Evaluation of hot isostatic pressed beryllium for low scatter cryogenic optics

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ABSTRACT

Coarse and fine variants and standard grades of I-70A beryllium powder manufactured by Brush Wellman were consolidated by vacuum hot pressing (VHP) and by direct hot isostatic pressing (HIP) techniques. Five cm diameter flat mirrors were fabricated from 7.5 cm diameter by 7.5 cm long billets. All billets had densities of essentially 100% after HIP’ing. The mirror blanks were polished bare and the resulting surface evaluated for surface roughness and surface scatter at 0.51, 3.39, and 10.6 μm. The general trend noted was that better optical properties were obtained with direct HIP’ed coarse powder (since designated as O-50 material). Surface roughnesses of 15 to 20 angstroms rms were obtained. The coarse powder was then used to fabricate a 0.5 meter diameter solid HIP’ed blank. This blank was polished as an F/1.5 sphere, scatter measurements taken, and the figure evaluated at cryogenic temperatures. Distortion of 0.19 waves rms at 0.63 μm was seen at 97 K, corresponding to a homogeneity of ~ 26 ppb/K rms.

1. INTRODUCTION

Beryllium is a favorite material of space sensor designers because of its high stiffness-to-weight ratio and excellent thermal properties at low temperatures. These properties led to its use for the scan mirrors in the Multi-Spectral Scanner and Thematic Mapper and for the telescope structure and/or mirrors in Infra-Red Astronomical Sensor (IRAS)2-4, Space Infra-Red Experiment (SIRE), Forward Acquisition Sensor (FAS), and many other sensors. In these systems either poor scattering characteristics were accepted with a bare polished surface or the mirrors were nickel plated to provide good surface finishes. Problems with material homogeneity were also encountered at low temperatures.

A cooperative investigation to develop a beryllium material with improved polishing characteristics and better homogeneity was begun in 1983. The approach taken in this study was designed to determine the material characteristics which could exert a significant influence on the optical properties of bare (uncoated) beryllium. Although it has been surmised that grain size, oxide content, and the presence of porosity can be important, a systematic study has not been conducted to quantify these effects.

Brush Wellman produced sets of samples consolidated by two different methods with three different powder particle sizes. These samples were polished by Micro-Robotics of Clearwater, Florida. Optical evaluation of surface roughness and bi-directional reflectance distribution function (BRDF) were performed by Hughes. Additional coarser grained material was examined later but the results simply confirmed this initial study.

After determining the best material, a 0.5 meter diameter mirror billet was fabricated by Brush Wellman using that material, machined into a blank by ABC Loral, polished by Tinsley Laboratories, and tested by Hughes.

Perkin-Elmer has been using HIP technology and I-70A beryllium powder to obtain improved optical properties from beryllium. Their results have shown improved homogeneity and surface finish over VHP material also.

2. SAMPLE PREPARATION AND TEST METHODS

2.1. Five centimeter samples

A batch of beryllium powder intended for processing to grade I-70A and prior to comminution to a final particle size was used as starting material. This material was screened, using standard Tylar screens, into a coarse, standard, and fine size fraction. The major difference in chemistries between the samples was in oxide content—the coarse having 0.22%, the standard having 0.67%, and the fine 0.91% BeO. Significant grain size differences due to powder particle size variations were also noted.
Two methods were used to consolidate the powder into billets. The first was vacuum hot pressing followed by hot isostatic pressing (VHP/ HIP). The second was direct hot isostatic pressing (HIP).

The VHP/ HIP samples were vacuum hot pressed using 7.5 cm diameter cylindrical graphite dies with graphite punches. The samples were pressed at a temperature of 1350 K and a pressure of 7 MPa for 3.5 hours. The resulting billets were HIP'ed without a capsule since the VHP porosity was isolated rather than interconnected.

The direct HIP samples used a mild steel capsule to contain the powder. The HIP capsules were 10 cm diameter by 10 cm long. They were HIP'ed at a temperature of 1100 K and a pressure of 102 MPa for 3.5 hours.

The resulting billets were approximately 7.5 cm diameter by 7.5 cm long. All of the HIP'ed billets had a density essentially 100% of theoretical, whereas the VHP/ HIP billets generally had a density approaching, but not equal to, 100% of the theoretical (Table I).

From each billet, 5 cm diameter by 0.6 cm thick blanks were machined. These blanks were then polished by the same person and in the same time frame.

2.2. Half meter mirror

The 50 cm mirror billet was fabricated using the coarse grained material by direct HIP. This material has since been designated as O-50 optical grade beryllium.

The HIP can have three vent tubes on one surface. They were located 120 degrees apart on a 30 cm diameter circle (Fig. 1). The surface with the vent tubes became the back of the mirror. The resulting billet was solid and 61 cm diameter by 7 cm thick. From this billet a 50 cm mirror blank was machined as well as metallurgical samples. The resulting mirror blank center was approximately 4.5 cm off-center from the billet center (Fig. 1).

