Use Advances in Beryllium Optical Technology Utilizing Spherical Powder, SPIE Astronomy Conference, March, 1998

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ABSTRACT

Beryllium has been used as an optical and structural material for astronomical/IR telescope applications for the past 20 years. Some of the most recent applications have been for the VLT (Very Large Telescope) M2 secondary mirror, SIRTF (Space Infrared Telescope Facility), plus many military space based IR sensors.

The traditional forms and optical grades of the material, I-70H and I-220H, are well characterized from a mechanical and thermal standpoint over a wide range of temperatures. Beryllium's limiting factors for astronomical and/or IR telescopes has been traditionally twofold: cryogenic stability and perceived higher cost than some of the other material options, such as glass, silicon carbide, and some composites.

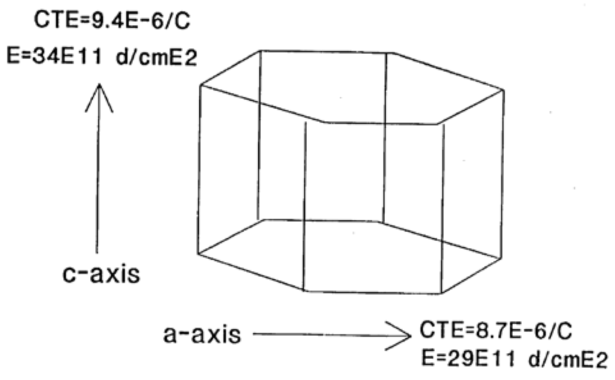
To address those two factors, Materion Corporation Beryllium & Composites group has developed a new optical grade of Beryllium produced by gas atomization (spherical powder) called O-30. This paper will detail the development of this grade of Beryllium, with emphasis on the cryogenic properties of the material from a thermal and mechanical view. It will also report on the results of the optical polishing/thermal cycling work done under government sponsored contracts. Finally the paper will describe the process of producing Near Net Shape parts utilizing O-30 spherical powder in order to reduce manufacturing cost and schedule.

Keywords: Beryllium, cryogenic stability, spherical powder, near net shape, O30 optical grade, isotropy

INTRODUCTION

In the past, Beryllium powders that were utilized for astronomical or IR telescopes such as VLT or SIRTF have been produced by conventional mechanical comminution processes, like impact or attrition grinding. The resultant powder reflects the crystallographic structure and deformation characteristic of Beryllium's close packed hexagonal lattice structure (Figure 1).

Figure 1. The Beryllium Close Packed Hexagonal Crystal



In the mechanical comminution powder process, fracture of the Beryllium chip occurs along the {0002} basal plane. This tends to produce flat, plate like particles, with faces corresponding to the basal planes. During handling and loading of these powders during the powder consolidation processes, Vacuum Hot Pressing(VHP) or Hot Isostatic Pressing(HIP), these powder particles tend to align and produce a non-random orientation in the consolidated powder. In critical components such as precision optics and alignment systems, this preferred orientation of the grains, could possibly lead to some anisotropy in both the mechanical and thermal properties of the material, which could detrimentally affect the performance of the instrument. The objective of developing a way to produce spherical Beryllium powder was to provide an isotropic material with predictable mechanical and thermal performance over a wide range of operating temperatures.

PROCESS DEVELOPMENT

In order to produce a truly random, spherical Beryllium powder particle, Materion Corporation Beryllium & Composites. in the late 1980's and early 1990's embarked upon a program to develop inert gas atomization of Beryllium and

Aluminum/Beryllium (AlBeMet). This process is mechanically simple and has been used to produce large quantities of other reactive metals like Magnesium and Aluminum. Under a Department of Energy contract, we developed inert gas atomization(Figure 2) of Beryllium, utilizing a small 3 kgs(6 Lb) melt furnace, with a helium atomization fluid under pressure. The process includes vacuum melting of solid beryllium and then pouring this liquid through a small orifice. This molten stream is impinged with a high velocity gas stream, breaking the molten stream into a fine droplet mist, which then by cooling turns into fine sphere's of high purity beryllium. The resultant material is then mechanically screened to remove the oversized material and provide a uniform powder product. This small unit produced powder particles that ranged from -325 to -100 mesh (44-110 microns). As the atomization gas pressure increased and the liquid metal diameter decreased, the resultant powder particle size distribution tended to decrease. Since this initial work, we have designed and built a production sized gas atomizer, with a capability of producing up to 500 kgs(1100 lbs) in a single week of atomization runs. The typical powder particle distribution of this production unit, is 99% less than 74 microns, -200/ + 325 mesh.

