Observations during the fabrication of spinel optics

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ABSTRACT

Exotic Electro-Optics recently conducted a basic evaluation of state-of-the-art spinel material from the standpoint of optical fabrication. The goal of this study was to characterize the behavior of several spinel samples as they passed through a complete optical fabrication sequence. Overall, the material was found to be compatible with conventional fabrication processes. Methodologies used in the manufacture of heritage optical components were employed successfully, without significant modification, in the fabrication of the spinel windows. A standard anti-reflective coating was used to coat polished spinel samples. Good coating-to-substrate adhesion was observed and the coated optic exhibited the expected spectral performance. In this paper, Exotic Electro-Optics reports on the results of this work.

Keywords: spinel, coating, grinding, polishing, strength, transmittance, index, surface roughness

1. INTRODUCTION

Exotic Electro-Optics (EEO) recently completed a comprehensive material evaluation of state-of-the-art spinel obtained from three of the most prominent spinel material vendors. This study was done as part of an internal objective to determine the current state of the art in the marketplace for spinel optical material, especially pertaining to its applicability as a substrate for infrared windows and domes.

Overall, the spinel samples were found to be compatible with conventional optical fabrication techniques. EEO's experience with hard materials, such as sapphire and AlON, ensured that suitable tooling, machinery, abrasives, and experienced personnel were readily employed. A variety of methodologies used in the manufacture of heritage optical components were employed successfully, without significant modification, in the fabrication of the spinel optical samples.

Polished spinel samples were successfully coated with an antireflective coating normally used for sapphire windows. Preliminary results show the coating exhibited the expected spectral performance in the desired wavebands, with excellent coating-to-substrate adhesion

2. DESCRIPTION OF EVALUATION

All of the spinel samples underwent analogous optical fabrication processes in order to examine spinel processing characteristics and capability. EEO performed a battery of tests designed to evaluate specific characteristics of interest chosen in order to thoroughly characterize the spinel pieces from the standpoints of optical fabrication and ultimate performance. These characteristics included a visual inspection by trained opticians, scratch/dig inspection and surface mapping, surface roughness by optical and contact profilometry, spectral performance by spectrophotometry, strength by equibiaxial flexural testing, and index of refraction testing by ellipsometry.

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Joseph R. Bashe, EEO; Douglas L. Hibbard, EEO, "Observations during the fabrication of spinel optics," Window and Dome Technologies and Materials XI, Randal W. Tustison, Volume 7302, Page 7302OC, 2009

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2.1 Preliminary Inspection

The spinel samples were received in states varying from unprocessed raw blanks to material pre-polished for inspection. Generally, spinel is received in a raw, unprocessed state characterized by a rough, rippling surface which makes it impossible to inspect for inclusions or test its transmittance.

All incoming samples were measured for surface roughness. Photographs of each sample were taken in order to record the samples' general visual appearance. The samples were also measured for size. The size of the samples was chosen to be near 4" x 4" wide plates as this is an economical size for studying fabrication properties.

2.2 Fabrication

The spinel samples were blocked using a technique which minimized deformation of the samples and ensured a flat first side grind and polish. The samples were then ground using conventional grinding techniques, equipment, and abrasives. A succession of smaller grit sizes was used in order to minimize sub-surface damage from grinding. Numerous in-process inspections of surface quality and figure were made in order to confirm that the samples were proceeding acceptably throughout the process of optical fabrication.

Once the samples had been ground on the first side, the surface characteristics of the ground samples were evaluated. A TalySurf contact profilometer was used to measure the surface roughness and a differential interference contrast microscope was used to record the appearance of the surface microstructure. Visual inspections were performed by trained optical inspectors in order to find any material or process induced anomalies.

The samples were then polished using relatively conventional techniques. As this work proceeded, we refined our optical fabrication process. As with any material, spinel exhibits idiosyncrasies unique to the material which require specialized optical fabrication processes suiting that material.

The surface characteristics were examined once again after first-side polishing. Visual inspections, surface photomicrographs, and surface roughness by MicroXAM optical profilometry were all employed to create a detailed picture of the spinel samples.

Second side fabrication was initiated by optically contacting each of the parts to a glass contact block. The parts were then ground in a fabrication process similar to the first side process described above. Finally, the samples were polished in accordance with EEO standard practices and methods, and released for evaluation.

2.3 Post-fabrication Evaluation

After polishing, the samples' characteristics of interest were again examined. Visual inspections, photomicrography, optical profilometry, and spectrophotometry were all employed. This assisted us in the development of an understanding of the ultimate performance of these materials, as well as the establishment of a baseline from which to compare post-coating performance.

Each of the test specimens was then sliced into an equibiaxial strength sample. These samples were prepared and tested in accordance with ASTM C1149. Testing was performed by an outside test laboratory, University of Dayton Research Institute.

After slicing the strength samples, the remaining spinel pieces were sent to J.A. Woollam Co., Inc. for index of refraction testing. There we obtained a broad range of index of refraction data which, when combined with transmittance data and reflectance data, gives us a detailed picture of spinel optical properties from each vendor.

Joseph R. Bashe, EEO; Douglas L. Hibbard, EEO, "Observations during the fabrication of spinel optics," Window and Dome Technologies and Materials XI, Randal W. Tustison, Volume 7302, Page 7302OC, 2009

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3. DATA AND ANALYSIS

Data are presented chronologically in groups, retaining the organization of the preceding description of the overall evaluation effort.

3.1 Preliminary Inspection

As explained in the section *Description of Evaluation*, investigations began with visual inspections of the samples. Figure 1 and Figure 2 are representative photomicrographs depicting the difference in the visual appearance and surface characteristics of incoming materials.

The raw material in Figure 1 was of a very coarse texture and a high degree of irregularity. The inspection polished material, shown in Figure 2, allows incoming inspection of material inclusions and haze. In the photograph, multiple inclusions are evident throughout the bulk of the material. A light hazy appearance characterizes the material. This hazy appearance is quite common in spinel material.

Figure 1. Representative photograph of raw, unprocessed spinel material.

Figure 2. Representative photograph of inspection polished spinel material.

