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Chemistry

MASTER

CRYSTALLIZATION AND PURIFICATION
OF BORON METAL POWDER

P. E. Trent

UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

operated for the ATOMIC ENERGY COMMISSION under U. S. GOVERNMENT Contract W-7405 eng 26

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ABSTRACT

Two methods have been found that can be used to transform impure, low-density amorphous boron to a higher-purity, higher-density crystalline boron metal powder. In the first method, amorphous boron metal powder was placed in HBR-grade boron nitride containers and heated to 2,000 - 2,150° C in an inert-atmosphere tungsten susceptor furnace. In the second method, the boron metal powder was passed through a sheath-stabilized helium plasma flame onto a water-cooled target to produce an essentially massive body. Amorphous normal boron produced by the reduction of the oxide (B_2O_3) by magnesium metal was crystallized and purified to greater than 99 weight percent boron by both of these methods. Electrolytic-cell-produced amorphous boron-10 metal powder was crystallized and purified to greater than 97 weight percent boron by both of these methods.

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SUMMARY

In order to maximize boron densities in compacted boron parts, it was necessary to use crystalline boron of at least 98 percent boron content. Two methods have been employed to produce a high-purity, crystalline normal boron powder from readily available amorphous metal powder. In the first method, amorphous boron with a boron content of as low as 89.95 percent, was placed in boron nitride containers and subjected to temperatures as high as 2,200° C in a tungsten-suscepted induction furnace using an inert (argon) atmosphere. The second experimental procedure passed the amorphous boron powder through a helium plasma flame onto a water-cooled target to produce an essentially massive metal boule. The amorphous boron metal was crystallized and purified to a greater than 99 weight percent boron by both of these methods.

Similar attempts were then made to produce high-purity product from boron-10 powder (electrolytic cell product) which contained such high-boiling-point impurities as nickel, copper, iron, and chromium in contrast to the low-boiling-point impurities in normal magnesium-reduced amorphous boron powder. The electrolytic cell enriched boron metal powder was converted by the two previously mentioned methods to crystalline boron metal of 95 - 98 weight percent pure enriched boron. It was demonstrated that the electrolytic-cell material with the higher-boiling-point impurities could not be processed by these methods to the high purity level that was obtained with the amorphous normal boron produced commercially by magnesium reductions.

Normal anhydrous boron oxide (B_2O_3) was reduced by thermite bomb reduction using magnesium powder and lithium metal to produce boron metal purities of 88 percent. This elemental powder produced by the magnesium reduction was purified and crystallized to 99.3 percent boron content. The boron metal powder produced by lithium reduction was ~ 83 percent boron. This powder, however, could not be further purified by thermal means due to the violent decomposition at 200 - 300° C.

INTRODUCTION

The Atomic Energy Commission, through its Oak Ridge Operations Office, has maintained a continuing interest in the boron field with respect to producing powdered metallic boron, enriched in B-10, of a high purity and crystalline structure. The Y-12 Plant initiated a series of studies to become familiar with new and existing methods for the production of elemental boron and develop methods to crystallize and purify the normal-boron metal powders and enriched-metal powders that are commercially available.

Elemental boron can be prepared most easily by using the oxide, carbide, or halide in several processes. Briefly, these methods include: (1) the electrolysis of a fused salt using boric anhydride to produce the amorphous product, and (2) the electrolysis by salt anode transfer using boron carbide or potassium fluoborate to likewise produce an amorphous product. An amorphous material may also be made from the direct reduction of boric oxide with magnesium, calcium, potassium, or possibly beryllium metal. Boron trichloride, trifluoride, or tribromide may be reduced on hot wires or rods to produce a crystalline product where the use of high temperatures increases the crystalline density of the product. More recently, a chemical plant has been operated that employs a chemical exchange process to separate the boron-10 and boron-11 isotopes. The boron-10 compound has then been converted to an amorphous boron-10 metal powder by using the electrolytic cell process.

A common characteristic of these processes is that they produce amorphous or semi-crystalline boron metal powders having an undesirably low boron content and relatively little crystalline structure. Truly crystalline, high-density, normal-boron or boron-10 powders have been obtainable only in small quantities and at high cost.

EXPERIMENTAL STUDIES

INITIAL EXPERIMENTS WITH AMORPHOUS NORMAL BORON METAL POWDER

High-Temperature Furnace Crystallization and Purification

Boron metal is extremely reactive with most materials at high temperatures and, due to this high activity, is very difficult to purify. Prior to crystallization and purification, attempts were made to find suitable containers to hold the boron at elevated temperatures. Magnesium oxide, zirconium oxide, uranium dioxide with and without tantalum, niobium, and titanium liners degraded and fell apart in the presence of boron powder metal below 1,500° C. Crucibles made of the borides of hafnium, titanium, zirconium, tantalum, and niobium were used and these also degraded and fell apart prior to reaching 1,500° C. Finally, several crucibles were fabricated from various boron nitride sources and used as containers to hold boron at temperatures up to 1,900° C. All of the boron nitride crucibles cracked, powdered, deteriorated, or were otherwise unsuitable except for one grade designated as HBR boron nitride. This material loaded with boron powder and held in argon up to eight hours at 1,900° C did not show any signs of degradation, and the boron powdered metal formed a button without wetting the boron nitride container. Therefore, HBR boron nitride was accepted as the crucible material for all of the boron high-temperature work. Analytical results of the normal boron powder before and after this treatment show a slight increase in the purity of the material (see Table 1). Figures 1 and 2 present electron micrographs of the powder before and after heat treatment, and they indicate that some crystallization took place. Results from normal boron sintered below 1,900° C did not indicate any improvement in purity, but X-ray diffraction indicated that the amorphous material was becoming crystalline. Results from normal boron runs held at temperatures from 1,900° C and up indicated a crystalline structure and improvement in the purity of the product, as reported in Table 1. An X-ray diffraction pattern of amorphous normal boron powdered metal prior to heating is shown in Column 1, Figure 3.

Plasma Crystallization and Purification

Amorphous normal boron was also successfully crystallized and purified by feeding the powder through a thermal-dynamics, sheath-stabilized, DC-generated plasma flame. Analytical results of the feed and product are listed in Table 2. Figure 4 is a photograph of hollow and solid boron spheres that were unintentionally produced by the plasma flame. Figure 5 is a photomicrograph of boron particles that were crystallized and purified from amorphous normal boron containing 90 percent boron. These particles were produced by crushing the solid chunks that formed in the nozzle of the plasma and plugged the nozzle. The particles are 99.6 percent pure boron with helium densities ranging from 2.34 to 2.41 gms/cc. Electron microscope examinations of boron powder samples were undertaken at this time. Two of the electron

Table 1
ANALYSES OF BORON METAL POWDER
(Heated in Argon)

Material	Before 1,900° C in Argon (amorphous)	After 1,900° C in Argon (crystalline)
Particle Size (μ)	7	-
Surface Area (m^2/gm)	5.6	1.46
Helium Density (gms/cc)	2.37	2.52
O ₂ (%)	0.681	2.88
Boron Content (gm B/gm sample)	0.9588	0.9626
Carbon (wt %)	0.32	0.30
Other Impurities (ppm)		
Al	700	500
Ba	70	20
Ca	200	1,000
Cr	30	10
Cu	100	100
Fe	1,000	200
Mg	3,000	> 10,000
Mn	700	200
Ni	50	50
Pb	2,000	40
Si	200	1,000
Sn	30	50
Sr	100	< 100
Ti	150	15
W	< 10	< 10

Table 2
ANALYSES OF BORON METAL POWDER
(Heated in Helium Plasma)

Material	Before Plasma(1) (amorphous)	After Plasma(2) (crystalline)
Particle Size (μ)	9	Large Chunk
Surface Area (m^2/gm)	~6	0.13
Helium Density (gms/cc)	2.37	2.40
O ₂ (%)	5.26	0.059
Boron Content (gm B/gm sample)	0.8995	0.9956
Carbon (wt %)	0.23	0.184
Other Impurities (ppm)		
Al	150	100
Ba	10	20
Ca	1,000	700
Cr	60	150
Cu	80	150
Fe	250	100
Mg	5,000	800
Mn	80	60
Ni	60	80
Pb	125	40
Si	100	100
Sn	40	50
Sr	150	-
Ti	10	100
W	< 10	200

- (1) X-ray diffraction pattern indicates some crystalline but mainly amorphous material.
(2) X-ray diffraction pattern indicates good crystalline structure with no intermediate or minor phases.

