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INTERNAL FRICTION IN ZIRCONIUM

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Abstract.- The effect of type (bending, tension or torsion) and temperature (100 K, 300 K) of deformation on the internal friction spectrum of well-annealed polycrystalline zirconium has been investigated at frequencies of about 1 Hz and about 100 Hz. The result of ageing at temperatures not higher than 300 K on both the modulus and the internal friction is also described. The observed peaks are discussed in terms of either dislocation relaxation or dislocation point defect interaction effects and combined with literature data to obtain better defined values for the relaxation parameters.

1. Introduction.- Since the work reported by Hasiguti et al. [1] in 1961, who observed several low temperature internal friction peaks in cold worked metals, this subject has been extensively studied (see e.g. [2]), mainly in f.c.c. metals. In this particular structure, the interpretation of these peaks in terms of either intrinsic dislocation relaxation processes or dislocation-point defect interaction processes seems to be rather well established and accepted.

In h.c.p. metals, however, the behaviour of those low temperature deformation induced internal friction peaks is rather more complicated. The relaxation spectrum appears to depend strongly on the impurity concentration and on the thermomechanical history of the specimens. As a result, there are still various controversial interpretations concerning the mechanisms responsible for the peaks. This is true in particular for zirconium. In this material, room temperature deformation results in a broad peak at about 150 K for 1 Hz. This peak was named Pd in the cited Hasiguti's paper [1], as dislocation movement was suggested to be responsible for it, though the hypothesis of dislocation-point defect interaction was not disregarded by those authors. Following low temperature deformation, another unstable peak was observed by some authors at temperatures much lower than Pd peak, and was attributed either to dislocation relaxation [3] or to the presence of self interstitials [4], while Pd was then interpreted as a dislocation-pinning effect [3]. Some higher temperature peaks were also observed after low temperature deformation [3] and the addition of H or

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O was found to influence the peak height [5-7].

In an attempt to try and clarify the conditions for observation of some of those peaks, in the present work experiments were carried out at two different frequencies on pure zirconium subjected to different types of deformation.

2. Experimental procedures.- The specimens were of 99,99 % pure Zr, either wire or plate shaped, obtained from Materials Research Corp.. The plates were ≈ 0.3 mm thick and cut into a dumbbell shape of 45 mm total length. The wire specimen was 0.5 mm diameter and 100 mm long.

The annealing treatment was performed in an electrical furnace for the plate, and by joule heating for the wire, under initial vacuum conditions better than 8 x 10^{-8} Pa in both cases. The specimen was heated stepwise and very slowly so that the pressure inside the system would never exceed 8 x 10^{-6} Pa. In this way it could be insured that, when the final temperature 1300°C was reached, the vacuum was better than 2 x 10^{-7} Pa. The specimen was kept for 30 min. at this temperature and then slowly cooled. For the wire, it was found necessary to maintain a temperature of 900°C for about 4 h, before heating further to 1300°C.

Electron microscopy observations of the plates after annealing showed the presence of a small density of precipitates which were identified as hydrides, after comparison with data in literature [8]. It might also be, however, that these precipitates were introduced during electro-polishing.

For damping measurements with the wire specimens a normal Kê-type torsion pendulum was used. The measurements were performed at constant strain amplitude, the damping being measured as the input energy required to keep the specimen vibrating at constant amplitude. Measurements were run at either a "low" strain amplitude of 2.10^{-5} or a "high" strain amplitude of 3.10^{-4}. The data were taken with a continuously increasing temperature (at a rate of about 0.5 K/min) and at a frequency of about 1 Hz. Deformation by torsion was carried out in situ by means of a gear mechanism adapted to the pendulum.

A flexural resonance system was used for the plates, vibrating as a cantilever at a frequency of about 80 Hz. Using electrostatic driving and F.M. detection, the specimen was again vibrated at constant strain amplitude, using input energy as a measure of the internal friction.

3. Experimental results.- A huge and well-defined peak (T_p = 140 K, 0.5 Hz) is observed in the as-received specimens (figure 1, curve 1). This peak has been repeatedly described in the literature and will be

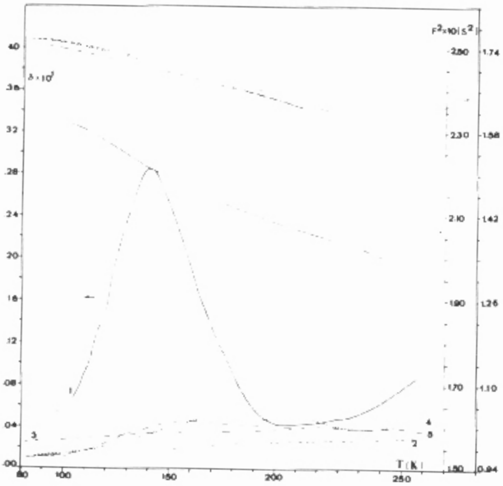

Fig. 1 : Low frequency internal friction in as-received Zr (1); effect of annealing (2); torsional deformation at L.T. (3,4) and at R.T. (5).

