Evaluation of sapphire at II-VI Optical Systems for HEL applications

M. Seitz*, N. Stoddard*, B. Glick*, J. C. Stover#
*II-VI Optical Systems
36570 Briggs Road., Murrieta, CA 92563
The Scatter Works, Inc.
2100 N. Wilmot Rd #202, Tucson, AZ 85712

ABSTRACT

There is an increasing industry need for high quality materials which can be used in high energy laser (HEL) applications. Currently, II-VI OS is growing large sapphire substrates for use in the defense industry on aerospace applications that operate in the visible and mid-wave infrared regions. II-VI OS is capable of producing a-plane and c-plane sapphire substrates of sufficient size for envisioned HEL applications, and in the following paper will present optical and material data on properties of interest. Specifically, data will be presented on BTDF and BRDF at 1.06µm in both s- and p-polarizations at angles of incidence (AOI) ranging from 0 to 60 degrees. These measurements are performed on a-plane and c-plane windows grown, fabricated and polished at II-VI OS Additionally, other critical properties evaluated include absorption at 1.06µm, as well as thermal expansion coefficient and Knoop hardness of c-plane sapphire.
Keywords: sapphire, edge fed growth, heat exchanger method, homogeneity, optical properties, material properties, high energy laser, HEL

1. INTRODUCTION

Over the last several years, II-VI Optical Systems (II-VI OS) has performed sapphire material characterization studies that are immediately relevant to many aerospace applications [1, 2, 3]. The properties that make sapphire an attractive option for these applications are also pertinent to HEL applications, such as high transmission in the VIS – MWIR wavelength regions, high optical uniformity, low thermal expansion, and extremely high durability and scratch resistance. This last property is correlated to its high Knoop hardness value. However, there are additional material properties that are specifically desirable for HEL applications. HEL applications require materials that demonstrate low surface scatter, low bulk scatter and low absorption [4].

The samples evaluated in the following paper were grown via the heat exchanger method [5]. The material tested was selected from routinely grown boules and is completely representative of II-VI OS sapphire material. Boules grown at II-VI OS are approximately 13.0” diameter and 10” tall. The boule can be oriented via x-ray diffraction techniques to yield parts in the a-plane, c-plane, r-plane or m-plane crystal orientation. A-plane and c-plane samples were evaluated for surface scatter via measurement of the bidirectional transmittance distribution function (BTDF) and the bidirectional reflectance distribution function (BRDF). Additionally, c-plane samples were measured for bulk absorption, coefficient of thermal expansion and Knoop hardness. It is important to emphasize that the samples evaluated in the following paper were taken from standard sapphire blanks used to make large windows at II-VI OS, and therefore the data is representative of a manufacturing environment. It is the goal of II-VI OS to provide high quality, large format windows to the HEL industry. Work is on-going to make continual progress to address desired key-attributes for this industry. Where applicable, measurements are compared to previously reported data on II-VI OS sapphire.

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* Further author information –
M. Seitz (correspondence): Email: Melissa.Seitz@ii-vi.com
N. Stoddard (correspondence): Email: Nathan.Stoddard@ii-vi.com
B. Glick (correspondence): Email: Bruce.Glick@ii-vi.com

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2. TEST METHODOLOGIES

2.1 BTDF and BRDF

Scatter measurements (BTDF and BRDF) [6,7], were made at The Scatter Works, Inc. using a CASI Scatterometer operating with a 1.06 µm source. This wavelength was selected because it is a typical operational wavelength in HEL applications. All of the measurements were done on a clean bench under HEPA filtered air. The CASI noise floor is between 10^{-7}/sr and 10^{-8}/sr. The system is calibrated before each set of scans using a diffuse Spectralon sample that has a near Lambertian response and a total reflectance of 0.99. After calibration, a short signature (no sample) scan is taken to check the noise floor and obtain a reading of the incident power to be used for the calculation of the BSDF. The incident laser power is monitored continuously to ratio out any changes during sample measurements. A sample alignment laser intersects the source beam at the goniometer center and the beam intersection is used to assure that the sample is correctly located.

