Letters

Examination of Neutron-Irradiated UO_2 using the Scanning Electron Microscope

One of the limiting factors in the use of uranium dioxide as a nuclear fuel is the behaviour of the gaseous fission products. Examination of neutron irradiated uranium dioxide using thin foils [1, 2] and replica techniques [3, 4] has revealed both intergranular and intragranular porosity. Thin foil techniques are not suitable for the examination of large grain-boundary pores (> 500 Å) or for the observation of bubble linkage. Replica techniques, although showing the extent of bubble linkage and grain-boundary porosity, suffer from the disadvantage of mechanical damage and distortion when removing the replica from the specimen surface. The scanning electron microscope (SEM) is useful in that it involves the direct observation of the specimen surface, aiding location of specific areas and it eliminates extraneous effects due to specimen preparation. This communication describes observations made on neutron-irradiated UO_2 using a Cambridge Stereoscan SEM. The material used was taken from irradiated UO_2 which had acquired a burn-up from 1.0×10^{26} to 5.4×10^{26} fissions/m^3 and the specimens examined covered a temperature range from 750 to 1750° C. Sections of thickness 1 mm were fractured and the freshly fractured surfaces coated with an evaporated layer of aluminium. The specimens were examined at 30 kV using a current of 200 μA. Typical micrographs are shown in figs. 1 to 3. Fig. 1 shows the structure at a calculated temperature of 1200° C and at a burn up to 10^{26} fissions/m^3. This is a typical cleavage fracture and is characterised by the "river patterns" on the fracture faces; a small amount of grain-boundary porosity can be observed. Fig. 2 shows the nature of fission gas bubbles on a grain face after irradiation at 1600° C to a burn up to 10^{26} fissions/m^3. This micrograph illustrates stages of bubble linkage. Thus we have discrete bubbles in the range 0.5 to 15 μm diameter together with larger bubbles formed by the coalescence of two or more bubbles. An interesting feature in fig. 2 is the association of gas bubbles with particles; these are thought to be precipitates of solid fission products. Fig. 3 shows the structure at an estimated temperature of 1500° C after a burn-up of 5.4×10^{26} fissions/m^3. The grain faces have numerous fission-gas bubbles in the range 0.2 to 2 μm. Gross bubble linkage has taken place along the grain edges to form "tunnels". The amount of gas per unit area of boundary was

Figure 1 Intragranular fracture of irradiated uranium dioxide showing both discrete fission gas bubbles and "river patterns" characteristic of cleavage (× 1600).

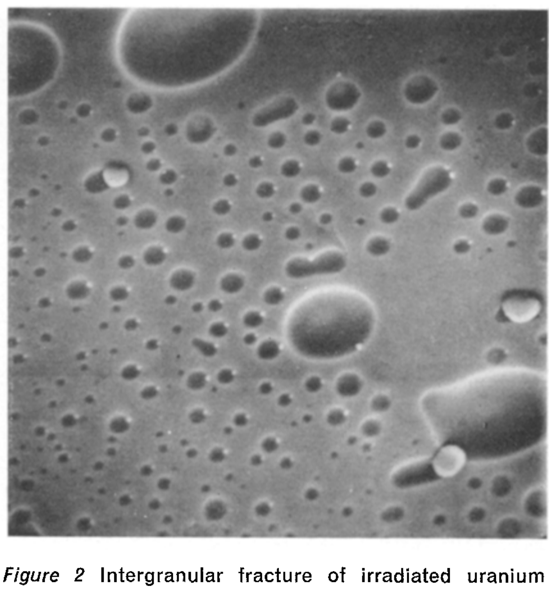

Figure 2 Intergranular fracture of irradiated uranium dioxide showing precipitates together with stages of bubble linkages (× 1600).

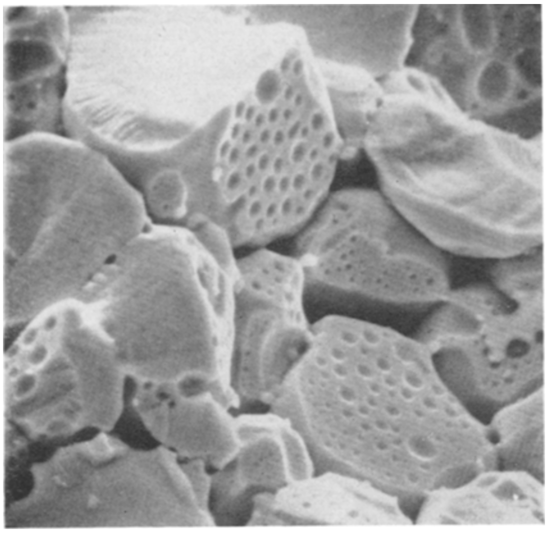

Figure 3 Intergranular fracture of irradiated uranium dioxide showing discrete bubbles on grain faces and tunnels on grain edges (× 1600).

calculated using the method of Speight [5] for lenticular bubbles whose pressure is assumed to be in equilibrium with the surface forces. The ratio of the major and minor axes of the bubbles was determined from a series of micrographs. Calculations gave a value of $\sim 10^{18}$ gas atoms/m^2 of boundary for the material examined. The results indicate the nature of the porosity in neutron-irradiated UO_2. With increasing temperature the amount and nature of the porosity changes. The small discrete bubbles on the grain faces grow and coalesce to form large irregular shaped bubbles (figs. 2 and 3). The linkage of bubbles along the grain edges results in the formation of tunnels, which will contain the gas until intersected by a free surface. This work illustrates that the SEM is a satisfactory means of examining the nature of grain-boundary porosity in irradiated UO_2.

Acknowledgement
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References
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Determination of Particle Size and Strain in a Filed Face-centred-cubic Copper-Silicon-Manganese Alloy by the Method of Variance

Tournarie [1] and Wilson [2-4] have shown the importance of "variance" as a measure of line-broadening in X-ray diffractometric investigations. Although the effect of the long tails associated with the diffraction peaks tends to detract from the accuracy of the results, this method still possesses some specific advantages, such as simplicity, to warrant its application in studies of line-broadening.

Variance is a measure of dispersion observed in a line profile. It is defined [4] as the mean square deviation from the mean (centre of gravity):

$$W_{2\theta} = \langle(2\theta - \langle 2\theta\rangle)^2\rangle = \frac{\int(2\theta - \langle 2\theta\rangle)^2 I(2\theta)\,d(2\theta)}{\int I(2\theta)\,d(2\theta)}$$

where $\langle 2\theta\rangle$ is the centre of gravity position.

Variance has the property of additivity and correction for experimental aberrations (such as lack of monochromation in the incident beam, finite size of specimen, source and detector, etc.) is done by simply subtracting the variance of the annealed line profile from that of the cold-worked one. Although technically the limits of the integral given above are $-\infty$ and $+\infty$, it is difficult to take them so in practice. So a finite range is taken for the line profile which gives a finite value for the variance. Naturally the establishment of the background level of the peaks gives a little uncertainty, which has been discussed [4].

Mitra [5] has described a graphical method for the determination of an apparent particle size and strain from the measured values of variances of line profiles. He shows that a plot of $W \cos \theta/\lambda\sigma$ against $n^2\lambda/\sigma \cos \theta$, (where W is the variance due only to deformation, θ is the Bragg angle, λ is the wavelength of the radiation