Report Documentation Page

Form Approved
OMB No. 0704-0188

1. REPORT DATE **2005**	2. REPORT TYPE	3. DATES COVERED **00-00-2005 to 00-00-2005**
4. TITLE AND SUBTITLE **Transparent Ceramics: Magnesium Aluminate Spinel**		5a. CONTRACT NUMBER 5b. GRANT NUMBER 5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S)		5d. PROJECT NUMBER 5e. TASK NUMBER 5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) **Naval Research Laboratory,Optical Sciences Division,4555 Overlook Avenue SW,Washington,DC,20375**		8. PERFORMING ORGANIZATION REPORT NUMBER
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)		10. SPONSOR/MONITOR'S ACRONYM(S) 11. SPONSOR/MONITOR'S REPORT NUMBER(S)
12. DISTRIBUTION/AVAILABILITY STATEMENT **Approved for public release; distribution unlimited**		
13. SUPPLEMENTARY NOTES		
14. ABSTRACT		
15. SUBJECT TERMS		

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT **unclassified**	b. ABSTRACT **unclassified**	c. THIS PAGE **unclassified**	**Same as Report (SAR)**	**2**	

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std Z39-18

Transparent Ceramics: Magnesium Aluminate Spinel

G.R. Villalobos, J.S. Sanghera, and I.D. Aggarwal
Optical Sciences Division

Motivation: There is a need for high-strength, rugged materials that transmit in the visible and mid-infrared (0.4 to 5 μm) wavelength region. These materials are needed for applications requiring transparent armor, including next-generation high-speed missiles and pods, as well as protection against improvised explosive devices (IED).

Material Selection: Silicate glasses are suitable for most common window uses, but they are too weak to be used as a practical transparent armor and as windows and domes in high-speed missiles and pods. Additionally, missile domes and pod windows must transmit to 5 μm but silicate glasses do not transmit well beyond 2 μm. Zinc sulfide transmits to beyond 5 μm, but it is too weak to be used. However, magnesium aluminate spinel is a polycrystalline ceramic material that has excellent optical and mechanical properties. It transmits from 0.2 to 5.5 μm, and ballistic testing has shown that 1/4 in. of spinel has the same resistance as 2.5 in. of bulletproof glass. Spinel is, therefore, an excellent candidate for transparent armor in light vehicles and goggles or face shields on infantry helmets. Unfortunately, there is no transparent spinel product in the marketplace, despite the fact that it has been studied sporadically since the 1960s. A review of the literature clearly indicates that attempts to make high-quality spinel have failed to-date because the densification dynamics of spinel are poorly understood.

Dynamics of Densification: Spinel is generally densified with the use of LiF sintering aid. Without LiF sintering aid, the material tends to be translucent, gray, and highly scattering. It is widely known that the LiF vaporizes during densification and must be removed from the material before complete consolidation or it will manifest itself as white cloudy regions.

We conducted extensive research into the dynamics involved during the densification of spinel. This research has shown that LiF, although necessary, also has extremely adverse effects during densification. Additionally, its distribution in the precursor spinel powders is of critical importance. These discoveries have allowed NRL to fabricate spinel to high transparency with extremely high reproducibility that will enable military and commercial use of spinel.

Figure 1 is a high-resolution scanning electron microscope (SEM) micrograph of a cloudy area showing that it is composed of small (300-500 nm) crystals that have not densified. Energy dispersive spectroscopy (EDS) analysis suggests that the small grains are made up of an MgO-rich phase. MgO has a considerably higher melting temperature than spinel and will not densify as readily at the lower sintering temperatures used for spinel. The pores and small crystals lead to optical scattering, resulting in the cloudy regions. It became apparent that LiF preferentially reacts with the Al_2O_3 in spinel, thereby leaving MgO-rich areas behind.

