VISCOELASTIC SPECTRA OF $Cd_{0.67}Mg_{0.33}$ IN TORSION AND BENDING

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Abstract

In both torsion and in bending, the alloy exhibited a viscoelastic relaxation which could be modeled as a Debye peak superimposed on a power-law low-frequency background. In torsion, the relaxed and unrelaxed shear moduli were 9 and 12.2 GPa, respectively; maximum loss tangent was 0.12. In bending, relaxed and unrelaxed Young's moduli were 15 and 35 GPa, respectively; maximum loss tangent was 0.11. Behavior was linear to at least 125 microstrain. These results are significant in that they represent a unique combination of stiffness and loss in a monolithic material.

Introduction

Mechanical damping is an important consideration in the design and analysis of dynamically-loaded structures. In the past, structures have been designed on the basis of strength and stiffness while damping was dealt with as an afterthought. For the aerospace structures of the future, however, the potential payoff for high-damping structural materials is great: decreased complexity of control systems, increased reliability, and decreased cost [1]. Hence, materials with high stiffness and high damping are desirable.

It is not anticipated that a monolithic material be found with both high stiffness and high viscoelastic loss. Thermodynamic considerations suggest that the simultaneous storage (high stiffness) and dissipation (high damping) of strain energy is unlikely. One alternative is composite materials. Chen and Lakes [2,3] have shown that composite materials with certain mesostructures can yield high stiffness and high loss. In particular, metal matrix composites are already under consideration for use in aerospace structures [1]. Candidate materials for the high stiffness-low damping phase of such a metal-matrix composite exist in abundance, whereas candidate materials for the moderate stiffness-high damping phase remain to be identified.

In connection with the search for a moderate-stiffness-high damping phase, a review of the literature revealed studies on the Cd-Mg system conducted by Lulay and Wert [4] and Enrietto and Wert [5] in the late 1950's. References 4 and 5 report the results of damping tests on Cd-Mg alloys ranging in composition from 0 to 50 at-% Mg. Tests were performed in a torsion pendulum operating near 1 Hz and at temperatures from about -50 to 70°C. A damping peak as a function of temperature was observed near room temperature for most alloy compositions tested. Peak magnitude displayed two maxima and one minimum as a function of alloy composition. The location of the minimum near the stoichiometric composition Cd_3Mg led the authors to attribute the observed damping to energy dissipated in stress-induced ordering.

In order to evaluate the alloys' potential for incorporation as constituents of high stiffness-high loss composites, stiffness and damping as functions of frequency must be assessed at a temperature near the anticipated service temperature. Techniques for the mutual interconversion of anelastic properties expressed as functions of temperature and of frequency are useful for this purpose, but rely upon assumptions that cannot always be justified [6]. Furthermore, stiffness values for these alloys were not reported in Refs. 4 and 5. The present study reports the results of stiffness and damping tests conducted on the alloy $Cd_{0.67}Mg_{0.33}$, the composition giving the largest damping peak according to Refs. 4 and 5. Tests were performed using a modified version [7] of the apparatus of Chen and Lakes [8] at 23°C in the frequency range of 10^{-3} to 10^4 Hz.

Materials and Methods

Specimen Preparation

Although Refs. 4 and 5 report the melting of the Cd-Mg alloy ingots under vacuum, we were unsuccessful in that regard. Early attempts to melt a powder charge in a small evacuated quartz tube 3.1 mm in diameter resulted in explosions sufficiently energetic to pulverize the specimen tube. A subsequent review of the literature revealed that the vapor pressure of Cd at the melting point of Mg is such that sublimation occurs readily at vacuum levels achievable with a mechanical pump; that pressure was considered a possible cause of the explosions. Plans to melt under vacuum were abandoned.

An argon port was fitted to our muffle furnace, but the volume of the furnace could not be filled economically with argon, and the amount of argon that could be injected was insufficient to suppress alloy oxidation at elevated temperatures. A quartz environment tube was obtained and a "tee" manifold was fabricated from Tygon tubing such that one end could be fitted to the open end of the environment tube, one end could be fitted to a mechanical vacuum pump, and one end fitted to an argon tank.

