

# Correlation of Sapphire Quality with Uniformity and Optical Properties

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## ABSTRACT

Large sapphire boules, up to 34cm diameter, 65kg, are being grown by the Heat Exchanger Method (HEM) and even larger sizes are sought to meet future requirements of advanced optical systems. These boules, especially in large sizes, exhibit lattice distortion and light scatter in a very narrow range. A qualitative grading system has been developed to characterize sapphire. Windows of five grades and different orientations were prepared and measured for refractive index homogeneity to evaluate transmitted wavefront distortion. The data showed that the refractive index homogeneity for all samples was in the  $10^{-7}$  (0.1 ppm) range. The fact that lattice distortion does not affect the transmitted wavefront allows fabrication of large sapphire windows in production mode at low cost.

**Keywords:** sapphire, crystals, interferometry, optical homogeneity, infrared materials, lattice distortion, scattering centers, Heat Exchanger Method (HEM).

## 1. INTRODUCTION

Sapphire is the current material of choice for midwave infrared (IR) optical systems for scientific, industrial and military applications requiring a stable, robust material with good transmissive properties and available in large size at relatively low cost. Sapphire is stable because it is a simple oxide with a high melting point and ability to withstand oxidizing, neutral and reducing conditions. Sapphire's high strength, toughness, hardness and other desirable mechanical properties, and ability to withstand harsh environments without degradation of its properties, make it robust compared to other materials. Recently, advances in mid-infrared wavelength detectors have minimized the requirement of long wavelength systems and emphasized the need to concentrate on materials with good transmittance in the 3-5 $\mu$ m wavelength region and high abrasion resistance. Higher-speed systems requiring good abrasion resistance with high thermal shock properties favor sapphire because of its robust characteristics in harsh environments.

In addition to the above-mentioned fundamental advantages attributable to properties of sapphire, it is necessary to evaluate the current state of production, quality, uniformity and consistency of the product, and availability in large size. This paper discusses the current state of production of sapphire crystals using the Heat Exchanger Method (HEM), characterization of grown crystals for lattice distortion and scattering centers, a method of grading the quality of sapphire, and correlating the optical properties with the various grades so that a determination can be made to use large sapphire components in optical systems without compromising optical performance.

## 2. ORIENTATION OF WINDOWS

The Heat Exchanger Method (HEM) has grown the world's largest diameter sapphire crystals<sup>1-3</sup>. Routine production of 20cm diameter, 20kg, and 25cm diameter, 30kg, boules has been carried out for over 15 years. Recent demands for large sapphire windows have necessitated production of 34cm diameter, 65kg sapphire<sup>4</sup> and there are plans to develop crystal growth of even larger sizes up to 50cm diameter to meet demands of an advanced optical system.

The most commonly grown sapphire boules are grown as *a*-plane ( $1\bar{1}20$ ), *m*-plane ( $10\bar{1}0$ ), or *r*-plane ( $\bar{1}102$ ). Because of its rhombohedral structure and its resulting anisotropic properties, sapphire components are sometimes specified with *c*-plane (0001) orientation to achieve symmetrical properties. The highest quality (0001) oriented components are fabricated from large diameter ( $1\bar{1}20$ ) or ( $10\bar{1}0$ ) oriented boules where the *c*-axis is orthogonal to the growth axis of the boule. The

(0001) orientation is commonly specified to reduce birefringence properties of sapphire. The commonly grown orientations of  $(11\bar{2}0)$  or  $(10\bar{1}0)$  are used in many optical applications, especially where large sizes are required, and (0001) orientation is used in other applications as size permits to minimize birefringence. The difference in refractive index between the ordinary and extraordinary rays is 0.008.

### 3. QUALITY OF SAPPHIRE

All sapphire boules grown in the industry exhibit some level of imperfections in crystal structure (lattice distortion) and light scatter. The melting point of sapphire is  $2040^{\circ}\text{C}$ ; therefore, growth from the melt will involve controlled solidification from temperatures of approximately  $2100^{\circ}\text{C}$ . Sapphire boule growth by HEM is carried out from the melt in a crucible under a vacuum environment using a heat zone and the appropriate insulation material. A schematic of a HEM furnace is shown in Figure 1.

