NEW TECHNIQUES FOR PRODUCING THIN BORON FILMS

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ABSTRACT

A review will be presented of methods for producing thin boron films using an electron beam gun. Previous papers have had the problem of spattering of the boron source during the evaporation. Methods for reducing this problem will also be presented.

1. Introduction

The production of thin films of boron, both on substrates such as carbon and those which are self supporting, has always been one of the most difficult challenges facing the "target maker". Some of the earliest work was reported more than fifty years ago. We shall review some of the history of the production of these boron films. Many of those most recently produced are to be used with accelerators such as the ATLAS facility at Argonne National Laboratory. However, in earlier work there was always the problem of sputtering of some of the source material when heated. We will discuss a method for solving this problem.

*Work was done in collaboration with John P. Greene.

2. Early Work

As early as 1934 O'Bryan[1] presented a method of producing thin films of platinum, boron, molybdenum, quartz and other substances by evaporation in a graphite crucible. His experimental apparatus is shown in Fig. 1. The crucible was heated by the bombardment of electrons (up to 4000 volts) from a tungsten filament. A tantalum shield was placed around the filament to reduce heating and deposition on the walls of the vacuum container, which was merely a five liter Pyrex flask. At this early stage, the hazards of radiation from the x-rays from the electrons was already realized as they did not operate above 5000 volts. They found that they could not use the commonly accepted vacuum of 10 Pa but that a vacuum of 10^{-3} Pa was needed. It is interesting to note that at first melting of the source it alloyed with the graphite, producing a thin layer of carbide which prevented further alloying. In some present day experiments, even after the alloying process, there might be an unacceptable amount of impurity produced.

Stafford[2] described a method for producing uniform coats of boron on parallel plate ionization chambers. He had success using a high purity graphite boat. This is one of the only reports of success of resistive evaporation of boron, although it was for a somewhat different application.

Hill[3] produced boron films by a technique similar to that of O'Bryan[1]. His experimental apparatus is shown in Fig. 2. The boron was heated by both electron bombardment and by radiation from the filament, which emitted electrons. The boron passed through a hole on the top of the radiation shield. By this method the evaporation temperature of 2300 degrees C was reached.

Self supporting films were produced by Muggleton and Howe[4] using focused electron bombardment. They found this method superior to induction heating. They used a tantalum crucible for the anode and electrons were emitted from a

3.2 mm x 0.128 mm tantalum filament. A water cooled shield protected the glass bell jar and base plate from the intense heat radiation generated by the evaporation.

Erskine and Gemmell[5] also used electron bombardment to produce their targets as shown in Fig. 3. However, they used a water cooled anode with a slightly concaved surface to reduce the heat generated by electron bombardment. The electrons are emitted from a 0.513 mm tungsten wire filament bent into a circular loop of 7.4 mm d. As a release agent they evaporated a thin film of boron onto a glass slide, exposed it to the atmosphere, and then evaporated the desired thickness of boron onto the slide. The two layers of boron could then easily be separated and the thicker layer floated off in water.

Adair and Kobisk[6] also used electron bombardment with a water cooled anode. However, they used a source consisting of a pellet of boron 12.8 mm d. which eliminated any contamination from the anode. In addition they used an accelerator collimator system which greatly increased their collection efficiency.

Riel[7] and Csihas[8] also presented papers in which they described some of the problems and possible solutions for producing boron thin films.

3. Present Work

In two previous papers by Thomas[9,10] a method was described by which boron targets would be rather routinely produced, both on substrates and self supporting. An older type electron beam gun (a Veeco Ve6 having a water cooled copper source holder) was used for the evaporations. This system, using an electron beam gun and a water cooled hearth, greatly reduced the heat generated from the evaporation as well as increased the purity of the target produced. The system used is shown in Fig. 4.

Four microscope slides which had been previously evaporated with 50 microgram per cm^2 of sodium chloride were placed on a fixture 12 cm from the source. A cold trap, diffusion pump system was used. A vacuum of 10^{-4} Pa or better was necessary to produce the desired high purity targets with low oxygen content.

One of the keys for the production of successful boron targets is heating the glass substrates to 175 degrees C as recommended by Arnison and Gilmore[11]. This minimizes the stress in the film, resulting in a better release from the slide. There is no rolling or breaking up into small pieces in the floating of the foil. A thermocouple placed on the slide determined the temperature of the substrates.

