LightPath® TECHNOLOGIES

Material Property Qualification for BD-2 Chalcogenide Glass

The batch production processes for chalcogenide glass raise concerns about the batch to batch variation in key physical and optical characteristics of chalcogenide glass. LightPath Technologies has extensively tested and qualified our glass sources to assure its customers that they will receive consistent quality of our BD-2 chalcogenide glass.

This white paper outlines the parameters and tests we conducted to verify the accuracy of the critical optical, thermal, and mechanical properties for our glass and verified them on a batch to batch basis.

1. COMPOSITION

The material samples were tested for their elemental make-up using proton-induced X-ray emission (PIXE) analysis. Disc samples with fine-ground surfaces were used for PIXE analysis. Various trace elements were found in these glasses but only the ratio between germanium (Ge), antimony (Sb), and selenium (Se) was studied in detail. The results for three boules of BD-2 are shown in Table 1. There is no significant difference in composition between the three boules of glass.

Table 1 – Comparison of the composition (in atom%) of samples from 3 BD-2 boules.

	Ge	**Sb**	**Se**
Sample 1	30.6	11.0	58.4
Sample 2	30.7	11.6	57.7
Sample 3	30.4	11.6	57.9

The composition results do vary from the theoretical composition of the glass (Ge28Sb12Se60). The element ratios are mixed before melting. During the manufacturing process, a small amount of selenium is lost due to its low melting point of approximately 221°C. The data is consistent from batch to batch and represents the final composition of the glass.

2. COEFFICIENT OF THERMAL EXPANSION

A sample of BD-2 glass was tested for the CTE and dilatometric softening point (T_d) using a dilatometer. The shape of the curve and the values of CTE and T_d are dependent on the heating rate, which was 3°C/min for this sample. The resulting curve is plotted in Figure 1.

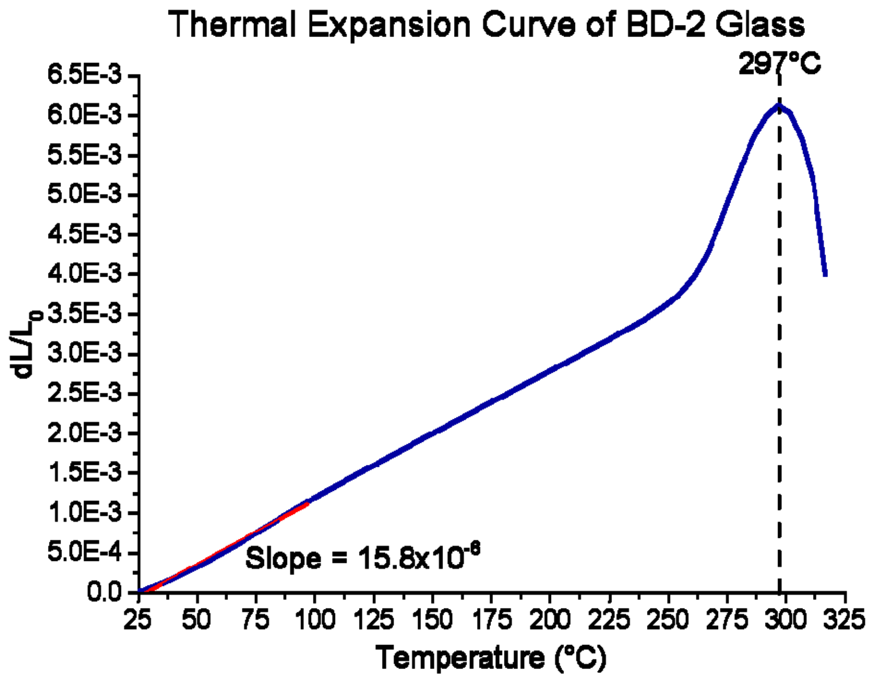

Figure 1 – Dilatometry curve of a BD-2 glass sample.

The CTE is found by calculating the slope of the curve in a particular temperature range and the softening point is the maximum of the curve. The properties determined for BD-2 are shown on the curve as well as listed below:

CTE (25-100°C): 15.8 x 10^{-6} /°C

T_d: 297°C

3. DENSITY

Archimedes' principle was used to determine the density of three BD-2 samples. The results are shown in Table 2. There is little variation between boules.