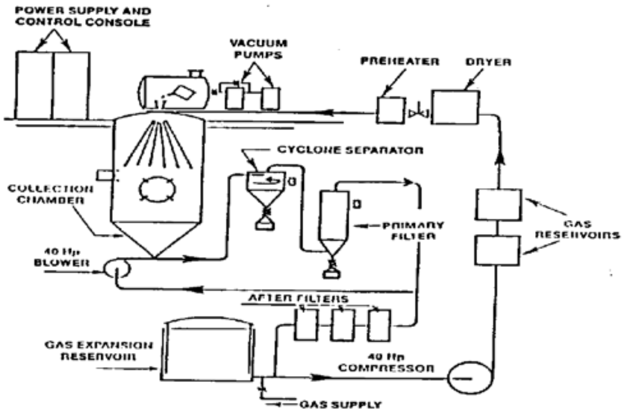


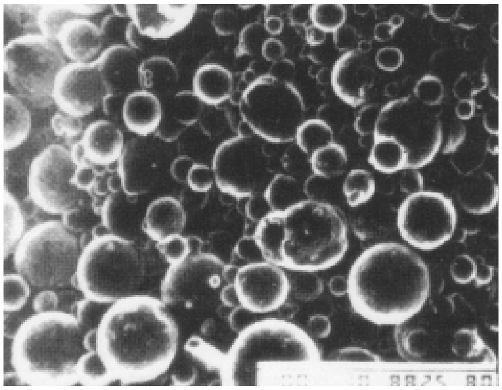
Figure 2. Schematic Gas Atomization Process

MATERIAL CHARACTERIZATION

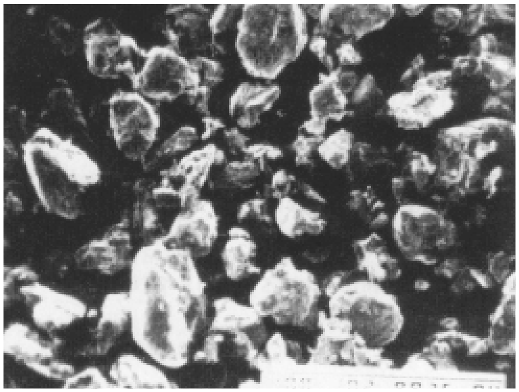
The as-atomized powder has been characterized in terms of its tap density, flow rate, chemistry, while the consolidated material by HIP has been characterized by its mechanical and thermal properties, thermal stability, and cryogenic optical properties.

POWDER CHARACTERIZATION

The powder produced by gas atomization was spherical as shown in Figure 3. The difference in particle shape between atomized and mechanically comminuted powders is striking as shown in Figure 3. The microstructure of the powder was polycrystalline. We also subjected the powder to a typical HIP consolidation temperature, 1000°C (1832°F), to determine if there would be any significant grain growth, there was no grain growth. The tap density of the -200 mesh spherical powder, O-30, was measured, 64% of theoretical, and was significantly improved over the 53% average of mechanically ground powders like I-70 and I-220. This along with the high flow rate of the powder, 50g/213 seconds, is significant in terms of loading HIP cans to thin cross sections/complex shapes, and too better predict final shape after consolidation by HIP. The mechanically ground powders, like I70, have virtually no flow rate, and therefore are less able to be used for producing Net Shape components. This early work has allowed Materion Beryllium & Composites and Matsys, under a DARPA contract[ref .1], to model the deformation process of HIPing spherical beryllium powders. Providing us with a process to predict the final part dimensions utilizing a Near Net Shape(NNS) process called Rapid Net Shape Forming(RNSF) being developed under the contract.



