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Scanning electron microscope selected area channelling patterns from 1 micron specimen areas

In the scanning electron microscope (SEM) selected-area channelling pattern (SACP) technique, the beam is rocked about a point on the specimen surface. This rocking action is achieved by the combined action of the scan coils and final lens as described by van Essen, Schulson and Donaghay [1]. In this condition, the smallest area which can be selected is determined by the spherical aberration of the final lens and the total included angle of rock. This limitation has been discussed from a theoretical standpoint by van Essen *et al* [2], and a careful series of practical measurements have been made by Booker and Stickler [3] to determine the variation in the selected area as a function of both angle of rock and specimen working distance.

To reduce the area below this limit set by aberrations, it is necessary to provide some form of correction signal to either the scan-wave forms or to the lens. The most practicable scheme is that suggested by van Essen [4] in which the final lens is dynamically focused. Since at any one time only one scan-ray orientation is passing through the lens, it is possible to vary the strength of the lens as a function of time so as to keep the crossover point of the scan rays exactly at the desired point. This is difficult to do successfully when using standard Cartesian scanning because the angle of scan changes from a maximum to a minimum at the line rate. The Cartesian scan is therefore replaced by a spiral scan with a frame repetition rate of approximately 5 sec and a circular frequency of 50 Hz. The angle of incidence in this mode increases linearly with time during each 5 sec frame scan period, and the necessary correction need only be supplied at the same rate. The system is set up by observing a piece of fine mesh grid (12 μm repeat) in the selected area mode and adjusting the correction amplitude until the distortion due to the spherical aberration of the lens is at a minimum. Once set the correction need not be adjusted again.

The other lenses in the system are set so as to produce a probe size of approximately 0.5 μm, a beam divergence of 3×10^{-3} radians, and a beam current of 3×10^{-9} A at the specimen. These values are chosen to give a pattern of adequate resolution together with sufficient beam current to provide the necessary signal to noise ratio to record the low level channelling contrast. For a source of given brightness no one of these figures can be improved without worsening the others. By changing the gun from a tungsten hair-pin source (of brightness 5×10^4 A cm^{-2} sr^{-1} at 20 kV) to a pointed filament (2×10^5 A cm^{-2} sr^{-1} at 20 kV) or a field emission source (10^7 A cm^{-2} sr^{-1} at 20 kV), it would be possible to increase the beam current, or reduce the probe size or beam divergence.

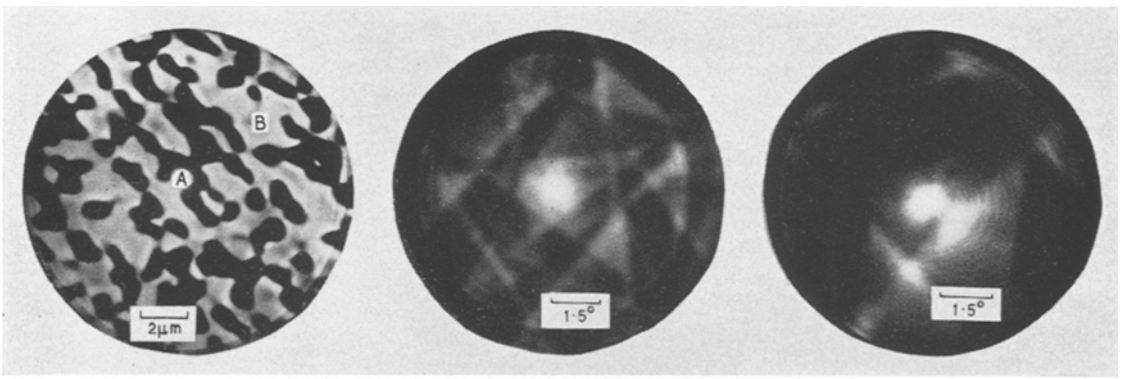
For a number of investigations at present underway in the Department, it was important for SACPs to be obtained with a total angle of rock of 9° and from areas only 1 μm across, a combination not hitherto achieved. In order to do this, it was decided (a) to work with the smallest selected-area size possible that could be obtained without corrections by working with the shortest practicable final-lens working distance, and then (b) to use spiral scan and apply dynamical focusing as described above.

It was found that a true working distance of 1 mm could be used as long as the specimen current was taken for the image signal, thereby avoiding the difficulty of collecting sufficient emitted electrons from a rather restricted geometrical configuration. From the results of Booker and Stickler [3] (see Fig. 1 of the previous paper in this journal), for a total rocking angle $2\phi = 9°$ and a true working distance $WD = 1$ mm, the selected-area size is $d = 3$ μm. This value was closely obtained in practice in the present work.