A-plane and c-plane sapphire samples were measured at five incident angles (0, 15, 30, 45 and 60 degrees from normal) in transmission and five incident angles again in reflection using 5 degrees instead of 0 to avoid blocking the incident beam when measuring near the specular reflection. The samples were 2" diameter and approximately 0.300" thick, and were taken from sapphire blanks which are routinely ground and polished at II-VI OS, so as to be completely representative of a large format sapphire window. The process was repeated with both s- and p-polarized sources. Scans started at -88 degrees with a large aperture and finished at 88 degrees from surface normal. The aperture size and the step size between points were decreased as the specular beam was approached to allow measurements near the specular beam. Step size varied from 0.05 degrees to 2.0 degrees as the detector aperture changed from 2.27×10^{-6} sr to 3.3×10^{-3} sr in four variations. Separation from the signature near specular was typically under 0.2 degrees.

2.2 Absorption

The c-plane sapphire samples described above were also used to measure the bulk absorption. A Photothermal Common-Path Interferometry (PCI) system was utilized. The II-VI OS PCI test system is outfitted with a 1.064µm pump laser. Again, this wavelength was selected for the same reason listed in the Section above. PCI is a well-known and reliable method for performing low-absorption measurements on bulk material samples as well as thin films [8,9]. The measurement is based on a pump/probe technique. The pump beam has comparatively higher power and smaller waist to allow measurement of a selected region of the sample under test. The beam is chopped to modulate the heating effect to a known frequency. The pump beam distorts and causes a small perturbation in the wavefront which results in an amplitude contrast, and this is proportional to known material properties. The interference maximum is detected by the probe beam, measured on a proportional photodetector and further processed by a lock-in amplifier. The bulk absorption is then calculated relative to the system calibration on known standards. II-VI OS has utilized this measurement technique and test system for assessing a number of bulk material types including YAG, YLF, GaF, and GaAs, for example, as well as other optical glasses and thin film coatings. Sapphire is the newest bulk material at II-VI OS to be studied in depth via PCI.

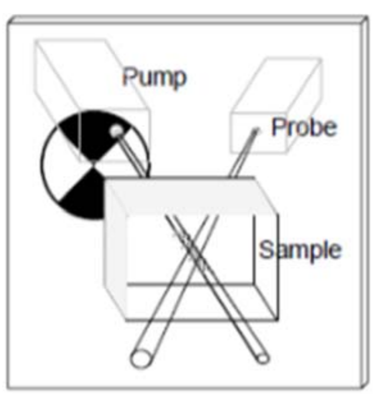

Figure 1: Example of PCI layout used in the system at II-VI OS, from Reference [9].

2.3 Coefficient of thermal expansion

Coefficient of thermal expansion was evaluated on c-plane sapphire bars using a Linseis dual pushrod dilatometer, measured differentially with an alumina standard in an argon atmosphere, per the guidelines and requirements of ASTM E228 [10]. The argon atmosphere aids in the reduction of frozen condensate in the furnace. Each sample was evaluated from -75 °C to +200 °C. Specimen lengths were measured using a 1-2 inch digital Mitutoyo micrometer.

2.4 Knoop Hardness

Knoop hardness was evaluated on 1” round c-plane sapphire samples using a Mitutoyo micro-hardness tester fitted with a Knoop diamond, per the guidelines and requirements of ASTM C730 [11]. A 100 gram load was used with a 20 second dwell time. The samples tested had polished surfaces. Due to the small size of the indents, the indents were measured using a Nikon metallograph to yield a more accurate measurement. The results presented are an average of 10 useable indents made on each sample.

