Recent Advances in Spinel Optical Ceramic

Thomas J. Mroz[a], Thomas M. Hartnett[b], Joseph M. Wahl[b], Lee M. Goldman[b], James Kirsch[c], William R. Lindberg[d]
[a]Surmet Ceramics Corporation, 699 Hertel Ave., Buffalo, NY 14207
[b]Surmet Corporation, 33 B Street, Burlington, MA 01803
[c]U.S. Army Research, Development and Engineering Command AMSRD-AMR-WS-PL, Redstone Arsenal, Alabama 35898-5000
[d]U.S Army Armament Research, Development and Engineering Command AMSRD-AMR-SE-MT, Redstone Arsenal, Alabama 35898-5000

ABSTRACT

New military requirements have reinvigorated the need for transparent magnesium aluminate ($MgAl_2O_4$) spinel. Surmet has developed a process that yields high quality transparent spinel at production scale. Several issues related to the extreme requirements of processing ultrafine spinel powders are described. Transmission data is presented for a significant dataset of parts made by this process.

More recently, the process has been expanded to include a capability for producing domes for the Joint Common Missile program. Domes at nominal 6" and 7" diameter have been successfully fabricated. Despite early challenges related to the forming portion of the process, a repeatable capability for these domes has been demonstrated.

Several challenges remain in spinel processing in order to support additional military requirements. In particular, the strength of the material needs further improvement. Also, improvements in optical quality with regard to inclusions are needed.

Keywords: spinel, MWIR windows, lenses, domes

1.0 INTRODUCTION

Future infrared sensors for missiles, unmanned aerial vehicles (UAV's) and other surveillance systems will require excellent optical transmission in the 0.5 to 5.0 µm range, refractive index homogeneity, and low scatter, all at an affordable price. Typical material requirements for windows and domes in these applications are:

- Excellent optical transmission (>80%) in the waveband of interest
- Refractive index homogeneity
- Low optical scatter
- High thermal shock resistance
- Low absorption/emission
- Low transmitted wave-front distortion (i.e., low dn/dT, high thermal conductivity)
- Low cost

Sapphire, introduced for these applications almost 30 years ago, is still the state-of-the-art material for windows and domes for applications in the visible to mid-wavelength infrared (MWIR) spectrum, where demanding optical, physical and environmental conditions exist. However, sapphire is expensive, and its an-isotropic mechanical properties make it difficult to fabricate. Consequently, sapphire is too expensive for many applications. The ideal material should combine the environmental durability of sapphire, with improved optical properties at a lower cost than sapphire.

tmroz@surmet.com; Phone: (716) 875-4091; FAX (716) 875-0106

Magnesium aluminate ($MgAl_2O_4$) spinel is such a material. It has a cubic structure, so it is transparent in polycrystalline form. Consequently, it can be made by conventional powder processing techniques into near net shaped dome blanks. Furthermore, its isotropic mechanical properties mean that it can be optically fabricated at a fraction of the cost of sapphire. The result is a material with better optical properties and the potential to be produced at a much lower cost than sapphire.

By comparison, ALON™ optical ceramic is a material very similar to spinel in that it is a transparent polycrystalline ceramic material with optical and mechanical properties similar to sapphire. ALON™ is inferior to spinel only in that it's MWIR cut-off is even shorter than that of sapphire. This consideration makes spinel preferable to ALON™ for applications where extended MWIR transmission is critical.

Polycrystalline transparent spinel has been developed over the last 30 years and the properties have been evaluated. Good optical material has been available in small quantities in the form of domes less than about 7" diameter and plates having dimensions less than about 11" x 11".(1) To date cost effective production processes for transparent spinel domes and windows have yet to be developed.

Based on our production experience with Aluminum Oxynitride (ALON™) optical ceramic and R&D experience with spinel, Surmet initiated a process development program for spinel in late 2002, with successful results transitioned to a production capability by early 2004. Additionally, fabrication methods have been developed by Surmet to form windows, optics and domes from processed transparent billets. Because of the isotropic nature of the material, as well as the somewhat lower hardness, fabrication costs are significantly lower for spinel as compared to sapphire.

The following provides a brief update on the state of the art of spinel production at Surmet Corporation.