In Figure 3 below, a photomicrograph of the surface of the unprocessed material is given. This magnified view confirms the disorder and irregularity of the surface visible to the unaided eye. Surface photomicrographs of the inspection polished spinel material are shown in Figure 4. This photomicrograph shows a surface that is generally polished, though it still exhibits significant pitting.

Joseph R. Bashe, EEO; Douglas L. Hibbard, EEO, "Observations during the fabrication of spinel optics," Window and Dome Technologies and Materials XI, Randal W. Tustison, Volume 7302, Page 7302OC, 2009

http://dx.doi.org/10.1117/12.818205

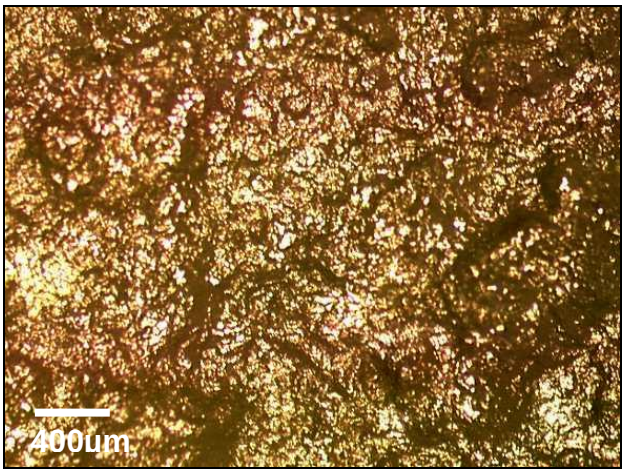

Figure 3. Representative photomicrograph of raw, unprocessed spinel material. 50X.

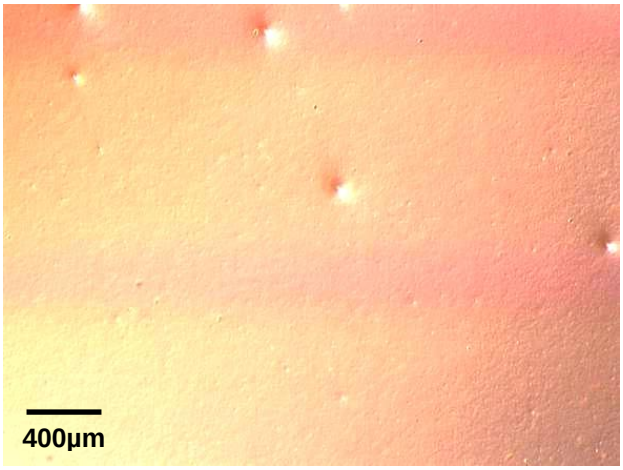

Figure 4. Representative photomicrograph of inspection polished spinel material. 50X.

Two of the inclusions seen in Figure 2 above are examined more closely in Figure 5 and Figure 6. In these figures, two separate examples of inclusions near the surface of the inspection polished spinel material are shown. These inclusions are quite large, the left measuring 0.02” in diameter and the right measuring over 0.06” when the outer halo is included. The chemical make-up of these inclusions is not known, but they are thought to be artifacts of the material consolidation process.

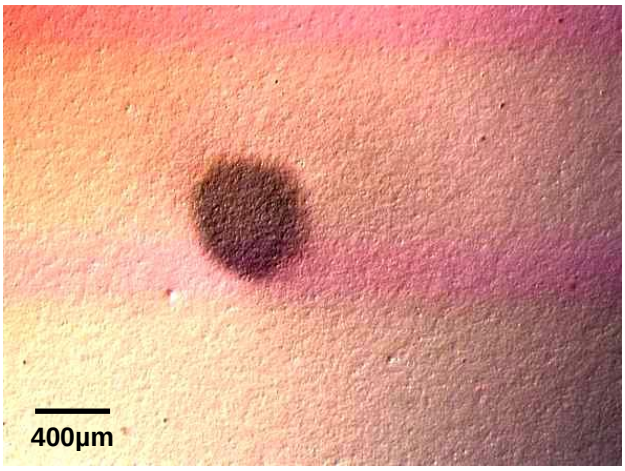

Figure 5. 0.02" diameter inclusion on the surface of inspection polished spinel sample. 50X.

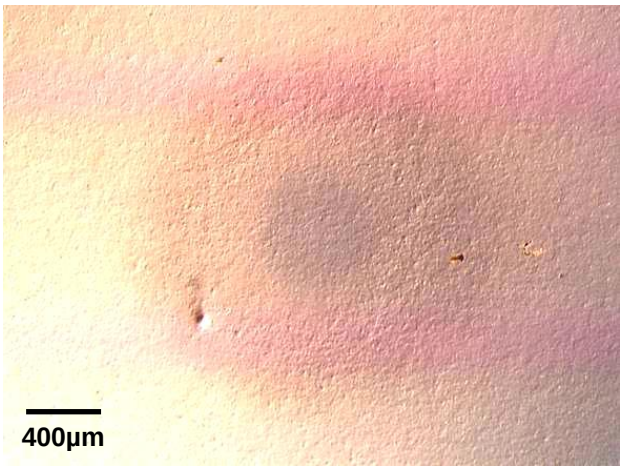

Figure 6. 0.06” inclusion on the surface of inspection polished sample. 50X.

Before processing, the mechanical dimensions of the samples were measured. The five samples ranged in size from 4.0” x 4.0” x 0.25” thick to 5.0” x 5.0” x 0.42” thick when received. The mechanical wedge across the raw samples was found to be anywhere from 0.013” to 0.078”, while the inspection polished sample only had 0.001” of wedge. Once preliminary evaluations were complete, optical fabrication of the samples began.

3.2 Fabrication

During fabrication we took these materials of varying initial quality and condition and subjected them to proprietary optical fabrication processes in order to examine their performance as deterministic, reliable optical substrates.

Joseph R. Bashe, EEO; Douglas L. Hibbard, EEO, “Observations during the fabrication of spinel optics,” Window and Dome Technologies and Materials XI, Randal W. Tustison, Volume 7302, Page 7302OC, 2009

http://dx.doi.org/10.1117/12.818205

Photomicrographs of the samples were taken after grinding in order to determine surface microstructure of the samples' starting point for polishing. Representative photomicrographs of the large-grained spinel surface are presented in Figure 7 and Figure 8. One can easily see grains in the material in Figure 7, and grains were also observed in Figure 8. In these large-grained samples, cracking at some of the grain boundaries was observed. These inter-granular crevices are removed by polishing though they can leave inter-granular pitting behind, as will be seen in Figure 25.

