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DECEMBER 1970

**BATH SHIELD MATERIAL
DEVELOPMENT**

TEST REPORT

MASTER

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BATH SHIELD MATERIAL
DEVELOPMENT

TEST REPORT

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ABSTRACT

The development of the manufacturing process and the materials properties of recent BATH (boron-aluminum-titanium hydride) round bar stock are discussed. Conventional extrusion of powder metallurgical compacts of the component powders is shown to be a practical process for producing BATH shield material which meets the program objectives for mechanical and physical properties. Optimum extrusion variables are listed.

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1.0 INTRODUCTION

The desired characteristics of a material suitable for use as the R-1 internal shield have been identified⁽¹⁾ as follows:

Characteristics include:

A. STRUCTURAL-THERMAL

1. Sufficient strength and ductility for use as a self-supporting structure.
2. High thermal conductivity and low thermal expansion coefficient.

B. CHEMICAL-PHYSICAL

1. Low vapor pressure.
2. Sufficiently high melting point.
3. Chemical compatibility with the reactor fluid environment and with adjacent materials.

C. NUCLEAR

1. Neutron moderating ability.
2. High neutron absorption cross section.
3. Resistance to radiation damage.

D. MANUFACTURING

1. Fabricability by standard techniques.
2. Low cost of raw materials.

A material composition consisting of 70 v/o* aluminum, 24 v/o titanium hydride, and 6 v/o boron carbide was designed to provide the required shielding characteristics. For convenience, this composition and material manufactured to this chemistry were assigned the acronym BATH.

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* v/o indicates percent by volume.

The aluminum component of BATH acts as a matrix for the other two constituents, provides for the strength, ductility, and thermal conductivity aspects of the BATH material, and results in a relatively low shield weight.

However, aluminum does not have the desired neutron moderating or absorbing characteristics⁽²⁾, which are provided instead by the other two BATH constituents.

Boron carbide, present in the form of dispersed particles, essentially acts as a vehicle to provide boron, a good neutron absorber. Alternate methods of incorporating boron in the BATH composition were evaluated. Use of the elemental form was considered and dismissed in favor of the carbide form because of the greater availability and lower cost of the latter. Substitution of boron nitride, a soft compound, for the hard boron carbide was attempted experimentally and is discussed below in greater detail.

Titanium hydride, also in the form of dispersed particles, provides the neutron moderating capability.

The nominal composition of the BATH material is shown in Table I.

Since this is a newly invented composition, it was necessary to develop a process whereby useful amounts of material could be manufactured, and then to measure the appropriate materials properties.

After due consideration of the standard processing techniques, a combination of powder metallurgy and extrusion was selected as the process having the highest probability of success in producing sufficient quantities of material with minimum development effort.⁽¹⁾ The process was defined to include (a) blending of the constituent powders, (b) compaction of the blended powders into a billet with adequate strength and density for further handling, and (c) extrusion into bars of the required size.

2.0 SPECIFIC OBJECTIVES

The specific objectives for the BATH material had been specified in detail as follows⁽³⁻⁴⁾:

A. MECHANICAL PROPERTIES

1. STRENGTH

10,000 psi minimum compressive and tensile yield
at 540°R (80°F).

2. ELONGATION

0.5 to 2.0 percent or greater at 540°R (80°F).
0.5 percent minimum at 60°R (-400°F).

B. PHYSICAL AND CHEMICAL PROPERTIES

1. DENSITY

95 percent of theoretical (minimum)*

2. DISTRIBUTION OF CONSTITUENTS

Uniform distribution is required.

3. CRACKS, VOIDS, AND SIMILAR FLAWS

Area of flaws is not to exceed 5 percent of any 1/4 inch by
1/4 inch section. The largest flaw shall be less than 0.03
inch in its largest dimension. No linear voids are accept-
able in the direction of extrusion.

4. CHEMISTRY

The desired composition of this material is shown in Table I.

5. THERMAL PROPERTIES

No specific thermal property objectives were listed. However,
a thermal conductivity of 50 BTU/hr-Ft-°R (nominal) had been
assumed in initial designs and was used as a guide in this program.

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* Theoretical density of the BATH reference composition is 2.94 gm/cc.

C. IRRADIATION REQUIREMENTS

After irradiation to an integrated neutron dose of 10^{19} nvt, the atom ratio of hydrogen to titanium must not be less than 1.51 due to loss of hydrogen.

D. MISCELLANEOUS FEATURES

In addition to the specific mechanical and physical property requirements, the material should be:

1. Dimensionally stable up to 900°R
2. Machinable
3. Usable without encapsulation
4. Non-reactive with the reactor environment.

3.0 SCOPE OF THIS REPORT

This report documents the entire development of the manufacturing process as well as materials properties of recent BATH extruded bar stock.

4.0 REVIEW OF PRIOR RESULTS

4.1 REVIEW OF PROPERTIES

The first lot of extruded 2-1/2 inch diameter bar (Lot No. 77265), with the nominal composition shown in Table I, was thoroughly tested in the as-received condition to obtain design information. The results were reported in detail previously⁽¹⁾. (The irradiation performance of BATH material has been discussed elsewhere⁽⁵⁾.) This early material met the mechanical, chemical, and physical objectives with the following exceptions or inconsistencies:

- (a) The primary inconsistency was in the trend of the Young's Modulus with temperature. As temperature increases, the modulus should decrease. The measured modulus of the BATH material increased with temperature up to at least 80°F (540°R), the highest temperature for which these data were obtained.
- (b) The thermal conductivity, varying from 69 to 77 Btu/hr-ft-°R, was higher than the nominal 50 Btu/hr-ft-°R value which was assumed in initial design studies, but was lower than expected. For example, the experimental value at ambient temperature was 75 Btu/hr-ft-°R, while the calculated value, using data for aluminum (corrected by assuming that the ceramic components of BATH act as voids and have virtually no conductivity) is at least 90 Btu/hr-ft-°R.

In addition, a statistically significant increase in thermal conductivity was found with specimen position in a bar from the nose (the first portion which was extruded) to the tail.

Radiographic examination of the test samples⁽¹⁾ showed that the Young's Modulus and thermal conductivity inconsistencies were caused by agglomerates of titanium hydride,

1/16 to 1/8 inch in size (see Figure 1), which resulted from improper blending of the constituent powders in the production of the billets. Results of the re-investigation of the questionable data are discussed below.

5.0 REVISED DATA

5.1 TENSILE PROPERTIES

The tensile properties of the first lot (No. 97265) of 2-1/2 inch diameter bar, which was used to determine the previously published data,⁽¹⁾ were re-evaluated using pre-selected samples. The testing methods used have been discussed previously.⁽¹⁾ The samples were radiographed to determine the extent and positions of the titanium hydride agglomeration, and those showing gross inhomogeneities were discarded. The samples which were selected for testing were those with the smallest agglomerates, spaced further apart than the specimen gage length, so that strain gages could be placed for data acquisition over material having only randomly-dispersed individual particles.