Atomized Powder



Impact Ground

POWDER CONSOLIDATION

During the early phase of spherical powder development, the powder was consolidated by both VHP and HIP to determine which consolidation process was best suited for producing fully dense products. While the VHP process was successful in producing a dense product, typical densities of 99.5% of theoretical, it was determined that the best overall consolidation process was HIP. This was due to the fact that HIP produces theoretical densities typically 100%, has better isotropy of properties, both mechanical and thermal, less than 1% variability in the three orthogonal directions of the material, due to isotropic pressure during consolidation. It also lends itself to Net and Near Net Shape technologies like RNSF, HIP bonding, and HIP with leachable mandrels.

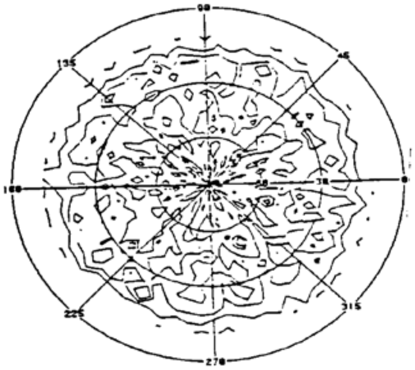


Figure 10. X-Ray Pole Figure for [0002] poles, VHP'd Spherical Powder. Note the Lack of Preferred Orientation

HIPing is a process of consolidating beryllium powders by the use of a high gas pressure, typically 15Ksi, in combination with a temperature that is lower than VHP, typically 850-1000°C. The HIP container is a metal can filled with beryllium powder, subjected to a high temperature degassing process to remove adsorbed gases, hermetically sealed, and then subjected to the HIP process. After the HIPing process the metal can is removed by acid etching and the part is then subjected to a stress relief cycle, 835°C(1450°F) to remove any thermal stresses and to measure for Thermal Induced Porosity(TIP). The part then is tested for mechanical and thermal properties.

MECHANICAL /THERMAL PROPERTIES

The isotropy of the HIP'd material was evaluated using X-Ray Pole figures by Lambda Research. The {0002} plane pole figure is shown in Figure 5. As one can see there is no preferred orientation of the grain structure and the grains are completely random, indicating a high degree of isotropy. Another way to measure isotropy is to test the material both mechanically and thermally.

The mechanical properties as shown in Table 1 indicates that the spherical powder grade, O-30, after HIPing has properties that meet or exceed the properties of the mechanically produced powders, like I70H. The tensile testing was performed in accordance with ASTM E8, at room temperature, 23°C (73°F). The isotropy of the mechanical properties is evident in both grades of beryllium, with variability on the order of less than 1% in any direction.

Table 1. Typical Mechanical Properties of Spherical Powder versus I-70H Grade

Grade	Ultimate Tensile Ksi	Yield Strength Ksi	Elongation %	CTE µin./in/C 0-65°C	Grain Size (µ)	Micro-Yield Strength Ksi
I70H - Longitudinal(L)	65	44	6	11.4	7	5
Transverse(T)	64	43	5	11.3	7	5
SPHERICAL 0-30 - L	59	43.5	3.0	11.38	7	4.5-5.2
Transverse(T)	58.5	42.8	2.9	11.38	7	4.5-5.2

Spherical powder grade 0-30 has also been tested for notched tensile strength (K_t=3) properties from room temperature (RT) to elevated temperature, >300°C, as part of a material characterization program $^{\text{Ref.2}}$. The data shows a slight rise in strength up to about 275°C, then a decrease with increasing temperature.

This data indicates that spherical powder is not notch sensitive, at a notch strength ratio of K_t=3, for all temperatures above 23°C (Figure 6). Also the fracture toughness of the material was measured using E399 (linear-elastic) K_{Ic} procedures up to temperatures of 250°C. The K_{Ic} values at room temperature are typical for beryllium, 10Ksi□in., increasing to 15 Ksi□in at 250°C.

