Characterization of ALON™ Optical Ceramic

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ABSTRACT

ALON™ Optical Ceramic is a durable window material for UV, Visible and Mid IR window and dome applications. The mechanical, thermal, and optical properties of ALON products produced commercially by Surmet Corporation have been measured and this new data will be presented. Comparisons to previously measured data will be made.

Optical quality, low scatter ALON having high strength that is nearly double previously reported has been made. Average strength values of 700 MPa at 21°C and 631 MPa at 500°C have been measured for ALON specimens prepared by precision surface finishing techniques. Polished optical domes tested have survived severe thermal shock tests. These strength levels are comparable to those for single crystal sapphire. Strength, thermal conductivity, thermal expansion, refractive index, emissivity and absorption coefficient will be presented. The possible mechanisms for the increased strength will be discussed.

Keywords: ALON, aluminum oxynitride, properties, strength, domes, transparent armor

1.0 INTRODUCTION

Aluminum oxynitride, ALON is a transparent polycrystalline ceramic which has high strength and hardness. It is transparent from ultraviolet (UV) wavelengths to mid infrared wavelengths (MWIR). High speed IR missile dome applications require a durable transparent material that can withstand severe thermal shock due to aerodynamic heating, remain transparent throughout the entire flight trajectory and be highly resistant to rain and sand damage. Light weight transparent armor applications require high strength and hardness in the outer layers of a ballistic laminate to defeat high velocity projectiles. Lighter weight ballistic laminate designs can be achieved by using thinner ALON panels having greater fracture strength.

The technology for the making ALON transparent ceramics was transferred from Raytheon Company to Surmet Corporation in 2002. The manufacture of dozens of large ALON panels for armor performance evaluations and the production of hundreds of domes, lenses and point of sale (POS) scanner windows have been achieved at Surmet over the past two years. In light of this recent manufacturing scale-up of the production of ALON it is important to reevaluate the critical material properties such as strength, thermal conductivity, thermal expansion, and transparency and demonstrate that the material is as good as or better than that which was produced in a laboratory/pilot scale process.

The data and results presented in this paper are part of Air Force sponsored Phase II SBIR program structured to addresses the need for an up to date and accurate material properties database for currently produced transparent ALON. Results of these measurements will be used in the future to model and predict the performance of ALON in missile dome and transparent armor applications. This materials properties study also successfully demonstrated that the same deterministic grinding and finishing operations could be used to produce high strength test samples as well as low-cost high strength thermal shock resistant missile domes.

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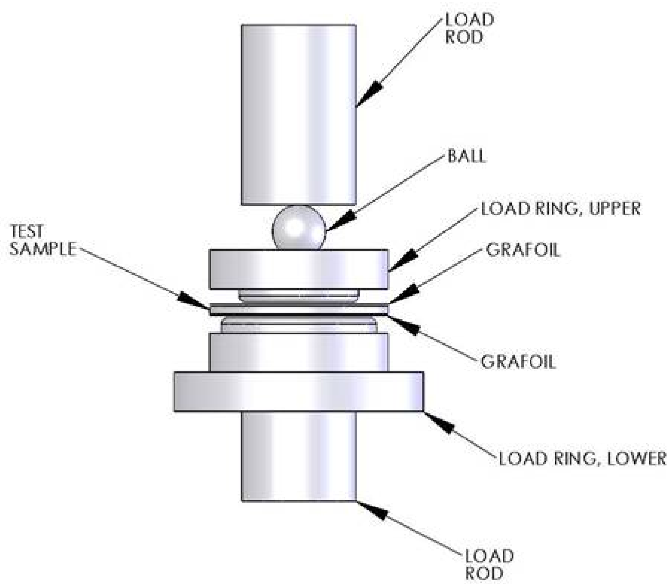
2.0 MECHANICAL PROPERTIES

Mechanical Properties Overview:

Mechanical properties testing of ALON was performed at the University of Dayton Research Institute (UDRI) in Dayton, Ohio. Biaxial Flexural Strength (at 21°C and 500°C), Young's Modulus, shear Modulus and Poisson's Ratio were measured using standard ASTM C1499-03(1) and ASTM C1259-01(2) test methods. Weibull modulus and characteristic strength were determined by fitting the measured data to a standard two parameter Weibull distribution given by Equation 1, ASTM C 1239-94a(3). Figure 1 shows the test fixture used for the biaxial flexure strength testing at room temperature and 500°C. Equation 2 is used to calculate the strengths from the sample and fixture dimensions (see Table 1) and the breaking load. Grafoil™ discs having a thickness of 0.005" thick were placed between the sample disc and the load and support rings to reduce the contact stresses that are thought to cause some samples to fail under the loading ring or support ring.



Schematic From: ASTM C1499-03

Figure 1: Biaxial Flexure Test Fixtures and Schematic of Test Setup used at UDRI for Testing ALON Samples. The loading rate used was 0.02"/min for all measurements; Humidity 50%; Temperature 21° C and 500 °C

Where: P_f = Probability of Failure
σ_θ = Weibull Characteristic Strength (MPa)
σ = Measured Strength (MPa)
m = Weibull Modulus

$$P_f = 1 - e^{-\left(\frac{\sigma}{\sigma_\theta}\right)^m} \tag{1}$$

Where: σ_f Breaking stress (psi or MPa)
F Breaking load (lb of N)
D_S Support Ring Diameter (in or m)
D_L Load Ring Diameter (in or m)
D Sample Diameter (in or m)
h Sample Thickness (in or m)
υ Poisson's Ratio

$$\sigma_f = \frac{3F}{2\pi h^2}\left[(1-\upsilon)\frac{D_S^2 - D_L^2}{2D^2} + (1+\upsilon)\ln\frac{D_S}{D_L}\right] \tag{2}$$

Table 1: Sample Size and Biaxial Flexure Fixture Dimensions.

