Low-temperature internal friction in metal films and in plastically deformed bulk aluminum

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Crystalline metal films on silicon substrates have been found to have a surprisingly large, nearly temperature-independent internal friction at low temperatures, with a magnitude similar to that of amorphous solids. This large internal friction is rather insensitive to chemical composition and impurities, mode of film preparations, and film thickness, but can be reduced considerably by proper alloying. In an attempt to understand its origin, plastically deformed bulk aluminum has been measured in the superconducting state. It has been found that its internal friction can be increased over two orders of magnitude by plastic deformation. For deformations exceeding 10% the internal friction saturates and resembles that of the metal films both in magnitude and temperature independence. It is suggested that the damping is caused by a broad spectrum of tunneling states associated with dislocations both in the bulk metal and in the metal films.
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I. INTRODUCTION

Metal films on substrates have long been of practical importance and theoretical interest. They are widely used in semiconductor-based microelectronics, for instance, as interconnects for integrated circuits and contacts for storage and display. These films, as commonly prepared, are highly disordered.[1,2] This disorder affects many of their electrical[3–5] and mechanical[6,7] properties. The purpose of the present investigation is to provide further insight into this disorder through measurements of their elastic properties below a few degrees K, a temperature range which has been ignored in the previous studies. We have found that in most of the films studied, their internal frictions below ~ 10 K resemble in magnitude those of structurally fully disordered, amorphous solids, regardless of the way in which the films have been produced. In order to understand the cause for this unexpectedly large internal friction, we have also studied systematically deformed high purity bulk Al in the same temperature range. While in fully recrystallized Al the lattice contribution to the internal friction in the superconducting state is found to vanish, as expected for perfect crystals, plastic deformation of such samples leads to a temperature-independent internal friction which for deformations on the order of 10% closely resembles that of Al films. Plastic deformation introduces dislocations into the metal. Therefore, in both the bulk and the films, we consider dislocation tunneling as the most likely cause for the large internal friction observed at the low temperatures of our measurements, although details of the tunneling entities remain unknown. The broad spectral distribution of the tunneling states resembles that of the atomic or molecular tunneling states that cause the elastic anomalies in amorphous solids.[8–11] Practical consequences of the large internal friction in the films for the application of microelectromechanical systems will be discussed, together with a possible way for its reduction.

II. EXPERIMENTAL MATTERS

The low-temperature internal friction of thin metal films was measured on substrates of ultrapure, boron-free silicon wafers shaped as double-paddle oscillators vibrating in their antisymmetric mode at ~ 5.5 kHz as described previously.[12] Most of the monatomic metal films studied in this work, Al, Ag, Au, Cu, In, Mo, and Ti, were deposited by *e*-beam evaporation at a base pressure of 10^{-7} Torr. Deposition rates varied between 10 and 20 Å/sec. The substrates were kept below 100 °C by water cooling. Pb films were deposited thermally in another vacuum chamber at a base pressure of 10^{-6} Torr to avoid cross contamination. Cu films were also prepared by electroless deposition[13] and by sputtering to study the dependence on the preparation methods. To study the possible influence of alloying on the internal friction, two aluminum alloys, Al 6061 and Al 5056, were deposited by sputtering to preserve the composition of the target materials. Sputtering was done in a dc magnetron sputter system in 10^{-2} Torr of Ar with a base pressure of 10^{-7} Torr at a deposition rate of 12 Å/sec with the substrate at room temperature. All films studied, regardless of their preparation methods, were between 300 and 1200 nm thick, except for the Au films on which we searched for the influence of film thicknesses on the internal friction. For this, film thicknesses were varied from as thin as 10 to 310 nm. In order to improve film adhesion, 3 nm Cr films and 2 nm NiCr film were evaporated before depositing the Au and Ag, respectively.

The purity of the monatomic metals used for the deposition varied, but was always better than 99.9%. Sputtering under the conditions used here should lead to an incorporation of Ar in the films estimated at 0.1 at. %.[14] For the electroless deposition process, hydrogen incorporation between 0.05 and 1.3 at. % has been reported.[15] Al 6061 with 0.6% Si, 0.3% Cu, 1.0% Mg, 0.25% Cr (all by weight), is a standard, easily machinable alloy, whereas Al 5056 with 5.2% Mg, 0.1% Mn, 0.1% Cr has an exceptionally high yield stress, which makes this alloy suitable even for nails.[16] Because of its extremely small internal friction in bulk form,[16] which is almost as small as that of high purity, single crystal silicon at low temperatures, it has been studied extensively as a potential antenna for the detection of gravitational waves.[17,18]

In order to vary its grain size, we annealed a Cu film in vacuum at 400 °C for 2 h after *e*-beam evaporation. For this, a 25 nm Ta film was evaporated to act as a diffusion barrier prior to evaporating the Cu film. Grain sizes of the *e*-beam Cu film were determined with an optical microscope after etching with a mixture of 5 ml NH_4OH, 3 ml H_2O, and 3 drops of H_2O_2 (30%). Mean grain sizes after deposition were less than 1 μm, and after a 400 °C vacuum anneal, $\sim$2.5 μm. These results agree with earlier published measurements. For example, on similarly produced unannealed 0.1 μm Cu films, Hentzell *et al.*[19] reported grain sizes between 5 and 50 nm. On 1.0 μm Cu films deposited by sputtering onto room temperature substrates, Gupta *et al.*[20] reported grain sizes between 60 and 600 nm, which increased to between 200 and 1500 nm after a 400 °C anneal. Recently, Keller *et al.*[21] reported medium grain sizes between 0.25 and 0.34 μm for room-temperature sputtered 0.6 μm thick Cu films which increased to 0.91 and 1.03 μm after a 600 °C anneal.

