfold increase in the ultimate strength. Furthermore, we show that our results do not fit the commonly used exponential and power-law kinetic relations. Instead, we suggest an elaborated power law, which takes into account a threshold stress for the plastic strain and is in good agreement our results over the entire range of strain rates.

The samples used in this study are free-standing Al films 1.2 μm thick, 25 μm wide and 70–120 μm long; they were fabricated on a microdevice that includes Si springs positioned to protect the sample (Fig. 1). Two circular holes located at both ends of the microdevice allow it to be mounted on gripper pins. The moveable part of the microdevice has a metallic grating on its surface to allow for the measurement of its displacement by an optical linear encoder located above the grating. A piezoelectric force sensor is connected to the static gripper, while the moving gripper is connected to a linear stage actuator. An XYZ stage and an optical stereoscope serve to align the microdevice along the tensile direction. Dedicated software and a user interface are used to operate the entire system and to record the measured response.

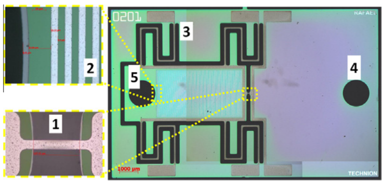

Figure 1. A top view of the microdevice (dimensions 13.8 × 8.5 mm^2), showing (1) the thin film, (2) the encoder grating, (3) the "S"-shaped silicon springs and (4, 5) the holes for the static and moving grippers, respectively.

Displacement control tensile tests were performed. As the gripper that is attached to the linear stage starts moving, tension is applied to the microspecimen until it is torn. Unloading is then performed, followed by a reloading step to measure the response of the springs alone. Thus, two tests are performed in series. In the first test, the responses of both the springs and the specimen are measured. In the second test, after the specimen is torn, only the spring's response is measured. The force measured over the springs alone is subtracted from the force measured over the springs and the specimen to extract the specimen's response alone. The experimental setup and procedure are described in detail in our recent study [11].

Five stress–strain curves measured under different strain rates from 0.0012 to 170 s^{-1} are presented in Figure 2. Significant increases in the yield and ultimate stresses are observed at high strain rates. At the highest rate, the ultimate stress is more than fourfold larger than the ultimate stress that is measured in the quasi-static experiment.

At strain rates between 0.0012 and 5.4 s^{-1}, the maximal slopes of the stress–strain curves are between 16 and 20 GPa. These values are considered to be very low for aluminum in comparison to the reported Young's modulus of 70.6 GPa for pure bulk samples [13]. An accurate measurement of Young's modulus of thin free-standing films is a major experimental problem, and several previous studies have reported values much lower than the known value for bulk materials [14–18]. In testing methods where the strain is determined by measuring the overall sample elongation, even a small compliance of the sample grippers may result in a significant underestimation of Young's modulus. In our case, we calculate the total sample elongation by assuming that the

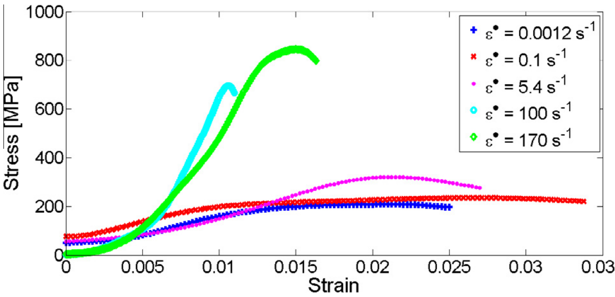

Figure 2. Stress vs. strain curves for different strain rates.

static pin gripper does not move at all. In fact, the mechanical part that connects the static pin gripper with the force sensor has some compliance. As a result, the evaluated elongation is larger than the actual elongation; therefore, the slope of the stress–strain curve is smaller than the real Young's modulus.

Interestingly, at high strain rates of 100 and 170 s^{-1}, Young's modulus values of 71 and 71.6 GPa (respectively) were been measured, which are in excellent agreement with the value for bulk aluminum. At these strain rates, the elastic loading time is less than 0.1 ms, and therefore the bulk mechanical parts of the setup do not have time to comply. Thus, our results explain the origin of the small Young's modulus values that are often reported for thin films [14–18], and indicate that an accurate Young's modulus can be measured in high rate tests. We emphasize that the difficulty associated with accurately measuring Young's modulus in some tests is due to an overestimation of the strain. The measured characteristic stress values, which are the main focus of the paper, are not influenced by this problem.

