



UNCLASSIFIED

-2- ABSTRACT

Methods of consolidating boron and boron carbide have been investigated. For production fabrication of boron, the hot-pressing method appears best. Boric anhydride and lead borate glass work well as bonding agents. Plastic bonding also has advantages as a method of consolidation.

  
UNCLASSIFIED

UNCLASSIFIED

-3-

THE CONSOLIDATION OF BORON

The experiments outlined in this report represent some attempts to consolidate boron into (such compacts, as would be suitable for use by the ordnance division.) The requirements for such a piece are first, that it must have a high boron concentration; and secondly, that it must possess the desired physical properties. In addition, any method of fabrication that can produce the desired piece must be readily convertible to production practice.

Because of the lack of pure boron at the start of these experiments, an appreciable amount of work was done with commercial grades of amorphous and crystalline boron of rather low purity. In other instances, (when the ordnance group required pieces for field use,) the limited amount of commercial boron made it necessary to use a substitutional material whose properties, in general, would resemble those of boron. In this case boron carbide was selected for the fabrication of large numbers of pieces of appreciable size. The rather urgent need for information on the consolidation of boron and boron carbide resulted in a series of rather hurried experiments in an effort to obtain suitable methods; hence much of the work is lacking in extended detail and many of the processes can be further refined to a considerable degree.

In contemplating methods of consolidation it is of interest to consider the different types of mechanisms by which bonding will occur. There are, presumably, three types of bonds: the film bond, the chemical bond, and the self-sintered bond.

The film type of bond is that in which the bonding agent is inert to the matrix substance but forms a film about the particles to be consolidated. The strength of the bonded compact is closely allied with the strength of the bonding

UNCLASSIFIED

UNCLASSIFIED  
[REDACTED]

-4-

material. In effect, this type of bond merely fills the voids in the compact; hence, the minimization of porosity in the compact by control of the grain size of the bonded material is of important consequence.

The chemical or binary type is that type of bond in which the bonding agent combines either chemically or in a solid solution with the matrix material. This type of bond is likely to give a greater ultimate density in the compact than will the film type of bond; however, in metal systems there is likely to occur a brittle phase which seriously affects the mechanical properties of the compacted pieces.

The self-sintered bond is that type of bond wherein the particles of the bonded substance exhibit plasticity below the melting point and consolidation occurs by the adhesion of the separate particles to one another. The highest densities of the bonded material are obtained by this method and there is the added advantage that no foreign substance is introduced. The physical properties of the compact are those of the matrix substance itself.

Metal bonds were attempted with no success; impregnation methods appeared favorable but did not lend themselves very well to production practice; and finally, hot-pressing methods with different bonding agents proved to be the most profitable from the standpoint of fulfilling the requirements. All of the sundry attempts at fabrication are discussed in some detail in this report.

#### A. METAL BONDING OF BORON

The first attempts to consolidate boron in these experiments were made by using metal powders as bonding agents. The commercial grades of crystalline and of amorphous boron were cold compacted with 10 wt. percent of sundry metal powders, and then subjected to a sintering treatment in a hydrogenous atmosphere.

[REDACTED]

UNCLASSIFIED

In all cases the results were negative; there was no evidence whatsoever of any tendency of the metals to bond the boron into a dense, mechanically strong compact.

Materials: boron, amorphous - (commercial) - boron below 80 percent

- balance, Mg, O, Si, Ca, etc.
- extremely fine powder
- loose pack density - 0.29 g/cc
- melting point, about 2350° C

boron, crystalline - (commercial) - boron about 63 percent

- balance mostly Al and O
- presumably  $AlB_{12}$
- coarse grain size, 65 - 100 mesh
- loose pack density - 1.1 g/cc

metal powders - commercial grades of Fe, Ni, Co, Mn, Cu, Si.

Experimental Procedure: Forty gram lots of amorphous and crystalline boron were ball milled (iron mill and steel balls) for five hours with ten wt. percent of each of the metal powders: iron, nickel, cobalt, copper, manganese and silicon. Sufficient naphtha was added to make a smooth paste. After the ball-milling operation the mixture was dried and a portion of it charged into a 1/4" steel die and compressed at 50,000 psi.

Sintering was accomplished under an atmosphere of dry hydrogen in a Burrell horizontal-tube globar furnace. Tank hydrogen was passed over heated copper chips, bubbled through concentrated sulphuric acid, and finally passed through a column of drierite before entering the furnace.

The compacts were placed in a porcelain boat and introduced into the furnace tube. The system was thoroughly flushed with dry hydrogen before the furnace was turned on. The compacts were sintered for five hours at 1325° C and then allowed to cool to room temperature in the hydrogen atmosphere.

In every case, there was no evidence of sintering and the compacts did not gain in mechanical strength. Both the amorphous and crystalline boron compacts were too weak to permit handling after treatment and those few that did hold together showed no improvement over the unsintered compacts.