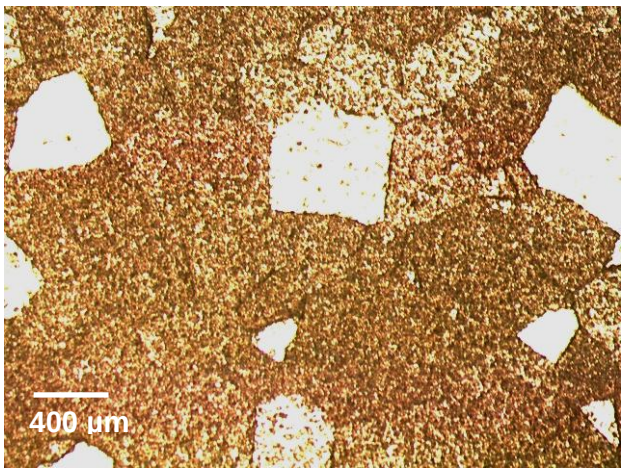

Figure 7. Spinel, ground. 50X.

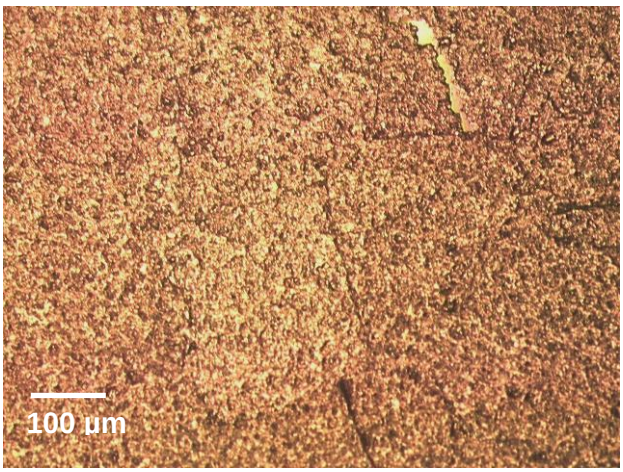

Figure 8. Spinel, ground, 200X.

Figure 9 is a representative photomicrograph demonstrating the appearance of a ground, small-grained spinel surface. This small-grained surface closely resembles the intra-granular spinel surfaces seen previously, or a single-crystalline ground surface such as sapphire.

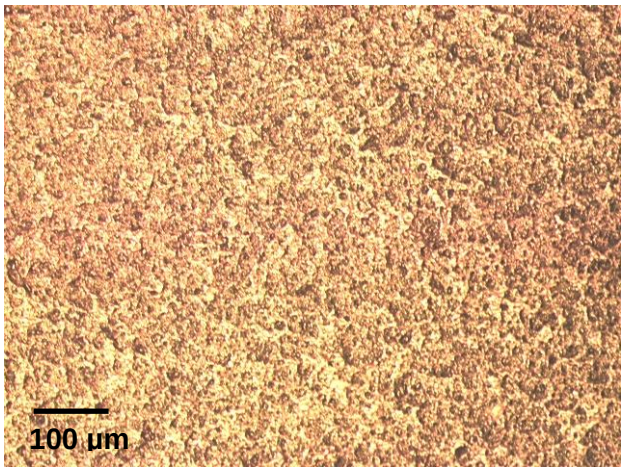

Figure 9. Spinel, ground, 200X. This shows surface morphology of a ground, small-grained spinel surface.

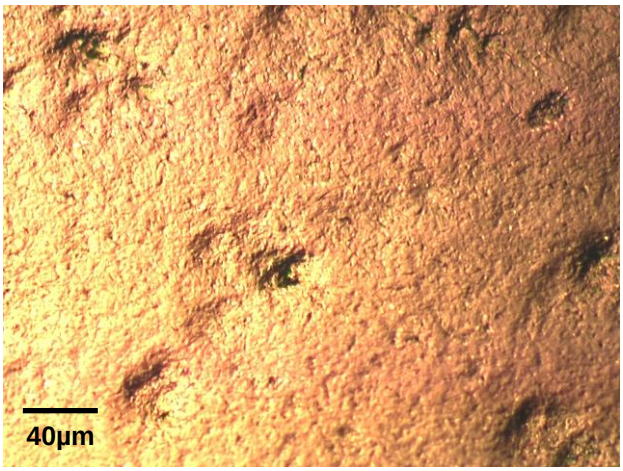

Figure 10. Spinel, ground. 500X. This representative photomicrograph shows the intra-granular surface morphology.

In Figure 10, a representative photomicrograph is given showing the appearance of a single grain. This grain is raised plateau seen at the center of Figure 7. It is apparent that the raised grain has been planarized more than the surrounding material. This shows that the granularity of spinel material affects its removal rate in grinding. Since the grain seen in Figure 10 was higher relative to the rest of the spinel surface, it follows that it has a lower removal rate that the

Joseph R. Bashe, EEO; Douglas L. Hibbard, EEO, "Observations during the fabrication of spinel optics," Window and Dome Technologies and Materials XI, Randal W. Tustison, Volume 7302, Page 7302OC, 2009

http://dx.doi.org/10.1117/12.818205

surrounding material. It is thought that the difference in removal rate from grain to grain depends upon the grain orientation. Certain facets of spinel granules yield more easily to grinding and, as we will see, polishing processes.

Visual inspections yielded no abnormal observations during the processing of the material. The spinel material was observed to grind and polish more quickly than sapphire material, in general. This accelerated the process of figure correction in comparison to sapphire.

3.3 Post-fabrication Evaluation

After polishing, the samples were visually inspected in order to determine the level of inclusions and haze in the bulk material. Surface photomicrographs were taken, and surface roughness and grain size were measured as well. The ultimate optical performance of the samples was measured by determining transmittance and index of refraction. Finally, the samples' strengths were evaluated by equibiaxial flexure testing.

