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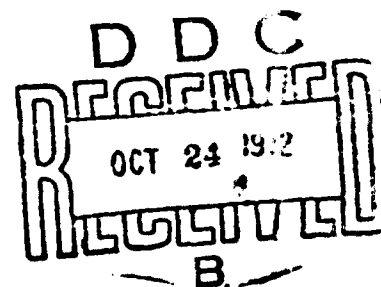
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AMMRC CTR 72-21

MANUFACTURING INVESTIGATION FOR IMPROVED FORMING OF BORON CARBIDE

September 1972

THOMAS VASILOS
AVCO CORPORATION
SYSTEMS DIVISION
LOWELL INDUSTRIAL PARK
LOWELL, MASSACHUSETTS 01851



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MANUFACTURING INVESTIGATION FOR IMPROVED FORMING OF BORON CARBIDE

THOMAS VASILOS
AVCO CORPORATION
Systems Division
Lowell, Massachusetts 01851

SEPTEMBER 1972

Final Report Contract Number DAAG46-72-C-0030
D/A Project 1706061
AMCMS Code 4097.92.O.P6061
Manufacturing Methods for Ceramic Armor Processing

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This project has been accomplished as part of the U.S. Army Manufacturing Methods and Technology Program, which has as its objective the timely establishment of manufacturing processes, techniques or equipment to insure the efficient production of current or future defense programs.

Prepared for

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

FOREWORD

In order to investigate the process for low temperature forming of boron carbide (B_4C), Contract No. DAAG46-72-C-0030 was awarded by the U.S. Army Materials & Mechanics Research Center, Watertown, Massachusetts, to the AVCO Corporation, AVCO Systems Division, Lowell, Massachusetts. Investigation of the process was to develop low temperature forming of improved boron carbide (B_4C) technology with greater cost effectiveness. Tiles 6" x 6" x 3/8" thick, were submitted to AMMRC for ballistic testing and evaluation.

This project was supervised by Dr. S. Dutta of the Ceramics Division, Materials Development Laboratory, U.S. Army Materials & Mechanics Research Center as PEMA Manufacturing Methods and Technology Project No. 1706061.

This project has been accomplished as part of the U.S. Army Manufacturing Methods and Technology Program, which has as its objective the timely establishment of manufacturing processes, techniques or equipment to insure the efficient production of current or future defense programs.

ABSTRACT

Lightweight, high modulus boron carbide was hot pressed to a density > 98 percent of theoretical by alloying additions at a temperature of 1750°C and a pressure of 3500 psi. The additives used were 5 percent sodium silicate and 0.5 percent magnesium oxide, added as magnesium nitrate and the mixture was ball milled with steel balls which introduced some iron in the final composition. Microstructure analysis indicated ultrafine-grained structure with average grain size of 0.55 micron. The process has been scaled up to provide 6" x 6" x 3/8" thick tiles, and eight tiles were supplied to AMMRC for ballistic evaluation, which will be the subject of a separate report.

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I. INTRODUCTION

Boron carbide is a very efficient lightweight armor material against armor piercing projectiles. With a theoretical density of 2.52 g/cc, it is the lightest standard ceramic armor material available. To be most effective, however, boron carbide must be consolidated to a high relative density, greater than 98% of theoretical density. For pure boron carbide, this high density can only be attained by high temperature hot pressing techniques. These techniques are relatively costly, at least twice as costly as silicon carbide - bonded - boron carbide and perhaps more than three times as costly as sintered alumina.

Thus, in order to make boron carbide more available and more attractive for armor protection, modifications to simplify the hot pressing process are necessary. Reducing the hot pressing temperature from the very high temperatures (over 2000°C) currently used to the 1700° - 1800°C region would be a major simplification. This temperature reduction would shorten the hot pressing cycle, would reduce the tendency for boron carbide to react with the mold materials, would allow less insulation, smaller hot pressing furnaces, and lower temperature insulating materials, and would reduce the danger of thermal cracking.

The reduction of hot pressing temperatures is not a new concept as it has been accomplished in other material systems. Significant reductions have been achieved in the densification of alumina, magnesia, and zirconia. In general, these reductions have been accomplished by using pure, reactive, fine (submicron) particle size raw material powders and/or by using densification aids. These approaches have been applied to the current program.

II. TECHNICAL DISCUSSION

A. Densification Mechanisms

In the simplified case, pressureless sintering proceeds via diffusion processes with the driving force normally being the achievement of the lowest surface energy state. Initially, the diffusion is relatively rapid surface diffusion during which contact points between particles grow "necks" and extend the area of contact. This process may continue to some degree until the necks coalesce while particle centers move closer together by volume or grain boundary diffusion. Pore removal or densification most likely occurs via diffusion of matter to the pore surface accompanied by counter diffusion of vacancies from the pore surface. These latter processes are typically much slower diffusion processes than surface diffusion. Although plastic flow sintering models have been evoked for some metallic and ceramic systems, it does not appear to be a likely process for a material with such limited plasticity as B₄C.

The effect of pressure on sintering has been studied by Vasilos and Spriggs^{1,2}. They concluded that stress enhanced diffusion was the principal mechanism operative under moderate pressure in pressure sintering of most ceramics. Other mechanisms which were operative in early and intermediate stages were 1) fragmentation of particles and/or agglomerates, 2) rearrangement of particles during early stages of densification, 3) grain boundary sliding, and 4) plastic flow at particle contact areas.

Additives may bring a variety of mechanisms to aid densification during sintering. A common type of additive which applies in the hot pressing of boron carbide is one which promotes liquid phase sintering. According to Kingery,³ there may be the following effects during liquid phase sintering:

1. A mechanical rearrangement promoted by the formation of the liquid phase and/or by the flow of the liquid, and
2. A solution-precipitation mechanism which enhances diffusion by transport of the sintering material as a solute in the liquid phase.

Reaction sintering occurs when a synthesizing reaction takes place during sintering and the sintering process is enhanced thereby. The ultimate in reaction sintering is to sinter the elemental ingredients in the proper proportions; however, reaction sintering where only a small fraction of the total material volume is utilized is suitable as long as it forms the desired product during sintering. Sintering is enhanced by the formation in situ of the desired product at rates that may be significantly faster than self-diffusion in the final compound. In some cases, particularly those involving a gas, the reaction fills voids resulting in little shrinkage during the reaction-densification process. An additional aid to the process is the release of the heat of formation of the product. Thus, the addition of elemental boron and carbon could cause beneficial reaction sintering effects in attempting to lower the hot pressing temperature.

