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The influence of hydrogen and dislocations on the internal friction in yttrium

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Abstract

The internal friction spectra of (i) plastically deformed Y, (ii) undeformed H-doped $YH_{0.1}$ and (iii) plastically deformed and H-doped $YH_{0.1}$ samples were measured with the help of the vibrating reed technique (polycrystalline foils, 5 K$\leqslant T \leqslant$320 K, frequencies f between 100 and 1300 Hz, plastic deformations between 5% and 25% due to rolling at room temperature). The deformed (undoped) Y samples exhibit an internal friction peak in the same temperature range as the undeformed doped $YH_{0.1}$ samples. We found further that combined plastic deformation and H-doping leads to a stronger peak than does sole deformation or H doping. The possible mechanisms for the observed relaxation process are discussed.

1. Introduction

The behavior of H and D in Y has been studied by several investigators with the help of internal friction measurements [1–3]. The results of these experiments show an isotope-dependent relaxation peak located, *e.g.* for $f=500$ Hz, at 272 and 279 K in the case of H and D, respectively. The relaxation process is thermally activated and can be described by an Arrhenius relation with a relaxation rate $\tau^{-1}=\tau_0^{-1}\exp(-E/k_BT)$, where the activation energy E is (0.60 ± 0.05) eV for both H and D. The relaxation strength of the internal friction peak increases nearly linear with the H(D) content. This fact and the isotope dependence of the peak temperature suggests that the appearance of the peak is directly correlated with diffusive H(D) jumps [1–4].

For symmetry reasons, diffusive jumps of isolated H(D) atoms between either tetrahedral (T) or octahedral (O) sites are not expected to yield an anelastic relaxation [5–7]. For this reason, the following two models were proposed for the microscopic mechanism of the observed relaxation process [1, 3, 4]: (i) a stress-induced formation or dissolution of H(D) pairs and (ii) a relaxation due to a stress-induced change of the H(D) distribution between T and O sites.

An important requirement for a meaningful analysis of the internal friction caused by H or D in Y is that no additional relaxation process exists in the same temperature range. Such an additional relaxation process may result from dislocations. Accordingly, we report in this paper measurements investigating the influence of dislocations on the internal friction spectra of Y. The measurements were carried out (i) on plastically deformed Y samples (not doped with H), (ii) on an H-doped and plastically deformed Y sample and, for comparison, (iii) on an H-doped undeformed Y sample. The main result of our study is that plastic deformation causes an internal friction peak in Y which is similar to the H(D) peak in undeformed samples.

2. Experimental details

The samples (~50 mm long, 5 mm wide) were prepared from 0.1 mm thick polycrystalline Y foils with a typical impurity content of 0.2 at.% (O, N, ...). The measurements were carried out on four samples. Sample 1 was doped with H ($YH_{0.1}$, no plastic deformation); sample 2 was doped with H and subsequently plastically deformed by 5%–10%, sample 3 was an undoped sample which was deformed by 5%–10%; and sample 4 was also an undoped sample that was deformed by 20%–25%.

We found in preparatory experiments that vacuum annealing of Y samples at temperatures of 1100 °C reduces the dislocation density far enough to eliminate internal friction peaks that were caused by plastic deformation. For this reason, all our samples were

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subjected to annealing at 1100 °C (4 h) either in a vacuum (undoped samples) or in an H_2-atmosphere (H-doped samples, H_2-gas pressure $p \leqslant 0.2$ mbar), in order to achieve a low initial dislocation level which was closely identical for all our samples. The H concentration x of the doped YH_x samples (samples 1 and 2) was determined from the amount of absorbed H_2 gas and from the weight increase due to H absorption. Plastic deformation of samples 2, 3 and 4 was performed after the above annealing procedure, with the help of room-temperature rolling of the samples in a direction perpendicular to their length. The relative increase of the sample width was taken as a measure of the resulting deformation.

The internal friction measurements were carried out with a computer-controlled vibrating-reed apparatus with FM detection and electrostatic drive. The samples were kept in a vacuum chamber in order to minimize background damping. The clamp, which fixed the sample, could be mounted at different positions along the sample so that it was possible to adjust different vibrational frequencies (100–1300 Hz) for a given sample.

3. Experimental results and discussion

Figure 1 shows a comparison of the internal friction peaks of (i) the hydrogen-doped sample 1 (no deformation, H-peak), (ii) the 5%–10% deformed sample 3 (no H-doping, d-peak) and (iii) the hydrogen-doped and 5%–10% plastically deformed sample 2 (dH-peak). All three internal friction peaks are measured as a function of temperature at nearly the same frequencies (109–126 Hz). The most surprising result of the data is that the temperatures (257–259 K) of the main internal friction peak (H-peak, d-peak and dH-peak for samples 1, 3 and 2, respectively) are found to be identical for all three samples within the limits of experimental accuracy. If we account for linear background damping, we can see that the peak heights of the d-peak and the dH-peak exceed that of the H-peak by a factor of 1.5 and 1.8, respectively. The deformed samples 2 and 3 show two additional smaller peaks, centered at ~130 and ~210 K, where the 210 K peak appears solely as a shoulder on the low temperature side of the main d-peak. These two additional peaks exist, with different intensity, in all deformed samples.