Sample Size and Test Fixture Dimensions				
Test Period	Sample Diameter	Sample Thickness	Load Ring Diameter	Support Diameter
(Historical) Raytheon 2002	0.998”	0.051”	0.4500”	0.9000”
(Surmet) May 2004	0.994”	0.055”	0.4166”	0.8330”
(Surmet) October 2004	1.244”	0.055”	0.4166”	0.8330”

Elastic Properties Measurements

The elastic properties of ALON were measured according to ASTM C1259-01 using the same disc specimens (1.25” diameter x 0.055” thick) used for biaxial flexure testing. The values measured at UDRI for the room temperature Young’s Modulus, shear modulus and Poisson’s ratio are given in Table 2 with comparisons to previously measured values. These current measured values are only slightly different from historical values [4, 5, 6, and 7]. Test specimens in all three set of measurements were 100% dense transparent ALON. The difference may be in the due to the greater accuracy of the flexural resonance method used in the current measurements.

Table 2: Room Temperature Elastic Properties (E, G and υ) of ALON

Value	UDRI (2004)	Historical*(ref 4,5,6) SORI (1988)	Historical*(ref 7) Raytheon (1984)
Young’s Modulus , E (GPa)	321.05	321.3	323.8
Shear Modulus, G (GPa)	127.35	124.55	130.24
Poisson’s Ration ,υ	0.26	0.24	0.24
* Historical (1988 and 1984) Data Measured in four point flexure using strain gages.			

Biaxial Flexure Strength Measurements

The testing program initially set out to compare three different grades of ALON and two test temperatures (RT and 500°C). The results of these measurements (May 2004) showed average strengths comparable to historical values (Historical Raytheon 2002) [8], however, the Weibull modulus was very low and the there were some very low and some very high strengths measured. Since the material is transparent it is relatively easy to inspect for the presence of strength limiting flaws. Having not found anything of significance the next step was to examine other possible reasons for the wide variation in strengths. There were some concerns that there was damage introduced into the samples by the fixed abrasive grinding process. Typically free abrasive grinding is used to lap the material prior to final finish. There were several other concerns which included the one-inch sample diameter, and the possibility of damage introduced during the edge beveling step.

It was decided that the next set of tests compare both fixed and free abrasive grinding as well as the benefits or detriments of chemical etching the ALON prior to mechanical testing. Chemical etching or a combination of chemical etching followed by a light polish has been demonstrated to reduce the average flaw size and increase the fracture strength of garnet (YAG, GSGG, and GGG) slab lasers [9]. Table 3 and Table 4 describe the complete set of samples and test conditions evaluated in this study.

Table 3: Test Matrix for ALON Biaxial Flexure Strength Measurements

	Number of Samples Tested at Each Condition							
Material Classification	**Loose Abrasive Grind**		**Fixed Abrasive Grind**		**Mild Etch**		**Deep Etch**	
Test Temperatures	**21°C**	**500°C**	**21°C**	**500°C**	**21°C**	**500°C**	**21°C**	**500°C**
Surmet CG Grade (May 2004)	0	0	**30**	**0**	0	0	0	0
Surmet HP Grade (May 2004)	0	0	**30**	**30**	0	0	0	0
Surmet LS Grade (May (2004)	0	0	**30**	0	0	0	0	0
LS Grade (September 2004)	**15**	**3**	**14**	**5**	**15**	**0**	**15**	0
Raytheon Historical (2002)	**28**	**31**	0	0	0	0	0	0

Table 4: Sample Set Descriptions

Sample Set	TEMP (°C)	RH	Number of Samples	UDRI TEST ID	Grain Size (µm)	Date Tested
Fixed No Etch RT	21	54%	14	SMG-04-2-46	233±29	9/13/04
Loose No Etch RT	21	48%	15	SMG-04-2-47	252±34	9/13/04
Combined No Etch	21	N/A	29	N/A		
Fixed/Loose No Etch 500	500	AIR	8	SMG-04-2-59	233±29	10/6/04
Fixed Etched RT	21	50%	16	SMG-04-2-48	233±29	9/14/04
Loose Etched RT	21	50%	14	SMG-04-2-49	252±34	9/14/04
CG ALON RT	21	75%	30	SMG-04-1-84		6/14/04
HP ALON RT	21	49%	29	SMG-04-1-58	309±60	5/10/04
LS ALON RT	21	64%	30	SMG-04-1-72	254±43	5/27/04
HP ALON 500C	500	AIR	30	SMG-04-1-43	309±60	4/29/04
2002-RT	21		28	N/A	250	2002
2002-500C	500	AIR	31	N/A	250	2002

A tabulation of the measured strengths and Weibull parameters for the samples tested is shown in Table 5. A wide variation in strength is seen for the May 2004 data set. In this data set the room temperature average strength for HP-Grade ALON is 344 MPa. The high strength for this set is 746 MPa, a low strength is 185 MPa and Weibull modulus is 2.8. The failure probability distribution for this sample set shown in Figure 2 illustrates this wide distribution of strengths. A comparison of the May 2004 data with the Historical 2002 data shows that the average strengths are in the same range, 310 MPa to 390 MPa, but the Weibull Modulus is much lower and there are no extremely high or low strengths.

The high strengths measured in the first round of testing were very promising since there had never been strengths at this level ever reported for ALON transparent ceramics. The reason for low strengths was of concern and an investigation of the possible causes was begun. A low fracture strength sample (#119) is shown in Figure 3. The sample fractured into only about 6 pieces indicating a low amount of stored strain energy prior to fracture. The area where the fracture originated was located by examining the fracture pattern of the disc. The portion of the sample which contained the fracture origin was etched to reveal the grain structure and any surface defects. It can be seen in Figure 3 that there are deep scratches in the sample near the origin. These scratches were not visible using light microscopy prior to etching and indicate that there is damage in this sample which was most likely introduced during the grinding operation. This damage was not removed in subsequent processing steps and resulted in a failure at a much lower than average strength.