Thin films increase the internal friction of the paddle oscillator Q^{-1}_{paddle}. From this, the internal friction of the film Q^{-1}_{film} was determined by[12]

$$Q^{-1}_{\mathrm{film}}=\frac{G_{\mathrm{sub}}t_{\mathrm{sub}}}{3G_{\mathrm{film}}t_{\mathrm{film}}}(Q^{-1}_{\mathrm{paddle}}-Q^{-1}_{\mathrm{sub}}), \qquad (1)$$

where t and G are thicknesses and shear moduli of substrate and film, respectively, and Q^{-1}_{sub} is the internal friction of the bare paddle. Film thickness t_{film} was determined with a quartz microbalance during deposition (assuming G_{film} to be equal to that of the bulk metal) and was confirmed by stylus profilometry.

The internal friction of bulk Al was measured with a torsional oscillator.[22] The method to determine the internal friction of the sample Q_s^{-1} from the internal friction of the composite oscillator Q_{co}^{-1} is described in detail in Ref. 31. In presenting the data, we use the following equation:

$$Q_s^{-1}=\left[\frac{(1+\alpha)I_q+I_s}{I_s}\right]Q_{\mathrm{co}}^{-1}-\left[\frac{(1+\alpha)I_q+I_s}{I_s}\right]Q_b^{-1}, \qquad (2)$$

where I_q and I_s are the moment of inertias of the quartz transducer and sample, respectively. The quantity α is inserted to account for the finite thickness of the pedestal to which the transducer is epoxied and was found empirically to be $\approx$0.06.[22] The background internal friction of the technique Q_b^{-1} was measured using a second quartz transducer as the sample. In the figures, we have not subtracted the background, instead we show the first and second terms of Eq. (2) separately. The internal friction of the samples can be determined by taking the difference between the two curves.

Polycrystalline rods, 2.5 mm diameter, 99.999% (5 N) nominal purity (supplied by Research and PVD Materials Corp., Wayne, NJ) were plastically deformed at room temperature by stretching in an Instron machine to a maximum deformation of $\epsilon=\Delta l/l=0.2$. All deformed samples were kept at room temperature for two to three days before measuring their internal friction at low temperatures in order to reach a time-independent stage of internal friction.[24] However, some recovery of the internal friction in the deformed samples has been observed even after storage for several months at room temperature. This recovery is known as the Köster effect.[23] Annealing was done for 5 h under vacuum (10^{-6} Torr) at 560 °C, which leads to complete recrystallization of bulk Al, with crystallite sizes on the order of millimeters.[25] All internal friction measurements were performed without any applied magnetic field.

The torsional strain amplitude ϵ_m at resonance of the neck in the double paddle oscillator and of the cylindrical bulk sample in the torsional oscillator is defined as the torsional displacement amplitude of the circumference at their ends divided by the length of the neck or half the length of the bulk sample. For simplicity, the neck of the double paddle oscillator, with a cross section of 0.3×1.1 mm, was taken to be circular, of radius 0.3 mm. The angular amplitude of the neck of the double paddle was determined by measuring the output voltage of the detector circuit, which was calibrated by reflecting a laser beam off the oscillator vibrating in air. The error of ϵ_m is estimated to be a factor of 4, because of the uncertainty with which the capacitor spacing between wing and detecting electrode was determined.

The strain amplitude of the torsional oscillator was measured using a photodiode to detect the angular amplitude of the laser beam which was reflected off the torsional oscillator. Once the reflected laser beam profile was measured with the oscillator at rest by moving the photodiode, the photodiode was placed in one position and the amplitude of the ac component was measured with a lock-in amplifier with the driving oscillator resonant frequency as the reference signal. Cahill and Van Cleve[22] estimated the strain amplitude at resonance for a torsional oscillator with the same geometry by calculating the static strain off resonance, then multiplying by the quality factor of the oscillator. Our measured strain amplitude ϵ_m agrees to within a factor of 2 with this theoretical estimate.

Since the determination of the dislocation density with an electron microscope requires considerable care, in particular because of the risk of losing dislocations during the thinning of the films or during the investigation of the film in the electron microscope,[26] we have so far only estimated the densities by comparison with published values determined for known deformations. Very little is known about the dislocation densities in thin metal films. This will be reviewed in the discussion section.

III. EXPERIMENTAL RESULTS

The solid curve at the bottom of Fig. 1 is the internal friction of the 300 μm thick bare double-paddle oscillator Q^{-1}_{paddle}. It decreases rapidly with decreasing temperature, and reaches a nearly temperature-independent value of $Q^{-1}_{\mathrm{paddle}}\approx 2\times 10^{-8}$ below 1 K, which is reproducible to within 10% for different paddles. Deposition of thin metal films increases Q^{-1}_{paddle} above this background. The large effect of even relatively thin metal films is shown with one pure Al and two Al alloy films. Using Eq. (1) and the shear modulus of bulk Al, the internal friction Q^{-1}_{film} of the films themselves has been computed and is shown in Fig. 2 (the data for the alloy Al 6061 have been omitted, since they are identical to those obtained on pure Al). Figure 2 also contains the internal friction of seven other monatomic metal films. At temperatures below 10 K, their internal frictions are temperature independent and lie in a range spanning less than one order

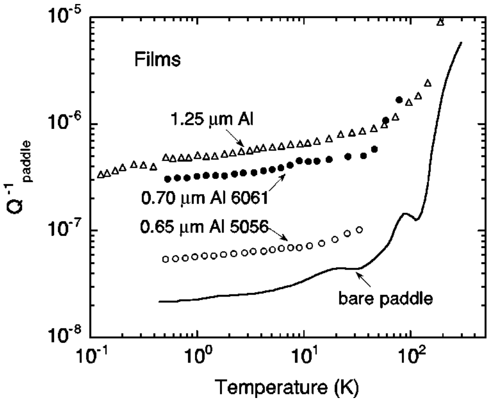

FIG. 1. Internal friction of the bare double paddle oscillator Q^{-1}_{paddle} vibrating in its antisymmetric mode at 5.5 kHz, and of paddles carrying three different aluminum films with thicknesses indicated: *e*-beam evaporated pure aluminum, and the sputtered alloys Al 6061 and Al 5056. None of the films were heat treated after deposition at room temperature.

of magnitude. In order to show the similarity with bulk amorphous solids, the curve for bulk amorphous SiO_2 (a-SiO_2) is also shown,[27] together with a vertical bar labeled ''glassy range.'' The internal frictions of all amorphous solids that have been studied in their bulk forms to date fall within this range. Reviews can be found in Refs. 8,27. The internal friction of all monatomic metal films studied, and also that of the alloy Al 6061, is remarkably close to that found in bulk amorphous solids. The only exception is the alloy Al 5056.

