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THERMAL AND MECHANICAL PROPERTIES OF CALCIUM LAMTHANUM SULFIDE

Final Report to

OFFICE OF NAVAL RESEARCH Department of the Navy 800 North Quincy Street Arlington, Virginia 22217

Contract Number N00014-83-X-0195

April, 1985



Southern Research Institute

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PREPARED BY:

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OCT 2 8 1985 Southern Research Institute 2000 Ninth Avenue, South Post Office Box 55305 Birmingham, Alabama 35255-5305





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THERMAL AND MECHANICAL PROPERTIES OF CALCIUM LANTHANIUM SULFIDE

1.0 INTRODUCTION

This is the final report of the effort conducted at Southern Research Institute for the Office of Naval Research, Arlington, Virginia, under contract number N00014-83-K-0195. The purpose of this effort was the determination of the mechanical and thermal properties of specimens fabricated from calcium lanthanium sulfide (CaLa₂S₄) by Raytheon Company, Research Division.

The CaLa S, was reported by Raytheon to have initial (slow) decomposition occurring at 700 to 800 °C. They suggested 1000 °C as an effective use temperature. It melts at 1800 °C. It is further reported to have favorable transmission properties in the 8-12 micron range and twice the hardness of ZnS.¹

The test matrix used is shown in Table 1. It is essentially identical to the test matrix utilized for the evaluation of ZnS and ZnSe under NWC contract number N60530-83-C-0031 and reported in SoRI-EAS-84-1096, Thermal and Mechanical Properties of Candidate Optical Materials for IR windows. It Was designed to obtain sufficient information for comparative design studies and screening of the material with emphasis on those properties which are anticipated to be most critical for a thermostructural environment.

^V The thermal stress resistance was investigated using thermal expansion, thermal conductivity, specific heat and flexure. Sensitivity

studies conducted on this class of materials (reference 2, see Appendix B) show the critical properties which control the thermal stress reponse for the general heating rates and geometries of interest here to be the thermal expansion, tensile strength or strain to failure and the ratio of the high temperature compressive modulus to low temperature tensile modulus. The other properties measured have a second order effect on the thermal stress response, but are important for other critical responses. For example, the thermal conductivity and specific heat, in addition to being necessary to predict the thermal fields for thermal stress prediction, also affect in depth heating of other system components and with the thermal expansion, aid in thermal deformation compatibility design with the collar. In addition to these properties, nondestructive measurements (NDC) were made to aid the interpretation of the data and fractographic studies were made of the tested specimens to aid in the understanding of the results.

Tensile and compressive tests, which were planned in the initial matrix, were not conducted as specimens were not received. This is unfortunate, particularly for the tensile tests, as these are more directly related to the response of the material as a dome under thermostructural loading than are the flexural results.

The flexure tests were conducted at a series of temperatures providing both flexural modulus and strength. Tests were conducted at 70, 500, 1000, 1500 and 1800 °F. (20, 260, 538, 816 and 982 °C). Thermal expansion and specific heat were measured up to the material limit (~1000 °C). Thermal conductivity was measured up to about 650 °C at which temperature the degradation of the material poisoned the thermocouples and additional data could not be taken. Several attempts to extend that range meet with limited success as will be discussed later.

The nondestructive tests consisted of measuring the bulk density and sonic velocity of each specimen plus a careful visual examination. The surface roughness was inspected using optical and SEM microscopy. SEM microscopy was used for the fractography.

2.0 APPARATUSES AND PROCEDURES

2.1 Flexure

Beam flexural evaluations were performed in the flexural apparatus shown in Figure 1 which consists of a load frame, load cell, load train, graphite resistance furnace, deflection measurement system and associated equipment for continuous measurement of load and deflection. Load was applied to the specimen from the lower end of the load train, and load measurements are made by the load cell at the upper end. As the load is applied to the specimen midpoint, deflection of the specimen is measured by means of a rod contacting the specimen midpoint and extending down to a differential transformer. The differential transformer is supported by a tube that attaches to the support bar eliminating load train motion from the deflection measurement. A new molybdinum loading system was designed and fabricated for this effort.

From the plot of load versus midpoint deflection, the values of modulus of rupture and flexural measured. The ultimate strength is calculated from the equation:

 $S = \frac{Mc}{I}$

which simplifies to

649363

$$S = \frac{PL}{bh}$$

for a specimen with a rectangular cross-section employing the third span loading method, and where fracture occurs within the middle one third of the specimen span length. The four point loading system is shown schematically in Figure 2. In these equations:

- S = modulus of rupture
- P = maximum applied load
- L = span length
- b = width of specimen
- h = height of the specimen

The initial modulus in flexure was calculated from the equation:

$$E_{f} = \frac{6P [a^{3} 3 + ac/2 (a + c/4]}{\delta bh^{3}}$$

where

 E_{f} = elastic modulus in flexure

 P/δ = ratio of load to corrected midpoint deflection at any point along the elastic portion of the curve

a and c = distances between the supports and loading points

The above equation neglects the deformation due to shear and assumes that the neutral axis coincides with the center of the cross-section.