2.0 STATUS DISCUSSION

2.1 Basic Spinel Processing

Spinel components are fabricated by typical ceramic processing methodology, starting with phase pure spinel powder. In order to facilitate the sinterability of the material, the powder must be very fine; less than 0.5 um in particle diameter, and have high surface area; typically higher than 15 m^2/gm. Such very fine powders are difficult to process. They tend to have very low bulk density and are difficult to disperse in a liquid medium to facilitate forming processes. Figure 1 provides and illustration of this point. The bag at the rear of the photo contains 2.2 Kg of spinel powder, at a density of about 8% of the theoretical density of spinel. Of course, the final spinel component must be 100% dense in order to be transparent. Therefore, the fabrication process must facilitate a consolidation factor of 12.5 times in order to successfully support optical applications. In comparison, a fabrication process that utilizes aluminum oxide exhibiting a more reasonable powder density of 20% requires a consolidation factor of only 5 times to result in a suitably dense component.

Figure 1: Representations of spinel at various steps in the fabrication process

The first step in spinel powder processing is to spray dry the powder into a denser, more flowable powder that contains organic processing aids. Figure 1 shows the relative amount of water (in the beakers) that must disperse all of the 2.2 Kg of powder in order to facilitate the spray drying process. Very careful selection of organic additives and mixing methodology are required to successfully disperse this quantity of powder while retaining sufficient fluidity of the resulting slurry. For comparison, 2.2 Kg of spray dried powder are shown in the container in the center of Figure 1. At this point, the bulk density of the material has increased to about 25% of the theoretical density of spinel.

Following spray drying, the powder is molded and pressed, most typically using isostatic pressure. This method of fabrication is suitable for small to moderate numbers of components with sizes ranging from small coupons to large plates, and geometries that can include tubes or even domes. Careful control of the process is required on one hand to provide sufficient densification to minimize the shrinkage (and related stresses) that occur in later thermal processes, while ensuring that components are not stressed too far to crack upon release or handling. Figure 1 additionally shows a 2.2 Kg pressed billet at a density of approximately 55% of theoretical.

Pressed parts must be prefired to remove the organic additives prior to sintering them to high density. This process step is similarly affected by the fine nature of the powder. In order to remove the organic additives as gaseous species, it is imperative to carefully control the heating rate at points in the cycle where they begin to decompose from solids into gas. Improper care can result in a rapid increase in pressure within the part as these gasses struggle to escape the part through very small pore channels between particles. Incorrectly done, this process can fracture a pressed part into many pieces and eliminate any chance of future use of the material. Because of the required level of control, this process step requires the longest cycle time of any process step for spinel.

The sintering process step follows debinding, and must densify the part up to at least 95%. Despite removal of organics in the prior processing step, a relatively long cycle is still required for sintering in order to remove any residual organics, as well as adsorbed water bound to the very high surface area particles. Additionally, the thermal cycles need to be controlled in order to minimize the opportunity for thermal shock, as the thermal conductivity of the material is relatively low compared to most ceramic materials.

A suitably sintered spinel component exhibits fully closed porosity in order to facilitate the final thermal processing cycle required to achieve transparency. Hot isostatic pressing (HIP) is used to pressurize the parts during a thermal cycle at temperatures similar to the sintering temperature. Under these conditions, the residual porosity is forced to shrink and ultimately exit the component along grain boundaries as the material further densifies. The effect of residual porosity on transparency is striking. Parts sintered even to 99.9% density are only slightly transparent to transmitted light. By removing the final 0.1% of porosity via HIP, the parts will convert to a fully transparent material.

2.2 Spinel Lenses

In late 2002, Surmet was approached by a military components producer with the request to develop a spinel fabrication capability suitable to produce a nominal 5” diameter lens for a MWIR – visible optical application. Primary spinel processing development was performed at Surmet throughout 2003, with increasingly good results obtained in the second half of that year. Multiple test blanks meeting suitable transmission properties for the intended application were produced late in the year, with reproductions demonstrated early in 2004. Figure 1 shows several nominal 5” spinel blanks at 0.5” thick produced in this timeframe.

Production repetition of parts commenced in Q2 of 2004 and continued throughout the remainder of the year. A standard set of procedures was developed based on the prior development process. Typical production rates have been in the range of 6-12 blanks per week. The process has been demonstrated to provide a theoretical throughput of 20 blanks/week, limited at this point by the size of the thermal processing equipment employed. Critical portions of the process have been demonstrated with larger equipment, ensuring that throughput can be increased as needed with no concern over production capability.

As of this writing, 65 blanks have completed processing and have subsequently been machined and evaluated. Table I provides a summary of the transmission obtained on these parts. It can be seen that the transmission of the material is quite good from mid-range IR through the visible, and the performance variation is suitable to support production purposes.