The material within the specimen gage length, selected in this manner, more closely represents the latest homogeneous material than the bulk of the early material.⁽¹⁾ Data from the selected samples are therefore judged to be more closely applicable to the latest BATH manufacturing process than are the earlier data.⁽¹⁾

The tensile data obtained in this manner are given in Tables II to VI, and include Modulus of Elasticity, Yield Strength, Ultimate Strength, Total Elongation, and Poisson's Ratio.

These data were examined statistically⁽⁷⁾ by the analysis of variance technique for significant differences due to:

- (a) specimen orientation with respect to the extrusion direction (specimen axis longitudinal and transverse to the long axis of the bar);
- (b) location within the original bar (nose and tail of bar as extruded);
- (c) extrusion sequence (bar-to-bar variation); and
- (d) test temperature.

As expected, test temperature was significant for each property.

The Young's Modulus data (see Table II) showed no significant differences due to orientation, bar, and/or location within a bar. The variation of Young's Modulus with temperature was now as originally expected, showing decreasing values with increasing temperature, contrary to the previous determination.⁽¹⁾ In addition, the present standard deviations are less than in the original data.

The 0.2 percent offset yield strength (see Table III) showed no differences bar to bar and/or location within a bar, but orientations within a bar are highly significant. Similar results were obtained previously, and the present yield values do not differ greatly from the previous values, but it is noted that the latest transverse specimen properties are consistently lower than for the longitudinally oriented samples, a relationship opposite to that of the previous data.⁽¹⁾ Also, the present standard deviation values are lower than the previous values.

The ultimate tensile strength data (see Table IV) show the same bar to bar, bar location, and transverse to longitudinal specimen relationships as the yield strength data. The present longitudinal sample data are similar in absolute value to the previous data except at the highest temperature (400°F), where the present data are 2.5 to 5 kpsi lower. No reason has been found for a difference of this magnitude. In addition, the previous data showed no significant difference between specimen orientations, contrary to expectations. The present data, which show differences between specimen orientations, are therefore more consistent in this respect. As with the yield strength, the revised ultimate tensile strengths are consistently greater for the longitudinally oriented specimens. In this respect, the behavior of the present yield and ultimate strength data follows the expectation for a directionally formed, i.e. extruded, material. The standard deviations of the longitudinally oriented tensile data are identical to the previous data, but the transversely oriented samples show lower standard deviations.

The total elongation of the selected specimens (see Table V) showed no differences due to specimen orientation, original position within the bar, or from bar to bar. The present data are similar in magnitude to the early data.⁽¹⁾

Poisson's ratio was also re-measured, using the preselected samples. No statistically significant differences were found due to location within a bar. Differences in the ratio due to bar extrusion sequence are significant with a level of significance less than 0.05. Differences in ratio due to orientation were significant, with these differences dependent upon test temperature for the transversely oriented specimens. For the longitudinally oriented specimens, test temperature produced no significantly differing ratios. No technical justification is available for this behavior. The data are given in Table VI.

The absolute values of Poisson's ratio and the standard deviations did not differ greatly from the previous data.

5.2 THERMAL PROPERTIES

The thermal conductivity of the first lot of BATH 2-1/2 inch bar was not re-measured in the manner described above for the tensile properties since the entire volume of the specimens would have to be free of titanium hydride inhomogeneities in order to obviate their influence, and it was not possible to identify a sufficient volume of material for this purpose. Alternatively, the data were re-examined and it was judged that the data previously reported⁽¹⁾ for the central section of the bars most nearly approximates what could be expected from more homogeneous material. The most recently fabricated material will be tested to verify the re-assessed data.

The re-evaluated thermal expansion data are given in Table VII.

Analysis of variance showed no significant difference due to specimen orientation, position within a bar, or from bar to bar. In general, the revised data are about 30 percent higher than the previous data.

6.0 FABRICATION PROCESS

6.1 SUMMARY OF PROCESS DEVELOPMENT

6.1.1 General Information

The BATH manufacturing process development included the extrusion of:

- (a) 1-1/4 inch diameter and 2-1/2 inch diameter bar of the 70 v/o Al reference composition;
- (b) 1-1/2 inch diameter and 2-1/2 inch diameter bar of a modified composition with 80 v/o Al (balance TiH_x and B_4C);
- (c) 1-1/4 inch diameter bar of 75 v/o aluminum (balance TiH_x and B_4C);
- (d) two sizes (1-1/8 inch and 2 inches across flats) of hexagonal bar of the reference composition, and
- (e) 3/4 to 1-1/4 inch diameter bars of a modified composition replacing the boron carbide with boron nitride.

Traceability information for each extrusion run is given in Table XIX, Appendix.

A flow diagram of the BATH manufacturing process is shown in Figure 2. The aluminum and titanium hydride powders for all runs were purchased to the same respective specifications (see Table XIX, Appendix). The boron carbide powders for each run were essentially similar in composition, but the particle size was varied between some of the trials to improve the machineability, with the finer particle sizes (-325 mesh) showing the best machining characteristics.

Blending of the powders for the early runs was performed in a barrel-type blender. The latest runs, including those for the 2-1/2 inch diameter bar, were blended in a V-type blender.

The blended powders were isostatically compacted at 60,000 psi into billets of 95 percent theoretical density prior to extrusion. A typical compacted billet, (11 inches diameter by 12 inches length), as produced for the first 2-1/2 inch diameter bar extrusion

(lot no. 97265), is shown in Figure 3. The superficial staining evident on the periphery of the billet is inherent in the compaction process, and is completely removed in machining the billet to the desired uniform diameter (10.938 ± 0.062 in. for the 2-1/2 in. bar) prior to extrusion.

Two extrusion trials of the small diameter (1-1/4 inch) bar and three trials of the 2-1/2 inch diameter bar have been made to develop the present manufacturing process for the reference composition in round bar. The latest runs are discussed in greater detail in section 6.2.

6.1.2 Hexagonal Bar

Concurrently with the development of the extrusion process for round bar stock, two reference composition extrusion runs were made to produce material of hexagonal cross section. In the first trial, (Lot No. 02481), hollow bars, measuring 2 inches across flats, each with a central hole of approximately 1 inch diameter, were extruded from 9 inch diameter billets with a 1 inch pre-machined central hole.

In general, this trial resulted in severe wear to the extrusion tooling, as well as edge cracking of the extruded bar (see Figure 4). Pertinent billet data are given in Table VIII. Extrusion data are given in Table IX. Tensile data were obtained for this material according to a 1/2 full factorial test plan, and are listed in Table X^{*}. In general, the yield and ultimate strength data are fairly consistent for each test temperature, and compare favorably with the most recent data for the 2-1/2 inch bar discussed in section 5.0. In fact, the hexagonal bar tensile yield data are significantly higher, except at 400-480°R, as can be seen by comparison of Tables III and X. The elongation data for the hexagonal bar, however, are noticeably inconsistent.

The superior yield properties of the hexagonal bar over the 2-1/2" diameter round bar had been attributed earlier to the fact that the hexagonal bar microstructure was uniform in comparison with the agglomerates found in the first lot of 2-1/2" diameter round bar. As

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* Data for the experiments involving hexagonal bars are included in detail because of the recurrent interest in developing this shape to reduce machining costs.

noted above, however, specimens of the round bar, pre-selected to avoid the inhomogeneities, continue to show lower yield strengths than the hexagonal bar. In the present view, therefore, the improved yield properties of the hexagonal bar over the round are principally due to a greater degree of compaction in the former. This hypothesis is supported by the higher density of the hexagonal bar (99.3 percent T.D.) compared with the round bar (97.4 percent T.D.⁽¹⁾).