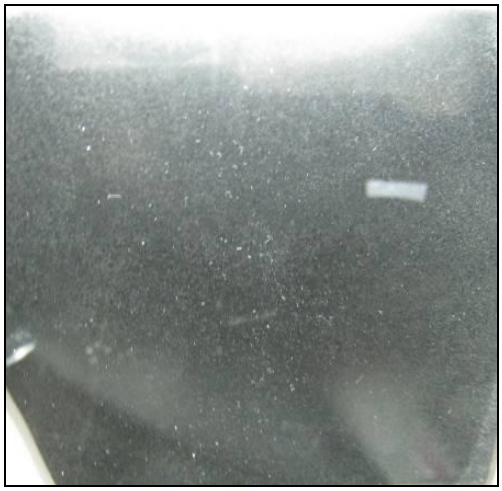

Figure 11. Photograph of polished spinel.

Figure 12. Photograph of polished spinel.

Figure 13. Photograph of polished spinel.

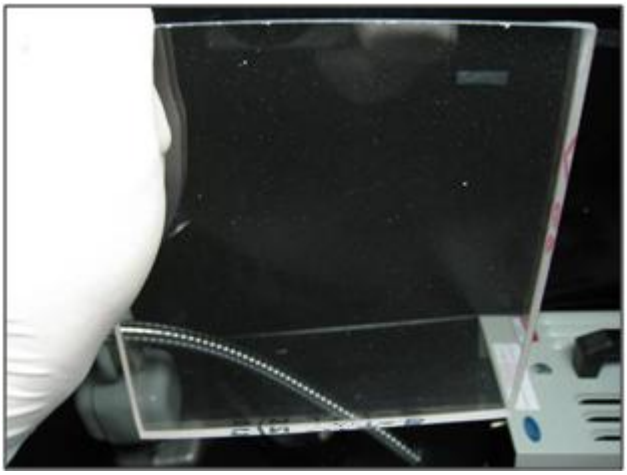

Figure 14. Polished spinel sample. This picture demonstrates that most haze is not visible unless inspecting closely under high intensity light.

Figure 11 through Figure 14 are representative photographs showing the visual appearance of the samples from all three vendors after polishing. In Figure 11 and Figure 13, the material is observed to be generally clear but has a speckled appearance attributable to both inclusions and granularity in the material. In Figure 12, the sample exhibits a less

Joseph R. Bashe, EEO; Douglas L. Hibbard, EEO, "Observations during the fabrication of spinel optics," Window and Dome Technologies and Materials XI, Randal W. Tustison, Volume 7302, Page 7302OC, 2009

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speckled, but hazy appearance. For reference, Figure 14 is given to show that only under intense lighting do all of the defects in the material become obvious, whereas under normal lighting some of the larger inclusions are visible but the material is quite transparent to the naked eye. The transmittance of these materials will be quantified in the *Optical Performance* results later in the section.

In Figure 15 and Figure 16, the representative photomicrographs of polished, larger-grained spinel show that the grain structure is readily discernable using a differential interference contrast microscope at 50X and 100X. Because spinel is polycrystalline, differently oriented grains in the material polish at different rates, causing the landscape of plateaus and ridges shown. This makes it easy to characterize the grain size. We will examine grain size more closely in the MicroXAM optical profilometer measurements.

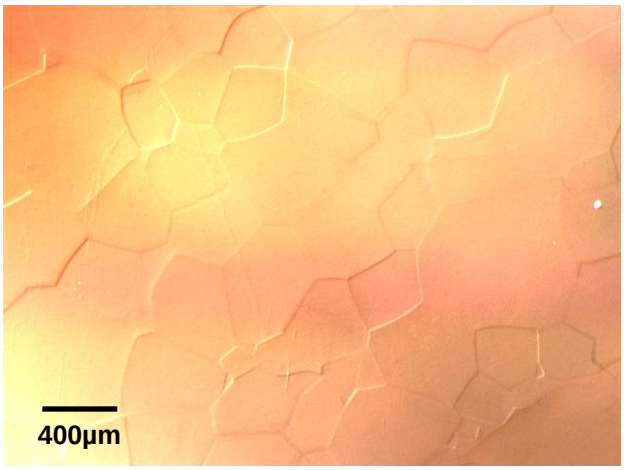

Figure 15. Larger-grained spinel, polished. 50X. Grain structure is plainly visible on the polished surface.

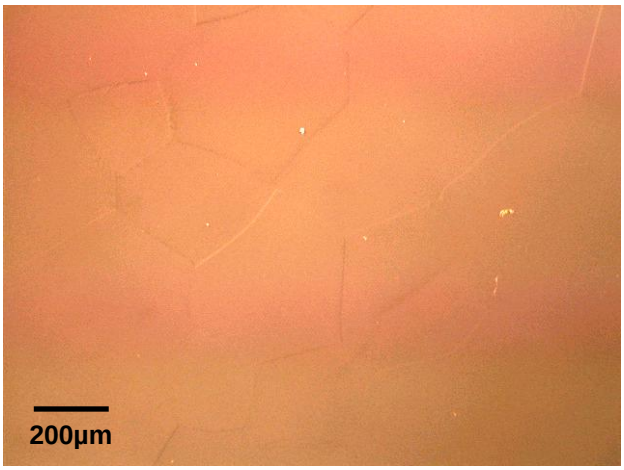

Figure 16. Larger-grained spinel, polished. 100X. Grain structure is slightly visible on the polished surface.

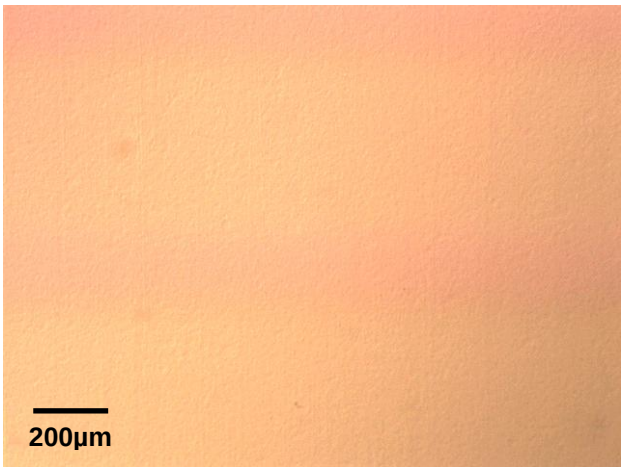

Figure 17. Smaller-grained spinel, polished. 100X. In this image, the grains are too small to resolve.