EM 0654

Figure 1. ELECTRON MICROGRAPH OF AMORPHOUS BORON - TYPICAL PARTICLE SHAPES. (Colloidion Dispersion; 6,750X)

EM 0640

Figure 2. ELECTRON MICROGRAPH OF BORON AFTER HEATING AT 1,900° C FOR SIX HOURS IN AN ARGON ATMOSPHERE – TYPICAL PARTICLE SHAPES. (Collodion Dispersion; 6,750X)

Natural Amorphous Boron (90% B)

Crystalline Boron (98.5% B)

Boron - 10 (93% B)

124139

Figure 3. X-RAY DIFFRACTION PATTERNS.

microscope photographs have already been presented in Figures 1 and 2. The complete electron microscope study of various amorphous and crystalline boron metal powders can be seen in the photographs of Appendix A.

Three types of normal boron powdered metal were characterized prior to the crystallization experiments: amorphous boron, crystalline boron, and boron-10. The X-ray diffraction patterns of the three types are presented in Figure 3. The amorphous boron is transparent to X rays and only faint lines are observed; the 98.5 percent boron

(a) With Internal Void

B175(b)

(b) Solid

B175(d)

Figure 4. CRYSTALLINE BORON METAL SPHERES. (As Polished; 90X; Polarized Light)

B360

Figure 5. CRYSTALLINE BORON METAL PARTICLES OF 99.6 PERCENT BORON CONTENT. (As Polished; 100X; Polarized Light)

crystalline material shows a pattern in agreement with the ASTM standard; the boron-10 sample, reportedly crystalline, does not give the characteristic X-ray boron pattern but one indicating that various impurities are present. Analytical results of these three powders are reported in Table 3.

Amorphous normal boron was extremely difficult to feed uniformly from a hopper into a helium carrier gas stream. A feed system consisting of a vibratory feeder with an enclosure, at a slight helium overpressure, proved satisfactory. The vibratory feeder discharged the powder into a line through which helium was flowing directly into the plasma. At this time an exploratory run was made in which a cold finger was inserted directly below the plasma flame to produce a target for depositing normal boron metal powder. Purity of the crushed, leached, and washed deposit was 99.8 percent. The

Table 3
ANALYSES OF BORON METAL POWDER
(Three Unheated Powders)

Material	Standard-Grade Natural Boron(1) (amorphous)	Natural Boron(2) (crystalline)	Boron-10(3) (crystalline)
Particle Size (μ)	7	40	25
Surface Area (m^2/gm)	5.6	0.82	2.72
Helium Density (gms/cc)	2.37	2.3	2.24
O ₂ (%)	0.681	0.449	0.720
Boron Content (gm B/gm sample)	0.9588	0.9851	0.9367
Carbon (wt %)	0.32	0.85	0.41
Other Impurities (ppm)			
Al	700	200	15
Ba	70	< 10	-
Ca	200	70	100
Cr	30	30	12
Cu	100	300	800
Fe	1,000	200	100
Mg	3,000	200	90
Mn	700	200	10
Ni	50	90	400
Pb	2,000	10	40
Si	200	2,000	100
Sn	30	10	3
Sr	100	< 100	-
Ti	150	100	10
W	< 10	< 10	20

(1) American Potash.

(2) US Borax and Chemical.

(3) Atomic Energy Commission.

deposition rate of the boron metal powder was approximately two grams per minute. Encouraged by this initial work with normal boron metal powder and with hopes that electrolytic-cell boron-10 metal powder could be as readily crystallized and purified, preparations including a cost estimate (as seen in Appendix B) were begun toward repeating the plasma and furnace work with boron-10 enriched metal powder produced by the electrolytic-cell process. The available boron-10 metal powder was the electrolytic-cell product of the Hooker Chemical Company's Lewistown Plant.^(a) This powder was the source of all feed used in the initial small-scale boron-10 experiments. All boron-10 powder used after the initial experiments was obtained from Numec who assumed responsibility for the operation of the Lewistown Plant.

(a) The batch designation was ASC Blend 5, Lot 8-12-64 C.

Figure 6 presents X-ray diffraction patterns of the crystalline product produced by the furnace and plasma purification and crystallization processes. It can be seen that all three patterns in Figure 6 correspond extremely close to the patterns of the 98.5 percent crystalline normal boron powder presented in Column 2 of Figure 3.

Figure 6. CRYSTALLINE BORON METAL X-RAY DIFFRACTION PATTERNS.

INITIAL EXPERIMENTS WITH BORON-10 METAL POWDER

Electrolytically produced boron-10 powdered metal was successfully crystallized by heating it in an induction furnace in an argon atmosphere for four hours at $2,000^{\circ}\text{C}$ using a normal boron nitride container. The massive metal was then removed from the container, crushed, and washed to remove the oxide (B_2O_3) and other materials that collected as the mass cooled. The analytical results, as reported in Table 4, of this crushed and washed powder before and after thermal treatment show a slight increase in the total boron content of the material, with negligible degradation of the boron-10 content due to its containment in the normal boron nitride crucible. The boron-10 solidified button after removal from the boron nitride crucible is shown in Figure 7.

Heating boron-10 metal powder at high temperature in an inert atmosphere does produce a purer crystalline boron-10 metal with very little degradation of the boron content, as indicated in Table 4. However, some difficulty exists in handling the impurities volatilized from the boron-10 powder mass. These lower-boiling-point impurities sublime to every surface in the furnace that is cooler than the $2,000$ to $2,150^{\circ}\text{C}$ temperature to which the boron-10 metal powder is being subjected. This action results in the need for extensive and costly furnace cleaning. The cloud of vapors moving from the boron-10 mass toward the cooler surfaces tends to plug all openings and obscures the pyrometric readings. Holding the material at $2,000^{\circ}\text{C}$

Figure 7. CRYSTALLINE BORON-10 BUTTON. (After Four Hours at $2,000^{\circ}\text{C}$ In Argon)

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Table 4
BORON-10 METAL POWDER
(Before and After the 2,000° C Treatment)

Material	As-Received Electrolytic Amorphous Powder ⁽¹⁾	Crystalline Powder ⁽²⁾
B-10 (at %)	92.48	91.69
B-11 (at %)	7.52	8.31
Total Boron (%)	93.2	96.63
Surface Area (m ² /gm)	2.72	0.38
Density (gms/cc)		
Helium	2.24	2.30
Pour	-	0.81
Tap	-	1.30
O ₂ (%)	0.720	0.166
N ₂ (%)	-	1.15
C (%)	1.76	1.89
Other Impurities (ppm)		
Al	-	750
Ca	4,000	2,500
Co	-	20
Cr	400	125
Cu	8,000	3,125
Fe	600	1,875
Mg	-	100
Mn	300	250
Mo	-	-
Na	-	375
Ni	8,000	4,375
P	-	-
Pb	< 20	5
Si	1,000	250
Ti	100	10
V	-	12
Particle Size (μ)	25	- 105

(1) Hooker Chemical Company, Lot 8-12-64C.

(2) After 2,000° C heat treatment in an argon atmosphere.

in a vacuum does not eliminate the problem, but transports these materials into the vacuum system causing rapid deterioration of the system efficiency. Also, in vacuum, the normal boron nitride crucibles holding the boron-10 metal tend to dissociate, causing high degradation of the normal boron nitride crucible and subsequent contamination of the boron-10 metal with normal boron from the crucible. This thermal treatment does, however, purify and crystallize the boron-10, as shown previously in Table 4 and Figure 7. The X-ray diffraction patterns shown in Figure 8 were taken from electrolytic-cell boron-10 before and after the furnace treatment. The lines on the film prior to the thermal treatment show an amorphous structure with a titanium carbide impurity. The X-ray diffraction film after furnace treatment is a typical Debye-Scherrer X-ray diffraction pattern of β -rhombus crystalline form. No intermediate or minor compounds are shown.

Col 1
Before Furnace
Treatment

(a) As-Received Powder

Col 2
After Furnace
Treatment

(b) Furnace Product

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Figure 8. X-RAY DIFFRACTION PATTERNS TAKEN FROM ELECTROLYTIC-CELL BORON-10.

Since boron is extremely reactive with other materials at elevated temperatures, it became important to utilize methods and equipment that eliminate containers. Therefore, a sheath-stabilized, high-frequency electric torch utilized previously on amorphous normal boron was adapted to a receiving chamber with a controlled-speed cold-finger device that could be retracted from the flame tip to seven inches below that point at previously controlled rates. The entire device showing the plasma feeder, torch chamber, and the cold-finger operating mechanism is illustrated in Figure 9. The amorphous, electrolytic-cell boron-10 powder was crystallized by passing it through the sheath-stabilized DC plasma torch and depositing it as a solid boule of crystalline boron-10 powdered metal. The massive metal was then removed from the collection target, crushed, and sized.