The strain rate sensitivity is better presented in Figure 3, where the ultimate stress values, normalized with respect to the value measured at the slowest strain rate, are plotted as a function of the strain rate. This allows comparison of the results from current experiments to results from the literature [19,20]. The results shown in Figure 3 are compared to values that were measured for pure bulk aluminum specimens at different strain rates in annealed and cold-worked conditions. Over the years, several studies [21–23] have shown that pure aluminum bulk specimens exhibit little, if any, strain rate sensitivity and that pure bulk aluminum has a slight rate sensitivity, evident in an increase in up to 20% in the stress at very high strain rates (above 10^3 s^{-1}). Karens and Ripperger [19] presented relatively large strain rate sensitivity for pure bulk aluminum, especially for annealed specimens. However, the strain sensitivity measured in their tests is much smaller than that in our tests. In particular, while in all previous studies the ultimate stress increases gradually with strain rate, our results demonstrate a very sharp increase in the ultimate stress at strain rates above ~10 s^{-1}. It should be noted that Karens and Ripperger conducted their experiments using the Hopkinson pressure bar in compression. The differences in strain rate sensitivity are most likely a result of different microstructures and defect contents. Scanning electron microscopy and X-ray diffraction measurements show that our Al thin films have an average grain size of 0.18 μm and a strong preferred orientation, in which the (1 1 1) crystallographic planes are parallel to the film surface.

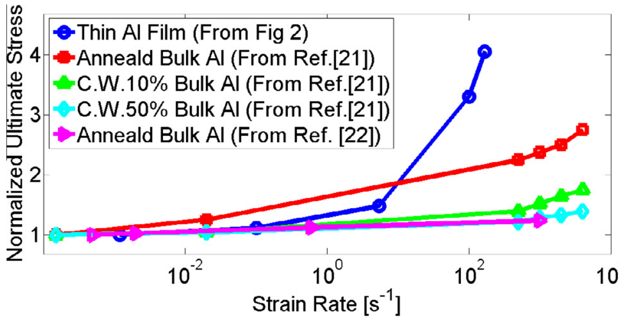

Figure 3. Comparison of the strain rate sensitivity of pure aluminum in different studies. The ultimate stress values are normalized with respect to values measured at the lowest strain rate for each type of sample.

A physical understanding of the mechanism of plastic deformation under different strain rates can be obtained by analyzing the kinetic relation and fitting kinetic laws. At the microscopic scale, the plastic strain rate is determined by the average velocity of crystal defects (usually dislocations). This velocity is determined by the force acting on the defects, which is proportional to the stress. Thus, kinetic relations should be expressed in terms of the plastic strain rate as a function of the stress. The overall applied or measured strain rate $\dot{\varepsilon}$ is a combination of the elastic $\dot{\varepsilon}_{el}$ and plastic $\dot{\varepsilon}_p$ strain rates. Above the yield stress, $\dot{\varepsilon}_{el}$ is much smaller than $\dot{\varepsilon}_p$, and therefore $\dot{\varepsilon}_p \cong \dot{\varepsilon}$. In particular, at the ultimate (maximal) stress $\sigma_{UTS}, \dot{\varepsilon}_{el} = 0$ and $\dot{\varepsilon}_p = \dot{\varepsilon}$.

In the following, we analyze the measured relation of $\dot{\varepsilon}$ as a function of σ_{UTS} and search for a kinetic law that fits this function. There are two accepted kinetic relations in the literature by which experimental results are commonly analyzed [24,25]. The first, which is often more relevant to body-centered cubic (bcc) metals, describes a thermally activated dislocation motion and has the form [24,25]

$$\dot{\varepsilon} = \dot{\varepsilon}_0 \exp\left(\frac{\sigma b A}{\sqrt{3}kT}\right) \tag{1}$$

where k is Boltzmann's constant, T is the absolute temperature and σ is the stress at some characteristic point above the yield stress (often taken as the yield stress). In expression (1), A is the activation area, i.e. the area covered by the dislocation motion during a discrete thermally activated event, and b is Burgers vector. The coefficient $\dot{\varepsilon}_0$ is also strain rate dependent [26–28], but this effect is minor with respect to the exponent term.