B. Boron Carbide Synthesis

Boron carbide powder is synthesized commercially by two essentially different ways.

The arc melt fusion process uses an electric arc furnace to react, melt and fuse a complete furnace charge of boron carbide. Basically, boric acid, carbon, and a flux are gradually added to the arc furnace. The electrodes are withdrawn as the boron carbide is formed, fused and solidified. The boron carbide product is removed from the furnace as large chunks which are crushed and graded using ordinary abrasive manufacturing technology. The product is normally boron deficient and not suitable for hot pressing. Special furnace runs are necessary to produce the high boron boron carbide for hot pressing. A powder particle size of one to five microns required for hot pressing is generated by comminution. The powder is then leached to remove iron contamination followed by a sequential settling to achieve particle size splits. This powder is typically characterized by poor reactivity due to the stability of fused material, high free carbon content (5 to 10 w/o), and high iron content (0.3 to 1.0 w/o), which is not easily removed from fine powders in the leaching step. In addition, a submicron average particle size is difficult to achieve even with new comminution techniques such as fluid energy milling or planetary milling.

Another commercial process uses magnesium to reduce the boron oxide in the presence of carbon at relatively low temperatures. After the reaction is complete, the magnesium oxide is removed by a leaching process, the powder

is then washed and dried. This process makes it possible to obtain a significant amount of material in the sub-micron size range.

C. Hot Pressing Procedures

For the current program, two hot pressing designs were used. One was a standard cylindrical 2" inside diameter mold for rapid evaluation of powder treatments and additives. In the contractor's hot press laboratory, the 2" mold is used in a 75-ton automatic air hydraulic hot press. This hot press has its own 50 KW power supply and is fully instrumented with dial gauge for measuring piston movement and optical pyrometer for measuring temperature.

The hot pressing set-up for the six-inch square plates consists of a sectional square mold of schematic design shown in Figure 1. A schematic of the powder charge, surrounding graphite, and parting agents is shown in Figure 2. This mold set-up is installed in a furnace of design shown in Figure 3. All graphite in the pressure train, including mold top, bottom, and I.D. is carefully machined flat, parallel, and/or perpendicular so that no excessive stresses are developed in the graphite. The furnace and mold may be located in either a 400-ton or a 1000-ton press located in Avco's hot press pilot plant. These presses are actually oversized for the current program. The power supply is an Inducto Tri-Line unit of 400 KW. These presses are also automatic in maintaining a preset load independent of platen movement. Dial gauges and pyrometer for piston movement and temperature measurement are also employed during runs.

The temperature is measured in the mold body at the bottom of the sight hole. The sight hole is located as near to the mold charge as possible for accurate temperature measurements.

The powder is very carefully loaded into the mold and levelled to avoid density variation within the pressed shape. The mold is then closed and pressure (a few tons for the 6" square mold) is applied to set the powder at room temperature and reduce the piston movement during hot pressing. Normal practice is to apply a nominal holding pressure (500-1000 psi) during heating. The temperature is increased as rapidly as possible reaching 1750°C well under an hour for the 6" square mold and hot press set-up. At a pre-determined temperature, such as 1400°C for a 1750°C hot pressing, e.g., at a rate of about one ton per minute, the maximum temperature and pressure are reached at about the same time. Temperature and pressure are held until a pre-determined compression rate is achieved (zero for small pressings and up to 5 mils/15 minutes for pressings 6" square and over, and then they are held for an additional period of 15 minutes to insure density. Total holding time at temperature and pressure is 4 hours. The pressure and power are then removed and the furnace is allowed to cool.

The above procedure represents the practice used for the boron carbide pressings.

