Zone-Axis Pattern Maps for Graphite

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Abstract

Zone-axis pattern maps for graphite have been obtained using multiple-micrograph montages. Maps have been constructed with bend-contour micrographs and with Kossel–Möllenstedt patterns, both obtained at 100 kV. The two kinds of map are compared.

As is well known, bend-contour patterns (BCP) obtained by electron microscopy form a powerful tool for crystallographic studies (Steeds, Tatlock & Hampson, 1973; Steeds, Jones, Rackham & Shannon, 1976). Such patterns are particularly useful when special attention is paid to zone-axis orientations (zone-axis patterns or z.a.p.'s) and when such patterns are laid out on a stereographic projection to form a map.

In an earlier paper (Silva, Van Dun & Riquelme, 1976), BCP maps from titanium and mica single crystals were presented and attention was drawn to the usefulness of such maps, specifically to determine the specimen orientation, to set up a required orientation and to measure angles of tilt. Even when the BCP is distorted, these may be carried out accurately and directly in the image plane (Eades, Riquelme, Silva & Van Dun, 1974).

This paper presents an experimental BCP map for single-crystal graphite and compares such a map with a corresponding Kossel–Möllenstedt pattern (KMP) map.

Kossel–Möllenstedt patterns are diffraction patterns obtained in transmission electron microscopy when the illumination is brought to a focus in the specimen plane and when the convergence angle is large. They are therefore closely related to convergent-beam patterns (Steeds, 1979). In the latter, the convergence angle of the illumination is chosen so that the discs associated with the diffracted beams do not overlap. On the other hand, KMP's are formed when the convergence angle is much larger than the Bragg angles. Thus the discs are much larger than their separation and there is extensive overlap. KMP's are similar in appearance to Kikuchi patterns (e.g. Thomas, 1970), to which they are also closely related. In Kikuchi patterns the wide-angle source is formed by the specimen itself by inelastically scattered electrons. They therefore require a thick specimen (thin specimens produce only spot patterns and spot patterns are superimposed on the Kikuchi pattern even for moderately thick samples). In contrast, KMP's can be formed by elastic scattering alone and may be obtained from specimens of any thickness. Moreover, since the beam is focused to a probe, KMP's will normally be formed from smaller regions of the specimen than Kikuchi patterns. This will be an advantage in many applications.


The BCP's were obtained in a Philips EM 300 operated at 100 kV. The specimens were prepared by cleavage in air using adhesive tape. Although cleavage causes some buckling of the specimens so that suitably bent specimens can be obtained, because of the rigidity of the material and the high density of dislocations usually found on the basal plane, it was a laborious business to obtain foils from which BCP's could be obtained. Only orientations up to about $35^\circ$ from [0001] could be investigated.

The KMP's were obtained on another Philips EM 300, also operated at 100 kV, but fitted with STEM pole pieces. The specimens prepared for BCP work were also suitable for the KMP's. A rather wider angular range could be explored.

In both cases, as will be seen from Figs. 1 and 2, the map was built up from a large number of micrographs. A double-tilt holder was used in both cases. The maps have been indexed using Miller–Bravais indices.

To aid interpretation of the maps, in Fig. 3 a calculated contour map is shown. This is a computed stereographic projection of the crystal with the contours drawn to scale for 100 kV (Okamoto & Thomas, 1968).

The BCP map, as is generally the case, shows considerable distortion, in contrast to the KMP map. As expected, the maps have a number of common features. On the (1010) contour, lines due to 2g and 3g are clearly visible, whereas along the (1120) contour, only the 2g line (as well as g) can be seen. In general (as might be expected for such a material) the contours and zone-axis patterns have the simpler form that results
from weak, or kinematical, diffraction. There are conspicuous exceptions to this only at [0001], [1213], [2423] and [1101] where the complexity which arises from dynamical diffraction is apparent. These z.a.p.'s show the same symmetries in both patterns; this will not generally be the case. The [0001] z.a.p. has the diffraction group 6mm11 (Buxton, Eades, Steeds & Rackham, 1976) and both patterns have symmetry 6mm. The other three z.a.p.'s, [1213], [2423] and [1101], have diffraction group 2Rmm; each of them has, in both maps, symmetry 2mm if the pattern is examined close to the zone axis only, and has symmetry m if a wider field of view is used. Now the symmetry of the bend-contour pattern should be the bright-field symmetry tabulated by Buxton, Eades, Steeds & Rackham (1976) whereas the Kossel-Möllenstedt patterns should have the 'whole-pattern' symmetry (Eades, 1980).

The thirty one diffraction groups can be divided into three groups.

(a) 16 diffraction groups have the same symmetry for 'bright field' as for 'whole pattern' and also have the same symmetry for 'bright field' as for 'whole pattern' when the projection approximation is used (i.e. when

Fig. 1. Bend-contour map from graphite. The montage covers a 60° sector extending out to about 35° from [0001]. 100 kV.
the symmetry of the region close to the zone axis only, is used).

(b) Four diffraction groups have the same symmetry for ‘bright field’ and ‘whole pattern’ but have different symmetries in the projection approximation.

(c) The remaining 11 diffraction groups have different symmetries in ‘bright field’ and ‘whole pattern’ (though not necessarily in the projection approximation).

Since both $6m1\text{R}$ and $2Rmm_{\text{R}}$ are in group (a) the symmetries in both maps should be the same, as observed. However, for diffraction groups in categories (b) and (c), the symmetries of bend-contour z.a.p.’s and Kossel–Möllenstedt z.a.p.’s will be different.

It is also very striking that the appearance of the BC and KM z.a.p.’s are similar. This similarity goes beyond the similarity of symmetry, it includes the shapes of the features and their extent. The eye is very good at recognizing such features and this ability can be exploited. Thus, it is, for example, standard practice to identify a specimen by recognizing that a BC pattern is one that has been observed before.

However, it would be premature, on the basis of these

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Fig. 2. Kossel–Möllenstedt pattern map from graphite. The montage covers a 30° sector extending out to about 45° from [0001]. 100 kV.
results, to say whether similarity between BC and KM patterns is general and whether z.a.p.'s obtained by one technique could be used for reliable ‘finger printing’ in the other. It will be especially interesting to see whether these similarities in the ‘look’ of the pattern are preserved in those cases when the BC and KM patterns from the same zone axis have different symmetries.

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References
