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Boron Modifications Produced in an Induction-Coupled Argon Plasma

Abstract. *Most of the small particles (50 to 100 micrometers in diameter) of microcrystalline β-rhombohedral boron that quickly transit an argon plasma maintained within a radio-frequency induction-coupled torch emerge as better crystallized spheroids of the same crystalline form and nearly the same size as the starting material. A few crystals of each of four distinctive, well-faceted habits are formed along with the general product. Three of these types are monocrystals of the β-rhombohedral polymorph, of the tetragonal-III modification, and of an unreported cubic form of boron. Specimens of the fourth type are polycrystals of another unreported form of boron, apparently consisting of many hexagonal platelets stacked in an imprecise fashion.*

Several different crystalline modifications of boron are recognized (*1–4*), but the structures of only three of these have been determined (*1, 3*). The large number of forms of the element and of structurally similar crystal species rich in boron (*5, 6*) has given rise to numerous discussions of the modes of formation of the structural variants, the nature of their bonding, and the ranges of their thermodynamic stabilities (*6*).

Many of these problems persist because it has been extremely difficult to prepare crystals of known composition which are of a sufficient size (100 μm in diameter) to allow the needed structure determinations to be made. Generally, the preparation of boron specimens has involved contact with other, supposedly inert, solids at high temperatures (*1–4, 6*), with the consequent risk of contamination and the formation of an ambiguous product.

In order to avoid such contamination during crystallization we dropped finely powdered (50 to 100 μm in diameter) polycrystalline β-rhombohedral boron through an argon plasma maintained within an induction-coupled radio-frequency torch (*7*). We used relatively pure starting material of widely varying isotopic content without significant effect on the results. By proper adjustment of the operating conditions of the torch, a large proportion of particles emerging from the plasma could be made spheroidal with some small flattened faces, but never truly spherical. This is in contrast to the commonly produced shape for oxides and metals after passage through such a plasma (*8*). Because the size range of the spheroids corresponded closely to that of the feed material and because vaporization appeared insufficient to produce the quantity of product obtained (a major fraction of the input sample), we believe that the spheroids were formed directly from individual feed particles by means of a liquid or solid-state conversion. X-ray diffraction photographs usually indicated one or at most a few β-rhombohedral crystallites per spheroid.

The overall purity of the product was improved by passage through the plasma, some of the more volatile foreign atoms having been removed. X-ray diffraction patterns from powder samples of the product material indicated its improved crystallinity and a predominance of the β-rhombohedral polymorph. However, a few faint lines foreign to that pattern were also present.

Careful microscopic examination revealed a small number of beautifully faceted crystals of four distinctive habits, each approximately 50 μm in diameter, within the bulk of the product (Fig. 1). X-ray diffraction photographs showed these specimens to be very well-ordered internally. When all of these distinctive specimens had been handpicked from a typical portion of product, the remainder gave a typical powder pattern for β-rhombohedral boron without extraneous lines.

We determined the densities of these well-formed crystals with a density-gradient column, using specimens of varied, but known, isotope ratios (assuming that no change occurred as a result of passage through the plasma). The density, adjusted to the normal isotope ratio of boron (*9*), was 2.367 ± 0.002 g cm^{-3} for each type of crystal.

Single-crystal x-ray diffraction techniques indicated that crystals of type (*a*) are simple tetragonal with lattice parameters $a = 10.061 \pm 0.005$ Å, $c = 14.210 \pm 0.005$ Å. The axial ratio obtained from optical observations ($c/a = 1.401 \pm 0.001$) is in good agreement with these values. Systematic absences in the diffraction patterns of these crystals limit their possible space groups to $P4_12_12$ (D_4^4) or $P4_32_12$ (D_4^8). The lattice parameters approximate those for a structurally unelucidated boron modification previously prepared only in the microcrystalline state (*2*); a powder pattern from our sample is in substantial agreement with that of this modification. The lattice type and parameters are also similar to those for α-AlB_{12} (*10*) and a form of SiB_6 (*11*) whose structures are unknown, although

