ELECTRON-MICROSCOPIC STUDY OF ARTIFICIAL POLYCRYSTALLINE DIAMONDS–BALLAS AND CARBONADO

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Abstract

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ELECTRON-MICROSCOPIC STUDY OF ARTIFICIAL POLYCRYSTALLINE DIAMONDS—BALLAS AND CARBONADO

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Recently artificial polycrystalline diamonds—ballas and carbonado—have been created (^1). The present article gives the results of a study of their microstructure.

The technique for obtaining thin diamond films suitable for transmission electron microscopy is rather complicated; therefore there are almost no experimental data on defects in the crystal structure of monocrystalline diamonds (^2), and ballas and carbonado had not been studied at all. However, since diamond is nonplastic at room temperature, the procedure for preparing electron-microscopic specimens can be simplified.

Specimens of artificial ballas and carbonado were comminuted mechanically. The powder was deposited on grids coated with a carbon film; excess powder was removed by shaking. The study was carried out in an electron microscope at an accelerating voltage of 100 kV. A sufficient number of thin (i.e., electron-transparent) diamond fragments were found. Usually they had a wedge-like shape with well-defined cleavage steps (Figs. 1 and 4). Judging from the electron diffraction patterns, the cleavage planes are often close to crystallographic planes of the types {112}, {110}, and {111}. Figure 2 shows a crystal with thickness extinction contours. According to contrast theory, on passing from one contour to another the crystal thickness changes by t_e. For diamond, for the reflection {111} operating in this case, the extinction distance is $t_e = 476$ Å. Thus, it may be considered that in electron-microscopic studies under focused conditions the thickness of the diamond specimen should not exceed 2500–3000 Å. No fundamental difference has yet been found in the microstructure of single crystals obtained by comminuting ballas and carbonado; therefore there is no point in discussing the data separately.

In almost all crystallites, shapeless dark spots were observed (Figs. 1, 3, and 4). Tilting the specimen did not change their contrast. Imaging in the dark field likewise did not change the contrast. Consequently, it is due not to diffraction but to absorption of electrons. This cannot be connected with local thickening of the crystallites, since, as is evident from the photographs, the cleavage is usually fairly even, and abrupt changes in thickness occur at cleavage steps. By studying spots located at the edges of crystallites, it was possible to ascertain that they are not associated with adhesion to the surface of the specimen under investigation of finer particles. Hence we conclude: impurities are present in the diamond crystallites, distributed in the form of local clusters. The size of a cluster, as a rule, does not exceed 0.5 μ. In one case (Fig. 4), smaller inclusions of approximately identical diameter ($\sim$ 100 Å) were found. Their nature is unclear. It is quite possible, however, that these are micropores formed during coagulation of vacancies, analogous to those found in natural diamond (⁀2).

Fig. 1. Carbonado. Cleavage steps, inclusions. Diffraction: zone [112]

Fig. 2. Carbonado. Arrows indicate stacking faults. Diffraction: zone [110]

Fig. 3. Ballas. Inclusions. Diffraction: zone [112]

Fig. 4. Carbonado. Cleavage steps, small inclusions

In one of the diamond fragments a system of stacking faults lying in parallel planes was found (Fig. 2). Since their planes are inclined to the incident electron beam, the stacking faults produce periodic contrast (an alternation of dark and light bands parallel to the line of intersection of the sample surface with the plane of the stacking fault).

Kikuchi lines are present in most of the electron-diffraction patterns. As is known, two conditions are necessary for their appearance: (a) the crystal must be sufficiently thick; (b) there must be a high degree of perfection with respect to angular misorientations of the atomic planes. The latter can be estimated from the width of the lines. For both ballas and carbonado, the degree of misorientation lies in the interval $3 \cdot 10^{-4}$—$2 \cdot 10^{-3}$ rad. Thus, despite the fact that during growth impurities enter the diamond lattice, the distortions that arise in this process do not spread throughout the entire lattice but are localized near the inclusions. In addition, the presence of Kikuchi lines confirms the correctness of our method for obtaining thin specimens.

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