2.2 Thermal Conductivity

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The apparatus used for the evaluation of the thermal conductivity of these materials through 1800 °F was the comparative rod apparatus (CRA). The CRA compares the conductivity of the specimen being characterized to the known conductivity of a reference piece. Several types of references are available and their conductivities are traceable to NBS data or other reliable sources. In this effort references of Pyroceram and slipcast fused silica were used. The CRA consists of a series of cylindrical pieces stacked in a vertical column as illustrated in Figure 3. The specimen and reference pieces are placed in the order reference-specimen-reference. Above the top reference is an electric heater that serves as the heat source for the thermal gradient in the experiment. AlSiMag insulators are placed above the heater and a steel rod forms the top of the column, receiving a compaction weight through a steel ball. Below the bottom reference is another electric heater or insulator, another steel rod, and a cooling sink, if required. The entire column is supported upon a steel ball for elimination of lateral forces that could deform the structure.

In assembling the apparatus, a large annular shell of alumina or transite is moved down around the stacked pieces. The central core of the shell is wound with five electric coils which are guard heaters, each separately controlled to match the temperature gradient through the stacked column, thereby controlling radial heat losses or shunting.

The annular space between the central core and the stacked column is filled with diatomaceous earth, thermatomic carbon, or other insulating material selected to be compatible with the specimen. The entire shell is also filled with insulation. In this case the insulation was thermatomic carbon.

Thermocouples are used to measure temperatures at all key points in the apparatus, including typically three points in the references and specimens, two points in the upper and lower guards, and one point in the middle guard.

For specimens with a conductivity of greater than 10 Btu-in./hr-ft-°F, the temperature gradient through the column is considered to be linear and the radial heat losses to be negligible. The calculations are made in a straightforward manner from Fourier's equation for one dimensional heat flow:

 $q'' = \Delta t k/l$

where q" = heat flow through the material

 Δt = temperature drop across the gage section

k = conductivity of the piece

1 = gage length of the piece

The heat flow is calculated for the reference pieces directly since their conductivities are known and the temperature drops and gage lengths have been measured. It is then solved for the conductivity of the specimen using the arithmetic average of the q" of the upper and lower references.

2.3 Thermal Expansion

Thermal expansion measurements were made using quartz tube dilatometers modified from the Bureau of Standards design for measurements up to 1800 °F. Improvements have been made to prevent lateral movement of the quartz tube, thus eliminating possible erroneous readings.

The specimen was placed in a quartz tube that is firmly secured to the body of the apparatus, which is mounted on a work bench. A second quartz tube of slightly smaller diameter was inserted into the outer tube such that it rests on the specimen end. A quartz rod that is free to move vertically was then placed into the apparatus such that one end is in contact with the dial gage piston. The body of the dial gage was firmly attached to the apparatus. Any expansion or contraction of the specimen is transferred through the inner quartz tube, the quartz push rod, and the dial gage piston to register a displacement on the gage. The quartz tube and specimen are located within a cylindrical electrical heater and can be heated to 1800 °F in the apparatus. The dial gage is calibrated in 0.0001 inch displacements and has an accuracy of ± 0.0001 inch at any point in its range (0.5 inches).

The use of quartz for both the fixed and movable parts of the apparatus eliminates any difference in expansion rates and allows the apparatus to record only the expansion or contraction of the specimen. The recorded raw data are then corrected based on calibration data for that dilatometer developed from regular calibration runs on traceable standards.

This facility is shown schematically in Figure 4.

2.4 Heat Capacity

The heat capacity to 1000 °F was determined from data obtained in an adiabatic calorimeter. In this apparatus the heated specimen was dropped into a thermally guarded, calibrated cup, and the enthalpy is measured as a function of the increase in temperature of the cup. The heat capacity is the slope of the enthalpy-temperature curve.

A tubular furnace was used to bring the specimen to temperature. The furnace pivots over the cup allowing the unit also to be used with a cold box for temperatures down to -300 °F. When the furnace is in place and the desired temperature is reached, the specimen is released from a suspension assembly which is triggered externally. Thermocouples are used to measure the specimen temperature.

