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Discovery of an Internal-Friction Peak in the Metallic Glass Nb_3Ge

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A well-defined internal-friction peak has been observed near 260 K in amorphous rf-sputtered films of Nb_3Ge, studied at audio frequencies by a vibrating-reed technique. The characteristics of the peak are consistent with a stress-induced ordering mechanism involving a presently unidentified center which undergoes reorientation by an atomic jump with a sharply defined activation energy of 0.52 eV. The peak appears to be the first example of its type found in a metallic glass.

Metallic glasses are presently under intensive investigation from many points of view, and the eventual use of selected alloys in some applications now seems highly probable. However, much remains to be learned about the detailed structure of these metastable amorphous alloys, and particularly about the mechanism and kinetics of the internal atomic movements that ultimately control their stability. As is well known from much work on crystalline materials, internal-friction peaks associated with the stress-induced directional ordering of point defects or local structural groupings can provide valuable information in both of these areas.[1] The purpose of this Letter is to describe results on amorphous films of Nb_3Ge which are thought to provide the first known example of such a peak in a metallic glass.

The measurements were performed on unsupported thin films by a vibrating-reed technique,[2] following the observation that amorphous films of Nb_3Ge are mechanically much superior to their crystalline counterparts.[3] Amorphous films of about 6 μm thickness have been detached from their substrates and found to be strong, flexible, and easily handled, in marked contrast to the extreme brittleness and fragility produced by a crystallization anneal. Although amorphous Nb_3Ge is a superconductor, the transition temperature is low ($T_c \simeq 3.6$ K) compared with the record T_c's of $\simeq 23$ K reported for films prepared directly with the A-15 crystal structure.[4,5] Nevertheless, the amorphous alloy has technological interest as an easily handled source from which the brittle high-T_c phase may be obtained by a final *in situ* anneal.[3]

Films of nominally stoichiometric Nb_3Ge were prepared in an amorphous condition by rf sputtering in krypton onto liquid-nitrogen–cooled substrates. The absence of crystallinity was verified by x-ray diffraction. The internal-friction measurements were performed on strips approximately 15 mm×1.5 mm, cut from detached films 6–8 μm thick. Test samples were selected on the basis of an acceptable flatness (as-prepared reeds tend to be curled either longitudinally or laterally), and on their freedom from edge cracks or other flaws. The curling of samples during preparation or subsequent annealing is a source of experimental difficulty not only from the viewpoint of avoiding contact with the electrodes of the internal-friction apparatus, but also because distortions from the simple reed geometry can markedly upset the higher modes of vibration and the ability to obtain data at a set of different frequencies. In the best case to date, measurements were possible at the first four tones of the reed, corresponding to a frequency variation in excess of a factor of 30. These results, shown in Fig. 1, illustrate a number of features which have been consistently observed with a number of different samples. Over the temperature range investigated (100–800 K), the internal-friction behavior of the amorphous Nb_3Ge films is dominated by a single well-resolved peak located somewhat below room temperature (~260 K for a reference frequency of 100 Hz). The peak heights observed with different tones often show erratic differences, sometimes of sizable magnitude (e.g., 2:1). Because a different stress distribution is associated with each tone, this observation implies that the films are prone to macroscopic inhomogeneities in the distribution of relaxation centers. However, it is not known whether the inhomogeneity is present in the form of random patches, or as a more uniform gradation over the whole film.

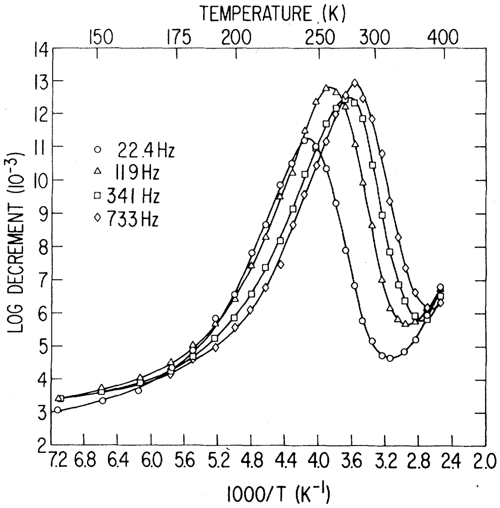

FIG. 1. The internal-friction peak exhibited by a sputtered film of amorphous Nb_3Ge, as observed at the first four tones of a vibrating reed. The reed had been exposed to 120°C for about 10 min prior to the measurements.