3. MEASUREMENT RESULTS

3.1 BTDF and BRDF

In total, four (4) samples were measured as described in Section 2.1 above. Two samples were a-plane material and two samples were c-plane material, and are identified as sample A1, B, C and D. Sample A1 and sample B are a-plane samples; sample C and sample D are c-plane samples. This resulted in 80 total measurements. A representative subset of these measurements is provided in the figures below. For each set of test conditions, one sample was chosen of the four as a representative data set for all the samples. This was necessary to provide a reasonable representation of the data collected in lieu of including all 80 measurements in this paper. Surface scatter is directly related to surface roughness, which is related to the fabrication processes used to grind and polish the surface of the sapphire substrate. The samples were evaluated for surface roughness and surface quality (SQ) prior to measurement. The surface roughness was measured with a white light interferometer. All samples had a measured surface roughness between 5 – 20 Angstroms. SQ is defined with a scratch/dig number per MIL-PRF-13830B where the scratch number is the maximum allowable or measured scratch width in microns, and the dig number is the maximum allowable or measured dig size in 1/100 of a millimeter. All samples had a measured SQ of < 5/0, which means the maximum measured scratch width was < 5 microns and no digs were observed when inspected per the requirements in MIL-PRF-13830B. Although there is not a direct, quantifiable relationship between SQ and scatter, it is generally accepted that higher SQ is correlated to higher scatter as well. The data provided in the following figures indicate that based on standard fabrication technologies used at II-VI OS for sapphire finishing; the measured BSDF is generally between 10^{-2}/sr to 10^{-3}/sr. The BSDF is consistent across the entire range of AOIs measured. Work is currently underway at II-VI OS to demonstrate enhanced fabrication processes for HEL applications to further reduce surface scatter, as well as to better characterize the contributions from surface roughness and subsurface damage to the overall measurement.

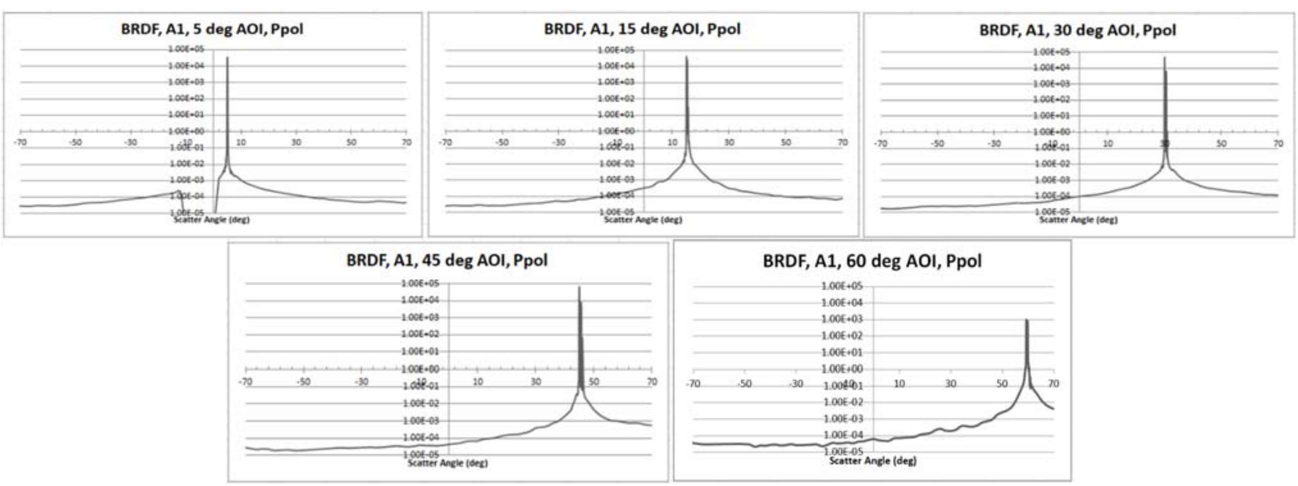

Figure 2: BRDF measurements made at 5, 15, 30, 45, and 60 degree AOI with p-polarized light at 1.064µm on an a-plane sapphire sample identified as sample A1. Sample B, C and D were evaluated under the same test conditions and demonstrated extremely similar results.