Table 5: Strength and Weibull Analysis for ALON Sample Sets

Sample Set	AVG σ (MPa)	STDEV (MPa)	Weibull Characteristic Strength σ_θ (MPa)	Weibull Modulus Biased m	Weibull * Modulus Unbiased m_u	R^2
Surmet September (2004)						
Fixed No Etch RT	700	169	750	8.5	7.7	0.982
Loose No Etch RT	753	179	811.9	5.6	5.1	0.968
Combined No Etch	*727*	*173*	*777.7*	*7.0*	*6.6*	*0.979*
Fixed/Loose No Etch 500°C	622	93	635.5	8.4	6.9	0.91
Fixed Etched RT (light etch)	422	59	451	10.1	9.2	0.951
Loose Etched RT (deep etch)	281	18	287.8	26.3	23.7	0.977
Surmet May (2004)						
CG ALON RT	308	126	325.1	2.9	2.8	0.952
HP ALON RT	344	146	361.6	3.0	2.8	0.953
LS ALON RT	389	135	425.2	3.2	3.1	0.988
HP ALON 500° C	364	123	410.4	3.0	2.8	0.990
Raytheon (2002)						
2002-RT	374.7	85.8	409.2	4.6	4.4	0.995
2002-500C	367.9	47.7	384.4	7.8	7.4	0.964
* The Unbiased Weibull Modulus is determined using Table 1 in ASTM C1239-94a. An unbiasing factor is used to best estimate the Weibull Modulus for small data sets. The factor approaches 1 as the # samples > 40.						

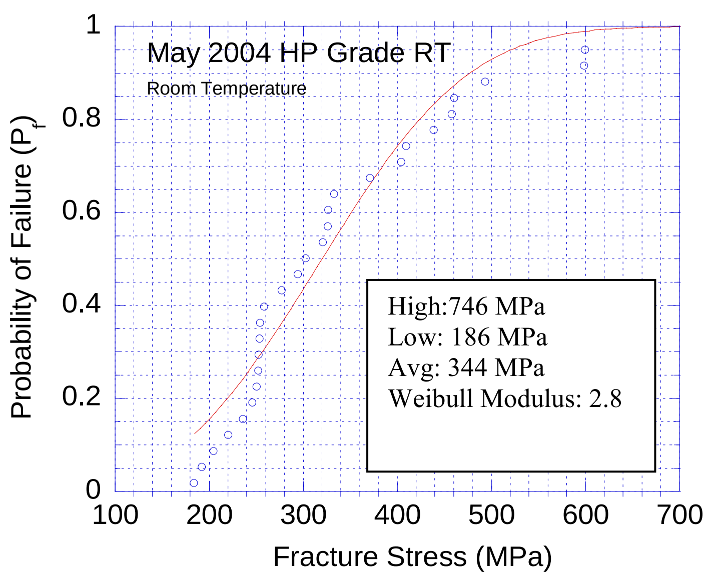


Figure 2: Weibull Failure Probability Distribution for HP-ALON (May 2004) Tested at Room Temperature.

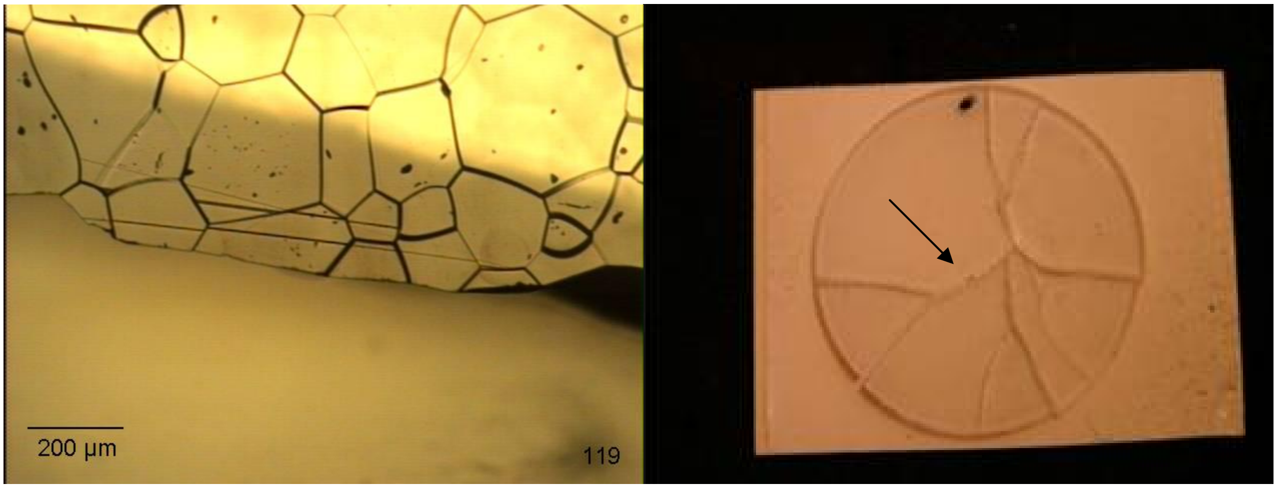


Figure 3: Photo of fracture test disc #119 (LS-Grade, May 2004 group) and an optical micrograph of the etched sample near the fracture origin. Strength: σ_f = 197 MPa and Average Grain Size = 277 µm. Scratches on the sample near the fracture origin are revealed by etching.

Several changes were made to the samples fabricated for the second round of testing (September 2004). These changes were: (1) The sample diameter was increased from 1-inch to 1.25 inch to eliminated the possibility of premature edge related failures; (2) Samples were prepared by fixed abrasive and free abrasive grinding prior to final polish and the amount of material removed at each successive grit size was increased; and (3) A chemical-mechanical final polishing step was used.

In addition, two sets of etched samples were prepared from the “super” polished sample groups. The etching was performed so that the effects of machining damage would be reduced. After polishing the samples were etched in 200°C phosphoric acid for two different time durations. The microstructure of polished and etched surfaces and the IR transmission before and after etching are shown in Figure 4. The light etching decrease the transmission in the IR only slightly and the deeper etch reduces the transmission by about 10%.

