

HEL quality polishing of large sapphire substrates

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ABSTRACT

II-VI Aerospace & Defense currently supports the defense industry on several aerospace platforms by providing high quality, large, single crystal sapphire substrates and windows. There continues to be a rapidly increasing interest in large substrates capable of use in high energy laser (HEL) systems. Such applications require materials with high thermal conductivity and high thermal shock resistance, high strength, and near-zero defect levels. Laser damage-inducing defects include those which are incorporated in the substrate during crystal growth, as well as defects incurred during routine optical fabrication and finishing processes. In the following paper, II-VI A&D presents data for a-plane and c-plane sapphire windows of sufficient size for projected HEL applications exhibiting laser-grade surface quality and surface roughness, while still maintaining low rms transmitted wavefront error. Included is a discussion evaluating the benefits of sapphire for envisioned HEL windows, as well as engineering challenges that should be considered.

Keywords: sapphire, polishing, edge fed growth, heat exchanger method, material properties, high energy laser, HEL

1. INTRODUCTION

II-VI Aerospace & Defense (II-VI A&D) is continuing their development of state-of-the-art single crystal sapphire substrates for a wide variety of aerospace and defense applications. II-VI A&D has completed several material characterization studies over the last few years [1, 2, 3]; in 2019, this work was specific to high energy laser (HEL) applications [4]. These applications include, but are not limited to: directed energy weapons, defensive countermeasures, guidance, target designators, or adaptive optic systems. Sapphire is an attractive option for HEL applications for a number of reasons; it has high transmission in the VIS – MWIR wavelength regions, high optical uniformity, low CTE and extremely high scratch resistance. Additionally, sapphire has low surface and bulk scatter as well as low absorption [4].

II-VI A&D is growing sapphire in sizes that are desirable for envisioned HEL platforms. Currently, material is grown via the heat exchanger method and the edge defined film fed growth method [6]. Via the heat exchanger method, boules are grown that are approximately 13” in diameter and 10” tall, yielding a-plane sapphire blanks 13” in diameter, as shown in Figure 1. The boule can also be oriented via x-ray diffraction to yield blanks in the c-plane, r-plane, or m-plane crystal orientation. Via edge defined film fed growth, large a-plane sapphire slabs are being grown 12” x 30”, as shown in Figure 2.

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In the following paper, work is presented on process development at II-VI A&D to support HEL requirements specific to surface quality and surface roughness. Multiple a-plane and c-plane blanks, 6.25" diameter x 0.500" thick were ground and subsequently polished with the goal of minimizing surface quality defects, such as scratches and digs, as well as surface roughness. The work presented here lays the foundation at II-VI A&D to scale-up the HEL-quality process for 13" diameter windows and larger pieces. Finally, the discussion section presents considerations and challenges for using single-crystal sapphire in HEL applications.

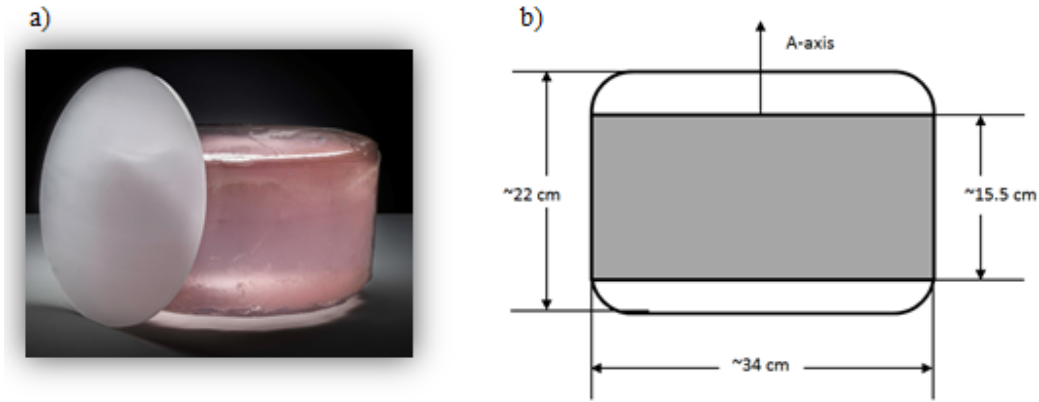


Figure 1: a) A typical boule grown at II-VI A&D produced via the heat exchange method with a representative 13" diameter blank and b) typical dimensions of an as-grown boule. The area shown in grey represents the portion of the boule considered to be high optical quality.