D. Low Temperature Hot Pressing

The goal of the proposed program was to achieve 98% dense boron

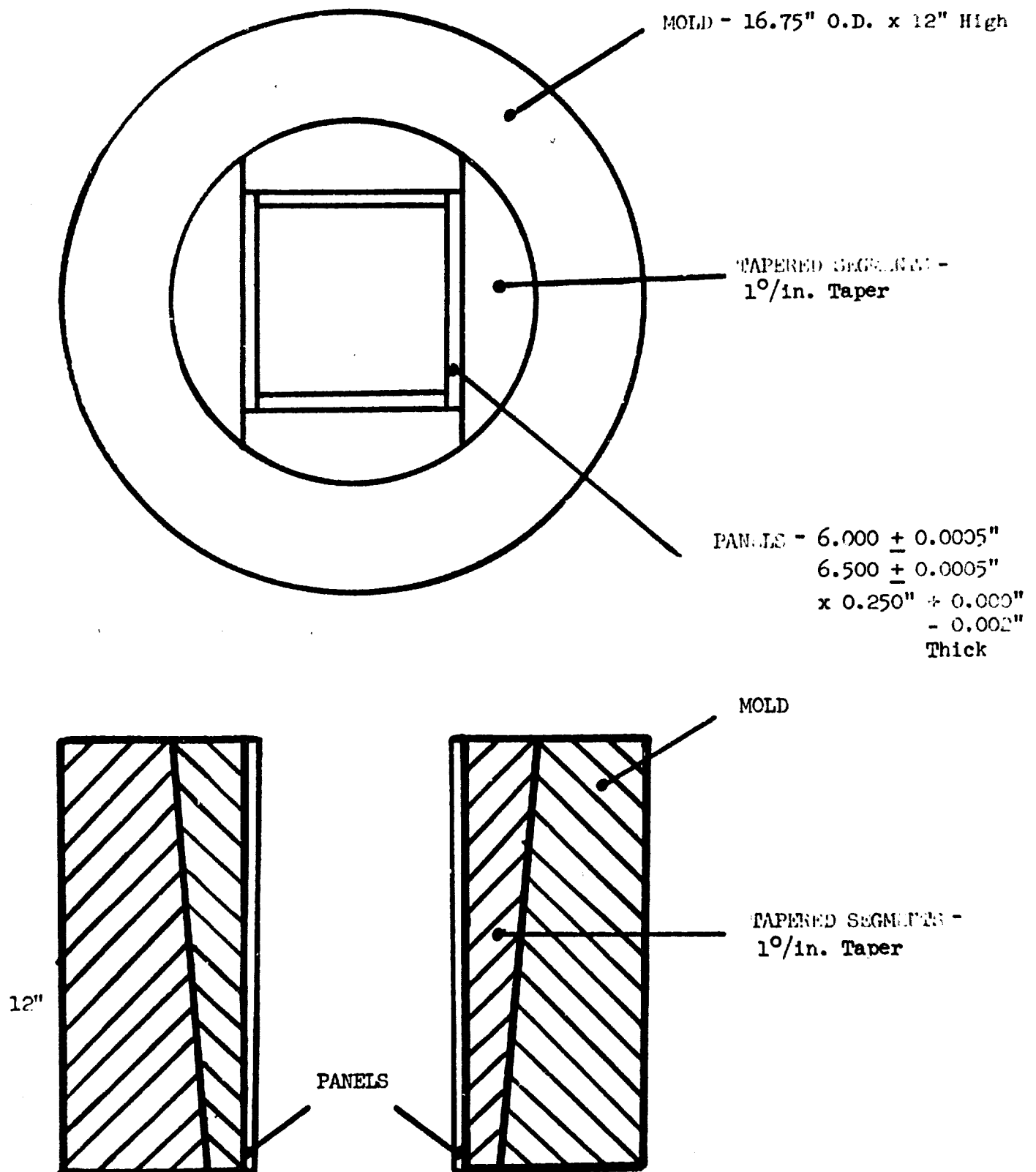


FIGURE 1.

MOLD DESIGN FOR 12" SQUARE PRESSINGS

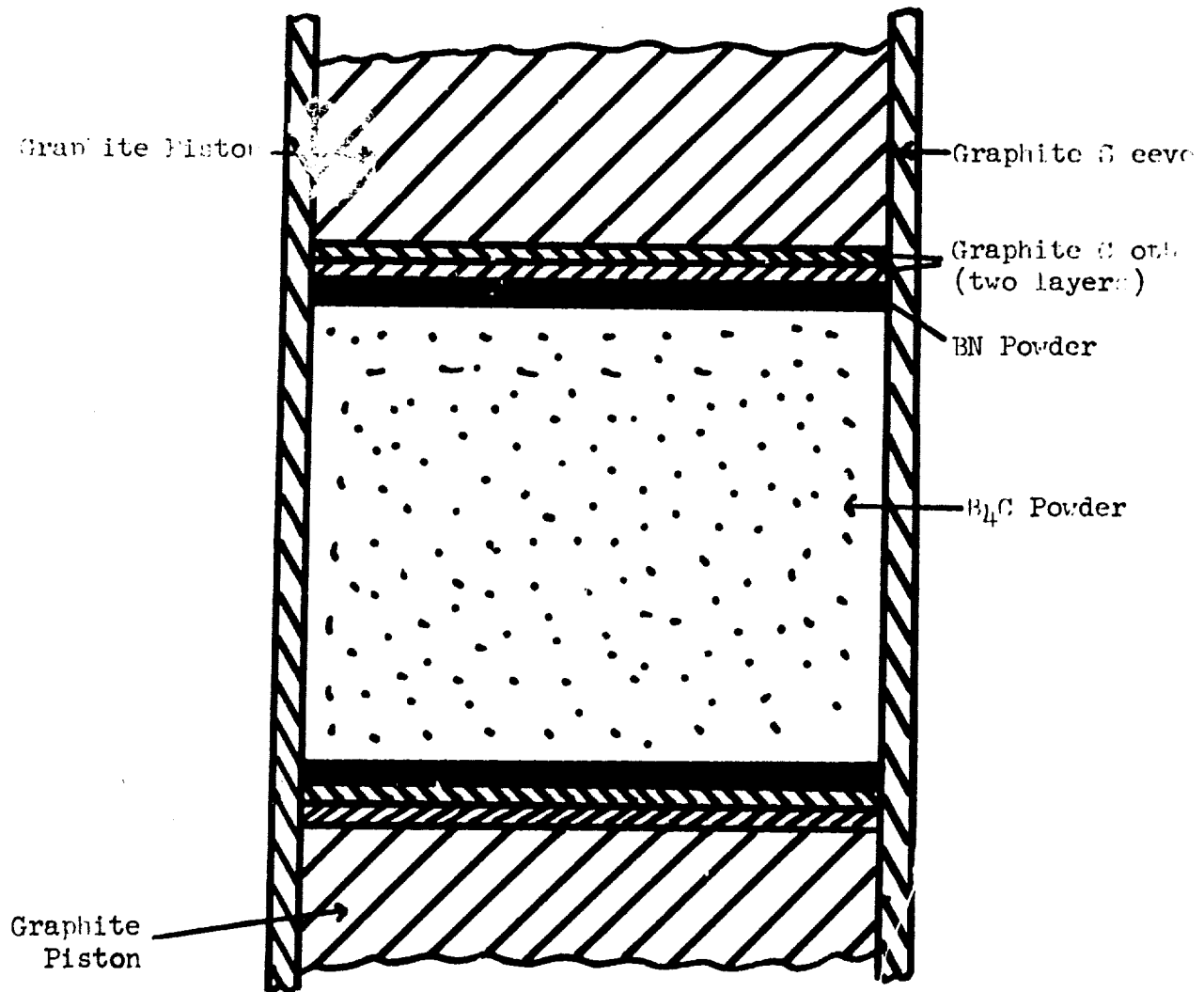


Figure 2.

Schematic illustration of diffusion barriers for prevention of reaction between boron carbide and piston faces.