Specimens of the material were heated to the desired temperature, and following a stabilization period, are dropped into the calorimeter cup. Adiabatic conditions are maintained during each run by manually adjusting the cup guard bath temperature.

The covered cup of the calorimeter is approximately 2.5 inches diameter by 2 inches deep. Three thermocouple wells are located in the bottom wall of the cup. The cup is mounted on cork supports, which rest in a silver plated copper jacket. The jacket is immersed in a bath of ethylene glycol, which is maintained at the temperature of the cup by means of a heater and copper cooling coils immersed in the liquid. A double bladed stirrer

maintains uniform bath temperature.

In the calorimeter six copper-constantan thermocouples, differentially connected between calorimeter cup and jacket, indicate temperature difference between the cup and bath. The six thermocouples allow a difference of 0.03 °F to be detected. This difference is maintained to within 0.15 °F. During the run, absolute temperature measurements of the cup are determined by means of the three thermocouple junctions, series connected, in the bottom of the calorimeter cup.

The enthalpy of the specimen at any initial temperature is calculated from the equation:

 $h = K/W_{S} (t_{2} - t_{1})$

where

- $h = enthalpy above t_2$
- K = calorimeter constant, 0.2654 Btu/°F
- W_{s} = sample weight in lbs

 t_1 = initial cup temperature in °F

 t_2 = final cup temperature in °F

The calorimeter constant of 0.2654 Btu/°F was determined by measuring the enthalpy of an electrolytic copper specimen of known specific heat. The enthalpy is referred to a common base temperature of 85 °F using linear interpolation. The enthalpy-temperature curve established is used to

determine heat capacity (specific heat) by measuring its slope at different temperatures. This is done both graphically and by analytical methods.

The accuracy of the apparatus has been confirmed by measuring the enthalpy of sapphire (SRM 734 from NBS) and other data which indicate that the overall uncertainty of the apparatus is ± 3 percent.

2.5 Bulk Density

The bulk density of the specimens was measured on the specimens by gravimetric techniques. The specimens were measured with micrometers and weighed on a Mettler balance. The reported density is simply the mass, in grams, divided by the calculated volume in cubic centimeters.

2.6 Ultrasonic Velocity

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The ultrasonic velocity was measured on each of the specimens in the direction of test. The measurement was made using a through transmission technique. The setup is shown schematically in Figure 5.

The basic apparatuses used for measuring ultrasonic velocity were a Sperry UM 721 Reflectoscope and a Tektronix oscilloscope. In using the through transmission, elapsed time technique for measuring velocity, a short pulse of longitudinal mode sound was transmitted through the specimen. An electrical pulse from the pulse generator was applied to the crystal in the transducer. The pulse generated by the transducer was transmitted through a short delay line and inserted into the specimen. The time of insertion of the leading edge of this sound beam was the reference point on the time base of the oscilloscope which was used as a high speed stop watch. When the leading

edge of this pulse of energy reaches the second transducer it was displayed on an oscilloscope. The difference between the entrance and exit times was used with the specimen length to calculate the sonic velocity.

3.0 DATA AND DISCUSSION

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3.1 NonDestructive Characterization

Table 2 shows the bulk density and ultrasonic velocity of each of the specimens tested under this effort. Note that some of the specimens received earlier (thermal specimens) were of slightly lower density than the bulk of the specimens. The average density was 4.611 gms/cc. The sonic velocity was typically 0.201 in./µsec. From these data a sonic modulus (neglecting Poisson's effect) can be calculated. It was typically 17.5 x 10^6 psi. Visual inspect saw no obvious defects except a few chips previously noted by Raytheon.

3.2 Flexure

The flexural test results are provided in Table 3. The tests were run on specimens as shown in Figure 6 with a 1.25 inch inner span and a 3.75 inch outer span. As requested the tensile face was opposite the marked face. This face was reported to have the better finish.

The 70 °F flexural strength was not very high, averaging 7240 psi. This was less than the value reported by Raytheon of about 14000 psi. A large portion of that difference may be attributable to specimen size, the Raytheon data having been generated on miniature specimens. The average 70°

flexural modulus was 13.57 x 10⁶ psi. Note that prior to every test a nondestructive mechanical (low stress loading, 1200 psi) test was run at 70° to get an initial modulus). The average value for all the NDM tests was 13.48 x 10⁶ psi, in good agreement with the average of the five 70° tests. The modulus is also in fair agreement with the calculated sonic modulus. The elevated temperature moduli show a steady decrease as a function of temperature. This is shown in Figure 7. At 1500 and 1800 °F the data became somewhat nonlinear and were represented by a bilinear fit. The secondary slope is shown as solid symbols on Figure 7 and an effective yield point provided in Table 3. The ultimate flexural strength as a function of temperature is shown in Figure 8. The strength drops slightly or is almost constant (eliminating the weakest specimen brings the average 500° strength to 6750 psi) through 1000 °F then has higher strengths at 1500 and 1800 °F. The higher strengths at 1500 and 1800 °F are concurrent with the nonlinearity of the flexural response curve.