Inhomogeneous distribution of the LiF in the precursor powders further degrades the ability to densify spinel. Areas with large amounts of LiF react to a greater extent than those having the correct amount, while the areas deficient in LiF do not densify completely and leave large amounts of porosity. Tradition-

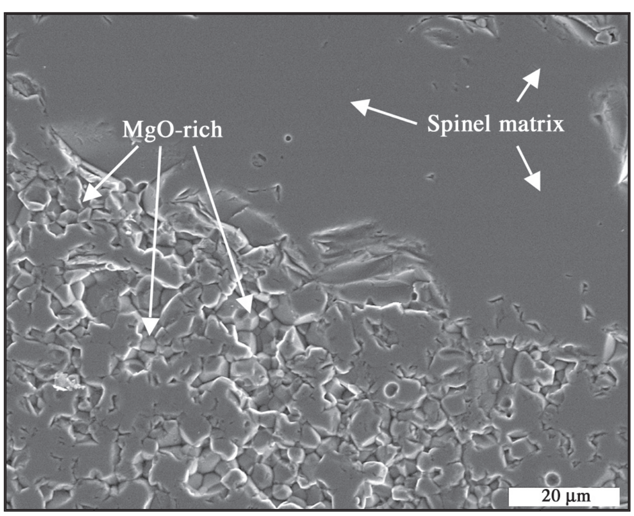

FIGURE 1
Small grained, partially sintered MgO-rich phase in $MgAl_2O_4$ spinel matrix.

ally, common wisdom has been used to increase the amount of LiF to increase the likelihood of having LiF throughout the spinel powder. Unfortunately, this just increased the extent of the LiF/Al_2O_3 reaction zones.

NRL Breakthrough: Traditional bulk mixing processes used to mix sintering aid into a powder leave a fairly inhomogeneous distribution of sintering aid that must be homogenized by extended heat treatment times at elevated temperatures. The homogenizing temperature for LiF/spinel occurs at the temperature of fast reaction between the LiF and the Al_2O_3. To avoid this, we developed a new process that uniformly coats the spinel particles with the LiF sintering aid. This allows us to reduce the amount of LiF necessary for densification (since we do not have to compensate for LiF-deficient regions) and to rapidly heat through the temperatures of maximum reactivity. Figure 2 compares spinel disks densified with and without using the NRL technique. The traditional sample appears opaque because of the high degree of optical scattering, while the NRL sample is transparent. The transmission of the NRL sample is within 1% of the maximum theoretical bulk transmission. Antireflection coatings will increase the total transmission to >99%. These results make spinel the de facto material of choice for transparent armor applications.

Conclusion: NRL has identified various reactions that had previously prevented the densification of magnesium aluminate spinel to high transparency and high yield. Based on these discoveries, we have developed particle-coating techniques that are used to routinely fabricate transparent spinel with extremely high reproducibility. Consequently, a company is pursuing a technology licensing agreement to manufacture spinel to enable its use as an armor material for personnel and light vehicle protection against small arms and improvised explosive devices as well as many other applications. The availability of spinel windows and domes will impact both military and commercial applications.

[Sponsored by ONR]

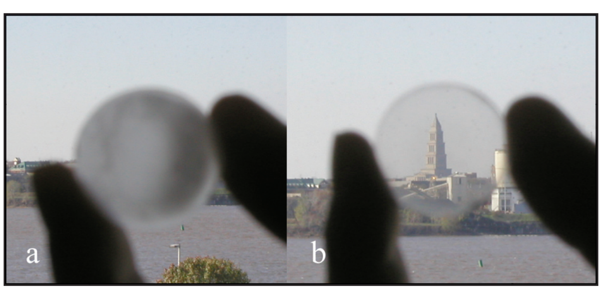

FIGURE 2
Looking across the Potomac River to the Masonic Temple, approximately two miles. (a) Traditional mixed spinel (b) NRL developed transparent spinel.

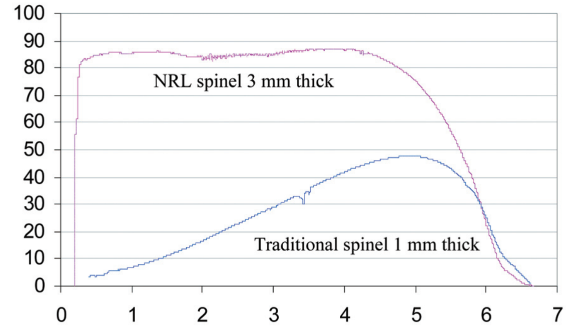

FIGURE 3
NRL spinel transmits within 1% of the theoretical bulk transmission throughout its range, while the traditional spinel has poor transmission resulting from scattering losses.