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FABRICATION
OF
CERAMIC INTERNAL REFLECTOR
FOR THE
SNAP 8 EXPERIMENTAL REACTOR

AEC Research and Development Report

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.

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By
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ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.
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ABSTRACT

This report describes the fabrication of internal reflector pieces for the SNAP 8 core. These reflectors were made of BeO, with and without the addition of Sm_2O_3 as a nuclear poison.

Because of the high density and dimensional tolerance requirements, the complexity of shapes, and the comparatively modest number of parts to be produced (approximately 1000), the blanks were hot pressed and subsequently machined with diamond wheels and cores. In almost all cases, the specified density of 98% of theoretical was achieved. The low eutectic temperature in the BeO- Sm_2O_3 system of about 1420°C necessitated special pressing parameters, which were arrived at by experimentation.

A rather coarse grit size (80) was used for the diamond wheels, which resulted in very little wear to the wheels, and an extremely low dimensional rejection rate on the blanks.

Because of the toxicity of BeO, all equipment was enclosed, and was held under negative pressure.

I. INTRODUCTION

A. BACKGROUND

The internal reflector pieces, the fabrication of which is described in this report, are intended for a test with the SNAP 8 Experimental Reactor. The SNAP 8 reactor is a larger version of the SNAP 2 reactor, and is designed to produce 30 kwe with one power conversion unit, and 60 kwe with two power conversion units, coupled to the reactor. The SNAP 8 reactor utilizes a homogeneous fuel moderator of zirconium hydride containing U^{235} . The reactor is partially reflected by BeO reflector pieces, located between the array of fuel elements and the core vessel. Poison in the internal reflector may provide a safeguard feature; safety elements poisoned with Sm_2O_3 may be evaluated in the experimental program.

B. SCOPE OF FABRICATION

The total fabrication effort called for 48 complete No. 1 BeO insert assemblies and 24 complete No. 2 BeO insert assemblies (Figure 1). Of this amount, 12 complete No. 1 assemblies and 6 complete No. 2 assemblies had to contain 8.85 wt % of Sm_2O_3 .¹ The design of the assemblies permitted the overall length of each insert to be comprised of an optional number of individual segments. Governed by the size limitation and by the unidirectional action of our presses, a height of about 1 in. was decided upon for the segments. As each assembly was 14.225 in. long, requiring 13 segments, the total requirements amounted to approximately 624 No. 1 segments and 312 No. 2 segments, one fourth of them containing Sm_2O_3 . The minimum density required was 98% of theoretical. The addition of 1 wt % of MgO, as a sintering promoter, was permissible. Originally, a more complete design for insert No. 1 was considered which had two curved surfaces; but later it was decided to use a plane surface adjacent to the fuel, as it was found that fabrication economies associated with the simple shape outweighed any slight nuclear disadvantages. These inserts are to be clad in Hastelloy Alloy "N"*. The cladding procedure will not be reported here.

---

*Brand name of Haynes Stellite Div. of Union Carbide Corp.

a. No. 1 Insert Assembly

b. No. 2 Insert Assembly

Figure 1. Assembly Design

C. FABRICATION CONSIDERATIONS

The high density requirement (98% of theoretical density, minimum), together with the small and complex shape and tight tolerances (Figure 1), suggested hot pressing and machining as the best fabrication method. This decision was furthered by the availability of a well equipped BeO hot pressing facility,² together with a wealth of experience in handling and hot pressing BeO,³ at Atomics International. Because of the toxic nature of BeO powder, the material preparation was handled in glove boxes under negative pressure; and, during the entire fabrication process, stringent health precautions were observed.

Figure 2. Fabrication Area

II. FABRICATION PROCESS

A. GENERAL

The fabrication of the ceramic inserts was conducted in the area shown in Figure 2.

The main steps of the fabrication process are shown in the process flow chart (Figure 3).

B. RAW MATERIALS

The following raw materials have been used in the fabrication of the BeO shapes, with and without nuclear poison.