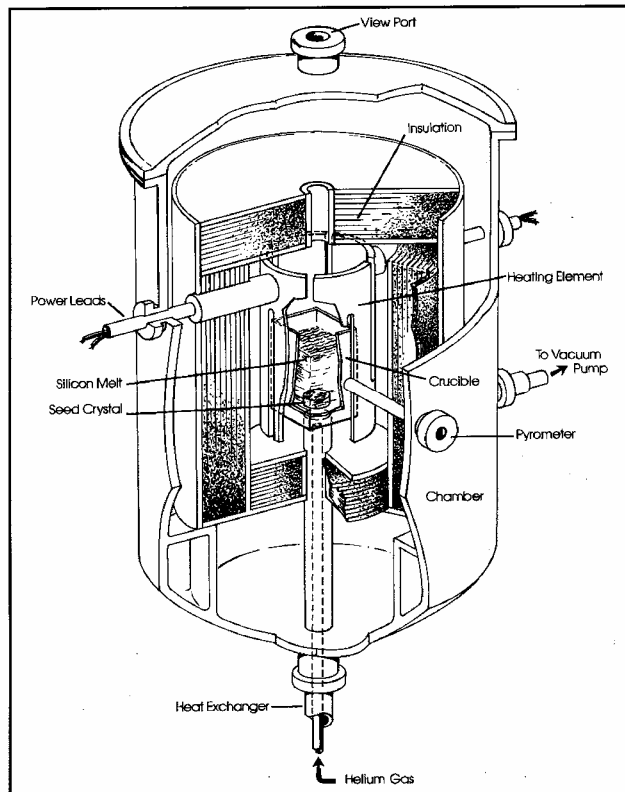


Figure 1. Schematic of a HEM furnace

In the HEM process a single crystal sapphire seed is centered at the bottom of a crucible and loaded with broken pieces of sapphire called crackle. CSI employs high purity crackle to ensure high optical transmission in the crystals and optics made from said crystals. A crucible is loaded with crackle and placed on the heat exchanger in the HEM furnace. The furnace is evacuated and the charge is heated by the heating element. The heat exchanger is located under the crucible and provides a cool spot that prevents seed melt out. After stabilization of the molten sapphire charge and appropriate melt back of the seed crystal, directional solidification is achieved by controlling the heat input and the heat extraction. The HEM process is the only crystal growth process that controls heat input and heat extraction without moving the crystal or crucible. After solidification is completed the crucible is still in the heat zone; the furnace temperature is lowered below the solidification temperature and the heat exchanger temperature is increased so that *in situ* annealing of the boule is achieved prior to controlled cool down to room temperature. *In situ* annealing of sapphire boules can thereafter be carried out after growth so that lattice distortion introduced during the growth can be annealed and further multiplication reduced. After a sapphire boule is grown and annealed, it is still subjected to stresses due to temperature gradients imposed during the cool down cycle; therefore, the cool-down cycle is tailored to minimize these stresses. Competing crystal growth processes impart larger stresses on the crystal because the crystal is physically pulled or flowed away from the heat source.