4. Results

In past work we found that use of high purity crystalling boron purchased from Eagle Pitcher Inc.[12] insured that there would be no spitting, sputtering or "blowing up" of the source when it was melted as was the case with other samples. This spitting, etc. often caused pitting, nonuniformity, or destruction of the foil. A new method has been developed to eliminate this problem. This involves resistive heated evaporations. The sputtering may be caused by lower melting impurity materials in the boron which, when the sample is heated, evaporate before the boron.

Materials which have been shown by their sputtering characteristics to have impurities were first heated to 1300 degrees C for at least ten minutes. An optical pyrometer was used to measure the temperature. Their characteristics are shown in Table 1. Most of the resistive evaporations have been successful. In some cases the loss of weight was dramatic and in others it was negligible, indicating the loss may have been from Hydrogen. Care should be used to insure that each sample is in contact with the evaporation boat in order to obtain the

desired temperature. In some cases we may not have heated the boron sample either long enough, hot enough, or both to completely evaporate the impurities out of the sample.

5. Conclusions

Resistive heating of impure boron samples usually evaporates the impurities out of the sample. This may be a useful technique for other high melting materials which contain hard to remove impurities. It should be noted that the newer electron beam gun systems which have more sensitive beam intensity control may make it possible to evaporate out these impurities by first heating the source at a low intensity until they are removed and then increasing the electron beam intensity enough to evaporate the boron.

6. Acknowledgments

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References

1. H.M. O'Bryan, Rev. Sci. Instr. 5 (1934) 125.
2. G.T. Stafford, Rev. Sci. Instr. 27 (1956) 972.
3. H.A. Hill, Rev. Sci. Instr. 27 (1956) 1086.
4. A.H.F. Muggleton and F.A. Howe, Nucl. Instr. and Meth. 13 (1961) 211.
5. J.R. Erskine and D.S. Gemmell, Nucl. Instr. and Meth. 24 (1963) 397.
6. H.L. Adair and E.H. Kobisk, Trans. 9th Nat. Vacuum Symp. (Chicago American Vacuum Society, 1962) p. 125.
7. W.D. Riel, Nucl. Instr. and Meth. 167 (1979) 1979.
8. L. Csihas, Nucl. Instr. and Meth. 167 (1979) 171.
9. G.E. Thomas, Nucl. Instr. and Meth. 177 (1980) 591.
10. G.E. Thomas, Nucl. Instr. and Meth. 200 (1982) 27.
11. G.T.J. Arnison and R.J. Gilmore, Nucl. Instr. and Meth. 45 (1966) 178.
12. Eagle Picher Inc. Miami, Oklahoma 74354, USA.

Figure Captions

Fig. 1. Layout of the experimental apparatus used in 1934 by O'Bryan[1] to produce thin films of platinum, boron, molybdenum, quartz and others.

Fig. 2. Layout of the electron bombardment system used by Hill[3] for the evaporation of boron.

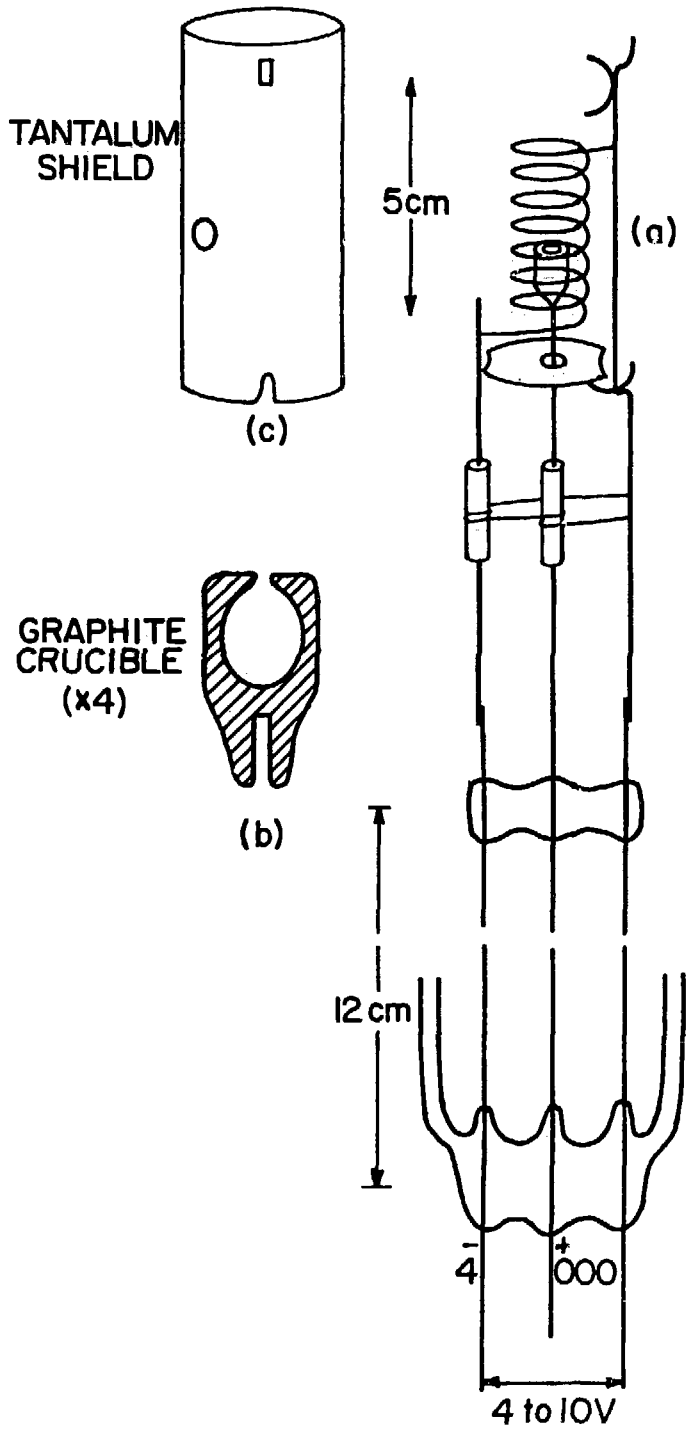
Fig. 3. General layout of the evaporation system by Erskine and Gemmell[5] using electron bombardment and having a water cooled anode.