Table 2 – Comparison of density values for 3 BD-2 boules.

	Density (g/cm^3)
Sample 1	4.6870
Sample 2	4.6816
Sample 3	4.6849

4. HARDNESS

For hardness measurements, a Berkovich diamond indenter was used. Polished discs were used for the measurements. For each sample, 5 indentations were made and the average Vickers hardness

(H_V) was calculated. The Young's Modulus (E) of the sample can also be calculated from the data by finding the slope of the linear portion of the stress-strain curve. The average values for each property are shown below in Table 3.

Table 3 – Hardness and Young's Modulus of three BD-2 boules.

	Hardness (Vickers)	**Young's Modulus (GPa)**
Sample 1	193.7	24.02
Sample 2	214.4	24.57
Sample 3	215.9	23.93

Differences in surface roughness can have a small effect on the hardness measurements, so this could account for some of the variation seen. However, BD-2 has relatively low hardness values and lenses molded from this material must have a hard coating applied. For this reason, small variations in hardness are not expected to affect the PGM process. There is no significant variation in E between boules.

5. INFRARED TRANSMISSION

Transmission FTIR was performed on samples of approximately 5 mm thickness with parallel optically polished surfaces using an FTIR spectrometer. Measurements were made at room temperature under a nitrogen atmosphere and a background spectrum was collected before each sample spectrum.

Differences in surface quality between samples prevented the determination of the absolute percent transmission. The spectra were normalized for easier comparison between them. The results for three boules of BD-2 are shown in Figure 2 below.

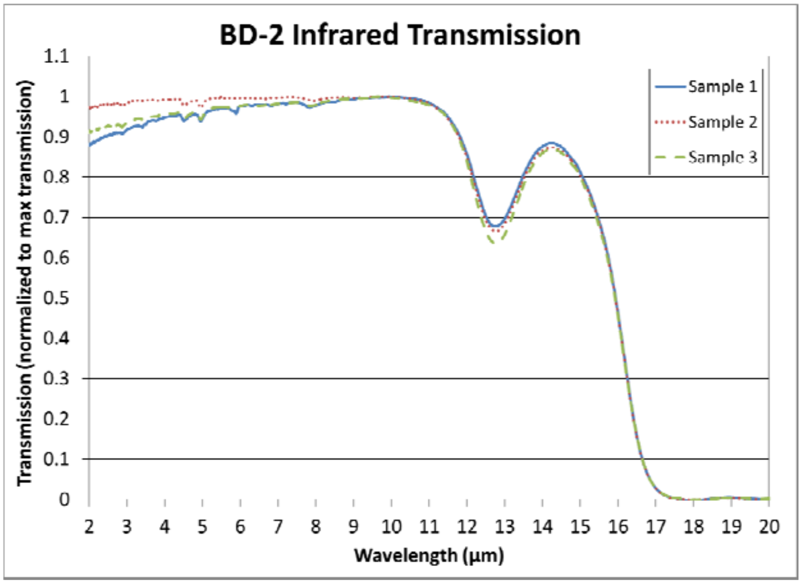

Figure 2 – Normalized infrared transmission of three BD-2 boules.

The absorption band located at approximately 12.5 µm is due to oxide impurities in the form of Ge-O bonds. All three boules have similar levels of oxide impurity based on the size of the absorption bands. This absorption band may vary between different glass suppliers due to their processing techniques, including whether or not they have an extra purification step in their manufacturing process. However, some oxygen will be introduced during the precision glass molding process so even a lens made from highly purified glass will have some absorption in this wavelength range. This only becomes an issue if the lens is used for a narrow band near 12.5 µm. For broad-band applications, such as thermal imaging using 8-14 µm, this small absorption does not impact performance. There is little to no effect on thermal imaging systems that use an 8-12 µm wavelength range.

6. REFRACTIVE INDEX

The refractive index of each glass boule was measured via a modified minimum deviation technique. Samples were in the form of a round diameter wedge. The index was measured at 20°C in air at 2, 4, 8, 10, and 12 µm. The results are shown in Figure 3.

