

Short Communication

The determination of size and spacing of second-phase particles by scanning electron microscopy

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A critical evaluation of the properties of a dispersion-hardened system requires an accurate estimate of both the mean particle size and mean planar spacing of second-phase particles. The difficulties associated with making such measurements from transmission electron microscopy of thin films have been reported previously^{1,2}. Ashby and Ebeling³ have shown that under certain circumstances, extraction replicas make the determination of the true size distribution of second-phase particles relatively simple. The technique has been used successfully to characterize the dispersions in internally oxidized copper-silicon and copper-beryllium alloys³⁻⁵. The purpose of this note is to show that scanning electron microscopy is a useful and in some cases advantageous method of determining both the planar spacing and size distribution of oxide particles in internally oxidized copper-aluminum single crystals.

Experimentally, a copper-0.035 wt.% Al single crystal was internally oxidized by the Rhines Pack method⁶ at 1010°C. The resulting dispersion of aluminum oxide particles was examined by electron microscopy of thin films and extraction replicas as well as by scanning microscopy. In order to ensure that each method sampled roughly the same area in the crystal, the following procedure was used. A thin slice of material was cut parallel to the slip plane in the crystal by spark erosion. It was subsequently used to prepare a thin film for transmission electron microscopy. Direct carbon extraction replicas were obtained from one of the remaining two cut surfaces of the crystal⁵. The other face was chemically polished, slightly etched and examined in a Cambridge Stereoscan microscope. The thin

films and replicas were examined in a Philips EM300 microscope operating at 100 kV.

Micrographs of the dispersed phase were obtained from roughly the centre of the cross-section of the crystal using all three techniques. The particle size distribution was determined by scanning the original negative plates in an optical microscope and measuring the edge length (x) of the triangularly shaped alumina platelets. Figure 1 shows an example of a micrograph obtained by scanning microscopy. The contrast between the alumina particles and the copper matrix is very marked. This is due to the large difference in electronic properties between the two phases. The oxide particles become highly charged by the electron beam and produce a very bright image. However, the charging also reduces the clarity of the image as compared with extraction replica micrographs.

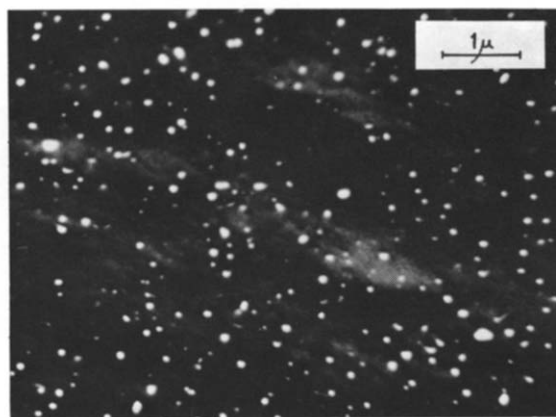


Fig. 1. Scanning electron micrograph of alumina particles in a copper matrix.

Ashby and Ebeling³ have shown that the size distribution of silica particles in copper approximates closely to a log-normal distribution. This was also found for the data obtained by all three methods in this investigation. Figure 2 shows the histogram of number of particles *versus* particle size for the scanning micrograph of Fig. 1. Superimposed on the histogram is a computer generated curve of a log-normal distribution using the geometric mean ($\bar{x}_g$) and geometric standard deviation ($\bar{\sigma}_g$) of the measured distribution.

A comparison of the particle size distributions as

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measured by the three techniques is shown in Fig. 3. It is a cumulative plot of the number of particles of size less than x against x on log-normal probability paper. Table 1 lists the geometric mean and standard deviation of the size distribution as

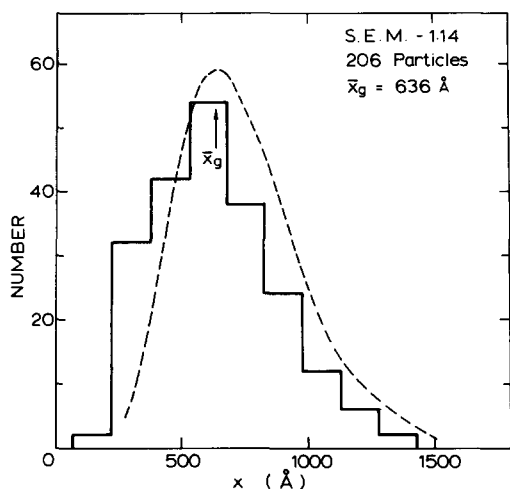


Fig. 2. Histogram of the alumina particle size distribution as measured from the scanning micrograph.

