Homogeneity of material and optical properties in HEM grown sapphire

M. Stout*, D. Hibbard

II-VI Optical Systems
36570 Briggs Rd., Murrieta, CA 92563

ABSTRACT

Sapphire crystal boules, approximately 34 cm in diameter and 22 cm tall, grown by the Heat Exchanger Method (HEM) are currently being sliced, ground and polished for use as window substrates in a variety of aerospace applications. As the need for larger volumes of higher quality material increases, it is necessary to evaluate and understand the homogeneity of optical and material properties within sapphire boules to ensure the needs of the industry can be met. The optical homogeneity throughout the full useable thickness of a representative sapphire boule was evaluated by measuring the transmitted wavefront error of multiple thin slices. This approach allowed the creation of a full-volume three-dimensional homogeneity map. Additionally, the uniformity of other critical characteristics of the material was evaluated at multiple locations within a boule. Specific properties investigated were equibiaxial flexural strength, index of refraction, Knoop hardness, coefficient of thermal expansion and modulus of elasticity. The results of those evaluations will be reported.

Keywords: sapphire, HEM, heat exchanger method, homogeneity, optical properties, material properties

1. INTRODUCTION

Sapphire remains the material of choice for many aerospace applications in the visible, near-infrared (NIR) and mid-wave infrared (MWIR) because of its high transmission in those wavelength regions, combined with its desirable material properties. Sapphire is most commonly grown in the a-plane. HEM boules, like the one shown in Figure 1, can then be oriented using x-ray diffraction techniques for use in either the a-plane, c-plane, r-plane, or m-plane. However, all measurements and data presented in this paper are for a-plane material.

* Further author information-
M. Stout (correspondence): Email: Melissa.Stout@ii-vi.com Telephone: 951-325-7568; FAX 951-926-3751
D. Hibbard (correspondence): Email: Doug.Hibbard@ii-vi.com Telephone: 951-9262994; FAX 951-926-3751
www.opticalsystems.com

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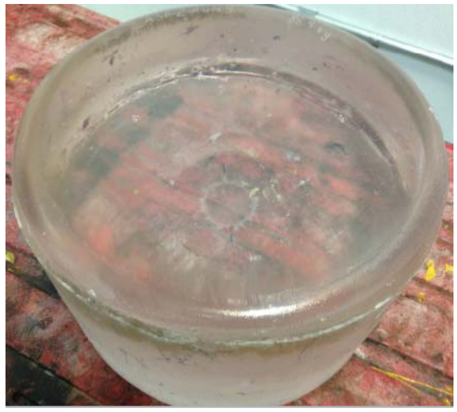

Figure 1: Typical boule produced from the HEM method

As the interest in sapphire continues to grow and requirements move toward more rigorous definition, it becomes more important to fully understand how the material will perform and what the reasonable expectation should be for the material regardless of where it comes from in a given boule. Currently, II-VI Optical Systems is growing sapphire crystal boules via the HEM method in the a-plane, 34 cm in diameter and 22 cm tall. Based primarily on visual inspection, approximately 15.5 cm of the height is considered usable material for aerospace applications, as shown in Figure 2. II-VI Optical Systems recently undertook an effort is to better understand the uniformity of refractive index homogeneity throughout the entire useable height of a given HEM boule, as well as the homogeneity of other material properties within a boule and across multiple boules. The results of that study are reported herein.

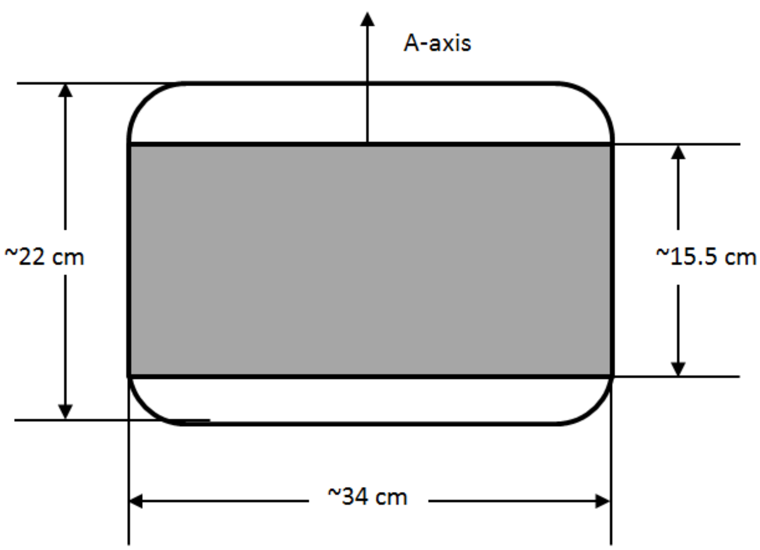

Figure 2: Typical dimensions of an as-grown a-plane HEM boule. The portion of the boule which is considered optical quality is shown in gray. The crystalline A-direction is shown.

