DAMPING AT HIGH HOMOLOGOUS TEMPERATURE IN PURE Cd, In, Pb, AND Sn

L. S. Cook and R. S. Lakes

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Introduction

Lazan (1) recognized in the late 1960's that the importance of material damping as an engineering property in the analysis and design of machines and dynamically loaded structures is an important motivation for material damping studies. Yet, it is only recently that the basic principles of materials science have been used to optimize the damping of engineering structures (2). Possibly the largest obstacle to the incorporation of material damping as a design parameter in structural members is the scarcity of high stiffness-high damping materials.

Typically, if a material possesses the stiffness necessary to be considered a structural material, its damping is low. Conversely, materials with high damping usually do not possess the stiffness necessary to be considered a structural material. Thermodynamic considerations mitigate against the simultaneous storage (high stiffness) and dissipation (high loss) of energy. Composite materials may provide a way to optimize the desired properties. Chen and Lakes (3,4) have shown that composite materials with certain mesostructures can yield high stiffness and high damping. Candidate materials for the high stiffness-low damping phase exist in abundance, whereas candidate materials for the moderate stiffness-high damping phase is metals at high homologous temperatures.

It is well known that above about $0.5T_{\rm H}$ (where $T_{\rm H}$ is homologous temperature, defined as the ratio of actual absolute temperature to melting temperature), mechanical damping increases rapidly with temperature in metals. Kê (5) was first to systematically study this effect in connection with his studies on the damping peak he believed to be due to grain boundary sliding. Initially, there was little interest in the phenomenon and it was mainly considered a nuisance as it obscured observation of the grain boundary peak. It was thus given the unflattering name of "high temperature background." Investigators studying high-temperature phenomena invariably observed the background; it managed to insinuate itself into the literature in this manner. These early results are summarized in the reviews of Niblett and Wilks (6) and Mason (7). Nowick and Berry (8), discussed the background in their classic reference on anelastic behavior. They observed that its magnitude is highly structure sensitive in that it is smaller in single crystals than in polycrystals; it is smaller in coarse-grained polycrystals than in fine-grained polycrystals; it is enhanced in deformed and partially-recovered or polygonized samples; and it is reduced by annealing treatments at successively higher temperatures. Its temperature dependence is usually well characterized by an expression of the form

$$\tan = A \exp(-U / kT).$$

(1)

where tan is mechanical damping, k is Boltzmann's constant, T is absolute temperature and A and U are empirical constants determined from the intercept and slope, respectively of a semi-logarithmic plot of tan against 1/T. Several mechanisms have been proposed for the background, but as all are thermally activated, their behavior with respect to the temperature cannot be distinguished. It is generally agreed upon, however, that the background is caused by a combination of thermally activated dislocation mechanisms. The frequency dependence of the background has been discussed by Schoeck, Bisogni, and Shyne (9). They supposed a generic thermally activated dislocation point defect mechanism and arrived at an expression of the form

$$\tan = \frac{K}{\left[\exp\left(U_o / kT\right)\right]^n},\tag{2}$$

where U_0 is the activation energy of the rate-controlling process, is circular frequency, and K and n are constants. They also observe that the parameter U from Eq. 1 should be related to the true activation energy of the rate-controlling process by U=nU₀.

The development by Woirgard, Sarrazin, and Chaumet (10) of a pendulum instrument capable of isothermal damping measurements over a wide range of frequencies (roughly 10^{-5} to 10 Hz, six decades) proved to be impetus for the more recent interest in the background. Experiments on 99.999% Al (11) yielded results which followed the form of Eq. 2 with n=0.45. Interestingly, in this case, the grain boundary peak obscured the now "low frequency background" to the extent that it could not be determined whether the background continued to higher frequencies or approached an asymptotic value.

A ten decade frequency range up to 10 kHz under isothermal conditions near or below ambient temperature is obtainable with the instrument developed by Chen and Lakes (12) for the dynamic characterization of foams, polymers, and viscoelastic elastomers. Recent modifications to the instrument (13) make possible experiments on stiffer and lower-damping materials such as metals.

The primary motivation for this study is the identification of candidate materials possessing moderate stiffness and high damping for use in composite materials with high stiffness and high loss. An important secondary motivation is to contribute to the state of knowledge of the mechanism of the high temperature-low frequency background. We present results of experiments conducted on specimens of In, Sn, Cd, and Pb. These are metals with low melting points: 156.6°C for In, 232°C for Sn, 320.9°C for Cd, and 327.5°C for Pb. All of them are above 0.45 T_H at ambient temperature. Tests were conducted at frequencies from 10⁻⁵ to 10⁴ Hz. Results and discussion are presented in the context of both motivations.