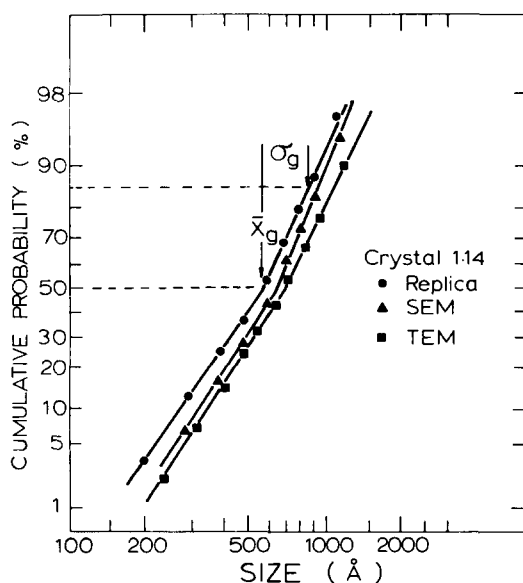


Fig. 3. Cumulative probability curves of the particle size distributions as measured by each of the three techniques.

obtained by each technique. The distribution measured from the extraction replicas has the lowest geometric mean while that from the thin films is 16% larger. This is expected since the individual particles are clearly defined in the replicas while in the thin films the edges of the particles are more diffuse and in many cases have strain contrast

TABLE 1

Values of the geometric mean and standard deviation of the particle size distribution and the mean planar particle spacing as measured by the three techniques

Method of examination	Geometric mean $\bar{X}_g$ (Å)	Standard deviation σ_g (Å)	Mean planar spacing $\bar{D}_s$ (Å)
Extraction replicas	570	285	6600
Scanning microscopy	636	274	6300
Thin films	664	356	—

effects associated with them. The geometric mean obtained from the scanning micrographs is approximately 10% larger than that from the replicas. While strain contrast effects are not present in this case, charging of the particles reduces the clarity of their image. The fact that the whole of the size distribution is progressively shifted to larger sizes as measurements are made from replicas, scanning micrographs and thin films, is a reflection of the increasing difficulty in defining the edges of the particles.

The three techniques of examining the dispersed phase were also applied to crystals oxidized under conditions which produce particle sizes in the range of from 50 to 350 Å. The small particles were readily observed in thin films through their strong strain contrast image. In comparison, extraction replicas suffered from the difficulties of extracting particles of less than 100 Å, as well as resolving them, when extracted, from among the background features of the replica itself. Resolving the small particles in the scanning microscope was not possible. Particles of less than approximately 150 Å were not observed, although the thin films indicated their presence. Thus the usefulness of scanning microscopy appears to be limited to dispersions containing oxide particles greater than approximately 200 Å.

With its limitations in mind, there are several advantages in using scanning microscopy which make it a particularly useful method. In the first place, it is a rapid technique requiring very little sample preparation. As in the case of replicas, measurements are made on a surface, avoiding particle overlap and strain contrast effects in the matrix. One advantage exclusive to this method is the ease of examining any area on the cross-section of a crystal. Thus particle size variations from the

surface to the centre of the crystal can be quickly determined and analysed. With replicas or thin films, different sections must be cut from the outside to the centre and separately examined. Similarly, the oxidation front of an incompletely oxidized specimen can be identified on the cross-section of the crystal. This situation is illustrated in Fig. 4. It shows a scanning micrograph of a Cu-0.035 wt.% Al crystal, internally oxidized at 1010°C for only 16 hours. The micrograph shows a sharp oxidation front with no resolvable oxide particles ahead of it. In this context, scanning microscopy is useful in obtaining quantitative data about the rate of internal oxidation.

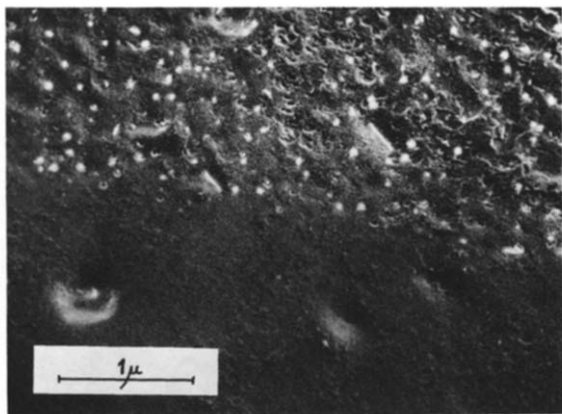


Fig. 4. Scanning micrograph showing the internal oxidation front in an incompletely oxidized crystal.

Apart from the size, the second major parameter needed to characterize the dispersion is the spacing of the particles. In testing the Orowan yield stress criterion, the mean planar spacing of the particles has been defined as the reciprocal of the square root of the number of particles per unit area of slip plane^{7,8}. Such measurements can be made by using either extraction replica or scanning microscope micrographs of surfaces cut parallel to the slip

plane. However, in calculating the spacing from scanning micrographs, care must be taken to ensure that the surface is observed perpendicularly, so that the projected image area corresponds to the actual area on the slip plane. As shown in Table 1, the calculated mean planar spacings ($\bar{D}_s$) from extraction replicas and scanning micrographs differ by only 5%.

In summary, the scanning electron microscope has been used to determine both the particle size distribution and the mean planar spacing of alumina particles in copper single crystals. A quantitative comparison of the technique with transmission microscopy of thin films and extraction replicas has shown it to be a useful and accurate method of determining dispersed phase parameters for particle sizes greater than approximately 200 Å. In such cases, the method has considerable advantages in ease of specimen preparation and examination of complete specimen cross-sections.

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