STUDY OF FILAMENTOUS CRYSTALS OF MAGNESIUM OXIDE

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Abstract

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STUDY OF FILAMENTOUS CRYSTALS OF MAGNESIUM OXIDE

Of great interest is the preparation and study of filamentous crystals of oxides with melting temperatures above 2500°. In particular, magnesium oxide belongs to such oxides.

Previously, filamentous single crystals of MgO were obtained by the method of a transport chemical reaction ([1]) upon heating MgO with carbon or tungsten, and also upon heating MgO in a hydrogen atmosphere. Analogous experiments were carried out ([2]) by heating magnesium oxide in molybdenum crucibles. By Hulse ([3]), filamentous crystals were obtained by compressing an MgO single crystal in the {100} direction.

We obtained filamentous single crystals of magnesium oxide by heating polycrystalline MgO in a cryptol furnace lined with tubes of magnesite, at a temperature of 1600–1700°.

Transport of magnesium oxide occurred as a result of transport reactions:

$$MgO_{(s)} + CO_{(g)} \underset{T_1}{\overset{T_2}{\rightleftarrows}} Mg_{(g)} + CO_{2(g)}.$$

$$2MgO_{(s)} + C_{(s)} \underset{T_1}{\overset{T_2}{\rightleftarrows}} 2Mg_{(g)} + CO_{2(g)}.$$

The reducing atmosphere in the working space of the furnace was due to diffusion of carbon through the furnace lining. As a substrate for crystal growth, a rod of sintered polycrystalline magnesium oxide was used, which was placed in the hot zone of the furnace.

Crystal growth is observed on the substrate and on the walls of the furnace in the temperature interval 1400–1600°. The length and shape of the crystals formed depend on the temperature at the growth point. At 1500–1600°, needle-like crystals grow up to 30 mm long with a thickness of up to 300 μ. The most intensive crystal growth occurs in the zone with a temperature of 1400–1500°.

Figure 1. Microphotographs and electron photographs of filamentary MgO crystals obtained by the transport-reaction method

Figure 1: Figure 1. Microphotographs and electron photographs of filamentary MgO crystals obtained by the transport-reaction method

In this temperature interval, filamentous crystals grow up to 15 mm long with a diameter from hundredths of a micron to 20—30 μ; under prolonged holding —growth time more than 2—3 hours—they form ribbed growth forms of MgO crystals.

The growth rate of filamentous crystals is 2—3 μ/sec. In those cases when the temperature in the reaction zone exceeded 1750° and the amount of MgO forming in the growth zone was large, a whitish-gray deposit, consisting of small crystals of magnesium oxide and particles of carbon, was deposited on the substrate. The color of the needle-like and filamentous crystals obtained was white or slightly yellow.

Microscopic studies of the crystals, carried out in transmitted light, showed that all the crystals investigated grow in the form of tetragonal prisms with square cross section. Crystals less than 3—4 μ thick have a very smooth surface (Fig. 1a). On crystals of greater thickness, growth planes are very often visible, which in some cases have the form of squares or rectangles. In Fig. 1a, e, microphotographs of filamentous crystals with growth planes are shown.

Fig. 1. Microphotographs and electron photographs of filamentary MgO crystals obtained by the transport-reaction method.

on the faces of the crystal (100). Often one or several offshoots form on the crystals, growing at an angle of approximately 90° to the main crystal (Fig. 1b). The offshoots have a tetrahedral shape, and their thickness is equal to that of the main crystal or somewhat smaller.

Electron-microscopic studies of crystals thinner than 2-3 μ were carried out with an ultra-high-voltage electron microscope with an accelerating voltage of 400 kV. The growth direction of the crystals, determined from selected-area electron diffraction patterns, in most cases coincides with the [100] direction and only in rare cases with [101]. In those cases where the crystals were about 1 μ thick, dark and light lines appeared in the electron diffraction patterns (Fig. 1d), arising from inelastic scattering of electrons (Kikuchi lines).

The surface of thin magnesium oxide crystals, as was also noted in optical microscopy, is smooth. The high energy of the electrons made it possible to examine crystals up to 1 μ thick in transmission. As in the case of beryllium oxide ([4]), bend extinction contours appeared in the images. The shape of the bend extinction contours depends on the position of the whisker crystal with respect to the electron beam. The principal shape of the contours is a rectangle (Fig. 1e), a rhombus, or a parallelogram. It should be noted, however, that bend extinction contours appear on magnesium oxide whisker crystals less often than on beryllium oxide whisker crystals. This is apparently explained by the small thickness of the basal ribbons or walls of the hexagonal prisms of beryllium oxide whisker single crystals, which to a considerable extent facilitates deformation of the crystals during specimen preparation.

Both in microscopic and in electron-microscopic examination, the presence of a capillary in the whisker crystals was not observed.

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