Materials and Methods

Indium (99.9%) was obtained in shot form (all materials were obtained from Johnson Matthey Alfa). The shot were cleaned chemically in a 50% solution of H₂SO₄ in water to remove surface oxide, cleaned mechanically to remove any film from the chemical cleaning process, then rinsed in acetone and in methanol. The shot were then placed in a Pyrex tube with inner diameter of 3.175 mm that was subsequently evacuated with a mechanical vacuum pump and backfilled with standard purity argon at atmospheric pressure. Tin (99.9985%) was obtained in the form of a 6 mm diameter rod. Indium and tin are both above $T_H = 0.6$ at ambient temperature, thus no further heat treatment was deemed necessary. After mounting, however, the In and Sn specimens were allowed to sit at room temperature for 24 hours before testing. Cadmium (99.9995%) was obtained in the form of 2 mm diameter rods. Lead (99.9999%) was obtained in the form of a 5 mm diameter rod. The Cd and Pb rods were annealed at 85°C ($T_H = 0.6$) for one hour to relieve the effects of any cold work that may have been inadvertently introduced during handling. Annealing took place in an environment chamber that had been evacuated with a mechanical pump and backfilled with standard purity argon at atmospheric pressure.

Tests were performed using the modified apparatus of Chen and Lakes (12,13). One end of the specimen was glued (with a cyanoacrylate cement) to the rigid framework and a high intensity neodymium iron boron magnet and mirror were glued to the other end. A sinusoidal voltage from a digital function generator was applied to the Helmholtz coil which in turn caused an axial torque on the magnetic disk, and thus the specimen. Angular displacement was measured using one of two methods. In the first method, light from the laser was first passed through a grating; an image of the grating was thus formed on the specimen's mirror. The reflected image was then passed through a second grating to a light detector. The number of interference fringes produced by superposition of the two gratings was recorded with the angular displacement being proportional to the fringe number. In the second method, light from the laser was reflected from the specimen's mirror to a split-diode light detector. The output from the detector was applied to a differential amplifier, the action of which was to produce a sinusoid in time proportional to the specimen's angular displacement.

Frequency was recorded from the function generator. From 10^{-5} to 10^{-2} Hz, the input and output voltages and the phase difference between the two signals were taken from a strip chart recorder; from 10^{-2} to 10^{0} Hz, the input and output voltages and the phase difference between the two signals were taken from a digitizing oscilloscope; and from 10^{0} to 10^{4} Hz, the voltages and phase difference were taken from a digital lock-in amplifier. At frequencies significantly below that of the specimen's first torsional resonance, stiffness was calculated using the quasistatic relation

$$G = \frac{T}{J}$$
(3)

where G is the shear modulus, T is the applied torque, *I* is the specimen length, J is the second moment of area, and is the angular displacement. Damping was taken to be approximately equal to the measured phase difference

tan (4) At frequencies approaching the specimen's first torsional resonance, stiffness and damping were calculated by numerical solution (12) of an exact relationship for the torsional behavior of a viscoelastic cylinder (8):

At the frequency of the specimen's first torsional resonance, damping was calculated using the shape of the frequency response curve:

tan	= ,	(:	(5))
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where is the difference between the two so called "half-power frequencies."

Each specimen was tested at audio and sub-audio frequencies at a temperature of 23° C. In most experiments, input voltage (and thus shear stress) was held constant. However, these specimens displayed amplitude-dependent damping at strains above about 10 microstrain. A plot of ln(tan) against 1/ gives a straight line for larger values of , thus it is reasonable to attribute the nonlinearity to dislocation breakaway as described by the theory of Granato and Lücke (14,15). This mechanism is well known and is not the subject of this investigation. Consequently, shear stress was adjusted so that the resulting shear strain was always less than 1 microstrain (10⁻⁶), thus ensuring linear damping.

Results and Discussion

Shear moduli of the specimens at 100 Hz are as follows: 4.1 GPa for indium, 5.7 GPa for lead, 15.7 GPa for tin, and 20.7 GPa for cadmium. Considering the behavior typical of metals, one may think of In and Pb as relatively compliant, while Sn and Cd could be called moderately stiff. Damping spectra are given in Figs. 1 and 2.



Fig. 1. Loss tangent vs frequency, for indium (G = 4.1 GPa at 100 Hz) and lead (G = 5.7 GPa).

Figure 1 clearly shows the presence of a low frequency background in indium. The presence of a peak, possibly the grain-boundary peak, is indicated near 0.1 Hz. The peak is partially obscured by the low-frequency background. The scatter in the very low frequency data renders subjective any attempt to separate the background from the peak. Indium exhibits a large loss tangent at very low frequencies, but the loss is much less in the audio range; moreover it also has the smallest shear modulus of the metals examined. We remark that indium wire tested in a previous study (3) showed larger loss tangent than the cast indium tested in the present study. Given that the low-frequency background is diffusion-controlled, one possible explanation for this observation is that the wire, being a wrought structure, contains more grain and subgrain boundary area, and thus more sources and sinks for vacancies, than the cast material. A broad peak is present in the loss for Pb. The shape of the peak suggests a broad grain-boundary peak, but the peak frequency is probably too high for that interpretation to be correct. Annealing in air may have affected results. If the specimen oxidized internally during annealing, the peak could correspond to the solid-solution grain-boundary peak which has been reported for other impure FCC metals (8). Although Pb is sometimes thought of as a high-damping material, we could find no literature references to substantiate this notion and we did not observe it ourselves. A damping peak, tan

=0.015 was observed in lead in bending by Kamel (16) near 100 Hz; it was attributed to thermoelastic effect which would be absent in the present torsional experiments. Both the stiffness and loss tangent of lead are low compared with values for other available metals examined here.