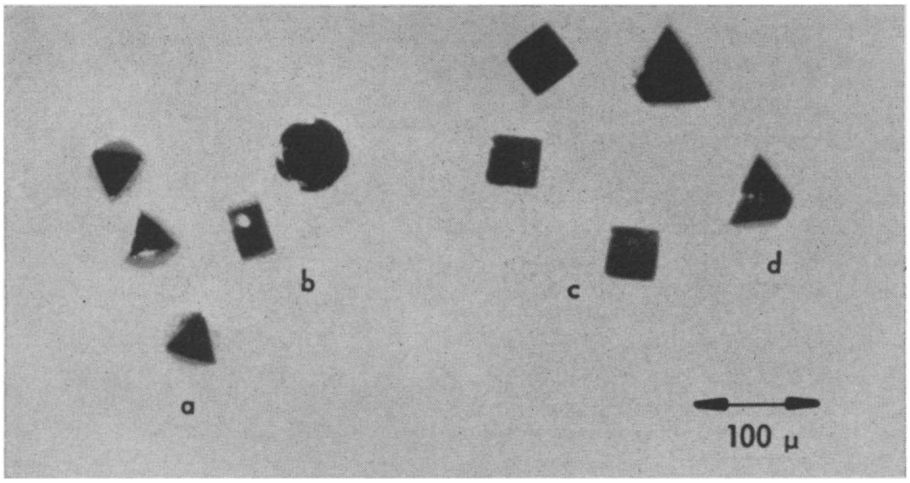

Fig. 1. Distinctive boron crystals produced in the plasma torch. Four habits are: *a*, slightly elongated octahedrons; *b*, hexagonal and dodecagonal right prisms (with some planar disparities on the lateral faces); *c*, square right prisms, some nearly perfect cubes and some slightly flattened (with small bevels on the edges also occasionally present); and *d*, truncated tetrahedrons (with some smaller faces evident).

single crystals have been reported for both compounds. In combination with the density of crystals of type (*a*), their lattice parameters indicate 190 boron atoms in the unit cell. At very high magnification and under critical lighting conditions, the triangular faces of these crystals are seen to be slightly raised toward smaller flat hexagons in their centers.

Crystals of habit (*c*) are of an unreported cubic form and have a lattice parameter $a = 23.472 \pm 0.006$ Å and space group *Pn*3*n*. The density and lattice parameters indicate 1708 atoms in the unit cell. Slightly raised central octagons are present on most square faces of these specimens.

The similarity of lattice parameters for this crystal with those for the face-centered cubic crystal YB_{66} (*12*) led us to check carefully the *hkl* x-ray diffraction intensities from a monocrystalline specimen for indications of structural similarity. Although many allowed reflections are nonsystematically absent, numerous reflections forbidden by face-centering are present in our data. However, these distinguishing reflections tend to be so much weaker than the "all-even" and "all-odd" reflections that they would be difficult to discern with certainty in an experimental powder diffraction pattern. Eick reported (*13*) that a primitive cell was required to index completely the lines of a diffraction pattern from a powdered sample of a compound PuB_{100}. Our calculated powder pattern (Table 1) is in better agreement with his experimental powder pattern (*14*) for PuB_{100} than with either the experimental YB_{66} pattern (*15*) or the "B_{66}" pattern derived from it by formal removal of the Y atoms. Therefore, we believe that there are two distinctive cubic frameworks for boron with large, nearly identical, lattice translations.

Crystals of type (*b*) superficially appear to be monocrystalline. However, steps and reentrant angles can be seen on their lateral faces; sometimes their opposite basal faces are not strictly parallel. X-ray diffraction patterns indicate that they are multicrystalline. The experimental powder pattern (Table 2) (obtained from several specimens ground between diamonds) is not reconcilable with any published pattern for any form of boron or boride, although it is compatible with hexagonal cell parameters of $a = 9.755$ Å and $c = 10.016$ Å.

Crystals of type (*d*) are of the β-rhombohedral form with typical lattice parameters (*3*) and are rare.

We believe that these distinctive crystals are essentially boron modifications because they do not appear to contain foreign atoms in the same concentration as the hectoborides (*12–14*, *16*). Scans upon several specimens of each variety of crystal with an electron beam microprobe instrument revealed no peculiar concentration of impurity atoms relative to the spheroidal particles from the same product. Barely detectable amounts (0.01 to 0.02 atom percent) of several heavier elements were present in each case, in general agreement with spectroscopic analyses for the bulk of the product material.

This does not rule out the importance of foreign atoms in establishing crystallization nuclei, however. Fewer of these distinctive crystals formed in runs in which starting materials of the highest purity were used, although there was no strict correlation between boron purity and the number of crystals formed. This hypothesis gained stronger support when we removed all the well-shaped crystals from one batch of product and repeatedly passed the remaining

Table 1. Powder x-ray diffraction pattern for cubic boron calculated from single-crystal intensity data; *d* represents interplanar spacing; *I* represents the x-ray diffraction intensity of the given set of atomic planes relative to the strongest intensity, I_0 taken as 100. The tabulated intensities are limited to $I/I_0 \geqq 1$ percent.