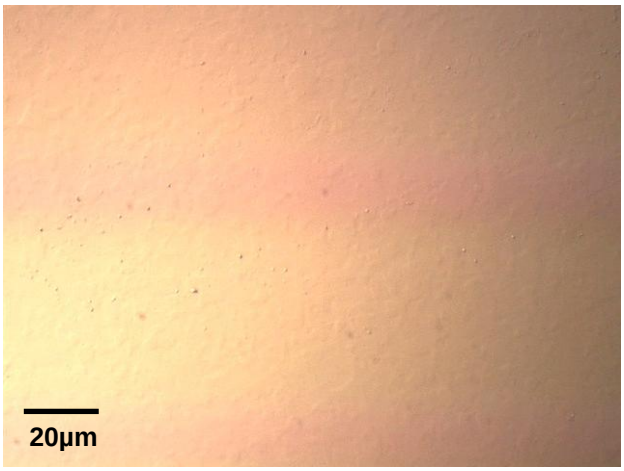

Figure 18. Photomicrograph of polished, smaller-grained spinel material. 1000X.

Conversely in Figure 17, it is not possible to see the individual grains in the spinel surface. This is due to the fact that the grains in this image are much smaller than those of Figure 15 and Figure 16. In the optical profilometer measurements of small-grained spinel surface roughness in Figure 23 and Figure 24, it is not possible to discern the grains in the smaller grained spinel sample. However, upon close inspection of Figure 18, grains on the order of 2 µm – 5 µm in diameter are visible (albeit with much difficulty).

Joseph R. Bashe, EEO; Douglas L. Hibbard, EEO, "Observations during the fabrication of spinel optics," Window and Dome Technologies and Materials XI, Randal W. Tustison, Volume 7302, Page 7302OC, 2009

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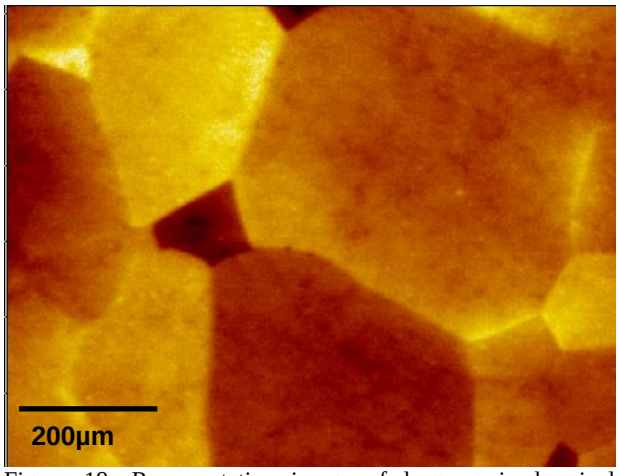

Figure 19. Representative image of larger-grained spinel sample, polished. 100X. 2D view.

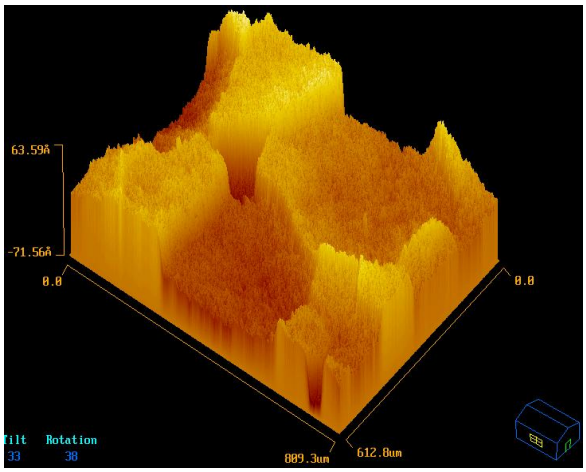

Figure 20. Spinel sample, polished. 100X. 3D view corresponds to Figure 19.

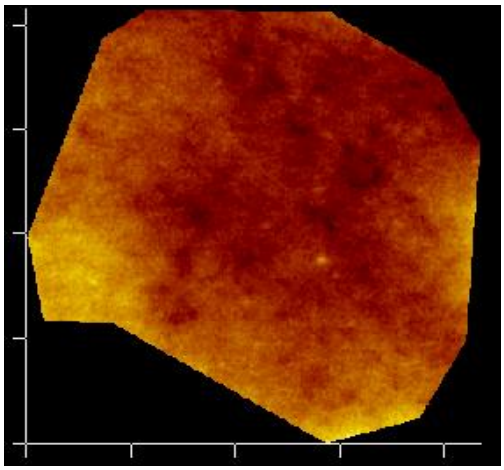

Figure 21. Spinel sample, polished. 100X. 2D view. All but a single grain has been masked. The dimensions of this grain are 416 µm X 436 µm.

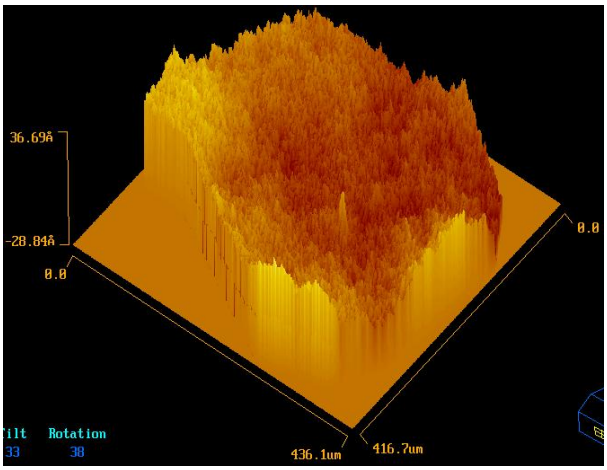

Figure 22. Spinel sample, polished. 100X. 3D view corresponds to Figure 21. All but a single grain has been masked.