2. TEST METHODOLOGIES

2.1 Refractive Index Homogeneity

Homogeneity measurements were made on 34 cm diameter by 5.7 mm thick a-plane sapphire samples using a 632.8 nm Fizeau interferometer and 25.4 cm (10.0”) aperture. Measurements were made of the test cavity, the transmitted wavefront, surface 1 and surface 2 of each sample. Using the Zygo Polished Homogeneity (PHom) application, the cavity and surface measurements were subtracted from the transmitted wavefront measurement, resulting in a homogeneity result for the entire 25.4 cm aperture. There is a clear advantage to this method versus the heritage oil-on-plate approach, as it allows for direct measurements of the surfaces and transmitted wavefront [7]. This minimizes error due to thermal effects, or possible error introduced by using index matching fluid. Samples were collected at several locations throughout the full useable thickness of a standard HEM boule.

2.2 Thermo-Mechanical Material Properties

Equibiaxial flexure strength, coefficient of thermal expansion, modulus of elasticity, and Knoop hardness were tested on three HEM boules, each grown in a different furnace. Samples were taken from the top and bottom of the useable height of each boule. All samples were a-plane sapphire. Each boule was grown using the same sources for raw alumina material, to eliminate any variation possibly caused by difference in impurities.

Table I lists the sample and test configuration for each test performed.

Table I: Sample and test configuration for material properties

Test	Sample Configuration	Test Method	Test Location
Equibiaxial flexure strength	Ring on Ring 7cm dia. x 5.7 cm	ASTM C1499	II-VI Optical Systems
Coefficient of thermal expansion	3 mm x 5 mm x 50 mm	ASTM E228	University of Dayton, Research Institute
Modulus of elasticity	3 mm x 5 mm x 50 mm	ASTM C1259	University of Dayton, Research Institute
Knoop hardness	2.54 cm x 5.7 cm	ASTM C730	University of Dayton, Research Institute

Equibiaxial flexure strength was evaluated via ring-on-ring samples created from the top, middle and bottom portions of each boule. All samples were configured per the guidelines and requirements per ASTM C1499 [2], and were fabricated using II-VI Optical Systems standard production processes. Samples were tested on an Instron Universal Testing machine, Model 5982. It is important to note that the results presented are only representative of II-VI Optical Systems processes, as previous work has indicated that surface quality has an impact on final results. [5,6] The data were processed using standard statistical analysis and Weibull analyses.

Coefficient of thermal expansion was evaluated on sapphire bars using a Linseis dual pushrod dilatometer, measured differentially with an alumina standard in an argon atmosphere, per the guidelines and requirements per ASTM E228 [4]. The argon atmosphere aids in the reduction of frozen condensate in the furnace. Each sample was evaluated from -75 ^oC to +200 ^oC. Specimen lengths were measured using a digital micrometer.

Elastic modulus was evaluated on the same sapphire bars used for evaluation of the coefficient of thermal expansion, per the guidelines and requirements of ASTM C1259 [1]. Each sample was weighed and measured prior to test. A flexible polymer rod with a silicon nitride ball bearing attached was used to strike the specimen at its center. A transducer was used to measure the sample frequency. The fundamental flexural resonance frequency of each sample was displayed on a GrindoSonic. Dynamic modulus is calculated using the following equation:

$$E = 0.9465 * \frac{m{f_f}^2}{b} * \frac{L^3}{t^3} * T_1 \quad (1)$$

where E is the modulus of elasticity, m is the mass of the bar, b is the width of the bar, L is the length of the bar, t is the thickness of the bar, f_f is the fundamental resonant frequency of the bar in flexure, and T_I is the correction factor for fundamental flexural mode to account for finite thickness of the bar. T_I is calculated as follow:

$$T_1 = 1 + 6.585(1 + 0.0752\mu + 0.8109\mu^2)\left(\frac{t}{L}\right)^2 - 0.868\left(\frac{t}{L}\right)^4 - \left[\frac{8.640(1+0.2023\mu+2.173\mu^2)\left(\frac{t}{L}\right)^4}{1+6.338(1+0.1408\mu+1.536\mu^2)\left(\frac{t}{L}\right)^2}\right] \quad (2)$$

where μ = Poisson's Ratio. For calculating elastic modulus, 0.285 was used for Poisson's Ratio.