Remarks: These preliminary tests gave such unsatisfactory results that this process was abandoned temporarily. At a later date, when a purer boron was available, a study of hot pressing with metal bonds was made.

#### B. IMPREGNATION WITH FUSED BORIC ANHYDRIDE

After preliminary trials of metal bonding of boron had proved unsuccessful, effort was directed to other types of bonding agents. Boric oxide was selected as a possibility. It is a desirable substance since in itself it possesses good mechanical strength; and secondly, the glass contributes approximately one third of its weight as boron atoms. The latter fact is significant in that a high concentration of boron is desired in the finished piece.

The results obtained from these experiments indicate that fused boric oxide is a good bonding agent for boron. It forms a tenacious film between the boron particles and provides a bonded compact with relatively high compressive strength. The process of impregnation has, however, certain disadvantages. The

~~SECRET~~

-7-

soaking time at temperature is a function of the cross section of the compact and the time element involved with the soaking of large compacts is undesirable. Secondly, any cracks in the cold compact (usually formed on ejection from the steel die) are greatly exaggerated during the soaking operation. The cracks enlarge and become filled with the oxide, making a nonhomogeneous piece. Finally, there is the difficulty encountered in removing surplus boric oxide from the outer surfaces of the compact after the impregnation has been completed. This is especially true of intricate sections.

Materials: boric oxide -  $B_2O_3$  - (commercial)

density - 1.84 g/cc

melting point - Ca 600° C

boron, amorphous - (commercial) - as in section A

boron, crystalline - (commercial) - as in section A

Experimental Procedure: The cold compacts of amorphous and crystalline boron in preparation for impregnation were made by compression molding in a 1/2" diameter steel die at 50,000 psi.

Impregnation of the boron compact was accomplished by immersing the piece into a bath of molten boric oxide at about 800° C, slowly bringing the bath to 1450° C, and allowing the compact to soak the required time. For the 1/2" diameter compacts the soaking time amounted to 1/2 hour. The salt was contained in a graphite crucible with a suitable lid for pyrometer sighting. The entire assembly was heated by induction. After the required treatment the compacts were removed from the bath, the excess oxide was scraped from the surfaces, and the compacts were allowed to cool in air.

~~SECRET~~

-8- ~~SECRET~~

Table I lists the average values for the amorphous and crystalline boron impregnations of the 1/2" compacts. Density determinations were made by the immersion method in bromobenzine.

TABLE I

| Boron       | Cold Compact Density g/cc | Density after Impregnation g/cc | Wt % B <sub>2</sub> O <sub>3</sub> Absorbed | Compressive Strength psi |
|-------------|---------------------------|---------------------------------|---------------------------------------------|--------------------------|
| Amorphous   | 1.3                       | 2.1                             | 45                                          | 20,000                   |
| Crystalline | 1.8                       | 2.4                             | 29                                          | 60,000                   |

Remarks: As suggested before, this process, although it does not lend itself well to production methods, illustrates that boric oxide makes a very desirable bonding agent for boron. Use is made of this fact in a later process of hot-pressing boron with boric oxide.

It might be mentioned here that boric anhydride has a fairly strong affinity for water and will, if exposed to the air, absorb a fair quantity of water vapor. A lead borate glass was later used to overcome this effect.

#### C. HOT-PRESSING OF BORON CARBIDE

The limited amounts of the amorphous and crystalline boron available made it necessary to seek a substitutional material for the fabrication of test pieces for the ordnance group. Boron carbide was selected as having physical properties approaching those of boron. The densities are fairly comparable, <sup>2.34</sup> 1.34 g/cc for boron, and <sup>2.52</sup> 1.52 g/cc for boron carbide. Both materials are extremely brittle and are of the same relative hardness.



~~SECRET~~

Boron carbide is capable of self-sintering at elevated temperatures, thereby eliminating the necessity for bonding agents of any kind. When subjected to heat and pressure (2200° C and 2000 psi) the carbide will consolidate into a dense compact of very high compressive strength. This process is called "hot-pressing." A previous report has been written on the hot-pressing of boron carbide but it seems desirable to give a brief summary here.

Material: boron carbide - (commercial)

- typical analysis: boron - 68.47 percent
- carbon - 25.75 percent, silicon - 0.2 percent
- iron - 0.3 percent, aluminum - 0.3 percent
- calcium - 0.1 percent
- 60F mesh size
- melting point, about 2450° C
- density, 2.52 g/cc

Experimental Procedure: The hot-pressing of boron carbide was carried out as follows: the charge of the carbide was tamped into the graphite mold cavity and the pressure piston secured in place. The entire assembly was placed within an induction coil and surrounded by lamp-black insulation, leaving only the top portion of the pyrometer tube exposed to permit readings. The coil rested upon the base of a manually operated hydraulic press of the yolk type and the hydraulic piston descended from the top onto the graphite pressure piston. Heat was induced into the charge from a 20 kw arc-gap converter (see Fig. 1).