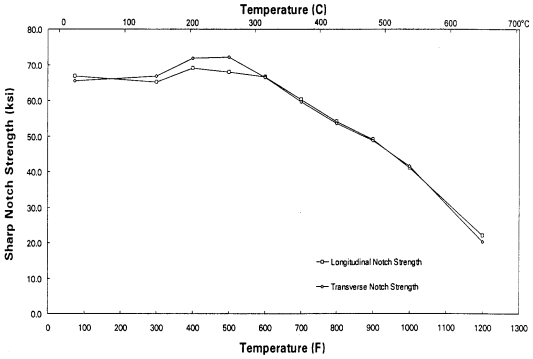


Figure 5.3.2.A Sharp Notch Strength ($K_t = 3$) of HIP'd GA Be for Longitudinal and Transverse Specimen Orientations

Spherical powder, grade O-30, was also tested for isotropy in its thermal properties, thermal conductivity and CTE, with CTE being the most sensitive to preferred crystallographic orientation. As the data in Table 2 indicates there was no difference in the CTE values over the temperature range of 0-65°C in any of the three orthogonal directions, demonstrating the excellent isotropy possible with HIP'd spherical beryllium powder. The thermal conductivity of spherical powder and another grade of beryllium similar to I70, were also tested from RT to 600°C. The results are consistent with other grades of beryllium already tested, RT 200 W/m-k, with decreasing thermal conductivity with increasing temperature, 124W/m-k at 316°C. The materials cryogenic thermal conductivity and CTE have not been tested at this time. One could make the assumption it will act like other low oxide beryllium materials at these temperatures, increasing thermal conductivity with decreasing temperature, and a CTE of 0 at 80°K. These properties will be tested under a contract supporting the NGST program. The last thermal property tested for was thermal shock. The thermal shock testing consisted of subjecting the specimens to heating rates from RT up to 700°C at 1.6°C/second(96C/hour) and then cooling rates from 700°C down to 250°C at 230°C/hour. There were no visual or dye penetrants inspected cracks observed in any of the 6 specimens after 32 cycles of both the heating and cooling cycles.

Table 2. Thermal Properties of Spherical Powder, Grade O-30

Test	25°C(77°F)	-166°C(-267°F)	148°C(298°F)
Thermal Conductivity W/m-k	204	365	161
CTE ppm/°C 65-5°C(149-41°F)	11.38 (L) 11.38(T)		
Electrical Resistivity (μ ohm-cm)	4.2		7.9
Thermal Shock Rt to 700°C@ 96°C/Hr 700°C to Rt @230°C/Hr	32 cycles no visual or dye penetrants inspected cracks observed		

CRYOGENIC TESTING

In the early and mid1990's Materion Beryllium & Composites, Tinsley Laboratories, and Hughes Aircraft worked under a contract from BMDO and Rome Labs, Beryllium Surveillance Mirror, to demonstrate the ability of using spherical beryllium powder in manufacturing high performance optics [Ref 3]. Also in the late 1980's work on optically characterizing spherical powder was done under a WPAFB contract with optical characterization done by Tinsley, Eastman Kodak, and Speedring Systems [Ref 4]. Basic material properties, powder processing methods, HIP parameters, bare-beryllium polishing methods were investigated, and a number of test mirrors were produced, up to 35cm lightweighted rectangles, for cryogenic figure and scatter testing down to 30K. This section of the paper will report on those results.

HIP EXPERIMENTS

Materion Beryllium & Composites determined through a series of smaller specimens, 76mm diameter flats polished by Tinsley in a block to a low scatter finish, 30-40Å, what was the optimum material process parameters for producing low scatter cryogenic beryllium optics. These tests / experiments determined that process to be: produce spherical powder with a particle size ranging from 38-74 microns, clean the powder, HIP at 830-850□C with 15Ksi of isostatic gas pressure, anneal the substrate after the HIP process at 835 □C for a minimum of 1 hour per inch of cross section and cool at a rate not to exceed 50 □C per hour. To verify these parameters, the 76mm test specimens were interferometrically evaluated. Interferograms were taken with a Zygo MK III phase measuring interferometer using a 4" diameter reference flat. The surface finish of 30-40 Å was performed to minimize scatter, the flatness of the surfaces were secondary. There were 6 specimens tested: L2,L3,L4, H2,H3,H4. Table 3 indicates the Post Polish P-V and RMS surface figure at 0.6328 microns, as polished, after annealing for 5 thermal cycles, and after cryo figure testing from 400K to 88K and back to 300K.