Figure 2: Sapphire sheet crystals growth via the edge defined film fed growth method at II-VI A&D, grown 12" wide and 30" in length.

2. SAPPHIRE SUBSTRATES AT II-VI A&D

Surface roughness and surface quality (SQ) are primary considerations for any material used in a high energy laser application. [5] Low surface roughness and high SQ are desirable, as these can be sources for defect sites in laser damage testing. For this effort, surface roughness was measured with a white light interferometer and is reported in angstroms ($\text{\AA}$). SQ is defined with a scratch/dig number that specifies the maximum allowable scratch width in microns and the maximum allowable dig size in 1/100 of a millimeter and was measured per MIL-PRF-13830B.

Other sapphire suppliers are reporting SQ numbers of 10/5 for sapphire windows up to 2” diameter. However, literature review on the need for HEL-quality windows indicates that window diameters will need to be 13” diameter and larger [5]. Therefore, there is a pressing industry need to demonstrate high SQ numbers on larger diameter windows to support HEL platforms and applications.

Over a two year development process, II-VI A&D investigated grinding and polishing process improvements with the primary goal of achieving high surface quality, SQ of 10/5 or better, while also minimizing surface roughness on optics of sufficient size for large HEL window platforms. A number of variables were investigated using a Design of Experiments (DOE) approach to determine influencing factors in controlling both SQ and surface roughness. These variables included crystal orientation, grinding parameters, polishing parameters, removal rates and tooling factors. In general, the DOEs indicate that the finish of the surface prior to final polishing is one of the most important factors to be considered, followed by tooling factors. Use of the DOE approach not only aided II-VI A&D in developing the process demonstrated on the 6.25” diameter parts described, but also lays a path for scalability to the larger HEL window sizes which are desired. A total of 8 windows, 6.25” x 0.500” thick, both in a-plane and c-plane crystal orientation were processed and evaluated following this development effort. Data are presented in Table 1. For comparison, standard conventional polishing of sapphire aerospace windows typically has a SQ of 60/40, with a measured surface roughness in the range of 5 – 20 Å.

Table 1: Surface quality of 6.25” sapphire development blanks

Sample ID	Crystal Orientation	Roughness (Å)	Scratch	Dig
1	c-plane	22.00	0	20
2	c-plane	25.87	0	0
3	c-plane	16.90	0	0
4	c-plane	22.30	25	0
5	a-plane	16.90	0	0
6	a-plane	12.97	0	0
7	a-plane	14.58	0	20
8	a-plane	22.65	0	0



Figure 3: 6.25” diameter sapphire development blank

The data demonstrates that via sufficient control of grinding and polishing parameters, and tooling factors, II-VI A&D was able to achieve desirable SQ of 10/5 or better on the 6.25” diameter sapphire windows, regardless of crystal orientation. However, there was a trade observed between the achieved SQ and surface roughness. Analysis of the DOEs indicate that one potential improvement path for decreasing surface roughness is further refinement of the surface finish prior to final polishing, and more work is planned to investigate this at II-VI A&D during their work to scale the HEL-quality polishing process to 13” diameter and larger sapphire windows.

3. CONSIDERATIONS FOR SAPPHIRE IN HEL APPLICATIONS

3.1 Material properties for HEL applications

As mentioned in the introduction, there are a number of requirements for HEL systems that must be considered when determining what material to use for these applications. Specifically, materials should demonstrate low coefficient of thermal expansion, low or negative thermo-optic coefficient, low bulk absorption, low bulk and surface scatter and high Knoop hardness [5], in addition to the other properties previously mentioned. Sapphire satisfies a number of these requirements. It is important to emphasize that the data presented in this section is fully representative to 13” or larger sapphire windows.

Sapphire exhibits a low thermal expansion coefficient, making it worthy of consideration for HEL applications. In general, the lower the thermal expansion coefficient, the better [5]. II-VI A&D has characterized our sapphire material from -75 °C to +200 °C. Additionally, measurements have been made on multiple samples to better characterize the expected range for thermal expansion coefficient, as show in Figure 4 below. The range of values is consistent with other literature references [9].