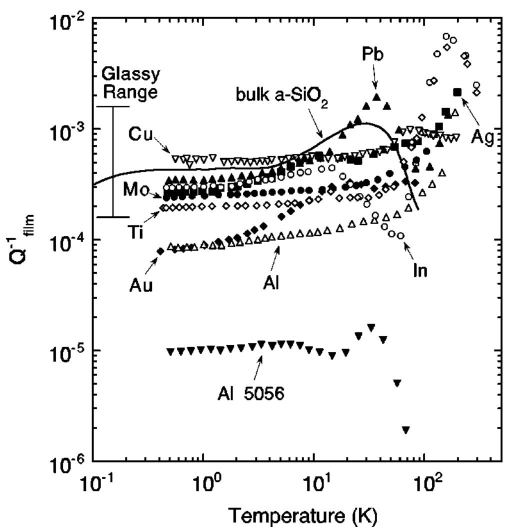

FIG. 2. Internal friction of several metal films Q^{-1}_{film} determined from the same or similar measurements as shown in Fig. 1, using Eq. (1). All films were ~ 1 μm thick, and had been deposited by *e*-beam evaporation, except for the lead film, which was thermally evaporated, and the alloy Al 5056 film, which had been sputtered. Q^{-1}_{film} for the alloy Al 6061 film (sputtered) (Fig. 1) is identical to pure Al and has been omitted for clarity. None of the films were heat treated after deposition at room temperature. The curve labeled a-SiO_2 was measured with a double-paddle oscillator etched out of a 0.125 mm thick wafer of Suprasil W, vibrating at 4.5 kHz (J. E. Van Cleve, Ph.D. thesis, Cornell, published in Ref. 27). The vertical bar indicates the range of internal friction in the temperature-independent region, measured on a wide range of bulk amorphous solids (Ref. 8).

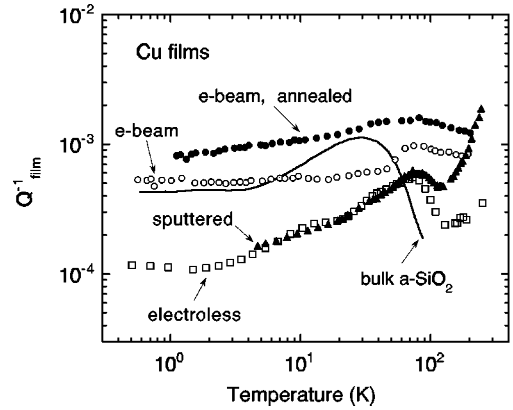

FIG. 3. Internal friction of three copper films produced by different techniques, and for the *e*-beam film after a vacuum anneal to increase the average grain size by at least a factor of 2. The internal friction of the film Q^{-1}_{film} was determined from the damping of the paddle Q^{-1}_{paddle} with the use of Eq. (1). A 25 nm thick Ta diffusion barrier had been used. Solid curve labeled bulk a-SiO_2 is the same as in Fig. 2.

Film preparation appears to have little influence on internal friction. This is demonstrated for Cu films in Fig. 3, in which an *e*-beam Cu film (already shown in Fig. 2) is compared with both sputtered and electroless Cu films. The relatively small range of Q^{-1}_{film}, about a factor of 5, indicates that neither the Ar in the sputtered films nor the hydrogen expected in the electroless films has a significant effect. Annealing of the *e*-beam film at 400 °C for 2 h increased the internal friction by a factor of 2. As a control for contamination of the Si paddle during the annealing, we subsequently removed the Cu film with a chemical etch which left the Ta diffusion barrier intact. The internal friction of the paddle was then found to be close to that expected for a paddle carrying a metal film of 25 nm thickness (using an average of the internal frictions shown in Fig. 2). From these measurements, we estimate the contribution of volume or surface contamination of the silicon oscillator to be less than 2% of the effect caused by the Cu film.

Most of the films shown in Figs. 1–3 were 0.5–1 μm thick. However, the internal friction of films was found to depend only little on film thickness. In Fig. 4, the thicknesses of Au films were varied over a factor of 30, from 10 to 310 nm. Their internal friction was found to be the same within a factor of 3.[28]

In an attempt to identify the defects leading to the unexpectedly large internal friction in these metal films, we also measured the internal friction of plastically deformed bulk Al with a torsional oscillator operating at ~ 90 kHz. For the well-annealed, fully recrystallized high purity Al, the internal friction Q^{-1} shows a steep drop off as the temperature decreases below $T_c = 1.19$ K (the superconducting transition temperature of Al), shown in Fig. 5. It is known that in perfect crystals the large internal friction above T_c is caused by electron-phonon interaction.[29] In order to separate structural and electronic contributions to the internal friction, the measurements were extended to ~ 0.06 K — well below T_c — where the electronic contribution becomes negligibly

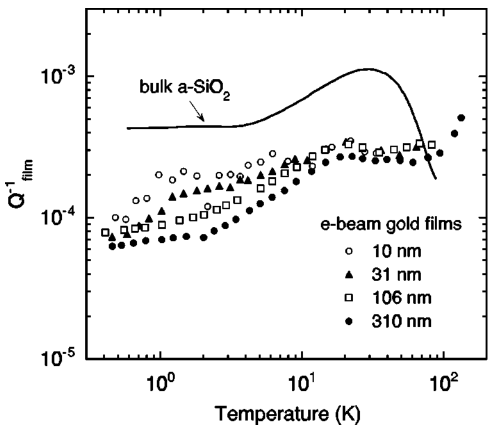

FIG. 4. $Q^{-1}_{\rm film}$ of four gold films of different thicknesses, as determined from the damping of the paddle $Q^{-1}_{\rm paddle}$ using Eq. (1). Solid curve labeled bulk a-SiO_2 is the same as in Fig. 2.

small. Using Pippard's theory,[29] which has been tested thoroughly for bulk Al, we tested our results by determining an electron mean free path l from the internal friction observed on the annealed sample above T_c, $Q^{-1}=5.7\times10^{-5}$, and obtained $l=4\times10^{-3}$ cm. The residual resistance ratio measurement of $RRR=3000$ made on a twin sample also gave the same mean free path. Such mean free paths are reasonable for the chemical purity of the Al we used.