Subsequent attempts at melting were still unsuccessful, however, and it was determined that pre-existing oxide layers on the constituent powders were interfering with the melting process. In an effort to reduce the ratio of surface area to mass, rods of the constituent materials were obtained. While an improvement in that some melting was observed, the resulting ingots did not meet expectations. Chemical cleaning of the surface oxides was pursued with good success. Finally, a mechanical cleaning was added to the procedure to remove the residue of the chemical cleaning.

The final fabrication process was as follows: A Cd rod (99.99+% purity) and an Mg rod (99.98% purity) were obtained from Johnson Matthey Alfa. Rectangular pieces of each rod were cut to fit the Al_2O_3 crucible used (quartz had been used initially, but the molten Mg reacted with the quartz to produce Mg_2Si). The Cd piece was cleaned by immersion in a solution of 20% concentrated HNO_3 in methanol (nital) and the Mg piece was cleaned by immersion in a solution of 10-15% concentrated H_2SO_4 in deionized water. Both pieces were subsequently cleaned mechanically with 1000-grit silicon carbide abrasive paper. Both pieces were rinsed in acetone and then methanol.

The pieces were placed into the crucible and the crucible was inserted into the environment tube. The environment tube was lowered into the furnace through its exhaust port and the manifold was connected to the environment tube. The environment tube was evacuated with a mechanical pump for 24 hours and was then backfilled with standard purity argon. The furnace was ramped to $680^{\rm o}C$ and allowed to soak for 20 minutes. Mechanical shaking was described by Refs. 4 and 5 as having "aided" the mixing process; we found it to be essential. The alloy ingot was allowed to furnace cool.

The alloy ingot was cut lengthwise into quarters with a low-speed diamond saw. The quarters were cleaned chemically in nital, cleaned mechanically with 1000-grit silicon carbide abrasive paper, and rinsed in acetone and in methanol. The quarters were melted, using the procedure above, in a Pyrex tube with inner diameter near the desired specimen size (3.1 mm in diameter). After melting, the specimen was soaked at $80^{\rm o}C$ for 24 hours to promote formation of the ordered Cd_3Mg phase. A section was cut from the specimen end for metallographic examination.

Stiffness and Damping Tests

Tests were performed using a modified version [7] of the apparatus of Chen and Lakes [8] (Fig. 1). The present version of the instrument incorporates improved phase resolution, and is not limited to high-loss elastomers. One end of the specimen, 3.1 mm (1/8") in diameter, was glued (with a cyanoacrylate cement) to a tungsten rod, 12.7 mm (1/2 in), fixed to a stiff framework of rods. The other end of the specimen was cemented to a high intensity neodymium iron boron permanent magnet. A sinusoidal voltage from a digital function generator was amplified and applied to a Helmholtz coil which generated a magnetic field which acted upon the permanent magnet to provide either an axial torque or a bending moment, depending upon the orientation of the coil with respect to the magnet. Light from the laser was reflected from a small mirror upon the magnet 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 proportional to the angular displacement. Torque was inferred from the Helmholtz coil current; torque calculations were supported by calibrations using the well-characterized 6061 aluminum alloy.

Frequency was recorded from the function generator. From 10^{-3} to 10^{-2} Hz, the input and output voltages and phase difference between the two signals were taken from a strip chart recorder; from 10^{-2} to 10^1 Hz, damping was obtained directly from the Lissajous figure resulting from a digitally-acquired plot [9] of the output signal against the input signal; and from 10^1 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 relations

$$G = \frac{Tl}{J_t \theta} \tag{1}$$

where G is the shear modulus, T is the applied torque, **l** is the specimen length, J_t is the second moment of area about the neutral axis in torsion, and θ is the angular displacement;

$$E = \frac{Tl}{J_b \theta} \tag{2}$$

where J_b is the second moment of area about the neutral axis in bending. Damping was taken to be approximately equal to the measured phase difference δ:

$$\tan \delta \tag{3}$$

At frequencies approaching the specimen's first torsional resonance, stiffness and damping were calculated by numerical solution of an exact relationship for the torsional rigidity (ratio of torque M* to angular displacement θ) of a viscoelastic cylinder of radius R length **l**, and density ρ with an attached mass of mass moment of inertia I_{at} at one end and fixed at the other end [10]:

$$\frac{M^*}{\theta} = [(1/2) \rho R^4][\Omega^2 l]\frac{\cot \Omega^*}{\Omega^*} - I_{at}\omega^2, \tag{4}$$

where $\Omega^* = (\rho \Omega^2 l^2 / KG^*)^{1/2}$, K is a geometrical constant (equal to 1 for a cylindrical specimen with circular cross section), and $I_{sp} = (1/2)\rho R^4$ is the mass moments of inertia of the specimen if it is a circular cylinder. Stiffness was taken to be the absolute value of the complex quantity G^*:

$$G = |G^*| \tag{5}$$

Damping was then obtained by the ratio of imaginary to real parts of G*:

$$\tan \delta = \frac{Im(G^*)}{Re(G^*)}. \tag{6}$$

A comparable treatment for bending is not presently available. Hence, results for bending are reported only for frequencies such that Eqs. 2 and 3 are valid.

At frequencies corresponding to a resonant torsional or bending mode of the specimen, damping was calculated using the shape of the frequency response curve:

$$\tan \delta \frac{\Delta \omega}{\omega_o}, \tag{7}$$

where $\Delta \omega$ is the difference between the two so-called "half-power frequencies."
The specimen was tested at various frequencies between 10^{-3} and 10^4 Hz. Test temperature was 23 $\pm$ 1$^{\rm o}$C.

Results and Discussion
Raw data were converted to |G|, |E|, and tan δ through use of Eqs. 1-7. The results are illustrated in Figs. 2 and 3. The first torsion resonance was at 3.9 kHz and the corresponding tan δ was 0.0072. Also shown in Figs. 2 and 3 are theoretical curves fitted by a nonlinear least squares routine. The curves are of the form

$$\tan = A \;{}^{-n} + \frac{}{1+ {}^2 {}^2} \tag{8}$$

where A and n are constants, is the relaxation of the modulus, is the relaxation time and is angular frequency.

 The first term in Eq. 8 corresponds to the "low frequency background" damping predicted theoretically by Schoeck, Bisogni and Shyne [11] and observed experimentally by Woirgard and de Fouquet [12] and by Cook, Chen and Lakes [13]. In torsion, the fitted values were A = 0.019 and n = 0.106. In bending, the fitted values were A = 0.015 and n = 0.090. The present state of knowledge of the low frequency background is not such that A and n can be interpreted in terms of material parameters, but those obtained in the present study seem to be reasonable in comparison to those published in Refs. 12 and 13.

 The second term in Eq. 8 is easily recognizable as the Debye relaxation equation. In torsion, the fitted values were $_G$ = 0.21 and = 0.22 s. In bending, the fitted values were $_E$ = 0.20 and = 0.20 s. The similarity between fitted values of $_G$ and $_E$ is indicative of isotropy. Given the polycrystalline nature of the specimen, it is probably more indicative of specimen isotropy than relaxational isotropy, although the latter is still certainly possible. Given the agreement of relaxation strengths, the similarity between values of obtained in torsion and bending is not surprising. It may be concluded that the mechanism operates over time periods of tenths of seconds.

 The shear modulus displays behavior typical of a viscoelastic relaxation. At high frequencies, the shear modulus has an unrelaxed value G_U near 12.3 GPa. At low frequencies, the shear modulus tends toward a relaxed value G_R near 8.8 GPa. However, the relaxation is not complete at 10^{-3} Hz because of contributions from the low-frequency background. At intermediate frequencies, the shear modulus takes on intermediate values.

 Young's modulus also displays behavior typical of a viscoelastic relaxation. At high frequencies, Young's modulus has an unrelaxed value E_U near 35 GPa. At low frequencies, Young's modulus tends toward a relaxed value E_R near 28 GPa. As in the case of torsion, contributions from the low-frequency background keep this from being a true asymptotic value, however.