Although the strength properties and density were adequate for the first hexagonal extrusions, the widespread edge cracking resulted in the trial extrusion of canned hexagonal shapes. Four extrusions were made (referred to in Table XIX as Lot No. 07530) consisting of two each of different internal billet configurations, (see Figure 5). In the first set (see Figure 5 a) hexagonal billets, 5 inch across corners, were enclosed in 6 inch O.D. jackets of 6061 T6 aluminum. The second set (see Figure 5 b), consisted of hexagonal shapes pre-machined with grooves (the reference BATH shield module design employs grooves as cooling channels). Bars of 6061 T6 aluminum were inserted in the canned assemblies to maintain groove dimensions. The canned assemblies were extruded at a 16:1 ratio (by cross-sectional area) to bars approximately 1-1/8 inch across the hexagonal flats. The results showed that the anticipated configurations were approximately achieved (see Figure 6), but close dimensional control would appear to be difficult to maintain. In addition, acceptable methods of de-jacketing the extruded bars would be required. Further work to develop this concept was suspended following the improvement in the extrusion process for round bars (Lot No. 08989) with the expectation of improved machineability by using a fine B₄C particle size in the raw powder compact for the round shapes. Thus, it was felt that the cost of machining the improved BATH material would be lowered sufficiently to obviate the need to develop the hexagonal extrusion process.

6.1.3 High Aluminum Compositional Variants

The high volume percent Al compositions were tested as a backup to the more desirable, 70 v/o Al material. In general, the high aluminum content material extruded more easily than the reference BATH composition. However, as the extrusion process for

the reference material improved, further work on the secondary compositions was terminated and the total development effort was successfully placed on optimization of production of the 70 v/o Al material.

6.1.4 Boron Nitride Compositional Variant

A second variant on the reference BATH composition, in addition to the high aluminum compositions, was the substitution of boron nitride for boron carbide as the vehicle for introducing boron into the shield material since it was expected that the nitride, being much softer than the carbide, would result in a more easily machinable product. This expectation was substantiated by tests on early material as summarized in Table XI.

A number of 3 inch diameter by 6 inch long billets containing 64-73 v/o aluminum, 24 v/o titanium hydride, and 3-12 v/o boron nitride were isostatically compacted prior to extrusion.

A total of 192 bars, 3/4 inch to 1-1/4 inch in diameter and 9 to 10 foot long was extruded under various conditions. The latest extrusion run, consisting of 132 extrusions, was a 1/16 factorially designed experiment based on the range of variables identified in the first BN experiments. The variables studied were:

- (a) billet temperature: 675, 725, 775, 825, and 875°F*
- (b) extrusion ratio: 12:1, 14:1, 16:1, 20:1
- (c) die land length: 0.125", 0.250"
- (d) ram speed: 20, 80, 140, 200 in/min
- (e) lubricant: heavy (lead base), light (lubriplate)
- (f) billet composition: four different variations in aluminum content, 64, 67, 70, and 73 weight percent Al
- (g) purity of aluminum powder: two levels of oxide content

The factorial experiment resulted in a wide variation in quality of the extrusions, as determined by visual inspection. The principal defect encountered was severe "pine-cone", or "rattlesnake" cracking (see Figure 7, bottom). Other defects included longitudinal

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* The 875°F temperature was not part of the original factorial plan, but was added at the end of the experiment as a supplement.

cracking, blistering, galling (extreme surface roughness), and generally distributed, small circumferential cracks. The condition resulting in the greatest defects was a combination of low billet temperature (675°F) and low ram speed (20 ipm). Use of the light weight lubricant always resulted in a greater number and severity of defects on the topside (with respect to the die) of the extrusions, probably due to gravity effects on the lubricant. In general, the best results (see Figure 7, top) were obtained with high billet temperature ($825\text{--}875^{\circ}\text{F}$), high extrusion ratio (16:1 or 20:1), high (140 ipm) rather than low ram speed, heavy lubricant, and high aluminum billet composition. The purity of the aluminum powder did not seem to be important in these runs. The effect of the die land length was greatest at the lower temperatures, where the defects using the 0.125" die land size were greater in severity at temperatures between $675\text{--}725^{\circ}\text{F}$.

The larger die land length was consistently superior to the small size in yield of defect-free material at all temperatures studied.

Sections of the extruded bar showing the best surface (shown in Figure 7, top) were inspected ultrasonically for internal defects. Some defect indications were found. Figure 8 shows a metallographic cross section through a portion showing the ultrasonic defect indications, where the quality of the outer surface can be clearly seen.

Mechanical property tests were conducted on the early lots of this material. Data for samples taken in the direction longitudinal to the extrusion direction are summarized in Table XII.

An indirect measurement of the transverse strength of the BN BATH alternate was made using short flexure bars. Results indicated that the transverse strength is only about $1/3$ that of the B_4C BATH. This is to be expected as a result of the considerable amount of BN stringers, as shown in Figure 9.

In summary, a combination of extrusion variables yielding visually acceptable bar stock was developed.

However, since large internal defects were found, even in bar with the best surface quality, and since the boron nitride tends to form longitudinal stringers with attendant loss of transverse mechanical properties, no confidence can be expressed in this material, at its present stage of development, as a substitute for the boron carbide version of the BATH composition. For these reasons, and since concurrent development of the reference composition was successful, further development of the BN-BATH composition was suspended.

6.1.5 Miscellaneous Experimentation

Before standard extrusion methods were adopted for the production of BATH material, several trials were made using the Dynapak method to compact and consolidate the powders. (Dynapak extrusion is a high-velocity, high-energy method, as contrasted to the low velocity and low energy of conventional extrusion practice.) Two-inch diameter extrusions of the reference composition were successfully fabricated without cracks, but the density achieved was only 83 percent of the theoretical 2.94 gms/cc. Only a cursory check was made of the properties of this material. The ultimate strength was only about 12,000 psi (contrasted to over 18,000 psi for conventionally extruded reference material), the elongation was less than 2 percent, and the thermal conductivity was about 55 BTU/hr/ft/°F (compared with 74 BTU/hr/ft/°F for the reference material), all at 80°F. Three-inch diameter extrusions were also made by this method, but excessive cracking and porosity was observed. As a result of the lower properties this method was abandoned in favor of standard extrusion practice.

Experiments were conducted early in the program to determine if extruded bar could be further reduced into strip by conventional rolling techniques, in the event that the shield design called for thin sections. Samples 1/2 inch thick of the 1-1/4 inch diameter extruded bars were rolled at 550°F and 700°F without any surface conditioning prior to rolling or between passes. Edge cracks formed at a thickness of 0.280 inch in the material heated to 550°F before rolling. Reductions were continued to a thickness of 0.115 inch before the cracks began to propagate across the sample width. At the 700°F rolling temperature, slight edge cracking began at a thickness of 0.238 inch. However, this sample

was continued in process to 0.060 inch thickness without extensive crack propagation. These experiments indicated that this material could be successfully fabricated into plate and strip by giving proper attention to surface condition and rolling ingot temperature. Further work such as determination of maximum possible reductions, was dropped since the shield design finally required material in bar form.