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The failures occurred at relatively low levels giving poorly defined fracture patterns. There were zones in the material which gave the appearance of being a visually more absorptive precipitate. These were present in all specimens but became more distinctive as the specimens had seen temperatures of 1000° and over. Also, specimens of that temperature range emitted a distinctive sulphurous order. EDS scans showed these "precipitate" zones to have the same elemental components as the other zones of the specimens. Only calcium, lanthanum and sulfur were detected as illustrated in Figure 9. Figures 10 through 13 show scanning electron microscopy documentation of the fracture initiation site in FS 10, one of the weaker 70 °F flexure tests. Most but not all of the specimens were even less definitive. There is the appearance of a mirror zone and some hackle in the left tensile corner of the specimen seen in Figure 10. The initiation site

(Figures 11 and 12) does not show any defect structure. It was in a "precipitate" zone. Figure 13 shows the structure in the hackle.

The raw load deflection curves are given in Appendix A.

3.3 Thermal Expansion

Figure 14 shows the specimen used for measuring the thermal expansion of the $CaLa_2S_4$. The measured response is shown in Figure 15 and the data given in Tables 4 and 5. The thermal expansion is higher than 0° sapphire which is shown as a reference. Above 1000° a sulfur odor was noted. There was also a slight deviation between the specimens at that temperature range. The two specimens both returned to near zero residual change after cooldown to room temperature. No significant weight loss was noted.

3.4 Specific Heat

Figure 16 shows the enthalpy plot generated for the $CaLa_2S_4$ specimens. Higher scatter than normal was seen above 1100 °F. Also the specimens tended to shatter due to thermal stresses when dropped from the higher temperatures. The calculated specific heat (slope of the curve in Figure 16) is shown in Figure 17. The material has a remarkably low specific heat typical of lanthanum compounds. It rises gradually through about 1200 °F then somewhat more rapidly thereafter. Tables 6 and 7 show the data obtained.

3.5 Thermal Conductivity

The thermal conductivity data generated on the $CaLa_2S_4$ material are shown in Figure 18. The initial data were well behaved at low

temperatures. However, at about 1000 °F on the first run the measured conductivity climbed dramatically and became erratic. Upon return to about 200 °F the data repeated fairly well for the one, lower temperature, specimen. Thermocouples could not be read on the other specimen and upon inspection it was seen that the thermocouples had been destroyed. New thermocouples were installed and cemented in place to protect them. The runs were repeated with similar results, this time obtaining an apparently valid data point at just in excess of 1000 °F. Again the thermocouples were destroyed. As with the expansion, a sulfurous order was detected. It was still present on the specimens after cooldown. No significant weight change was noted.

A third attempt was made this time changing to Pyroceram references with large (10 mil) thermocouple wire. It was planned to extrapolate surface temperatures as a backup after the failure of the internal thermocouples. The results showed permanent change in the conductivity of the material even at room temperature. Again the thermocouples were destroyed including those in the Pyroceram hot reference. Additional data were obtained by extrapolating temperatures to the surface for calculation of the temperature drop across the specimen. Figure 18 shows the apparently valid data only. A line value through the data up to 1000 °F is provided. Tables 8 through 11 provide the measured data.

4.0 SUMMARY OF THE DATA

Table 12 gives a summary of the data and compares the data with ZnS and ZnSe data from NWC Contract N60530-83-C-0031. The strength was somewhat lower and the modulus slightly higher than the ZnS. The comparison to ZnSe was similar except that at 70 °F the strength of the CaLa₂S, was

slightly higher. Specific heat was slightly lower than the comparison materials but all had low specific heats, the density was intermediate and, as expected from the moduli and densities, the sonic velocity was similar. The most remarkable differences were in the thermal conductivity and thermal expansion. The thermal conductivity is dramatically lower (a factor of about five) than either of the materials and the thermal expansion was a factor of about two higher.

This combination of properties will give a material that is not very good in thermostructural response, as was indicated by the fracturing of the heat capacity specimens, and therefore probably not a good candidate for high velocity IR dome applications. The reported optical and hardness properties may make this appropriate for a low velocity 8-12 micron window application where rain erosion is a concern.