Four similar runs were made on four different types of cold fingers. One was made on a stationary cold finger with a convex top; the other three runs were made on a retractable cold finger with different types of top designs. The three cold-finger tops that were utilized are shown in Figure 10. A typical boule formed on the fingers prior to crushing and grinding is presented in Figure 11. The analytical results of the starting boron-10 feed and the four crystallized and purified boron-10 runs are reported in Table 5. The boules formed on the cold fingers were collected, crushed, and screened through a 140-mesh (100-micron) screen prior to analysis. The X-ray film of the four plasma runs (Figure 12) are typical of the Debye-Scherrer rhombic crystal pattern.

After this experimental work on boron-10 metal powder was completed, an interest was expressed in producing boron-10 metal powder in production quantities and also in how much it would cost to produce this powder in production quantities. A cost estimate was prepared and is presented in Appendix B.

EXPERIMENTAL PRODUCTION OF BORON-10 POWDERED METAL

Furnace Method

Thirteen kilograms of boron powdered metal with a total boron content of approximately 93 percent and enriched to approximately 91 percent in boron-10 were successfully converted to highly crystalline metal by heating in an induction furnace for four hours at $2,000^{\circ}\text{C}$ in argon at atmospheric pressure. The 13 kilograms were processed in six runs averaging slightly over two kilograms of material in each run. The crystallized product from two of the runs can be seen in Figure 13. One, two-kilogram button illustrates the appearance as removed from the crucible after furnace heating; the second was broken to show the internal crystalline structure of the button.

The analytical results of the enriched boron metal powder used as the feed material in the furnace are presented in Table 6; the analytical results of the same material

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Figure 9. PLASMA ARC DEVICE. (Feeder, Torch, Chamber, and Cold-Finger Mechanism)

Table 5
COMPOSITION OF BORON-10 METAL POWDER
(Plasma Cold-Finger Runs)

Type of Finger	As-Received Material	Furnace Product			
		Plasma 1 Convex Top (1)	Plasma 2 Straight Top (2)	Plasma 3 Concave Top (2)	Plasma 4 Convex Top (2)
B-10 (at %)	92.48	92.77	-	-	92.69
B-11 (at %)	7.52	7.23	-	-	7.31
Total Boron (at %)	93.2	97.41	97.37	94.09	97.21
Surface Area (m ² /gm)	2.72	0.42	0.43	0.46	0.39
Density (gms/cc)					
Helium	2.24	2.21	2.23	2.22	2.24
Pour	-	0.858	0.82	0.82	0.84
Tap	-	1.35	1.30	1.33	1.33
O ₂ (%)	0.720	0.060	0.30	0.481	0.85
N ₂ (%)	-	0.013	0.229	0.059	0.138
C (%)	1.76	0.79	1.44	1.80	1.79
Other Impurities (ppm)					
Al	-	37	< 2	< 2	2,188
Ca	4,000	1,000	150	190	2,188
Co	-	13	12	13	20
Cr	400	125	38	65	250
Cu	8,000	3,750	1,875	1,950	3,125
Fe	600	1,000	1,875	1,000	2,188
Mg	-	75	62	100	250
Mn	300	300	150	150	250
Mo	-	62	10	80	< 10
Na	-	625	250	250	500
Ni	8,000	7,500	4,600	3,200	5,000
P	-	250	< 250	< 250	< 250
Pb	< 20	< 20	15	15	250
Si	1,000	7,500	150	190	438
Ti	100	< 10	< 10	< 10	25
V	-	25	10	25	20
Particle Size (μ)	25	- 105	- 105	- 105	- 105

(1) Stationary cold finger.
(2) Retractable cold finger.

Figure 11. BORON-10 BOULE.

116156

Figure 10. COLD FINGER TOP DESIGNS.

Plasma 1 - Stationary Finger; Convex Top

Plasma 2 - Retractable Finger; Straight Top

Plasma 3 - Retractable Finger; Concave Top

Plasma 4 - Retractable Finger; Convex Top

117204

Figure 12. BORON-10 METAL POWDER X-RAY DIFFRACTION PATTERNS.

117775

Figure 13. CRYSTALLIZED ENRICHED BORON METAL FURNACE PRODUCT. (Heated to 2,000° C in Argon for Four Hours)

Table 6
ENRICHED BORON POWDERED METAL
(Furnace Feed Material)

	Run Number					
	1	2	3	4	5	6
Numec Batch	49	39X46A	39X46B	53	51	48
Total Boron (%)	94.24	94.76	94.52	93.24	93.62	94.17
Enriched Boron (wt %)	90.50	90.30	90.35	90.56	90.48	90.73
Normal Boron (wt %)	9.50	9.70	9.65	9.44	9.52	9.27

after heating, grinding, screening, leaching, washing, and drying are presented in Table 7. Comparison of the batches both before and after processing indicates a total

Table 7
ENRICHED BORON POWDERED METAL
(Furnace Powdered Metal)

	Run Number					
	1	2	3	4	5	6
Numec Batch	49	39X46A	39X46B	53	51	48
Total Boron (%)	96.53	97.64	96.17	95.27	96.06	95.93
Enriched Boron (wt %)	87.44	86.62	89.73	90.54	90.08	90.27
Normal Boron (wt %)	12.56	13.38	10.27	9.46	9.92	9.73
Furnace Atmosphere	Argon	Argon	Argon	Argon	Argon	Argon
Time at 2,000° C (hrs)	4	6	4	4	4	4

boron purity increase of two to four percent and an initial boron-10 assay decrease of three percent. However, additional furnace runs using the same normal boron nitride container resulted in a decreasing interchange of the enriched and normal boron nitride in the product and crucible. The boron nitride crucible used showed no obvious sign of deterioration at any time. This enriched boron/normal boron exchange and apparent approach to an equilibrium condition was not unexpected. The one normal boron nitride crucible of HBR grade material with one-inch-thick walls, a one-inch-thick bottom, and an inside diameter of eight inches, was used for all six enriched boron furnace purification and crystallization runs. Photographs of the crucible prior to and after use are presented in Figures 14 and 15. The particles seen on the outside of the one crucible are crystallized droplets of enriched boron powdered metal. These droplets appeared on the second run and did appear to become larger or more numerous on the following runs. The crucible and button in the crucible shown in Figure 15 are as they appeared when removed from the furnace.

The two-kilogram button was approximately eight inches in outside diameter and one inch in thickness. The depth of the powder in this crucible prior to the four-hour, 2,000° C treatment was four inches, leaving a one-inch freeboard at the top of the crucible. The lighter-colored ring appearing on top of the button is a nickel boride compound impurity that was removed from the button easily, leaving the entire body with the crystalline appearance seen in the center of the crucible. This crystalline mass of enriched boron was easily crushed and sized to the desired particle size by using an impact-type crusher. This step was accomplished by repeated blows of a hydraulic ram and passage of the powder over a 140-mesh screen. This work resulted in the rapid sizing of the purified and crystallized enriched metal product. Since only diamond and boron carbide are harder than boron metal, attrition-type or rotating grinders would have a very short life expectancy and would add excessive impurities to the boron metal being ground.

118010

Figure 14. HBR-GRADE NORMAL BORON NITRIDE CRUCIBLES. (Outside View)

118011

Figure 15. HBR-GRADE NORMAL BORON NITRIDE CRUCIBLES. (Inside View)

Plasma Crystallization and Purification

Nine kilograms of the same type of feed material were also crystallized and purified by passing it through the sheath-stabilized DC plasma arc and depositing it as a solid boule of crystalline metal. The product was then removed from the water-cooled collection target and ground, screened, leached in hydrochloric acid solution, washed, dried, and sampled for isotopic and spectrochemical analyses. The nine kilograms were processed in five runs averaging approximately 1.8 kilograms per run. The analytical results of the enriched boron metal powder used as feed material in the plasma arc are summarized in Table 8; the analytical results of the same material after it was processed in the plasma arc, sized, and cleaned can be seen in Table 9. A comparison of the same batches before and after processing indicates a total boron content increase of 2 - 4 percent and no boron-10 degradation.

Table 8
ENRICHED BORON POWDERED METAL
(Plasma Feed Material)

	Run Number				
	1	2	3	4	5
Numec Batch	56	34	54	52	36
Total Boron (%)	93.21	93.64	93.14	92.36	93.60
Enriched Boron (wt %)	90.90	90.10	91.07	90.29	89.49
Normal Boron (wt %)	9.10	9.90	8.93	9.71	10.51

Table 9
ENRICHED BORON POWDERED METAL
(Plasma Product Material)

	Run Number				
	1	2	3	4	5
Numec Batch	56	34	54	52	36
Total Boron (%)	95.64	96.22	95.21	96.09	95.61
Enriched Boron (wt %)	90.71	90.17	90.88	90.27	89.54
Normal Boron (wt %)	9.29	9.83	9.12	9.73	10.46
Plasma Atmosphere	Helium	Helium	Helium	Helium	Helium
Amperage	400	400	400	400	400
Voltage	50	50	50	50	50

An overall program product material balance from the furnace and plasma processing of the 21,693 grams of enriched boron metal powder indicated an overall product yield of 91.88 percent; the remainder was in the form of recoverable leach solution, solids, paper salvage, unprocessed samples, or losses. A breakdown of the total recovery efficiency in the plasma and in the furnace can be seen in Table 10.