6.2 THE REFERENCE PROCESS

The reference process was optimized as a result of the extrusion of three lots of 2-1/2 inch diameter bars of the 70 v/o Al composition. Earlier extrusions of smaller diameter (1-1/4 inch) bars of the same composition were needed to identify the major processing variables in compacting and extrusion, i.e. a high ram speed (greater than 50 ipm.), billet temperatures above 550°F, an extrusion ratio (based on cross-sectional areas of billet and extruded bar) of about 20:1, and the use of a high viscosity lubricant.

The first 2-1/2 inch diameter batch (lot no. 97265, see Table XIX, Appendix), yielded sufficient material for a thorough investigation of the major mechanical and physical properties and chemical composition, results of which have been reported in detail previously⁽¹⁾. In general, this early material met the mechanical, physical and chemical program objectives. Significant anomalies, in the data, i.e. in the Young's Modulus and thermal conductivity data, have been attributed to inhomogeneities, as discussed in Section 4.1 above. Revised data, using samples of the same batch of material pre-selected to avoid the inhomogeneities, have been discussed in Section 5.0 above. The contributions of this extrusion run were (a) identification of the need to carefully blend the powders to avoid inhomogeneities and (b) verification with large diameter bar (versus earlier 1-1/2 inch bar) of the need for sufficiently high billet temperatures, about 720°F, and sufficiently high extrusion ram pressure (50-80 kpsi) and ram speed (greater than 50 in./min.) to keep the "pine-cone" effect to a minimum (one to three feet at the initial end of each bar) in this run; see Figure 10, and (c) verification of easier machining compared to earlier material, due to a smaller B₄C particle size (-325 mesh versus 200 mesh).

The second batch of 2-1/2 inch diameter bar (lot no. 08989), was blended more carefully than the first, by using an agitator bar in the V-blender to comminute the titanium hydride agglomerates. (Titanium hydride tends to agglomerate whenever it encounters moisture in the atmosphere. This tendency is probably accentuated during periods of high atmospheric humidity; therefore dryness of the powder during storage and blending is essential). A further note concerning the titanium hydride inhomogenities is in order. It has been earlier reported⁽¹⁾ that the effect of the agglomerates on the mechanical strength properties is not great, as shown by comparison with earlier, homogeneous material (hex bar). However, because of the effect on the elastic modulus and thermal conductivity, homogeneity is considered necessary for material specification purposes.

Metallographic examination of cross-sections of the extruded bar, taken at three positions in each bar, show no evidence of titanium hydride agglomerates and show generally evenly-distributed microconstituents. In Figure 11, three positions in a single bar (no. 5) are compared with a single position in another bar (no. 1) to show their similarity. These sections are representative of all sections taken in each of the five bars of this lot.

This material, therefore, is expected to show no data anomalies. A study of the mechanical and physical properties of this material is underway and the results will be reported later.

Fracture mechanics studies were initiated using this material. Acceptable fracture toughness specimens have been machined. Preliminary tests have been made and are being evaluated.

The extrusion parameters of this batch are listed in Table XIII. The yield of acceptable material (judged visually) was quite high, with an average bar length of 15 feet, and with "pine-cone" being minimized at the highest temperatures and with the fastest extrusions, as illustrated in Figure 12. In this figure, the dark striations are superficial surface stains from the extrusion press runout table and storage rack. Surface roughness, or grooving, can be seen on the nose section at the right of the figure. This defect was caused by material

pickup on the die from the previous extrusion and can be eliminated by changing dies prior to each extrusion, as is discussed below. The lack of straightness at the nose of the extrusion was due to the absence of guide rails on the runoff table, an error which was corrected on the subsequent run.

Chemical analyses of various sections of each bar were made, with results as given in Table XIV, which shows that the as-processed hydrogen content is within the program objectives.

Close inspection of the surface of the extrusions showed tiny circumferentially-oriented cracks, which penetrate the surface of the material to a maximum depth of less than 8 mils (see Figure 13). A much greater amount of material is normally removed during subsequent machining into the shield modules. Cracks of this nature are also smaller in depth than the amount of metal removal expected in bars which will be manufactured to "near" design-size and shape; therefore, they do not present a materials problem. In summary, this run of material is expected to show the beneficial effect of careful powder blending on the mechanical properties, and has shown the advantage of high extrusion temperatures (760°F) and high extrusion speed (about 100 ipm) in eliminating the "pine-cone" effect. In addition, a large amount of material was made available for other testing purposes, including additional fracture mechanics studies, flexure (bend) tests, machining of shield modules, and radiation streaming studies, results of which will be reported separately.

The third and latest 2-1/2 inch diameter extrusion run (lot no. 15627) was made to verify the results of the previous run and to determine the utility of using aluminum alloy sections fore and aft of the BATH billet in the extrusion die as a means of further increasing the yield of useful material. Use of the fore-piece was designed to eliminate the "pine-cone" effect under marginal temperature conditions. Use of the aft-piece was designed to permit extrusion of a greater length of the original billet than has been possible previously. (A 3-4 in. long by 11 in. diameter section of BATH is normally unextruded, since the process is stopped before the extrusion ram reaches the die wall.) The experiment with the fore-piece (billet no. 2, Table XV) was unsuccessful, since the aluminum (alloy 7075) coextruded back

over the BATH bar with many severe surface cracks. The 7075 alloy was selected since the vendor's* experience with this material indicated that its extrusion characteristics are similar to those for BATH. It is possible that this technique could be successful if an alternate aluminum alloy or a different die design were used. However, further development of this concept is probably unwarranted since the "pine-cone" defect can be minimized by proper control of the extrusion press and billet temperature, as discussed above.

The use of an aluminum piece against the aft end of the BATH billet was similarly unsuccessful since the aluminum alloy intrudes into the BATH bar to an unacceptable degree.

An important result of this run was the observation that BATH material strongly adheres to the die surface after an extrusion. The adhering material is not forced out of the die during the initial part of the next extrusion, contrary to experience in the extrusion of commercial aluminum alloys, but remains largely in situ, causing severe galling or scoring of the extruded BATH bar. In this run, a single die was used for three extrusions. The first showed only a short "pine cone" in the nose section. The second extrusion used the 7075 aluminum alloy nose piece previously discussed. The third run showed severe surface galling. The subsequent two runs, in which a fresh die was used for each, showed results comparable to the first run, in which only a short "pine cone" section, and no galling were observed.

A summary of the extrusion press data for this run is given in Table XV. The die I.D.'s were measured before and after extrusion, at identical temperatures, with results as tabulated in Table XVI, which shows an average die wear of 1 mil with each extrusion.

Evaluation of all of the reference composition BATH runs have resulted in the specification of the combination of extrusion parameters yielding the best results, as given in Table XVII.