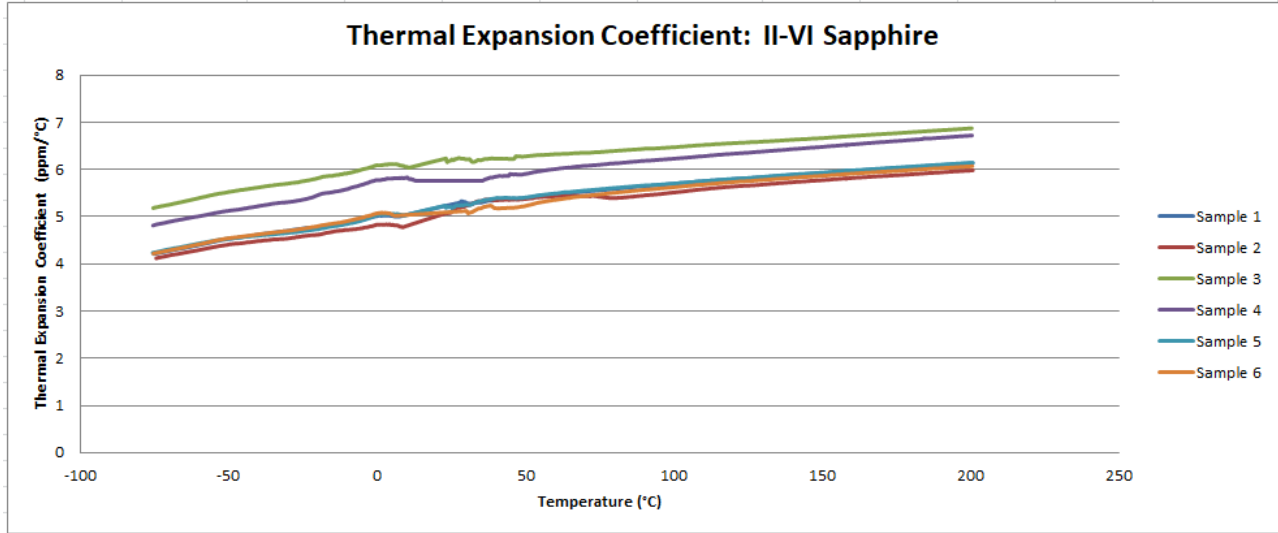


Figure 4: Thermal expansion coefficient measurements of II-VI A&D a-plane sapphire on multiple samples over a temperature range of -75 °C to +200 °C.

Work was completed in 2001 regarding measurement of sapphire refractive index and thermo-optic coefficient by Johns Hopkins University and NIST [7]. Based on this work, sapphire demonstrates a relatively low thermo-optic coefficient for the e-ray and o-ray over wavelength and temperature [7]. However, as with thermal expansion coefficient, the lower this value the better suited the material is for HEL applications, given that this property impacts photothermal heating effects due to the localized temperature changes caused by the laser passing through the window [5].

In 2019, II-VI A&D completed an extensive scatter study for both a-plane and c-plane sapphire material, measuring BRDF and BTDF at multiple angles of incidence (AOIs) and in both s- and p-polarization using a 1.06 μ m source [4]. The measurements demonstrate reasonably low surface scatter, with the BSDF being measured between 10⁻²/sr to 10⁻³/sr. More importantly, no bulk scatter was observed during the surface scatter measurements. Examples of these data are provided in Figure 5 below. In this same paper, measurements were reported for the absorption coefficient and Knoop hardness. Average bulk absorption was measured to be 35 $\pm$ 2 ppm, and more work is underway at II-VI to understand how to better control and reduce absorption in sapphire. Knoop hardness measurements are consistent with previously reported values for a-plane and c-plane sapphire, reported as high at 1830 kg/mm² [1]. High Knoop hardness indicates the superior scratch resistance of sapphire. A table summarizing these properties of interest is provided in Table 2.

