

MAGNESIUM BORIDES PREPARED UNDER SUPERHIGH-PRESSURE CONDITIONS

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Translated from Doklady Akademii Nauk SSSR, Vol. 175, No. 6,
pp. 1266-1269, August, 1967
Original article submitted May 25, 1967

During the synthesis of cubic boron nitride under superhigh-pressure (40-70 kbar) conditions and at high temperatures (1500-2200°K) from the system components Mg-B-N, magnesium borides are obtained as by-products [1], and these were used as the objects for our investigations.

According to the chemical analyses done by M. V. Kharitonova, these borides have the compositions corresponding to the formulas for magnesium diboride and hexaboride (Table 3). Magnesium borides are generally synthesized from a mixture of metallic magnesium and boron at atmospheric pressure in a hydrogen medium; the products obtained by this method are in the form of dark-brown dispersed powders, the particle size of which does not exceed 0.005 mm [2].

The magnesium borides obtained by us under superhigh-pressure conditions are usually well crystallized. Magnesium diboride is in the form of goldish-yellow platelets, the size of which, depending on the experiment, varies between 0.1 and 0.8 mm. Magnesium hexaboride crystallizes in the form of light-green isometric grains, whose size ranges from 0.02-0.04 to 0.1 mm.

The magnesium diboride and hexaboride which were separated from the products of the synthesis were subjected to x-ray diffraction and microscopic analyses, and their chemical stability and microhardness were determined.

The results of the x-ray diffraction analysis of magnesium diboride by the Debye method are shown in Table 1.

The calculation of identity periods from the 210 and 211 lines showed that $a = 3.083 \text{ \AA}$ and $c = 3.521 \text{ \AA}$; according to data of [2], $a = 3.083 \text{ \AA}$ and $c = 3.521 \text{ \AA}$. Thus, within the accuracy of the measurements, our data for d , a , and c are in good

TABLE 1. X-Ray Diffraction Characteristics of Magnesium Diboride, Obtained from a Debye Pattern Using Co K α Radiation

Line No.	hkl	Our data		Data of [2], Fe K α	
		I	d, \AA^*	I	d, \AA^*
1	001	—	—	< 5	3.54
2	100	m.s	2.671	25	2.673
3	101	v.v.s	2.123	100	2.126
4	002	m	1.763	10	1.760
5	110	s	1.542	30	1.542
6	102	m.w	1.466	10	1.469
7	111	w	1.414	5	1.412
8	200	w	1.335	5	1.337
9	201	s	1.251	20	1.2488
10	112	s	1.158	25	1.1596
11	103	m.s	1.073	15	1.0738
12	202	v.w	1.062	5	1.0638
13	210	m.w	1.009	10	1.0099
14	211	v.s	0.970		

*Calculated by us from kX in \AA by multiplying by 1.00202. Notation: v.v.s., very very strong; v.s., very strong; s., strong; m.s., medium strong; m., medium, m.w., medium weak; w., weak; v.w., very weak.

agreement with those of [2]. Consequently, the structure of magnesium diboride prepared by the superhigh-pressure technique does not differ from that of magnesium diboride synthesized under normal conditions.

Results of the x-ray diffraction study of magnesium hexaboride (Table 2) showed that most of the d values are close to the corresponding values for magnesium boride (phase A) synthesized under atmospheric pressure [2]. It must be mentioned that several lines which were given for phase A [2] were not present on our Debye patterns, namely:

radiation
of [2], Fe K α

d, Å*

1.392
1.346
1.324
1.313
1.303
1.273
1.263
1.256
1.221
1.205
1.168
1.160
1.140
1.125
1.100
1.083
1.070
1.063
1.050
1.047
1.028
1.018

ed; m.w., medium

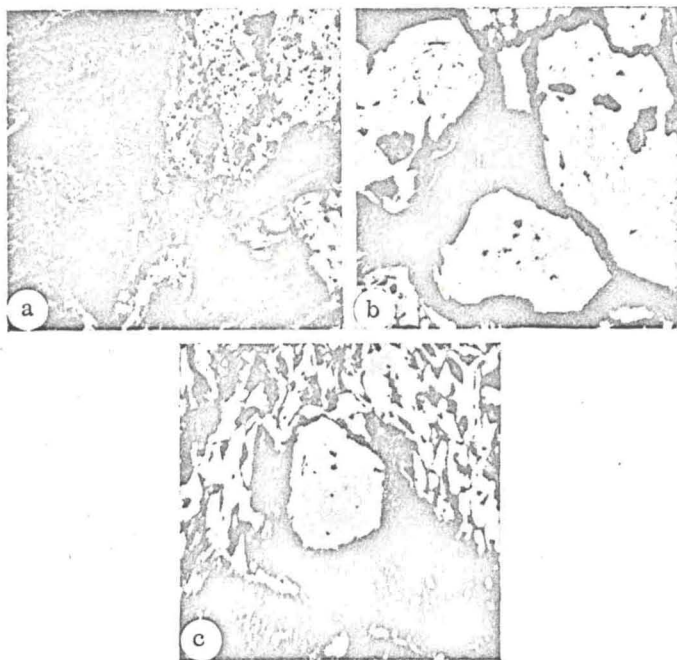


Fig. 1. Magnesium borides, at $\times 200$. a) Magnesium diboride, reflected and polarized light; b) magnesium hexaboride, reflected light; c) iso-morphous crystal of magnesium hexaboride and thin magnesium diboride platelets, reflected light.

medium at 2.26, medium at 2.01, medium at 1.86, very weak at 1.60, very weak at 1.346, weak at 1.263, weak at 1.063, strong at 1.050, and medium at 1.047 Å. The intensities of many of the lines do not agree. In particular, a number of lines of the A phase have higher relative intensities than those obtained from our Debye patterns. All these differences can, apparently, be explained not only by the peculiarities of crystallization under pressure, but also by the fact that the material which was named phase A by the authors of [2], and presumably considered by them to be magnesium hexaboride, is actually not single-phase.

Microscopic investigation of magnesium borides prepared by the superhigh-pressure technique was done by observation under a binocular, by the immersion method and by examination of polished sections; characteristic polished sections of magnesium borides are shown in Figs. 1a and 1b. In these polished sections the formation of magnesium as a result of the pyrolysis of magnesium diboride (Fig. 1c) was also observed.

Results of the microscopic investigation and the determination of some properties of magnesium

borides prepared under superhigh-pressure conditions and at high temperatures are shown in Table 3.

Thus, the present work showed that under superhigh-pressure conditions favorable conditions are created for the synthesis of well-crystallized magnesium borides of stoichiometric composition. It seems expedient to try to synthesize other magnesium borides, which, as is known, cannot be prepared in pure form by the conventional method [2, 3], in order to define more precisely their chemical composition, crystal structure, and properties.

LITERATURE CITED

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