Knoop hardness was evaluated on 1" round a-plane sapphire samples using a Buehler micro-hardness tester fitted with a Knoop diamond, per the guidelines and requirements of ASTM C730 [3]. A 100 gram load was used with a 20 second dwell time. The samples tested had polished surfaces. The results presented are an average of 10 useable indents made on each sample.

3. MEASUREMENT RESULTS

3.1 Refractive Index Homogeneity

Index homogeneity was evaluated on 11 a-plane sapphire samples, 34 cm in diameter by 5.7 mm thick, using an approximate 25.4 cm aperture. They were measured on an unpolarized Zygo interferometer at a wavelength of 632.8 nm. All samples were sliced from a single sapphire boule grown via the HEM process. For each result presented, a measurement was made of the interferometer cavity, the transmitted wavefront, and a reflectance measurement of surface 1 and a reflectance measurement of surface 2. For convention, surface 1 was maintained to always be the side towards the top of the HEM boule. For each test, temperature was allowed to stabilize before a measurement was captured. Samples were taken at various locations throughout of the useable height of the boule (see Figure 2). Sample 1 came from near the top of the useable boule height; sample 11 came from near the bottom. The homogeneity results are presented in Table II and Figure 3.

Table II: Homogeneity Results

Sample Identifier	Result RMS (ppm)
1	33.9
2	63.2
3	27.2
4	35.5
5	63.7
6	41.1
7	52.9
8	40.9
9	41.2
10	76.1
11	25.7
Average	45.6
Standard Dev.	15.5

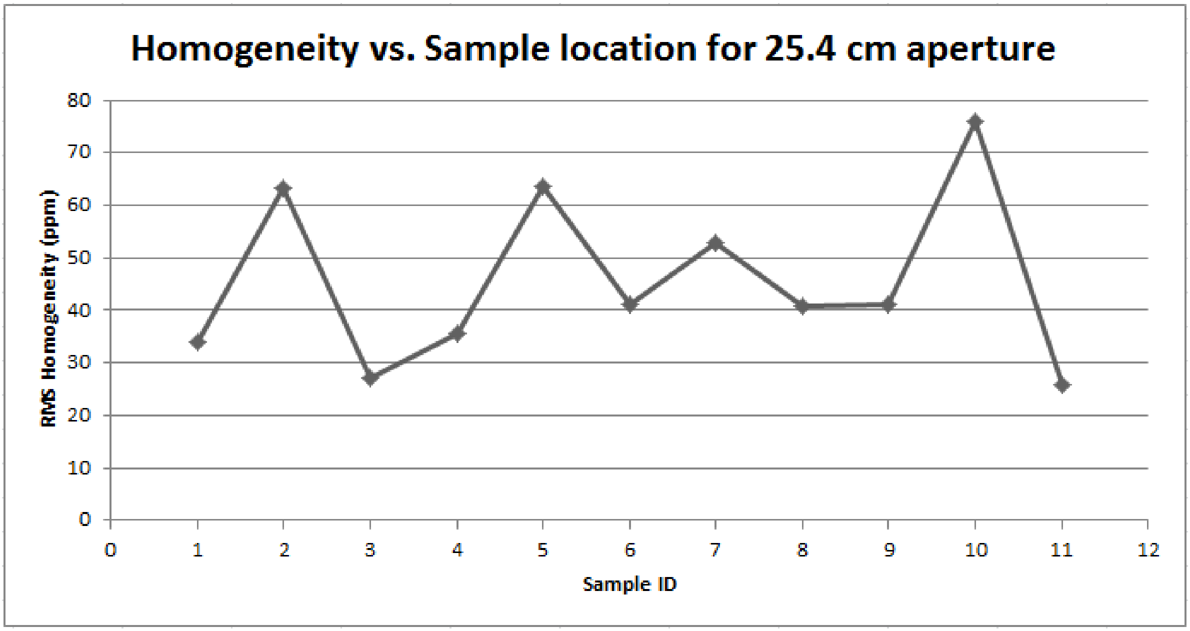

Figure 3: Graphical plot of homogeneity results for 25.4 cm aperture vs. sample ID. Sample 1 came from near the top of the useable boule height. Sample 11 came from near the bottom of the useable boule height. All samples were 5.7mm thick.