Pressing began when the charge approached 2200° C and continued at a slow rate until consolidation was completed. After cooling under pressure, the

~~SECRET~~

assembly was removed and the graphite machined from the carbide compact. The finished piece was characterized by high density and very high compressive strength. The densities of the hot pressed compacts approached 2.4 g/cc as compared with the theoretical density of 2.52 g/cc for the carbide. The compressive strengths were in the order of 200,000 psi.

Remarks: After several tests had been made by the ordnance division with the hot-pressed boron carbide compacts, it was discovered that the compressive strengths were much too high. These strengths were undesirable; hence, further investigation had to be undertaken to meet more closely the strengths desired.

D. HOT-PRESSING PURE BORON (95-98%)

At this particular time a much purer grade of boron (95-98%) was made available and experiments were made to determine methods of consolidation.

The first attempt was the hot-pressing of the boron without the use of any bonding agent. The process was identical with that used for hot-pressing the boron carbide. It was discovered that boron of such high purity did not lend itself to consolidation by self-sintering as did the carbide. The boron attacked the graphite severely, presumably to form the carbide, when the sintering temperature was approached; i.e., the graphite walls were attacked before any consolidation occurred.

It may conceivably be that very accurate temperature control during the hot-pressing cycle would show more favorable results. Accurate temperature control with induction heating of large and long sections is difficult, and in this case, it may be that resistance heating would be preferable.

E. HOT-PRESSING OF BORON WITH METAL POWDERS

After attempts to consolidate boron by hot-pressing without the use of bonding agents proved discouraging, metal bonding agents were again tried; in this case the hot-pressing technique was again used. The results showed no promise since there was no appreciable evidence of consolidation or sintering.

Materials: boron - Norton's product #26 - about 92-95 percent boron, balance carbon

boron - best purity - about 98 percent pure - balance carbon

metal powders - commercial grades of iron, nickel, aluminum, and silicon

Experimental Procedure: The method employed was the usual hot-pressing operation except that different temperatures were used for the different metals. Both the product #26 and the high-purity boron were tried. The boron was intimately mixed with 10 wt. percent of the metal powder in a mortar and then charged into a 3/4" graphite die. The assembly was heated by induction during the hot-pressing cycle. After cooling under pressure the compacts were removed from the graphite die and examined. The results for each type of metal bond are presented in Table II.

TABLE II. HOT-PRESSING OF BORON WITH METAL POWDERS

| Metal Powder | Wt. % | Diameter Die inches | Pressing Temp. °C | Pressure psi | Remarks                                                                                                                                                                        |
|--------------|-------|---------------------|-------------------|--------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Iron         | 10    | 3/4                 | 1900              | 2000         | There was no consolidation but there was evidence of some sintering although this might have been caused by the mild sintering action of the boron itself at this temperature. |
| Aluminum     | 10    | 3/4                 | 1000              | 2000         | No consolidation or sintering.                                                                                                                                                 |
| Silicon      | 10    | 3/4                 | 1920              | 2000         | No consolidation or sintering.                                                                                                                                                 |
| Nickel       | 10    | 3/4                 | 1930              | 2000         | No consolidation or sintering.                                                                                                                                                 |

-12-

Remarks: Just as in the case of the sintering of boron with metal powders these preliminary tests on hot-pressing with metal bonds were not encouraging and no further investigation of metal bonds was made. Different types of non-metallic bonding materials had now to be considered. Further study of premilling would undoubtedly give better results.

F. HOT-PRESSING BORON AND BORON CARBIDE WITH BORIC ANHYDRIDE

The process of hot-pressing boron carbide and boron with a fused boric oxide as the bonding agent is an attempt to introduce a simplified method of consolidation to replace the previous method of impregnating a compact of boron in a bath of molten boric oxide. This simplified process lends itself much better to production practice than does the impregnation. Hot-pressing permits use of lower temperatures and is comparatively faster; in general, less difficulty was experienced with hot-pressing.

In practice, this process can be applied to boron and boron carbide with equally good results. Work has been done on both. Pieces which were used by the ordnance group were prepared with boron carbide, whereas the limited amount of high-purity boron available made it necessary to restrict the work to small-scale experimental development.

Previous results with the impregnation process indicated that approximately 30 wt. percent of boric oxide was absorbed at the completion of the run. In view of this fact the preliminary work on hot-pressing was made with the use of a bond of 30 wt. percent of the oxide.

The results obtained by hot pressing boron and boron carbide with the boric oxide bond proved very encouraging. At temperatures in the order of 800° to 1000° C the oxide is quite fluid, wets the boron particles, and allows them to

~~SECRET~~

-13-

slide readily over one another to form a dense mass. After cooling, the compact is tough, dense, and mechanically strong.