The second kinetic relation, which is often more relevant to face-centered cubic (bcc) metals, describes a viscoplastic flow and has the form [25]

$$\frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} = \left(\frac{\sigma}{\sigma_0}\right)^{\frac{1}{m}} \tag{2}$$

The coefficient m is known as the rate sensitivity parameter and is given by $m = \partial \ln \sigma / \partial \ln \dot{\varepsilon}$. Although Eqs. (1) and (2) have very different forms, both of them can be explained in terms of a thermally activated dislocation motion, where in the case of Eq. (1) the activation area A is constant, while in the case of Eq. (2) A depends on the stress as approximately $A = 1/\sigma$. It has been shown that both m and A depend on the grain size [29].

Our experimental results fit neither Eq. (1) nor Eq. (2), i.e. plots of the measured values of $\ln \dot{\varepsilon}$ vs. either σ_{UTS} or $\ln(\sigma_{UTS})$ do not exhibit a linear relation. There are cases in which the experimental results do not fit a single kinetic law using a single parameter of A or m over the entire range of strain rates. These cases often fit two kinetic laws [30,31] or fit the same law but with two different values of A or m, each valid in a different range of the stress [18]. These cases are interpreted as having two different mechanisms for dislocation motion;

each has a different kinetic law. The overall plastic strain rate is a combination of both mechanisms, and it follows the dominant mechanism, i.e. the faster mechanism, at each different range. In our case, fittings using two different values of A or m reveal an impossible situation in which the combination of the two mechanisms follows the slower mechanism rather than the faster one. Thus, we conclude that our results do not fit the common kinetic laws in the form of Eq. (1) or (2).

We therefore suggest a different kinetic law of the form

$$\frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} = \left(\frac{\sigma_{UTS} - \sigma_0}{\sigma_0}\right)^{\frac{1}{m}} \tag{3}$$

Eq. (3) is an elaboration of Eq. (2) for cases in which there is no plastic strain at all below some threshold stress σ_0, regardless of the strain rate. In this case, $\dot{\varepsilon}_0$ has no physical meaning and is a constant that results from the mathematical fit. Figure 4 shows a fitting of this kinetic relation to our measured values, which demonstrates an excellent agreement over the entire range of strain rates. The best fitting is obtained for $\sigma_0 = 212$ Mpa, $\dot{\varepsilon}_0 = 22\,s^{-1}$ and $m = 0.55$. The obtained value of m is in good agreement with measurements of the velocities of individual dislocations in fcc metals [32], in which a kinetic relation similar to Eq. (3) was fitted using m values in the range 0.2–1.

In summary, in this paper, we report new high strain rate tensile tests of thin free-standing films using a newly developed experimental system. The obtained results demonstrate a large strain rate sensitivity effect in aluminum thin films under strain rates as high as 170 s^{-1}. A comparison with previous studies of bulk specimens shows that this effect is far beyond what is known for pure aluminum. An analysis of the kinetic relation for plastic flow, i.e. the relation between $\dot{\varepsilon}_p$ and σ, indicated that our results do not fit either of the two well-known kinetic laws that are frequently used in the literature. We therefore suggest a newly elaborated kinetic law that is suitable for materials in which there is no plastic strain at all below some threshold stress regardless of the strain rate. A comparison of our experimental results with this kinetic law demonstrates excellent agreement over the entire range of strain rates. The extracted value for the threshold stress, $\sigma_0 = 212$ Mpa, is two orders of magnitude higher than the typical yield stress in bulk specimens of pure aluminum, a fact that explains the brittleness of thin films. The obtained results also have an important practical implication because they indicate that small-scale devices, such as metallic nanowires, which are designed to operate under very high strain rates, can sustain much higher stresses (more than four-fold based on our results) without failure.

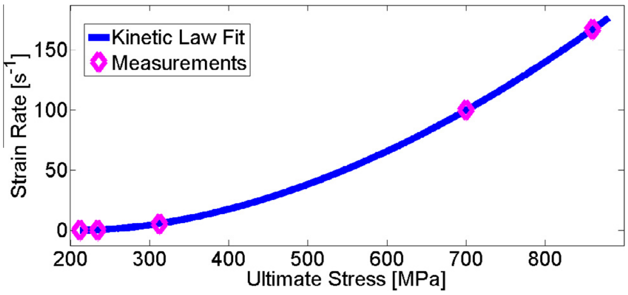

Figure 4. A fitting of the newly elaborated kinetic law (Eq. (3)) to the experimental results shown in Figure 3.

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