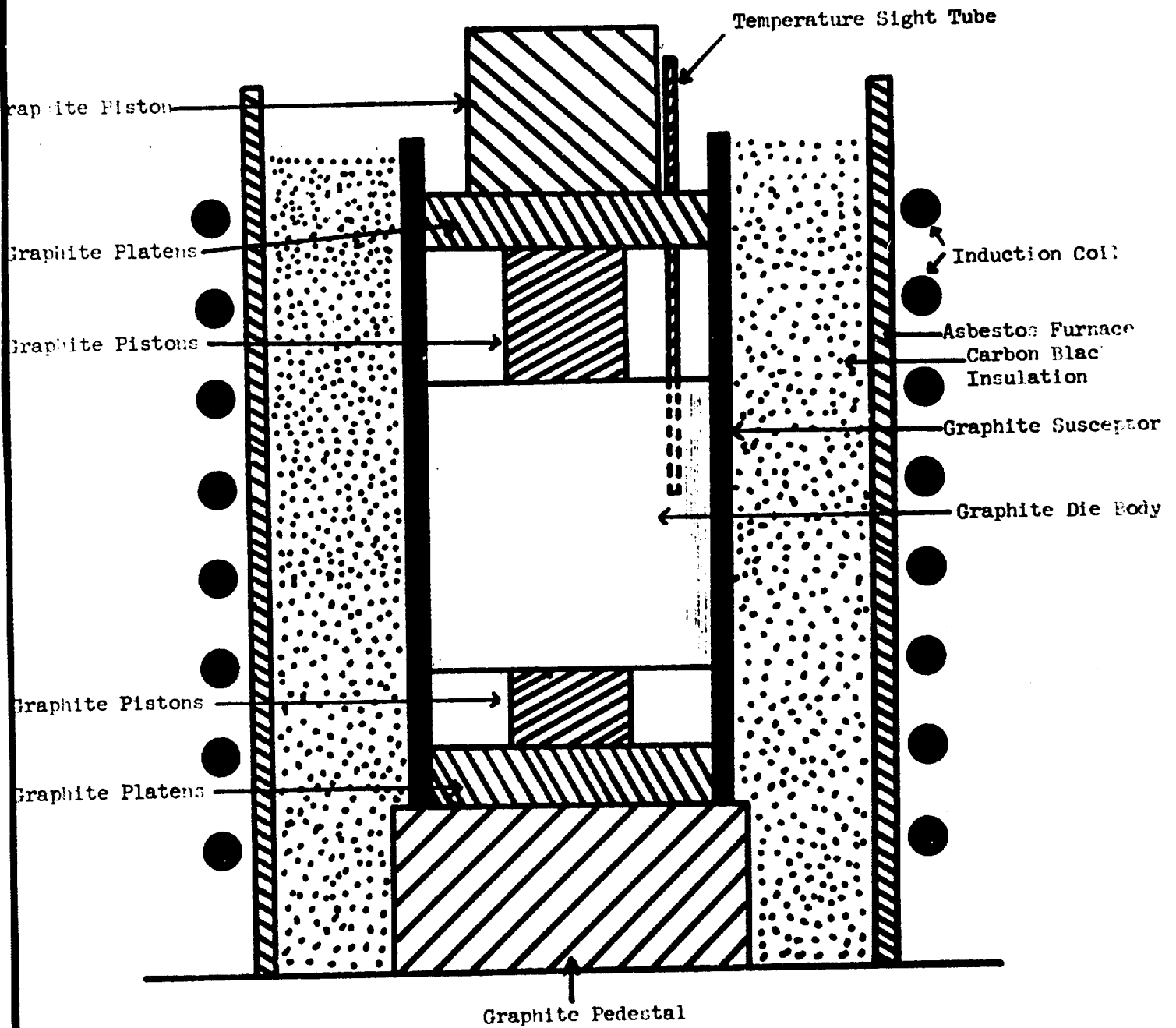


Figure 3.

Schematic Illustration of Hot Pressing Assembly

<u>Name</u>	<u>Designation</u>	<u>Dimensions</u>
Coil O.D.	A	28"
Coil I.D.		25"
Asbestos Furnace O.D.	B	24"
Asbestos Furnace I.D.		22"
Graphite Pedestal	C	19" diameter x 6" high
Graphite Platens	D	16½" diameter x 2" high
Graphite Pistons	E	6.000 + 0.000" x 6.000 + 0.000" x - 0.002 - 0.002 8" high
Graphite Susceptor	F	17.00 ± 0.020" I.D. x 18" O.D. x 28" high
Graphite Mold	G	16.75 + 0.000" O.D. x 12" long - 0.020
Follower Piston	H	8" diameter x 6" long

carbide by hot pressing at less than 1700°C and 2500 psi. The contractor was able to densify boron carbide to $>98\%$ density by employing a temperature of 1750°C and a pressure of 3500 psi for a period of 4 hours.

Initial experiments employing >1 micron average size particulate B_4C were unsuccessful in reducing the densification temperature below 1800°C with moderate pressures ~ 5000 psi. The best results were obtained by employing B_4C from Boride Products Co. with particles 95% finer than 1 micron, as shown in Figure 4. An electron micrograph of this powder grade is shown in Figure 5. The structure reveals some agglomeration.

Without densification aids and agglomeration break-up, the best density obtained at 2500 psi and 1900°C was 2.11 or 84% of theoretical.

Approximately 30 small scale two-inch diameter hot pressing runs were made to evaluate the effects of various additives on both the coarse and finer sized B_4C powders. (Cold pressing prior to hot forming was not employed.) These included alumina, silica, iron oxide, magnesia, sodium silicate, and titania, some in combination. In each case, densification was enhanced by the presence of these additives which developed liquid phases at hot pressing temperatures. The optimum combination to date resulted from combination of long ball milling (rubber lined steel mill) (75 hrs.) with 5% sodium silicate, and 0.5% MgO added as magnesium nitrate. The ball milling served to break up the agglomerate structure (Figure 6) and to introduce finely divided iron additive as a principal densification aid. The iron content introduced by this procedure was estimated to be over 5%.

Temperatures as low as 1750°C and pressures of 3500 psi with a 4-hour soak at temperature and pressure provided samples with densities in excess of 2.5 gms/cc. Electron fractographs taken at 1500X and 7500X show a dense fine grained structure (Figures 7 and 8) (average grain size of .55 micron).

X-ray diffraction analysis (Figure 9) of such hot pressed specimens reveals identifiable impurities as principally iron boride (FeB) in concentration $>5\%$, and a F.C.C. spinel, probably magnesium aluminate and silicon carbide. There is also a trace of iron silicide either FeSi or FeSi_2 .

The total impurity content is probably of the order of 10% and requires further quantitative characterization.

The starting boron carbide powder from Boride Products Co. was known to contain some F.C.C. spinel (magnesium aluminate) to begin with, as well as silicon and some free carbon (1-3%). A chemical analysis is tabulated below in Table I.

The process has been scaled up to provide several 6" x 6" x 3/8" thick tiles. Their densities were each better than 2.5 g/cc. The hot pressing process itself has previously been described in Section C.