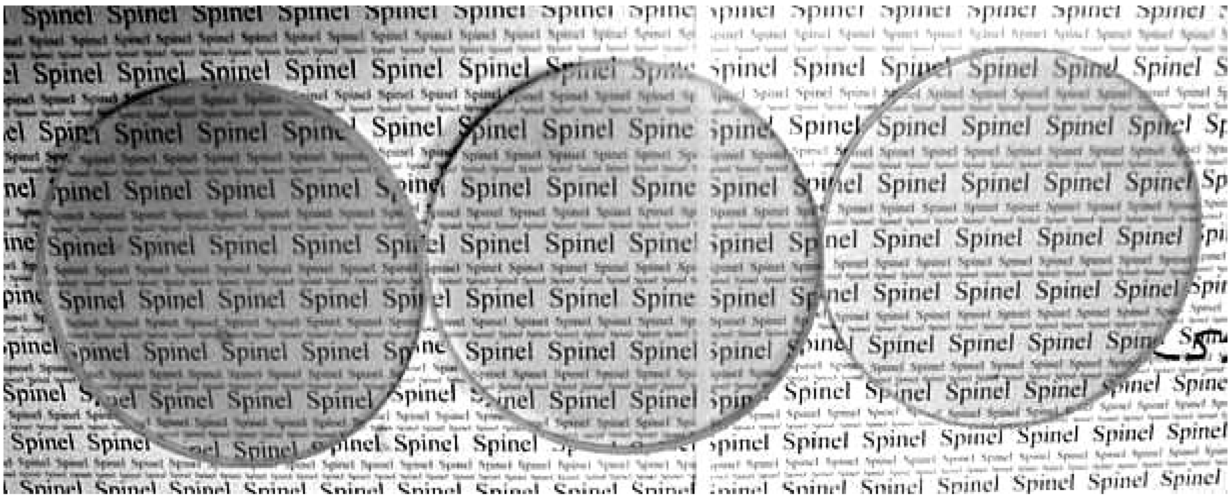

Figure 2: Spinel lens blanks produced during development efforts

Meanwhile, in 2004, Surmet developed the process operations and capability to final finish these spinel blanks into lenses meeting the physical and optical requirements of the customer. The majority of the material delivered to the customer has thus been in the final lens form. In this manner, Surmet has successfully provided a “one-stop” source for this lens design for the customer, integrating the materials processing and component machining into one fluid process flow. Figure 3 shows a finished lens created in this manner.

Figure 3: Spinel lens produced using production process

Table 1: Transmission and Haze Measurements of Spinel Lens Blanks

	Transmission			Haze
	% @ 0.65 um	% @ 1.064 um	% @ 4.5 um	(%)
Average	79	84	82	5.1
St. Dev	1.4	0.7	0.6	1.3
# of Samples	65	65	59	65

2.3 Spinel Domes

Surmet was awarded a Phase I SBIR program that started very early in 2004, with the primary goal of developing a process for producing spinel domes. The goal of the program was a 6" dome with 150^{o} of included angle for the Joint Common Missile program.

Our primary development effort centered on the available isopressing fabrication method and thermal processing steps previously developed for the MWIR - visible lenses. As can be expected, the tooling required to fabricate a dome shape by isopressing is quite a bit more sophisticated than a plate mold, given that the molding must locate both an internal as well as external surface. A number of test iterations were required in mold design in order to successfully create unfired parts suitable for thermal processing.

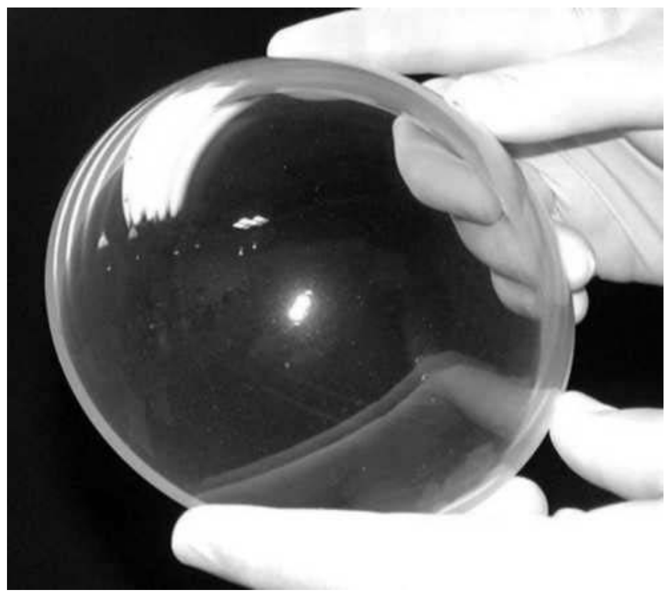

Figure 4: A finished 6" spinel dome

A nominal 6" was successfully fabricated early in the development program. Aside from some consideration of fixturing methods, the thermal processing of these domes represents little additional challenge compared to the flat billets used for producing lenses. This dome blank was further generated, ground and machined into an excellent quality transparent dome at Surmet Precision Optics in California. Figure 4 is a photo of this completed dome.

