Development in EFG sapphire at II-VI Optical Systems

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ABSTRACT

Large sapphire crystals are currently being grown at II-VI Optical Systems with dimensions of approximately 12" x 36" x 0.300" thick via an Edge Defined Film Fed Growth (EFG) method. These crystals are being used to make large sapphire windows for aerospace applications which require high strength and optical performance in the visible to mid-wave infrared regions. In the following paper, II-VI OS will present material data for their EFG sapphire and compare it to that for sapphire grown via the HEM approach, a method which is also used at II-VI OS. Reported data includes index homogeneity evaluated over the entire grown crystal. Other properties to be presented are equibiaxial flexural strength, index of refraction, Knoop hardness, thermal expansion coefficient and modulus of elasticity. The results presented herein will demonstrate that the material performance of sapphire grown at II-VI OS by either method is entirely comparable.

Keywords: sapphire, EFG, edge defined film fed growth, homogeneity, optical properties, material properties, HEM, heat exchanger method

1. INTRODUCTION & PRIOR WORK

II-VI Optical Systems (II-VI OS) is growing large sapphire sheet crystals via the Edge Defined Film Fed Growth (EFG) method. The as-grown crystals are 12" x 36" x 0.300" thick, with the large face of the sheets being a-plane. These crystals are subsequently ground, polished, shaped and coated to yield large sapphire windows for aerospace applications.

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Figure 1: a) Typical sapphire sheet crystals produced from the EFG method at II-VI OS

Previously, II-VI OS characterized sapphire grown via the Heat Exchanger Method (HEM) for various optical and material properties to develop a thorough understanding of how the material will perform and what the expectation for performance should be regardless of where a window comes from a single boule [1]. These properties included refractive index homogeneity and thermo-mechanical material properties. The following paper presents an evaluation of the same material properties for EFG sapphire to demonstrate that the performance expectations for sapphire grown via either method are entirely comparable

2. TEST METHODOLOGIES

2.1 Refractive Index Homogeneity

Homogeneity measurements were made along the entire length and width of representative sapphire sheets from different growth runs identified as sheets A – G in Section 3. The thickness of the sapphire sheets after grinding and polishing is approximately 5.7mm and measurements were made over various aperture sizes, depending on the technique used. Measurements were performed using two different techniques. The first technique used was the oil-on-plate method; the second technique used interferometric data processed via the Zygo Polished Homogeneity (PHom) application.

The oil-on-plate method was performed by sandwiching a polished sapphire sheet between two test plates and utilized a Cargille index matching fluid with a nominal index of 1.7600 to effectively eliminate effects due to the surface irregularity of the sapphire sheets. Measurements were made of the test cavity and subtracted from the transmitted wavefront measurement. Index homogeneity was calculated using the following equation:

$$Index\ homogeneity = \frac{TWE\ measurement\ (nm)}{Part\ Thickness\ (mm)} \tag{1}$$

The PHom application takes inputs from interferometric measurements made of the test cavity, the transmitted wavefront, surface 1, and surface 2. The PHom application then subtracts the test cavity and surface measurements from the transmitted wavefront measurement which results in a homogeneity measurement. Upon further investigation by II-VI and discussed in [9], it is important to note that this technique does not entirely eliminate surface irregularity contributions in the same way as the oil-on-plate method, and therefore any results presented from this technique should be considered worst case.

2.2 Index of Refraction

Index of refraction measurements were made on a-plane sapphire prisms fabricated from sapphire sheets A, B, and C, The prisms were 1” in diameter with a 0.0787” minimum thickness and an apex angle of 10°. Measurements were made at room temperature, 70°F ± 10°F, of the ordinary refractive index (n_o) at wavelengths of 0.7, 1.06, 1.57, 3, 4 and 5 microns. The measurements were performed by M3 Measurement Solutions, Inc. in Escondido, CA.

2.3 Thermo-Mechanical Material Properties

Additionally, after evaluation of the refractive index homogeneity, samples were collected from sapphire sheets A, B, and C, and evaluated for equibiaxial flexure strength, coefficient of thermal expansion, modulus of elasticity and Knoop hardness. All samples are a-plane sapphire. The same source of raw alumina was used for each growth run, and is also the same source material for the HEM boules evaluated in [1], which will be compared in the later sections of this report. The test methodologies used are also identical to what is reported in [1], but are repeated here for ease of reference. Table 1 lists the sample and test configuration for each test performed.