The spiral scan system and circuitry used to subsequently apply the correction were those described by van Essen [4]. However, special

precautions were taken to improve the stability, and accuracy of setting, of the electronics. Provision was also made so that the axis of the scan coils could be aligned with the axis of the final lens. This was important because the system only corrects for spherical aberration and so other aberrations need to be minimized. In this regard, care was also necessary with the initial cleaning of those portions of the SEM in the neighbourhood of the lens, limiting aperture, etc. If this was not done, additional aberrations arising from electrostatic fields associated with surface contamination occurred and these caused difficulties. When these modifications were made and precautions taken, the system proved straightforward to set up in practice, and it was found that SACPs could then be obtained with $2\phi = 9°$, and from selected areas 1 μm across or slightly less.

magnification micrograph, but with the image rotated through 180°, and then the magnification decreases. When a polycrystalline material is being examined and the grains exhibit surface topography, atomic number, crystallographic, etc. contrast, as with the present specimen, then the procedure is to choose a particular grain of interest using the out-of-focus SACP mode, move the specimen laterally so that the centre of the grain corresponds to the marked centre of the viewing screen, and then to progressively focus the final lens. As the micrograph magnification increases, the boundaries of the grain move outwards towards the edges of the viewing screen. At the focus position, the SACP occurs, with superimposed topographical, etc. contrast. If the boundaries have moved completely out of the field of view, then it is unambiguously demonstrated that this particular SACP is being



(1) (2) (3)

Figure 1 Lead-tin eutectic alloy specimen. Scanning electron micrograph.
Figure 2 Selected-area channelling pattern obtained from area B (2 μm across) of Fig. 1.
Figure 3 Selected-area channelling pattern obtained from area A (1 μm across) of Fig. 1.

A typical result demonstrating the operation of the system once set up is as follows. The specimen used was the lead-tin eutectic alloy shown in Fig. 1. The white areas are tin grains when viewed in the specimen current mode of operation. With the instrument operating in the SACP mode but with the final lens slightly defocused, a high magnification image of the specimen is formed. As the lens is progressively brought into focus, the magnification increases until the scanned rays are rocking about the specimen surface. The magnification reaches a maximum and the channelling pattern from the area selected is then visible on the screen. A further change in focus again gives rise to a high

generated entirely from material within the chosen grain.

Fig. 2 shows the channelling pattern obtained from the grain labelled B in Fig. 1. This grain is 2 μm in diameter. Fig. 3. shows the pattern from the grain labelled A in Fig. 1. This grain is 1 μm in diameter. In each case, the previously observed boundaries have moved outside the field of view, and the angular width of the pattern is 9 to 10°. The centre portion of each pattern is brighter than the outer edges because the scan lines are more closely spaced in the centre. However, the contrast is clearly visible on both patterns. The concentric circles of brightness variation are due to small instabilities in the

specimen current amplifier.

With further improvements in the design of the correction circuitry and in the setting-up procedure, it is believed that it will be possible to obtain adequate SACPs with $2\phi = 9°$ and from areas as small as 0.5 μm. For areas smaller than this, it will probably be necessary both to improve the final lens and also to use a brighter electron source, compared with the standard tungsten hair-pin filament used here, in order to obtain sufficient probe current to observe the channelling contrast.

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An aluminium—α-alumina composite

The recent availability of single-crystal α-alumina in filamentary form [1] has resulted in marked interest in the use of this materal as a reinforcing agent in metal-matrix composites [2-3]. Apart from having excellent mechanical properties (tensile strength $\sim$ 2000 MN/m^2 and modulus of $\sim 4.65 \times 10^5$ MN/m^2), these filaments have been shown to exhibit good stability in metal matrices up to temperatures of $\sim$ 1373 K [2].

The present programme was aimed at studying some of the mechanical characteristics of alumina filaments embedded in an aluminium matrix. For effective reinforcement, the length of fibre embedded in matrix material must exceed a critical value (l_c), given by the equation [4]:

$$\frac{l_c}{d} = \frac{\sigma_f}{2\tau_y} \qquad (1)$$

where d is the fibre diameter, σ_f the fracture stress of the fibre, and τ_y the shear stress of the fibre/matrix interface. Values of the critical aspect ratio (l_c/d) have been determined by casting aluminium around single filaments and performing draw-out tests similar to those described by Kelly and Tyson for metal wires embedded in various metal matrices [5-6].

Materials used in the present study were 0.51 mm diameter filaments of single crystal α-alumina, and high purity (99.999%) aluminium. The technique used to produce the composite specimens involved casting the aluminium at a temperature of 1073 K into a graphite mould containing the alumina filament. This technique, which is described in detail elsewhere [7], resulted in good filament alignment and interfacial bonding.

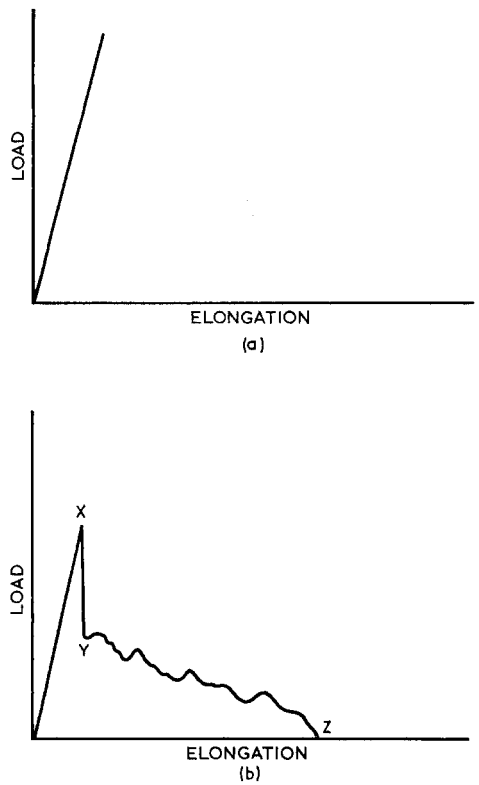

Figure 1 Typical load-extension plots for specimens exhibiting (a) fibre fracture, and (b) fibre pull-out.