1. Beryllium Oxide

Beryllium Corporation of America, Grade 1, high purity beryllium oxide was used for all shapes, Blend No. 16 was used for the shapes containing nuclear poison, Blends No. 14 and 16 for all others. An analysis of this material is given in Table I. It should be pointed out that this table, following the common usage, omits anions. Analyses conducted at Atomics International showed a fluorine content, varying from 0.5 to 0.75 wt % of this material. Analysis of plates, hot pressed of this material, gave a fluorine content from 0.04 to 0.30 wt %, showing that most of the fluorine was volatilized during hot pressing.

2. Magnesium Oxide

Mallinckrodt Chemical Works Analytical Reagent Grade magnesium oxide was used. The maximum impurities content of this material is shown in Table II.

3. Samarium Oxide

American Potash and Chemical Corp, Lindsay Chemicals Div. Code 823-325 samarium oxide, Lot 72572, was used. The analysis of this material is shown in Table III.

4. Water

All water employed in processing, prior to hot pressing of the ceramic, was deionized.

WITHOUT NUCLEAR POISON

WITH NUCLEAR POISON

Figure 3. Process Flow Chart

TABLE I
CHEMICAL ANALYSIS OF BERYLLIUM OXIDE*
(%)

	Blend Number	
	14	16
Fe	0.002	0.003
Si	0.019	0.018
Al	0.006	0.007
Co	<0.001	<0.001
Pb	<0.001	<0.001
Sn	<0.002	<0.002
Ni	<0.001	<0.001
Ca	0.001	0.001
Mn	0.002	0.001
Mg	0.005	0.006
Ag	<0.001	<0.001
Cu	0.001	0.001
Na	0.025	0.035
Ca	0.007	0.005
B	0.00005	0.00005
Li	0.0001	0.0001
Cd	<0.00005	<0.00005
Be	<0.001	<0.001
Ti	<0.001	----
Mo	0.001	----
P	----	0.001
H ₂ O	0.50	0.62
LoI †	1.05	1.09

*By Nuclear Division of the Beryllium Corp.

†Loss on ignition

TABLE II
MAXIMUM LIMITS OF IMPURITIES OF MAGNESIUM OXIDE*
(%)

Ammonium Hydroxide ppt.	0.020
Ba	0.005
Ca	0.05
Cl	0.010
Heavy Metals (as Pb)	0.003
Insoluble in HCl	0.020
Fe	0.01
Mn	0.0005
NO ₃	0.005
SiO ₂	0.040
Soluble in H ₂ O	0.40
Sulfate and Sulfite (as SO ₄)	0.005
Loss on ignition	2.0

*By Mallinckrodt Chemical Works

TABLE III
CHEMICAL ANALYSIS OF SAMARIUM OXIDE*
(%)

Gd ₂ O ₃	0.016
Eu ₂ O ₃	0.009
Y ₂ O ₃	<0.005
Ce ₂ O ₃	<0.05
Nd ₂ O ₃	0.014
Th O ₂	Not Detected

*By Lindsay Chemicals Div., Am. Potash and Chemicals Corp.

5. Auxiliary Materials

A number of additional materials which were used in processing were analyzed by the Atomics International Analytical Chemistry Group. The results are shown in Table IV.

TABLE IV
IMPURITY CONTENT OF AUXILIARY MATERIALS
(ppm)*

Impurities	CARBON PRODUCTS				
	Baked† Carbon	Lamp § Black	ATS** Graphite	Porous Carbon	Carbon Rod††
Ag	< 5	< 5	< 5	< 5	< 5
Al	500	< 50	< 50	50	< 50
B	-	-	-	-	-
Be	50	< 1	100	100	10
Bi	< 10	< 10	< 10	< 10	< 10
B	1	< 1	5	5	5
Ca	50	< 10	50	50	50
Co	500	< 10	500	500	500
Cr	< 10	< 10	< 10	< 10	< 10
Cu	100	< 10	50	100	50
Fe	> 1000	< 10	500	500	500
Mg	100	10	5	50	5
Mn	50	< 10	10	50	10
Mo	< 100	< 100	< 100	< 100	< 100
Na	-	-	-	-	-
Ni	500	< 50	< 50	100	< 50
P	-	-	-	-	-
Pb	50	< 10	< 10	10	< 10
S	1.5%	< 0.1%	< 0.1%	1.6%	< 0.1%
Si	1000	> 1000	100	500	100
Sn	< 10	< 10	< 10	< 10	< 10
Ti	50	< 50	50	50	50
V	500	< 50	< 50	100	< 50
Zn	< 100	< 100	< 1000	< 100	< 100

