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ANELASTIC PHENOMENA ASSOCIATED WITH THE LONG-RANGE DIFFUSION OF SELF-INTERSTITIALS AND THEIR INTERACTION WITH DISLOCATIONS IN ELECTRON IRRADIATED MAGNESIUM

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Résumé - Le frottement interne et le module élastique du magnésium de haute pureté ont été étudiés pendant l'irradiation à 2 K avec des électrons de 0.4 ou 0.6 MeV et durant des cycles de recuit. Un stade d'ancrage très marqué se produit entre 9 et 13 K. Il montre que les autointerstitiels diffusent à grande distance à ces températures. Lors de recuits ultérieurs, deux pics de relaxation apparaissent à 6 K et à 15 K. Ils trouvent leur origine dans l'épingleage des dislocations par arrivée des interstitiels, qui interagissent avec les décrochements géométriques.

Abstract - The internal friction and elastic modulus of high purity magnesium were studied during irradiation at 2 K with 0.4 - 0.6 MeV electrons and subsequent anneal sequences. The salient feature of the results is the evidence of a marked pinning stage in the temperature interval 9 to 13 K. This indicates that long-range diffusion of elementary interstitial defects occurs at these temperatures. Post-irradiation anneals enabled also the discovery of two relaxation peaks centered at 6 K and 15 K, respectively. These stem from the arrival of the diffusing self-interstitials at dislocations, and presumably from their interactions with geometrical kinks.

I. INTRODUCTION.

From a general viewpoint, after a long-standing controversy, the state of affairs about the configurational and migratory properties of the split-interstitials is now settled in a number of metals. However, in a few of them, there is still some suspense about the very existence of these defects and their observability. This is precisely the case for magnesium. In this metal, it was deduced from Huang X-ray scattering measurements that interstitial clustering was below 6 K [1]. In contrast, on the basis of electrical resistivity studies between 1.5 K and 150 K, none of the existing stages in the recovery spectrum of electron irradiated magnesium were interpreted as due to the random diffusion of self-interstitials [2-4]. The present research is intended to elucidate the situation for the freely migrating self-interstitials, using dislocations as sensors. An attractive feature of magnesium is the existence of large anelastic effects, in the highly pure material [5-6], at very low temperatures.

II. IN-LINE MEASURING DEVICE

The sample used is a rectangular strip 2 cm long, 0.4 cm wide, with thickness 300 μm , obtained by cold-rolling. It is clamped in a copper block in which a heat exchanger is incorporated. This system works between 2 K and 900 K by combined use of a liquid helium stream and furnace heating. The copper block includes also a helium III container. By pumping on the helium III bath, temperatures as low as 0.7 K (with no flux) and about 2 K (for a moderate electron flux rate) are attainable. A Rh-Fe thermocouple is used to monitor the sample temperature. For damping and dynamic modulus measurements, the strip is operated in flexural vibrations with use of electrostatic excitation [7].

The material studied is high purity magnesium prepared by zone melting at the Centre d'Etudes de Chimie Métallurgique de Vitry sur Seine. Neutron activation indicated that the total impurity concentration is below one atomic part per million. After mounting, the sample was heat treated under vacuum at 440 K for 20 min. to produce long dislocation lines.

III PREIRRADIATION DAMPING AND MODULUS SPECTRA

Most features of the overall damping spectrum of high purity hcp magnesium are quite similar to the ones observed in a number of fcc metals [6-7]. In particular, after anneals in the range 400 to 500 K followed by cold-work, two Bordoni peaks labelled B_1 and B_2 exist at 60 K and 120 K, at a measuring frequency of about 1000 Hz [8]. Subsequent annealing at temperatures higher than 340 K produces the disappearance of B_2 .