TABLE I

Analysis of B₄C Powder from Boride Products

<u>Element</u>	<u>Amount</u>
Boron	77.0%
Carbon	22.15%
Aluminum	0.1%
Magnesium	0.2%
Silicon	0.1%

III. SUMMARY AND CONCLUSIONS

The objective of the proposed program was to achieve essentially theoretical dense boron carbide by hot pressing at a temperature not exceeding 1700°C and a pressure of 2500 psi. The contractor was able to densify boron carbide to > 98 percent density at a temperature of 1750°C and a pressure of 3500 psi. The reduction of required pressing temperature is a significant accomplishment even though the use of slightly higher pressures are required. It is anticipated that the cost effectiveness can be attributed mainly through the reduction of fabrication temperature (than pressure) due to the following reasons:

1. Improved mold life due to low temperature processing;
2. Reduction of total cooling time cycle and less power consumption;
3. Lower oxidation rate and minimized reaction with the sintering compositions;
4. Use of less expensive dies and fixtures; and
5. Better process control and reduced scrap rates.

The contractor has supplied eight 6" x 6" x 3/8" thick tiles which were scaled up in this development program. The ballistic evaluation, to be published separately, was made by Dr. S. Dutta at the Army Materials and Mechanics Research Center's Ballistic Range. The result is particularly noteworthy, since in spite of alloy additions and impurity content ranging between 5 and 10 percent, the ballistic performance is almost equivalent to that of presently available standard armor grade boron carbides when examined under identical conditions.

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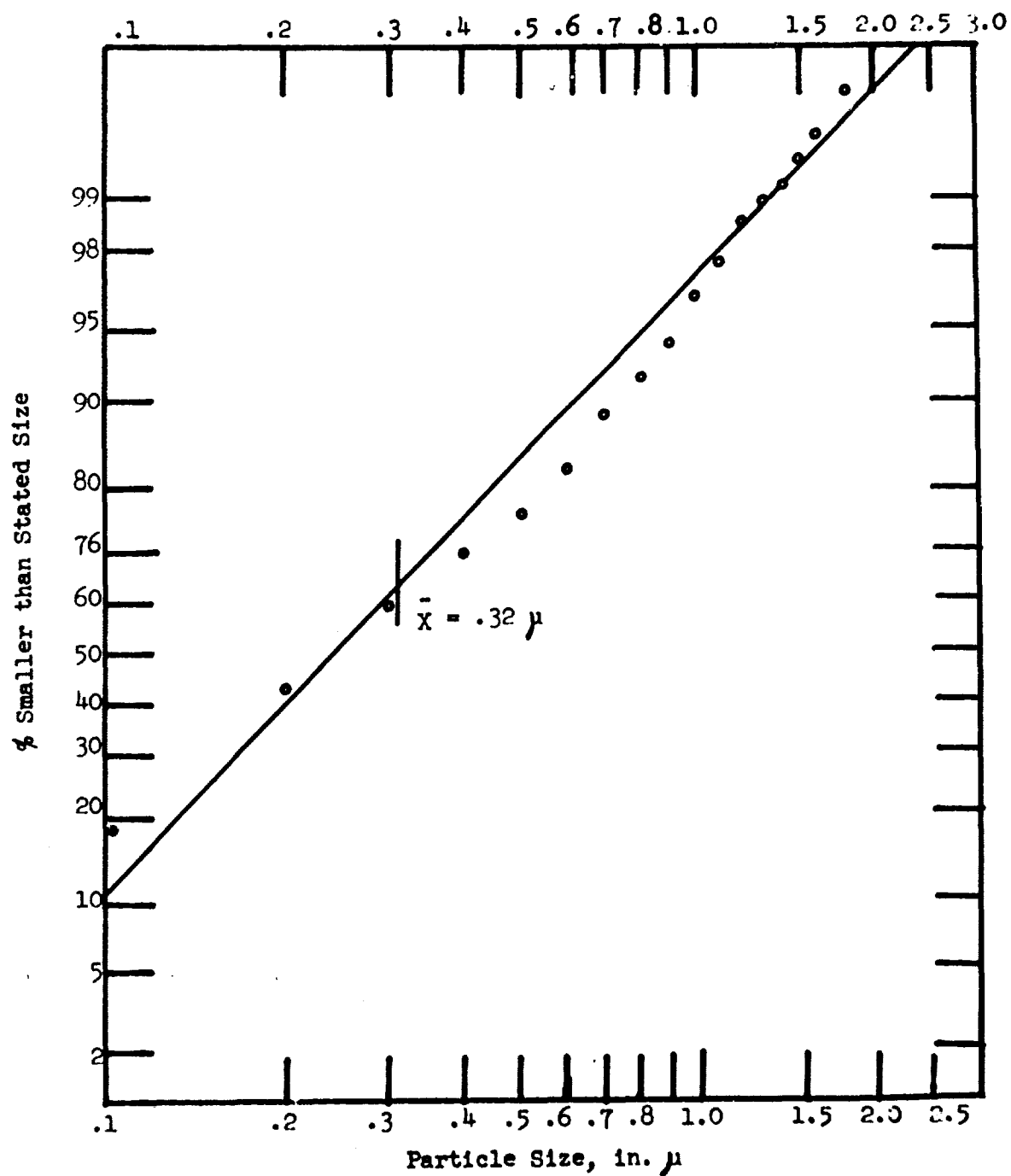
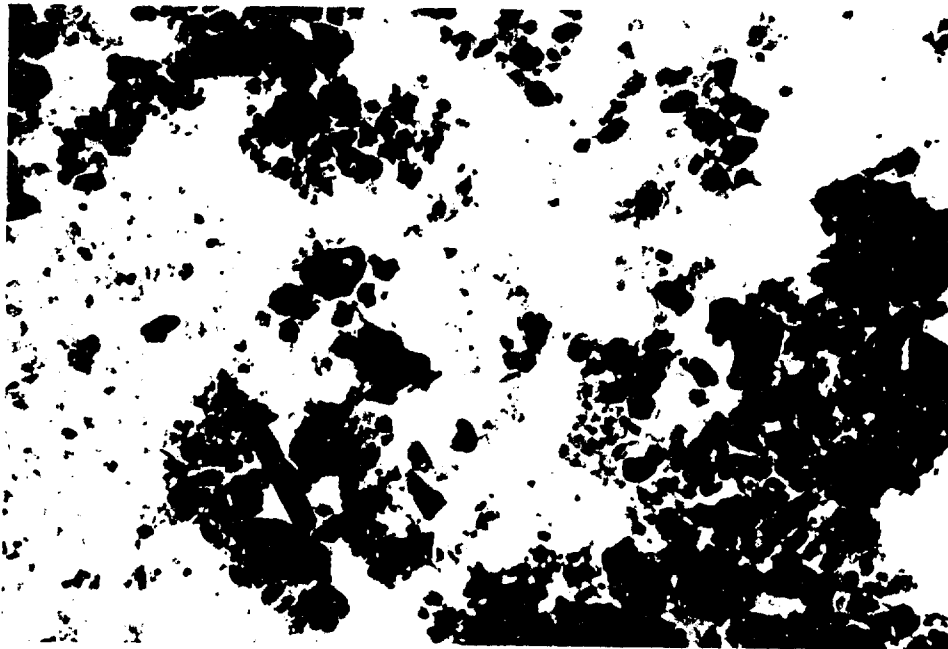