From Figure 19 and Figure 20 above, it is evident that the larger-grained spinel material compromised final surface roughness. In these images produced by the MicroXAM optical profilometer, we can see that the steps from one grain to another are typically from 50 Å to 100 Å in height. These steps affect measurements of surface roughness in the polished state when the grain sizes are on the order of the area under measurement. Because of this, intra-granular surface roughness was also examined by masking all but one large grain. These data are given in Figure 21 and Figure 22. Within these intra-granular areas, we observed that the surface roughness is typical of polished glasses and crystals, as well as smaller-grained spinel. Quantitative data is provided in Table 1 at the end of the section. Further modification of the surface finishing process is expected to alleviate differential material removal rates and yield improvement in the overall surface roughness.

It may seem that in Figure 23 and Figure 24 that the samples do not contain grains. However, this is an illusion as Figure 18 shows. These grains were measured to be at approximately 2 µm – 5 µm in size, and thus are not readily discernable in the optical profilometer measurements shown in Figure 23 and Figure 24.

Joseph R. Bashe, EEO; Douglas L. Hibbard, EEO, "Observations during the fabrication of spinel optics," Window and Dome Technologies and Materials XI, Randal W. Tustison, Volume 7302, Page 7302OC, 2009

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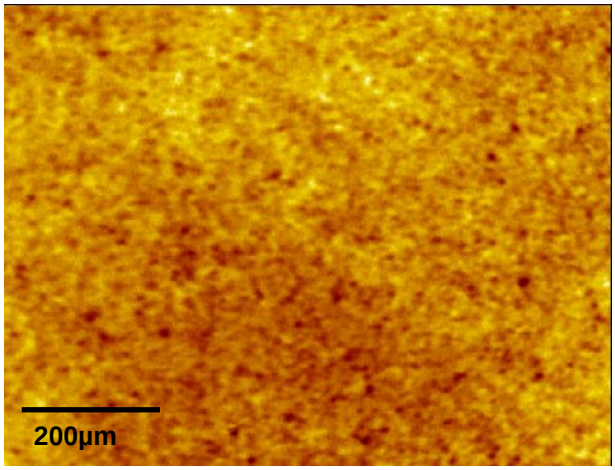

Figure 23. Spinel sample. 100X. Side 1, polished. 2D view.

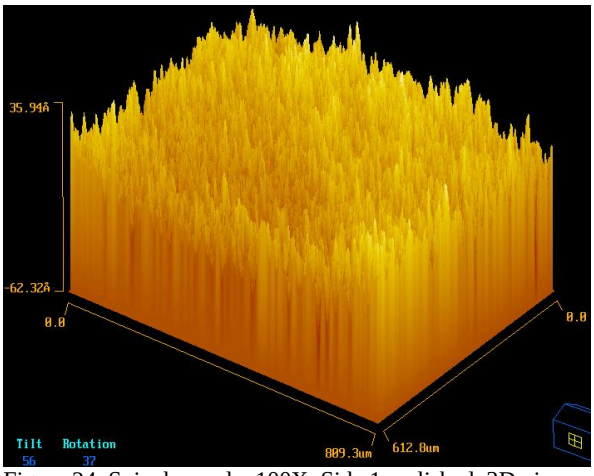

Figure 24. Spinel sample. 100X. Side 1, polished. 3D view.

Figure 25 shows another example of large-grained spinel material. In this example, pitting can be seen at the grain boundaries, apparent as dark spots along the boundary. This pitting is the remnant of the crevices observed after grinding. Figure 26, we see an inter-granular step height of approximately 110 Å.

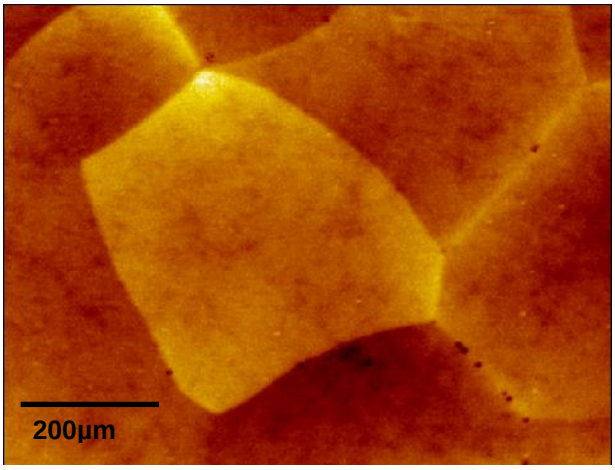

Figure 25. Spinel sample. 100X. 2D view.

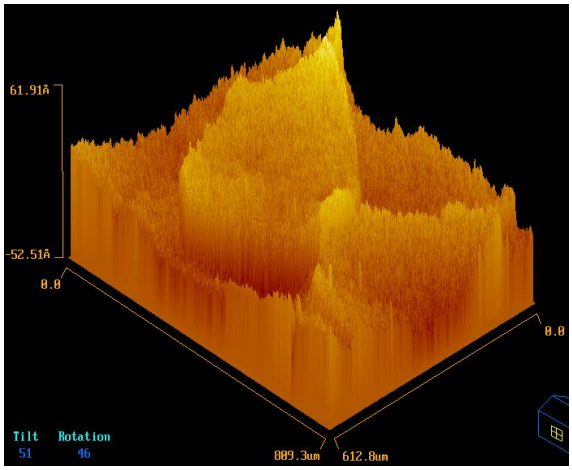

Figure 26. Spinel sample. 100X. 3D view.

Table 1 shows that spinel in the ground state exhibits high RMS and PV surface roughness, while the standard deviation of the measurements is approximately 10% from one spinel sample to the next. In polishing, the surface roughness has been reduced substantially, but at the expense of a high standard deviation from sample to sample. This is solely due to the fact that large-grained and small-grained materials were considered together. When we look only at the intra-granular surface roughness of the large-grained samples, we see that it is significantly lower in both magnitude and variance. These values are equivalent to the surface roughness found in small-grained and crystalline polished optical materials.

With these observations we have inspected the surface characteristics of polished spinel material. These results show that using relatively conventional processes, spinel can be polished to typical optical specifications.