3.2 Thermo-Mechanical Material Properties

Equibiaxial flexure strength was evaluated using a set of ring-on-ring samples made from slices from the top, middle and bottom of the useable height of each boule. Equibiaxial flexure strength coupons were prepared using II-VI Optical Systems fabrication processes, and were all polished to the same surface quality. The Weibull modulus for Boule 1, Boule 2, and Boule 3 was 3.5, 3.6 and 4.4, indicating that the failure mode and distribution was consistent for the samples made for all 3 boules. No unusual distributions of sample results were observed. The mean strength for each set of samples is presented in Table III.

Coefficient of thermal expansion, modulus of elasticity and Knoop hardness were evaluated on 2 samples from each of the 3 boules grown, with each boule being grown in a different furnace. The intention of these tests was to gain an understanding on how material properties varied within a single boule and across multiple boules. The sapphire bars used to measure coefficient of thermal expansion and modulus of elasticity had a ground finish on all surfaces. Knoop hardness samples were polished to the same surface quality as the ring-on-ring samples used for equibiaxial flexure strength testing. Results are presented in Table III.

Table III: Thermo-mechanical material properties results

Property		Results			Units
		Boule 1	Boule 2	Boule 3	
Equibiaxial Flexure Strength	Strength	57.2	73.6	62.21	ksi
	St. Dev	18.3	24.6	15.8	
Coefficient of Thermal Expansion (-75 to +200 ^{o}C)	Sample 1	4 – 6	5 – 7	4 – 6	^{o}C/ppm
	Sample 2	4 – 6	5 – 7	4 – 6	
Modulus of elasticity	Sample 1	56E+06	65E+06	57E+06	psi
	Sample 2	56E+06	66E+06	56E+06	
Knoop Hardness	Sample 1	1550	1660	1770	N/A
	Sample 2	1500	1670	1830	

4. DISCUSSION

4.1 Refractive Index Homogeneity

What is particularly interesting about the homogeneity results is there appears to be no clear correlation between the measured homogeneity and the location along the boule's axial direction from which the sample was sliced. Homogeneity data have been reported previously for HEM sapphire, but for much smaller aperture sizes [7,8]. For example, values of 2-9 ppm were reported for specimens that were only 5 cm diameter by 10 mm thick [8]. Based on those prior results, the currently observed slice-to-slice variation for the 25.4cm aperture indicates that aperture size is critically important when specifying an index homogeneity requirement. Using the standard textbook approach, a 45 ppm inhomogeneity level for a 25.4 cm aperture is calculated to be equivalent to an 8-10 ppm inhomogeneity level for a 5 cm aperture. Given that HEM sapphire inhomogeneity has been reported for smaller aperture sizes with relatively consistent results [7,8], the current data indicate that there can be localized variations in index that aren't observed for small apertures, but can significantly affect results as the aperture size is increased. These fluctuations can be due to lattice distortions, lattice vacancies, impurities, or some combination of these factors [8]. As performance requirements for index homogeneity grow more stringent for larger and larger windows these data indicate that further work is required to better predict and ultimately control index inhomogeneity.

4.2 Material Properties

The results for the material property testing indicate that values within a single boule tend to agree very well, but there can be variation in properties from boule to boule. There is some boule-to-boule variation in the equibiaxial flexure strength testing, but this can mostly be attributed to variations in surface quality. Surface quality varied from 20/10 to 60/40 on samples tested. Coefficient of thermal expansion and modulus of elasticity samples that came from the same boule agreed very well with each other; however, the samples from Boule 2 did vary compared to Boule 1 and Boule 3. These are fundamental material properties that are dependent on lattice structure, meaning variations could be caused by lattice distortions, dislocations or impurities. The Knoop hardness results also showed some boule-to-boule variation. This can likely be attributed to the location of the other crystal planes, namely the c-plane and the m-plane within the sample. However, all values measured are consistent with values reported for a-plane sapphire material [5].

5. CONCLUSION

Refractive index homogeneity and other material properties were evaluated for a-plane sapphire HEM boules grown at II-VI Optical Systems. Homogeneity was evaluated on 34 cm diameter by 5.7 mm thick sapphire samples at various locations within a HEM boule. There appears to be no correlation between where the sample came from in the boule and the resulting homogeneity values measured; however, it is clear that when specifying homogeneity for optical applications, it is important that the aperture size be specified, as larger aperture sizes show larger variations in homogeneity. Other material properties showed a relatively small degree of boule-to-boule variation, another important attribute to keep in mind when specifying material properties for sapphire material. However, the values measured are consistent with current published values for a-plane sapphire.

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