Powder Size	Part Number	Initial P-V	Initial RMS	Post Thermal Cycle P-V	Post Thermal Cycle RMS	Post Cold Test P-V	Post Cold Test RMS
38 microns	L-4	1.280 cc	0.287	1.683 cc	0.405	1.841 cc	0.402
38 microns	H-4	2.213 cc	0.545	4.138 cc	1.090	3.780 cc	1.024
<74 microns	463-A2	0.1543 flat	0.0233			0.1172	0.0153
<149/>74	466-A2	0.1680 flat	0.0257			0.1632	0.0163

Measurement wavelength = 0.6328 cc = concave surface

Table 3. Cryogenic Figure Testing Results Spherical Powder Coupons

The figure changes were the largest, as expected, going from initial testing after optical processing to post-thermal cycles. This indicates that there were residual stresses remaining in the blanks after machining, grinding and polishing. The figure changes after the 5 thermal cycles shows small changes, indicating that the thermal cycling removed a significant portion of the processing stresses, but additional thermal cycles were needed to reach a true equilibrium in the blanks.

SCATTER TESTING COUPONS

Scatter testing at □= .6328, 3.39 and 10.6µm was performed immediately after the polishing of the discs. All testing was performed at 5 degree angle of incidence. The samples were measured in 2 places, spot 1 and 0.75" to the left of center on the 76mm (3") diameter coupons (Table 4). In all testing at 0.6328,it was observed that the cleaned powder material samples, regardless of grain size had a factor of two(2) improvement in scatter over the uncleaned powder lots. This does not appear to be the case at the higher wave lengths. Testing done by Kodak at 10.63µm during the WPAFB contract indicated that the best scatter results at 3 degrees from specular was 2.7 x 10^{-4} and 1.2 x 10^{-4} at 5 degrees from specular (see Figure 7).

Scatter Results Spherical Powder

Part Identification/Grain Size µm	3° From Specular @ 10.6µm	5° From Specular @ 10.6µm
HIP 463-A2 / 7µm	2.7 x 10^{-4}	1.2 x 10^{-4}
HIP 518-A2 / 9µm	2.9 x 10^{-3}	9.6 x 10^{-4}
C38L-1 / 7µm	6 x 10^{-4}	4 x 10^{-4}
C38H-1 / 9 µm	1 x 10^{-3}	4 x 10^{-4}