The mechanical and physical properties of the latest batches of 2-1/2 inch diameter bar have not yet been evaluated and will be the subject of a separate report. Comparison of the available results i.e., chemical composition, visual appearance, extrusion

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* Aluminum Corp. of America, Lafayette Works.

parameters, of the three large-diameter extrusion runs indicates that the properties of the last two lots are also expected to be within the program objectives.

The compositions of the raw materials of the last two runs is given in Table XVIII. These compare well with the compositions of the raw materials used in the first 2-1/2 inch diameter bar run.⁽¹⁾

7.0 SUMMARY AND CONCLUSIONS

Conventional extrusion has been shown to be a workable process for producing a high yield of BATH shield material of the reference composition, which meets the program objectives for mechanical and physical properties and chemical compositions. The process development progressed from small bar sizes to a size acceptable for the shield design. Several compositional variants were studied and discarded when it became certain that acceptable material of the reference composition could be produced.

Machinability was significantly improved through the use of fine particle size (-325 mesh) B_4C , although even greater ease of machining is desirable. (The very easily machined BN version was discarded due to low yield of intact material and poor transverse mechanical properties). Homogeneity of the powdered constituents, primarily titanium hydride, was found to be important for uniformity of properties (elastic modulus and thermal conductivity). The extrusion parameters have been optimized (for round bar). The most significant parameters are:

- (a) adequate temperature, 750 -760°F is recommended,
- (b) rapid extrusion of the material through the die as soon as "breakout" is achieved,
- (c) die must be thoroughly cleaned or a fresh die must be used prior to each extrusion to consistently yield good quality surfaces,
- (d) proper attention to the billet temperature, extrusion speed and die condition can nearly eliminate "pine-cone" and
- (e) use of a high-viscosity lubricant produces the best results.

In conclusion, the development of a process for producing acceptable shield material of the BATH reference composition in sizes suitable for machining to design configuration, has been completed. Accordingly, WANL Purchasing Department Specification No. 30259 has been prepared. Future developmental work is presently designed to

adapt the extrusion process to produce material as close to the design configuration as possible, thereby minimizing the amount of material to be machined, further reducing machining time and costs.

8.0 TABLES

TABLE I
BATH Shield Material Reference Composition

<u>Component</u>	<u>Volume Percent</u>	<u>Weight Percent</u>
TiH _x *	23-24	29.5-30.8
B ₄ C	5.5-6	4.7-5.1
Al	70 balance, nominally	64.1 balance, nominally
Total Impurities	1% max.	

* In the stoichiometry of TiH_x, where "x" is the hydrogen concentration, x must be 1.81 minimum as fabricated. After irradiation to a fluence of 10¹⁹ nvt, x must not be less than 1.51 due to loss of hydrogen.

TABLE II

Modulus of Elasticity

LONGITUDINAL & TRANSVERSE SAMPLES

Temp. °F	$\bar{X}$ psi ($\times 10^6$)	n	S	S (previous) ⁽¹⁾
-320	9.10	8	0.538	0.967
-120	8.53	8	0.538	0.967
80	8.73	8	0.538	0.967
400	6.85	8	0.538	0.967

$$f = 24$$

$\bar{X}$ refers to the mean value of a number of observations "n".

S is the statistical standard deviation with f degrees of freedom.

TABLE III
Tensile Yield Strength *
LONGITUDINAL SAMPLES

<u>Temp. °F</u>	<u>$\bar{X}$ ksi</u>	<u>n</u>	<u>S</u>	<u>S (previous)⁽¹⁾</u>
-320	15.39	8	0.343	0.678
-120	13.73	8	0.343	0.678
80	12.03	8	0.343	0.678
400	10.13	8	0.343	0.678

f = 23

TRANSVERSE SAMPLES

<u>Temp. °F</u>	<u>$\bar{X}$ ksi</u>	<u>n</u>	<u>S</u>	<u>S (previous)⁽¹⁾</u>
-320	15.09	8	0.343	0.678
-120	13.42	8	0.343	0.678
80	11.72	8	0.343	0.678
400	9.82	8	0.343	0.678

f = 23

$\bar{X}$ refers to the mean value of a number of observations "n".

S is the statistical standard deviation with f degrees of freedom.

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* 0.2% Offset

TABLE IV
Ultimate Tensile Strength
 LONGITUDINAL SAMPLES

<u>Temp. °F</u>	<u>$\bar{X}$ ksi</u>	<u>n</u>	<u>S</u>	<u>S (previous)⁽¹⁾</u>
-320	22.68	4	0.307	0.95
-120	19.58	4	0.307	0.95
80	18.75	4	0.307	0.95
400	12.78	4	0.307	0.95

f = 12

TRANSVERSE SAMPLES

<u>Temp. °F</u>	<u>$\bar{X}$ ksi</u>	<u>n</u>	<u>S</u>	<u>S (previous)⁽¹⁾</u>
-320	17.93	4	0.954	0.95
-120	16.43	4	0.954	0.95
80	13.60	4	0.954	0.95
400	11.50	4	0.954	0.95

f = 12

$\bar{X}$ refers to the mean value of a number of observations "n".

S is the statistical standard deviation with f degrees of freedom.

TABLE V
Total Elongation
LONGITUDINAL & TRANSVERSE SAMPLES

<u>Temp. °F</u>	<u>$\bar{X}$ %</u>	<u>n</u>	<u>S</u>	<u>S (previous)⁽¹⁾</u>
-320	1.09	8	0.852	0.536
-120	1.14	8	0.852	0.536
80	2.24	8	0.852	0.536
270	2.83	8	0.852	0.536
400	3.19	8	0.852	0.536

f = 24

$\bar{X}$ refers to the mean value of a number of observations "n".

S is the statistical standard deviation with f degrees of freedom.

TABLE VI
Poisson's Ratio
 LONGITUDINAL SAMPLES

<u>Temp. °F</u>	<u>$\bar{X}$</u>	<u>n</u>	<u>S</u>	<u>S (previous)⁽¹⁾</u>
-320 to 400	0.318	16	0.055	0.069

f = 13

TRANSVERSE SAMPLES

<u>Temp. °F</u>	<u>$\bar{X}$</u>	<u>n</u>	<u>S</u>	<u>S (previous)⁽¹⁾</u>
-320	0.412	4	0.055	0.069
-120	0.322	4	0.055	0.069
80	0.228	4	0.055	0.069
400	0.330	4	0.055	0.069

f = 13

$\bar{X}$ refers to the mean value of a number of observations "n".

S is the statistical standard deviation, with f degrees of freedom.

TABLE VII

Mean Coefficient of Linear Thermal Expansion

Temp. °F	$\bar{X}^*$ as 10^{-6} in/in-°F	n	S
-400	7.94	20	0.334
-300	8.38	20	0.318
-200	8.81	20	0.294
-100	9.24	20	0.275
0	9.67	20	0.260
100	10.01	20	0.268
200	10.51	20	0.257
300	10.94	20	0.203
400	11.36	20	0.275

* Indicated values are the average between 80°F and the indicated temperature of a number of observations, "n." "S" is the statistical standard deviation.

