stand with small amounts of barium hydroxide or of tetraethylammonium hydroxide, or with soft-glass beads; in every case, the refractive index decreased to the value of 1.4779, cited above, and then remained constant for at least three weeks.

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X-Ray Powder Patterns of Boron Coated Mo and W Filaments

BY ST. V. NARAY SZABO AND CHARLES W. TOBIAS[1]

In 1945 we conducted a series of experiments on the preparation of pure crystalline boron. Our object was to obtain boron in such a form that X-ray studies concerning the crystallographic structure would be possible. After the beginning of World War II, scientific information, especially from the United States, was unobtainable. This is why important papers, such as the extensive study of Laubengayer, *et al.*,[2] were unknown to us. In most respects the above-mentioned authors attained more significant results than we did. Nevertheless, the techniques we used and the data obtained might eventually be of some use in further studies.

1. Preparation of Pure Boron.—A modification of van Arkel's method for thermal dissociation of boron tribromide[3,4] seemed to us the most promising. The regulation of boron tribromide vapor stream in van Arkel's original apparatus (Fig. 1) was effected by separate temperature control of two boron tribromide containers, making it difficult to attain a steady state. Also the quantity of boron tribromide necessary to fill such an apparatus would have been too large. Having only a few grams of boron tribromide at our disposal, we used a much smaller cylinder, 3 cm. in diameter and 4 cm. high, and only one boron tribromide container was connected to it. The apparatus used for the deposition of pure boron is shown in Fig. 2.

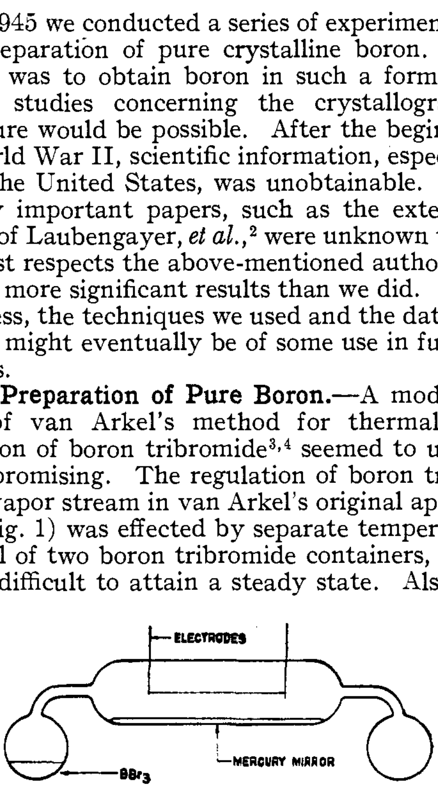

Fig. 1.

Boron tribromide was prepared by using Meyer and Zappner's modified Moissan method.[5] After purification by distillation the boron tribromide was sealed in small glass capsules and stored, when not used immediately. In preparation for the thermal decomposition, the apparatus already containing the boron tribromide was cooled under −50°, flushed with hydrogen and then evacuated to 1–1.5 mm. By controlling the temperature of the boron tribromide container, the density of boron tribromide vapor in the reaction space was fixed by the vapor pressure of boron tribromide at the given liquid temperature. The bromine liberated at the filament was instantaneously bound by the mercury mirror at the bottom of the cylinder. To keep the mercury surface fresh, small

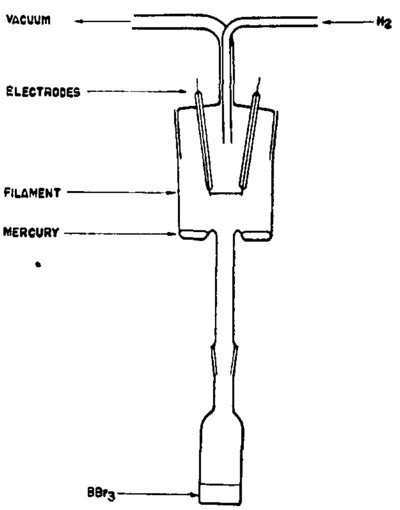

Fig. 2.

TABLE I

POWDER PATTERN OF THE "GRAPHITIC" TYPE BORON

B on W filament		B on Mo filament Sin² θ		Calculated	Miller's index
	I_{est}		I_{est}		
0.0326	7	0.0325	7	0.0324	200
.0362	9	.0356	7	.0360	002
408	3	412	4	414	201
445	3	440	3	441	102
732	2	734	2	729, 738	300, 221
888	8	880	6	891	311
0.1020	8	0.1020	10	0.1008	222
1100	2	1120	2	1089, 1134	302, 203
1210	2	1205	2	1215	213
1310	7	1320	9	1296	400
1462	2	1470	2	1458, 1467	330, 223, 411
1650	6	1620	4	1620	420
?		1780	2	1764	204
1980	3	1995	3	1980	422
2160	2	2150	2	2169	304
?		2400	1	2412	115
2550	1	2540	2	2574	205
2630	6	2620	5	2592, 2655	440, 215
2780	2	2780	2	2817	414
2850	1	2850	1	2835	433
?		2930	4	2916	513
3110	4	3080	2	3087	611
3260	4	3260	4	3276	006
3340	3	3350	4	3357	612
3550	3	3560	4	3546	514, 405

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[2] A. H. Laubengayer, D. T. Hurd, A. E. Newkirk and J. L. Hoard, THIS JOURNAL, **65**, 1924 (1943).