Table 4 - Scatter @ 10.6 µm

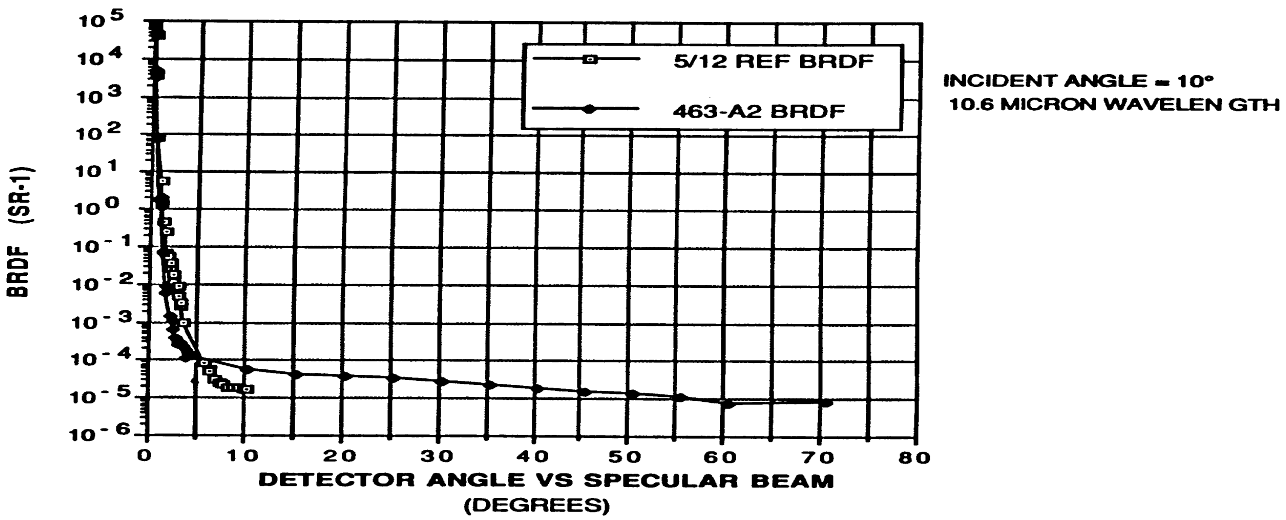


Figure 7 - Kodak Scatter Testing

All of these samples were then subjected to a post annealing process, 835°C, to simulate the normal annealing processing in fabricating stable Beryllium optics. The optimum powder size, 38 microns, showed very little difference in pre and post annealing scatter at any wavelength. Some of the other specimens, in particular the 74 micron size, cleaned and uncleaned, had measurable differences in scatter after the annealing process. The spectral reflectance of the spherical powder averaged 98.4% at 10.6µm using a Perkin Elmer Model 983G infrared spectrophotometer at an angle of incidence of 15 degrees.

The results of the HIP experiments, altering particles size, cleaning powders, HIPing at lower temperatures, indicates that improved performance of the finished mirror surfaces, scatter and surface finish, can be achieved through the use of aggressively cleaned 38 micron powders.

ANNEX MIRROR FABRICATION AND TESTING

A number of small open back lightweighted, 22Kgs/sq.meter areal densities, 130mm x 146mm rectangle (Annex 4 mirror) and 30.5cm x 35.2cm(Annex 1 mirror) were produced to evaluate Near Net Shape HIPing using a leachable mandrel technology, as well as cryogenic testing of figure and scatter on larger size blanks using the 38 micron powder process defined in the test coupons reported above. The goal of the mirror design was to achieve: areal density of < 22kg/sq.M, first mode frequency of greater than 100Hz, maximum deflection between the ribs of less than 0.075 microns, with a load of 20.7kpa (3psi), a mirror operating temperature of 40K, figure error of 0.3 microns rms maximum, and scatter at 10.6 microns less than 0.0001 per steradian at 2 degrees from specular.

RESULTS - ANNEX 4 MIRRORS

The blank fabrication for the Annex 4 mirror was done by loading cleaned 38 micron powder, as previously selected for its improved cryogenic scatter and surface finish, into a HIP can, with a leachable Monel 400 mandrel. These were then HIP'd at the same process parameters as the 76mm diameter coupons reported in section 4.1. All of the material test coupons for mechanical properties, chemistry, grain size were integral to the mirror blank, and therefore represented the process.

The final mirrors were pitch polished by Tinsley Laboratories using a full lap and diamond slurry abrasive process developed by Tinsley to produce low scatter surfaces on bare beryllium. The final figure of the Annex 4 mirrors using a phase measuring interferometer were, 0.31 □ P-V, 0.055□ rms at 0.6328 microns. The mirror was not polished to control the figure out to the edge, only to produce a low scatter surface. The surface profile was measured using a Chapman profilometer, with results of 8 angstroms.

The Annex 4 mirror was cryogenic holographic tested at Hughes Space Systems, using their 1.5M diameter holographic chamber (Figure 8). The holograms were taken every 25 degree Kelvin from room temperature, 298K, down to 75K. The final hologram was taken at 61K. The test was run at 0.514 microns wavelength. The mirror figure changed during the test with no signs of quilting or print through. The figure change occurred right after cool down, 273K, and stayed within those bounds, 0.25-0.5 □ P-V, all the way down to 61K. The mirror figure returned to its starting values upon return to 298K, with no signs of hysteresis or permanent figure change.