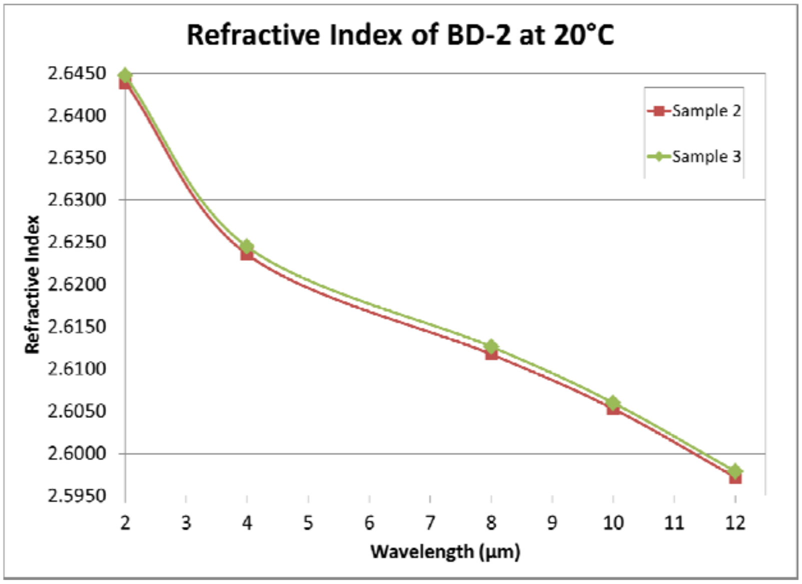

Figure 3 – Refractive index of two BD-2 boules

The refractive index has a tolerance of ±0.005 between boules. The two samples shown here have very similar index values, well within the tolerance range. Also, the refractive index changes after molding, making small differences beforehand insignificant.

7. CHANGE OF INDEX WITH RESPECT TO TEMPERATURE (dn/dT)

The change of index with respect to temperature is important in the design of lenses that may be used at different temperatures depending on the application. The index of a BD-2 prism sample was measured from -40°C to 80°C at 1.5, 2, 3, 5, 8, 10, 12, and 14 µm. Then the dn/dT was calculated and the results are shown in Figure 4.

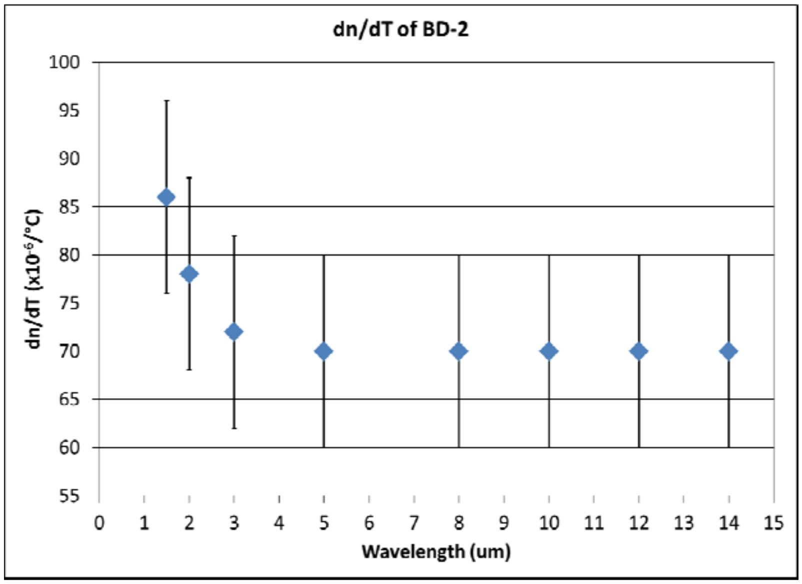

Figure 4 – Change in refractive index with temperature for BD-2 at various wavelengths.

Compared to crystalline germanium, BD-2 is clearly superior with regards to dn/dT. Crystalline germanium has a dn/dT that is on the order of 4 x 10^{-4}/°C near room temperature [1].

8. CONCLUSION

Based on the testing LightPath has completed and ongoing quality monitoring, our BD-2 glass specifications are indeed consistent and within all stated tolerances on a batch to batch basis. The material is well suited for high volume production without concern about the quality of the raw glass material.

References

[1] Frey, B.J., Leviton D.B., Madison, T.J., "Temperature-dependent refractive index of silicon and germanium," Proceedings of SPIE Vol. 6273 (SPIE, Orlando, FL 2006) 62732J.