Below T_c, the steep drop off of the internal friction is caused by the decreasing concentration of the unpaired electrons. This is demonstrated by comparing the measured internal friction with a theoretical calculation done by Kogure *et al.*[30] from T_c down to 0.4 K. These authors used this calculation to fit measurements of the ultrasonic attenuation in single crystal Al oriented along a $\langle 100\rangle$ direction. Using as the input parameter the internal friction we had measured on the annealed Al at T_c, our data on the annealed sample below T_c also agree well with the theoretical calculation, as shown in Fig. 5 as a solid curve. Below $\sim$0.3 K, the internal friction of the annealed Al sample has a value of $\approx 10^{-6}$ after subtracting the background.

A plastic deformation of $\epsilon=1\%$ in the Al sample causes an increase of the internal friction over the entire temperature range. For $\epsilon>10\%$, Q^{-1} is nearly temperature independent, with a magnitude approaching that of amorphous dielectrics (the ''glassy range,'' see Fig. 2). Note that the largest internal friction occurs at $\epsilon=15\%$; it decreases slightly for $\epsilon=20\%$. We repeat, however, that some decrease of the deformation-induced internal friction occurred after storing samples at room temperature for several months. Thus, a quantitative relation between plastic deformation and low-temperature internal friction cannot be made at this time. The as-received sample has an internal friction which is nearly identical to that of the Al film. Both sets of data are plotted in Fig. 5 for comparison. Annealing at 560 °C for 5 h restores the small internal friction of the undeformed bulk sample. (In fact the curve labeled ''annealed'' was measured on the as-received sample after such an anneal.)

The attenuation of the elastic waves also appears to affect the speed of sound. For the same bulk samples as those in Fig. 5, we show in Fig. 6

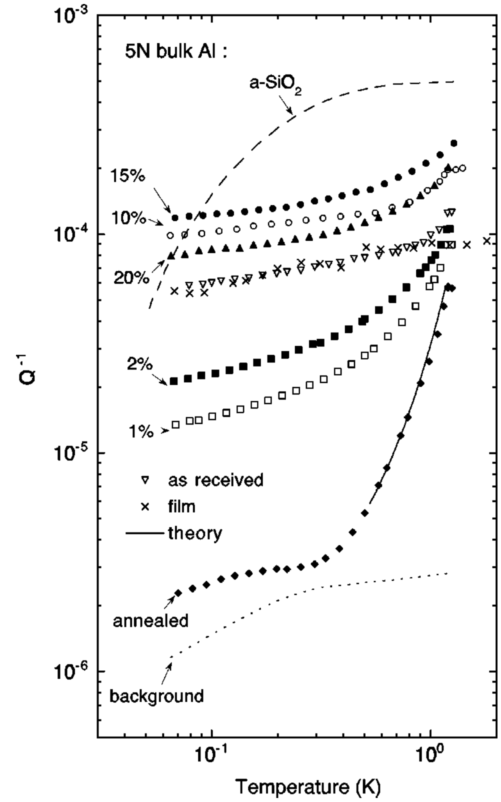

FIG. 5. Internal friction of annealed high purity (5 N) aluminum rods 2.5 mm diameter, 18 mm long, measured with a torsional oscillator vibrating at 90 kHz. In presenting the data, we show the first and second terms of Eq. (2) separately. The internal friction of the Al samples can be determined by taking the difference between the first term and the second term (dotted line) as described in the text. Thus for the annealed sample, the internal friction below $\sim$0.3 K is $\approx 10^{-6}$. Above T_c, the internal friction is temperature independent (not shown). The solid curve at temperatures $T\leqslant T_c$ is a fit as explained in the text. The ''as-received'' sample has an internal friction almost indistinguishable from that of the Al film (same data as in Fig. 2). The dashed curve is bulk a-SiO_2, measured at 66 kHz, see Ref. 8 (based on data contained in Ref. 34).

$$\left(\frac{\Delta v}{v_0}\right)=\frac{v(T)-v_0}{v_0}, \tag{3}$$

where $v(T)$ is the transverse speed of sound at temperature T, and v_0 that of the lowest temperature of measurement, $\sim$60 mK. $\Delta v/v_0$ varies nearly linearly with T, and its temperature derivative is generally larger for samples with a larger internal friction. An exception is the 10% deformed sample which has a $\Delta v/v_0$ comparable to that of the 2% deformed sample, although their internal frictions differ by a factor of 5. For the films, a similar temperature dependence of $\Delta v/v_0$ was observed, but these measurements do not yet extend below 0.5 K, and are not shown. For comparison, the temperature dependence of $\Delta v/v_0$ in a-SiO_2 is also plotted in Fig. 6.

A striking dependence of the internal friction of plastically deformed bulk Al on the strain amplitude was also observed; see Fig. 7. For the sample which was plastically deformed by 1%, the internal friction at 65 mK increased by a factor of 5 when the strain-amplitude increased from ϵ_m

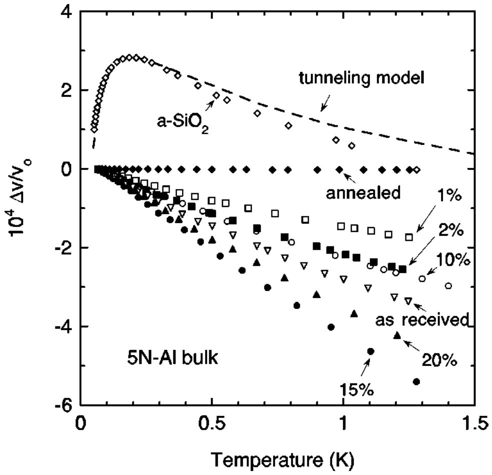

FIG. 6. Variation of the transverse speed of sound $\Delta v/v_0$ in the pure Al samples shown in Fig. 5. The linear variation with temperature extends to the lowest temperatures of measurement, T_0 at which the velocity v_0 was measured. Data for a-SiO_2, at 66 kHz, and the tunneling model fit (dashed curve), after Ref. 34.