 The present results may be compared with those of Enrietto and Wert [5] who reported the damping peak vs composition. The peak tan was about 0.145 at 1 Hz at 'room' temperature in torsion for 33 atomic % magnesium. Lulay and Wert [4] found a peak in tan at 0.75 Hz vs temperature of 0.13 near 18°C for an alloy of 29.3% Mg and inferred a Debye peak by assuming a single thermally activated mechanism. The present torsional results, tan at 23°C, vs frequency, showed a Debye peak, tan = 0.12 at 0.6 Hz. This is considered satisfactory agreement and supports the assumption of Lulay and Wert of a single thermally activated relaxation process.

 Metallographic examination of the specimen cross-section via optical microscopy revealed only five grains across the cross section (Fig. 4). Longitudinal sections were also examined. Micrographic study of longitudinal sections disclosed equiaxed grains with no obvious orientation effects. Specimens had a shiny metallic luster when first prepared. They became visibly tarnished after several weeks' exposure to room air. Re-testing of the tarnished specimens disclosed no obvious change in the damping. Any oxygen impurity would give rise to a Snoek relaxation, well out of the frequency range considered. The specimens became grossly corroded after six months in humid air. Future investigators of this alloy will be well advised to protect it from corrosion.

 Since results for both shear and Young's moduli were obtained, it is of interest to consider the Poisson's ratio computed under the assumption of isotropy. The calculated Poisson's ratio from 1 to 100 Hz is about 0.42, and its phase angle is between -0.002 and -0.03 rad with considerable scatter. The calculated bulk modulus proved to be too sensitive to small uncertainty in the input data for any meaningful inference to be made. A major caveat here is whether a specimen with so few grains across the cross section can be regarded as quasi-isotropic, even approximately. Even so, inference of a Poisson's ratio within the isotropic range from -1 to 1/2 and of a 'normal' magnitude,

suggests an un-oriented specimen. Further study involving fine grained specimens would be of interest.

In terms of the goals of the study, results are satisfactory. This statement may be better appreciated through use of a stiffness-loss map. The stiffness-loss map is a useful way to compare the stiffness and damping properties of various materials. Stiffness is plotted as the ordinate and loss tangent is plotted as the abscissa. High stiffness-high damping materials will thus appear in the upper right-hand corner of the plot. Figure 5 is a stiffness-loss map wherein Cd-Mg is compared to various representative engineering materials. The loss in the common metals shown at low frequency is mostly due to thermo-elastic damping in flexure. In torsion or at higher frequency, the loss tangent of common metals can be considerably lower. Indeed, type 6061 aluminum alloy exhibits $\tan = 3.6 \times 10^{-6}$ in torsion at room temperature [20]. It may thus be appreciated that the combination of stiffness and high damping in the Cd-Mg alloy studied is unusual.
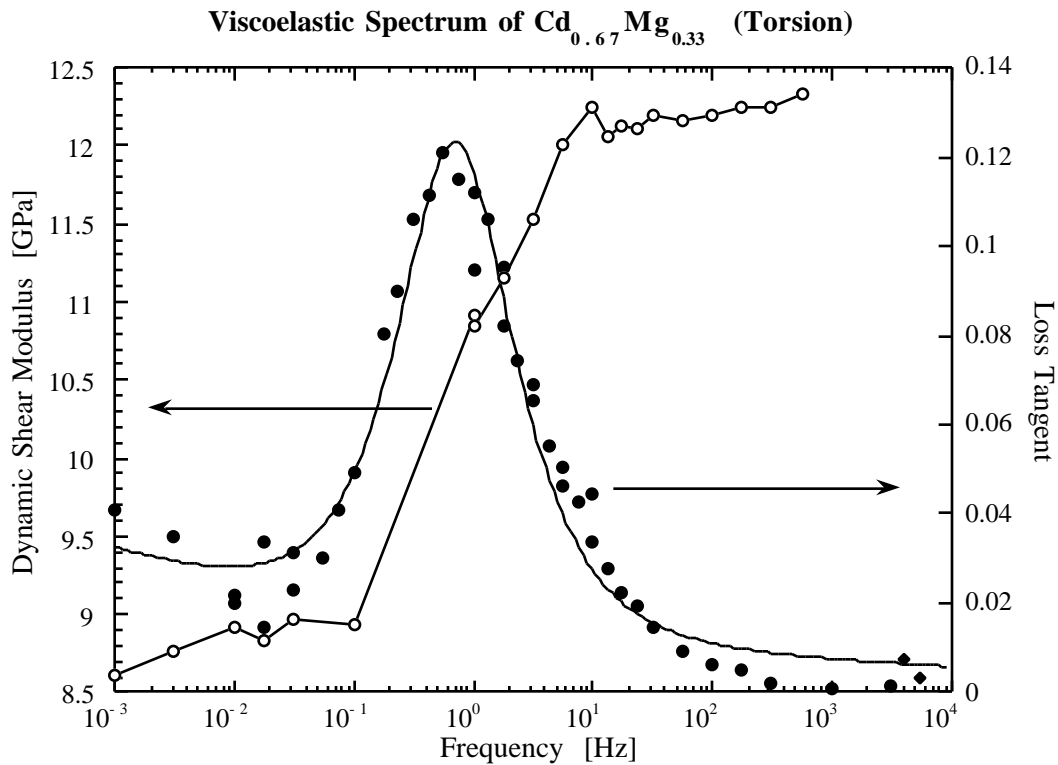
Conclusion