Materials: boron carbide - Norton Co. ---- 60F mesh

density - 2.52 gms/cc

boron - 95-98% pure, balance carbon

60F mesh ---- density - 2.34 gms/cc

boric oxide - ( $B_2O_3$ ) - commercial - fused

density - 1.84 gms/cc

melting point - ca 600° C

Experimental Procedure: The apparatus used is similar to that used in hot-pressing the boron carbide (see Fig. 1). It consisted essentially of the yolk-type manual press enclosing an induction coil. The quartz tube within the coil contained the graphite die and its lampblack insulation.

The graphite dies were of the double-acting piston type and the pressures were of the order of 2000 psi. Because of the adherence of the boric oxide to the die walls, the production of one compact cost one die.

The hot-pressing cycle was usually completed in about an hour from the start. The procedure was as follows: the boron or boron carbide was dry mixed in a mortar with 30 wt. percent of the fused boric oxide and then tamped into the graphite die. The assembly was subjected to the usual hot-pressing operation. The charge was brought up to heat slowly without the application of pressure to allow any residual water retained by the oxide to escape. When the charge reached temperature (800° - 1000° C) as indicated by the optical pyrometer, pressure was applied (2000 psi) and consolidation proceeded rapidly. The pressure was main-

-14-

tained until there was no further evidence of consolidation; then the assembly was allowed to cool under pressure. Because of the adherence of the oxide to the walls of the graphite die it was necessary that the graphite be machined from the compact.

Table III lists some of the results of hot-pressing.

TABLE III

| Material              | Dia. Die<br>inches | Wt. %<br>B <sub>2</sub> O <sub>3</sub> | Pressing<br>Temp.<br>°C | Pressure<br>psi | Density of<br>Compact<br>g/cc |
|-----------------------|--------------------|----------------------------------------|-------------------------|-----------------|-------------------------------|
| B - #26               | 3/4                | 30                                     | 1000                    | 2000            | 2.06                          |
| B - #26               | 3/4                | 30                                     | 975                     | 2000            | 1.85                          |
| B - #26               | 3/4                | 30                                     | 1000                    | 2000            | 1.94                          |
| B - #26               | 3/4                | 30                                     | 885                     | 2000            | 1.94                          |
| B - #26               | 1.5                | 30                                     | 985                     | 2000            | 1.97                          |
| B <sub>4</sub> C-60F  | 3/4                | 30                                     | 1015                    | 2000            | 2.16                          |
| B <sub>4</sub> C-600F | 3/4                | 30                                     | 1005                    | 2000            | 2.04                          |
| B <sub>4</sub> C-60F  | 1.0                | 30                                     | 780                     | 2000            | 2.14                          |
| B <sub>4</sub> C-60F  | 3/4                | 30                                     | 1015                    | 2000            | 2.16                          |

Compressive tests made on the second and third specimens indicated values at 30,000 psi and 29,800 psi respectively.

Remarks: The hot-pressing technique as applied with the boric oxide bond is to be preferred to the impregnation method and showed considerably more promise. Here again refinement of process should be of concern, first, in the matter of synthetic grain sizes in the boron to produce minimum porosity, and, secondly, in the percentage of bonding agent needed to meet particular specifications.

G. HOT-PRESSING BORON AND BORON CARBIDE WITH LEAD BORATE GLASS

Boric oxide, although it functions very well as a bonding agent for boron and boron carbide, has the singular disadvantage of absorbing water at atmospheric conditions. The fused anhydrous salt will in a very short time pick up appreciable amounts of water vapor from the air. Although the compact apparently suffers no disintegration, the presence of hydrogen is undesirable.

It was solely to overcome this defect that a lead borate glass was considered for use as a bonding agent. The glass proved stable under ordinary atmospheric conditions and the bond provided a compact with greater compressive strengths than those compacts which were bonded with boric oxide.

Materials: lead borate glass - density - 3.73 g/cc

- 17% boron, 40% lead, balance oxygen

- prepared by fusion of 60 wt. percent of lead borate,

$Pb(BO_2)_2$  and 40 wt. percent of boric oxide,  $B_2O_3$ .

boron carbide - Norton Co. 60F mesh

boron - 95% pure, balance carbon

Experimental Procedure: The technique involved in hot-pressing boron and boron carbide with a lead glass bond is exactly the same as the process in which the boric oxide is used as the bonding agent. Effective temperatures and pressures for the operation are essentially the same. Here again 30 wt. percent of the lead borate glass was used to provide the bond. It can be seen that because of the difference in densities between the lead borate glass (3.73 g/cc) and the boric oxide (1.84 g/cc) the same volume percentage of bond was not represented, hence the hot-pressing of the lead borate glass appeared more sluggish. Consolidation was satisfactory however.