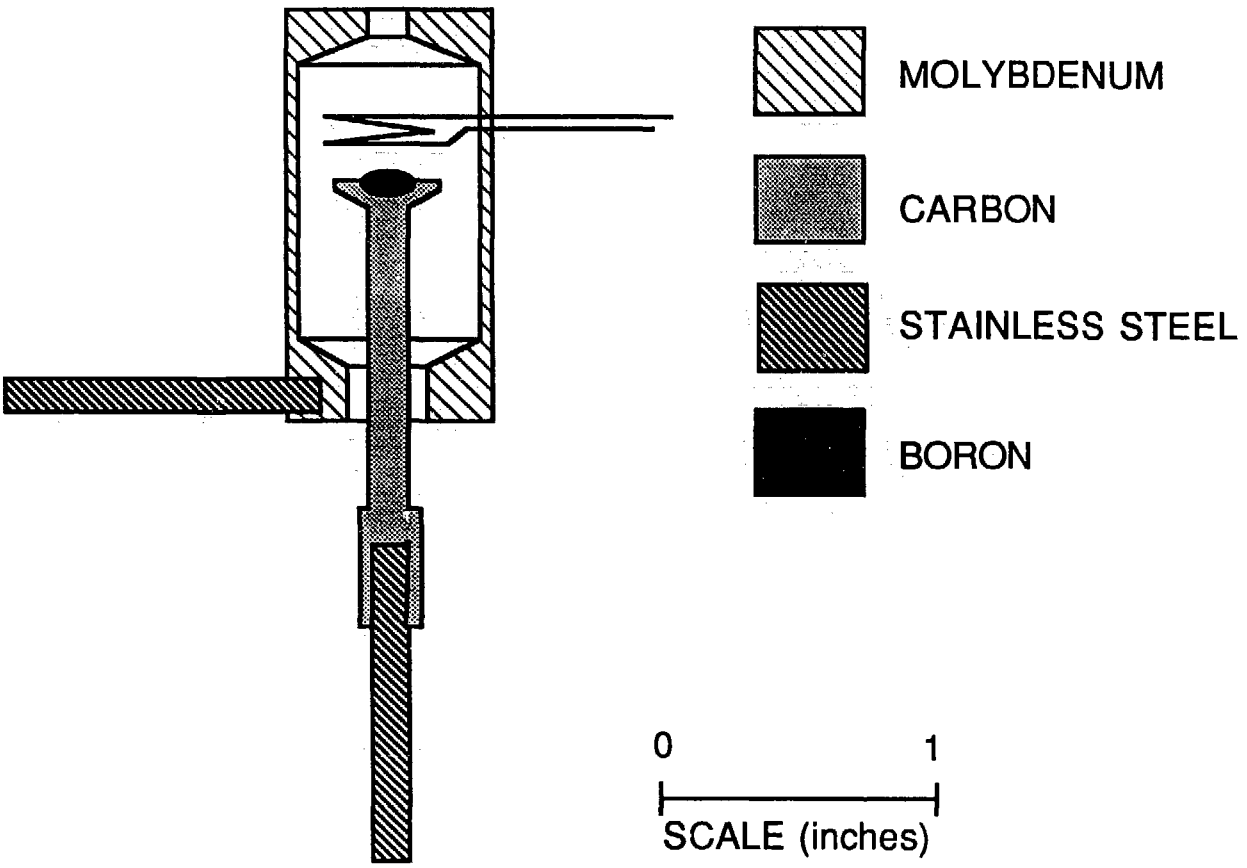
Fig. 4. System for producing stress free self-supporting boron targets. It uses a Veeco Ve6 electron beam gun with a copper water cooled source holder.