In contrast to the variability observed in the peak height, the location and shape of the peak have been found to be essentially constant. This implies that the kinetic parameters involved in the relaxation are also constant. The thermally activated nature of the relaxation is brought out in Fig. 1 by the shift of the peak to higher temperatures as the measurement frequency f is increased. Figure 2 shows the Arrhenius plot obtained for the mean relaxation time $\bar{\tau}$, which is determined by the condition $2\pi f\bar{\tau}=1$ at the peak temperature, T_p. The straight line corresponds to the relationship

$$\bar{\tau}=\bar{\tau}_0 \exp(\bar{Q}/kT), \tag{1}$$

where k is Boltzmann's constant and T is the absolute temperature, and yields the values 2×10^{-13} sec for $\bar{\tau}_0$ and 0.52 eV for the activation energy $\bar{Q}$. These values have been used in Fig. 3 to cal-

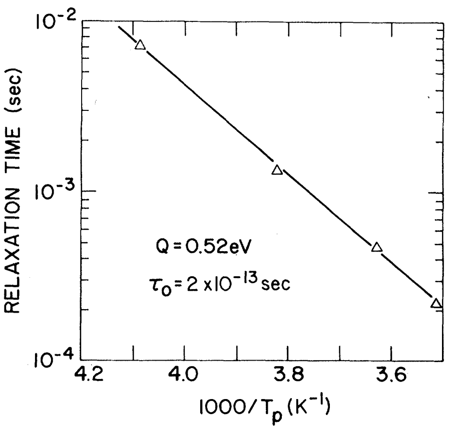

FIG. 2. Arrhenius plot for the 260-K peak in amorphous Nb_3Ge.

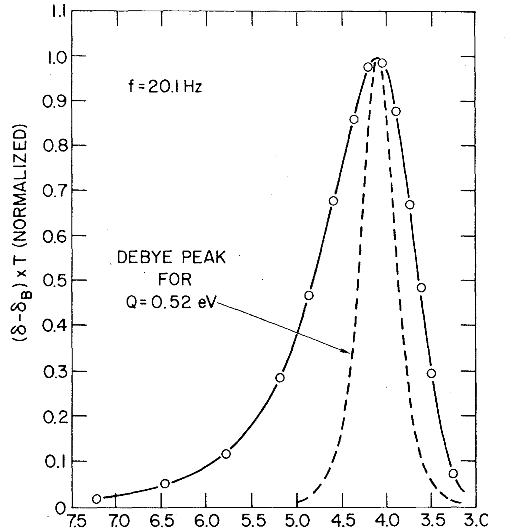

FIG. 3. Comparison of the experimental peak with the equivalent Debye peak. The data were obtained in the fundamental mode prior to heating the sample above room temperature.

culate the equivalent Debye peak for the case of a single relaxation time, for comparison with the experimental peak obtained after removal of the background and application of a temperature correction to compensate for the usual $1/T$ dependence of the relaxation strength. The experimental peak has a width at half-maximum that is 2.8 times broader than the Debye peak, and therefore is clearly seen to involve a distribution of relaxation times. An interesting feature of the dispersion is reflected in the noticeable asymmetry of the peak, which is skewed in a manner that corresponds to a heavier weighting of the short relaxation times (low activation energies) in the distribution.

The results of annealing experiments, conducted in vacuum for periods of up to an hour at temperatures up to 550°C, are given in Fig. 4. Annealing produces a marked decrease and eventual elimination of the peak, without a significant change in either the shape or location of the peak. The form of Fig. 4 suggests that annealing occurs in two separate stages. The first and larger stage appears almost immediately after the films are heated above room temperature. The second stage, which appears to be relatively sharp, is centered near 500°C. It should be noted that annealing is complete well before the film crystallizes, which occurs on heating above 700°C.

The present findings may be briefly discussed

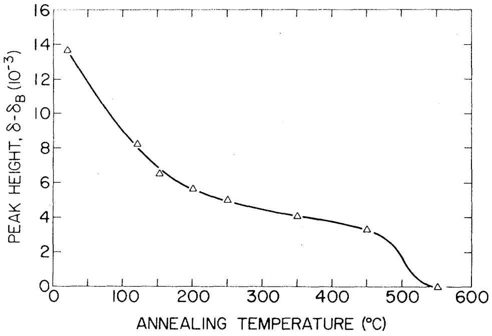

FIG. 4. Annealing behavior of the 260-K peak. The peak heights shown are corrected for the background damping, δ_B.