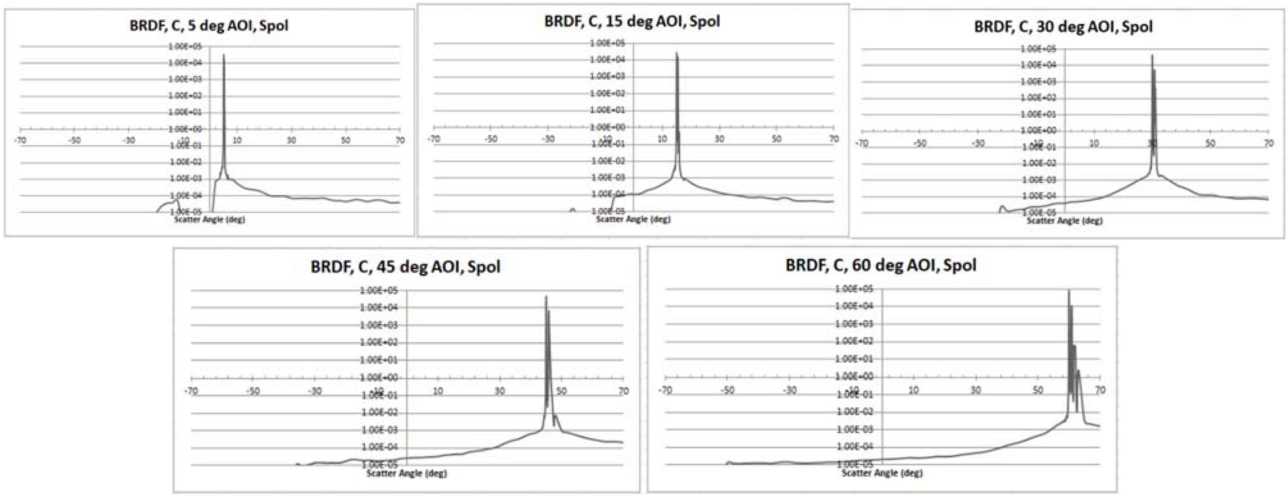

Figure 3: BRDF measurements made at 5, 15, 30, 45, and 60 degree AOI with s-polarized light at 1.064µm on a c-plane sapphire sample identified as sample C. Sample A1, B, and D were evaluated under the same test conditions and demonstrated extremely similar results. In some graphs, there is a weak response to the left of the primary specular reflection peak. This is an artifact of the measurement and should be ignored.