The presently measured temperature variations of internal friction and elastic modulus are shown in Fig. 1 for the non-irradiated condition. In the considered case of a 440 K heat treatment, only the first of the two Bordoni peaks exists. In agreement with prior work. It is seen in Fig. 1 that a large peak termed D shows up around 15-20 K. In addition a large background damping exists at still lower temperatures. Further inspection of Fig. 1 reveals that the relaxation strength of peak B_1 keeps almost unchanged when the vibrational strain amplitude is varied by a factor of 5, while the background damping is markedly increased with increasing amplitude. Noteworthy, for the lower strains, a small peak, called π_1 , appears to be superimposed on D. In prior work this peak has been traced to interactions between geometrical kinks and vacancies [6,9,10].

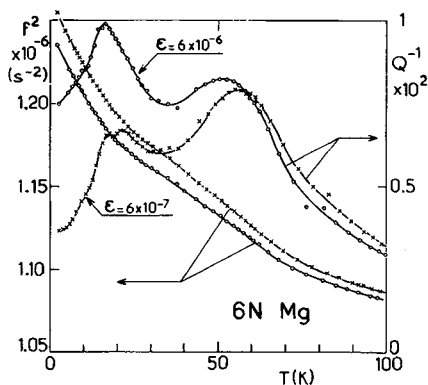


Fig. 1. Experimental damping and modulus spectra of high purity magnesium obtained during linear heating at a rate of $2 \text{ K} \times \text{min}^{-1}$ following a preannealing treatment at 440 K. Two vibrational strain amplitudes were used, with respective values of 6×10^{-6} and 6×10^{-7} .

IV ARRIVAL OF FREELY MIGRATING SELF-INTERSTITIALS AT DISLOCATIONS

The mobility of the self-interstitials in magnesium is still controversial. It is recalled that during low energy (0.12 to 0.4 MeV), low temperature (1.5 K or 4.2 K) electron irradiation, only a slight thermally activated recovery of electrical resistivity has been detected, if any [2-4]. The immediate inference that was drawn is that self-interstitials are not mobile below 4.2 K. In sharp contrast to these results, Huang X-ray scattering experiments carried out at 4.5 K or 6 K after irradiation with 1.1 MeV or 3 MeV electrons have revealed that essentially small interstitial clusters were present, but no isolated defects. A hypothesis emerging from this observation is that self-interstitials become mobile below 6 K. However this interpretation is objectionable, since the relatively high energy of the impinging particles in that case may have resulted in the formation of multiple, rather than single defects, on account of the very low displacement threshold energy of magnesium, 10 eV [11].

Radiation induced modulus variations are of special usefulness in trying to solve this dilemma. Fig. 2 shows the modulus variations with temperature measured during two successive heating runs between 2 K and 30 K, starting from the respective following states : (a) non-irradiated, well-annealed condition, as for Fig. 1 ; (b) after a 2 K irradiation at a dose of $3 \times 10^{14} \text{ e}^- \times \text{cm}^{-2}$. The notable features of the curves are that (1) the modulus was increased by about 1 % during the irradiation (2) a still more marked modulus increase occurs during the subsequent heating. This increase, which amounts to more than 2 % of the preirradiation value is localized between 9 K and 13.5 K.

The moderate modulus variation detected during the 2 K irradiation is due to dislocation pinning by the defects directly created on the dislocations or in their immediate vicinity. Now the marked pinning stage centered on 11 K cannot be explained in terms of close pair or correlated recombination. It is plain that it originates in the arrival of interstitials at dislocations as a result of their long distance random diffusion.