Figure 4. Size Distribution for Fine Particle B₄C

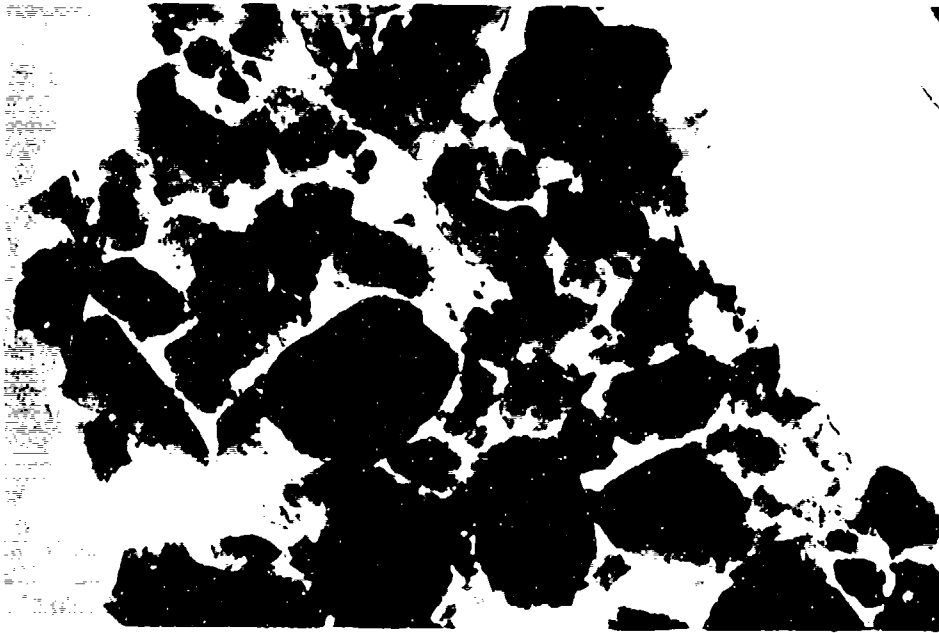
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Figure 5. Electron Micrograph of Fine Particle B_4C

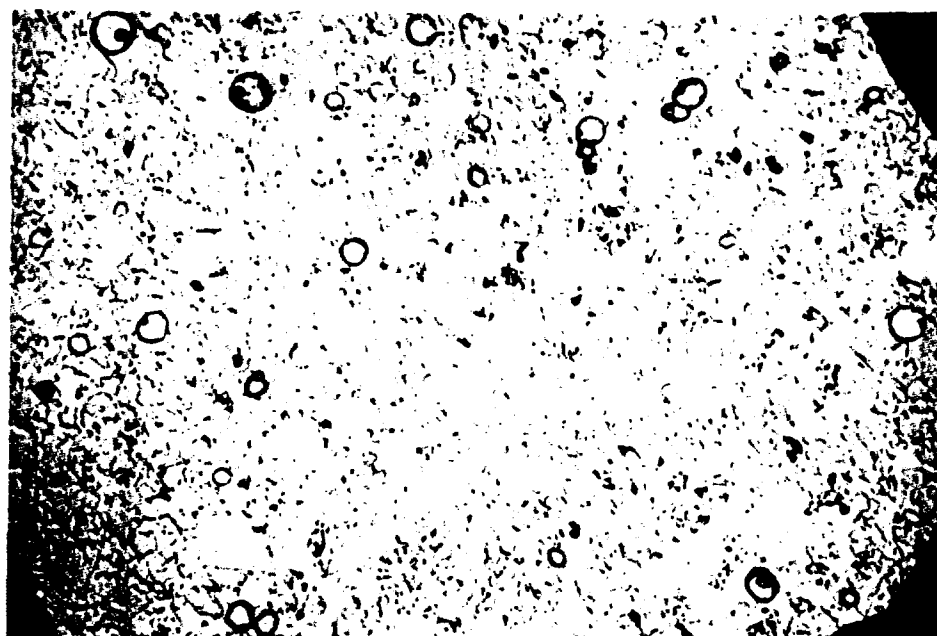


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Figure 6. Electron Micrograph of Fine Particle B_4C After Milling