The strengths of the polished and etched ALON samples are lower than the strengths of the “super” polished samples. The average strengths decrease for the “super” polished and etched samples but the Weibull modulus increased. In the case of the lightly etched samples the average strength of 422 MPa is still higher than the average strengths reported for the May 2004 testing or for the Historical 2002 testing. The Weibull modulus for this lightly etched sample set is 9.2. The more deeply etched samples have an average strength of 281 MPa and a very high Weibull modulus of 23.7. So, unlike the data of Marion (1987) the strength of ALON is not increased by etching in phosphoric acid. The potential reliability, however, is increased. This increased reliability may not be practical without re-polishing because of the transmission decrease due to the increased surface roughness. One interesting observation is that there was no evidence of machining damage revealed by the etching process as was seen for the May 2004 test samples (see Figure 3). Many samples from that earlier May 2004 test group also show scratches revealed by light acid etching.

The Weibull failure probability distribution and representative samples from the Fixed Abrasive Group are shown in Figure 5. The average strength of this set of samples was 700 MPa and the Weibull modulus is 7.7. The highest strength sample in the fixed abrasive group had a strength of 966 MPa and fractured into numerous small fragments. The zone under the load ring appears pulverized which is an example of a very high strength fracture. There is only one low strength sample in this group with the majority of the samples having strengths exceeding 600 MPa.

Similar strength behavior was demonstrated for free abrasive ground ALON test at room temperature and both free and fixed abrasive ground ALON tested at 500°C. The average strength at 500°C is 622 MPa with a Weibull modulus of 6.9.

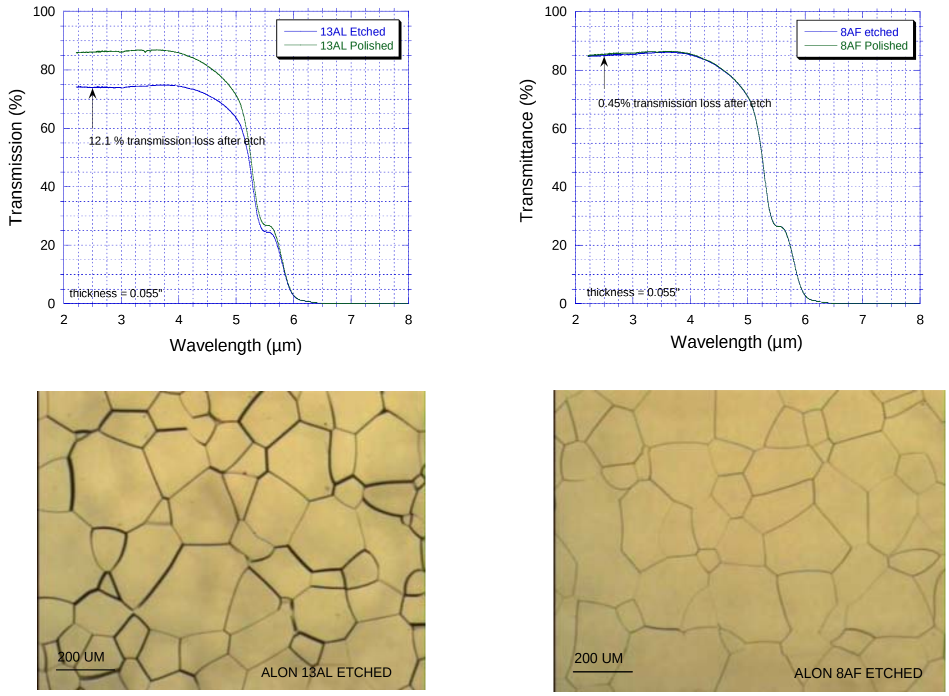


Figure 4: Microstructure and IR transmission of deeply etched (13AL) and lightly etched (8AF) ALON biaxial flexure test samples.

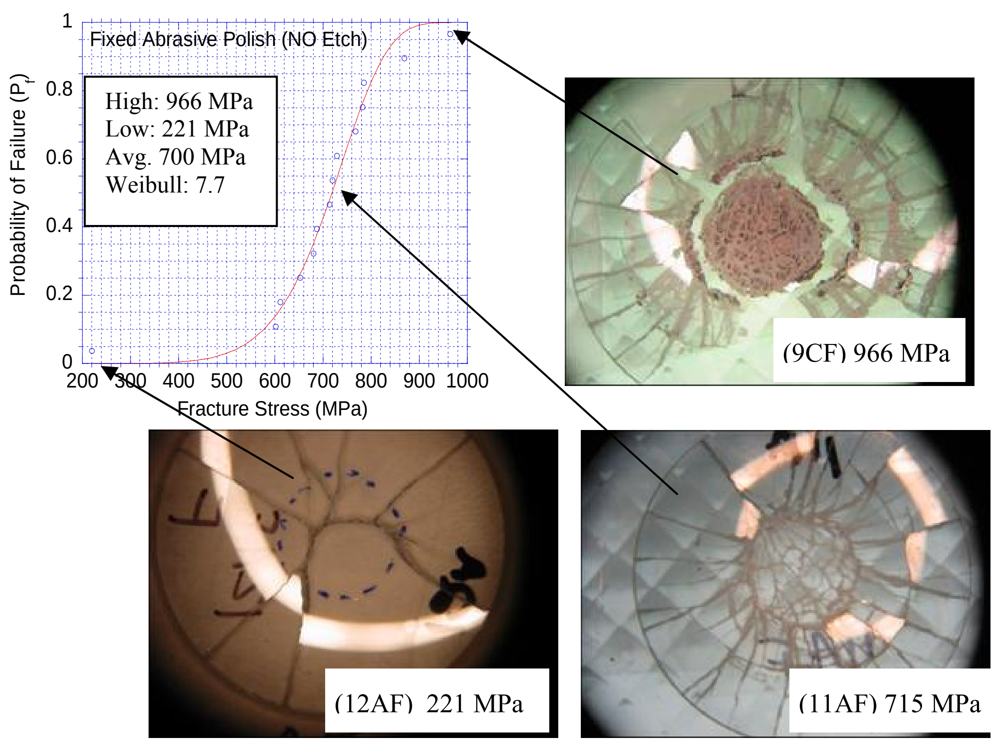
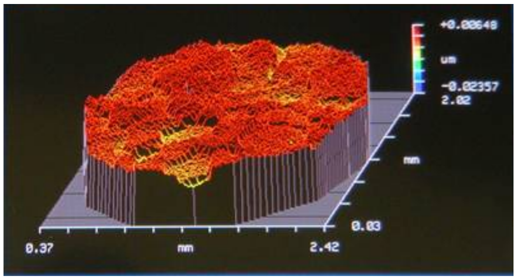
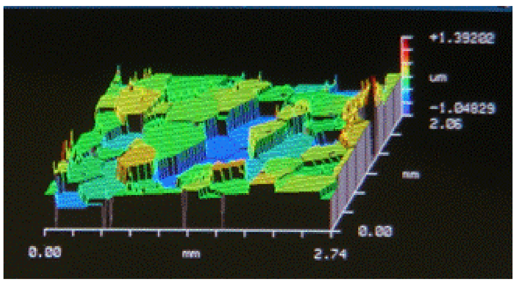