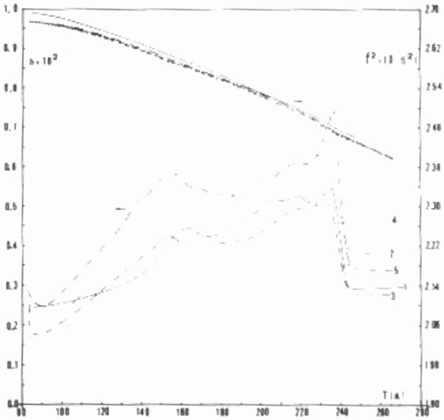

Fig. 2 : Internal friction in zirconium.
(1):annealed and lightly handled; (2):deformed by bending at R.T.; (3):after ageing; (4):measured at high strain amplitude; (5):measured again at low amplitude.

called here Pd following the nomenclature introduced by Hasiguti and co-workers [1]. The peak disappears after the annealing treatment (curve 2) and can be reintroduced, although with a much smaller peak height, after 1 % deformation by torsion at room temperature (curve 5). When the same amount and type of deformation was applied at 80 K to the specimen in the condition (2), the peak did not appear immediately (curve 3). After annealing at 210 K, however, the peak was defined (curve 4) and the presence of other higher temperature peaks was slightly suggested.

Besides its dependence on the temperature of deformation, the observation of these higher temperature peaks seems to depend also on the mode of deformation. Several peaks between 160 K and 240 K were detected in one specimen annealed and slightly deformed by handling during mounting (figure 2, curve 1). After a 2 % deformation by bending at room temperature (curve 2), the peak Pd also appeared (curve 3 corresponds to the same deformation, after one day at R.T.). When subsequent measurements were made at a larger strain amplitude, the overall damping level as well as the peak heights were found to decrease (curve 4). By measuring again at lower amplitude after one day resting, the peak heights increased again and in addition there was also a larger and sharper peak at 230 K (curve 5).

In order to verify this behaviour and to try and obtain activation parameters for the observed peaks analogous experiments were

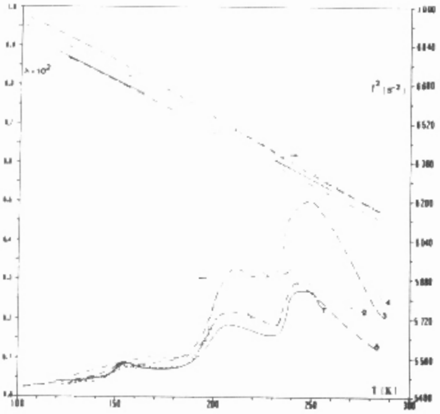

Fig. 3 : Higher frequency internal friction in one specimen deformed 1 % by bending at 100 K (1). After annealing at 260 K(2); effect of ageing 7 days at R.T. (3); after 12 days at R.T. (4); after new deformation at 100 K(5).

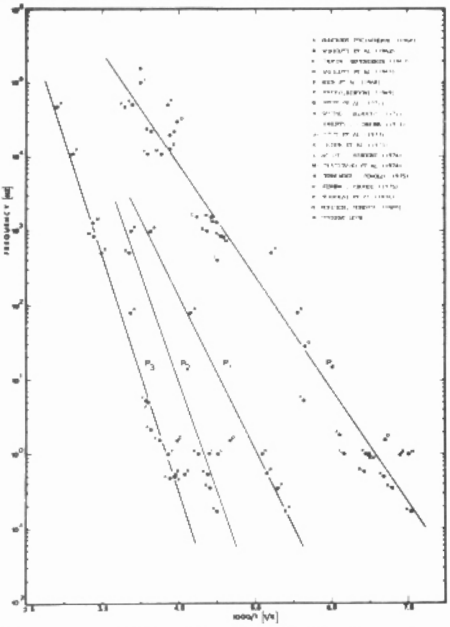

Fig. 4 : Arrhenius graph for Zr including data from different authors. 1-peak Pd; 2-peak P_1; 3-peak P_2; 4-peak P_3.

carried out at higher frequencies. The specimens used had a dumbbell shape, and were deformed after the annealing treatment, by bending in situ at ≈ 100 K. This bending deformation was performed by using the (mobile) electrodes, and the amount of deformation was roughly estimated from the shape of the specimen after bending (≈ 1 %). In figure 3 (curve 1 to 4) one observes the presence of at least two peaks in the curve obtained just after deformation. Ageing at room temperature results in an increase in peak height and in a shift to higher temperatures. The peaks were found to decrease again when the specimen was once more deformed in the same way (curve 5).