Joseph R. Bashe, EEO; Douglas L. Hibbard, EEO, "Observations during the fabrication of spinel optics," Window and Dome Technologies and Materials XI, Randal W. Tustison, Volume 7302, Page 7302OC, 2009

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Measurements in Angstroms	Inter-granular				Intra-granular (masked data)
	Ground Spinel		Polished Spinel		Polished Spinel
	RMS	PV	RMS	PV	RMS
AVERAGE	6524	34507	28.61	138.76	11.05
STDEV	750	4283	13.73	112.88	2.74
STDEV %	11.5%	12.4%	48.0%	81.4%	24.8%
MIN	5626	29359	9.42	9.58	7.55
MAX	7352	39985	45.58	415.50	15.72

Table 1. Overall and intra-granular surface roughness of ground and polished spinel.

3.4 Strength Evaluation

Strength evaluations were carried out using equibiaxial flexure samples. These samples were used because they offer the clearest indication of bulk material strength. However due to the size of the sample required, we were only able to obtain one strength sample per sample of spinel. Thus we were unable to obtain a data set large enough to be statistically precise while remaining economical. However, the values obtained are in line with expectations as to the strength of spinel material, and are tabulated in Table 2.

	Equibiaxial Flexure Strength	
	(ksi)	(MPa)
MEAN	19.97	137.7
MIN	12.01	82.8
MAX	29.92	206.3
STDEV	7.60	52.4
STDEV %	38%	

Table 2. Spinel equibiaxial flexure strength data.

3.5 Optical Performance

The optical performance of polished spinel was of particular interest. Spinel is widely known to have excellent transmission in the 3 – 5 µm wavelength band. After polishing, each spinel sample was examined for transmittance from 400 nm to 7000 nm. Although the spinel samples were finished to different thicknesses, transmittance values were normalized to theoretical transmittance at 0.25" thickness before averaging the values from each sample together to get the curve of average spinel transmittance. The average transmittance of the spinel samples is given in Figure 27.

Joseph R. Bashe, EEO; Douglas L. Hibbard, EEO, "Observations during the fabrication of spinel optics," Window and Dome Technologies and Materials XI, Randal W. Tustison, Volume 7302, Page 7302OC, 2009

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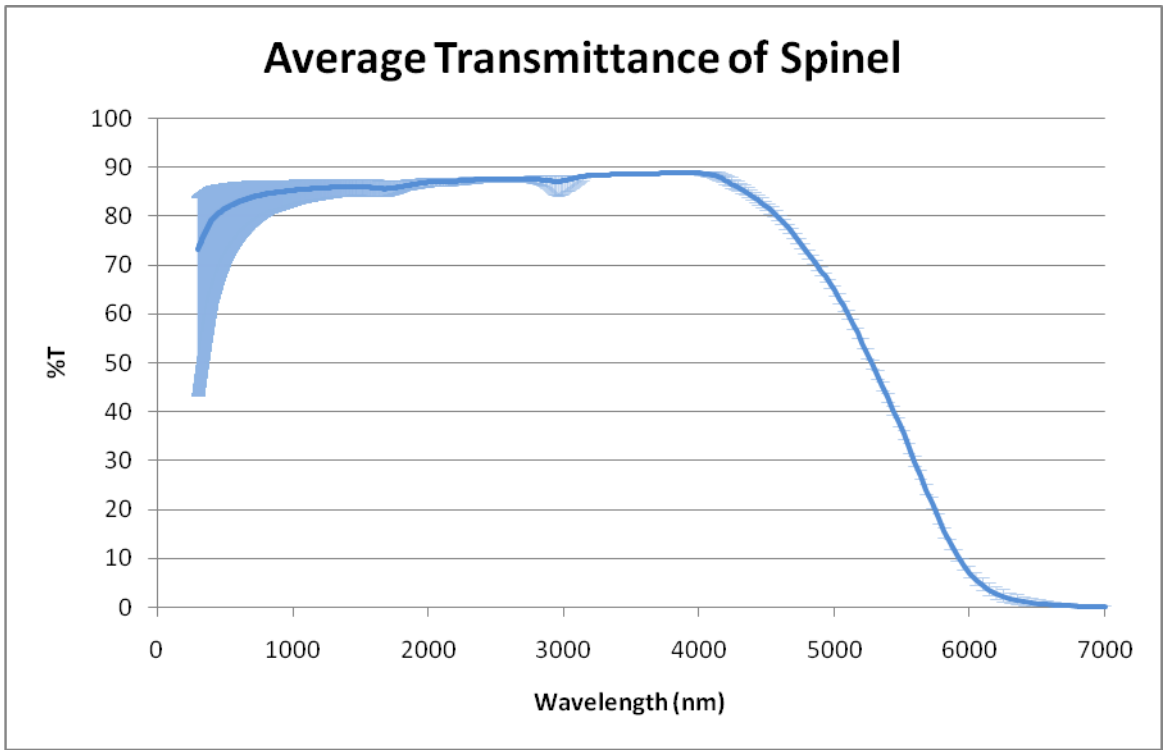

Figure 27. Average transmittance of spinel samples. Error bars represent maximum and minimum values measured. These values have been normalized to represent transmittance at 0.25" thickness.