**HEM sapphire is characterized into five grades based upon lattice distortion and scattering center concentrations.**

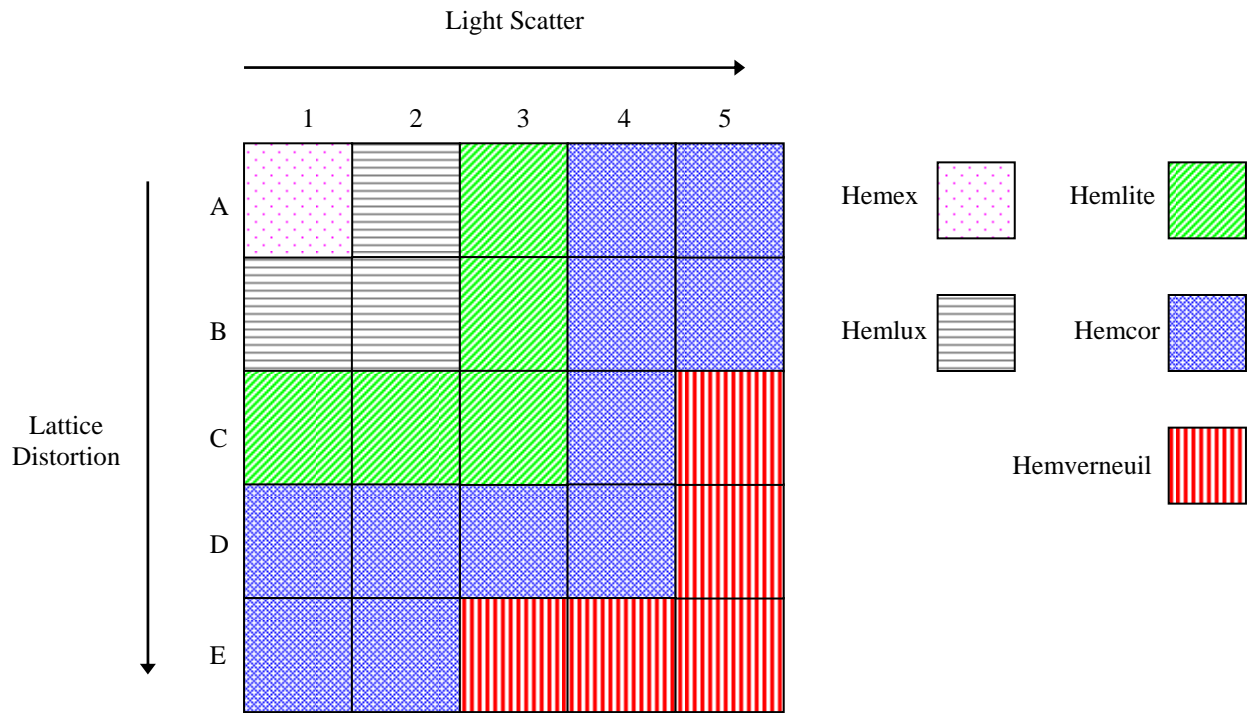


Figure 2. Representation of lattice distortion and light scatter in various grades of sapphire

Lattice distortion is characterized by observation between cross polarizers. Scattering centers in sapphire were observed by illuminating with an intense white light source. For critical applications He-Ne lasers and argon ion lasers were also used as final inspection on polished samples. Based upon qualitative analysis of lattice distortion and scattering centers, standard samples were fabricated which were used periodically to compare with test specimens. These standard samples were the basis for a grading system used to characterize HEM sapphire. Figure 2 shows the range of lattice distortion and/or scattering centers used to specify the grades of sapphire, viz., Hemex, Hemlux, Hemlite, Hemcor and Hemverneuil. The highest quality grade, Hemex, has no observable lattice distortion and light scatter. The lowest optical quality sapphire produced in HEM is called the Hemverneuil grade. The other grades bridge the gap between Hemex and Hemverneuil grades. Small sapphire components can be fabricated using one grade of sapphire; however, for windows larger than approximately 10cm diameter or for production quantities, the material will generally contain a mix of grades.

A testing procedure was developed to correlate the optical properties of sapphire with various grades of sapphire so that this data could be used for specifying sapphire for large windows in optical systems.

#### 4. OPTICAL PROPERTIES

The most important optical properties of large sapphire windows are the optical transmission and transmitted wavefront distortion. The transmitted wavefront distortion is evaluated by measuring the homogeneity of refractive index of sapphire. This measurement is affected by the quality of surfaces of the window as well as by the quality of bulk sapphire. It is possible to prepare samples so that the distortion from surfaces can be extracted from the data and the effect due to

lattice distortion can be evaluated. Changes in lattice distortion could change the refractive index of sapphire to cause wavefront distortion.

Test samples of the grades of sapphire with various degrees of lattice distortion were prepared and characterized for refractive index homogeneity so that the data could be extended to fabrication of large windows. The test windows were 5cm diameter, a size large enough to obtain statistical data, and approximately 1cm thick to meet requirements for 20cm to 30cm size windows. Most windows are specified as  $c$ -plane or orthogonal to it. Therefore, test samples of the various grades were fabricated with (0001) and ( $\bar{1}1\bar{2}0$ ) orientations. The test samples were polished to a 60/40 scratch/dig specification and a slight wedge of approximately  $1^\circ$  was intentionally incorporated in the surfaces of the samples. This allowed extraction of the effect of surface flatness on transmitted wavefront, thus allowing the wavefront distortion to be attributed to the lattice distortion of the sapphire.