~~SECRET~~

Table IV lists some of the results obtained by hot-pressing:

TABLE IV

| Material          | Dia. Die inches | Wt. % Lead Borate Glass | Pressing Temp. °C | Pressure psi | Density of Compact g/cc |
|-------------------|-----------------|-------------------------|-------------------|--------------|-------------------------|
| Boron -# 26       | 3/4             | 30                      | 1105              | 2000         | 2.11                    |
| Boron carbide 60F | 3/4             | 30                      | 1010              | 2000         | 2.12                    |

A compressive test on the first sample ran 37,100 psi.

Remarks: It is interesting to note that the lead borate glass will yield a greater boron concentration per unit volume than the boric oxide. Boric oxide yields 0.54 grams of boron per cc, whereas the lead borate glass provides 0.63 grams of boron per cc.

Not very much work has been done with this type of bonding agent up to the present time, but it is proposed that the future work should include a slightly greater weight percentage of this bond so that the volume composition will more nearly approach that of the boron-oxide-bonded compacts. A second consideration would again be the investigation of synthetic grain sizes to minimize the number of voids and hence make for a greater boron concentration in the fabricated compact.

#### H. PLASTIC BONDING OF BORON AND BORON CARBIDE

Compression molding of boron with plastic resins opens a potential field as a new method of consolidation where the presence of hydrogen in the compact is desirable. From the standpoint of mechanism of fabrication the process has a de-



oiled advantage since plastic bonding is a low-temperature operation and since the steel molds can be used to manufacture very many pieces.

Because of the general similarity of boron and boron carbide, and because of the scarcity of boron, most of the work in plastic bonding was done with boron carbide. It is quite reasonable to believe that if the plastic bonding of the boron carbide is successful, so likewise will be the plastic bonding of the boron since the plastic will behave much in the fashion of an inert adhesive.

Preliminary work has been done only with the thermoplastic resins, lucite and polystyrene, with promising success. The compressive strengths were relatively low (below 20,000 psi), but further refinement of the process may well bring the compressive strengths to an appreciably higher value. Work has been contemplated for thermosetting materials which would give greater compressive strengths, but as yet no detailed work has been done.

Materials: lucite - commercial molding resin

- excellent molding qualities
- compression molding temp. - 150° - 190° C
- very slight tendency to cold flow
- specific gravity - 1.18 - 1.19
- softening point - 105° - 120° C
- solvents - ketones, esters, aromatic hydrocarbons

polystyrene - commercial molding resin

- excellent molding qualities
- molding temp. - 150° C - 160° C
- specific gravity - 1.05 - 1.07
- solvents - esters, aromatic hydrocarbons

boron carbide - Norton Co. - 60F mesh

boron - about 95 percent pure - balance carbon

Experimental Procedure: The first work that was done with plastic bonding made use of a limited amount of plastic, in this case 10 wt. percent, in order to observe whether or not there would be a fair degree of consolidation. It was found that consolidation did occur but that the voids were not completely filled. In order to determine the amount of plastic necessary to fill the voids, pressure/density data were compiled for both boron and boron carbide. This was done by pressing compacts in a steel die of 1/2" diameter to various pressures and then determining the percentage voids for each pressure (see Table V). From the observation of these data, the weight percentage of plastic bond was chosen such that the voids should be completely filled by the plastic at the particular working pressure.

Table VI lists the results of sieve tests performed on materials used.

TABLE V

Pressure/Density Data - 1/2" dia. Steel Die

| Material                                  | Pressure<br>psi | Density<br>g/cc | Percent<br>Porosity |
|-------------------------------------------|-----------------|-----------------|---------------------|
| Boron carbide-60F;<br>density - 2.52 g/cc | 10,000          | 1.75            | 31                  |
|                                           | 30,000          | 1.85            | 27                  |
|                                           | 50,000          | 1.91            | 24.2                |
|                                           | 70,000          | 1.92            | 23.8                |

~~SECRET~~

~~SECRET~~

-19-

TABLE V (cont'd)

| Material                             | Pressure<br>psi | Density<br>g/cc | Percent<br>Porosity |
|--------------------------------------|-----------------|-----------------|---------------------|
| Boron - D7267;<br>density - 2.3 g/cc | 15,000          | 1.66            | 28                  |
|                                      | 30,000          | 1.70            | 26                  |
|                                      | 50,000          | 1.74            | 24                  |
|                                      | 70,000          | 1.77            | 23                  |