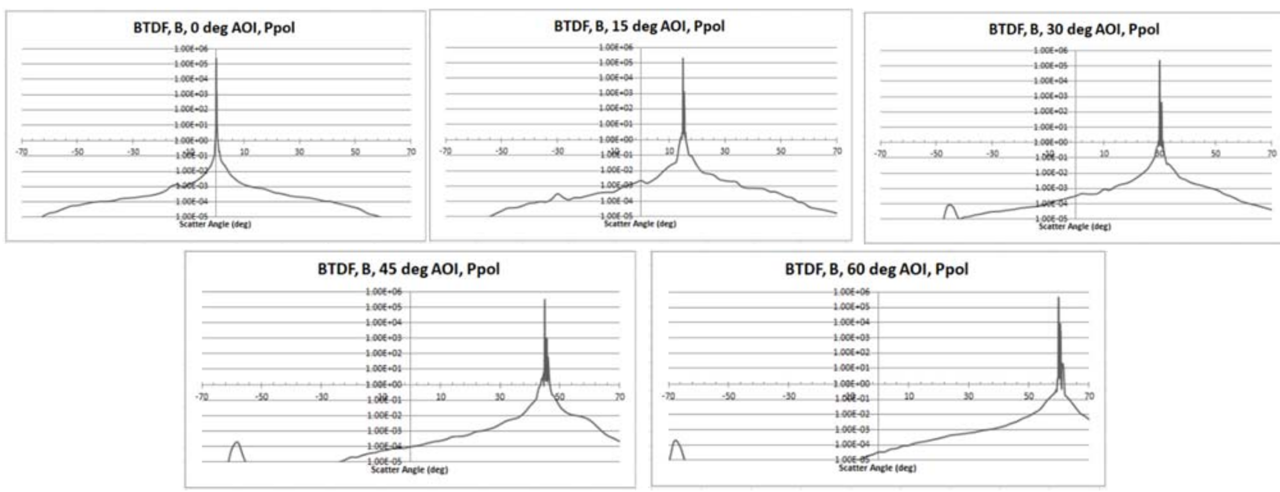

Figure 4: BTDF measurements made at 0, 15, 30, 45, and 60 degree AOI with p-polarized light at 1.064µm on an a-plane sapphire sample identified as sample B. Sample A1, C, and D were evaluated under the same test conditions and demonstrated extremely similar results. In some graphs, there is a weak response to the left of the primary specular reflection peak. This is an artifact of the measurement and should be ignored.

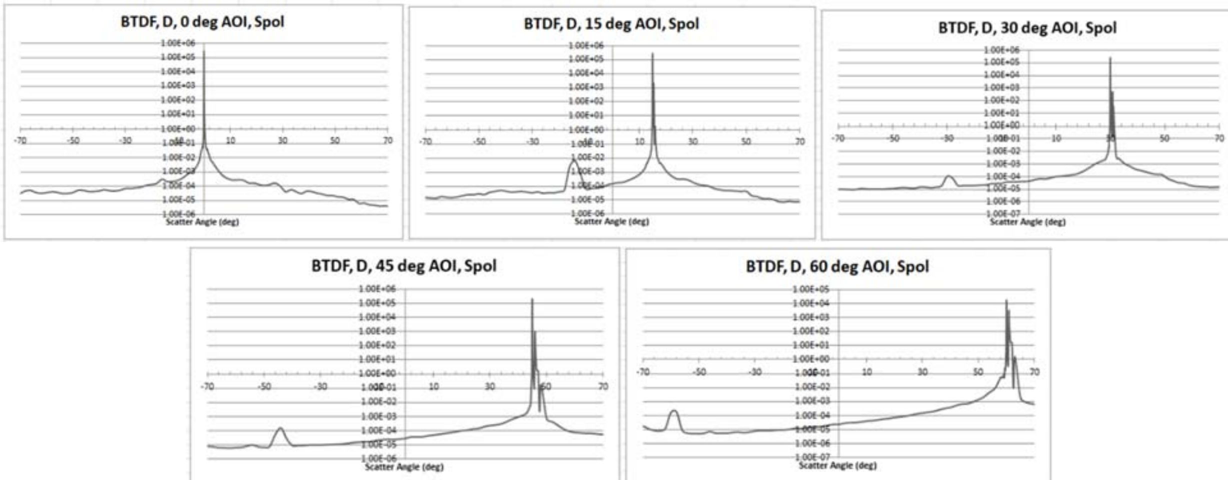

Figure 5: BTDF measurements made at 0, 15, 30, 45, and 60 degree AOI with s-polarized light at 1.064µm on a c-plane sapphire sample identified as sample D. Sample A1, B, and C were evaluated under the same test conditions and demonstrated extremely similar results. In some graphs, there is a weak response to the left of the primary specular reflection peak. This is an artifact of the measurement and should be ignored.

3.2 Absorption, CTE and Knoop Hardness

Absorption measurements were made on the c-plane samples prior to scatter measurements. As mentioned in Section 2, II-VI OS has just begun to characterize its sapphire material for bulk absorption. On-going work will continue at II-VI OS and with other respected industry experts to compare results and confirm precision and accuracy on these and other selected samples via “round-robin” studies. Measurements indicate an average bulk absorption of 35 ± 2 ppm at 1.064µm for II-VI OS sapphire grown via the heat exchanger method. An example of the PCI plot generated by these measurements is provided in the figure below.