<table>
<thead>
<tr>
<th>TABLE I. Density results for five centimeter sample billets.</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAMPLE TYPE</td>
</tr>
<tr>
<td>--------------</td>
</tr>
<tr>
<td>VHP/ HIP Billets</td>
</tr>
<tr>
<td>Coarse</td>
</tr>
<tr>
<td>Standard</td>
</tr>
<tr>
<td>Fine</td>
</tr>
<tr>
<td>HIP Billets</td>
</tr>
<tr>
<td>Coarse</td>
</tr>
<tr>
<td>Standard</td>
</tr>
<tr>
<td>Fine</td>
</tr>
</tbody>
</table>

Figure 1. HIP configuration showing vent tube geometry and mirror blank center offset. Note that clocking of vent tubes is not known relative to mirror.

The mirror was figured as an F/1.5 sphere with a 152 cm radius of curvature. The finished mirror was 50 cm diameter with an edge thickness of 6.8 cm. Polishing was done twice—once in May 1988 and then again in September 1988. This was done to improve the surface figure to provide more accuracy in the cryo testing.

2.3. Surface characterization

Direct measurements of surface roughness were done using a WYKO surface profilometer. These measurements were correlated with results of visible total integrated scatter (TIS) measurements.

BRDF was measured in the Hughes scatter facility from 2 degrees to 70 degrees. Test wavelengths of 0.5145, 0.6417, 3.39, and 10.6 μm were used. Integration of the BRDF curves was used to determine TIS.

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2.4. Figure evaluation

Optical surface figure was measured using a ZYGO interferometer with an F/1.5 diverger. The test configuration allowed the mirror to be supported on three teflon pads with the mirror looking up (Fig. 2). Copper braid thermal straps were bonded to the mirror using a 50:50 mixture Carter's rubber cement and copper powder assisted by strips of aluminum tape. Five thermocouples were attached to the back of the mirror. Cross hairs and fiducial marks were attached to the front of the mirror (Fig. 3) to allow unambiguous determination of the mirror orientation in the interferograms. Liquid nitrogen flowing into a reservoir underneath the mirror was used to cool the mirror. The mirror temperature was recorded automatically every 15 minutes.

Figure 2. Interferometer and cryo-chamber configuration used to test the 50-cm mirror.

Interferograms were taken approximately every 50 K from room temperature to 100 K. Four interferograms were taken at each temperature and averaged during data analysis. Cryo distortion was determined by subtracting the starting interferograms with the window in place because of significant higher order spherical aberration introduced by the window (0.59 waves r.m.s.). Warm to warm hysteresis was determined with the window removed to improve test accuracy. Interferogram analysis was performed using WISP with 36 Zernicke terms (spline fits could not be used because of large fringe slopes). Tilt and focus terms were subtracted. Only the central 95% of the mirror was used to prevent problems with the Zernicke polynomials at the edge.

Three cryo-tests were performed. The first, in May 1988, used a different test configuration than shown here. The mirror was supported on edge. The test configuration and significant distortion seen led to reconfiguration of the test as described above and the refiguring of the mirror to obtain better accuracy. The second and third cryo-tests were performed in September and October 1988.

3. EVALUATION AND TEST

3.1. Mechanical properties

3.1.1. Samples

As expected, métallographic examination revealed a strong dependency of grain size on powder particle size. The VHP/HIP samples had grain sizes of 16.9, 8.9, and 6.0 μm while the HIP had 6.9, 4.9, and 3.6 μm respectively for the coarse, standard, and fine powders (Fig. 4). The effects of processing history are also evident in that the VHP/HIP samples have ~2 times larger grains than HIP for the same starting powder. This is expected because of the higher consolidation temperatures used in the VHP process. No difference in microstructure, other than the grain size, was observed.

The tensile and precision elastic limit (PEL) properties (Table II) of these samples exhibited several significant trends. First, the dependence of yield strength upon grain size is clearly shown (Figure 5). The behavior of direct HIP and VHP/HIP are not noticeably different insofar as the relationship of grain size to strength. It appears that tensile properties are a function of grain size rather than of the path taken.
The PEL does exhibit a dependence upon consolidation technique. Direct HIP follows a different relationship than does VHP/HP (Fig. 6). The cause of this phenomenon has not been established. The PEL is a very sensitive measure of flow stress and the differences in dislocation configuration can have significant effects on this property. The critical resolved shear stress on the (0001) slip planes of oriented versus random materials can contribute to the effect noted.

3.1.2 Mirror

The grain size of the mirror blank (Table III) is considerably larger than the grain size in the HIP samples with the same coarse starting material. This is because the blank was HIP-ed at 1273 K as opposed to 1100 K used for the test specimens. This temperature difference was necessitated by the production requirements (i.e., furnace availability).