At this time it became necessary to ascertain if the Y-12 Plant analyses were accurate. Therefore, check samples were sent to the New Brunswick Laboratory. Analytical results from Y-12 and New Brunswick are presented in Table 11. As indicated, the results show good agreement in nearly every analysis. Since the New Brunswick Laboratory has served as the referee in boron analysis, the Y-12 analytical procedures and results can be accepted as being confirmed.

Thermal Decomposition of the Metal

Initial experiments with boron-10 powdered metal, electrolytically produced, indicated that boron-10 metal powder could be crystallized and purified by thermal decomposition in both the furnace and plasma. Therefore, all the available electrolytic-cell boron-10 powdered metal was transferred to the Y-12 Plant for purification and crystallization. This material, as received, was processed by the two methods previously reported. Five batches, as previously noted in Figure 9, were processed by passing the boron-10 metal feed through the hot zone of the DC arc helium plasma flame and collecting on a movable cold finger. Altogether, 7.45 kilograms of powder was fed through the plasma and 6.47 kilograms of crushed, leached, washed, and dried crystalline boron-10 metal product was obtained, for an overall efficiency of 86.8 percent. The other 28 batches of feed were processed by placing two kilograms of boron-10 metal powder feed at a time into a HBR-grade boron nitride crucible and heating it in a tungsten susceptor induction furnace for four hours at 2,000° C under an argon atmosphere. The 28 batches of feed, consisting of 51.2 kilograms as boron-10, were processed by the thermal method producing 20 batches of crushed, leached, washed, and dried boron-10 metal product weighing 48.36 kilograms—an overall product efficiency of 94.43 percent. The salvage from both the plasma and furnace processes is in the form of combustibles, furnace and plasma cleanings, and concentrated hydrochloric acid solutions. Efficient recovery processes could probably account for most of the material not accounted for as product. Analytical analysis and weights of all the batches received and of all the product produced are listed in Appendix C.

BOMB REDUCTION OF NORMAL BORIC OXIDE

Previous experience indicated that normal boron powder produced by magnesium reduction techniques could be crystallized and purified to total boron contents in excess of 99 weight percent; whereas, the electrolytic cell product purity only approached 98 percent total boron when similarly processed. It appeared feasible,

Table 10
MATERIAL BALANCE FOR ENRICHED POWDERED BORON METAL

	Quantity (grams)		
	Furnace	Plasma	Total
Feed	12,866	8,747	21,613
Shippable Product	12,019	9,564	19,583
Sample Product	181	94	275
Total Product	12,200	7,658	19,858
Product Yield (%)	94.8	87.55	91.88
<u>Salvage</u>			
Solids Recovered	470	731	1,201
Feed Samples	407	173	380
Leach Solution	-	-	-
Paper Salvage	-	-	-

Table 11
ENRICHED ELEMENTAL BORON
(Sample Exchange)

Source Analysis	Sample Number					
	1		2		3	
	Feed		Plasma Arc Product		Furnace Product	
	Y-12	NBL(1)	Y-12	NBL	Y-12	NBL
Total Boron (%)	93.16	93.0	95.16	94.9	94.72	93.6
Enriched Boron (wt %)	91.977	92.064	91.977	92.052	90.873	90.839
Normal Boron (wt %)	8.023	7.936	8.023	7.948	9.127	9.161
Atomic Weight	10.0862	10.0855	10.0862	10.853	10.0963	10.0966
Other Impurities (ppm)						
Ag	< 3	ND(2)	3	ND	< 3	ND
Al	625	95	315	100	625	390
Cu	1,250	2,300	1,250	1,900	1,875	2,800
Cr	1,000	100	75	110	75	130
Cu	9,800	9,000	7,500(3)	6,000	10,000(3)	9,000
Fe	1,500	1,500	1,375	1,700	1,800	2,300
K	6,250(3)	7,000(3)	< 15	50(3)	< 15	100(3)
Li	< 2.5	ND	< 2.5	ND	< 2.5	ND
Mg	400	115	85	95	125	110
Mn	375	450	225	350	375	450
Na	1,625	700(3)	625	100(3)	600	200(3)
Ni	6,250	9,000	6,875	9,700	6,875	10,000
P	< 250	ND	< 250	ND	< 250	100(3)
Pb	125	38	8	16	5	12
Si	2,315	2,100	2,190	2,500	1,875	2,400
Carbon (%)	2.28	1.78	2.26	1.36	2.03	1.68
Oxygen (%)	0.89	0.93	0.12	0.19	0.14	0.14

(1) NBL - New Brunswick Laboratory.

(2) ND - None detected.

(3) Approximate.

therefore, to modify the present metal-winning processes so that the boron-10 metal will be produced by thermite reduction techniques. The product impurities inherent in the metal so produced are the easily removed low-boiling-point metals rather than the difficult-to-remove metal contaminants of the electrolytic cell process. At present, the dimethyl ether boron-10 trifluoride complex is available as source material and could be hydrated as follows to produce a desirable feed for a bomb-reduction process.

Enriched potassium borofluoride was also readily available as feed material for bomb reduction processes but was so low in boron content that it had to be ruled infeasible when compared to the oxide with three times as much boron. Reactions visualized with boric oxide and the potassium salt are as follows:

The magnesium oxide of Reaction 4 can be readily separated from the boron powder by using dilute hydrochloric acid, while the potassium fluoride of Reaction 5 can be removed from the boron powder using hot water. In both cases the boron product achieved should be available with only potassium, magnesium, or other low-boiling-point metals as the major impurities. Magnesium, calcium, potassium, and the other metals boil at fairly low boiling points when compared to impurities imported by the electrolytic-cell processes. Since both compounds are readily available at low cost in the normal boron state and also can be readily obtained in the enriched state, bomb reduction of both compounds was contemplated.

Twenty-one thermite bomb reductions were fired in an attempt to produce a high-quality boron metal powder that could be thermally crystallized and purified to better than 98 percent. Three of the reactions were vented to the atmosphere with the metal product containing as much as 80.7 percent boron and an overall boron metal yield of 61.9 percent. The remaining thermite reactions were carried out in sealed bombs. Fourteen of the sealed bombs used magnesium as the reducing agent on the oxide and the other four sealed bombs used lithium metal as the reducing agent. The boron metal product produced by the magnesium reduction in the sealed bombs had a boron

content as high as 88 percent with a total yield of 86.2 percent. The boron metal product produced by lithium reduction of the oxide in the sealed bomb had a boron content of 82.1 percent and a total yield of 42.4 percent. The analytical results of the magnesium and lithium metal reductions are summarized in Table 12. Table 12 also shows the analysis of the oxide powder and the magnesium powder charged into the reactor. The lithium metal used was not analyzed but is a pure lithium metal produced routinely at the Y-12 Plant obtained from a pure lithium metal production stream. The photomicrographs of Figure 16 show that the powder produced in the vented reactor is much smaller than the powder produced in the sealed reactor.

Analytical results also indicate that the Fisher sub-sieve size of the powder produced in the open bomb is approximately one micron, while the particle size of the boron metal produced in the sealed bomb averages approximately 23 microns in size. Figure 17 is a photomicrograph of the sealed-bomb boron metal powder product that was taken under polarized light to show the particle crystalline structure. X-ray diffraction patterns, shown in Figures 18 and 19, also show that the open or vented reactions produced amorphous powder, while the sealed reactions produced a crystalline pattern.

The first sealed bomb was prepared by mixing a charge of 866 grams of boric oxide and 908 grams of magnesium powder and placing the blended powders into a Type 309 stainless steel reactor. The reactor was then heated in an induction coil until the exothermic reaction started (at 850° C). The reactions then carried the external temperature of the reactor to 1,100° C where it leveled out for ten minutes before the reactor started cooling. The pressure at the beginning of the reaction rose to 475 psig, held at this level for approximately 1 - 5 seconds, then dropped to less than 100 psig. By utilizing encapsulated chemical igniters composed of 65 weight percent potassium iodate, 10 weight sodium peroxide, and 25 weight percent magnesium powder, the ignition point of the reaction was reduced to 550° C; and, with 1,600 grams of boric oxide and 1,679 grams of magnesium powder, the pressure was held to 600 psi. The lower pressure and lower ignition temperature can be handled more safely by the 1/2-inch-thick, 6-inch-ID, 13-inch-long Type 309 stainless steel reactor. Table 13 lists the charges and pressures that were obtained during the firing of six different reactors. In reactions where chemical igniters were used, the capsule was inserted one inch from the wall and one inch deep in the charge.