Figures 2, 3 and 4 present internal friction spectra, taken at different frequencies, for H-doped and additionally 10%–15% deformed sample 2, for 10%–15% deformed (undoped) sample 3 and for 20%–25% deformed (undoped) sample 4, respectively. For the spectra shown in Figs. 2 and 3, the main relaxation peak (temperature range 260–290 K) is clearly identifiable. For the 20%–25% deformed sample 4, on the other hand, the shoulder at the low temperature side of the main peak (d-peak) has practically the same height as the main peak itself. Accordingly, the shoulder and the

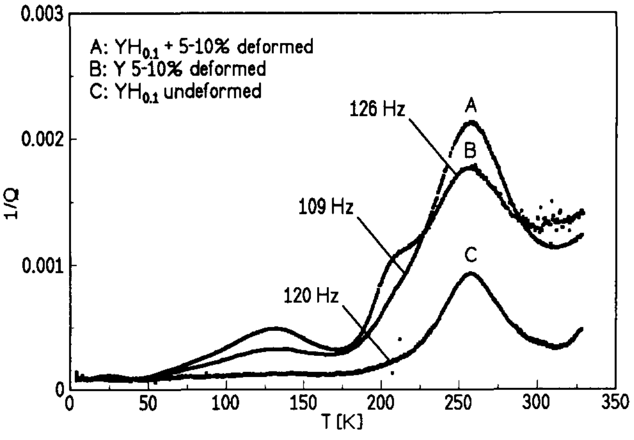

Fig. 1. Comparison of the internal friction spectra for (A) hydrogen-loaded and deformed, (B) deformed and (C) hydrogen-loaded Y samples.

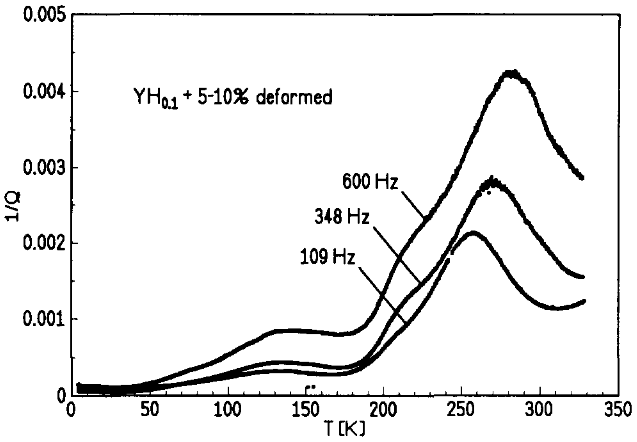

Fig. 2. Internal friction Q^{-1} of a $YH_{0.1}$ sample deformed (5%–10%) by rolling at room temperature.

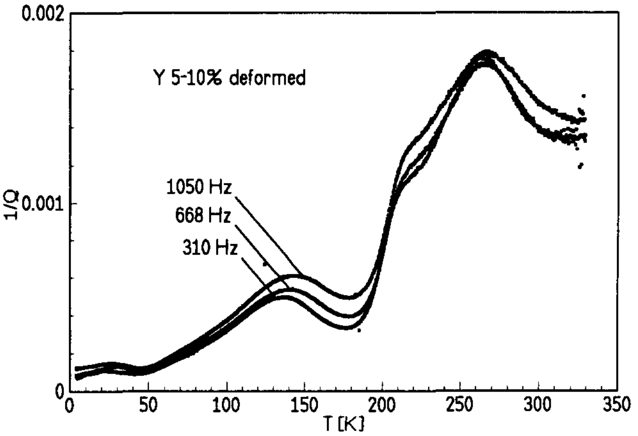

Fig. 3. Internal friction Q^{-1} of an undoped Y sample deformed (5%–10%) by rolling at room temperature.

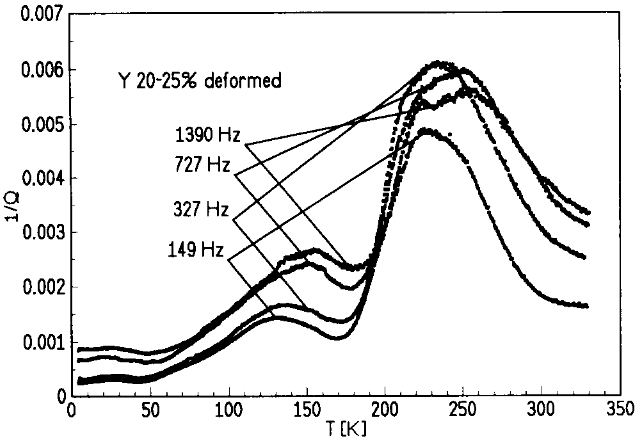

Fig. 4. Internal friction Q^{-1} of an undoped Y sample deformed (20%–25%) by rolling at room temperature.