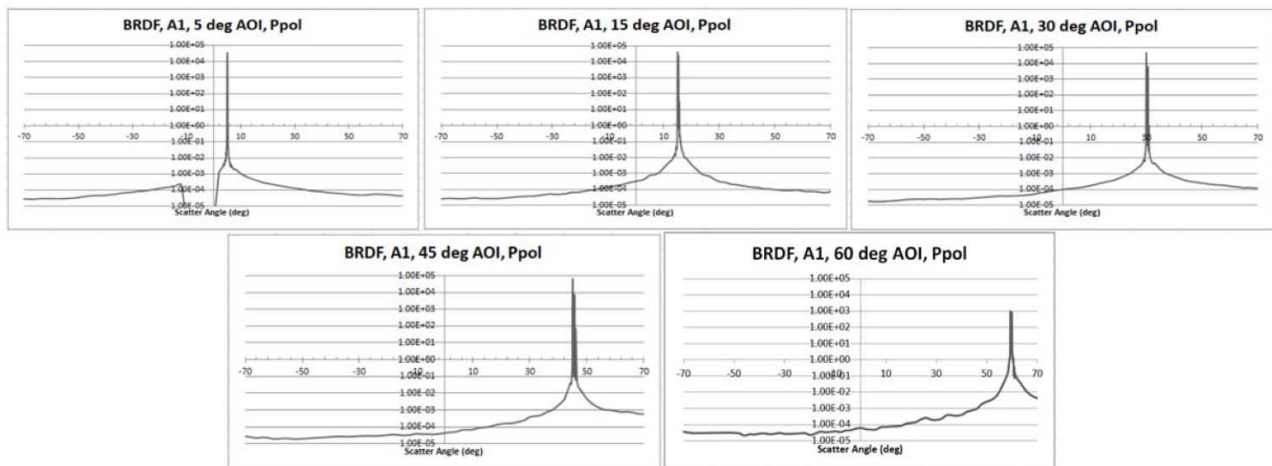


Figure 5: Example BRDF measurements made at 5, 15, 30, 45, and 60 degree AOI with p-polarized light at 1.064 μ m on an a-plane sapphire sample identified as sample A1, published by II-VI A&D in 2019 [4].

Table 2: Sapphire properties pertinent to HEL applications

Property	Results	Unit	Target for HEL windows
Coefficient of Thermal Expansion (-75 °C to RT)	4 - 5	ppm/°C	Low as possible [5]
Coefficient of Thermal Expansion (RT to +200 °C)	6 - 7	ppm/°C	Low as possible [5]
Thermo-optic coefficient [7] dn_o/dT	8.2 (1 μ m) 8.8 (1.67 μ m) 9.9 (2.5 μ m) 11.3 (3.3 μ m) 14.1 (5.0 μ m)	ppm/K	Low or negative [5]
Thermo-optic coefficient [7] dn_e/Dt	11.51 (2.86 μ m) 12.4 (3.3 μ m) 14.03 (4 μ m) 15.56 (5 μ m)	ppm/K	Low or negative [5]
BSDF (s- and p-polarization, 0 – 60 AOI)	10^{-2} to 10^{-3}	sr ⁻¹	$< 10^{-3}/\text{cm}$ [5]
Knoop hardness [1, 4]	1400 - 1830	kg/mm ²	High as possible [5]
Absorption (1.06 μ m)	35 ± 2	ppm	$< 10\text{ppm}$ [5]

Other considerations for HEL use include crystal quality, such as twins, bubbles, and inclusions. Given that sapphire has a non-cubic crystal structure and is anisotropic [6], twins or grains in the crystal can have a significant impact on laser and transmitted wavefront performance. Bubbles and inclusions can be sources for laser damage sites, potentially causing catastrophic failure of the window or optic which has these defects. Therefore, it is recommended the sapphire for HEL applications be specified to be free of twins or grains, and be free of defects such as bubbles or inclusions, as reasonably possible.

Refractive index homogeneity is another important characteristic, as this is a measure of the optical uniformity and can impact transmitted wavefront error and stress birefringence. II-VI A&D has completed studies on both their heat exchanger method and edge fed grown sapphire [1,2,3] and reported values as low as 3.5 ppm for 6” apertures at 632.8nm.

3.2 Design considerations and challenges

The main consideration when using sapphire in the design of a window or dome is that it is naturally birefringent in the a-plane. This means that the e-ray and o-ray travel different paths through the crystal, as explained in detail in Reference 8, “Refractive index of infrared-transparent polycrystalline alumina.” For a plane parallel plate, or a window, this is less of a concern, as the e-ray and o-ray will exit the window parallel to each other. However, for a dome application, this means that the rays will not focus at the same spot, which can cause image blur on the detector for the optical system behind the dome. For this reason, it is important to note that when designing dome systems using sapphire, the orientation of choice is c-plane to minimize the effects of birefringence on performance.