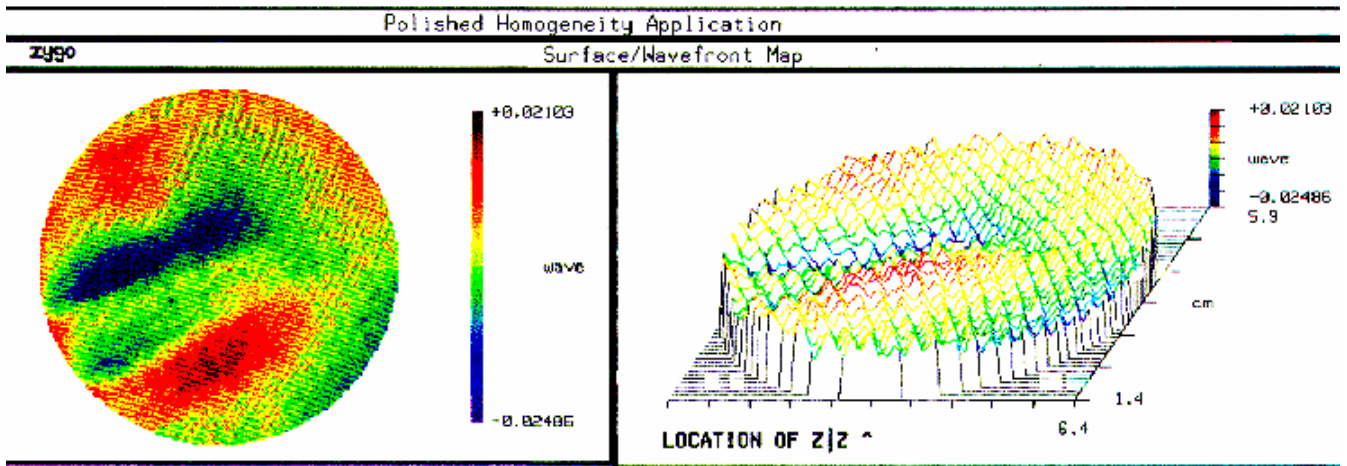
A Zygo Mark IV Interferometer was used for transmitted wavefront distortion measurements of test samples. The data on refractive index, homogeneity rms value and peak-to-valley (pv) value for the five grades of sapphire in two different orientations is shown in Table I.

Table I. Transmitted wavefront distortion data for various grades and orientations of sapphire samples

Sample			Homogeneity	Homogeneity, rms	pv (Wave)
Orientation	ID	Grade			
( $\bar{1}1\bar{2}0$ )	10	Hemex	2.78349E-06	3.340857E-07	0.041
( $\bar{1}1\bar{2}0$ )	9	Hemlux	3.41919E-06	4.347430E-07	0.051
( $\bar{1}1\bar{2}0$ )	7	Hemlite	3.05817E-06	3.660218E-07	0.045
( $\bar{1}1\bar{2}0$ )	8	Hemcor	5.11889E-06	5.744561E-07	0.076
( $\bar{1}1\bar{2}0$ )	6	Hemverneuil	4.21734E-06	6.179205E-07	0.063
(0001)	5	Hemex	1.47575E-06	1.867685E-07	0.022
(0001)	3	Hemlux	1.68367E-06	2.175566E-07	0.025
(0001)	1	Hemlite	3.05309E-06	4.535550E-07	0.046
(0001)	4	Hemcor	3.83489E-06	6.469233E-07	0.058
(0001)	2	Hemverneuil*	9.40220E-06	7.169225E-07	0.141

\*This sample had a severe localized lattice distortion, and it was included in the analysis.

