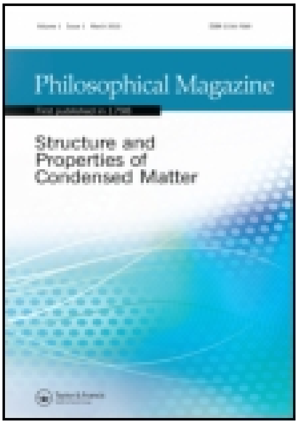

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The determination of dislocation densities in thin films

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CORRESPONDENCE

The Determination of Dislocation Densities in Thin Films

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I wish to describe (1) a new method of estimating the density of dislocations observed in a thin metal film by electron microscopy, and (2) an investigation of the sources of random error in estimates of the mean density corresponding to a particular treatment.

(1) Bailey and Hirsch (1960) have described a method of estimating the density of dislocations. They measure the total projected length R_p of dislocation line in a given area A on a typical micrograph. Then, on the assumption that the dislocation segments are randomly orientated with respect to the plane of the film, the dislocation density is $\rho = (4/\pi)R_p/At$, where t is the thickness of the film.

A disadvantage of this method is the labour of measuring R_p. However, an estimate of R_p may be obtained easily by making use of the results of Smith and Guttman (1953). They showed that if a set of random lines with a total length L is marked on A, and the number of intersections N which dislocations make with the grid lines is measured, then $R_p = \pi NA/2L$, giving $\rho = 2N/Lt$, provided N is large enough.

The two methods of finding R_p have been compared for 20 micrographs taken from thin films of a specimen of aluminium cold-rolled to about 9% reduction. The new method proved to be about ten to twenty times faster in measuring time alone. Five lines drawn in random directions on a plate taken at $\times 20\,000$ gave enough intersections (about 50), and it was not necessary to enlarge the micrographs, as it was for the direct measurement of R_p. The mean difference between the dislocation densities (of about $2\times10^9/\text{cm}^2$) estimated by the two methods was $\simeq 4{\cdot}1\times10^7/\text{cm}^2$, the standard deviation of the mean difference being $\simeq 3{\cdot}6\times10^7/\text{cm}^2$. Hence there was no significant difference between the two methods.

(2) As part of another programme, the new method has been used in a preliminary investigation of the sources of random error in determinations of dislocation density. Three specimens of aluminium were prepared at each of three levels of deformation, namely 18, 45 and 88% reduction, by cold rolling. A thin film was prepared from each specimen and two micrographs taken from each of two grains. The specimens were treated in three groups, all levels being represented in each group. In this way

systematic variation during the measurement was shown to be absent. The dislocation density was determined for each micrograph, and an analysis of variance made of these data. In what follows, references have been given to specific sections of Davies (1958) which describe the procedure. For each level of deformation the specimens, grains, and micrographs form a hierarchic classification with three sources of variation (§ 6.3). The additive property of variance (§ 6.11) was used in the separation and estimation of components of variance associated with specimens, grains, and micrographs. These estimates are given in the table. The variance of the mean dislocation density (§ 6.11) based

Level	Components of variance		
	Between specimens, S_s^2	Between grains, S_g^2	Between micrographs, S_m^2
18	0	0·87	0·68
45	0	6·03	2·14
88	1·07	0·17	1·23

on n_s specimens, n_g grains per specimen and n_m micrographs per grain is $S_s^2/n_s + S_g^2/n_s n_g + S_m^2/n_s n_g n_m$. Hence it is seen from this formula and the estimates in the table that at levels 18 and 45 the most efficient way to reduce the error in the mean is to take more than two grains per specimen ; two specimens and two micrographs per grain are enough. At level 88, however, the most efficient procedure is to take more specimens. The variance estimates are fully efficient, but since they are based on few observations these suggestions should be regarded as a guide to the design of further investigations.

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