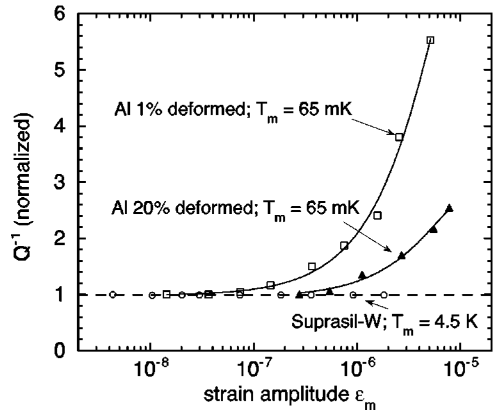

FIG. 7. Dependence of the internal friction on the strain amplitude ϵ_m of high purity (5 N) bulk Al plastically deformed in tension by 1 and by 20 %, measured at the temperature $T_m = 65$ mK, normalized to the internal friction at low strain amplitude. In the same range of strain amplitude, a-SiO_2 shows no strain-amplitude dependence.

$=1\times10^{-8}$ to 5×10^{-6}. For the more heavily deformed sample ($\epsilon=20\%$), the strain-amplitude dependence was somewhat smaller. We also measured the line shapes of the torsional resonant mode. With increasing strain amplitude, the line shapes became distinctly non-Lorentzian. In bulk a-SiO_2, we found no dependence in this range of strain amplitude, as is also shown in Fig. 7. For the sake of completeness we mention that at temperatures below 1 K, the internal friction of the bulk a-SiO_2 does appear to increase with increasing strain amplitude. However, this has a purely experimental origin. The increase in strain amplitude causes an increase in power dissipation which leads to an increase in sample temperature. Since the internal friction of a-SiO_2 increases with temperature at these low temperatures (see Fig. 5), an increase in internal friction is observed. In the deformed Al, this effect is absent, since its internal friction is nearly independent of temperature. We also observed hysteresis in tracing out curves on Al such as the ones in Fig. 7, which we interpret as evidence for microplasticity. This effect needs to be studied in more detail. A similar strain-amplitude dependence was also observed in metal films, as shown for Al in Fig. 8. The alloy Al 5056 is again an exception. Its very small internal friction is shown in Fig. 2. This film has almost no strain-amplitude dependence, as shown in Fig. 8. All measurements shown in Figs. 1–6 were taken at low strain-amplitudes in which neither strain-amplitude dependence nor hysteresis was noticeable.

IV. DISCUSSION

A. Comparison with amorphous solids

In amorphous solids, the temperature-independent internal friction at low temperatures, which indicates a wide, uniform range of energies and relaxation times, is believed to be caused by tunneling of atoms or groups of atoms.[9–11] The low-temperature internal friction of the heavily deformed bulk aluminum and of the Al films resembles that of amorphous solids. We will begin by inspecting this similarity as well as differences more closely, and will then turn to the question of possible causes for the elastic properties of the crystalline metal.

In amorphous solids, the low-temperature internal friction is independent not only of the temperature, but also of the measuring frequency,[8] a fact that is well described with the tunneling model. The metal films were measured at 5.5 kHz, and the bulk Al at 90 kHz, and the internal frictions of the Al film and of the most heavily deformed (''as received'') bulk Al are equal. This apparent independence of measuring frequency is suggestive, although it must be emphasized that a proper test would be to measure either the films or the bulk over a wide frequency range. A difference between amorphous solids and the metals, however, is that the internal friction of the latter does not decrease at the lowest temperatures of measurement, in contrast to dielectric amorphous solids, see Fig. 5. According to the tunneling model, this decrease occurs when the shortest relaxation time of the tunneling defects exceeds the period of oscillation as the temperature decreases. Yet, the absence of a similar drop off for the metal may only indicate a stronger coupling between the low-energy excitations and the phonons, and not a disagreement with the tunneling model. Another difference is observed in the temperature dependence of the speed of sound shown in Fig. 6. For metals, a linear increase of $\Delta v/v_0$ with decreasing temperature dominates the low-temperature speed of sound down to the lowest temperature measured. A similar linear increase, although at temperatures above a few K, is also observed in amorphous solids, which is called the Bellessa effect.[32] It is not explained by the tunneling model,[11] and is also observed in disordered crystals, even if their lattice vibrations do not resemble those of amorphous solids.[33] However, in amorphous solids, the linear increase of $\Delta v/v_0$ usually gives way to a logarithmic one as the temperature is decreased below a few K, and this slower rise is followed by a peak at the so-called crossover temperature T_{co}, and then a logarithmic decrease below T_{co}, in agreement with the tunneling model.[11] The dashed curve at the top of Fig. 6 is such a fit to $\Delta v/v_0$ for bulk a-SiO_2, which describes the data well.[34] In contrast, no evidence for such a slow, logarithmic rise followed by a crossover is found in any of the deformed bulk Al samples studied here. Conceivably, T_{co} could occur at a temperature below the range of our measurement, also as a result of strong coupling. We will return to this point. We therefore conclude that although there is some evidence that the metal films and the heavily deformed bulk Al have excitations similar to those of amorphous solids, clearly, more work is required, in particular at lower temperatures, to settle this issue.