~~SECRET~~

TABLE VI. SIEVE TESTS - TYLER STANDARD SCREEN SCALE SIEVES

Screen Scale Ratio: 1.414

Rutap -- 20 min. with 100 6M charge

Date: January 11, 1944

| Screen<br>Openings<br>inches | Mesh | Norton Co.<br>60F        |                        |           | Norton Co.<br>#26        |                        |           | Norton Co.<br>D-7195     |                        |           | Norton Co.<br>D-7267     |                        |           |
|------------------------------|------|--------------------------|------------------------|-----------|--------------------------|------------------------|-----------|--------------------------|------------------------|-----------|--------------------------|------------------------|-----------|
|                              |      | Wt.<br>Between<br>Sieves | %<br>Between<br>Sieves | Cum.<br>% | Wt.<br>Between<br>Sieves | %<br>Between<br>Sieves | Cum.<br>% | Wt.<br>Between<br>Sieves | %<br>Between<br>Sieves | Cum.<br>% | Wt.<br>Between<br>Sieves | %<br>Between<br>Sieves | Cum.<br>% |
| .0082                        | 65   | 13.4                     | 13.4                   | 13.4      | 29.8                     | 29.8                   | 29.8      | 13.8                     | 13.8                   | 13.8      | 14.5                     | 14.5                   | 14.5      |
| .0058                        | 100  | 10.2                     | 10.2                   | 23.6      | 17.5                     | 17.5                   | 47.3      | 15.7                     | 15.7                   | 29.5      | 16.7                     | 16.7                   | 31.2      |
| .0041                        | 150  | 7.5                      | 7.5                    | 31.1      | 12.0                     | 12.0                   | 59.3      | 12.2                     | 12.2                   | 41.7      | 12.4                     | 12.4                   | 43.6      |
| .0029                        | 200  | 10.5                     | 10.5                   | 41.6      | 11.6                     | 11.6                   | 70.9      | 12.2                     | 12.2                   | 53.9      | 12.4                     | 12.4                   | 56.0      |
| .0021                        | 270  | 9.6                      | 9.6                    | 51.2      | 6.5                      | 6.5                    | 77.4      | 6.5                      | 6.5                    | 60.4      | 6.0                      | 6.0                    | 62.0      |
|                              | PAN  | 48.5                     | 48.8                   | 100.0     | 22.8                     | 22.6                   | 100.0     | 39.2                     | 39.6                   | 100.0     | 37.3                     | 38.0                   | 100.0     |
| TOTALS                       |      | 99.7                     | 100                    |           | 100.2                    | 100                    |           | 99.6                     | 100                    |           | 99.3                     | 100                    |           |

APPROVED FOR PUBLIC RELEASE

APPROVED FOR PUBLIC RELEASE

Double-acting steel dies were used in these experiments. Pressure was applied to the assembly with a hand-operated Carver hydraulic press. The temperatures of compression molding differed with the type of resin and were in the ranges recommended by the commercial molders of plastic materials.

The heating of the charges was effected by placing the entire assembly (including charge, steel die, and pistons) in an oven and holding the assembly at the required temperature until it was heated uniformly throughout.

The plastic was intimately mixed with the boron and boron carbide by making a solution of the plastic in a suitable organic solvent (i.e., ethyl acetate) and then adding the required amount of solution to the charge. The mixture was agitated frequently while drying. After drying the boron and plastic mixture was reground and charged into the steel die. After the assembly had been sufficiently soaked at temperature in the oven it was quickly transferred to the press and maintained at pressure until it cooled to slightly above room temperature. The compact was ejected from the mold and all the necessary data were recorded.

Table VII lists some of the results obtained.

TABLE VII

| Material         | Die Dia.<br>inches | Bond   | Wt. %<br>Bond | Pressing<br>Temp.<br>°C | Pressing<br>Press.<br>psi | Density<br>g/cc |
|------------------|--------------------|--------|---------------|-------------------------|---------------------------|-----------------|
| B <sub>4</sub> C | 1-1/8              | poly.  | 17            | 155                     | 10,000                    | 2.04            |
| B <sub>4</sub> C | 1-1/8              | poly.  | 17            | 155                     | 10,000                    | 1.87            |
| B <sub>4</sub> C | 1-1/8              | lucite | 18            | 165                     | 10,000                    | 2.02            |
| B <sub>4</sub> C | 1/2                | lucite | 10            | 165                     | 25,000                    | 2.22            |
| B <sub>4</sub> C | 1/2                | lucite | 10            | 170                     | 25,000                    | 2.20            |
| B-D7145          | 1/2                | lucite | 10            | 160                     | 25,000                    | 1.88            |

~~SECRET~~

The compressive strength of the second sample measured 16,350 psi.

The compressive strength of the third sample measured 11,280 psi.

Remarks: This program on consolidation of boron had been temporarily concluded with this small amount of work on plastic bonding. The results were encouraging and indicated good potentialities for this type of bonding.

If in the future the hydrogen-bearing bond is acceptable, further work could be done on the following:

- 1) synthetic grain size mixtures to minimize porosity,
- 2) thermoplastic resins with suitable hydrogen contents,
- 3) thermosetting resins, and
- 4) possible boron-bearing plastics.

Conclusion: Table VIII lists the various methods for consolidation of boron which thus far have appeared most attractive. In comparison, it can be seen that sintered boron carbide, in general, will not yield such high boron concentrations as other methods for consolidating boron of 98 percent purity.

