



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

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/m² of boundary for the material examined. The results indicate the nature of the porosity in

neutron-irradiated UO₂. 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 UO₂.

Acknowledgement

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30 September 1969

G. L. REYNOLDS
G. H. BANNISTER

*Central Electricity Generating Board
Berkeley, Glos, UK*

Determination of Particle Size and Strain in a Filled 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):

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

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

used, σ the angular range in 2θ over which the intensity distribution is appreciable and n is the order of a reflection), for two orders of the same reflection, would give a straight line whose slope and intercept would be proportional respectively to the mean square strain and the reciprocal of the apparent particle size in the corresponding direction. Sometimes one can take different sets of (W , σ) values, estimate the particle size and strain for each set and obtain an average value for these quantities through a least square fit.

The variance method was applied to the case of a copper-silicon-manganese alloy (containing about 6.7 at. % silicon, 1.3 at. % manganese, rest copper) possessing a face-centred-cubic structure. Copper $K\alpha$ radiation and a Siemens diffractometer with a scanning speed of $\frac{1}{8}^\circ \text{ min}^{-1}$ in 2θ were employed to record the diffraction patterns of the cold-worked and annealed filings. Variance measurements were carried out on the $\{111\}$ and $\{222\}$ profiles for one set of (W , σ) values. (The very weak cold-worked $\{400\}$ reflection precluded any accurate studies along the $\langle 100 \rangle$ direction.) The variance due only to deformation was obtained by subtracting the variance of the annealed sample from that of the cold-worked one [6].

The value of the apparent particle size and strain obtained from the linear plot were 98 Å and 0.0018 respectively. This can be compared with the values obtained for the effective domain

size and strain by Fourier analysis [7] and integral breadth [8] measurements on the same alloy for the same direction (viz. $\langle 111 \rangle$) which were 97 Å, 0.0025 and 163 Å, 0.0041 respectively. It can be seen that the values obtained by variance analysis are closer to those given by Fourier analysis than by integral breadth measurements.

Acknowledgement

The author is indebted to the Alexander von Humboldt Foundation for the award of a research fellowship.

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8 September 1969

R. VASUDEVAN
*Institut für Metallkunde
 Max-Planck-Institut für Metallforschung
 Stuttgart, Germany*

Etching of Fracture Surfaces

In the course of a fractographic investigation of fatigue in a 0.1% plain carbon steel it became necessary to devise a technique that would enable the principal features of the fracture surface to be related to the underlying microstructure.

In addition to taper-sectioning, an etching technique was adopted. The fracture was immersed in a solution of 2% nitric acid in ethyl alcohol for progressively increasing times and was then examined on a Cambridge Stereoscan scanning electron microscope. Before etching, a composite photograph of an area of the fracture was produced at a magnification of 2400 times and this same area was re-examined in detail after each etching step.

Pearlite colonies were revealed after 20 sec and their numbers increased with longer etching times as indicated in table I. Up to 100 sec, little

damage occurred to the fractographic features, but after 300 sec general attack was obvious, although isolated areas remained unetched.

Fig. 1a shows an enlarged area of the composite photograph before etching, and figs. 1b and c show the same area after etching times of 30 and 300 sec respectively. The pearlite colony in figs. 1b and c clearly lies in the surface of the

TABLE I

Etching time, sec	Number of pearlite colonies observed	Estimated % pearlite
As-fractured	0	0
10	0	0
20	2	less than 0.5
30	5	less than 0.5
50	18	1.5
70	34	3.0
100	48	4.0
300	166	12.0