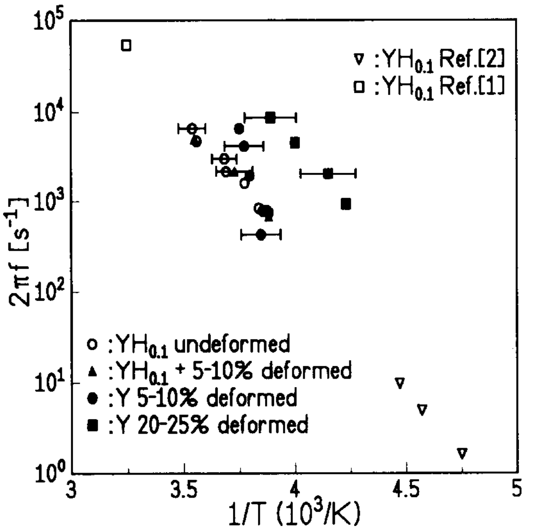

Fig. 5. Arrhenius plot of the peak temperature variation with applied frequency for $YH_{0.1}$ samples and deformed Y and $YH_{0.1}$ samples.

main peak form essentially a single strongly broadened peak, which makes it difficult to determine precisely the temperature of the main peak. For this reason, all the temperature values subsequently given for the main d-peak of this sample (sample 4) can only be considered to represent estimates.

Figure 5 shows an Arrhenius plot in which the vibrational frequencies f are presented as a function of the inverse temperature T_p of the main peak of all our samples. For comparison, the figure also indicates corresponding results of previous internal friction measurements carried out on H-doped (undeformed) $YH_{0.1}$ samples [1–3].

It is possible to determine values for the activation energies of the relaxation process of the main peak from the data in Fig. 5, if any potential temperature dependence of the relaxation strength is neglected. In this case, the H-doped and 5%–10% deformed sample 2 and the undoped and 20%–25% deformed sample 4 have the same activation energy $E \simeq 0.60$ eV, which is also identical to that obtained for H-doped and undeformed $YH_{0.1}$ samples [1–3]. On the other hand, the main d-peak of the undoped and 5%–10% deformed sample 3 exhibits a much larger activation energy. This can also be seen from Fig. 3 in which the temperature of the main peak presented there practically does not shift with frequency.

The above results suggest large differences in the activation energies of the main peak of the two differently deformed (undoped) samples 3 and 4. We think that this reflects differences in the actual plastic deformations of the two samples since inaccuracies in determining the temperature of the main peak for the 20%–25% deformed sample 4 are not likely to simulate a sufficiently large change for the activation energy of the main peak.

The internal friction spectra of the deformed samples 2, 3 and 4 show a shoulder at the low temperature site of the respective main peaks. This is a behavior characteristic for a Bordoni relaxation (observed mainly in face-centered cubic metals) [5, 6]. If the relaxation process in the deformed samples is, in fact, a Bordoni relaxation, the main internal friction peak (dH-peak) of the H-doped and deformed sample 2 may be explained by a simple superposition of an H-peak, caused by diffusive hydrogen jumps, and a d-peak that corresponds to the Bordoni relaxation. In this case, the fact that both peaks exist in practically the same temperature range must be considered to be purely accidental. On the other hand, what makes it difficult to assume a Bordoni relaxation is the high value of the pre-exponential factor found for the relaxation rate τ^{-1} [8]. This factor can be estimated to be $\tau_0^{-1} \simeq 10^{15}$ in the case of the 20%–25% deformed sample, although this sample seems to have the smallest activation energy of those presented in Fig. 5.

It may also be speculated that the main peaks of the deformed samples 2, 3 and 4 result from a type of Snoek–Köster relaxation [8], caused by the presence of H. Such an explanation implies that the deformed samples 3 and 4, which were not intentionally doped with H, contained a small residual amount of H in spite of vacuum annealing at 1100 °C. For instance, it can be estimated that an impurity concentration of $\sim 10^{-3}$ at.% H could already saturate the interstitial sites in direct neighborhood of the dislocation lines, even for dislocation densities as high as 10^{11} cm^{-2}. It cannot entirely be excluded that such a small residual amount of H was, in fact, present in our undoped samples.

A final fact to be noted is that the internal friction peak of the more severely deformed sample (20%–25%

deformation) is found at a lower temperature than in the case of samples with a 10%–15% deformation. Such behavior is indeed characteristic for both Bordoni and Snoek–Köster relaxations [5, 6, 8].

In conclusion, our experimental results show that dislocations in Y yield an internal friction peak (main peak) in the same temperature range as that previously found in the presence of H (D). In fact, the height of the peak caused by dislocations can drastically exceed the peak height in the presence of H (D), even for H (D) concentrations as high as ~10 at.%. Independently of the question of the microscopic mechanism of the dislocation peak (Bordoni or Snoek–Köster–(H) relaxations), the height of this peak shows that reliable discussion of the H-induced relaxation behavior requires consideration of the possible influence of dislocations.

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