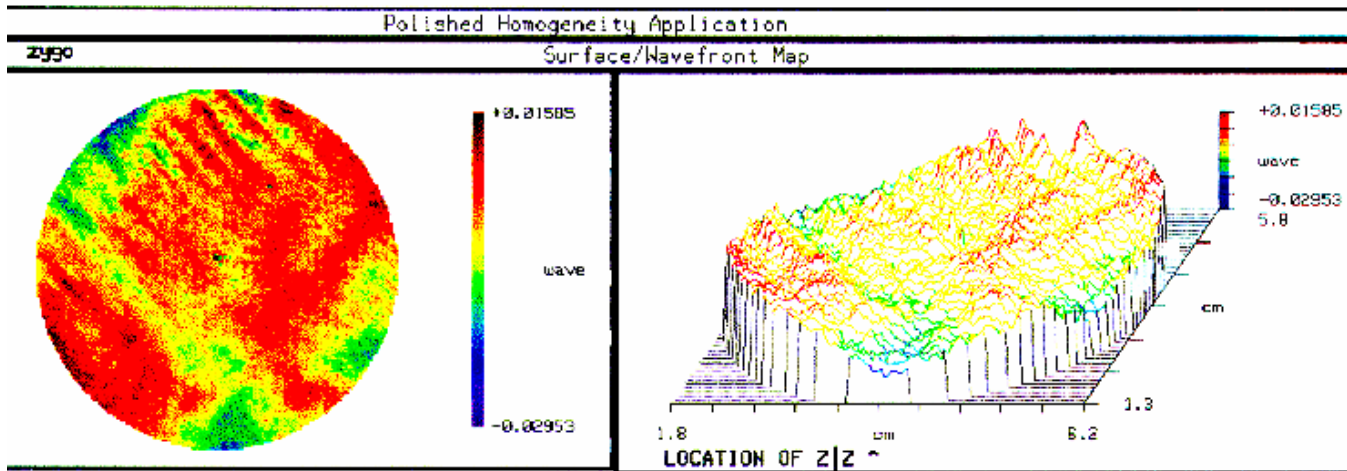
Representative interferograms for a Hemlite grade in ( $\bar{1}1\bar{2}0$ ) and (0001) orientation are shown in Figure 3. This data shows, as expected, that for highest grade, Hemex, the refractive index homogeneity exhibited minimal variation in the  $10^{-7}$  rms value (0.1 part per million, ppm) and 0.04 wave pv value. This data confirms that the homogeneity shows improvement with higher grades of sapphire and that the grades reflect quality of sapphire. Further, the various grades of lattice distortion have only small effect on the refractive index homogeneity of sapphire. This is illustrated by the fact that the Hemverneuil grade sample of (0001) orientation had a severe localized lattice distortion and, even including this severe distortion, the sample showed a homogeneity rms value of only  $7 \times 10^{-7}$  (0.7 ppm) and pv of 0.14 wave (see Figure 4). The test samples were also measured for transmission in the midwave infrared wavelength region. No measurable difference could be measured which could be attributed to orientation or effect of the grade of sapphire. A typical transmission curve is shown in Figure 5.



Homogeneity 3.05309E-06

Homogeneity RMS 4.535550E-07

PV 0.046 wave

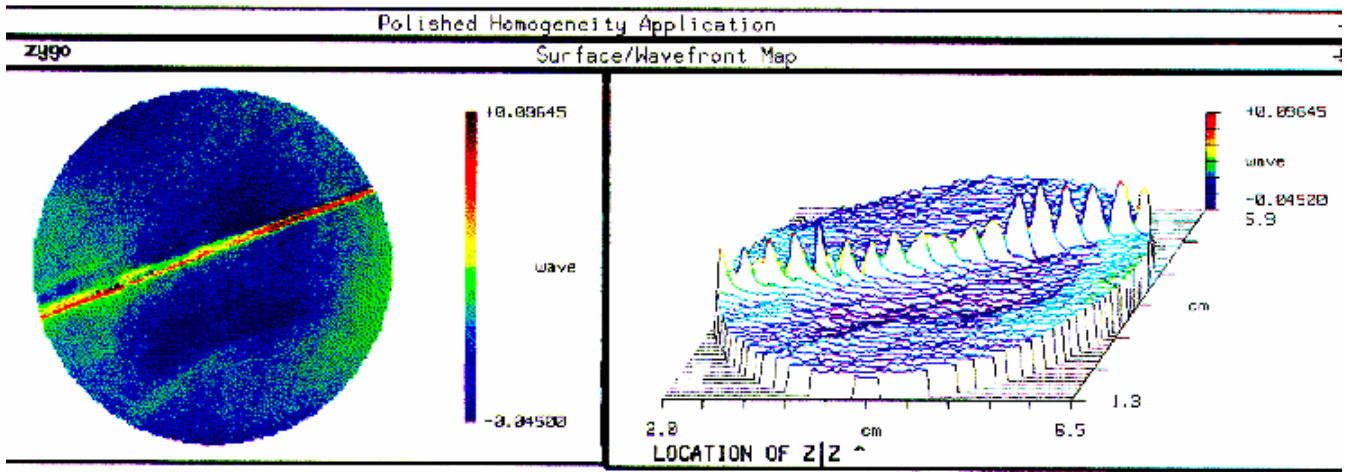


Homogeneity 3.05817E-06

Homogeneity RMS 3.660218E-07

PV 0.045 wave

Figure 3. Refractive index homogeneity of Hemlite grade sapphire with  $(\bar{1}120)$  (above) and  $(0001)$  (below) orientations