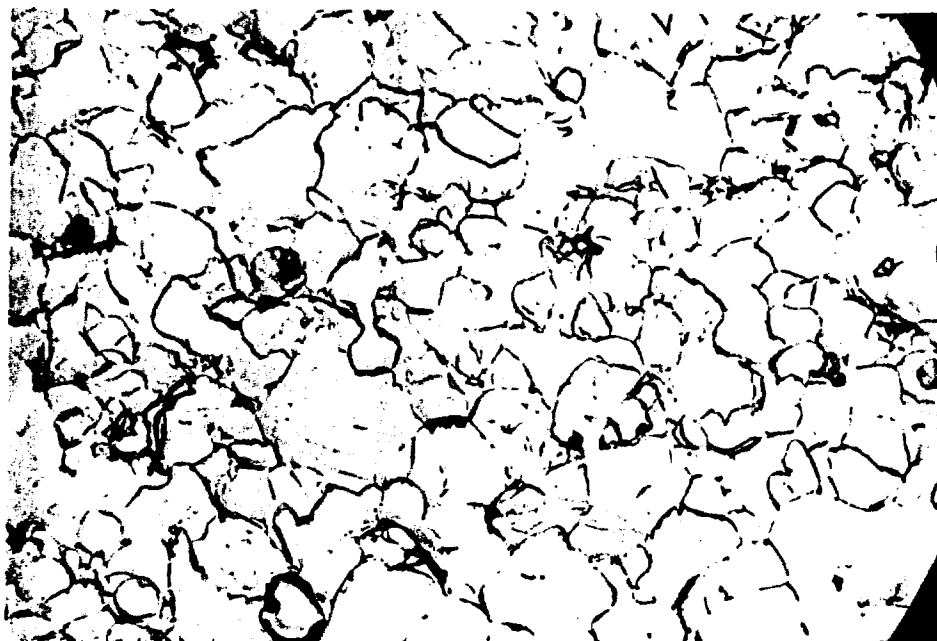
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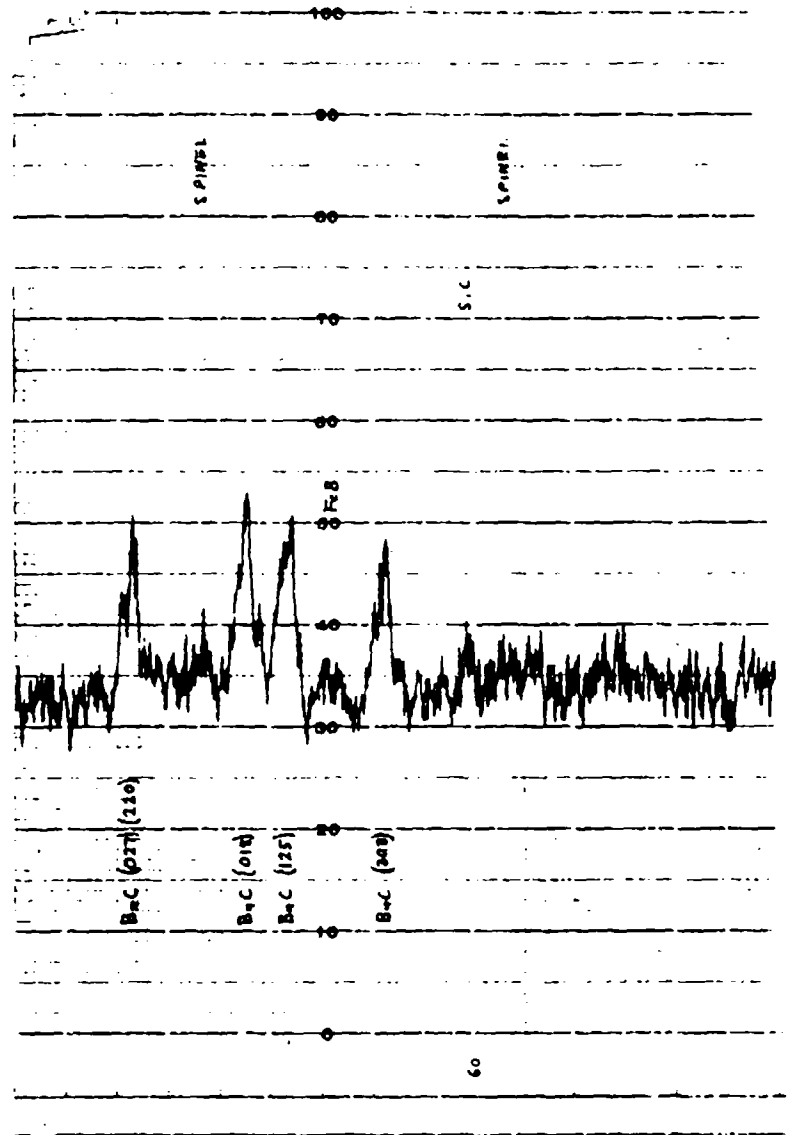
Figure 7. Electron Microscope Fractograph of Fine Particle B_4C Hot Pressed at $1750^{\circ}C$ and 3500 psi.



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Figure 8. Electron Microscope Fractograph of Fine Particle B_4C Hot Pressed at $1750^{\circ}C$ and 3500 psi.



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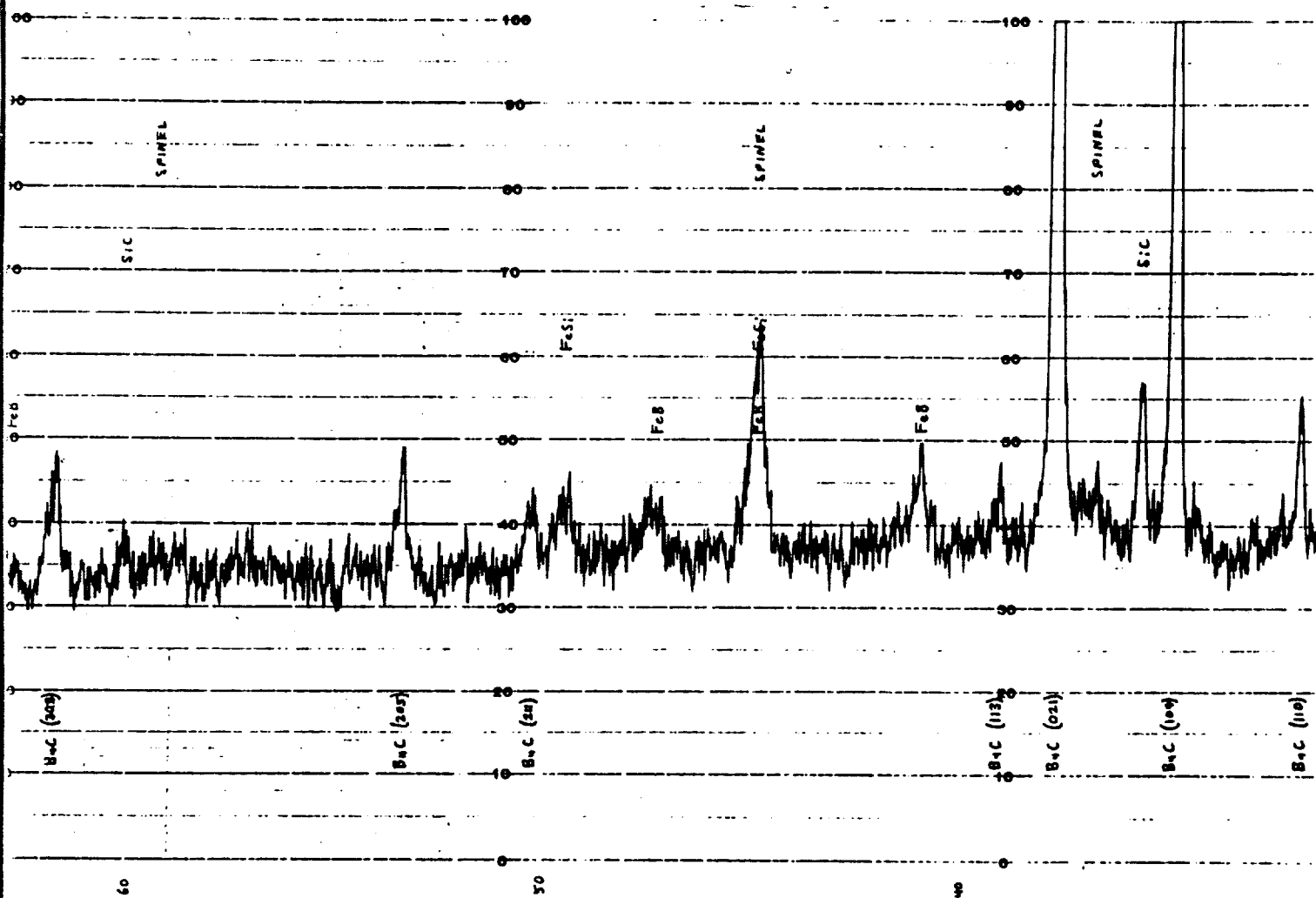