Obviously, ultimate achievable size is a consideration as well. This is not just an issue of what size the material can be grown, and as already detailed in the Introduction, II-VI A&D is growing sapphire of the necessary size for envisioned HEL applications. A considerable challenge with large sized optics is controlling transmitted wavefront error (TWE). II-VI A&D can achieve rms TWE errors of $< \lambda/10$ at 632.8 nm over a 5” diameter aperture, and has the necessary tools and technical skills to achieve more difficult TWE requirements, if required.

4. DISCUSSION AND FUTURE WORK

The results presented herein are exciting developments for sapphire windows. Material characterization studies on sapphire material over the last several years at II-VI A&D support a compelling argument for consideration of sapphire for HEL platforms. Previous experience has shown that, given the high hardness and strength of the material, it can be difficult to achieve high SQ for sufficiently sized windows. Work is underway at II-VI A&D to scale the process development for HEL-quality sapphire windows from a 6.25" diameter window to 13" diameter. Of particular interest in this scale up work, beyond continuing to sufficiently control SQ, we will also be reducing surface roughness to a target of $< 10 \text{ \AA}$. Additionally, II-VI A&D plans to perform laser damage testing on representative test samples at completion of this development work, additional surface scatter testing, as well as strength testing to demonstrate improved modulus of rupture on high SQ parts. Finally, work is also underway to characterize material from multiple boules to better understand how to control and reduce sources of absorption in sapphire crystals, beyond known impurities, which are already controlled via high purity raw materials.

5. CONCLUSION

Work was completed at II-VI Aerospace & Defense to develop a polishing process capable of achieving high SQ on large sapphire windows. Work was completed on a-plane and c-plane windows 6.25" in diameter. II-VI A&D successfully achieved a SQ of 0/0 on both a-plane and c-plane parts; however, surface roughness was slightly increased to as high as $25 \text{ \AA}$, versus a surface roughness of $5 - 20 \text{ \AA}$ typically observed on conventionally polished substrates. Work is planned to scale up the process to 13" sapphire windows, with additional process refinement to reduce surface roughness to $< 10 \text{ \AA}$. Once complete, II-VI A&D intends to perform laser damage testing, modulus of rupture testing, and repeat surface scatter testing on representative samples.

Material characterization data was presented on sapphire material, based on previous work by II-VI A&D and others, which indicates sapphire is a compelling choice for HEL applications given its high strength, high hardness, low CTE and thermo-optic coefficient, reasonably low surface scatter and low absorption. However, given the natural birefringence of a-plane sapphire, this needs to be considered in the design domes or curved parts. For these types of optics, c-plane sapphire is recommended.

REFERENCES

1. Stout, M., D. Hibbard. "Homogeneity of material and optical properties in HEM grown sapphire." *Proceedings of SPIE* 9574 (2015)
2. Stout, M., Neff, B. "Refractive index homogeneity TWE effect on large aperture optical systems." *Proceedings of SPIE* 10179 (2017)
3. Seitz, M., Peterson, G. "Development in EFG sapphire at II-VI Optical Systems." *Proceedings of SPIE* 10627 (2018)
4. Seitz, M., Stoddard, N., Glick, B., Stover, J.C., "Evaluation of sapphire at II-VI Optical Systems for HEL applications." *Proceedings of SPIE* 10985 (2019)
5. Thomas, M.E., Brown, D.M., Spicer J.B., "Requirements for a high energy laser window and components." *16th Electromagnetic Window Symposium* (2018)
6. Dobrovinskaia, E. R., et al. *Sapphire Material, Manufacturing, Applications*. Springer, 2009.
7. Yang, Di, Thomas, Michael E., Kaplan, Simon G., "Measurement of the infrared refractive index of sapphire as a function of temperature." *Proceedings of SPIE* 4375 (2001).
8. Harris, Daniel; Johnson, Linda; Cambrea, Lee; Baldwin, Lawrence; Baronowski, Meghan, et al., "Refractive index of infrared-transparent polycrystalline alumina." *Proceedings of SPIE* 10179 (2017).
9. Harris, Daniel C. *Materials for Infrared Windows and Domes*. Bellingham: SPIE, 1999. Print.