Figure 5: Weibull Failure Probability Distribution for LS-ALON (September 2004) Tested at Room Temperature

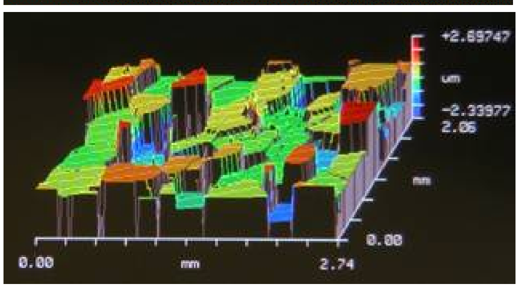
Surface roughness measurements using a ZYGO NewView 100 optical profilometer were measured for samples from the two etch groups and an un-etched sample from the high strength group. Figure 6 illustrates the nature of the surface roughness features and the level of surface roughness produced by the etching process. The most deeply etch surface sample has a roughness PV roughness of 5.0 µm and an Ra of 0.657 µm compared to a PV roughness of 2.44 µm and an Ra of 0.215 µm for the lightly etched sample. The Ra of the "super" polished samples is 10 Å and the PV roughness is 30 nm.



ALON Fixed Abrasive Polish
PV = 0.030 µm
Ra = 0.001 µm
RMS = 17.71 Å
Measured Area = 2.07 mm x 1.99 mm



ALON 8AF Fixed Abrasive Mild Etch
PV = 2.44 µm
Ra = .215 µm
RMS = .261 µm
Measured Area = 2.74 mm x 2.06 mm



ALON 13AL Loose Abrasive Deep Etch
PV = 5.036 µm
Ra = 0.657 µm
RMS = 0.887 µm
Measured Area = 2.74 mm x 2.06 mm

Figure 6: Surface roughness of polished and etched ALON biaxial flexure test samples.

The Griffith relationship given by Equation 3 commonly used to explain the relationship between the fracture strength (σ_f) and the flaw size (c_f) of a material. The fracture toughness (K_{1c}) of the material determines how susceptible the strength of the material is to the size of the flaw or crack. The factor (Y) is related to the shape and the location of the flaws of the material. [10, 11, 12, 13] For brittle ceramics and glass which fail in tension due to surface flaws the factor Y is equal to about 1. The fracture toughness (K_{1c}) of ALON is in the range of 2.0 to 2.4[7, 14].

$$\sigma_f = \frac{K_{1c}}{Y\sqrt{c_f}} \tag{3}$$

Table 6 compares the predicted flaw size calculated using equation 3 and the measured average strength with the peak to valley (PV) surface roughness and the grain size. There is no apparent relationship between the fracture strength and the grain size. There is a strong correlation between the surface roughness and the strength although there is not an obvious scaling factor to convert PV or Ra to the flaw size. The lower surface roughness "super" polished sample group has the highest strength and the increased roughness caused by etching lowers the strength accordingly. Based on the higher Weibull modulus the non-uniform etching rate of the adjacent ALON grains provides creates a more uniform distribution of flaws on the surface. The greater etch depth smoothes out the distribution of larger subsurface flaws introduced by machining and creates a new controlled distribution of larger flaws that lowers the average strength but increases the Weibull modulus. The high strength "super" polished and un-etched ALON has a low level of subsurface damage that is not revealed by etching as was the case for the low strength sample #119 and other in that group tested in May 2004. The nature of this subsurface damage that cannot be exposed by etching will need to be examined in greater detail using TEM and X-Ray topography which can reveal dislocations, twins and strains in the polished surfaces.[15, 16]

Table 6: Predicted Flaw Size for ALON Fracture Test Samples Compared to the Surface Roughness

Sample Set	Measured Average Strength (MPa)	Weibull Modulus	Calculated Flaw Size (µm)	Measured PV Surface Roughness (µm)	Measured Grain Size (µm)
Polished Fixed Abrasive 9/2004	700 ±169	7.7	12	0.030	233±29
Mild Etch Fixed Abrasive 9/2004	422 ±59	9.2	32	2.44	233±29
Deep Etch Loose Abrasive 9/2004	281 ±18	23.7	73	5.04	233±29
Sample #119 from LS-Group	197(individual)		148	scratched	277±48
LS-Group May 2004 testing	389 ±135	3.1			254±43

3.0 THERMAL PROPERTIES

The thermal properties of transparent ALON have been evaluated in the past by several labs [6, 17] There is a need for more recent and up to date data measured for currently available transparent ALON. These material properties will be used in predictive models for thermal shock performance in high speed missile dome application. The measurements reported in this paper are more accurate than historical data and cover a wider temperature range. Samples were prepared using LS-Grade ALON (low scatter optical grade) produced at Surmet Corporation, Burlington, MA. The measurements of the thermal properties of ALON were made at TPRL, Inc. [18].