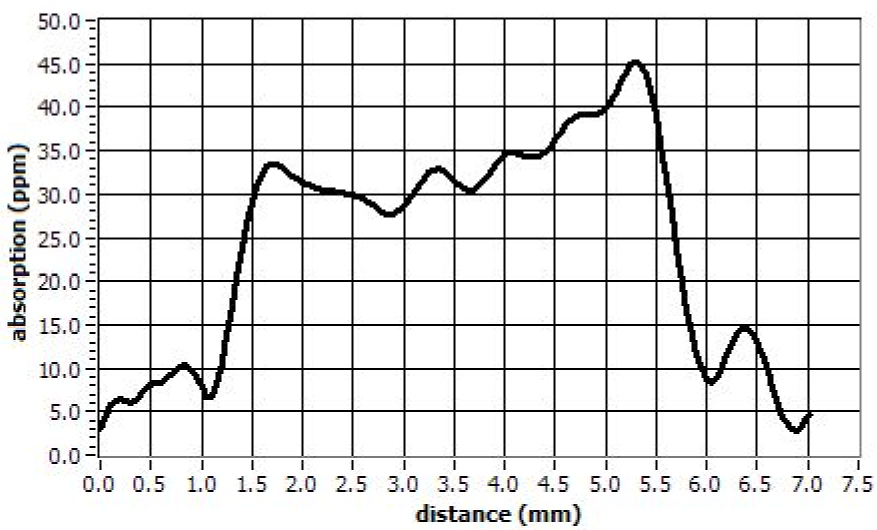

Figure 6: PCI plot of absorption measurements on II-VI OS sapphire. The plot shows the absorption in ppm vs. depth in sample.

Coefficient of thermal expansion (CTE) and Knoop hardness were evaluated on c-plane samples from the same blanks used to create the samples described in the previous sections. Two samples were made for each test. The intention of these tests was to provide data on c-plane material in comparison to measurements previously performed on a-plane material. [1,3]. The sapphire bars used to measure coefficient of thermal expansion had a ground finish on all surfaces. The range in CTE values correlate respectively to the temperature range reported, that is: the lower CTE value represents measured CTE from -75 °C to room temperature; the higher CTE value represents measured CTE from room temperature to +200 °C. Knoop hardness samples had polished surfaces. Results are presented in Table 1.

Table 1: Thermo-mechanical material property results

Property	Sample ID	Result		Units
		-75 °C to RT	RT to +200 °C	
CTE	Sample 1	4.1	6.0	ppm/°C
	Sample 2	4.2	6.0	
		Result		
Knoop hardness	Sample 1	AVG. 1464; ST. DEV. 56		kg/mm^2
	Sample 2	AVG. 1469; ST. DEV. 32		

4. DISCUSSION

4.1 BTDF and BRDF

BTDF and BRDF plots should ideally be smooth delta-functions, with the primary peak being specular reflection. The scatter measurements performed on II-VI OS sapphire demonstrates reasonably low surface scatter. The measured range for BSDF is between 10^{-2}/sr to 10^{-3}/sr. Importantly, no bulk scatter was observed in any of the measured samples, indicating high crystal quality in II-VI OS sapphire material. This fact is based on visual observation when aligning the samples during the test set up with a HeNe (632.8µm) laser. The observed scatter performance in a-plane and c-plane samples is nearly identical. One interesting artifact in the measurement is the appearance of additional peaks to the right of the specular reflectance peak in all the non-zero AOI measurements; refer to the example in Figure 7 of a zoomed plot of the BRDF of sample A1 at 30 degree AOI. The first peak is the specular reflection at the measurement angle; the next two peaks are the result of internal reflections of the laser that are consistent with Snell's law. Further work is necessary to understand how an AR coating might affect these internal reflections. Development work is also actively underway at II-VI OS to improve fabrication processes to reduce BSDF for large sapphire windows.