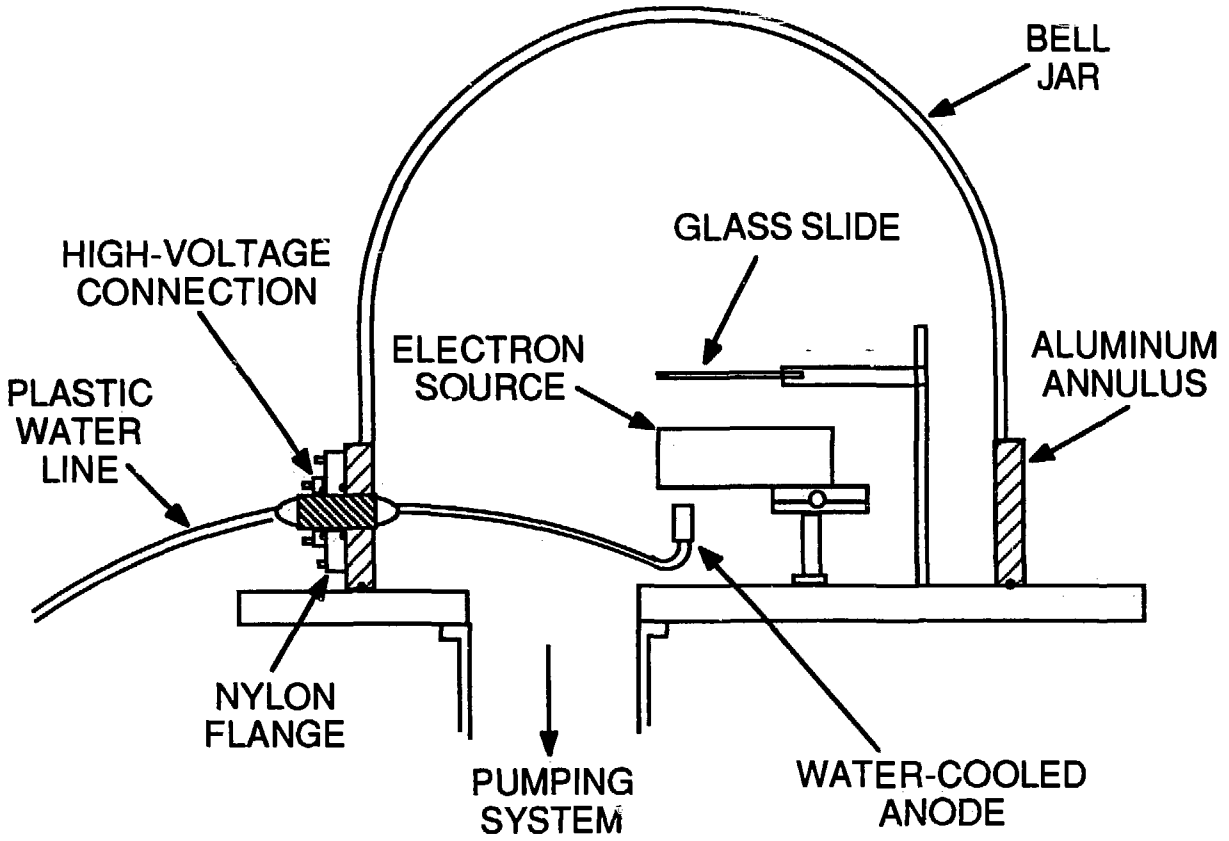
Table 1

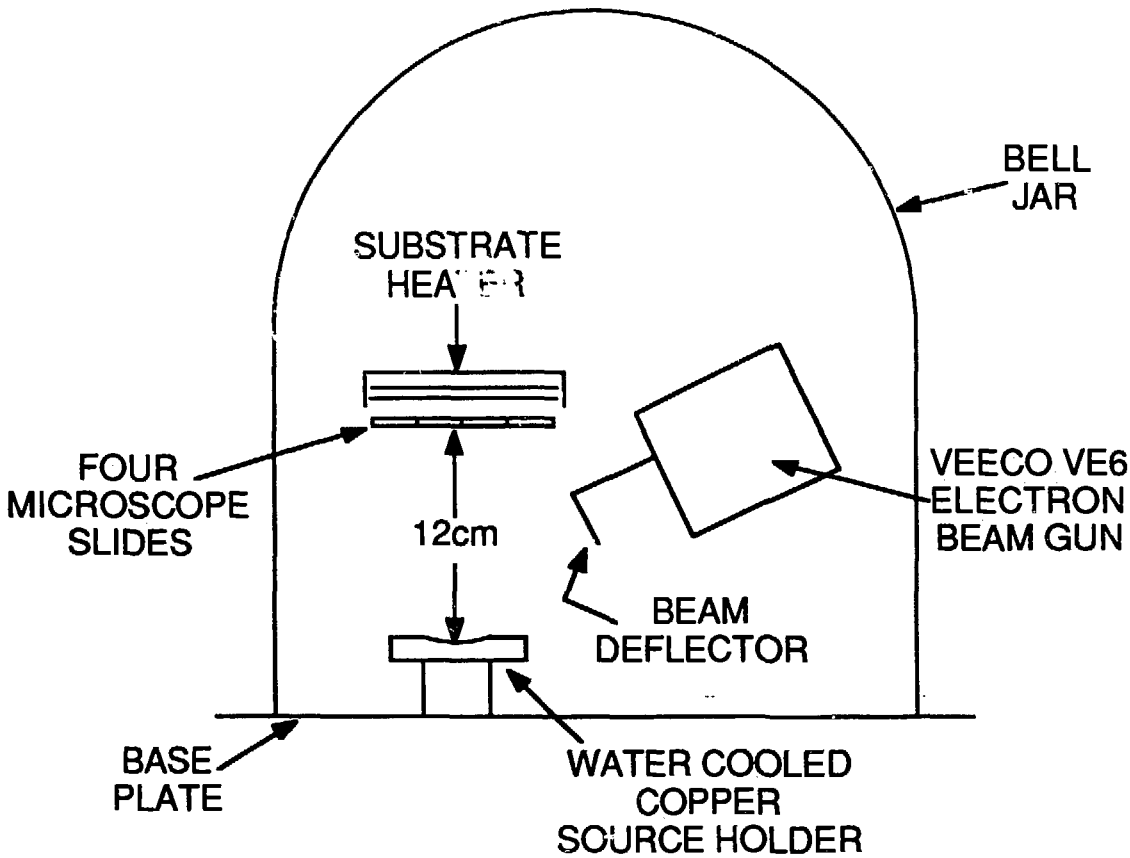
Characteristics and treatment of samples of boron used for electron beam gun evaporation.

Boron Source	Isotope	Form	Sample Heating Temp. (°C)	Sample Heating Time (min)	Original Wt (gms)	Final Wt (gms)
Aldrich	nautral (99.7)	Crystalline chunks	1285	15	266	266
Eagle Pitcher EXP3N-Run 38	natural	chunks	1250	15	442	442
Electronics Spare Products	natural	chunks	1220	15	205	205
Eagle Pitcher QB-58	^{10}B	Crystalline chunks	1500	20	268	268
Eagle Pitcher 5010026	^{10}B 98.59	small granules	1100	35	100	97
Eagle Pitcher 63290059	^{10}B 99.49	powder	1290	10	100	86
UK-20th Century Electronics	^{11}B 92.5	powder	1225	15	101.3	89.2
Eagle Pitcher R7902A0	^{11}B 98.6	chunks	1270	15	299	299
Eagle Pitcher B 11-85-1	^{11}B	chunks	1225	15	382	381


TANTALUM
SHIELD
5cm
(a)
(c)
GRAPHITE
CRUCIBLE
(x4)
(b)
12 cm
4 to 10V


MOLYBDENUM
CARBON
STAINLESS STEEL
BORON
0
1
SCALE (inches)


BELL JAR
GLASS SLIDE
HIGH-VOLTAGE CONNECTION
ELECTRON SOURCE
ALUMINUM ANNULUS
PLASTIC WATER LINE
NYLON FLANGE
PUMPING SYSTEM
WATER-COOLED ANODE


BELL
JAR
SUBSTRATE
HEATER
FOUR
MICROSCOPE
SLIDES
12cm
VEECO VE6
ELECTRON
BEAM GUN
BEAM
DEFLECTOR
BASE
PLATE
WATER COOLED
COPPER
SOURCE HOLDER