RESULTS ANNEX 1 MIRROR

The mirror substrate is a rectangular off-axis segment of a rotationally symmetric aspheric mirror. The finished mirror is 30.5 x 35.2 cm with rounded corners. The mirror blank was a fully machined substrate, followed by optical polishing, and was measured for thermal distortion at cryogenic temperatures using holography. The polished mirror blank was tested at 300K and had a P-V measurement of 0.17 waves and a RMS of 0.023 waves at 0.6328 microns. It was thermally cycled 5 times from 300K up to 423K and back to 300K, and down to 77K and back to ambient temperature. After these thermal cycles the mirror figure error was 0.3 waves P-V and 0.028 waves RMS, within the specification requirements. This was a difference of 0.24 waves P-V and 0.033 waves RMS from the initial figure. A final polish was performed which reduced the final figure to 0.22 P-V and 0.033 RMS prior to the final cryogenic distortion test.

The mirror was installed in the holographic chamber and tested from 300K down to 30K and back up to 300K. At 200K the mirror had a 5K thermal gradient across the part due to the thermal straps and the chamber characteristics. This resulted in a figure error of 0.88 waves P-V and 0.177 waves RMS (0.514= □). At 300K the figure was 0.132 P-V and 0.025 RMS, at 100K it was 0.237 P-V and 0.052 RMS, at 30K it was 0.274 P-V and 0.052 RMS, indicating that after 100K the beryllium's CTE was zero and stayed that way down to 30K. During cool down the mirrors figure changes slowly with no signs of print through or quilting. Upon heating back up to 300K the mirror's figure returned to its initial values, 0.146 P-V and 0.028 RMS, indicating no hysteresis.

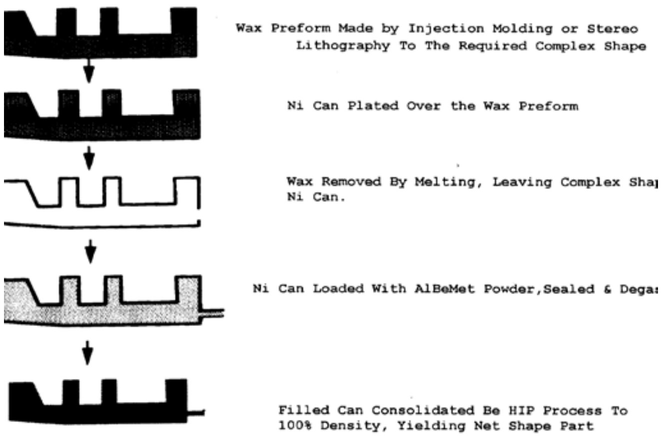
NEAR/NET SHAPE TECHNOLOGY

During the 1980's considerable work was performed in developing a Net Shape technology for Beryllium, utilizing leachable mandrel technology. This work was performed by numerous companies, Materion Beryllium & Composites, Inc., Perkin Elmer/Hughes Danbury Optical Systems, and OCA Applied Optics [Ref. 5&6]. The main mandrel material in all of these processes was copper tooling machined to the final dimensions of the lightweight cell structure. The other mandrel material that was utilized was Monel used during the development demonstration phase of the Beryllium Surveillance Mirror contract [Ref. 3]. This process, HIP with leachable mandrels, was demonstrated to be capable of producing up to 1 meter diameter with either closed or open back structures, and areal densities of < 20kg/sq meter. The main issues with this approach has been the time and cost to produce precision machined mandrels that can only be used once, predicting dimensional shrinkage of the blank during HIP consolidation, and potential thermal mismatches of the mandrel to the beryllium during the cool down cycle of the HIP process, potentially causing cracking in the beryllium.

To address these issues, cost, schedule, compatibility of mandrels to beryllium during the HIP process, Materion Beryllium & Composites and MATSYS are developing a net shape process called Rapid Net Shape Forming(RNSF) under a DARPA funded program [Ref. 7]. This process utilizes an " investment casting like" process(Figure 9), where in this case the injection molded wax form is a conductive wax in order to allow the plating of a metal on to the wax, thereby producing a net shape HIP can. This can is then loaded with spherical beryllium powder, degassed, sealed and HIP'd as we would conventional HIP processing of beryllium.