TABLE VIII
Billet Data Hollow Hexagonal Extrusions
 (Batch No. 02481)

<u>Billet Number</u>	<u>Pre-Extrusion</u>		<u>Wall Variance (Inches)</u>	<u>Weight (lbs.)</u>	<u>Length (Inches)</u>	<u>Extrusion Size⁽¹⁾ (Inches)</u>	<u>Post-Extrusion I.D. (Inches)</u>
	<u>O. D. Inches</u>	<u>I. D. Inches</u>					
1	8.996	1.040	0.012	61	9-7/8	2.132	0.993
	8.994	1.046	0.007			2.131	
						2.131	
2	9.005	1.046	0.016	63	10	2.137	0.994
	9.007	1.051	0.006			2.135	
						2.135	
3	9.003	1.064	0.020	57	9	2.149	0.998
	9.006	1.056	0.010			2.147	
						2.147	
4	8.998	1.029	0.015	62	9-9/16	2.142	0.995
	8.997	1.033	0.012			2.142	
						2.155	
5	9.000	1.025	0.007	65	10	2.144	0.995
	9.000	1.027	0.007			2.143	
						2.141	
6	8.999	1.032	0.014	61	9-3/4	2.147	0.990
	9.004	1.027	0.014			2.145	
						2.146	

(1) Extrusion size is average across flats, shown for three locations (nose, center, tail).

TABLE IX
Extrusion Data - Hollow Hexagonal Extrusions (Batch No. 02481)

Billet Number	Billet Temp. (°F)	Furnace Time (hrs.)	Upset Pressure ⁽¹⁾ (psi)	Running Pressure ⁽¹⁾ (psi)	Ram Speed (ipm)	Extrusion Time (sec.)	Extrusion and Tool Condition
1	835	21.6	1900	1500	50	10.7	Slight edge cracking, severe tool galling
2	835	22.5	2300	2000	130	6.3	Best extrusion, least edge cracking, slight tool galling
3	835	22.9	3050	2700	130-150	5.3	Severe edge cracking; least tool galling
4	800	17.1	2050	1700	150	4.9	Mild edge cracking; slight tool galling
5	800	17.4	2500	1750	130	6.1	Mild edge cracking; most severe tool galling
6	800	17.9	2400	1900	110	6.5	Severe edge cracking; slight tool galling

DATA PERTINENT TO ALL RUNS:

Liner Diam.	9.188"
Liner Temp.	800°F
Die Size	2.150" across flats
Die Temp.	700°F
Cone Angle	90° Incl.
Lubricant ⁽²⁾	FISKE 604-D
Reduction (area) Ratio	21:1

NOTES:

- (1) Extrusion pressures are to ± 25 psi.
- (2) In addition to this lubricant (used on liner, die, cone and mandrel), the billets were precoated with a mixture of colloidal graphite, molybdenum disulfide and oil.

TABLE X
Tensile Data - Hollow Hexagonal Extrusions
(Batch No. 02481)

<u>Specimen Number</u>	<u>Temp. °F</u>	<u>Yield Strength 0.2% Offset ksi</u>	<u>Ultimate Strength</u>	<u>Measured Elongation</u>	<u>Young's Modulus</u>
1	-320	17.9	21.6	1.67	9.61
5	-320	17.7	22.9	4.16	---
9	-320	18.0	21.3	0.83	8.09
13	-320	18.1	22.3	2.50	---
2	-120	14.6	18.8	4.16	---
6	-120	14.0	17.9	1.67	---
10	-120	14.1	18.7	1.67	---
14	-120	13.0	17.2	2.50	---
3	80	13.8	18.2	4.16	10.7
7	80	13.3	17.8	5.0	---
11	80	13.2	18.1	4.16	8.76
15	80	12.4	17.2	3.30	---
4	480	7.6	9.3	5.8	---
8	480	8.0	9.6	5.8	---
12	480	7.7	9.3	9.17	---
16	480	7.9	9.1	5.0	---

NOTE: All data are for samples whose gage length is parallel to the direction of extrusion, designated as the longitudinal direction.

TABLE XI

Machinability of Shield Material Containing Either B₄C or BN as Boron Carrier - Early Tests

Operation*	Tool	Cutting Speed (rpm)	Feed (in.)	Depth of Cut (in.)	Tool Wear, in. for		Remarks
					B ₄ C Shield**	BN Shield	
Turning	High Speed	370	0.005	0.050	0.030	< 0.001	
	Carbide Tip	370	0.005	0.050	0.013	None	Also used 580 rpm on BN
Threading 100-12NS	High Speed	125		0.005/each pass	0.020	None	Rough torn finish on all threads with B ₄ C. Smooth finish on root of threads with BN. Top of thread slightly chipped with BN.
Drilling	1/2" dia. High Speed	370		0.750	0.005	None	
Milling	High Speed End 3/4"	825	2-3/8 in/min	0.2	0.030 max. + 0.005 taper in 0.8 in.	None in 0.8 in.	
	High Speed End 7/32"	1000	0.002 in/rev.	2 holes in B ₄ C 3 holes in BN 5/8"	0.010	None	With B ₄ C, end mill became loaded with mat'l. Hole drilled oversized and tapered 0.020 in. from top to bottom, chipping on corners. With BN, good hole and no chipping was observed.
	High Speed Saw Cutter 5/32" x 3" diameter	150	1-3/8 in/min	5/16	0.010	None	Slight chipping where cutter broke through.

* No cutting fluid used. ** Early material, average boron carbide particle size, about 140 micron.

TABLE XII

Longitudinal Tensile Data
for 1-1/4" Diameter BN-BATH Compared to 2-1/2" Diameter B₄C-BATH Material

Temp. °F	Yield Strength (ksi)		Ultimate Strength (ksi)		Elongation (%)	
	BN (S = .735)	B ₄ C (S = .343)	BN (S = .413)	B ₄ C (S = .307)	BN (S = 1.5)	B ₄ C (S = .852)
-320	20.8	15.4	25.3	22.7	2.5	1.1
80	14.8	12.0	20.6	18.8	4.6	2.2
480	9.4	9.95*	11.2	12.3*	6.2	3.4*

* Extrapolated

NOTES: 1. S is the statistical standard deviation.
2. The B₄C-BATH data are taken from Tables III, IV and V.

TABLE XIII

Extrusion Press Data, Second Batch of 2-1/2 Inch Diameter Bar (Lot No. 08989)

Billet Iden. Number ⁽¹⁾	Cylinder Temp. (°F)	Billet Temp. (°F)	Rod Temp. at End of Charge (°F)	Average Ram Speed ipm ⁽²⁾	Maximum Breakout Unit Press. (lbs./in. ²) ⁽³⁾	Minimum Running Unit Press. (lbs./in. ²) ⁽³⁾	Remarks
1	690	710	900	42	74,280	55,240	(4)
2	700	715	900	42	70,470	55,240	(4)
3	700	760	920	60	70,470	51,430	(5)
4	700	760	920	58	68,570	51,430	(5)
5	700	760	920	60	68,570	49,520	(5)

(1) Extruded rod numbered on the rear-most end of each cut. X is the rear cut, X-1 is the front cut.

(2) Calculated from average speeds of the extruding bars.

(3) Liner Dimension - Sealing End 11.245" dia.
Middle 11.244" dia.
Pressure Ram End 11.250" dia.