Thermal Conductivity (κ):
Method: Laser Flash Diffusivity (ASTM E1461), Sample gold coated to minimize the effects of radiation heat transfer.
Temperature Range: -50°C to 700°C

Bulk Density: Measured by precision geometry and mass measurements, value = 3.666 g/cc @ 23

Specific Heat (c_p):
Method: Scanning Differential Calorimeter Perkin Elmer Model DSC-2, (STM E1269)
Temperature Range: -50°C to 700°C

Thermal Expansion Coefficient (CTE):
Method: dual push-rod dilatometer (Theta Dilatronics II)
-150°C to 800°C

Thermal Conductivity Comparisons are referenced as follows:

- TPRL (2004) – This Study, Reference 18
- JHAPL (1989) – Summary of Data Measured at SORI in 1988. Reference 4
  - Comparative Rod Method (CRA) – Referenced To Stainless Steel
- SORI (1986) – Earliest Measurements on ALON – Reference 17
  - Comparative Rod Method (CRA) – Referenced To Stainless Steel
- RAYFIT (1988) - Raytheon (1988) – Polynomial Fit to SORI 1988 Data

A summary of the thermal properties measured is listed in Table 7 and comparison plots to historical data are shown in Figures 7 thru 9. Figure 7 shows that the thermal conductivity of ALON measured at TPRL is slightly higher in the temperature range from -50°C to 600°C than previously measured. The high temperature values (T > 600°C) reported previously are most likely in error due to the radiation effects of heat transfer through the transparent samples. In the current measurements the radiation heat transfer effects were minimized by applying a gold coating to both sides of the test sample. Thermal Expansion values are slightly higher than previously reported and these new measurements extend the temperature range to include temperatures from -150°C to 800°C. A greater number of points were measured on the specific heat vs. temperature curve and the temperature range of the measurements was extended to include points below 0°C to -50°C. The thermal shock figure (500°C) of merit, R' of ALON is compared to three other IR dome materials in Figure 8. It can be seen in this table the predicted thermal shock resistance of ALON for "mild" thermal shock conditions is equivalent of equal to sapphire, spinel, and MgF_2 in terms of calculated thermal shock performance.

Table 7: Thermal Properties of Transparent ALON Measured at TRPL (2004)

T emp.	MEAN CTE	Specific Heat	Thermal Diffusivity	Thermal Conductivity
°C	10^{-6} in/in - °C	J/g-K	cm^2/s	W/m-K
-150.0	3.067	-	-	-
-100.0	3.591	-	-	-
-50.0	4.070	0.600	0.0789	17.36
-20.0	4.336	0.687	0.0587	14.78
0.0	-	0.733	0.0500	13.44
23.0	-	0.781	0.0430	12.30
100.0	5.230	0.916	0.0312	10.48
200.0	5.783	1.015	0.0236	8.79
300.0	6.196	1.084	0.0201	7.98
400.0	6.507	1.132	0.0176	7.32
500.0	6.758	1.165	0.0161	6.89
600.0	6.986	1.191	0.0154	6.71
700.0	7.211	1.219	0.0137	6.12
800.0	7.432	1.235	0.0113	5.13

Table 8: Thermal Properties and Thermal Shock (@ 500°C) F.O.M Comparison Transparent Dome Materials

Material	C.T.E. α (10^{-6}/K)	Young's Modulus E (GPa)	Thermal Cond. κ (W/m-°C)	Poisson's Ratio ν	Strength σ_f (MPa)	Thermal Shock F.O.M. R' (W/m)
MgF_2	10.4 (ref 28)	114 (ref 32)	5.0 (ref 28)	0.28 (ref 32)	120 (ref 24)	365
Sapphire	7.70 (ref. 29)	345 (ref 32)	10.9 (ref. 29)	0.27 (ref 32)	708 c-plane (ref 25) 535 c-plane (ref 30) 375 c-axis 60° (ref 31)	2120 1602 1123
ALON	6.76 (ref 18)	321 (ref 26)	6.9 (ref 18)	0.26 (ref 26)	622 (ref 26)	1464
Spinel	7.77 (ref 27)	193 (ref 32)	7.88 (ref 27)	0.26 (ref 27)	190 (ref 27)	739
Thermal Shock F.O.M.: $R' = \sigma_f (1-\nu)\kappa/\alpha E$						