*Unless otherwise specified

†Die plungers

§Insulation

**Dies

††Spectrographic

7513-1849 @

Figure 4. Unloading and Weighing Glove Boxes

C. POWDER PREPARATION

1. General

The operations described in this section were designed to produce mixed powders containing BeO and MgO, or BeO and Sm_2O_3 , in the specified ratio and in a form suitable for loading into the hot pressing dies.

Batching of the materials was used to make strict production control possible and to facilitate homogenization.

2. Material Without Nuclear Poison

Steel drums, each containing approximately 25 lb of BeO powder, were taken from storage and placed, one at a time, into the BeO unloading box, Figure 4. The sealed plastic bags which contained the beryllium oxide powder were then removed from the drums. Batches, weighing 700 g each, were then weighed out in the next glove box, which was connected to the unloading box. The size of the batch was dictated by the capacity of available mixing equipment. Before weighing the BeO, representative samples were removed from each drum for the loss on ignition determination (the loss on ignition was found to be consistently close to 1 wt %, and this value was used in material balance calculations). Magnesium oxide was weighed out, in the same glove box as the BeO, in the computed amount to produce a BeO/MgO ratio of 99/1. Both materials were then transferred to a mixing glove box and placed in a 1-gal capacity Waring Blender (Figure 5) containing 2800 ml of deionized water. The blender was operated at 8,000 rpm for 10 min. To prevent segregation, the slurry was quickly poured into an 11-3/4-in. diameter Buchner funnel, Figure 6, and filtered through a No. 50 filter paper. Analysis of the filtrate has shown that, while approximately 5% by weight of the MgO was dissolved, only traces of BeO passed into the filtrate. To minimize the loss of MgO, care was taken not to allow the temperature of the slurry during the blending operation to exceed 28°C; this was accomplished by cooling the blender vessel with dry ice. At this temperature, less than 1 wt % of the MgO added was found in the filtrate. The filter cake was dried by infrared heat lamps, while still in the Buchner funnel, to a moisture content of approximately 8 wt %. This predried material was then broken up by passing through an 8-mesh stainless steel screen, and approximately 400-g portions were loaded into platinum boats. The platinum boats were then transferred, using a rolling transfer glove box (Figure 7), into an

7513-1869

Figure 5. Waring Blender Being Loaded

7513-1870

Figure 6. Buchner Funnel Used for Filtering and Drying

7513-1882

Figure 7. Rolling Glove Box, Showing Platinum Boats

electrically heated, moving chain link bed, atmosphere conveyor furnace (Figure 8). The material was dried by passing it through the 22-ft long furnace in 14 hr, being exposed for 3 hr to a maximum temperature of 500°C. The thoroughly dried material was then passed through a 16-mesh stainless steel screen, weighed into 1230-g batches, and stored in plastic bottles.

3. Material With Nuclear Poison

The nuclear poison used was Sm_2O_3 . The BeO was weighed out, as described previously; and the calculated amount of Sm_2O_3 , to give 8.85 wt %, was added. One batch, containing an addition of 1 wt % MgO , was also prepared. Originally, an attempt was made to homogenize this mixture, using the wet blending procedure used with the BeO-MgO material; but it was found, on analysis of the filtrate from the Buchner funnel, that a considerable amount of the Sm_2O_3 hydrolyzed and went into solution. Therefore, this approach was discontinued, and two other methods of homogenization were tried. One consisted of blending by dry grinding in a rubber-lined jar mill. This experiment was discontinued because the material packed and stuck to the jar walls. The other method consisted of dry blending in a Twin Shell Blender with intensifier bar.

