Free Migration of Interstitials in Tungsten

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The internal friction and dynamic modulus of W single crystals were measured after neutron irradiation near liquid-helium temperature. The results on dislocation pinning and the associated relaxation peaks suggest that in W the ⟨110⟩ split interstitials perform a free migration at ~15 K (substage I_1) and the ~30-K stage (substage I_2) is associated with detrapping of the ⟨110⟩ split interstitials from impurity atoms.

The internal-friction technique is a powerful tool for the study of point defects in metals in two respects. With a low defect concentration, one can study the long-range migration of the defects by measuring dislocation pinning. The arrival of the point defects at a dislocation causes dislocation pinning or shortens the dislocation-loop length, and decreases both the internal friction and the modulus defect associated with dislocation motion. With a higher defect concentration, on the other hand, if one can find relaxation peaks associated with the point defects, the type of these defects can be determined through the study of the anisotropy of their strain field.[1,2] In this note, results of this type of study on the stage-I defects of W will be reported.

Specimens were plates of W single crystals with ⟨100⟩ crystallographic direction (stress axis of flexural vibration parallel to ⟨100⟩). They were cut from a single-crystal rod obtained from Materials Research Corporation (nominal purity 99.99%), etched, and then annealed at 2100°C in a vacuum of 2×10^{-8} Torr. Their residual resistivity ratio (RRR) after annealing was about 8200. They were irradiated near liquid-helium temperature by fast neutrons, 1×10^{12} neutrons/cm^2 sec with energies greater than 0.1 MeV. The internal friction and dynamic modulus were measured by flexural vibration at ~500 cps. The experimental procedures were similar to those described elsewhere.[3]

Figures 1(a) and 1(b) show an example of the results for high irradiation doses. Two pronounced relaxation peaks at 8 and 27 K can be seen in the figure. From these results together with other results from various irradiation doses and from the ⟨111⟩ and ⟨110⟩ specimens which are not shown here, the following features were found for these two peaks: (1) The 8- and 27-K peaks anneal out in recovery substages I_1 (~15 K) and I_2 (~28 K),[4] respectively; these are the first two substages of stage I found in the electrical-resistivity measurements by Kunz *et al.*[5] on electron-irradiated W. (2) Dislocation pinning by radiation-induced defects gives no essential effect on

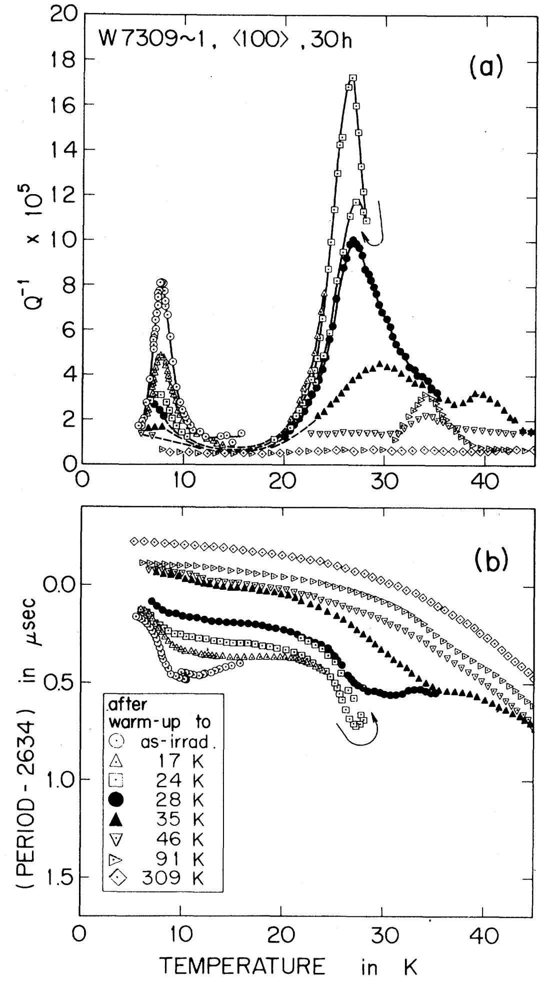

FIG. 1. (a) Internal friction and (b) dynamic modulus of the ⟨100⟩ single-crystal W during warmup after fast neutron irradiation near liquid-helium temperature for 30 h. Dynamic modulus was represented by the period of resonant vibration. Warmup runs were made to successively higher temperatures as shown in the figure.

the peaks. (3) The peak heights of both peaks are roughly proportional to defect concentrations. (4) From the orientation dependence of the peak heights, it can be concluded that the defects associated with both peaks have a maximum strain field along a ⟨110⟩ crystallographic direction.

From these results, one can conclude that the 8- and 27-K peaks are associated with the stress-induced rotation of the defects in the substages I_1 and I_2, respectively, and these defects are ⟨110⟩ split interstitials of two different types.

An example of the results for low irradiation doses is shown in Figs. 2(a) and 2(b). In these figures, two pronounced pinning stages centered at about 15 and 30 K can be seen clearly, where the internal friction decreased and the dynamic modulus increased. This shows that there are point defects of two types which perform a long-range migration in substages I_1 and I_2, respectively. From the results of high irradiation doses, these point defects must be ⟨110⟩ split interstitials. Incidentally, new types of interstitials proposed by Afman[6] and Moser[7] are not reconcilable with the above interstitials with respect to the symmetry found in the present experiments. The present results on the stage-I defects in W have a striking analogy with those found in Mo.[8-10] In Mo, ⟨110⟩ split interstitials of two types perform a long-range migration at around 30 K (substage I_2) and 40 K (substage I_4), respectively.