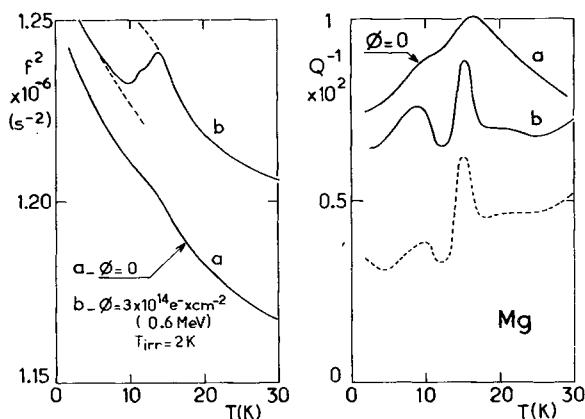


Fig. 2 (left). Modulus versus temperature curves for the unirradiated condition (curve a) and after irradiation at 2 K with 0.6 MeV electrons, to a flux of $3 \times 10^{14} \text{ e}^- \times \text{cm}^{-2}$ (curve b).

Fig. 3 (right). Damping curves traced in the same conditions as for Fig. 2. Maximum strain amplitude was also 5×10^{-6} except for the dotted curve, for which strain amplitude was 1×10^{-6} .

This finding enables interesting comparison with the resistivity recovery spectrum observed after low temperature electron irradiation. It mainly consists of two pronounced stages located in the two regions 4.5 to 8 K and 9 to 13 K, respectively. No other well-resolved structure is present between 15 K and 140 K. The location of the above stages has been found not to depend on irradiation dose. Hence they were supposed to reflect first-order recovery processes, that is close-pair recombination events. However on account of their width and shape characteristics, the superimposition of several close-pair, first-order processes had to be invoked. The present results show that the first stage is indeed due to close pair recombination, since the modulus defect is not affected. Now, interestingly, the only modulus stage evidenced is located in the very same temperature interval as the one for the second resistivity stage. This definitely shows that this stage corresponds to random interstitial diffusion, that is to stage I_E , or possibly stages $I_D + I_E$ in the usual terminology of irradiated metals.

V. DISCOVERY OF LOW-TEMPERATURE RELAXATION PEAKS

It is apparent in fig. 3 that when temperature is raised further, a well defined relaxation peak is observed. It is centered on 14 K. Its height keeps practically unchanged when the maximum strain amplitude is enhanced from 1×10^{-6} to 5×10^{-6} . More details about the characteristics and stability of this peak are given in Fig. 4. For this purpose, the sample

was given successive linear heating runs at progressively increased temperatures followed by cooling down to 4 K. Before this anneal sequence was started, the sample had been heat treated at 440 K to anneal out the damage and most changes induced by the irradiation just considered. Then it was exposed to a second irradiation, to a flux of $2.5 \times 10^{15} \text{ e}^- \times \text{cm}^{-2}$. Curves 2 to 5 totally confirm the previously observed pattern during the direct anneal to 30 K. Additionally, after annealing up to 15.5 K, a new peak shows up around 6 K (curve 4). It is further developed by the further anneals, until it goes through a maximum after a 20K anneal (curve 6) before it starts getting slowly decreased by anneals at still higher temperatures.

Clearly, on account of the estimated very low defect concentration, presumably of the order of 10^{-7} , neither of these peaks can be produced by stress-induced reorientation of relaxing entities of interstitial type. A more plausible interpretation is in terms of interstitial-dislocation interactions. A redistribution of the pinning points by their diffusion along the dislocation lines would cause the growth of the 6 K peak at the expense of the initially existing 15 K one.

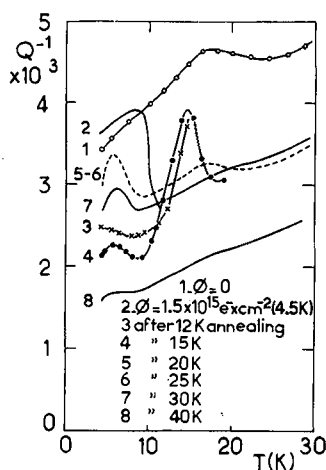


Fig. 4. Damping curves traced during successive anneal sequences at a heating rate of $2 \text{ K} \times \text{min}^{-1}$, at progressively increased temperatures. These treatments have revealed the existence of two relaxation peaks, centered at 6 K and 15 K, respectively.

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