B. Mechanisms for the damping

We now turn to the discussion of the kind of defects causing the elastic properties observed in this work. Possible defects are electrons, impurities, grain boundaries, interfacial bonding, and dislocations. In pure, well annealed crystalline materials, the influence of *electrons* can be large.[29] An example was shown in Fig. 5 close to T_c, where damping by electrons leads to an internal friction $Q^{-1}=5.7\times10^{-5}$. In even more perfect Al single crystals ($RRR>20\,000$, electron mean free path $l\approx1$ mm at 4.2 K), Kogure *et al.*[30] measured a temperature-independent ultrasonic absorption above T_c which corresponds to an internal friction $Q^{-1}=2\times10^{-4}$. In the metal films we studied, in which we measured values of $RRR\approx2$, however, the influence of electrons should be negligible according to Pippard's theory. This conclusion is confirmed by the complete absence of any change of the internal friction at T_c in the films of Al, In, and Pb (in the bulk, T_c $=1.2$, 3.4, and 7.2 K, respectively); see Figs. 1 and 2. *Impurities* can also be ruled out as causes for the large internal friction, since it is improbable that their effect should be similar for all films studied, which are expected to contain a wide range of impurities, or admixtures in the case of the alloy Al 6061. Further evidence for the insensitivity to impurities is contained in Fig. 3: In the Cu films, neither Ar nor H has a noticeable effect on the internal friction, as compared to the *e*-beam film, which contains only the impurities of the starting material and those picked up from the residual gas during the evaporation. Weakly bonded atoms at or near *grain boundaries* could lead to relaxations at low temperatures, and thus to internal friction. By annealing, the grain sizes of the copper film were increased by more than a factor of 2, which decreased the total length of the grain boundaries contained in the film by about the same factor. Yet, as seen in Fig. 3, this grain growth did not lead to a decrease of the internal friction, but rather to an increase. This observation rules out grain boundaries as cause for the large internal friction. The increase observed upon annealing will be discussed further in the following section. If poor *interfacial bonding*, or disorder, or contamination at the interface would dominate the internal friction, their effect on the measured internal friction of a paddle (Q^{-1}_{paddle}) carrying a film should not scale with the film thicknesses. Yet, our measurements of paddles carrying gold films ranging in thicknesses from 10 to 310 nm show Q^{-1}_{paddle} to increase linearly with film thickness, as expected if the internal friction occurred in the film itself [see Eq. (1)], rather than at the bonding interface. Evaluating the measurements using Eq. (1), an internal friction of the film Q^{-1}_{film} is obtained which varies very little with thickness, as shown in Fig. 4. This demonstrates that at least the majority of the damping occurs in the film and not at the interface.

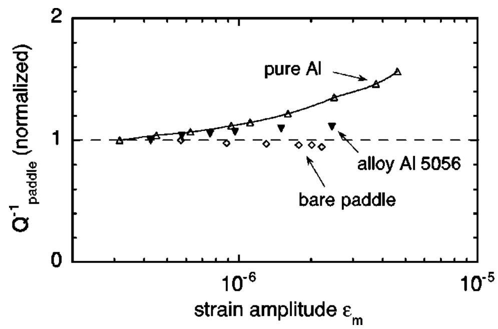

FIG. 8. Dependence of the internal friction on the strain-amplitude ϵ_m of a bare double paddle oscillator and of oscillators carrying a pure Al film, and a film of the alloy Al 5056, respectively. Internal friction is normalized to its value at low strain-amplitude.

These observations leave *dislocations* or pieces thereof, like kinks, as the most likely causes for the large internal friction in the films, and will be discussed below. To begin, we will consider dislocations in bulk metal. Ultrasonic attenuation by thermally activated relaxation of dislocations in bulk metals is well known. Since we are concerned with the elastic properties below 1 K, we will review only work done in that temperature range, in which relaxation is expected to occur via tunneling of dislocations,[35,36] as first suggested by Mott.[37] The first observation of dislocation relaxation at low temperatures was reported by Alers and co-workers,[38] who observed through ultrasonic measurements a nearly temperature-independent damping and an almost linear rise of the speed of sound with decreasing temperature in bulk, undeformed, high purity copper at temperatures as low as 0.1 K. They concluded that a broad range of activation energies E, extending over at least two orders of magnitude (E/k_B ~0.1 to 10 K), was required in order to describe these results. The slope of the temperature variation of $\Delta v/v_0$ was found to decrease with γ irradiation, which was explained by dislocation pinning. Measurements between 2 and 9 K on an Au bar yielded qualitatively similar results. Furthermore, both the damping and the slope of $\Delta v/v$ of the Au bar increased after a plastic deformation by 4%. Hikata and Elbaum[39] performed at low temperatures sound velocity measurements on a plastically deformed single crystal Al while a second ultrasonic wave was traveling simultaneously in a perpendicular direction, in order to provide periodic stresses. They explained their results with the tunneling model and suggested that the broad range of tunneling states was caused by the motion of dislocation kinks. Strain amplitude dependent internal friction (commonly referred to as ADIF), a characteristic feature of dislocations in metals, is explained with the unpinning of dislocations,[40,41] and evidence for this at low temperatures in bulk high purity, undeformed single crystal Al has been studied in the helium temperature range.[30,42] Recently, Kosugi *et al.*[43] extended these measurements to 0.1 K. They interpreted the temperature-independent ADIF observed below 0.5 K in undeformed high purity single crystal Al (in zero magnetic field) with a relaxation by tunneling of dislocation loops consisting of 100 atoms. All of the observations reviewed here support the picture of dislocation tunneling at low temperatures, and some of the measurements also indicate the existence of a broad range of tunneling state splittings.