7513-1828

Figure 8. Conveyor Furnace

7513-1865A @

Figure 9. Loading Glove Boxes

7570-1871H

Figure 10. Graphite Die

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Radiographic investigation of hot pressed plates, prepared by this method, showed a very satisfactory degree of homogenization. To densify this material, an attempt was made to mix-mull it, with the addition of 8 wt % of water, and then drying it with infrared lights. But radiographic investigation of the resulting granulation revealed that some segregation of the originally well-homogenized mixture occurred. The reason for this segregation seemed to be that the small amount of water used in mix mulling was sufficient to cause solution of the Sm_2O_3 , with subsequent precipitation on drying. The hydrolysis of Sm_2O_3 is reported in the literature.⁴ The method finally adopted consisted of dry blending a 550-g batch for 30 min in a Twin Shell Blender, cold pressing the entire batch in a 5-in. diameter graphite die at 1000 psi, breaking up the pressed disk in a porcelain mortar, and screening through a 16-mesh stainless steel screen. This "slugging" operation densified the originally very fluffy powder sufficiently to make it fit the die. The granulated powder was then stored in 1280-g batches, ready for pressing.

D. HOT PRESSING

1. General

The weighed batch of the prepared powder (1230 g for material without, and 1280 g for material with, nuclear poison) was introduced into the loading glove box, Figure 9, where it was loaded into the graphite die, Figure 10. The cylindrical dies (8-3/4 in. OD, 5-1/16 in. ID, 5-5/8 in. high) consisted of an outer sleeve, matching plungers, and 1/4-in. thick disposable plates which bore directly on the powder. The dies were made of ATJ grade graphite in Atomics International's graphite shop.

Each of the four hot presses used, one of which is illustrated in Figure 11, consisted of a single 6 in. hydraulic ram, capable of a 4000-lb force, and operated by a gas ballast system which limited the use of the mechanical pumps for moving the fluid. The hydraulic system and the power transformers and control panels (designed to deliver approximately 5-v, 4000-amp maximum current to the heaters) were built to Atomics International specifications.

Figure 11. Hot Press

7513-1867A

Figure 12. Cooling Rack

7513-1838

Removable heater barrels were used to increase the production rate. These consisted of an 8-in. ID carbon resistance heater and a carbon packing inside a 20-in. diameter Transite case. End clamps, intercooling, argon-purging, sight-tube (for optical pyrometry), and quick-connect provisions completed the assembly.

The loaded die was secured inside a furnace barrel, which was next placed in a hot press. An initial pressure of 100 psi was applied to the powder; while the temperature was raised, over a 3 hr period, to the maturing temperature. Over a 10-min period, the pressure was then increased, in equal increments, to the specified pressure; and both the temperature and pressure were maintained for 2 hr. After a 16-hr cooling period in place, the furnace and its contents were removed to a cooling rack (Figure 12) for 15 hr, and then unloaded in the loading glove box, to complete a 36-hr overall pressing cycle.

2. Plates Without Nuclear Poison

Seventy 99 wt % BeO - 1 wt % MgO plates, 5 in. in diameter and approximately 1-1/8 in. high, were hot pressed. The MgO acted as a mineralizer or sinter-aid. This material was hot pressed at 1500 psi and 1600°C for 2 hr, to achieve the specified density of over 98% of theoretical.

3. Plates With Nuclear Poison

Twenty-one 91.15 wt % BeO - 8.85 wt % Sm₂O₃ plates, 5 in. in diameter and approximately 1-1/8 in. high, were hot pressed at 2,700 psi and 1450°C for 2 hr.

To establish these optimum parameters, hot pressing experiments were conducted at 2,000 psi and 1600°C with material containing 90.15 wt % BeO - 8.85 wt % Sm₂O₃ and 1 wt % MgO as additive to promote sintering, and at 2700 psi and 1400 to 1750°C without MgO addition. The time at temperature was held constant at 2 hr. The rate of pressure and temperature increase was always the same as was described previously. The results of these experiments are shown in Table V. At the time these experiments were conducted, AI had no access to the BeO-Sm₂O₃ phase diagram, published by S. G. Tresvyatskii,⁵ which shows a eutectic temperature at 1420 ± 10°C (Figure 13). These data are in good agreement with the results of AI experiments.