Homogeneity 9.40220E-06

Homogeneity RMS 7.169225E-07

PV 0.141 wave

Figure 4. Refractive index homogeneity for a Hemverneuil grade sapphire with a severe lattice distortion across the sample

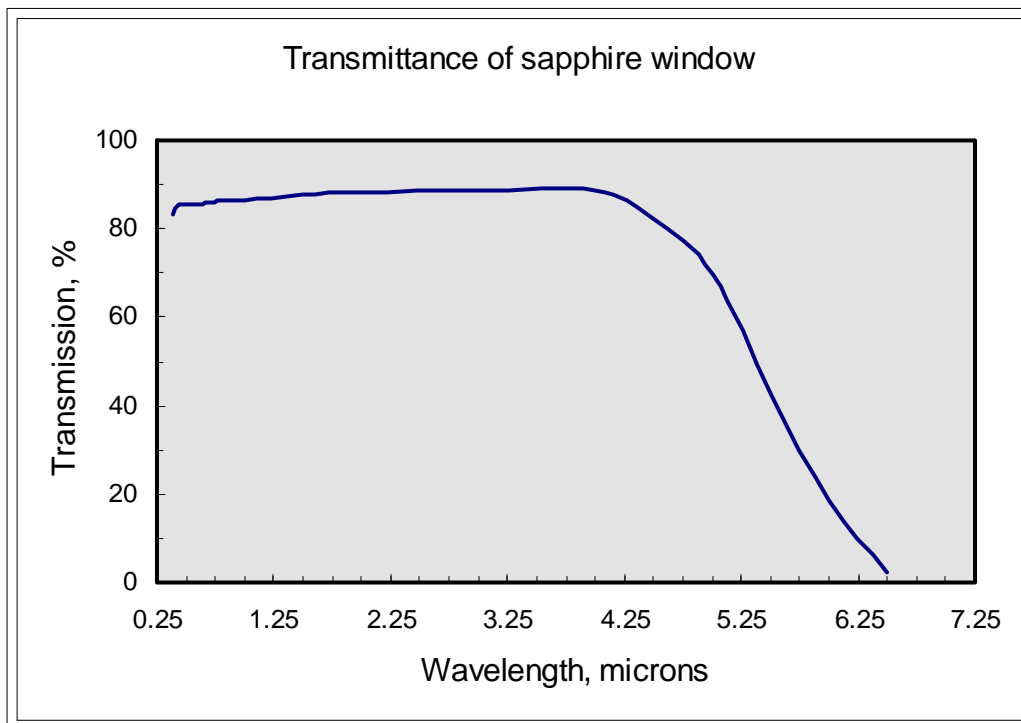


Figure 5. Typical transmission curve of a 3mm thick sapphire window

## 5. CONCLUSIONS

Large sapphire boules are currently produced by the HEM method to meet the requirements of next generation infrared optical systems requiring abrasion resistant windows. Qualitative analysis has been developed to characterize five grades of sapphire based on degree of lattice distortion and light scatter. Sapphire boules from production contain various grades. Therefore, large sapphire windows will contain more than one grade of sapphire. Lattice distortion in sapphire is most easily observed as deviation from symmetry when viewed along  $c$ -axis or (0001) orientation under cross polarizers. The most commonly used sapphire windows are of (11 $\bar{2}$ 0) or (0001) orientation. Samples were prepared in two orientations of five grades and characterized for refractive index homogeneity. This data showed that the refractive index homogeneity for all the samples was in the  $10^{-7}$  (0.1 ppm) range and the refractive index was more uniform for the higher grades of sapphire. The qualitative measurements correlated with the quantitative measurements of refractive index homogeneity. Based on this data, large sapphire windows containing various grades of sapphire could be used without significantly affecting the transmitted wavefront of large sapphire optics. The fact that lattice distortion does not have a large effect on transmitted wavefront allows fabrication of larger plates in production mode at low cost.

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