The Cd-Mg alloy exhibits strong viscoelastic damping combined with moderate stiffness. The behavior in torsion and bending is describable by a Debye peak superimposed upon a power law low frequency background.
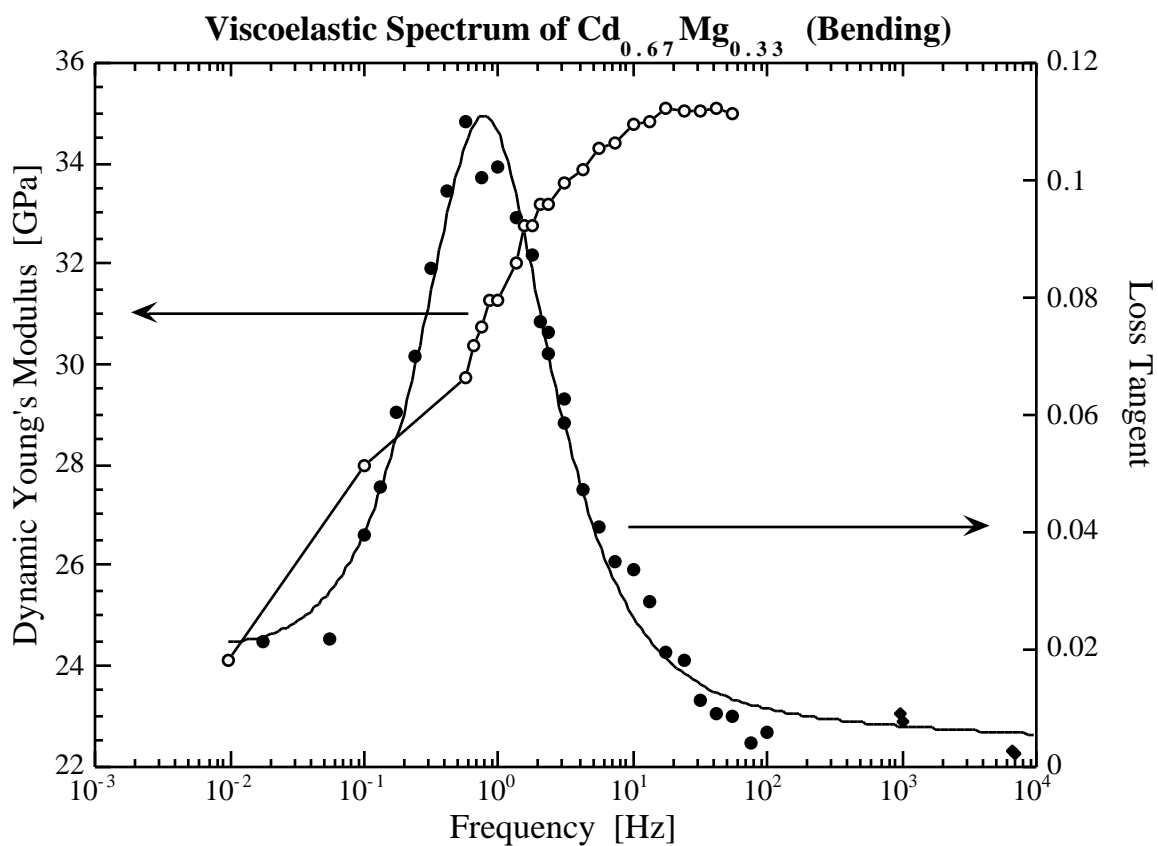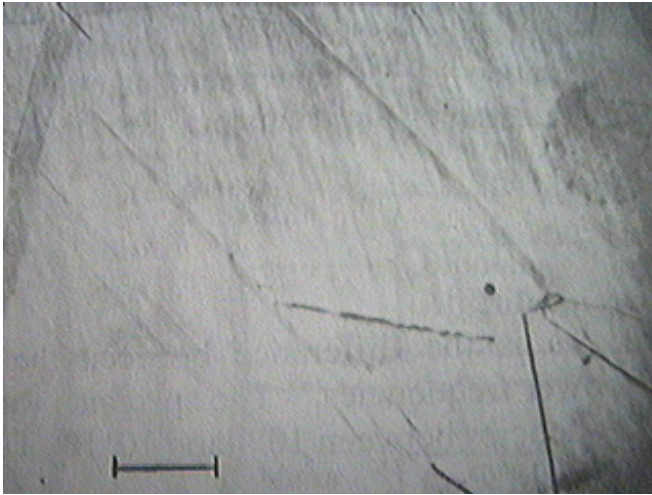
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