Fig. 2. Loss tangent vs frequency, for cadmium (G = 20.7 GPa) and tin (G = 15.7 GPa).

Figure 2 shows the presence of a peak near 1 Hz in Sn. This peak is probably the grain-boundary peak, since it is very near the grain-boundary peak frequency reported by Pearson and Rotheram (17). The damping curve for Cd contains two regions obeying power-laws with respect to frequency.

The results are of some technological interest in view of the utility of materials with moderately high stiffness and damping. The combination of moderate stiffness and reasonably high loss tangent makes Cd the most promising metal tested with respect to technological applications. The shear modulus of Cd was highest of the metals tested (and very near that of aluminum (G = 27 GPa), which exhibits a loss tangent of about 0.001 at room temperature) The loss tangent of Cd at audio-frequencies was as high or higher than that of the other metals. In addition, frequency dependence of loss tangent was not as large as that observed in the other metals. No clear pattern relating damping to melting point emerged. An understanding in terms of viscoelastic mechanisms is not forthcoming at this time.

Conclusions

Among the metals studied, cadmium exhibited a substantial loss tangent of 0.03 to 0.04 over much of the audio range, combined with a moderate stiffness, G = 20.7 GPa.

Acknowledgments

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References

1. B. J. Lazan, *Damping of Materials and Members in Structural Mechanics*, Pergamon Press, New York, 37, (1968).

2. R. Plunkett, "Damping Analysis: An Historical Perspective," in *M*³*D*: *Mechanics and Mechanisms of Material Damping, ASTM STP 1169*, V. K. Kinra and A. Wolfenden, Eds., American Society for Testing and Materials, Philadelphia, 562-569 (1992).

3. C. P. Chen, and R. S. Lakes, "Viscoelastic Behaviour of Composite Materials with Conventional- or Negative-Poisson's-Ratio Foam as One Phase," *Journal of Materials Science*, 28 4288-4298 (1993).

4. C. P. Chen, and R. S. Lakes, "Analysis of High-Loss Viscoelastic Composites," *Journal of Materials Science*, 28, 4299-4304 (1993).

5. T. S. Kê, "Internal Friction of Metals at Very High Temperatures," *Journal of Applied Physics*, 21 414-419 (1950).

6. D. H. Niblett and J. Wilks, "Dislocation Damping in Metals," Advances in Physics, 9, 63-69 (1960).

7. W. P. Mason, *Physical Acoustics and the Properties of Solids*, Van Nostrand-Reinhold, Princeton, 272-285 (1958).

8. A. S. Nowick, and B. S. Berry, *Anelastic Relaxation in Crystalline Solids*, Academic Press, New York, 435-462 (1972).

9. G. Schoeck, E. Bisogni, and J. Shyne, "The Activation Energy of High Temperature Internal Friction," *Acta Metallurgica*, 12, 1466-1468 (1964).

10. J. Woirgard, Y. Sarrazin, and H. Chaumet, "Apparatus for the Measurement of Internal Friction as a Function of Frequency Between 10⁻⁵ and 10 Hz," *Review of Scientific Instruments*, 48, 1322-1325 (1977).

11. J. Woirgard and J. de Fouquet, "High Temperature Internal Friction Measured as a Function of Frequency Between 10⁻⁵ Hz and 10 Hz on High Purity Metals," *Proceedings of the Sixth International Conference on Internal Friction and Ultrasonic Attenuation in Solids*, University of Tokyo Press, Tokyo, 743-747 (1977).

12. C. P. Chen, and R. S. Lakes, "Apparatus for Determining the Viscoelastic Properties of Materials Over Ten Decades of Frequency and Time," *Journal of Rheology*, 33, 1231-1249 (1989).

13. M. Brodt, and R. S. Lakes, "Refinements to an Apparatus Capable of Measuring Viscoelastic Spectra over Ten Decades of Time and Frequency," in preparation.

14. A. Granato, and K. Lücke, "Theory of Mechanical Damping Due to Dislocations," *Journal of Applied Physics*, 27, 583-593 (1956).

15. A. Granato, K. Lücke, "Application of Dislocation Theory to Internal Friction Phenomena at High Frequencies," *Journal of Applied Physics*, 27, 789-805 (1956).

16. R. Kamel, "Measurement of the Internal Friction of Solids", Physical Review, 75, 1606 (1949).

17. S. Pearson and L. Rotherham, Trans. AIME 206 (1956) 661, cited in A. S. Nowick and B. S. Berry (8).