C. Dislocation motion in bulk and films

In order to analyze our data on bulk Al, we need to know the dislocation densities in the deformed samples. Numerous measurements of dislocation densities in plastically deformed metals have been reported. For aluminum, we refer to Refs. 44–52. Although the absolute magnitude for a given deformation varies within one order of magnitude between different investigators, they all reported an approximately linear variation with plastic strain ϵ for $0.01<\epsilon<1.0$. The strain ϵ is not to be confused with the measuring strain amplitude ϵ_m introduced above. We will therefore assume in our samples a dislocation density of

$$n=(10^{11}\ \mathrm{cm}^{-2})\epsilon, \tag{4}$$

with the prefactor accurate to within a factor of 3 up or down. In carefully annealed samples, densities $n<10^8\ \mathrm{cm}^{-2}$ have been reported.[25,53,54]

We now consider our measurements. For plastic deformation ϵ up to 15%, i.e., dislocation densities on the order of $10^{10}\ \mathrm{cm}^{-2}$, the low-temperature internal friction increases with increasing plastic deformation, see Fig. 5. A similar behavior is seen for the temperature variation $\Delta v/v_0$ of the sound velocity — see Fig. 6 — which shows that in the more highly deformed samples the sound velocity generally varies more rapidly with temperature, although there is an exception observed for the 10% deformed sample, indicating that the connection is not that simple. Larger plastic deformation leads to a saturation or even to a small decrease of the internal friction and of the slope of $\Delta v/v_0$. This saturation is particularly apparent for the ''as-received'' sample. Given the way such rods are drawn[55] at room temperature with no subsequent anneal,[56] their plastic deformation ϵ should greatly exceed $\epsilon=100\%$, and their dislocation density might be larger than $10^{10}\ \mathrm{cm}^{-2}$, although highly nonuniformly distributed.[44] The fact that the internal friction and the magnitude of the temperature derivative of $\Delta v/v_0$ increase with deformation for small ϵ, as does the dislocation density, suggests that the elastic effects are caused by individual dislocations, at least up to dislocation densities of $\sim 10^{10}\ \mathrm{cm}^{-2}$. At higher dislocation densities, they become entangled, which reduces their mobility. This may lead to the observed saturation.

The measurements discussed so far were done in the limit of small measuring strain-amplitude ϵ_m and were independent of the latter. Convincing evidence that the elastic properties of the deformed bulk Al are caused by dislocation motion is obtained at large strain amplitudes, when ADIF is observed; see Fig. 7. The fact that the ADIF decreases with increasing plastic deformation indicates that the dislocation mobility decreases at larger dislocation densities. Further evidence for dislocation motion is obtained from the irreversible changes of the internal friction which are noticed after measuring the sample at high strain-amplitudes. The latter effect is explained as microplasticity caused by dislocation motion.[57] Similar irreversibilities have been previously reported on undeformed Al.[42]

Based on this experimental evidence, we conclude that the elastic effects observed on the plastically deformed Al are caused by the relaxation of dislocations, or parts thereof, such as dislocation kinks, although it must be emphasized that convincing evidence for this interpretation is only observed at large strain amplitudes. At small strain amplitudes, other mechanisms cannot be ruled out at this point. Because of the low temperatures, relaxation by tunneling is considered as a likely mechanism. Following Hikata and Elbaum,[39] we suggest that the independence of the internal friction on temperature we observe indicates the existence of a broad spectrum of tunneling splittings, similar to those observed in amorphous solids. The linear temperature dependence of the speed of sound remains without explanation, just as it does for amorphous solids[11] and disordered crystals.[33]

At this point, we refer to a series of experiments which appear to be closely related to the work reported here. Esquinazi and co-workers[58–60] have studied elastic constants and internal friction of thin metal wires and foils, and in several cases found a behavior resembling that of amorphous solids. In a commercial polycrystalline Al wire, for example, 0.02 mm diameter, 99.99% purity, and vibrating at $\sim$7.7 kHz,[59] they reported a nearly temperature-independent internal friction $Q^{-1}\sim 1.9\times 10^{-4}$, close to the values found on the highly deformed Al rods shown in Fig. 5. They also reported that a similar anomaly in a 0.025 mm Pt wire was reduced by a factor of 3 by an anneal at 1300 K.[59] According to Bordoni *et al.*,[61] this heat treatment should reduce the number of dislocations observed in the Bordoni peak by about an order of magnitude. Although no attempt was made to identify the defects and impurities involved, the samples used by Esquinazi and co-workers must have contained high dislocation densities, similar to those in our ''as received'' Al sample, since all commercially prepared wires and foils have undergone considerable plastic deformation during their manufacture. We therefore suggest that the phenomena they reported were also caused by dislocations. However, some of their results disagree with our findings. For example, on the Al wire just mentioned, they observed a maximum in the temperature-dependent variation of $\Delta v/v_0$, occurring at $T_{\mathrm{co}}=0.25\ \mathrm{K}$,[59] which we did not observe (Fig. 6). Moreover, they reported good agreement of both their speed of sound and internal friction measurements with the tunneling model,[11] while clear deviations are found in the present study (see Figs. 5 and 6). Recently, Classen *et al.*[62] have performed vibrating reed measurements at 5.5 kHz on a single crystal Al, which had not been intentionally deformed.[63] They reported a maximum of $\Delta v/v_0$ at a much lower temperature, 0.03 K, while the nearly temperature-independent internal friction was close to that of our 2% deformed sample (see Fig. 5). A damping of this magnitude appears reasonable, since the reed was almost certainly deformed during its preparation, and had not been annealed prior to the measurement. In the temperature range of overlap, their speed of sound data agree with those we obtained on deformed Al, in contrast to those by König *et al.*,[59] as shown in Fig. 9. Classen *et al.*[62] found their experimental results to be inconsistent with the tunneling model. We suggest that these discrepancies are the results of trace impurities in the samples, which are known to influence greatly the dislocation motion.[38,42,64] It is also conceivable that the

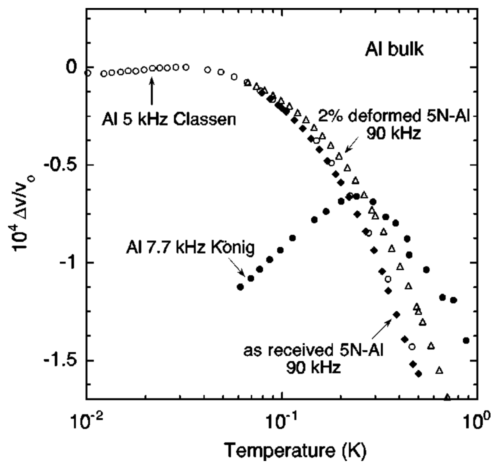

FIG. 9. $\Delta v/v_0$ of bulk Al after König *et al.* (Ref. 59), Classen *et al.* (Ref. 62), and this work.

somewhat irregular variation of the speed of sound observed in the 10% deformed sample shown in Fig. 6 has also been caused by accidental impurities. We conclude that while some quantitative discrepancies exist among the measurements by different research groups, the results agree qualitatively with each other, and can be explained by the tunneling of dislocations caused by the plastic deformation.