4. Discussion.- The observation of a well-defined Pd peak in zirconium seems to require a rather high amount of deformation, while the peak temperature shifts to lower temperatures for a heavily deformed specimen like in an as-received condition (see e.g. ref. [11]). This fact, combined with other factors like purity, previous thermomechanical history etc., explains the large scatter in an Arrhenius graph for Pd data from different authors (figure 4, curve 1). The activation parameters derived from these data are listed in table 1. These values differ considerably from those calculated by Hasiguti et al. [1] who obtained 0.18 eV and 3 x 10^7 s^{-1} for activation energy and frequency factor, respectively.

The peak does not appear immediately after liquid nitrogen

Peak	Tp (K) (f = 0.5 Hz)	E (eV)	fo (s^{-1})
Pd	140	0.30	5.3×10^{10}
P_1	188	0.41	3.1×10^{11}
P_2	205	0.59	1.6×10^{14}
P_3	219	0.63	5.3×10^{13}

TABLE 1 : Activation parameters for deformation induced internal friction peaks in zirconium

deformation (figure 1, curve 3). This behaviour was also observed by Savino and Bisogni [3] and those authors considered as necessary for the peak to appear that point defects migrated to the dislocations. Hydrogen was suggested as the migrating point defects and the interaction of dislocations with these point defects as causing the peak. Yet, in zirconium charged with hydrogen [7], it was found that the peak height was much larger in a specimen which did not contain hydrogen. As one does not expect the specimens used in the present investigation to contain much hydrogen (or oxygen) after the heat treatment, it appears reasonable to interpret the Pd peak in terms of dislocation relaxation. The necessity of an ageing treatment following deformation at liquid nitrogen temperature for the Pd peak to appear can then be explained as follows. Immediately after low temperature deformation there is a modulus decrease (curve 3 of figure 1) when compared to the annealed condition (curve 2). This is caused by the creation of dislocations containing a large concentration of geometrical kinks, thus inhibiting the observation of a dislocation relaxation peak. During the internal friction measurement, dislocation rearrangement brings them into the valleys of the Peierls potential. In this condition then, the dislocations cannot move at temperatures below Pd, so that there is a recovery in the modulus, but they can contribute to Pd which can now be observed at 160 K (curve 4).

The higher temperature peaks (figures 2 and 3) have also been plotted in an Arrhenius graph together with data reported in the literature (figure 4, curves 2,3,4). The estimates for peaks P_1 and P_2 so obtained (see table 1) are in good agreement with those reported by Hasiguti [1] for Pα (0.50±0.05 eV, $2\times10^{13\pm1}s^{-1}$) and Pβ (0.51±0.07eV, $9\times10^{12\pm2.4}s^{-1}$) *respectively. As first considered by those authors,* these peaks might be caused by an interaction between dislocations and point defects: deformation of the specimen is required and the peak appears at a temperature region where both, dislocations and point defects, are mobile. For some reason that might be related to deformation mechanism and mode of deformation, these peaks are better defined when the specimen is deformed by bending rather than by torsion or uniaxial tension. According to Fromont et al. [9] the amount of point defects created in zirconium is much higher after cold-rolling at room temperature than after tensile deformation; deformation by torsion at 4.2 K creates 10 times more defects than extension at room temperature.

When a large strain amplitude is used, the peak heights are strongly suppressed (curve 4, figure 2). This can be explained by assuming that as a result of the high amplitude oscillation the point defects have been dispersed and can no longer contribute to the dislocation point defect interaction peaks. During ageing at room temperature, the point defects are mobile and return to the dislocations, thus restoring (and even enhancing), the relaxation strengths. For specimens deformed at low temperature, the dislocation point defect interaction peaks develop during heating to room temperature. Ageing at R.T. causes more point defects to migrate to dislocations and thus leads to the final peak shape (figure 3).

The sharp peak at 235 K (figure 2) could be related with twin boundary movements. It is, however, difficult to prove this hypothesis before a careful electron microscopy study of the various material conditions used for the internal friction experiments has been been.

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