~~SECRET~~

TABLE VIII

Summary of Best Methods for Consolidation of Boron

| Material                                                           | Density<br>gms/cc | Wt.%<br>Bond | Boron<br>gms/cc | Carbon<br>gms/cc | Oxygen<br>gms/cc                                                               | Lead<br>gms/cc                                       |
|--------------------------------------------------------------------|-------------------|--------------|-----------------|------------------|--------------------------------------------------------------------------------|------------------------------------------------------|
| Sintered boron carbide                                             | 2.4               | -----        | 1.64            | 0.62             | { Fe - 0.06 gms/cc<br>Al - 0.06 gms/cc<br>Si - 0.04 gms/cc<br>Ca - 0.02 gms/cc |                                                      |
| " " "                                                              | 2.3               | -----        | 1.57            | 0.59             |                                                                                |                                                      |
| " " "                                                              | 2.2               | -----        | 1.51            | 0.57             |                                                                                |                                                      |
| " " "                                                              | 2.1               | -----        | 1.44            | 0.54             |                                                                                |                                                      |
| " " "                                                              | 2.0               | -----        | 1.37            | 0.51             |                                                                                |                                                      |
| <u>30% Porosity</u>                                                |                   |              |                 |                  |                                                                                |                                                      |
| B + B <sub>2</sub> O <sub>3</sub>                                  | 2.16              | 25.4         | 1.75            | 0.03             | 0.37                                                                           | 0.45                                                 |
| B + PbB <sub>2</sub> O <sub>4</sub> ·B <sub>2</sub> O <sub>3</sub> | 2.73              | 41.0         | 1.77            | 0.03             | 0.48                                                                           |                                                      |
| B + Lucite                                                         | 1.86              | 18.8         | 1.58            |                  |                                                                                |                                                      |
| B + Polystyrene                                                    | 1.82              | 17.0         | 1.58            |                  |                                                                                |                                                      |
| <u>25% Porosity</u>                                                |                   |              |                 |                  |                                                                                |                                                      |
| B + B <sub>2</sub> O <sub>3</sub>                                  | 2.18              | 21.0         | 1.82            | 0.04             | 0.32                                                                           | 0.37<br>(Composition of Molding Powder<br>Uncertain) |
| B + PbB <sub>2</sub> O <sub>4</sub> ·B <sub>2</sub> O <sub>3</sub> | 2.65              | 35.0         | 1.84            | 0.04             | 0.41                                                                           |                                                      |
| B + Lucite                                                         | 2.01              | 14.4         | 1.69            |                  |                                                                                |                                                      |
| B + Polystyrene                                                    | 1.97              | 13.0         | 1.69            | "                | " " " "                                                                        |                                                      |
| <u>20% Porosity</u>                                                |                   |              |                 |                  |                                                                                |                                                      |
| B + B <sub>2</sub> O <sub>3</sub>                                  | 2.21              | 16.7         | 1.91            | 0.04             | 0.29                                                                           | 0.30                                                 |
| B + PbB <sub>2</sub> O <sub>4</sub> ·B <sub>2</sub> O <sub>3</sub> | 2.59              | 29.0         | 1.93            | 0.04             | 0.32                                                                           |                                                      |
| B + Lucite                                                         | 2.07              | 11.5         | 1.80            |                  |                                                                                |                                                      |
| B + Polystyrene                                                    | 2.05              | 10.0         | 1.80            |                  |                                                                                |                                                      |
| <u>15% Porosity</u>                                                |                   |              |                 |                  |                                                                                |                                                      |
| B + B <sub>2</sub> O <sub>3</sub>                                  | 2.22              | 12.5         | 1.99            | 0.04             | 0.23                                                                           | 0.22                                                 |
| B + PbB <sub>2</sub> O <sub>4</sub> ·B <sub>2</sub> O <sub>3</sub> | 2.47              | 22.7         | 2.00            | 0.04             | 0.25                                                                           |                                                      |
| B + Lucite                                                         | 2.08              | 8.5          | 1.91            |                  |                                                                                |                                                      |
| B + Polystyrene                                                    | 2.06              | 7.6          | 1.91            |                  |                                                                                |                                                      |

Note: In these calculations the boron is considered to be 98 percent pure.

Having selected porosities of 30, 25, 20 and 15 percent in the boron compact, calculations have been made for each of the types of bonds to determine the ultimate boron concentrations in the finished compact if all the voids are filled. This table will be useful for future work.

It will appear from all the methods considered for the fabrication of boron that the hot-pressing method is the most adaptable to production fabrication.

As for bonding agents, both the boric anhydride and the lead borate glass work well, the latter having an advantage because of its stability in air and because of its greater boron yield.

Plastic bonding shows considerable promise as a method for consolidation. Excluding its bonding properties, the function of the hydrogen-bearing plastic is quite different from that of the boron-bearing glasses and their respective merits must be considered before detailed work can be directed toward a specific method of consolidation.