Late in the Phase I portion of this program, the overall JCM contract was awarded to Lockheed Martin. With Lockheed's formal entry to the program, design requirements for the dome changed in a number of ways. The overall dome diameter increased to 7". More importantly, the requirement for introduction of a EMS screen within the dome structure added a significant degree of difficulty to the overall design.

Another development cycle for isopressing was performed throughout the final 4 months of 2004, primarily under Phase I option funding. Despite the seemingly small increase of 1" in diameter, significant further improvement was required in the mold design and pressing methodology to result in good quality unfired domes with reasonable consistency.

An additional complication was encountered with the requirement for the screen structure within the dome. The most straight-forward means of providing this feature was determined to be bonding two dome shells together with the screen located in the center. This, however, required the preparation of 2 dome shells for each final dome. Given the time constraints, it was decided to produce a sufficiently thick dome to yield either an inner or outer dome shell from the same blank rather than produce two different mold designs. While this simplified design requirements, time and tooling costs, the thicker dome blanks further increased the difficulty of pressing, thus requiring additional process development.

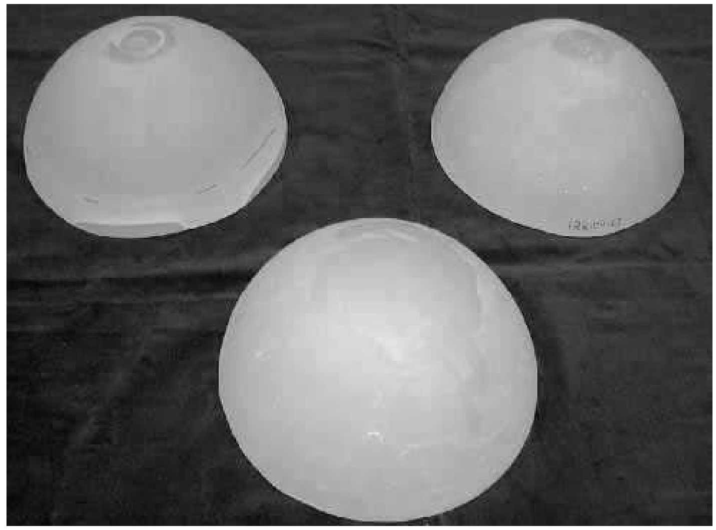

Figure 5: Several 7” spinel dome blanks for JCM

Success in this area was demonstrated in late 2004. Early in 2005, a number of good quality dome blanks have been produced and forwarded to Surmet Precision Optics for generation and finishing. Figure 5 shows a number of these dome blanks ready to be generated. Fully polished dome shells should be completed by the time this paper is presented in late March of 2005.

3.0 ANTICIPATED FUTURE WORK

3.1 Process Capability Scale-up

By the presentation date of this paper, Surmet expects to be under contract with the U.S Air Force to scale-up its spinel fabrication capability to yield large panels for the Joint Strike Fighter (JSF). Ultimately, panels 12” x 18” are desired at the end of the development program. The development sequence will demonstrate several sizes as part of the scale-up, including 9” x 9” and 12” x 12” as well as the final 12” x 18” size. Similar to the dome development, it is anticipated that the forming process will require several iterations of development in order to yield crack-free unfired bodies. By stepping through several panel sizes as part of the development, it is anticipated that powder and process requirements will continuously improve to a point sufficiently suitable for forming the largest panel.

Of course, additional issues will come into play, including handling requirements for the larger panels prior to firing, and fixturing of parts during sintering. In both cases, the significantly higher weight of the panels will add complications and thus, additional process improvements will be necessary.

On the other hand, Surmet Precision Optics, Murrieta, CA has already demonstrated a capability for finishing panels of this size and even larger from ALON™ optical ceramic. Thus, it is expected that with successful spinel billet process development, the means to convert the billet to a final window with wavefront surface quality will be relatively straightforward.