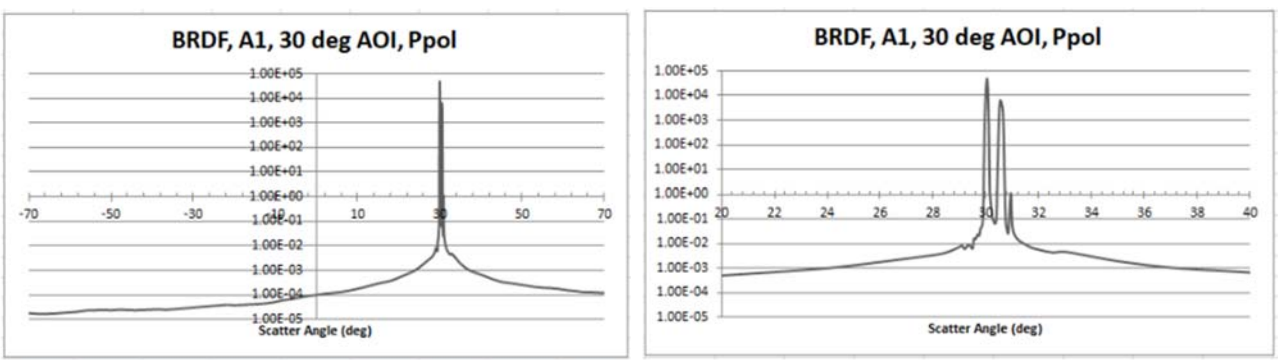

Figure 7: Example of BRDF/BTDF measurements at non-zero AOI. Right plot is typical for BRDF; left plot shows a zoomed view of the same data. The secondary and tertiary peaks to the right of the primary reflection peak are the results of internal reflections within the material.

4.2 Absorption, CTE and Knoop Hardness

Absorption measurements resulted in an average bulk absorption of 35 ± 2ppm for samples tested. Material absorption in wavelength regions of high transmission is caused principally by impurities in the substrate material. Impurities incorporated in the final boule are most commonly caused by impurities in the raw material. Measurements were performed via Glow Discharge Mass Spectrometry (GDMS) on the raw material used for sapphire growth at II-VI and measured between 5N – 7N purity. Measurements have also been made on final boules and indicate an average purity of 6N. Further work is planned to understand how raw material purity impacts bulk sapphire absorption.

CTE and Knoop hardness measurements are consistent with previously reported values for c-plane sapphire [12]. Sapphire demonstrates a low CTE value over the temperature range measured, which is desirable for HEL applications. As mentioned previously, the high Knoop hardness value indicates superior scratch resistance.

5. CONCLUSION

II-VI OS sapphire material, grown via the heat exchanger method, and fabricated using standard fabrication technologies at II-VI OS for large area windows, was evaluated for material performance properties that are important to HEL applications. Specifically, BSDF was evaluated on a-plane and c-plane samples at 1.064µm. Bulk absorption, CTE and Knoop hardness was evaluated on c-plane samples only. The wavelength was selected based on typical HEL applications. II-VI OS expects similar performance at other wavelengths based on previously reported scatter data for synthetic sapphire [13]. The intention of this effort is to demonstrate that in addition to sapphire being an excellent material for many challenging aerospace applications; it also has many desirable performance properties for HEL applications as well. The BSDF measurements indicate reasonable scatter performance for II-VI OS sapphire, but more work is necessary to improve the BSDF. Importantly, visual observations during the measurements indicate that there is essentially zero bulk scatter, indicating II-VI OS sapphire material is high quality and an excellent material candidate for HEL applications. Further work is also planned to understand and fully characterize bulk material absorption. The results for CTE and Knoop hardness are consistent with published data for c-plane sapphire.

II-VI OS recognizes for window materials used in envisioned HEL applications, both the bulk material performance and the surface processing techniques used are critical properties and must be well understood and characterized. Work at II-VI OS is on-going to make continual progress towards desired key attributes, not just for sapphire, but for other materials as well.

6. ACKNOWLEDGEMENTS

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