Therefore, from the above results, one will be led naturally to the conclusion that in W, ⟨110⟩ split interstitials perform a free migration in substage I_1, and the same interstitials, but trapped by impurity atoms of a certain kind, release from the trap in substage I_2. Furthermore, for low irradiation doses, dislocation pinning proceeded slowly but almost continuously above 30 K. This suggests that the other substages of stage I are also associated with the detrapping of interstitials from various impurities. The concentration of metallic impurities estimated from the RRR, following Schultz,[11] is ≲ 40 at. ppm, and the concentration of Frenkel defects after a 30-h irradiation is estimated to be about 60 ppm from the resistivity measurements.[12] Therefore, the present model of impurity trapping does not seem unreasonable.

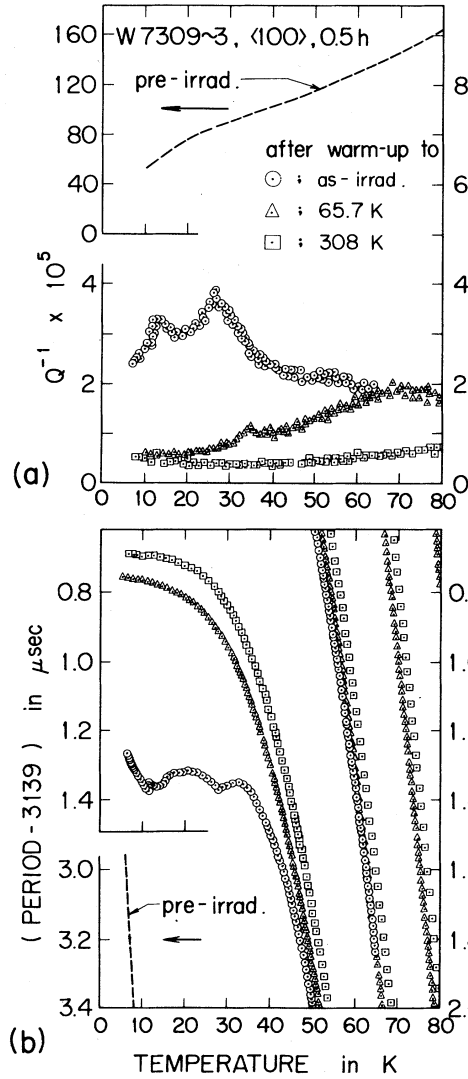

FIG. 2. (a) Internal friction and (b) dynamic modulus of the ⟨100⟩ single-crystal W during warmup after fast neutron irradiation near liquid-helium temperature for 0.5 h. Warmup runs were made to successively higher temperatures as shown in the figure.

DiCarlo, Snead, and Goland[13] found a relaxation peak at ~30 K in electron-irradiated W which is considered to correspond with the present 27-K peak. They assigned this peak to close pairs, but the present assignment is not in contradiction with their findings. The present 8-K peak does not seem to correspond with the 10-K peak found by Townsend *et al.*,[14] since their 10-K peak was said to be stable up to 30–40 K. Scanlan, Styris, and Seidman[15] studied the stage-I recovery of very pure W [RRR of (4–5) $\times 10^4$] by field-ion microscopy. After ion irradiation of W at ~15 K, they observed interstitials appearing at the specimen surface in a broad temperature range beginning at ~20 K. According to the present model, the interstitials they observed above ~20 K were those released from impurity traps. As they mentioned in their paper, even with this high

RRR, there was a considerable probability for impurity trapping. Kunz *et al.*[5] found that prequenching enhanced all the substages of stage I except substage I_1. If the prequenching had introduced only vacancies, their results suggest that substage I_1 is associated with close pairs, but not with free migration. If, however, the prequenching had caused dispersion of impurity clusters, the present model does not necessarily contradict their results. At present, we consider that the latter possibility is the case.

In conclusion, it is considered that a free migration of ⟨110⟩ split interstitials occurs at ~15 K in W and ~30 K in Mo and that the other recovery stages at higher temperatures (below room temperature) are probably associated with detrapping of ⟨110⟩ split interstitials which were trapped by various impurity atoms. A detailed account will be published elsewhere.

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Selection-Rule Effects in Electron-Loss Spectroscopy of Ge and GaAs Surfaces*

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Low-energy electron-loss spectroscopy on Ge and GaAs surfaces reveals opticlike selection-rule behavior for transitions involving *d*-core states and empty dangling-bond surface states. From the observed breakdown of these rules at low incident primary electron energies the symmetry of the dangling-bond states may be estimated. The Ga dangling bond is found to be largely *s* like on all surfaces, whereas the Ge dangling bond exhibits *p*-like character on the (111)-(8×8) surface, and mixed *s*-*p* character on the (100)-(2×2) surface.

We present in this Letter new experimental results of low-energy electron-loss spectroscopy (LEELS) on Ge and GaAs single-crystal surfaces which indicate strong primary-energy and surface-orientation dependence for electronic transitions involving core levels and empty localized surface states. It is concluded that the results allow the determination of the symmetry of the final states, information generally not available by other surface techniques.