(4) Pine cone at nose of extrusion, about 3 ft. in length.

(5) Best yield of material, no "Pine-Cone" defect.

NOTES: Die Code XH1

Furnace Charged 4:30 p.m. 10/10/69

Furnace Setting: 800°F

After Charge No. 2, furnace setting
increased to 850°F

Extrusion Ratio: 20:1

TABLE XIV
BATH Shield Material Chemical Composition
Second Batch of 2-1/2 Inch Diameter Bar (Lot No. 08989)

Extrusion Sequence (Bar No.)	Sample Position In Bar	Ti Weight %	H ₂ ⁽¹⁾ Weight %	B ₄ C Weight %	TiH _x Average Weight %	X in H _x ⁽²⁾
1	Nose	29.5	1.12	5.60	30.65	1.81
	Center	29.7	1.11	5.53		
	Tail	29.4	1.11	5.46		
2	Nose	30.3	1.12	5.47	31.12	1.80
	Center	29.7	1.12	5.47		
	Tail	30.0	1.11	5.50		
3	Nose	30.0	1.13	5.38	31.02	1.81
	Center	29.8	1.12	5.36		
	Tail	29.9	1.11	5.42		
4	Nose	29.8	1.11	5.45	30.68	1.81
	Center	28.9	1.11	5.45		
	Tail	30.0	1.11	5.51		
5	Nose	29.3	1.14	5.36	30.73	1.84
	Center	29.6	1.13	5.38		
	Tail	29.9	1.12	5.41		

NOTES:

(1) Hydrogen was determined by a combustion procedure with the hydrogen collected as water.

(2) See Appendix for sample calculation.

TABLE XV

Extrusion Press Data, Third Batch of 2-1/2 Inch Diameter Bar (Lot No. 15627)

Billet Iden. No. (1)	Cylinder Temp. (°F)	Extrusion Temp. (°F)	Rod Temp. at End of Charge (°F)	Average Ram Speed, ipm. (2)	Maximum Breakout Unit Press. (lbs/sq. in) (3)	Minimum Running Unit Press. (lbs/sq.in) (3)	Remarks
1 (XH3)	700	720	900	32	74,300	58,500	(4)
2 (XH3)	700	730	890	16	85,700	56,500	(5)
3 (XH3)	700	700	870	27	80,600	58,500	(6)
4 (XH2)	700	730	890	38	80,600	58,500	(7)
5 (XH1)	700	680	870	36	82,500	58,500	(8)

NOTES:

(1) Extruded rod numbered on the rear most end of each cut. X is the rear cut, X-1 is the front cut. Parenthesis indicates die used.

(2) Calculated from average speeds of the extruding bars.

(3) Liner Dimension -   Sealing End   11.245" Dia.
                                          Middle   11.244" Dia.
                                          Pressure Ram End   11.250" Dia.

Billets charged 3:00 p.m. 7/14/70; billets discharged about 11:00 a.m. 7/15/70; furnace setting 850°F.

(4) 4" thick 7075 cap at 700°F used on rear end of billet. First 1-1/2' of ext. length "pine coned".

(5) 4" thick 7075 cap at 620°F used on front and rear of billet. First 28" good surface, next 4' severely broken and torn surface.

(6) 4" thick 7075 cap at 700°F used on rear of billet. Surface very rough, pickup on die.

(7) 4" thick 7075 cap at 710°F used on rear of billet. Pickup on die. First 2-1/2' of extruded length "pine coned".

(8) 4" thick 7075 cap at 700°F used on rear of billet. First 2' of extruded length "pine coned".

(9) Extrusion Ratio 20:1.

TABLE XVI
Die Wear, Extrusion of BATH Lot No. 15627 *

<u>Die Insert Identification</u>	<u>Number of Extrusions Through Die</u>	<u>Die Insert, Measured I. D.</u>	
		<u>Before</u>	<u>After</u>
XH1	1	2.534	2.535
XH2	1	2.536	2.537
XH3	3	2.531	2.534

NOTE: Each die is made of AISI-type H12 tool steel; nominal composition
 0.35C, 1.5 W, 1.5 Mo; 5.0 Cr, 0.40 V.

Average die wear, 1 mil with each extrusion.

* See Table XV.

TABLE XVII
Optimum Extrusion Variables
2-1/2 Inch Diameter Bar

Extrusion Ratio	20:1
Billet Temperature	760°F
Ram Speed (Avg.)	50-60 ipm.
Cylinder Temperature	700°F
Minimum Running Unit	
Pressure (kpsi)	50-60
Lubricant	high viscosity, (lead-base)

TABLE XVIII
 Compositions of Raw Materials
 (For Extrusion Lot Nos. 08989 and 15627)*
 (Weight Percent)

	<u>Aluminum Powder</u>	<u>Titanium Hydride</u>	<u>Boron Carbide</u>
Al (free)	99.09	0.01	0.036
Al (by difference)	99.79	---	---
Ti	---	95.5	---
H ₂	---	3.8	---
B	---	---	76.11
C	---	---	22.32
B + C	---	---	98.43
B ₂ O ₃	---	---	0.12
Ca	---	0.02	0.01
Mg	---	0.01	0.003
Fe	0.12	0.03	0.04
Si	0.07	0.02	---
Mn	0.00	---	---
Zn	0.00	---	---
Zr	---	0.04	---
Others, each	0.02	---	---
Others, total	0.02	---	---
Mesh size	99.5% thru 200 mesh	99.8% thru	100.0% thru 200 mesh
(bulk)	85.0% thru 325 mesh	325 mesh	78.7% thru 400 mesh

*As determined by suppliers.