3.2 Strength/Microstructure Improvements

As part of its ongoing spinel development in 2005, Surmet anticipates spending considerable time improving the strength of spinel in support of the JSF application. The current manufacturing process for spinel results in components exhibiting a bimodal grain size distribution, such as that shown in Figure 6. While this microstructure is sufficient to yield suitable optical properties, the large grains are stress concentrators and are expected to limit the strength of the overall material. Improvements to the process, primarily through limitation of time and temperature, are expected to control the growth of the large grains and thus maintain a more monomodal, fine-grained microstructure as typically exhibited by advanced structural ceramics such as aluminum oxide or zirconium dioxide. The technical challenge will be to facilitate this microstructural improvement while maintaining the excellent optical properties of the material.

In addition to microstructural control, Surmet expects to further improve the strength of spinel by controlling the method of surface finishing. Similar methods have recently been shown to significantly improve the strength of ALON without any microstructural improvement (See "Characterization of ALON^{TM} Optical Ceramic", T. M. Hartnett, et al. in these proceedings for further details). By limiting the amount of damage induced during machining and finishing the material, bulk strengths can be significantly improved in the ceramic.

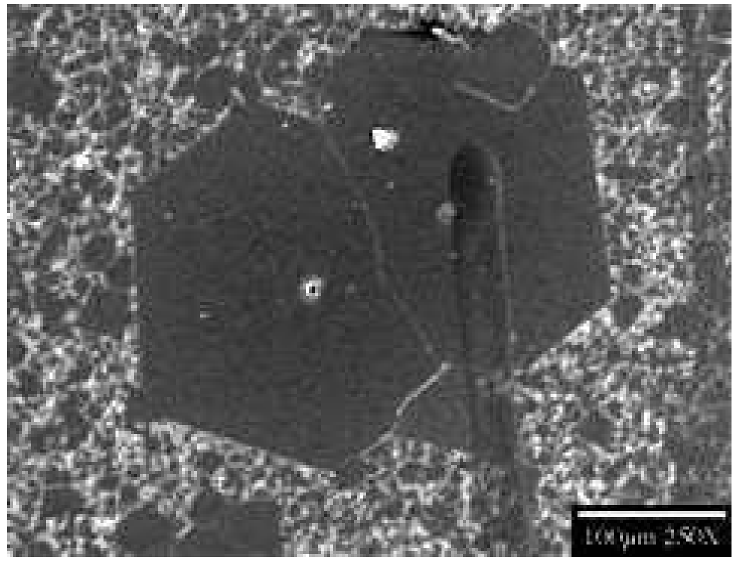

Figure 6. Typical spinel microstructure showing large grains

3.3 Inclusion Removal

An additional area of process development involves improving the optical clarity of the ceramic. Current spinel materials, while exhibiting excellent transmission and low haze, are affected by periodic inclusions in the range of 10 – several hundred microns in diameter. Macroscopically, these inclusions appear as black spots within the material. Microscopically, they exhibit a structure of hundreds of very fine pores in a tight cluster surrounding a central, larger pore. The structure suggests some form of contaminant that disperses into the material structure at some point during the thermal process, leaving behind the pores as light scattering defects. These inclusions have the obvious impact on the visual quality of the material, and may also limit the strength of the material in cases where they represent a large flaw in a stress field. Efforts to limit these inclusions will include improved process and handling cleanliness, as well as further control of the starting powder and associated processing additives.

4.0 CONCLUSIONS

The current capabilities of Surmet's spinel fabrication process have been described. The ability to produce flats, lenses and even domes has been demonstrated. A significant number of lenses have been produced to date, and the related optical performance shows excellent quality and consistency.

The opportunity to further increase the capability for producing spinel windows to large size, such as 12" x 12" or even 12" x 18" exists, though additional process development will be required in order to consolidate and handle such large parts while using these ultrafine powders. Additionally, further improvements in the material strength, as well as limitation of optical defects such as inclusions will be needed in order to take full advantage of spinel in suitable applications.

ACKNOWLEDGEMENTS

Portions of the described development work was performed under contract #W31P4Q-05-C-R028 with the U.S. Army Aviation and Missile Command Research, Development , and Engineering Center.

REFERENCES

1. M.C.L.Patterson, A.A. DiGiovanni, L. Fehrenbacher, and D.W. Roy, "Spinel: Gaining Momentum in Optical Applications", Window and Dome Technologies VII, R. Tustison Editor, Proceedings of SPIE Vol. 5078 pp 71-89 (2003).