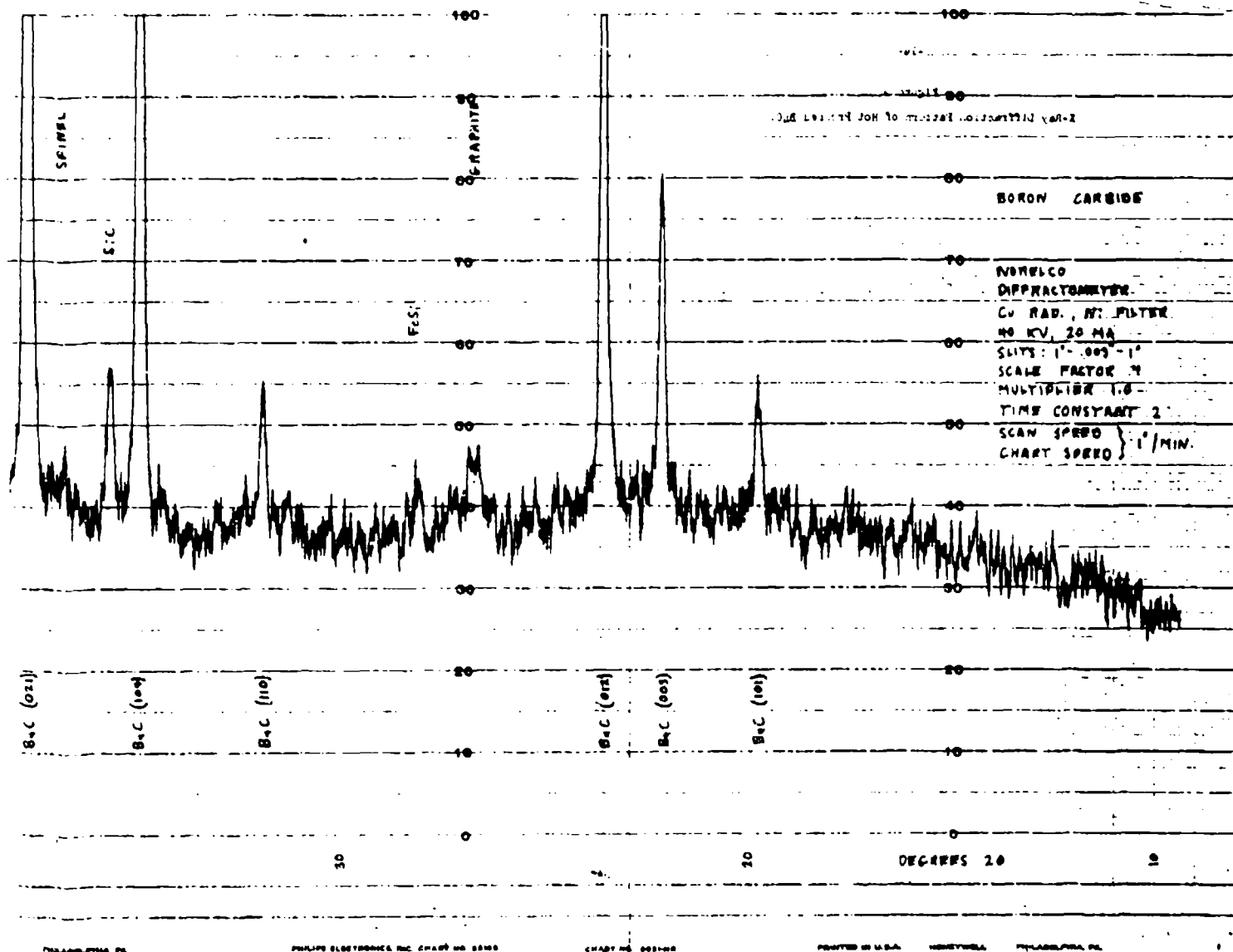
Figure 9.

X-Ray Diffraction Pattern of Hot Pressed B₄C

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Army Materials and Mechanics Research Center, Watertown, Massachusetts 02172
MANUFACTURING INVESTIGATION FOR IMPROVED FORMING OF BORON CARBIDE
Thomas Vasilos, AVCO Systems Division, Lowell, Massachusetts 01851
Technical Report AMMRC CTR 72-21, September 1972, 19 pp - illus - tables, Contract DAAG72-C-0030
D/A Project 1706061, AMCMS Code 4097.92.0.P6061
Final Report, September 1972

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Key Words

Boron Carbide
Hot Pressing

Lightweight, high modulus boron carbide was hot pressed to a density > 98 percent of theoretical by alloying additions at a temperature of 1750°C and a pressure of 3500 psi. The additives used were 5 percent sodium silicate and 0.5 percent magnesium oxide, added as magnesium nitrate and the mixture was ball milled with steel balls which introduced some iron in the final composition. Microstructure analysis indicated ultrafine-grained structure with average grain size of 0.55 micron. The process has been scaled up to provide $6" \times 6" \times 3/8"$ thick tiles, and eight tiles were supplied to AMMRC for ballistic evaluation, which will be the subject of a separate report.

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