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Measurement of Relative Void Surface Energies in Irradiated Metals by Small-Angle Scattering*

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Voids formed in neutron irradiated metals are faceted on various crystallographic planes. This faceting has been shown to cause significant anisotropy in the small-angle scattering pattern [Epperson, J. E., Hendricks, R. W. & Farrell, K. *Studies of Voids in Neutron Irradiated Al Single Crystals. I. Small-Angle X-Ray Scattering and Transmission Electron Microscopy*, To be published; Hendricks, R. W., Schelten, J. & Schmatz, W. *Studies of Voids in Neutron Irradiated Al Single Crystals. II. Small-Angle Neutron Scattering*, To be published]. In the case of neutron-irradiated Al single crystals, the voids may be generally characterized as octahedral having (111) faces with a varying degree of truncation on (100) faces. If it is assumed that the voids are in an equilibrium shape (which can be achieved by a low-temperature anneal), the specific surface energies γ_{hkl} and the truncation parameter t are related by $\gamma_{100} = \sqrt{3}(1-t)\gamma_{111}$. Hendricks, Schelten & Schmatz proposed that the truncation parameter t could be measured by studying the anisotropy of the tail of the small-angle scattering curve. In such an experiment, the scattering is measured at constant $|k|$ in a given plane in reciprocal space as a function of rotation of the crystal about an axis perpendicular to the diffraction plane. In this paper, the sensitivity of the proposed experiment to (i) the distribution of void sizes $N(D)$ and (ii) various crystallographic planes is numerically explored. It has been found that for certain low-index planes, the effect is sufficiently independent of $N(D)$, that reasonable estimates of t can indeed be found. Thus, it is possible to obtain the specific void surface energies from small-angle diffuse scattering data.

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Small-Angle Scattering in Neutron Irradiated Copper

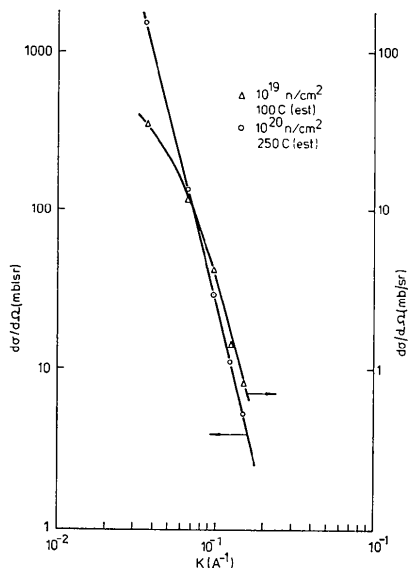
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Work which has been started at Pelindaba on small-angle scattering in neutron irradiated copper is described. The facility consists of a 15 m long *S*-curved waveguide, which transmits a neutron spectrum with a maximum at 9 Å and a FWHM of 4 Å [Hofmeyr, C. & Isebeck, K. (1964). *Nucl. Instrum. Meth.* **117**, 9–16]. The collimator has an angular divergence of 0.01 rad and a comparative measurement is made of a sample against a reference sample. Samples of ASARCO copper purified by annealing at 960 C for 1 hour in a vacuum of 5×10^{-4} torr were subsequently irradiated in the SAFARI reactor to 10^{19} and 10^{20} n/cm² ($E > 0.1$ MeV) at estimated temperatures of 100 C and 250 C respectively. The small-angle scattering effect was shown to be due to radiation-induced damage through measuring a sample before irradiation, after irradiation and after annealing at 450 C for 10 minutes, which restored the original scattering characteristics. The differential coherent-scattering cross sections ($d\sigma/d\Omega$) have been plotted on a log-log scale against the wave vector K (Figure). Samples irradiated to 10^{20} n/cm² show an increase in scattering over the lower-dose-irradiated samples, the increased scattering being particularly strong for $K < 0.05 \text{ \AA}^{-1}$. For the former, the plot is linear over three orders of magnitude and has a gradient of -4.0 . Such a dependence is expected if voids are present (Porod's equation) provided that they are randomly oriented. In a polycrystalline material this will be true even if the voids are faceted. We therefore conclude that voids have been formed at this fluence and temperature, which accords with

data taken by Adda, Y. [*Radiation-Induced Voids in Metals*. USAEC Symposium series 26, p. 40. Albany: USAEC]. For the 10^{19} n/cm² samples, a gradient of 3.6 was obtained indicating that a mixture of voids and loops contributes to the small-angle scattering (dislocation loops would produce a K^{-3} dependence). The size and concentration of the voids have been estimated from that portion of the Porod plot, which is linear whilst another estimate of the voids as well as the loops has been made from the low- K side of the Guinier plot (Table). In the sample irradiated to 10^{19} n/cm², both loops and voids are present and the concentrations in the table were derived assuming only one or the other species to be present. For the high-temperature, high-dose sample, a definite interpretation can be made in terms of voids of density $\sim 10^{14}$ cm⁻³ and diameter between 130 Å and 180 Å.



Figure

Table				
Irradiation	Type	Diameter (Å)	Density cm ⁻³	Plot
10^{19} n/cm ² $T = 100\text{C (est)}$	Loop	85	4×10^{16}	Guinier
	Void	85	1×10^{13}	Guinier
10^{20} n/cm ² $T \approx 250\text{C (est.)}$	Void	65	7×10^{13}	Porod
		130	6×10^{14}	Guinier
		180	8×10^{13}	Porod

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The Determination of the Dislocation Structure in Copper Single Crystals from X-ray Diffraction Profiles

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A careful analysis of X-ray diffraction profiles obtained from copper single crystals with dislocation densities between 10^4 and 2×10^6 dislocations per cm² has been performed. Lattice tilts and strains introduced by the as-grown dislocations have been deduced from double-crystal rocking curves recorded in a large range of Bragg angles, up to 89° . A new numerical deconvolution method [Mendes, M. & Delestre, C. Submitted to *Acta Cryst.*] has been applied successfully to unfold the broadened diffraction patterns. The dislocation density ρ has been determined by etch-pit counting and by the application of Wilkens' theory [Wilkens, M. (1970). *Phys. stat. sol. (a)* **2**, 359-370] to the diffraction profiles. Very good agreement has been obtained between the methods. The samples were free from low-angle grain boundaries. However, as a function of ρ the lattice tilts increased much faster than the strains, thus revealing the non-statistical distribution of the dislocations. The results indicate an arrangement of the dislocations in walls or cells as proposed by Bassim, M. N. & Kuhlmann-Wilsdorf, D. [*Cryst. Lattice Defects* (1973). **4**, 9-27]. The refined X-ray diffraction technique applied in this work has proved to be useful for obtaining information about the average arrangement of a great number of dislocations which is difficult to extract from topography or electron microscopy experiments. It can be used even for nearly perfect crystals.