Table 1: 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 mm	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 mm	ASTM C730	University of Dayton, Research Institute

Equibiaxial flexure strength was evaluated via ring-on-ring samples made from single sapphire sheets and fabricated using II-VI OS standard production processes. This testing was performed using the same support and load ring diameters for the strength data provided on HEM material in [1]. All samples were configured per the guidelines and requirements per ASTM C1499 [3]. 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 [6,7]. 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 [5]. 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 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 [2]. 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:

$$E = 0.9465 * \frac{m{f_f}^2}{b} * \frac{L^3}{t^3} * T_1 \tag{2}$$

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_1 is the correction factor for fundamental flexural mode to account for finite thickness of the bar. T_1 is calculated as follow:

$$T_1 = 1 + 6.585(1 + 0.0752\mu + 0.8109\mu^2)\left(\frac{t}{L}\right)^2 - 0.868\left(\frac{t}{L}\right)^4 - \left[\frac{8.340(1+0.2023\mu+2.173\mu^2)\left(\frac{t}{L}\right)^4}{1+6.338(1+0.1408\mu+1.536\mu^2)\left(\frac{t}{L}\right)^2}\right] \tag{3}$$

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 [4]. 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

Refractive index homogeneity was evaluated on multiple sapphire sheets along the entire length of the crystal using two techniques over different size apertures. The results of these measurements are presented in Table 2 and Table 3.

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Table 2: Homogeneity results; oil-on-plate method. All results are in ppm. Aperture size is 6".

	LEFT			CENTER			RIGHT		
	Ap1	Ap2	Ap3	Ap1	Ap2	Ap3	Ap1	Ap2	Ap3
Sheet A	5.46	7.64	7.09	7.14	8.30	8.18	8.80	12.28	12.84
Sheet B	8.56	9.52	9.63	7.68	9.57	5.92	5.25	3.50	3.81
Sheet C	NA	7.78	5.64	NA	8.95	11.00	NA	10.15	8.40

Table 3: Homogeneity results; PHom Application. All results are in ppm. Aperture sizes are noted

	Aperture 1	Aperture 2
Sheet D	29.6 (11.6" aperture)	23.0 (11.6" aperture)
Sheet E	44.8 (8.9" aperture)	76.5 (8.9" aperture)
Sheet F	93.3 (10" aperture)	83.7 (10" aperture)
Sheet G	60.4 (10" aperture)	55.8 (10" aperture)

3.2 Index of Refraction

Index of refraction measurements was evaluated on a total of 6 wedges fabricated from sheets A, B and C; 2 wedges per sheet. The results are presented in Table 4.

Table 4: Index of refraction measurement of n_o in a-plane sapphire wedges

Wavelength (μm)	Sample 1 SHEET A	Sample 2 SHEET A	Sample 1 SHEET B	Sample 2 SHEET B	Sample 1 SHEET C	Sample 2 SHEET C	Avg.	St. Dev
0.700	1.7551	1.7564	1.7584	1.7574	1.7541	1.7609	1.7571	0.002
1.06	1.7467	1.7535	1.7454	1.7479	1.7466	1.7532	1.7489	0.003
1.57	1.7384	1.7448	1.7456	1.7416	1.7382	1.7443	1.7422	0.003
3.0	1.7047	1.7113	1.7121	1.7104	1.7048	1.7111	1.7091	0.003
4.0	1.6687	1.6748	1.6758	1.6725	1.6693	1.6746	1.6726	0.003
5.0	1.6192	1.6241	1.6250	1.6224	1.6206	1.6238	1.6225	0.002

3.3 Thermo-Mechanical Material Properties

Equibiaxial flexure strength was evaluated using three sets of ring-on-ring samples, one set each made from Sheet A, Sheet B and Sheet C. Equibiaxial flexure strength coupons were prepared using II-VI Optical Systems fabrication processes. The Weibull modulus for Sheet A, Sheet B, and Sheet C is 3.7, 3.6 and 6.6, respectively. No unusual distributions of sample results in the Weibull plots were observed. The mean strength for each set of samples is presented in Table 5.

Coefficient of thermal expansion, modulus of elasticity and Knoop hardness were evaluated on two samples from each of the same three sheets listed above. The intention of these tests was to gain an understanding on how material properties varied across different growth runs with the same raw material used, and how that data compared to previously collected data on II-VI OS HEM sapphire. The sapphire bars used to measure coefficient of thermal expansion and modulus of elasticity had a ground finish on all surfaces. The range in CTE values correlate respectively to the temperature range reported, i.e.: the lower CTE value represents measured CTE from -75 ^oC to room temperature; the higher CTE value represents measured CTE form room temperature to +200 ^oC. 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 5.