as follows. Amorphous sputtered films of Nb_3Ge have been found to exhibit a thermally activated relaxation with a surprisingly narrow distribution of relaxation times and a relatively unique activation energy located near 0.52 eV. The magnitude of $\bar{\tau}_0$ is of typical magnitude for an atomic jump, and it seems clear that the peak represents an example of stress-induced ordering of a basically similar nature to the point-defect relaxations known in crystalline solids. To account for the sharpness of the peak it appears that, despite the presence of both local and long-range disorder, the amorphous structure must contain structural units or groupings (relaxation centers) which, apart from differences in orientational configuration, exhibit a marked degree of similarity. To produce the peak, these centers must possess an axial character which makes them susceptible to a thermally activated reorientation to a stress-preferred direction. While the annealing behavior is not yet understood, it is clear at least that the annealing kinetics, particularly for the second stage, must be controlled by a process with a much higher activation energy than the 0.52-eV reorientation jump. This suggests that the relaxation is caused by the localized motion of a bound or trapped atom contained in a rather stable grouping which is itself relatively immobile. However, further experiments, presently in progress and to be reported elsewhere,[6] are needed to help identify the nature and number of these groupings, and the identity of the atom making the 0.52-eV jump. It is of particular interest to establish whether the relaxation center is an intrinsic grouping containing only Nb and Ge atoms, or whether it contains an impurity incorporated into the film during sputtering. It is possibly significant that none of the liquid-quenched amorphous alloys previously studied has shown a peak of the type now seen for the first time in a sputtered film. If the peak is impurity related, it remains to be seen whether there is an impurity effect on the superconducting behavior of crystallized films, since the annealing behavior may indicate that rejection of the impurity by precipitation or desorption is complete prior to crystallization.

Finally, and from a more general viewpoint, it can be remarked that the principal significance of the present work lies in the conclusion that a well-defined internal-friction peak associated with a stress-induced ordering mechanism has been identified for the first time in a metallic glass. If the history of internal-friction studies on crystalline materials is used as a guide, it now appears likely that other peaks await discovery in a variety of metallic glasses, and that these will be of considerable use in the characterization of these interesting new materials at an atomic level.

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Resonant Nonlinear Mode Conversion in He II

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The results of an experiment to observe the nonlinear conversion of second sound to first sound within a waveguide show that this resonant process occurs at precisely the predicted frequency. Unique procedures are used to calibrate the first-sound transducers and we find the amplitude of the mode-converted first sound has its predicted value value which is determined principally by $\partial\rho/\partial(w^2)$ where w is the difference between the normal-fluid and superfluid velocities and ρ is the density.

The purpose of this Letter is to describe an experiment which results in the first observation of a propagating first-sound (pressure-density) wave generated by the nonlinear interaction of two second-sound (temperature-entropy) waves. This resonant mode-conversion process is in some ways similar to the generation of a longitudinal sound wave through the interaction of two transverse waves in a solid, in that the difference in the propagation velocities of the two modes places severe restrictions on the frequencies and wave vectors of the interacting waves.[1] This has been observed in solids[2] but no absolute measurements were made which could be used to determine coupling coefficients. In our experiment, a new application of the reciprocity principle[3] allows absolute measurements of the primary and mode-converted wave amplitudes and thus a direct quantitative measurement of the coupling between the mode unique to superfluid (second sound) and the ordinary sound wave (first sound).

In the nondissipative approximation, the general hydrodynamic description of He II is given by the Landau two-fluid equations.[4] When wavelike solutions to the two-fluid equations are sought, retaining terms which are second order, the wave equations for the second-order pressure or temperature variations in the superfluid are not homogeneous but have driving terms which are proportional to quadratic combinations of the first-order quantities. The following is the wave equation for second-order pressure disturbances, p_2, neglecting the isobaric coefficient of thermal expansion which is small in the region of experimental interest and vanishes at 1.17°K:

$$\frac{\partial^2 p_2}{\partial t^2} - u_1{}^2\nabla^2 p_2 = u_1{}^2\left[\frac{\partial^2}{\partial r_i\,\partial r_j}\left(\rho v_i v_j + \frac{\rho_n\rho_s}{\rho} w_i w_j\right) - \frac{1}{2}\frac{\partial^2\rho}{\partial p^2}\frac{\partial^2 p_1{}^2}{\partial t^2} - \frac{1}{2}\frac{\partial^2\rho}{\partial T^2}\frac{\partial^2 T_1{}^2}{\partial t^2} - \frac{\partial^2\rho}{\partial p\,\partial T}\frac{\partial^2 p_1 T_1}{\partial t^2} - \frac{\partial\rho}{\partial(w^2)}\frac{\partial^2(w^2)}{\partial t^2}\right], \tag{1}$$