3.2 Surface roughness and scatter

3.2.1 Samples

The TIS ranged from 0.19 to 0.41% at 0.5145, from 0.03 to 0.062% at 3.39, and from 0.0088 to 0.083% at 10.6 μm. The BRDF at 10.6 μm of the coarse grained direct HIP sample is 5.5E-4 at 2 degrees (Fig. 7). The measurement of surface roughness at two locations gave 12 angstroms rms (Fig. 8) and 20 angstroms rms. Surface roughness calculated from the visible TIS is 26 angstroms rms.
TABLE II. Tensile properties of the VHP and VHP/HIP sample billets.

<table>
<thead>
<tr>
<th>SAMPLE TYPE</th>
<th>SPECIMEN ORIENT.</th>
<th>P.E.L (MPa)</th>
<th>0.2% Y.S. (MPa)</th>
<th>U.T.S. (MPa)</th>
<th>ELONG. (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>VHP/HIP</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coarse</td>
<td>Long.</td>
<td>0.31</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td>Trans.</td>
<td>0.29</td>
<td>3.07</td>
<td>6.04</td>
<td>3.9</td>
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<tr>
<td>Standard</td>
<td>Long.</td>
<td>0.28</td>
<td>4.48</td>
<td>7.58</td>
<td>3.2</td>
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<tr>
<td></td>
<td>Trans.</td>
<td>0.39</td>
<td>4.60</td>
<td>7.48</td>
<td>2.6</td>
</tr>
<tr>
<td>Fine</td>
<td>Long.</td>
<td>0.33</td>
<td>5.08</td>
<td>8.27</td>
<td>3.9</td>
</tr>
<tr>
<td></td>
<td>Trans.</td>
<td>0.38</td>
<td>5.11</td>
<td>8.29</td>
<td>3.3</td>
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<tr>
<td>Direct HIP</td>
<td>Coarse</td>
<td>0.46</td>
<td>--</td>
<td>--</td>
<td>--</td>
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<tr>
<td></td>
<td>Long.</td>
<td>0.50</td>
<td>5.04</td>
<td>7.56</td>
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<tr>
<td></td>
<td>Trans.</td>
<td>0.45</td>
<td>--</td>
<td>--</td>
<td>--</td>
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<tr>
<td></td>
<td>Long.</td>
<td>0.57</td>
<td>5.07</td>
<td>8.01</td>
<td>3.9</td>
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<tr>
<td>Standard</td>
<td>Long.</td>
<td>0.70</td>
<td>7.28</td>
<td>6.54</td>
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<tr>
<td></td>
<td>Trans.</td>
<td>0.60</td>
<td>7.10</td>
<td>9.23</td>
<td>0.9*</td>
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<tr>
<td>Fine</td>
<td>Long.</td>
<td>0.89</td>
<td>5.04</td>
<td>7.84</td>
<td>3.1</td>
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<tr>
<td></td>
<td>Trans.</td>
<td>1.13</td>
<td>10.66</td>
<td>--</td>
<td>--</td>
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<tr>
<td></td>
<td>Trans.</td>
<td>1.11</td>
<td>8.44</td>
<td>11.27</td>
<td>2.1</td>
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</table>

*Broke outside gauge marks.

TABLE III. The mechanical properties of the 0.5 meter mirror.

A. Tensile Properties.

<table>
<thead>
<tr>
<th>Orient.</th>
<th>P.E.L (MPa)</th>
<th>0.2% Y.S. (MPa)</th>
<th>U.T.S. (MPa)</th>
<th>Elong. (%)</th>
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</thead>
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<tr>
<td>Long.</td>
<td>0.21</td>
<td>4.01</td>
<td>5.91</td>
<td>2.9</td>
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<tr>
<td>Trans.</td>
<td>0.23</td>
<td>4.04</td>
<td>5.94</td>
<td>2.4</td>
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</table>

B. Coefficient of Thermal Expansion

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Long.</th>
<th>Trans. 1</th>
<th>Trans. 2</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>11.3</td>
<td>11.4</td>
<td>11.4</td>
</tr>
<tr>
<td>2</td>
<td>11.3</td>
<td>11.4</td>
<td>11.3</td>
</tr>
</tbody>
</table>

C. Grain Size = 16.9 micrometers.

Figure 5. Effects grain size on tensile properties.

Figure 6. Dependence of precision elastic limit on grain size and consolidation method.