One of the key components of this technology is the ability to model and predict the compaction forces of the HIP process on the net shape metal can loaded with spherical beryllium powder. MATSYS has developed a modeling and simulation program to predict the powder consolidation process and its affect on the shape of the final part after the consolidation by HIP.

Figure 9. Shell Mold NNS HIP Process



This model takes into account the following variables: temperature, pressure/displacement, heating and pressurization rate. Under the DARPA contract the modeling of the spherical beryllium powder consolidation process has been completed. During the verification phase of the modeling we utilized on-line sensors to measure the dimensional change during compaction of the powder and compared that to the model prediction. The final consolidated part dimensions on these small pieces, 25mm O.D. x 12.5mm I.D. x 125mm long tubes, S-200F type powder, were within the dimensions as predicted by the model, ± 0.0004mm. The material properties of these demonstration parts met or exceeded the properties of HIP'd S-200FH material: Density 1.85 g/cc, UTS 55 Ksi (380Mpa), Y.S. 38 Ksi (260Mpa), and elongation of 3%.

The other key component of this technology is the utilization of spherical beryllium powder. Spherical beryllium powder has a higher packing density than traditional impact ground beryllium powders, 64% of theoretical versus 53% for impact ground. Plus it has a higher flow rate, 50grams/213 seconds versus no measurable flow rate, and isotropy of properties after consolidation by HIP. This allows, along with the modeling data, the ability to design thin walled, 0.050 in (1.5mm) HIP cans that can be filled without significant variability of the wall thickness over large aspect ratios, 6-8 to 1. The flow characteristics of the powder also allow the design of complex shapes utilizing this technology. This has been demonstrated on a hollow turbine blade application, utilizing Titanium powders with the following results:

- wall thickness tolerances typically ± 0.015 inches (0.3mm) for walls as thick as 0.250 in(6.35mm)
- true position of 0.001 in. (0.0254mm) - center to center distance between two holes on each end of part
- internal radii of 0.060 in. (1. 5 mm)
- twist ± 1 degree over entire length of part - 15.0 in (381mm)

ADVANTAGES OF RNSF TECHNOLOGY

1. Mechanical properties meet or exceed standard HIP'd beryllium grades
2. Thermal properties are isotropic - CTE 13.8 ppm/°C, Thermal conductivity of 210 W/m-k.
3. Shape capability like investment casting, with better mechanical properties
4. Potential use of internal coring for actively cooled optics.
5. Low oxide material may provide a diamond turnable surface for precision snap together optical systems.
6. Reduced material and fabrication cost versus the traditional beryllium technology.

CONCLUSIONS

1. Spherical beryllium powder provides improved thermal and mechanical isotropy versus impact ground powders for optical substrates.
2. Spherical beryllium powder provides the lowest oxide beryllium material available to date which improves the polishability to produce low scatter optics.
3. HIP'd spherical beryllium powder has improved cryo figure stability versus the traditional beryllium powders, with no hysteresis.

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4. Spherical beryllium powder provides high flow rates and packing densities allowing the use of Net Shape technologies like RNSF.
5. The use of RNSF technology provides the potential for producing complex Net Shape beryllium mirror blanks thereby reducing the cost and schedule to produce beryllium substrates.

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3. Cliff Bugle of Dynamet

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Note:
Handling Aluminum-Beryllium Alloys in solid form poses no special health risk. Like many industrial materials, beryllium-containing materials may pose a health risk if recommended safe handling practices are not followed. Inhalation of airborne beryllium may cause a serious lung disorder in susceptible individuals.

The Occupational Safety and Health Administration (OSHA) has set mandatory limits on occupational respiratory exposures. Read and follow the guidance in the Material Safety Data Sheet (MSDS) before working with this material.

For additional information on safe handling practices or technical data on Aluminum Beryllium Alloys, contact Materion Beryllium & Composites.

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