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1957

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Abstract

Full Text

Reports of the Academy of Sciences of the USSR
1957. Volume 115, No. 3

PHYSICAL CHEMISTRY

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INTERCRYSTALLITE INTERLAYERS IN GYPSUM

(Presented by Academician P. A. Rebinder on 29 IV 1957)

Most crystalline bodies used in technology are aggregates consisting of individual grains (crystallites) bonded to one another. Numerous investigations, a detailed critical review of which has been given by M. V. Klassen-Neklyudova and T. A. Kontorova ⁽¹⁾, show that the boundaries between grains play a very substantial role in a whole series of processes occurring in polycrystalline materials. The destruction of metals and alloys under certain conditions proceeds along grain boundaries (hot shortness, creep); in other cases (lower temperatures, other loading conditions) the boundaries manifest themselves, on the contrary, as the strongest regions of the metal. Intercrystallite boundaries change in a definite way the character of plastic deformation of crystals ⁽²⁾, promote the development of diffusion processes in metallic alloys ⁽³⁾, influence the electrical properties of bodies ⁽⁴⁾, etc. Therefore, special study of the structure and properties of intercrystallite boundaries is of very current importance.

Many investigators at present believe that grains are separated from one another by an intercrystallite interlayer ("transition zone") binding them, which has a many-atomic thickness and a structure different from that of the grains themselves ⁽⁵⁾.

Another point of view is that no special intermediate layer exists between grains, and that the lattices of neighboring crystallites adjoin one another directly, forming a two-dimensional, and not a three-dimensional, interface ⁽³⁾.

Thus, despite the large number of works (carried out, moreover, almost exclusively on metals), the question of the nature of the intercrystallite bond remains unresolved to this day. This situation is explained in part by the fact that, owing to the small thickness of the interlayers, it has not been possible to apply methods of X-ray structural analysis to their study. In addition, intercrystallite boundaries in nonmetallic substances have been little studied; there are almost no works performed on transparent crystals. An extremely numerous class of substances obtained by crystallization from solutions has also been studied in

this respect quite insufficiently. Meanwhile, many important building materials are polycrystals precisely of this type.

We therefore considered it expedient to undertake a study of intercrystallite boundaries in polycrystalline gypsum dihydrate—a substance satisfying the requirements indicated above and widely used in building practice. The material for the investigation was a fairly pure fine-grained natural gypsum from the Peshelan deposit of the Arzamas region.

The following experiments were carried out.

1. From polycrystalline specimens of natural gypsum stone, plane-parallel sections with a thickness from 0.03 to 0.1 mm were prepared. The -

examination of such thin sections with a polarizing microscope reveals, along grain boundaries, strips 0.002–0.01 mm wide, which do not change their color and remain dark when the microscope stage is rotated. Such strips are observed in the thinnest sections; they do not cease to be visible under any rotations and inclinations and therefore cannot be explained merely as the result of mutual overlap of the edges of neighboring grains. The presence of narrow dark strips indicates the absence of a regular structure of the crystallites near their boundaries, i.e., in other words, the presence in gypsum of special intercrystalline interlayers.

2. On the surfaces of thin sections subjected to 15-minute etching in HCl, shallow grooves up to 0.01 mm wide appear, readily observable under oblique illumination or with a binocular microscope and running along the grain boundaries; this indicates an increased solubility of the interlayers as compared with the grains.
3. If a thin section is immersed for half an hour in an alcoholic solution of methyl violet and then the upper layer is slightly ground off or the excess dye is washed away with alcohol, then along the grain boundaries a more intense and deeper coloration is preserved, forming a characteristic network of intergranular boundaries.
4. Observation under the microscope of the surface of a thin section heated in a special electric furnace mounted on the microscope stage reveals, when the temperature of the section is raised to 80–100°, darkening of the crystallites, beginning, as a rule, at their boundaries. These experiments give grounds for believing that the process of dehydration of dihydrate gypsum begins primarily in the intercrystalline interlayers.
5. When fracture surfaces of polycrystalline gypsum are examined under the microscope in reflected light, it is found that the rupture of individual grains occurs predominantly along the planes of most perfect cleavage {010}, which are readily recognized by their characteristic luster; in addition, the method of pressure figures was used to identify the fracture planes. However, in many places what is revealed are not smooth crystal faces but rough surfaces of irregular shape, apparently indicating the

presence in these places of fracture along the boundaries of crystalline grains.

6. Pieces of gypsum stone were comminuted by various methods at room temperature into a powder with a maximum fragment size close to the largest crystallite size in the given specimen. Careful and repeated examinations of the powder in crossed nicols did not reveal a single fragment that consisted of two, three, or more grains: all fragments proved to be single-crystalline. These experiments (as well as those described in item 5) testify to the low strength of the intercrystalline interlayers in gypsum as compared with the strength of the grains themselves. In this respect gypsum behaves differently than, for example, metals, in which fracture at room temperature occurs, as is known, by rupture or sliding through the body of the grain. The very same result was given by analogous experiments repeated with gypsum powder obtained at $+50^\circ$ and -20° ; under these conditions it was not possible to detect any influence of temperature on the strength of the interlayer. All these experiments indicate the existence in natural gypsum of special, comparatively weak and porous intercrystalline interlayers, which may represent places of incomplete intergrowth of grains with entrapment of air films between them. The characteristics of such interlayers undoubtedly determine to a great extent the mechanical strength of natural gypsum stone under various conditions (humidity, etc.).

When this work had already been completed, an article by E. E. Segalova, V. N. Izmailova, and P. A. Rebinder ⁽⁶⁾ appeared in print, in which the importance of contacts of intergrowth of crystallites in the formation of a spatial crystallization structure (porous skeleton) in the process of crystallization of dihydrate gypsum from a supersaturated solution during the setting of concentrated suspensions of hemihydrate gypsum is noted.

In connection with this, it seems of interest to study the transition of the conditions of coalescence in the genesis of natural gypsum, which is always much less porous, and of artificial gypsum stone.

Z. V. Sarazova and N. S. Igonina took part in carrying out the experiments.

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Received
27 X 1956

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Note: Figure translations are in progress. See original paper for figures.

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