Based on these samples, the following generalizations were made: 1) in terms of TIS, HIP is always better than VHP/HIP, and 2) in terms of surface roughness coarse grained is better than standard is better than fine and HIP is always better than or equal to VHP/HIP. These conclusions would be more reliable if the data base were larger (1 to 2 samples of each type were examined). Although, other samples (not shown here) polished by other optical facilities tend to support the basic conclusion — direct HIP coarse grain gives the best surface finish.
3.2.2. Mirror

The mirror was figured two times, once in May 1988 and then again in September 1988. More care was given to the surface finish in the May polish than in the September polish (Table IV). In September, effort was concentrated on obtaining good figure quickly to provide more accuracy to the second set of cryogenic tests.

The steep rise in the 10.6 μm BRDF at small angles indicates contamination of the measurement by the specular beam (Fig. 9). Ignoring the point at 2 degrees and extrapolating back gives ~1E-3 at 2 degrees and 2E-3 at 1 degree. Unfortunately the measurement was not repeated. Comparison with the best coarse direct HIP sample BRDF of 5.5E-4
at 2 degrees is good, considering the mirror is ten times larger in diameter. TIS data is even closer with the May mirror having a TIS of 1.1E-4 and the sample 9E-5 at 10.6 μm.

Calculation of the surface roughness from the visible data gives 31 angstroms for May and 42 angstroms for September. The coarse direct HIP sample gave 26 angstroms.

3.3. Mirror figure

The mirror figure at the start of the September tests was 0.04 wave rms (1/4 wave P-V). As the mirror was cooled, a "smiley face" distortion appeared (Fig. 10). At 100 K the change from room temperature was 0.18 wave rms, 1.1 waves P-V (Table V). The second cooldown gave similar results with 0.19 wave rms change at 97 K (Table V). In both tests a permanent distortion of 0.04 waves rms was observed after warming back to room temperature. This instability probably results from changing strain patterns due to the interaction of residual stress from fabrication with the cryo-cycle. Examination of the data from the May test shows the same "smiley face" distortion of approximately the same magnitude (large test errors made accurate comparison difficult).

Surface distortion data is plotted as a function of temperature to obtain a measure of CTE homogeneity (Fig. 11). Data from both the September and October tests for the 95% aperture peak to valley and rms and the "smiley face" peak to valley are plotted. The slope of the graph is a measure of CTE homogeneity. By dividing the slope by half the thickness of the material (assumes equal distortion on both sides of the mirror), an apparent CTE homogeneity of 1.1E-7/K-P-V is obtained from both the 95% aperture and the "smiley face." An rms CTE of 2.6E-9/K is obtained from the rms surface data.

One potential cause of the "smiley face" distortion relates to the HIP can configuration. As described above, the HIP can had three vent tubes equally spaced on a 15 cm radius about its center (Fig 1). The mirror axis was offset by ~4.5 cm from the HIP can center. Examination of the "smiley face" (Fig. 11) shows that the "nose" is displaced from the mirror center by ~4.1 cm. Further, the "nose" is separated from the "eyes" by ~16 cm and ~15 cm and from the "mouth" by ~14.5 cm. The angular separation of the eyes and mouth are 166, 115, and 80 degrees. Unfortunately, the exact orientation of the mirror within the HIP can was not recorded, but the similarities are striking.

The tubes act to reinforce the HIP can, causing lower pressures at these locations. This produces compression in a preferential direction and could result in the varying values of CTE. The effects seen at the edges of the mirror may be related to the HIP can's increased stiffness at the edges in a manner similar to that of the vent tubes.
4. CONCLUSIONS AND FUTURE WORK

These tests have yielded much valuable information concerning the characteristics of the HIP’d special grade of beryllium (O-50) and the constraints that must be imposed for HIP’ing optical grade materials. The following characteristics of O-50 were demonstrated:

1. HIP beryllium grade O-50 is suitable for use as optical components in infrared optical systems.

2. The tests reconfirm previous experience on the importance of proper fabrication methods to produce stable cryogenic optical components.

3. The scatter from the bare polished surface meets the requirements of infrared optical systems.

Work is continuing with the mirror. The next phase will lightweight the mirror by machining a triangular pocketed core into the back of the mirror. Polishing of the lightweighted mirror will concentrate on achieving good surface figure and low scatter simultaneously. The mirror will be retested for scatter and cryogenic distortion. It is expected that the distortion will be reduced since there will be a smaller volume of material with varying CTE.

Before lightweighting the mirror, eight plugs will be removed from the back of the mirror. CTE and X-ray diffraction pole figure analysis of the pieces will be measured and correlated with the cryo-distortion data.

5. ACKNOWLEDGMENTS

The authors wish to thank Fred Thompson of ABC Loral in Televast, Florida, for machining of the 0.5 meter blank; Bill Caithness and Frank Montone of Micro-Robotics in Sarasota, Florida, for preparing polished samples, and Dan Bajuk of Tinsley Laboratories in Richmond, California, for polishing the mirror—all done on internal funding within each company.
6. REFERENCES

1. E. W. Gossett and P. M. Winslow, NASA Tech Briefs, 121 (Fall 1983).