TABLE V
HOT PRESSING OF BeO WITH 8.85 wt % Sm₂O₃
(Time at Temperature 2 hr)

Additive	Pressure (psi)	Temperature (°C)	Apparent Density After Surface Grinding (g/cc)	Homogenization As Shown by Radiography	Remarks
1 wt % MgO	2000	1600	--	--	Formation of liquid phase. Bad sticking in die.
None	2700	1750	--	--	Formation of liquid phase. Bad sticking in die.
None	2700	1550	3.04	Segregation	Slight sticking in die.
None	2700	1500	3.08	Segregation	Slight sticking in die.
None	2700	1450	3.18	Satisfactory	No sticking in die.
None	2700	1400	3.04	Satisfactory	No sticking in die.

Figure 13. Phase Diagram for
BeO-Sm₂O₃ System Between
1300 and 2500°C
(From Reference 8)

E. SURFACE GRINDING

After removal from the dies, the plates were first ultrasonically cleaned (Figure 14). The plates were also ultrasonic cleaned, every time the machining was interrupted for inspection purposes. Because of the toxicity hazard, this and the other machinery were housed in Plexiglas, vented, and provided with an internal negative pressure. A preliminary inspection eliminated those plates with cracks and other visual imperfections. The plates were then surface ground on both sides on a Blanchard rotary surface grinder (Figure 15), using an 80-grit diamond metal bonded wheel. This grinding was sufficient to form smooth and parallel surfaces, but no attempt was made to equalize plate thicknesses.

F. INTERMEDIATE INSPECTION

The faced and cleaned blanks were first inspected again for visual imperfections, and then their densities were determined geometrically. Assuming computed theoretical densities of 3.02 g/cc for the 99 wt % BeO - 1 wt % MgO, and 3.17 g/cc for the 91.15 wt % BeO - 8.15 wt % Sm₂O₃ material, 90% of the BeO-MgO and 78% of the BeO-Sm₂O₃ hot pressed plates met the specified minimum density of 98% of theoretical. A detailed analysis is shown in Table VI.

TABLE VI
DENSITIES OF HOT PRESSED PLATES

Percent of Theoretical Density	Number of Plates	Percent of Total Measured
A. 99 wt % BeO - 1 wt % MgO		
Calculated Theoretical Density 3.02 g/cc		
> 98	63	90
97 - 98	5	7
96 - 97	1	1-1/2
95 - 96	1	1-1/2
B. 91.15 wt % BeO - 8.85 wt % Sm ₂ O ₃		
Calculated Theoretical Density 3.17 g/cc		
> 98	14	78
97 - 98	2	11
96 - 97	2	11

Figure 14. Ultrasonic Cleaning

7513-1855

Figure 15. Blanchard Surface Grinder

7513-18251

A radiographical inspection of the plates containing Sm_2O_3 followed, showing a satisfactory homogenization. Specimens from each plate have been identified and retained for future chemical analysis.

G. MACHINING

1. Number 1 Assembly

The surface ground plates were mounted on a Hardinge milling machine, Figure 16, sliced with a diamond wheel into bars 0.325 in. wide, and these bars were cut to 1.425 in. lengths, Figure 17. Approximately 30 blanks were obtained from each plate.

Shaping of these blanks to the required configuration was accomplished with the Hardinge milling machine in three operations, each with a differently shaped wheel, the curvatures blending into each other. Diamond wheels with plated bond, 80 grit, were used. They withstood this operation without any noticeable wear. The sequence of these three forming operations, and the wheels used, are shown in Figure 18.

2. Number 2 Assembly

For this assembly, the surface ground plates were core drilled on a Buffalo Drill Press, Figure 19, with diamond core drills of 0.260 in. ID. Approximately 40 cylindrical blanks were obtained this way from each plate (Figure 20).

These cylinders were then shaped on the "Hardinge" milling machine with a suitably formed diamond wheel, as shown in Figure 21.

H. DIMENSIONAL INSPECTION

After ultrasonic cleaning, the machined segments for the No. 2 assembly were dimensionally inspected on a Kodak Optical Comparator, Figure 22.