TABLE XIX
Materials Summary of BATH Extrusion Development Runs

Size	Composition/ Designation (1)	Lot No. Extrusion (2)	Date	Vendor	Lot No. IsoPress (2)	Date	Vendor	Powder	Lot No. Description (2)	Vendor	Pounds
1-1/4" Dia.	70/30 B ₄ C/OD, E 75/25 B ₄ C/B, C 80/20 B ₄ C/OA, F*	95884	10/18/67	Whittaker	97264	10/6/67		Al TiH B ₄ C	95886 95885 94004	Reynolds, 30006-B, ChemPly Grade E Ventron Nuc. Grd. 325, Norton	600 20 10
2-1/2" Dia.	80/20 B ₄ C/1,2,4, 5,6 70/30 B ₄ C/3,7*,8, 9,10	97265	5/1/68	Alcoa	97264		Sylvania	Al TiH B ₄ C	94412 96064 94409	30006-B, ChemPly Grade E, Ventron -325 mesh, Carborundum	800 200 60
1-1/4" Dia.	70/30 B ₄ C/1,2,3, 4,5 70/30 BN/6*,7,8	02435	9/18/68	Reactive Metals	99418	9/11/68	Sylvania	Al TiH B ₄ C	97452 99447 97451	30006-Alcoa 123 Grade E, Ventron Nuc. Grd.-100 +325, Carborundum Hi Purity (Fine)	600 500 60 10
Hex	70/30 B ₄ C/4,5,7, 10	02481	12/13/68	Reactive Metals	99418	9/11/68	Sylvania	Al TiH B ₄ C	97452 99447 97451	See above See above See above	600 500 60
Hex	70/30 B ₄ C/1,2	02481	12/13/68	Reactive Metals	99418	9/11/68	Sylvania	Al TiH B ₄ C	97452 99447 94409	See above See above See above	600 500 120
1-1/4" Dia.	70/30 BN/1*,2*, 3*	06447	2/6/69	Whittaker	94418	9/11/68	Sylvania	Al TiH BN	97452 99447 03811	30006-Alcoa 123 Grade E, Ventron Hi Purity (Fine), Carborundum	600 500 10
Canned Hex	70/30 B ₄ C/1,2, 3,4	07530	5/27/69	Whittaker	99418	9/11/68	Sylvania	Al TiH B ₄ C	97452 99447 97451	See above See above Nuc. Grade, Carborundum	600 500 60
3/4" dia. to 1" dia.	70/30 B ₄ C/1-27	07527	6/10/69	Whittaker	99418	9/11/68	Sylvania	Al TiH B ₄ C	97452 99447 97451	See above See above See above	600 500 60
3/4" to 1" dia.	70/30 BN/1-132	07527-2	10/3/69	Whittaker	07528	6/16/69	Sylvania	Al TiH BN	09457 09454 09455	Alcoa Ventron Carborundum	1800 1000 100
3/4" to 1" dia.	70/30 BN/1-27	09249	8/4/69	AFB ® No cast P.O. Wright Patterson	07528	6/16/69	Sylvania	Al TiH BN	09457 09454 09455	Alcoa Ventron Carborundum	1800 1000 100
2-1/2" dia.	70/30 B ₄ C/1,2,3, 4,5	08989	10/23/69	Alcoa Lafayette Ind.	07528	6/16/69	Sylvania	Al TiH B ₄ C	09457 09454 09456	Alcoa Ventron Carborundum	1800 1000 102
2-1/2" Dia.	70/30 B ₄ C/1,2*, 3*,4,5	15627	7/15/70	Alcoa, Lafayette Ind.	07528	6/16/69	Sylvania	Al TiH B ₄ C	09457 09454 09456	See above See above See above	1800 1000 102

(1) OD, E, B, 1, 2, 3, etc. refer to identifications of individual bars.

(2) Lot No. is identical to the WANL Purchase Order number for traceability purposes.

* Bars marked by asterisk were scrapped.

9.0 ILLUSTRATIONS

FIGURE 1

Titanium Hydride Agglomerate in Early BATH Material
Tensile Specimen (Lot No. 97265) Magnification: Approx. 7X

FIGURE 2
Flow Diagram of BATH Manufacturing Process

FIGURE 3
Isostatically Compacted Billet - BATH Reference Composition

FIGURE 4

Extruded Hexagonal Bar - First Trial
(Lot No. 02481)

(a)
Plain Hexagonal Section

(b)
Hexagonal Section with
Pre-formed Grooves

FIGURE 5
Canned Billet Assemblies - For Hexagonal Bar Extrusion, (Lot No. 07530)

(a)
Plain Hexagonal Section

(b)
Hexagonal Section with
Pre-Formed Grooves

FIGURE 6
Canned Hexagonal Extrusions - Cross-Sections

About 1/3 Actual Size

Top: Representative Section of Best Material

Bottom: Typical "Pine-Cone" Effect

FIGURE 7

Extruded BATH Bars, Containing Boron Nitride

Magnification 4X
Section Through Bar Where Ultrasonic
Defect Indications Were Found

FIGURE 8
Extruded BATH Bar Cross Section - Boron Nitride Variant

Magnification 50X

White Angular Particles are TiH
Black Stringers are BN
Matrix is Aluminum

FIGURE 9

Longitudinal Section of Extruded BATH Bar
Containing Boron Nitride

FIGURE 10

Localized Extrusion Defect Termed "Pine-Cone"
First Batch of 2-1/2 Inch Diameter Bar
(Lot No. 97265)

Light Angular Particles are Titanium Hydride.
Dark Angular Particles are Boron Carbide.

(a)
Bar No. 5 Nose of Extrusion 100X

(b)
Bar No. 5 Center of Extrusion 100X

FIGURE 11

Photomicrographs of BATH Bar Cross Sections - Second Batch of 2-1/2 Inch Diameter Bar (Lot No. 08989).

(c)

Bar No. 5 Tail of Extrusion 100X

(d)

Bar No. 1 Center of Extrusion 100X

At Right: Nose of Extrusion. This Bar was Bisected after Extrusion. The Black Striations are Superficial Stains from the Extrusion Runoff Table and Storage Rack.

FIGURE 12

BATH Bar Showing Elimination of "Pine-Cone" Defect
(Bar No. 4, Lot No. 08989)

Mounting
Material

BATH

Magnification 100X

NOTE: Maximum Crack Depth from Surface
is Less Than 8 MILS.

FIGURE 13

Photomicrograph of Generally-Distributed Circumferential
Surface Cracks on Extruded BATH Bar

10.0 REFERENCES

- (1) Initial Characterization of BATH Shielding Material, WANL-TME-1947, Aug. 1969, F. D. Seaman.
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- (3) "Objectives for the NR Shield Material Development Program", memo no. ARD 270 (CRD) from Lloyd Cooper to D. C. Goldberg, June 8, 1967.
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- (5) "Irradiation of BATH Shield Material at Ambient Temperatures," WANL-TME-1898, March 1969, R. P. Shogan.
- (6) BATH Shield Module Thermal Shock Test, WANL Test Scope 62 DE-189.
- (7) Summary of Analyses on Some BATH Material Data, DRM-52284, W. H. Horton, May 12, 1970.

APPENDIX

SAMPLE CALCULATION

Calculation of stoichiometric ratio of hydrogen to titanium in TiH_x (for bar 5, Table XIV).

1. Weight percent Ti = 29.6% (by analysis)
2. Weight percent H = 1.13% (by analysis)
3. Weight percent TiH_x = 30.73% (add 1 and 2)
4. Weight percent H in TiH_x :

$$\frac{1.13}{30.73} \times 100 = 3.68$$

5. Weight % H in TiH_2 (stoichiometric composition):

$$\frac{2}{49.9} \times 100 = 4 \text{ w/o}$$

6. Ratio of H to Ti in bar 5, Table :

4% H : 3.68% H as 2 atoms in TiH_2 : X atoms in TiH_x

$$\frac{4}{3.68} = \frac{2}{x}$$

$$x = \frac{2}{4} (3.68) = 1.84$$

GENERAL:

$$"x" \text{ in } \text{TiH}_x = 0.5 \times \text{w/o H in } \text{TiH}_x$$

	TITLE	
	BATH Shield Material Development	
DOCUMENT NO.		DATE
WANL-TME-2737		October 1970
ABSTRACT		
The development of the manufacturing process and the materials properties of recent BATH (boron-aluminum-titanium hydride) round bar stock are discussed. Conventional extrusion of powder metallurgical compacts of the component powders is shown to be a practical process for producing BATH shield material which meets the program objectives for mechanical and physical properties. Optimum extrusion variables are listed.		
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