[3] v. Arkel, *Metallwirtschaft*, **XIII**, 23, 29 (1934).

[4] v. Arkel, "Reine Metalle," Springer Verlag, 1939.

[5] Gmelin-Kraut, "Handbuch der anorganischen Chemie."

TABLE II

POWDER PATTERN OF THE "CRYSTALLINE" TYPE BORON

B on W filament		B on Mo filament		Calculated	Miller's index
$\sin^2\theta$	I_{est}	$\sin^2\theta$	I_{est}	$\sin^2\theta$	
0.0190	2	0.0188	2	..	
0217	2	0218	1	0.220	
233	8	232	8	230	111
260	8	257	7	260	002
280	10	280	8	285	201
301	3	310	2	315	102
442	7	440	5	455	211
482	2	483	2	480	202
508	3	506	1	495	300
585	3	585	1	585	003
715	6	715	6	715	310
800	3	805	2	805	203
880	9	876	9	880	400
972	2	?		975	213
0.1040	8	0.1020	10	0.1040	132, 004
1110	3	1110	3	1110	231
1200	2	1205	2	1205	104
1310	4	1310	1	1305	130, 232
1416	3	?		1415	142
1444	7	1450	4	1440	501
1600	4	1600	2	1625	005
1910	6	1910	5	1920	404
1985	2	1995	4	1980	600
2150	2	?		2125	250, 243
2330	2	2300	1	2340	135, 006
2490	5	2480	5	2505	116, 405
2580	5	2610	3	2580	244
2800	3	2800	3	2780	145
2900	6	2900	5	2900	442
3130	4	3120	3	3110	335
3250	5	3260	6	3225	443

pieces of steel were continuously circulated on the surface by means of an outer magnetic field. This method enabled us to carry out experiments with extremely small quantities of liquid boron tribromide. Constant stream of boron tribromide vapor was secured by the sole control of liquid boron tribromide temperature. Electrodes were made of Pt, and Mo and W filaments of different thicknesses were used alternatively. Deposits which formed a dense cylindrical layer around the W or Mo core were obtained. In no case could the deposit be stripped from the filament by simple means.

On both W and Mo two virtually different deposits resulted depending on the temperature of the filament during the process. At lower temperatures, 1000–1300° "graphitic," grayish, virtually amorphous deposits were obtained, while around 1500–1600° dark gray crystalline surfaces resulted. Single crystal growth was observed in a few cases, but the specimens were too small, 0.2–0.3 mm., for further investigations. (Apparently the reduction of boron tribromide with hydrogen is more favorable for the growth of single crystals.) The diameter of boron coated Mo and W filaments after thirty to sixty minutes of decomposition varied between 400–500 μ, while the core diameter was 50–100 μ. We were not able to check the purity of our samples thoroughly, however there is good reason to believe that the purity of our samples was not inferior to those of other investigators. Specific density measurements averaged 2.33 (possible error ±0.02). Electrical conductivity and general chemical behavior were qualitatively investigated. Our findings substantially agree with the data of Laubengayer, *et al.*

TABLE III

POWDER PATTERN OF THE "AMORPHOUS" (SCHUCHARD) BORON

$\sin^2\theta$	I_{est}
0.0279	4
.0340	10
422	4
500	1
575	6
715	2
945	2
0.1170	3
1460	2
2420	1
2860	2
3130	1

2. X-Ray Powder Diffraction Pattern.—All boron collected on the W and Mo filaments was investigated in a 5-cm. radius cylindrical camera with Cu Kα-ray. We found it unnecessary to eliminate the W or Mo filaments since X-ray patterns of W and Mo could easily be singled out. As mentioned previously, we obtained two types of deposits on both Mo and W. These gave different X-ray patterns; however, the same type of deposit on the two filament materials gave identical ones, so that there was no disturbing effect due to any alloy formation between boron and the filament material.

The reflections of the "graphitic" type of boron coating could be indexed using a tetragonal elementary cell (a = 8.57 Å., c = 8.13 Å., 78 atoms/cell (Table I).

The "crystalline" deposit gave a pattern which suggested a hexagonal cell (a = 11.98 Å., c = 9.54 Å., 180 atoms/cell) (Table II).

X-Ray patterns were taken from the "amorphous" boron which was used for the preparation of boron tribromide (supplied by the Schuchard Ltd., Germany); 12 line pairs were observed (Table III).

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Interpretation of the Parachor

BY M. S. TELANG

Sugden's parachor P is based on Macleod's empirical equation[1]

$$\gamma = C(D - d)^4 \qquad (1)$$

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[1] D. B. Macleod, *Trans. Faraday Soc.*, **19**, 38 (1923).