Four lithium reduction bombs were prepared by mixing boric oxide powder and lithium metal and placing the blend in the same reactors used in the magnesium reductions. However, chemical igniters were not used on any of the four lithium reactions. The first lithium reduction using 220 grams of lithium and 350 grams of the oxide resulted in a solid fused mass that coated the bottom and sides of the reactor and was almost impossible to remove. The charge was increased on the second reduction to 440 grams of lithium and 700 grams of the oxide. The second reduction resulted in a solid mass that was more friable and could be easily broken into chunks and removed from the reactors. The summary of charges used and the firing temperatures

(a) Vented Reactor

C950-1

(b) Sealed Reactor

C950-2(a)

Figure 16. BORON POWDER METAL BY BOMB REDUCTION. (Bright Field Illumination; 100X)

C950-2(b)

Figure 17. NORMAL BORON POWDERED METAL FROM BOMB REDUCTION 4 (SEALED). (As Polished; Polarized Light; 100X).

Table 13
SEALED BOMB REDUCTIONS

Reduction	B ₂ O ₃ (gms)	Mg Powder (gms)	Ignition Temperature (° C)	Reactor Pressure (psi)
1 ⁽¹⁾	866	908	850	475
2	866	908	550	450
3	1,000	1,049	540	500
4	1,200	1,249	525	550
5	1,400	1,469	540	600
6	1,600	1,679	520	600

(1) This reduction run did not have a chemical igniter and reacted at the normal oxide-magnesium ignition temperature.

121223

Figure 18. X-RAY DIFFRACTION PATTERN OF BOMB REDUCTION PRODUCT FROM A VENTED REACTOR.

121224

Figure 19. X-RAY DIFFRACTION PATTERN OF BOMB REDUCTION PRODUCT FROM A SEALED REACTOR.

and pressures on the four lithium metal-boric oxide reductions are reported in Table 14. The equation theorized from the reaction is as follows:

The product recovered from the magnesium-boric oxide reductions was placed in boron nitride crucibles and heated in a tungsten susceptor induction furnace to temperatures of 2,000 - 2,150° C and held for four hours in an argon atmosphere. Heating under vacuum was tried on several batches, but vacuum dissociated the boron nitride crucibles and did not improve the crystallization and purification of what little of the boron batch that could be recovered. The buttons resulting from furnacing the magnesium-reduced boron powder were crushed, sized to -100 mesh, and then processed as follows:

1. The crushed reaction product was dissolved by adding the powder slowly to boiling 37.7 percent hydrochloric acid solution. Failure to have the solution boiling would result in the accumulation of undissolved magnesium compounds that could result in a violent reaction when the temperature did reach boiling. After all powder had been added to the hydrochloric acid, the slurry was boiled for one hour. Solution level was maintained at all times by adding fresh hydrochloric acid solution.
2. The slurry was then cooled and filtered. The product, a fine black powder, was retained on the filter. It was water washed thoroughly to remove all traces of hydrochloric acid.
3. The powder was leached in ethylenediaminetetraacetic acid solution for one hour, filtered, and water washed again.
4. Steps 1 and 2 were repeated.
5. The wet powder was washed with acetone and then dried in a vacuum oven at 100° C.

Table 14
LITHIUM METAL-BORIC OXIDE POWDER BOMB REDUCTION

Reduction	B ₂ O ₃ (gms)	Lithium (gms)	Ignition Temperature (° C)	Reactor Pressure (psi)
1	350	220	625	25
2	700	440	550	25
3	1,050	665	625	25
4	1,400	875	640	25

This procedure was performed on all magnesium bomb reduction product obtained. The dried boron powders were then placed in boron nitride crucibles and held from 4 to 7 hours under argon in a tungsten-suscepted furnace. The resultant crystalline button was then crushed under a hydraulic ram to - 100 mesh and then the leaching process, as herein described, was repeated on the crystallized and purified boron metal powder.

The product from the lithium reaction was a sintered, friable mass somewhat less friable than the mass produced by magnesium reactions. This lithium-boric oxide reaction product was crushed into small pieces and then processed as follows:

1. The reaction product was dissolved by adding the pieces very slowly to cold water. The reaction was very vigorous and the water would soon become hot and boil. As the water became more alkaline, due to the formation of lithium hydroxide, the boron was allowed to settle. After the hydroxide solution was decanted, more water was added.
2. After all the reaction products were dissolved and the solids remaining were in water close to the pH of 7, the slurry was filtered and the solids washed thoroughly with water and then dried on the filter.
3. The solids from the filter were added to boiling 37.7 percent hydrochloric acid and leached for one hour.
4. The slurry was then cooled and filtered. The product, as a fine boron powder, was retained on the filter. It was washed thoroughly with distilled water to remove all traces of hydrochloric acid.
5. The powder was releached in an ethylenediaminetetraacetic acid solution for one hour, filtered, and water rinsed once more.
6. The boron powder product was then dried with acetone and placed in a vacuum furnace at 100° C until thoroughly dried.

The product recovered from the second lithium-boric oxide reduction was placed in a boron nitride crucible and heated in an argon-atmosphere tungsten-suscepted furnace. At somewhere around 200 - 300° C the powder decomposed violently. The force of the explosion was sufficient to lift the 44-pound furnace lid.

Prior to the violent decomposition of the Reduction 2 product, the product from Reduction 3 had been dissolved in water and filtered. The solids were in the process of being removed from the filter funnel when they decomposed violently causing serious burns to the operator. Analysis of Product 2, which had been processed the same way without decomposition, indicated the formation of a borane compound that evidently triggered the violent reaction. The only source of hydrogen available

during the process and necessary for formation of the borane compound (probably $B_{18}H_{22}$) had to be the decomposition of water during the dissolution of the bomb reduction product.

Table 15 gives the analytical results of the furnace product produced from the magnesium-boric oxide and the lithium-boric oxide bomb reduction products. It can be seen that 99.3 percent crystalline boron was produced in Run 18 by holding the impure bomb reduction product at $2,150^{\circ}C$ for four hours. Therefore, it would be possible to produce 98 - 100 percent crystalline boron-10 material by preparing the boron-10 metal powder from enriched oxide as normal boron powder is now prepared commercially by a number of industries throughout the country. This boron-10 metal powder could then be thermally crystallized and purified to the desired purity by the pyrometallurgical techniques developed at Y-12.

Table 15
BORON CRYSTALLIZATION AND PURIFICATION PRODUCT ANALYSES

	Run Number																
	1 & 2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Source of Feed (bomb reduction number)	3,6	4	4	4	7	7	7	11	12	13	14	15	16	17	17	17	17
Furnace Temperature (° C)	2,000	2,000	2,000	2,000	2,000	2,000	2,000	2,000	2,000	2,000	2,050	2,050	2,100	2,000	2,050	2,100	2,150
Furnace Atmosphere(1)	Argon													Vacuum		Argon	
Time at Temperature (hours)	4	4	4	4	5	5	5	7	7	7	7	7	5	1	0.25	0.08	4
Furnace KW	11.5	12.0	13.0	12.9	13.0	12.5	13.0	13.0	13.0	13.2	13.8	13.8	14.7	12.0	12.4	12.7	15.5
Total Boron (wt %)		97.6	97.2	95.8	98.4	96.6	97.4	98.5	96.2	89.9	98.1	98.0	97.0	97.7	96.3	97.8	99.3
Magnesium (wt %, by chemical analysis)		0.168	0.596	1.862	0.664	0.274	0.102	0.052	0.029	0.465	0.268	0.040	0.020	0.06	0.12	0.10	0.05
Impurities (ppm)	Did not Consolidate to Button																
Al		1,875	375	438	375	625	625	500		250	375	500	500	3,750	3,750	500	750
Ca		188	375	438	625	1,250	1,500	1,500		375	375	375	2,500	2,500	2,000	375	2,000
Cr		200	150	150	15	20	10	15		15	15	15	20	62	125	15	45
Cu		250	375	250	100	100	38	100		50	25	75	38	62	100	75	75
Fe		1,875	1,875	750	312	500	1,000	438		875	875	875	1,000	938	1,250	875	938
Mg		1,000	3,125	10,000	13,125	500	1,000	750		3,750	1,875	500	500	188	100	500	375
Mn		1,250	312	375	225	250	188	175		625	1,000	625	750	2,500	3,438	625	3,125
Na		3,750	1,250	1,250	75	188	125	750		750	938	750	750	938	750	750	1,000
Ni		150	250	175	50	88	75	50		38	38	38	38	75	100	38	75
Si		2,188	1,250	2,188	1,250	500	1,250	750		750	1,500	1,000	500	750	750	750	1,000
O ₂															1,144	5,904	1,326
N ₂															2,431	914	652
X-Ray Diffraction																	
Major Constituent		B	B	B	B	B	B	B		B	B	B	B	B	B	B	B
Minor Constituent		B ₂ O ₃	B ₂ O ₃	B ₂ O ₃ & Mg	MgO	MgO	MgO	MgO		B ₂ O ₃	MgO	MgO	B ₂ O ₃	B ₂ O ₃	B ₂ O ₃	-	-

(1) All runs under vacuum resulted in complete destruction of the boron nitride crucible.