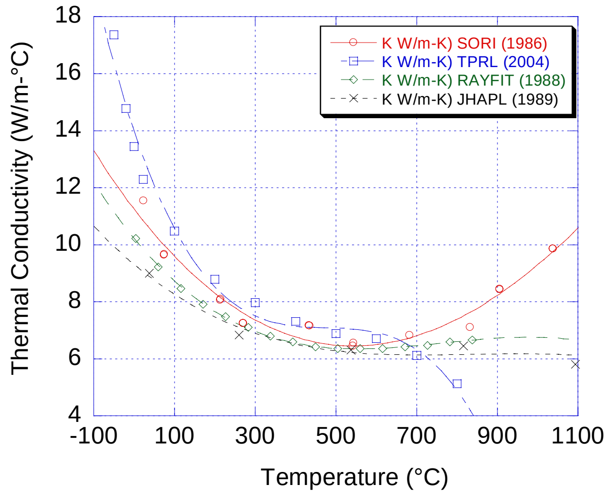


Figure 7: Thermal Conductivity or ALON - Historical Data Comparison

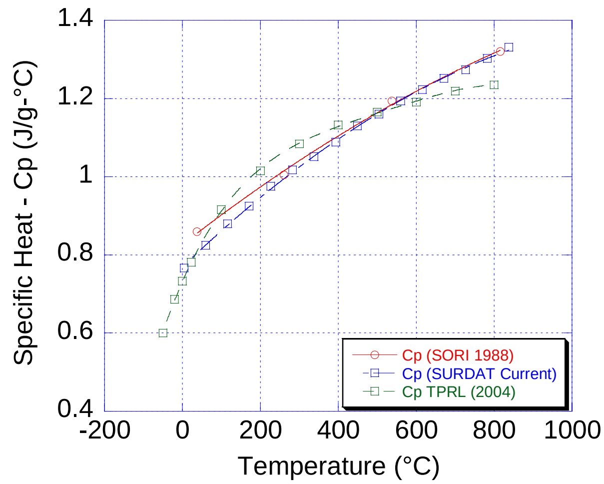


Figure 8: Specific Heat of ALON – Historical Data Comparison

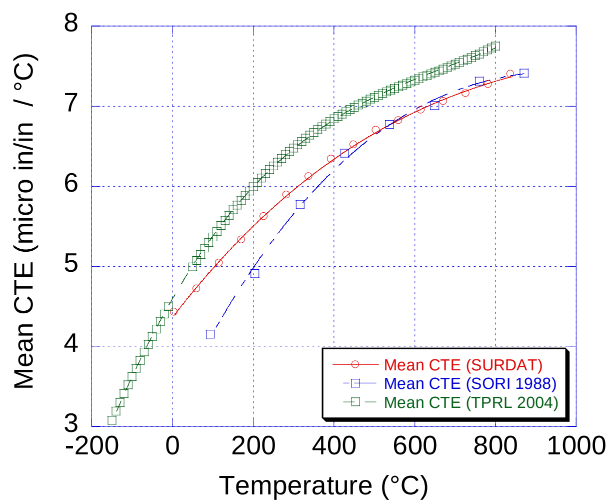


Figure 9: Thermal Expansion Coefficient of ALON – Historical Data Comparison

4.0 THERMAL SHOCK PERFORMANCE (DOME TESTING)

Finished domes may have damage at the surface or contain internal flaws that are not visible or detectable by normal optical inspection methods. This damage can greatly reduce the strength of the domes and skew the predicted reliability that was based on Weibull statistical analysis of test coupons. Reliability testing of finished optical components such as domes is an important operation for screening out weak components. It is desirable that the method be a non-destructive method to screen domes for such defects and also provide a threshold which insures that only structurally robust domes make it to the missile level. The test must not weaken the component. Several methods have been proposed that would provide a non-destructive thermally or mechanically induced stress: quartz lamps, laser heating, hydraulic pressure and liquid quench have all been proposed or evaluated as suitable methods for proof testing domes.

Of these methods, the liquid droplet quench provides the thermal stress similar to that produced during actual flight and can be easily implemented as a 100% screen or proof test. Predictive models to determine the stress levels introduced by the liquid droplet quench test have been developed but are difficult to verify. The non-linear and highly dynamic phase change occurring as jets of water are impinged on the dome make analysis of the thermal contour and resulting stress field nearly impossible. It is only with the aid of experimental measurement using IR cameras and carefully time mapping individual elements that a relation between quench temperatures and peak dome stress can be ascertained.

While this is a rather laborious technique requiring extensive testing the test has been verified using sapphire domes having the same geometry as the ALON domes that were tested recently. The first order assumptions for the ALON dome test parameters were based on sapphire material property data. This assumes that the properties of ALON and sapphire of the materials vary similarly and do not significantly affect the stresses induced by the test.

Testing was performed on three ALON domes polished using the same grinding and polishing media and techniques as were used for the high strength biaxial flexure test specimens (σ_f =700 MPa ± 169 MPa, m = 7.7). The liquid droplet quench testing was performed at Raytheon's test facilities in Tucson, AZ. using the methods and parameters that were established for sapphire domes of the same geometry. ALON domes were heated to a uniform temperature of 150°C and water spray quenched on the concave surface to room temperature using a water temperature of 25°C. *All three domes survived this test.*

Testing of additional ALON domes is currently is process and predictive models of the thermal stresses generated during the quench testing are being refined using the newly measured temperature dependent thermal properties of ALON. The models will be used to alter if necessary the quench conditions so that the test conditions of liquid droplet reliability test for ALON domes can be finalized.

5.0 OPTICAL PROPERTIES

The purpose of this analysis is to quantify the emissive properties of ALON and compare these values with two other MWIR dome materials. This is important because a dome in flight will be aerodynamically heated and will begin to emit radiation to the detector behind the dome.

Several characteristic optical properties of ALON were measured and analyzed at JHAPL, Laurel, MD.[19] The data was further analyzed and reduced to final form by Optics Solution, Dayton, Ohio.[20] A brief summary of the data will be presented in this paper and a more complete description of the measurement methods and results can be obtained from Reference 21.

The ALON material measured was optical LS-grade ALON made by Surmet Corporation, Burlington, MA. LS-Grade ALON is large grained polycrystalline material having low optical scatter. The LS-Grade and HP-grade ALON are essentially the same in terms of purity and grain size. The primary difference is the optical scatter in the visible region of the spectrum. The spinel is polycrystalline optical grade material produced at Surmet Corporation. The sapphire was made by Crystal Systems and the comparisons made here are based on previous extensive measurements on sapphire made at JHAPL. Measurements of the transmission vs. temperature were made to obtain the extinction coefficient. The scatter (see Table 9) at 3.39 µm was measured to determine the contribution of scatter to the extinction coefficient.

By knowing the index of refraction of the material and using the measured transmittance values, the total extinction coefficient can be calculated using equation 4.

$$\beta_{\text{ext}} = \frac{\ln\left(2R^2\tau\right) - \ln\left[\sqrt{(1-R)^4 + 4R^2\tau^2} - (1-R)^2\right]}{L}. \tag{4}$$

Where: $R = \left(\frac{n-1}{n+1}\right)^2$ is the single surface reflectance (5)

And: n is the index of refraction,
L is the sample thickness
τ is the transmittance.

The refractive index values for ALON (Reference 22) were computed using the Sellmeier Equation 6

$$n^2 = 1 + \frac{A\lambda^2}{\left(\lambda^2 - B\right)} + \frac{C\lambda^2}{\left(\lambda^2 - D\right)} \tag{6}$$

Where: A = 2.1578, B = 0.010507, C = 3.6683 and D = 243.02

The refractive index values for Spinel and Sapphire (Reference 23) were computed using the Sellmeier Equation 7

$$n^2 = A + \frac{B\lambda^2}{\left(\lambda^2 - C\right)} + \frac{D\lambda^2}{\left(\lambda^2 - E\right)} \tag{7}$$

For Spinel A = 1.9370478, B = 0.95795641, C = 0.01832141, D = 3.7654327, and E = 300.

For Sapphire A = 1.6257584, B = 1.457221, C = .0113346735, D = 6.6760415, and E = 400.

The absorption coefficient is then calculated using the following relationship:

$$\beta_{ext} = \beta_{scatter} + \beta_{abs} \tag{8}$$

Table 9: Total integrated scatter (TIS) and scatter coefficient (β).