h,k,l	*d*(Å)	I/I_0
0,0,2	11.736	100
1,1,2	9.582	1
0,2,2	8.299	9
0,1,3	7.423	1
2,2,2	6.776	51
0,0,4	5.868	4
0,2,4	5.248	3
2,3,3	5.004	1
2,2,4	4.791	12
0,4,4	4.149	7.5
0,3,5	4.025	1
1,3,5	3.967	7
2,4,4 0,0,6	3.912	48
2,2,6	3.538	2
4,4,4	3.388	1
0,4,6	3.255	1
3,4,6	3.005	1
0,2,8	2.846	1
1,5,7	2.710	2
2,6,6	2.692	1
3,5,7	2.576	4
2,4,8	2.561	4
4,6,6	2.502	6
1,3,9	2.461	4
4,4,8	2.396	5
0,6,8 0,0,10	2.347	14
2,6,8	2.301	1
1,5,9	2.269	2
6,6,6	2.258	2
0,0,12	1.956	1
2,2,12 4,6,10	1.904	1
0,4,12	1.856	1
6,6,10	1.790	1
8,8,8	1.694	2
0,0,14	1.677	3
0,10,10 6,8,10	1.660	1
8,8,10	1.554	1
0,8,14 0,2,16	1.456	1
2,2,16 2,8,14	1.445	1
8,8,12 0,4,16	1.423	1
5,9,13	1.415	1
4,8,14	1.413	1
1,11,13	1.376	1
0,6,16	1.374	1
3,11,13 5,7,15 7,9,13 1,3,17	1.357	4
10,10,10	1.355	2
4,12,12	1.346	1
8,8,14 6,12,12	1.304	2
2,2,18	1.288	1

Table 2. Powder x-ray diffraction pattern for plasma-produced boron crystals of hexagonal platelet habit, with CuKα radiation; s=strong intensity; m=medium; w=weak; vw=very weak; vvw=very, very weak.

d(Å)	I	d(Å)	I
4.1990	s	1.8201	w
3.7600	w	1.6662	vw
3.3684	s	1.5398	w
2.5230	vw	1.3745	w
2.4626	vw	1.2526	vw
2.2899	vw	1.1959	vw
2.2382	vvw	1.1752	vvw
2.1247	vvw	0.8507	vvw
2.0229	m	0.8489	vvw
1.8639	vvw		

particles through the plasma, taking care not to contaminate the sample while handling. By this treatment the typical spheroidal particle became more spherical through a rounding of faces and an increased beveling of edges. However, no more of the distinctively shaped crystals formed. The powder pattern of the bulk product material contained no lines extraneous to the β-rhombohedral pattern after the reprocessing.

Whether the plasma is responsible for other effects in addition to supplying heat, for example, electromagnetic influences, we do not know. Certainly the thermal gradient within the plasma is immense, and the quench of particles falling from the plasma is severe because of their small heat capacity. Environments experienced by different particles falling through the same plasma may be quite varied. We have obtained all of the distinctive habits from product samples on occasion. More generally, one or two types were considerably more numerous than the others. We have discovered no underlying cause for the variation, but it seems to depend on the sample, repeated runs with any given sample being reproducible. With our instrument the particle size must be less than roughly 150 μm in diameter in order for decisive thermal effects to occur. Variation in instrument power, plasmoid volume, or other operating conditions might conceivably change this limitation.

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Cytotoxic Effects of Leukocytes Triggered by Complement Bound to Target Cells

Abstract. *Chromium-51-labeled chicken erythrocytes (E), treated with rabbit anti-Forssman antibody (A) and the first four (C1–4) or the first seven (C1–7) components of human complement (C), released isotope upon exposure to human leukocytes. Isotope release from EAC1–7 cells proceeded more rapidly and was more extensive than that from EAC1–3 cells. Lysis of these cells was suppressed by pretreatment of leukocytes with antimycin A. Monocyte-enriched leukocyte preparations affected both types of target cell-complement intermediates, whereas purified lymphocytes lysed EAC1–7 cells but not EAC1–3 cells.*

Cell-mediated cytotoxic reactions are operative in delayed hypersensitivity, homograft rejection, and in certain autoimmune diseases. Cytolytic effects of purified lymphocytes may be initiated in vitro by mixing lymphocytes from (i) sensitized donors with antigen-bearing target cells (*1*), (ii) normal donors with target cells in the presence of humoral antibodies to target cell antigens (*2*), and (iii) normal donors with target cells in the presence of lymphocyte stimulants such as phytohemagglutinin (*3*). Phagocytosis does not appear to play a role in these reactions. Similarly, macrophages from sensitized donors (*4*) or monocytes in combination with antibody (*5*) may damage in vitro a variety of target cells by a nonphagocytic mechanism.

Although complement (C) has been reported to potentiate the cytotoxic action of sensitized lymphocytes in a few cases (*6*), it is generally assumed that C is not essential for cell-mediated cytotoxic reactions (*1*). The present experiments were performed to ascertain whether complement, bound to target cells, renders these cells susceptible to cell-mediated lysis.

Chicken erythrocytes were used as target cells (*7*) which were sensitized with four hemolysin units (*8*) of a rabbit antiserum to boiled sheep stromata (*9*). The antibody was primarily of the 19*S* variety and readily cross-reacted with chicken erythrocytes. Intermediate complexes consisting of chicken erythrocytes (E), rabbit antibody (A), and human complement components 1–7 (C1–C7) were prepared essentially as in previous studies in which sheep erythrocytes were utilized. We prepared EAC1,4 with mac-