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APPENDIX A

ELECTRON MICROSCOPE EXAMINATION OF BORON POWDER SAMPLES

Sample History

In preparation for the boron-10 program, development studies were made in the processing of boron powders. Five samples of boron powder were submitted for electron microscopic examination of particle size and shape. The samples and their history are reviewed in Table A-1.

Experimental Work

Small amounts of the boron powder were dispersed on a suede glass^(a) with a steel spatula in a 2.5 percent solution of collodion in amyl acetate. The mixture was worked to dryness several times using amyl acetate as the vehicle. After the last working, the dry material was slurried with a minimum amount of ethyl acetate, smeared on a glass slide, and allowed to dry. A carbon film was vacuum deposited over the collodion dispersion. The carbon-collodion film was scored into 1/8-inch squares and removed from the glass slide by floating on water. The squares were picked up on 1/8-inch-diameter, 200-mesh copper grids and the collodion dissolved away with amyl acetate. The boron powder imbedded in the carbon film was examined in the electron microscope, and electron micrographs were taken.

Discussion

Samples 2, 3, and 5 were very gritty while being dispersed on the glass plate. Sample 1 formed a paste-like mixture immediately with the collodion-amyl acetate solution. Sample 4 exhibited both characteristics.

Examination in the electron microscope showed all five samples to have both crystalline and amorphous particles; electron diffraction patterns from single crystals can be found even in the amorphous samples. In general, Sample 1 appears to be predominately noncrystalline (Figures A-1 and A-2) while Samples 2 and 5 (Figures A-3, A-4, A-9, and A-10) are predominately crystalline. Although Samples 3 and 4 (Figures A-5, A-6, A-7, and A-8) are not so easily characterized, they appear to be more crystalline than amorphous.

As can be seen in the electron micrographs, the particle sizes cover a large range—from less than 0.1 micron in all samples to greater than 5 microns in the amorphous samples and greater than 30 microns in the crystalline samples.

(a) E. F. Fullam, Inc, Dispersion Kit; Fullam Catalog Number 1138.

Table A-1
BORON POWDER SAMPLES SUBMITTED FOR
ELECTRON MICROSCOPIC STUDY

Sample Number	Source and/or Treatment	Boron (%)
1	Standard Grade Amorphous Boron (American Potash Company)	95.9
2	100-Mesh Crystalline Boron (US Borax and Chemical Corporation)	98.5
3	Electrolytic Crystalline Boron-10 (received from the AEC for experimental studies)	93.7
4	Same Material as Sample 1, but after Heating at 1,550° C. (argon atmosphere) for 4 Hours	
5	Same Material as Sample 1, but after Heating at 1,900° C (argon atmosphere) for 4 Hours

EM 0654

Figure A-1. TYPICAL PARTICLE SHAPES OF AMORPHOUS BORON. (Sample 1; Collodion Dispersion; 6,750X)

EM 0652

Figure A-2. SELECTED GROUP OF LARGE PARTICLES OF AMORPHOUS BORON. (Sample 1; Collodion Dispersion; 6,750X)

EM 0650

Figure A-3. TYPICAL PARTICLE SHAPES OF CRYSTALLINE BORON. (Sample 2; Collodion Dispersion; 6,750X)

EM 0649

Figure A-4. SELECTED GROUP OF LARGE PARTICLES OF CRYSTALLINE BORON. (Sample 2; Col-
lodion Dispersion; 6,750X)

EM 0658

Figure A-5. TYPICAL PARTICLE SHAPES OF ELECTROLYTIC BORON-10. (Sample 3; Collodion Dispersion; 6,750X)

EM 0659

Figure A-6. SELECTED GROUP OF LARGE PARTICLES OF ELECTROLYTIC BORON-10. (Sample 3; Collodion Dispersion; 6,750X)

EM 0661

Figure A-7. TYPICAL PARTICLE SHAPES OF AMORPHOUS BORON THAT WAS HEATED AT 1,550° C FOR FOUR HOURS IN AN ARGON ATMOSPHERE. (Sample 4; Collodion Dispersion; 6,750X)

EM 0663

Figure A-8. SELECTED GROUP OF LARGE PARTICLES OF AMORPHOUS BORON THAT WAS HEATED AT 1,550° C FOR FOUR HOURS IN AN ARGON ATMOSPHERE. (Sample 4; Collodion Dispersion; 6,750X)

EM 0640

Figure A-9. TYPICAL PARTICLE SHAPES OF AMORPHOUS BORON THAT WAS HEATED AT 1,900° C FOR FOUR HOURS IN AN ARGON ATMOSPHERE. (Sample 5; Collodion Dispersion; 6,750X)

EM 0641

Figure A-10. SELECTED GROUP OF LARGE PARTICLES OF AMORPHOUS BORON THAT WAS HEATED AT 1,900° C FOR FOUR HOURS IN AN ARGON ATMOSPHERE. (Sample 5; Collodion Dispersion; 6,750X)

APPENDIX B

COST ESTIMATE FOR THE PREPARATION OF CRYSTALLINE BORON-10 METAL POWDER

Introduction

Costs for the preparation of crystalline boron-10 metal powder from the electrolytic cell product of the Hooker Chemical Company have been estimated for two different processes: (1) the plasma-torch process in which the feed material is passed through the hot zone of a DC arc plasma, collected as a boule on a water-cooled target, and finally crushed and sized; (2) a high-temperature furnace operation in which a mass of the feed material is placed in a suitable container in a high-temperature furnace and taken to the melting point of boron in a flowing inert gas atmosphere. Upon cooling, the consolidated metal is crushed and sized as in the plasma torch method.

In both cases, the estimates do not include any recycle salvage costs, but are based merely on the cost of operating each process, the materials involved, special equipment where necessary, and the associated analytical and packaging costs.

Plasma Torch Conversion

The powdered boron-10 metal product from the Hooker Chemical Company is fed into the helium plasma flame and deposited as a solid boule of crystalline boron-10 metal. The massive metal is then removed from the collection target, crushed, and washed to remove oxide and other impurities that collected as the boule cooled. The extremely hard boron-10 crystalline boule is crushed to -140 mesh with no more than 15 weight percent less than 10 microns, then given a gentle leach in dilute hydrochloric acid prior to a final water wash. The sized crystalline boron-10 powder is sampled as a two-kilogram batch and packaged for shipment.

Conversion Costs - Cost for the conversion of amorphous electrolytic cell produced boron-10 metal to 10 kilograms of crystalline boron-10 product are listed as follows:

Labor	\$4,400
Materials	1,400
Analytical Services	1,600
Packaging and Shipping	<u>200</u>
Total	\$7,600

The analytical costs are based on one sample for each two kilograms of crushed product. The cost is approximately \$760 per kilogram of crystalline boron-10 product.

Estimated Yield - Estimated yields from the conversion of boron-10 electrolytic cell product to crystalline boron-10 based on several 200-gram and one 1,000-gram runs have been approximately 75 percent by weight. The remaining 25 percent would be collected and returned for salvage recovery.

Material Requirements - Based on a 75 percent recovery efficiency, it would be necessary to process 14.0 kilograms of feed material in order to produce 10 kilograms of the crushed, cleaned, and sized crystalline boron-10 metal powder product.

Program Tenure - The crystalline boron-10 metal powder, with analytical results on each batch, could be ready to ship within six to eight weeks after receipt of the boron-10 feed material.

High-Temperature Conversion

The electrolytic boron-10 metal powder is placed in normal boron nitride crucibles and sintered in an argon atmosphere at 2,000° C for four hours. The crystallized boron-10 solidified mass is then removed from the boron nitride crucible and crushed, leached, and washed. The sized boron-10 powder is sampled and packaged for shipment.