Sample #	Sample Description	Wavelength (µm)	%Transmittance @ 3.39 µm	TIS	scatter coefficient β (cm^{-1})
11	ALON (1" x 0.050")	3.39	86	0.005	
14	ALON (1" x 0.236")	3.39	88	0.011	0.0127
15	Sapphire (1" x 0.050")	3.39	88	0.005	
16	Sapphire (1" x 0.236")	3.39	87	0.012	0.0148
17	Spinel (1" x 0.080")	3.39	88	0.005	
18	Spinel (1" x 0.236")	3.39	87	0.02	0.0379

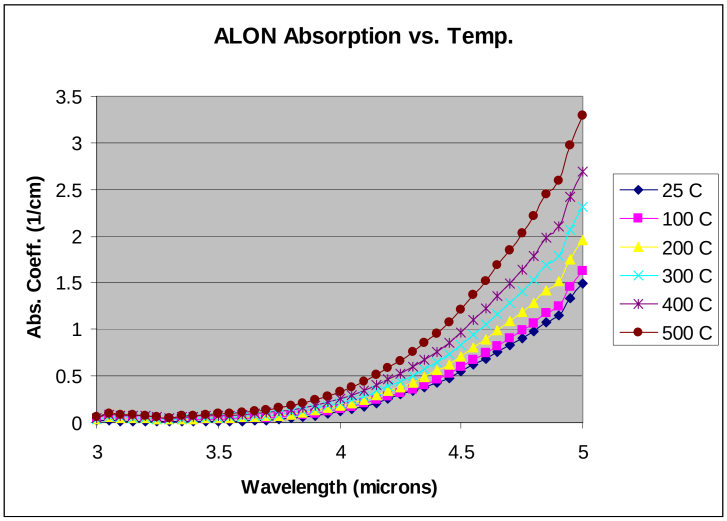


Figure 10: ALON: Absorption Coefficient vs. Wavelength and Temperature.

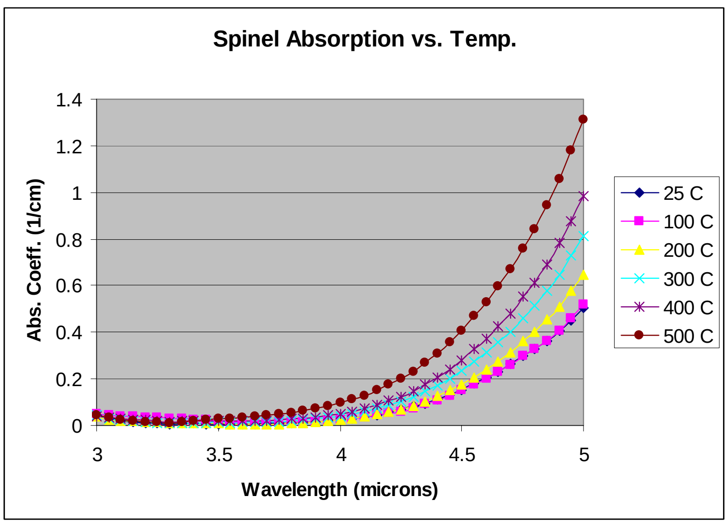


Figure 11: Spinel: Absorption Coefficient vs. Wavelength and Temperature.

The temperature dependence of the absorption coefficient of ALON in the 3 to 5 μm wavelength range is shown in Figure 10. The absorption coefficient of spinel (Figure 11) and sapphire exhibit a similar temperature and wavelength dependence. A comparison of the calculated emittance behavior of these three materials is given in Table 10.

A first order evaluation of the emittance is simply given by the equation below by Equation 9. The emittance is determined by the product of the absorption coefficient and the path length or sample thickness. Table 10 compares the band averaged emittance of ALON, spinel and sapphire domes having a thickness (L) of 0.090".

$$\varepsilon = \beta_{abs} L \quad (9)$$

Table 10: Band averaged emittance of ALON, Spinel and Sapphire

	Band Averaged Emittance					
Material	**25C**	**100 C**	**200 C**	**300 C**	**400 C**	**500 C**
Spinel	0.024	0.025	0.029	0.037	0.045	0.064
ALON	0.076	0.087	0.104	0.122	0.144	0.179
Sapphire	0.044	0.048	0.058	0.071	0.087	0.107

The lower emittance of spinel makes it the most suitable optical material if dome emission is of primary concern. In general the emittance of ALON is about three times that of spinel and about slightly less than 2 times that of sapphire. The higher strength of ALON compared to spinel and its lower cost compared to sapphire and spinel makes ALON an attractive and viable dome material for high speed MWIR missile domes.

6.0 SUMMARY

Complete thermo-physical and optical properties of ALON have been tested for various grades of material produced using full-scale production processes. Complete data for the design of critical optical components incorporating ALON is given, including Weibull modulus and characteristic strength values. Deterministic grinding and polishing of ALON has resulted in characteristic strength values which are over 160% greater than that previously reported at high temperature (500C) and over 180% greater for room temperature samples. Through use of Surmet's proprietary finishing processes, the reliability of ALON has also increased significantly from previous reports. It is expected that the finishing techniques developed for ALON will also be useful for strength improvements and increased reliability of other hard polycrystalline ceramics and single crystal MWIR window materials such as $MgAl_2O_4$-spinel and sapphire.

ALON Low Scatter (LS) grade material has been found to have the best combination of mechanical and optical properties for use in severe environments. For thermal shock applications, ALON is comparable to Sapphire, while clearly superior to MgF2 and Spinel. Due to its lower cost for raw material and fabrication it will continue to replace sapphire in many applications.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the support of the follow organizations and individuals:

(AFRL) Materials and Manufacturing Directorate, Air Force Research Laboratory,(WPAFB,) Dayton OH

(UDRI) The University of Dayton Research Institute, Dayton, OH
Steve Goodrich
(TPRL) Thermophysical properties Research Institute Inc. West Lafayette, IN
Jozef Gembarovic
(JHAPL) John Hopkins Applied Physics Lab, Laurel, MD
Dave Blodget, Mike Thomas, and Dave Hahn

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