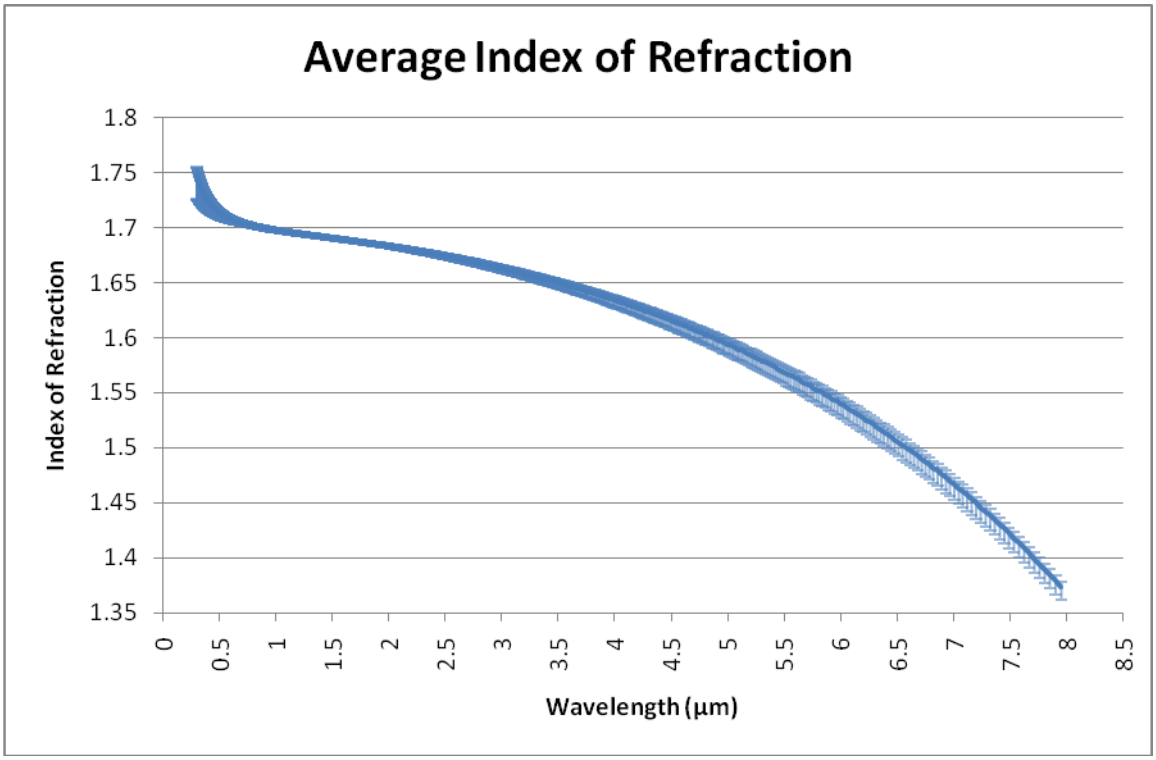

Figure 28. Average index of refraction of spinel samples. Error bars represent maximum and minimum values measured.

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Spinel sample pieces from each vendor were sent to J.A. Woollam Co., Inc. for index testing. Refractive index was determined using spectroscopic ellipsometery over the wavelength range from 0.3 µm to 8.0 µm. An M-2000DI was used to collect data from 0.3 µm to 1.7 µm and an IR-VASE was used to collect data from 1.7 µm to 8.0 µm. These data are shown in Figure 28.

3.6 Coating

Spinel samples were coated using a standard AR coating developed by EEO for sapphire. This coating is optimized for sapphire windows over a particular set of wavebands. The coating was chosen for this demonstration due to spinel's similarity in refractive index and coefficient of thermal expansion when compared with sapphire. With further development, a coating can be created to compliment spinel material more fully. The main purpose of this trial was to allay concern that coating-to-substrate adhesion could be a problem. Several photomicrographs are presented in order to show the superior adhesion achieved with this un-optimized coating. Visual inspections yielded no differences in surface appearance after coating. No severe environmental testing was performed.

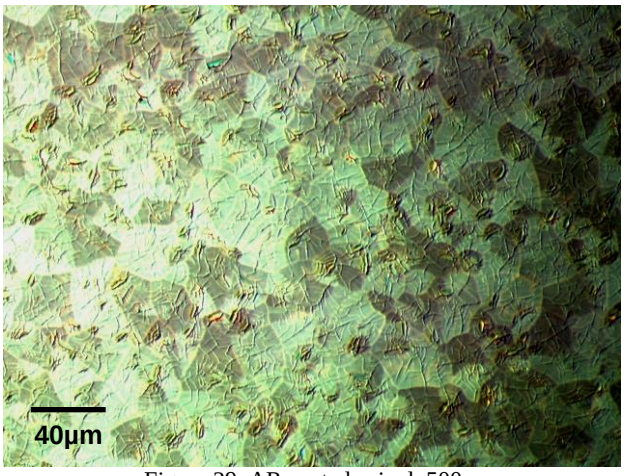

Figure 29. AR coated spinel, 500x.

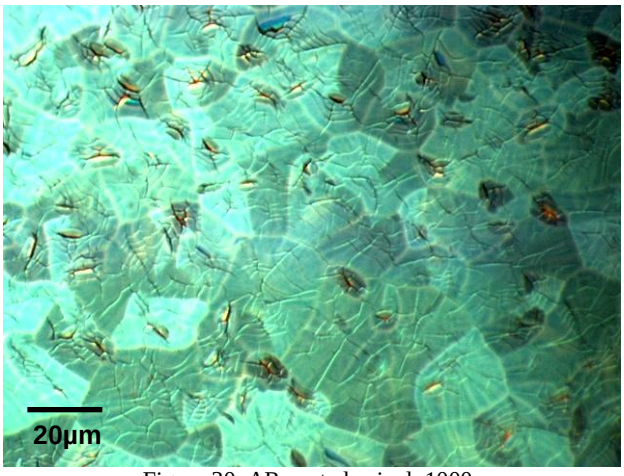

Figure 30. AR coated spinel, 1000x

4. CONCLUSION

EEO has determined that spinel is a viable substrate material for optical components including domes and window panels. Its strength makes it competitive with materials such as AlON, while the fact that it is softer and more compliant than sapphire makes it easier to machine, grind, and polish. Furthermore, it is evident that spinel material's transmittance is of higher performance than that of AlON or sapphire at the far end of the 3 µm – 5 µm band. An additional advantage of spinel is that it can be made near-net shape, making spinel an attractive material for domes and other non-planar optical components. Finally, spinel is a polycrystalline material meaning that it is optically isotropic. All of these characteristics make spinel an attractive, viable optical substrate for both windows and domes. The goal of this study was to characterize the behavior of spinel through typical optical fabrication processes. We were successful in employing conventional optical manufacturing techniques in order to fabricate spinel optics, and were able to obtain a large set of data including visual inspections, surface roughness, grain size, transmittance, strength, index of refraction, and coating characteristics.

5. ACKNOWLEDGEMENTS

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