Only 15 No. 2 assembly segments out of a total of 810 inspected, or not quite 2%, were rejected for dimensional deficiency. The dimensional inspection of the No. 1 assembly segments was postponed, because the Hastelloy cladding for this assembly, as received from the supplier, deviated slightly from the specified configuration, due to stresses during extrusion of this complex shape. Therefore, it became necessary to machine the No. 1 segments, to fit them in the cladding.

Figure 16. Hardinge Milling Machine

7513-1825B

Figure 17. Forming of Blanks from Hot Pressed Plates

7570-1871N

a. First Operation

7570-1871E

b. Second Operation

7570-1871G

7570-1871I

c. Third Operation

Figure 18. Forming of Segments for Assembly No. 1

7513-1879

Figure 19. Buffalo Drill Press

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Figure 20. Core Drilling of Blanks

7570-1871K

Figure 21. Forming of Segments for Assembly No. 2

7570-1871F

7570-1875B

Figure 22. Optical Comparator

I. DENSITY DETERMINATION

The water immersion method was used to determine the densities of the machined segments. The densities were determined for segments from each of the 27 plates without, and the 16 plates with, nuclear poison that were selected for machining. The results are shown in Table VII.

J. ASSEMBLY

The machined and inspected ceramic pieces are to be assembled in No. 1 and No. 2 assemblies, consisting of 13 segments each. The height of the assemblies will then be measured, and the top segment of each assembly surface will be ground to bring the height of all assemblies down to the specified 14.225 in. The assemblies will be ultrasonically cleaned, remeasured, and clad with Hastelloy N cladding. The cladding pieces, and the stacked inserts to be assembled, are shown in Figure 23. The cladding operation is not reported here.

TABLE VII
DENSITIES OF SEGMENTS

99 wt % BeO - 1 wt % MgO (Calculated theoretical density 3.02 g/cc)		91.15 wt % BeO - 8.85 wt % Sm ₂ O ₃ (Calculated theoretical density 3.17 g/cc)	
Parent Plate No.	% of T. D.	Parent Plate No.	% of T. D.
BO 50369	100.	BO 22593	98.
50532	99.3	22578	99.4
65088	100.	22580	97.5
50362	98.1	22596	99.6
50384	100.	22592	100.
50358	100.	22573	100.
50363	99.9	22595	100.
50355	96.8	22575	100.
66596	100.	22588	100.
50371	99.8	22589	100.
65086	99.6	22577	96.8
66595	97.7	22582	98.5
50381	100.	22585	97.3
65083	99.8	22590	96.8
50588	100.	22581	99.6
50329	95.3	22574	99.7
50357	100.		
50361	99.3		
65098	100.		
65094	98.8		
50374	100.		
66594	99.3		
65078	98.7		
50366	100.		
66597	99.3		
50353	99.3		
50367	98.3		

7570-1871L

a. Assembly No. 1

7570-1871J

b. Assembly No. 2

Figure 23. Cladding and Inserts

III. EVALUATION

As a result of previous experience with hot pressing dense (99/1) BeO/MgO bodies, no difficulties were encountered in this part of the fabrication program. The preparation of homogeneous BeO-Sm₂O₃ presented difficulties, because of hydrolysis of Sm₂O₃, which restricted the mixing procedure to dry blending. A satisfactory blending can be achieved by using "Twin Shell" blenders with intensifier bars to break up agglomerations. In sintering BeO-Sm₂O₃ compositions, Sm₂O₃ acts as a sintering promoter, facilitating densification at lower temperatures. Care must be taken not to go much over the eutectic temperature (about 1420°C), as the formation of appreciable amounts of liquid phase causes dehomogenization.

Although the use of diamond wheels for forming dense BeO bodies is expensive, well manufactured wheels of correct grit size, properly applied, have long life, and allow good machining speed.

The experience gained in this fabrication effort reaffirmed the opinion that, for the fabrication of a limited number of small high-density BeO shapes of complex configuration and close tolerances, hot pressing of blanks and subsequent machining to shape is the most straightforward and economical method.

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