Table 5: Thermo-mechanical material property results

Property		Results			Units
		Sheet A	Sheet B	Sheet C	
Equibiaxial Flexure Strength	Strength	53.1	56.0	65.3	ksi
	St. Dev	18.3	13.7	12.0	
	Samples Tested	12	12	16	
Coefficient of Thermal Expansion (-75 to +200 ^oC)	Sample 1	4.3 – 5.9	4.1 – 5.9	4.1 – 6.1	ppm/^oC
	Sample 2	4.0 – 5.9	4.2 – 5.9	4.1 – 6.1	
Modulus of elasticity	Sample 1	61E+06	61E+06	62E+06	psi
	Sample 2	61E+06	61E+06	62E+06	
Knoop Hardness	Sample 1	1627	1647	1510	kg/mm^2
	Sample 2	1656	1600	1590	

4. DISCUSSION

4.1 Refractive Index Homogeneity

As described previously for HEM sapphire, the EFG homogeneity results indicate that aperture size and measurement technique have an impact on reported values. For example, the homogeneity results from the PHom technique cover a range of 23.0 – 93.3 ppm for the large aperture sizes measured on multiple sapphire sheets. II-VI OS reported homogeneity values ranging from 33.9 – 76.1ppm for similar aperture sizes also measured with the PHom technique on blanks fabricated from HEM sapphire boules [1]. Therefore, the current data correlates well with measurements made on HEM material using the same technique. Additionally, measurements made via the oil-on-plate method for 6” apertures are also consistent with previously reported data on EFG type sapphire with a similar aperture size [8]. Therefore, the data confirms that II-VI OS EFG sapphire exhibits consistent performance with previously self-reported values for HEM material, and with previously reported data for sapphire grown via a similar technique, and further confirms that aperture size should be an important consideration when specifying homogeneity values for materials.

4.2 Index of Refraction

The index of refraction measurements for n_o indicate that there is some small degree of variation in the refractive index of sapphire and this should also be an important consideration when specifying the material. As shown in Figure 2 below, when plotted against the dispersion curve based on the dispersion formula reported in [10], the current data shows good correlation with the shape of the historical curve.

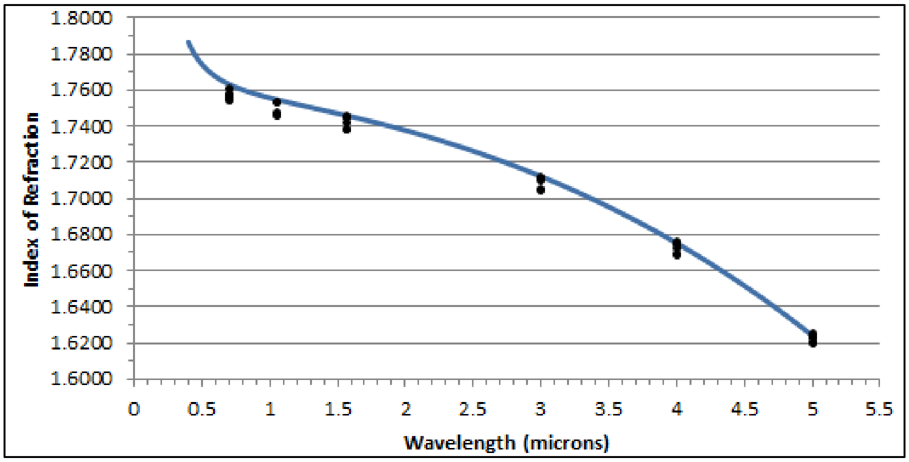

Figure 2: Measured index of refraction data plotted against the dispersion curve for synthetic sapphire as reported in [10].

4.3 Thermo-Mechanical Material Properties

The results for the material property testing indicate that values within a single sheet agree very well, and that there is also little variation sheet-to-sheet (i.e., run-to-run). As observed with the HEM material, there is some sheet-to-sheet variation in the equibiaxial flexure strength testing, but as before this can be attributed to differences in surface imperfections and surface quality [6,7]. In general, localized surface quality varied from scratch/dig tolerance of 20/10 to 60/40 on the samples tested. The other material properties tested demonstrated very small variations sheet-to-sheet. Given that these are fundamental material properties that are principally dependent on lattice structure, this indicates that the EFG process used at II-VI produces a product with consistent and predictable crystal quality. There was some boule-to-boule variation observed in the data reported on HEM material, however, the data reported here is in the same family as what was reported for HEM [1], and all measurements are consistent with historical values reported for a-plane sapphire material [6].

5. CONCLUSION

A number of optical and thermo-mechanical material properties were evaluated for a-plane sapphire grown via the EFG method. The intention of this effort was to demonstrate that the material properties and performance of II-VI OS EFG sapphire are consistent with II-VI OS HEM sapphire, and historical data for synthetic sapphire [1,6,10]. The data reported herein show that all the data is in agreement. Specifically, regarding the index homogeneity measurements, the data shows that homogeneity values measured on II-VI OS EFG material is consistent with data reported for different aperture sizes and different measurement techniques for other synthetic sapphires. Additionally, however, it is clear based on what was reported that there is some variation in material properties that should be considered when specifying properties of interest for sapphire. Regardless, all values measured and reported are consistent with published data for a-plane sapphire.

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