A major cost in producing crystalline boron-10 metal powder by this process is the expense of the normal boron nitride billet from which the crucibles are fabricated. A crucible sized to hold approximately 2,000 grams of electrolytic boron-10 feed material will cost approximately \$2,300. This total includes the cost of the massive stock and all machining operations. While the life expectancy of the crucibles is not known at this point in the development program, three units are believed to be a satisfactory number to complete this work. The desirability or necessity of fabricating crucibles from boron-10 nitride powder is presently being evaluated.

Cost of Conversion - Costs for the conversion of boron-10 electrolytic cell product to 10 kilograms of crystalline boron-10 metal powder by the high-temperature furnace method are as follows:

Labor	\$4,600
Materials	
(3 boron nitride crucibles)	7,000
Analytical Services	1,600
Packaging and Shipping	200
	<hr/>
Total	\$13,400

The analytical costs are based on one sample for each two kilograms of crushed product. The cost is approximately \$1,340 per kilogram of crystalline product.

Estimated Yield - Estimated yields on converting boron-10 cell product to crystalline boron-10 by high-temperature furnace methods, based on several 200-gram runs, is estimated to be 88 percent by weight. The salvage of the remaining 12 percent would probably be more complicated than the plasma method, but all recovered material would be returned for salvage.

Material Requirements - Based on an 88 percent recovery efficiency, it would be necessary to process 12.0 kilograms of boron-10 feed material in order to produce 10 kilograms of crystalline boron-10 metal powder product.

Program Tenure - The crystalline boron-10 metal powder, with analytical results on each batch, could be ready to ship within eight to ten weeks after receipt of the feed material.

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APPENDIX C

ANALYTICAL DATA ON BORON-10 METAL POWDER (AS RECEIVED AND AS PRODUCED)

Table C-1
MATERIAL RECEIVED, BORON-10 METAL POWDER
(Analytical Data)

Batch	56	34	54	36	46A	46B	48	51	52	53	49	59A	59B	79A	79B	79C	80	81
Boron (%)	93.21	93.64	93.14	93.60	94.76	94.52	94.17	93.62	92.36	93.24	94.24	92.48	92.80	93.80	94.54	94.38	94.94	94.24
Boron-10 (wt %)	90.90	90.10	91.07	89.49	90.30	90.35	90.73	90.48	90.29	90.56	90.50	91.45	91.45	91.41	91.41	91.42	91.54	91.44
Boron-11 (wt %)	9.10	9.90	1.16	10.51	9.70	9.65	9.27	9.52	9.71	9.44	9.50	8.55	8.55	8.59	8.59	8.58	8.46	8.56
N ₂ (%)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
O ₂ (%)	0.99	1.11	0.76	0.57	0.95	0.84	0.75	1.30	0.88	0.70	0.64	1.39	1.22	1.02	0.75	0.95	0.63	1.05
Carbon (%)	0.72	0.35	1.16	0.51	0.43	0.36	0.56	0.44	0.69	0.92	0.69	1.36	0.89	1.12	1.23	1.76	1.05	1.45
Other Impurities (ppm)																		
Ca	1,280	1,280	670	1,440	740	680	650	690	540	800	830	770	1,130	2,660	1,080	900	760	330
Cr	20	20	70	20	8	6	6	110	220	60	6	270	150	180	220	170	120	230
Cu	10,400	10,400	8,650	7,200	5,000	9,350	9,100	6,100	4,900	8,000	8,100	11,400	11,200	900	980	900	940	10,000
Fe	1,730	1,730	960	1,580	4,250	2,200	1,320	5,080	15,000	1,440	1,290	350	1,340	1,710	2,600	1,090	840	1,999
K	6,650	6,650	2,500	10,150	5,350	4,000	6,200	5,080	3,380	1,650	2,630	1,820	2,250	3,800	1,800	2,400	500	2,600
Mg	900	900	2,000	800	860	800	1,500	1,200	540	800	900	1,200	1,200	1,700	900	1,500	1,500	750
Mn	330	330	460	280	360	290	310	300	470	410	330	340	410	410	470	430	320	530
Na	500	500	250	500	200	400	800	500	200	200	500	400	-	400	800	300	200	80
Ni	17,400	17,400	2,380	16,200	17,200	21,300	19,100	19,500	13,100	23,200	18,400	15,800	16,400	15,300	12,800	16,400	13,000	12,600
Si	500	500	600	1,000	1,500	1,300	800	20	800	800	10	400	800	600	400	500	500	500

Table C-1 (Continued)

Batch	82	83	87B	88B	87C	89A	89	89B	91A	90	90B	91B	91A	86A	94B	95	90A	86B
Boron (%)	96.74	94.38	93.23	95.30	94.06	94.00	95.14	93.77	94.54	94.10	95.26	94.12	93.52	93.59	93.67	94.46	93.26	94.58
Boron-10 (wt %)	91.53	91.57	91.60	91.73	91.60	91.81	91.84	91.64	91.80	91.93	91.67	91.87	91.85	91.75	91.54	92.06	91.87	91.75
Boron-11 (wt %)	8.47	8.43	8.40	8.27	8.40	8.19	8.16	8.36	8.20	8.07	8.33	8.13	8.15	8.25	8.46	7.94	8.13	8.25
N ₂ (%)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
O ₂ (%)	0.58	1.12	0.80	0.67	0.65	0.57	0.47	0.69	1.50	0.79	0.68	0.88	1.01	0.68	0.77	0.70	0.93	0.70
Carbon (%)	0.92	1.36	2.70	0.40	0.43	0.50	0.66	0.61	1.03	0.97	0.78	1.29	0.78	0.50	1.29	1.14	1.60	1.21
Other Impurities (ppm)																		
Ca	2,220	1,350	590	690	400	1,320	1,220	2,100	590	690	400	1,320	1,220	2,100	2,250	1,120	1,150	1,900
Cr	130	150	330	40	30	10	10	40	40	260	20	500	70	10	40	360	370	60
Cu	9,000	8,900	8,400	8,600	9,600	7,100	7,800	10,100	8,400	8,600	9,600	7,100	7,800	10,100	13,900	6,600	9,400	8,600
Fe	1,420	2,320	1,530	2,420	800	2,180	4,600	1,210	1,530	2,420	800	2,180	4,600	1,210	1,220	2,100	1,770	1,740
K	5,600	4,500	4,400	2,500	7,100	2,220	2,700	6,400	4,400	2,500	7,100	2,200	2,700	6,400	3,400	1,500	1,900	6,400
Mg	2,400	2,000	500	1,400	2,500	5,000	5,700	5,000	500	1,400	2,500	5,000	5,700	5,000	4,000	4,000	3,600	5,700
Mn	390	390	300	590	210	680	240	370	300	590	210	680	240	370	500	480	760	240
Na	400	450	500	200	300	500	200	600	500	200	300	500	200	600	150	400	300	700
Ni	13,700	9,800	14,000	11,500	14,900	7,400	15,100	17,000	14,000	11,500	14,900	7,400	15,100	17,000	18,200	7,400	10,400	8,200
Si	1,000	800	400	500	200	250	300	800	400	500	2,000	2,500	3,000	800	1,000	1,300	300	-

Table C-2
MATERIAL RECEIVED, BORON-10 METAL POWDER
(Batch Weights)

Batch	Total Weight (gms)	Net Boron (gms)	Net Boron-10 (gms)	Cumulative Net Boron-10 (gms)
56	1,446.1	1,347.9	1,225.3	1,225.3
34	1,659.9	1,554.33	1,400.5	2,625.8
54	1,421.5	1,323.99	1,205.8	3,831.6
36	2,341.9	2,192.02	1,961.6	5,793.2
46A	2,481.4	2,351.38	2,123.3	7,916.5
46B	2,201.2	2,080.57	1,879.8	9,796.3
48	2,191.8	2,064.02	1,872.7	11,669.0
51	2,143.1	2,006.37	1,815.4	13,484.4
52	1,990.3	1,838.24	1,659.8	15,144.2
53	2,051.7	1,913.01	1,723.4	16,876.6
49	2,004.1	1,888.66	1,709.2	18,585.8
59A	2,340.5	2,164.49	1,979.4	20,565.2
59B	2,093.4	1,942.68	1,776.6	22,341.8
79A	1,870.7	1,754.22	1,604.0	23,945.8
79B	1,851.2	1,750.12	1,599.8	25,545.6
79C	1,737.6	1,639.95	1,499.2	27,044.8
80	2,328.7	2,210.87	2,023.8	29,068.6
81	3,044.2	2,868.85	2,623.3	31,691.9
82	1,969.5	1,865.90	1,707.9	33,399.8
83	2,526.6	2,384.61	2,183.6	35,583.4
87B	1,713.4	1,597.40	1,463.2	37,046.6
88B	2,355.0	2,244.32	2,058.7	39,105.3
87C	1,621.7	1,525.37	1,397.2	40,502.5
89A	1,348.6	1,267.68	1,163.9	41,666.4
89	2,016.3	1,918.31	1,761.8	43,428.2
89B	1,300.9	1,219.85	1,117.9	44,546.1
91A	1,242.1	1,174.28	1,078.0	45,624.1
90	2,091.7	1,968.29	1,809.4	47,433.5
90B	1,783.8	1,699.25	1,557.7	48,991.2
91B	1,277.0	1,201.91	1,104.2	50,095.4
91A	1,730.1	1,617.99	1,486.1	51,581.5
86A	1,681.8	1,580.05	1,449.7	53,031.2
94B	1,431.4	1,340.36	1,227.0	54,258.2
95	1,924.4	1,817.79	1,673.5	55,931.7
90A	1,998.2	1,863.52	1,712.0	57,643.7
86B	1,167.0	1,103.75	1,012.7	58,656.4

Unless otherwise specified, the X-ray diffraction patterns on all batches indicate an amorphous structure.