We now consider dislocations in the films and the effects they may have on their internal friction. Thin films are under large stresses, either as a result of thermal mismatch between substrate and film, or as internal or growth stress.[6] When these stresses exceed the yield stress, dislocations will form. Although there is wide agreement that their densities range from 10^9 to as high as 10^{12} cm^{-2} (Refs. 65–67), measurements appear to be scarce. In continuous Au and Ag films on mica and MoS_2, Pashley and co-workers[68] determined with electron microscopy typical dislocation densities of 10^{10} cm^{-2}. In very thin discontinuous films (<30 nm), however, dislocation densities of 10^9 cm^{-2} and less were observed.[68] In Cu films ~ 1 μm thick, Keller *et al.*[21] recently reported $3\sim 4\times 10^{10}$ cm^{-2}, determined from x-ray line broadening. In Pb films, Murakami[69] reported densities on the order of 10^{10} cm^{-2}, although only in grains larger than 0.6 μm in diameter. In smaller grains, no dislocations were detected. Based on this admittedly limited knowledge, a dislocation density on the order of 10^{10} cm^{-2} is not an unreasonable assumption for the thin films studied here. Such a high dislocation density can explain why the Al film has an internal friction equal to that of the most highly deformed, ‘‘as received,’’ bulk sample, as shown in Fig. 5.

Damping by dislocations can perhaps explain why the Cu film showed an increase in internal friction upon annealing, as shown in Fig. 3. In Cu films, the internal stress at room temperature has been shown to increase after a high-temperature anneal.[21] This leads to an increase in dislocation density, and thus may increase the low-temperature internal friction. This argument is not entirely convincing, since at very high dislocation densities their entanglement may also lead to a decrease of the damping, or at least to a saturation. More convincing evidence for the role of dislocation motion in the metal films is obtained with the alloy Al 5056. In bulk form, it has a high yield stress and also an exceptionally small internal friction, less than 2×10^{-8} from 10 K to the lowest temperatures of measurement, 50 mK.[16] These observations have been explained assuming that its dislocations (or dislocation kinks) are immobile.[16] We suggest that in the sputtered thin film of this alloy, even without any special heat treatment, this low mobility has been partly preserved as shown by the unusually small internal friction of this film in Fig. 2. Our conclusions are supported by the observation of the strain amplitude dependence in the Al film, and its absence in the alloy Al 5056. The fact that the ADIF in the Al film is observed at relatively large strain amplitudes compared to bulk Al particularly for $\epsilon = 1\%$, see Figs. 7 and 8, is explained by the fact that thin metal films have greatly enhanced yield stress relative to that of pure bulk metals, which has been explained by strong pinning of the dislocations in the films.[2,6,70] Previously, ADIF in Al films on silicon substrates has been observed in measurements above 180 K,[71] where it has been shown to set in at strain amplitudes exceeding $\epsilon_m \sim 10^{-5}$.

One potentially serious objection to the picture of dislocation tunneling in the films may be the observation that the internal friction is unchanged even in the thinnest gold films we investigated, shown in Fig. 4. According to Pashley,[68] dislocation densities in Au on MoS_2 substrates vanish for film thicknesses less than ~ 30 nm, while we continue to observe an unchanged internal friction even at 10 nm. In order to settle this question, dislocation densities will have to be determined on the same films as used in our elastic measurements on Si substrates.

Regardless of the mechanism underlying it, the large internal friction observed in the metal films is expected to have practical consequences. We mention two recent examples of micromachined mechanical oscillators in which gold films were used to drive and detect the vibrations of the silicon beams. With the dimensions given, and using the internal friction of the gold films determined here, we estimate for the oscillator of Greywall *et al.*[72] a contribution of $\Delta Q^{-1} = 1.2\times 10^{-7}$ or 5% of the temperature-independent part of the measured damping at low temperatures, $Q^{-1} = 2.2\times 10^{-6}$ to be caused by the gold film. For the oscillator described by Cleland and Roukes,[73] the gold film is calculated to contribute $\Delta Q^{-1} = 2\times 10^{-5}$ or 40% of the internal friction observed at 4.2 K ($Q^{-1} = 5\times 10^{-5}$). In both cases, the use of a metal film with little damping, such as the alloy Al 5056, would have reduced the film losses by an order of magnitude. Heat treatment may decrease the internal friction of these films even further, as it does in bulk samples.[16]

V. CONCLUSION

We have found a large internal friction in numerous metal films and have shown that it resembles that obtained in highly deformed bulk Al in the superconducting state. Our measurements indicate the presence of a uniform density of states of defects in both the films and in the deformed bulk Al. These states can relax at the lowest temperatures investigated, which is 0.065 K in the bulk Al. This low-temperature relaxation suggests that tunneling is the most likely mechanism. The nearly linear temperature variation of the speed of sound below 1 K remains without explanation. The tunneling entities are also unknown, but we suggest dislocations or pieces thereof, such as dislocation kinks. Apart from the fundamental issue of understanding their physical nature, the point to be stressed is that we have found that many metal films and also highly deformed bulk aluminum have a remarkably large internal friction at low temperatures, almost comparable to that of structurally fully disordered amorphous solids. This comparison illustrates the large disorder present and should be considered, for example, when designing mechanical oscillators in which large elastic quality is desired. Conceivably, this disorder may also influence the electrical transport properties of metal films. For these reasons, controlling this source of the damping may be desirable.

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