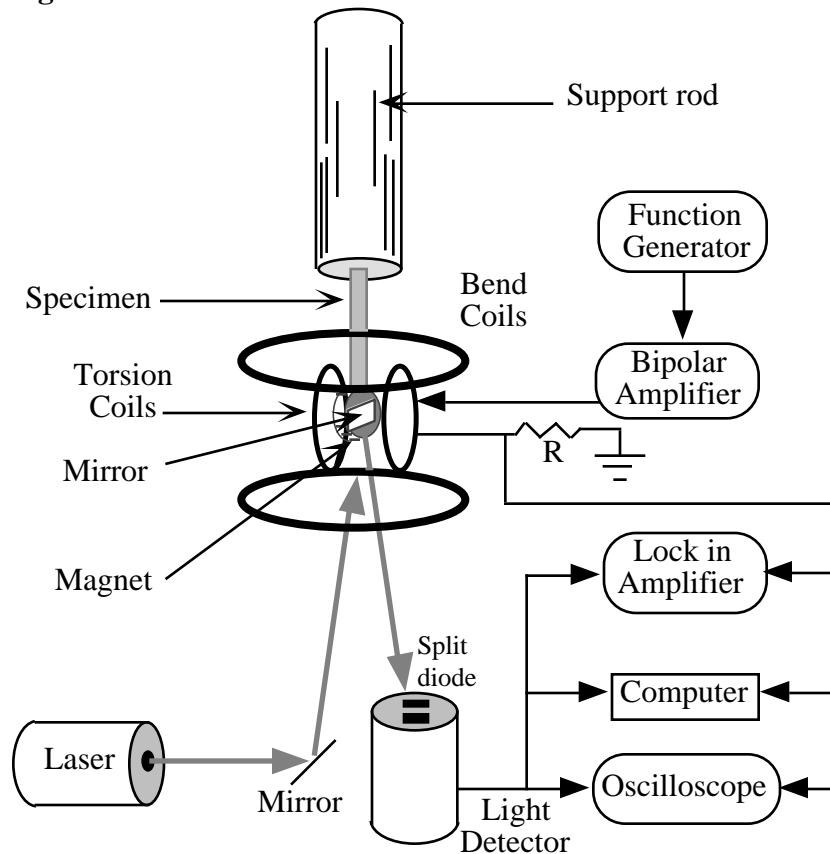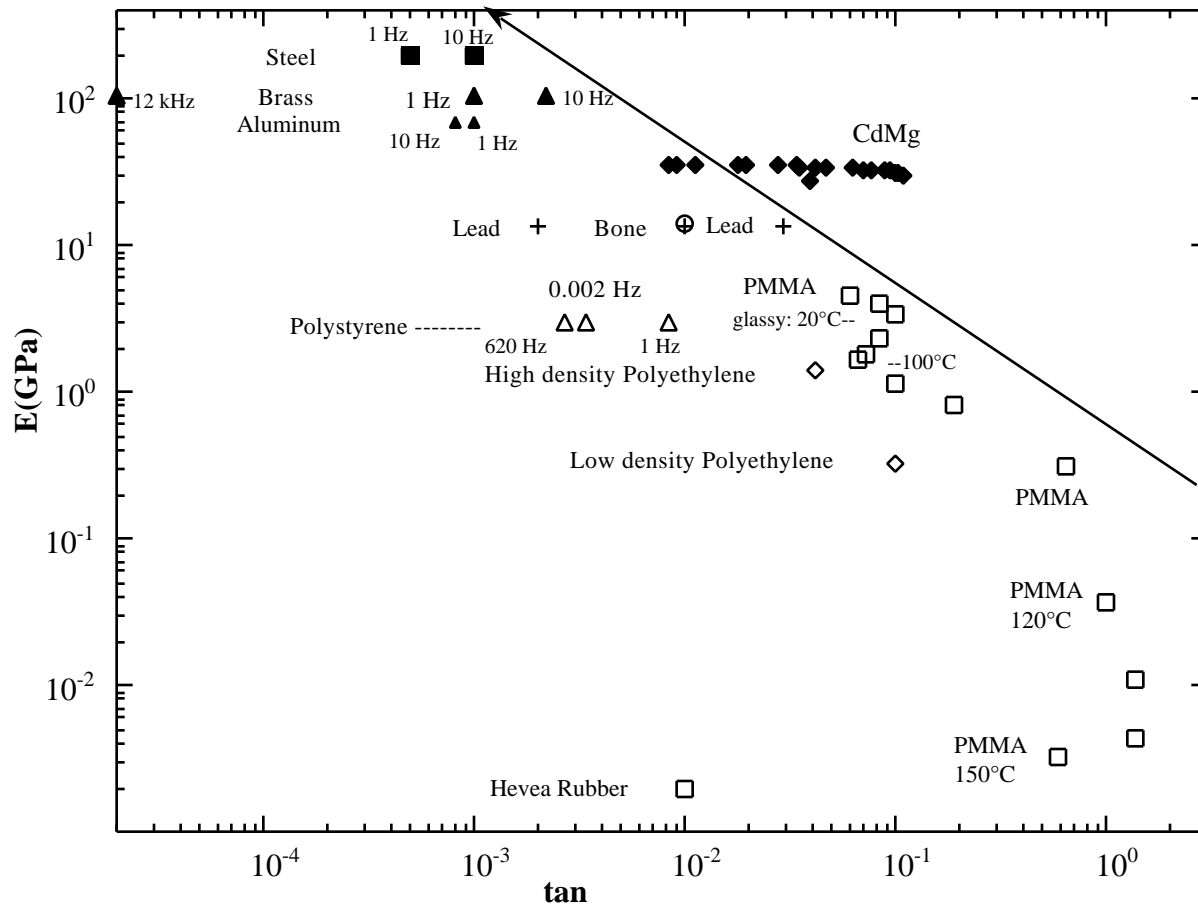

1   Experimental configuration.

2   Viscoelastic response of Cd-Mg alloy: torsion.

3   Viscoelastic response of Cd-Mg alloy: bending.

4 Cross sectional optical micrograph of specimen. Scale mark: 0.2 mm.

5 Stiffness-loss map showing a comparison of Cd-Mg alloy and common materials.
  Data are adapted from the following sources. PMMA, 1 Hz, various temperatures, [14].
Lead, 1-15 kHz, [15]; bone, 1-100 Hz [16]; single crystal lead, 64 kHz, [17]; Hevea rubber, [18, p.50], polystyrene, 0.001 Hz to 1 kHz [18, p.468]; steel, flexure, [19]; aluminum, flexure, [19]; brass, flexure, [19].

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