Table C-3
 PRODUCT PRODUCED, BORON-10 METAL POWDER
 (Analytical Results)

Batch	56	34	54	36	46A	46B	48	51	52	53	49	59A	59B	79A	79B	79C
Process	Plasma	Plasma	Plasma	Plasma	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace
Boron (%)	95.64	96.22	95.21	95.61	97.64	96.17	95.93	96.06	96.09	95.27	96.53	95.75	94.12	95.74	97.81	95.80
Boron-10 (wt %)	90.71	90.17	90.88	89.54	86.62	89.73	90.27	90.08	90.27	90.54	87.44	90.54	90.94	90.61	90.44	89.18
Boron-11 (wt %)	9.29	9.83	9.12	10.46	13.38	10.27	9.73	9.92	9.73	9.46	12.56	9.46	9.06	9.39	9.56	10.82
O ₂ (%)	0.249	0.129	0.133	0.208	0.177	0.163	0.256	0.087	0.204	0.25	0.234	1.03	1.446	0.25	0.25	0.013
Carbon (%)	0.67	0.59	1.00	0.61	0.49	0.63	0.52	0.48	0.73	0.80	0.40	1.23	0.83	0.90	1.38	0.80
Other Impurities (ppm)																
Al	50	125	188	100	125	75	100	188	125	125	100	565	375	435	300	500
Ca	188	250	750	1,875	3,750	625	375	750	125	500	7,500	200	2,450	1,800	465	2,250
Cr	312	250	1,000	500	625	188	375	500	187	312	500	3,750	1,950	2,350	6,875	2,150
Cu	6,250	7,500	7,500	6,250	6,250	6,250	5,625	5,625	3,750	3,438	5,625	1,125	10,000	11,250	5,000	5,000
Fe	1,688	1,875	2,188	2,500	2,500	1,500	1,000	1,875	8,750	1,500	1,562	6,850	2,100	2,750	4,062	1,675
K	< 15	50	25	138	< 15	< 15	< 15	50	62	15	< 15	75	75	60	50	25
Mg	125	250	188	625	250	375	125	1,500	312	188	500	170	560	1,258	500	160
Mn	150	375	375	312	250	188	150	150	375	125	312	625	310	280	375	170
Na	250	625	375	312	250	250	312	312	312	188	250	1,875	1,250	1,875	1,250	1,250
Ni	10,000	12,500	12,500	11,250	11,250	11,250	9,375	9,375	10,000	12,500	11,250	10,000	12,500	12,500	5,000	5,000
Si	1,875	2,500	6,250	3,438	1,688	2,500	2,500	1,875	3,750	100	3,750	3,125	2,500	1,530	1,250	2,250

Table C-3 (Continued)

Batch	80	81	82	83	86A	87C	88B	89A	89B	91A	90	90B	95	90A	94B
Process	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace	Furnace
Boron (%)	95.84	96.12	95.66	95.91	95.51	95.88	96.26	96.87	95.81	96.37	96.65	97.41	94.56	94.53	98.30
Boron-10 (wt %)	90.82	91.07	90.82	90.60	90.83	90.71	90.73	90.74	90.83	90.86	90.31	90.66	90.45	90.53	90.33
Boron-11 (wt %)	9.18	8.93	9.18	9.40	9.17	9.29	9.22	9.26	9.17	9.14	9.69	9.34	9.55	9.47	9.67
O ₂ (%)	0.20	0.71	0.20	0.20	0.23	0.61	0.28	0.17	0.72	0.53	0.136	0.17	-	-	-
Carbon (%)	0.89	1.21	0.82	0.96	0.90	1.15	0.34	0.57	-	0.81	0.88	0.57	0.82	1.54	0.57
Other Impurities (ppm)															
Al	500	475	500	2,500	750	500	250	375	750	1,875	2,500	1,875	1,250	1,250	750
Ca	1,875	465	250	5,000	2,500	2,500	1,875	1,250	1,875	200	750	1,000	1,562	1,000	1,250
Cr	3,125	2,750	4,375	6,250	1,875	3,750	625	1,000	1,875	438	3,750	438	5,625	5,312	275
Cu	3,750	12,500	3,750	5,625	2,500	5,000	3,750	3,750	6,250	8,750	8,125	8,750	500	1,875	1,250
Fe	1,250	2,500	3,750	3,438	1,875	2,500	1,562	1,562	1,688	10,000	8,750	1,250	3,125	2,188	1,625
K	25	125	25	50	250	50	50	125	100	38	38	< 15	38	75	75
Mg	250	170	750	375	750	750	750	750	750	625	438	438	500	375	250
Mn	125	345	375	312	375	500	188	250	375	200	750	500	250	625	1,875
Na	625	1,500	625	1,250	1,250	1,250	1,000	1,250	1,250	12,500	12,500	6,250	6,250	6,250	7,500
Ni	5,000	12,500	5,000	5,938	6,250	3,755	10,000	8,750	6,250	1,875	15,000	1,875	10,000	10,000	8,750
Si	1,125	2,500	1,250	2,500	500	1,500	500	-	1,250	2,500	3,125	12,500	4,375	2,500	2,500

Table C-4
 PRODUCT PRODUCED, BORON-10 METAL POWDER
 (Batch Weights)

Batch	Total Weight (gms)	Net Boron (gms)	Net Boron-10 (gms)	Cumulative Net Boron-10 (gms)
56	1,254.6	1,199.9	1,088.4	1,088.4
34	1,265.8	1,218.0	1,098.3	2,186.7
54	1,350.4	1,285.7	1,168.5	3,355.2
36	2,004.6	1,916.6	1,716.1	5,071.3
46A	2,361.1	2,305.4	1,996.9	7,068.2
46B	2,092.4	2,012.3	1,805.6	8,873.8
48	2,051.1	1,967.6	1,776.2	10,650.0
51	1,802.5	1,731.5	1,559.8	12,209.8
52	1,611.4	1,548.4	1,397.8	13,607.6
53	1,852.7	1,765.1	1,598.1	15,205.7
49	1,752.4	1,691.6	1,479.1	16,684.8
59A	2,014.8	1,929.2	1,746.7	18,431.5
59B	2,111.6	1,987.4	1,807.4	20,238.9
79A	1,860.9	1,781.6	1,614.3	21,853.2
79B	2,078.2	2,032.7	1,838.4	23,691.6
79C	1,891.4	1,812.0	1,616.0	25,307.6
80	1,756.3	1,683.2	1,528.7	26,836.3
81	2,325.3	2,235.1	2,035.5	28,871.8
82	2,474.4	2,367.0	2,149.7	31,021.5
83	2,301.5	2,207.4	1,999.9	33,021.4
86A	2,364.0	2,257.9	2,050.8	35,072.2
87C	2,357.0	2,259.9	2,049.9	37,122.1
88B	2,295.2	2,209.4	2,005.7	39,127.8
89A	2,759.2	2,672.9	2,425.4	41,553.2
89B	2,844.8	2,725.6	2,475.7	44,028.9
91A	2,666.8	2,570.0	2,335.1	46,364.0
90	1,987.9	1,921.4	1,735.2	48,099.2
90B	1,814.3	1,767.3	1,602.2	49,701.4
95	1,710.4	1,617.4	1,462.9	51,164.3
90A	1,836.1	1,735.7	1,571.3	52,735.6
94B	2,352.8	2,312.8	2,089.2	54,824.8

Unless otherwise specified, the X-ray diffraction patterns on all batches indicate a crystalline structure.