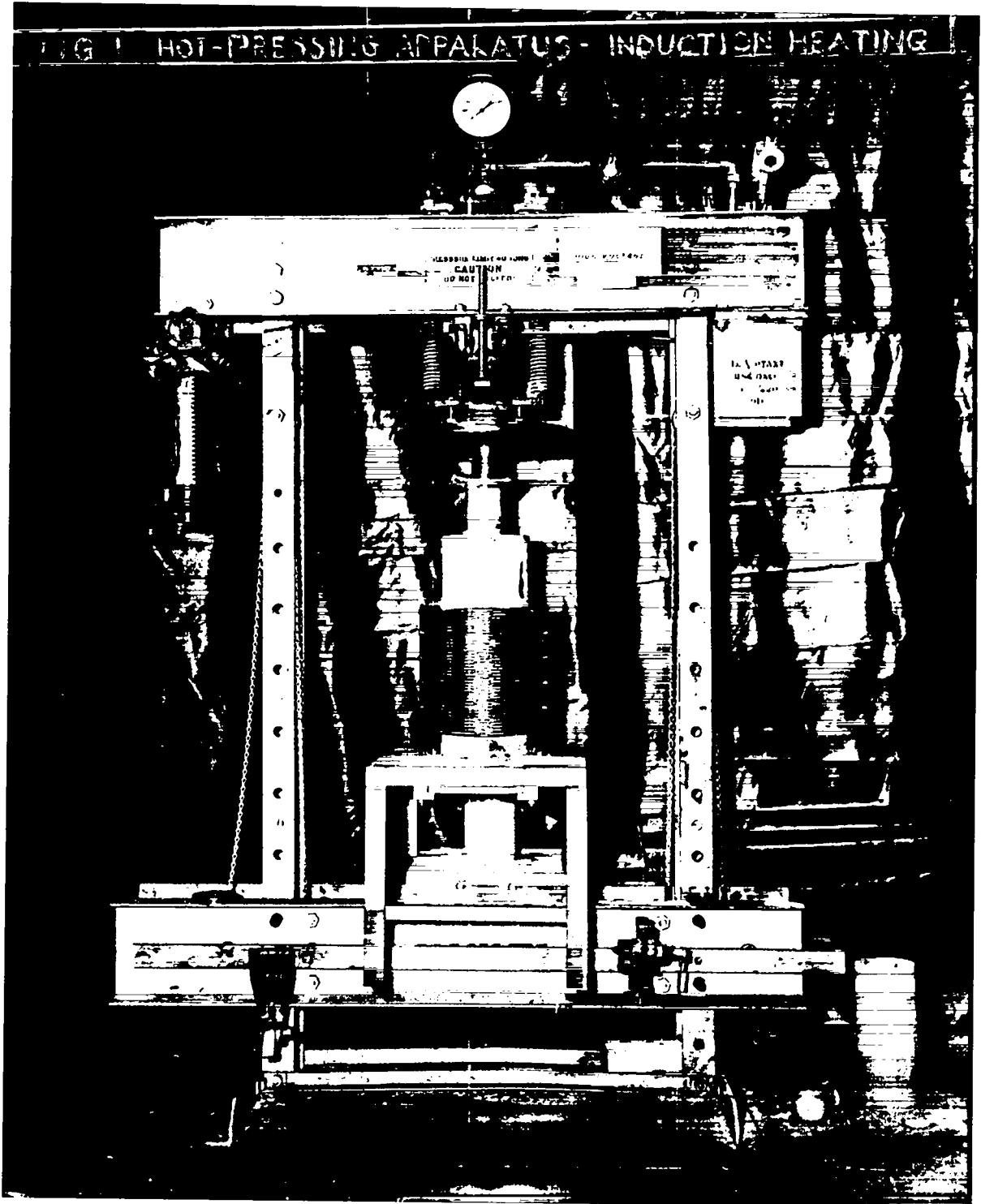
Another consideration of importance, which will apply to either of the above cases, is that of particle size. Since boron is hard, abrasive, and not subject to cold flow as are most powdered metals, the question of packing densities and particle size is of importance. It is reasonable to assume that an adjustment of particle sizes to produce a synthetic mixture with optimum packing characteristics (i.e., obtain the least percentage of voids) is a desirable goal towards increasing the concentration of boron in the fabricated compact.

It must be mentioned that the work that has been done so far is of a preliminary and investigatory nature and that a great deal of further effort could be directed to the refinement of those methods which showed promise. Another field of investigation may well lie in the search for bonding agents which in themselves have higher boron concentrations.

~~SECRET~~



SECRET



SECRET

UNCLASSIFIED

DOCUMENT ROOM

REC. FROM ed

DATE 8/25/44

REC. NO. REC.



UNCLASSIFIED