5.0 REFERENCES

1. Personal Communication, R. Gentilman to J. R. Koenig, 1984

2. Koenig, J. R., Presentation to AMRAAM JSPO, 1980



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Figure 4. Assembly of Quartz Tube Dilatometer for Thermal Expansion Measurements



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Notes: 1. Break corners on all edges

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2. Surfaces to be ground parallel to length

Figure 6. Flexural Specimen Configuration





Strength versus Temperature for CaLa $_2S_4$







Figure 10. Failure Surface of F-10

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Figure 11. Probable Initiation Site in F-10



Figure 12. Higher Magnification of Probable Initiation Site in F-10





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Figure 15.







Table 1. Test Matrix for CaLa₂S₄

			Temperature	°F	
	7 0	500	1000	1500	1800
Flexural Strength and Modulus	5	4	4	4	2
Thermal Expansion	2				
Specific Heat	2				
Thermal Conductivity	2 —				
Density	x				
Sonic Velocity	x				

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Specimen Number	Density (gm/cc)	Velocity (in./µsec)	ρ V² (10 ⁶ psi)
RHP-145-HV1-26 #22 (TE)	4.591	0.201	17.46
RHP-220-HVI-47 #31 (TE)	4.558	0.201	17.33
RHP-155-HVI-30 #22-1 (TC)	4.591	0.204	18.05
RHP-207-HVI-41 #32 (TC)	4.608	0.202	17.69
RHP-155-HVI-30 #22-2 (SH)	4.604	0.202	17.68
RHP-155-HVI-30 #29 (SH)	4.609	0.204	18.05
F-1	4.616	0.201	17.55
F-2	4.631	0.201	17.61
F-3	4.632	0.201	17.62
F-4	4.643	0.201	17.66
F-5	4.641	0.201	17.65
F-6			
F-7	4.621	0.201	17.66
F-8	4.621	0.201	17.66
F-9	4.619	0.201	17.57
F-10	4.619	0.201	17.57
F-11	4.611	0.201	17.54
F-12	4.619	0.201	17.57
F-13	4.608	0.201	17.52
F-14	4.610	0.201	17.53
F-15	4.618	0.201	17.39
F-16	4.603	0.201	17.50
F-17	4.597	0.201	17.48
F-18			
F-19	4.603	0.201	17.50
F-20	4.588	0.200	17.27
F-21	4.602	0.200	17.33
F-23	4.586	0.201	17.44

Table 2. Density and Velocity of CaLa₂S₄ Specimens

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Table 3. Flexural Properties of CaLa₂S, IR Window Material

Bilinear Yield Point (in.)	ı	ı	ı	ŀ	ı			ı	ı	ı	ı			ı	•	ı	ı			0.0068	0.0044	0.0071	0.0080	0.0066	0.0015	0.0081	0.0052	0.0067
E _{II} (Test)	ı	ı	ı	ı	ı			I	1	ı	ı			ı	1	ı	۱			7.15	7.98	7.57	7.13	7.46	0.40	4.58	5.30	4.94
ral Modulus (Msi) E _{Init} (Test)	14.05	13.44	13.29	13.78	13.30	13.57	0.33	12.88	12.81	12.51	13.36	12.89	0.35	11.70	12.48	11.96	11.99	12.03	0.33	10.30	10.69	10.53	10.78	10.56	0.21	7.95	9.53	8.74
E Init (70 °F)	13.88	13.48	12.71	13.75	13.89	13.54	0.49	13.21	13.44	13.05	14.34	13.51	0.58	13.48	14.30	13.47	13.46	13.68	0.42	13.47	12.71	13.58	13.41	13.29	0.39	13.34	13.15	13.25
Ultimate Deflection (in.)	0.0086	0.0064	0.0073	0.0091	0.0092	0.0081	0.0012	0.0085	0.0079	0.0077	0.0047	0.0072	0.0017	0.0068	0.0077	0.0104	0.0120	0.0092	0.0024	0.0237	0.0164	0.0164	0.0212	0.0194	0.0036	0.0307	0.0251	0.0279
Strength (ksi)	7.97	5.67	6.45	8.20	7.89	7.24	1.11	7.24	6.62	6.39	4.05	6.08	1.40	5.11	6.23	8.23	9.34	7.23	1.91	12.40	9.20	9.35	11.85	10.70	1.67	10.40	9.71	10.06
Density (g/cm ³)	4.616	4.641	4.619	4.610	4.603	4.618		4.631	4.611	4.619	4.618	4.620		4.632	4.645	4.621	4.608	4.627		4.643	4.643	4.597	4.586	4.617		4.588	4.602	4.595
Temp (°F)	70	70	70	70	70		ation	500	500	500	500		ation	1000	1000	1000	1000		ation	1500	1500	1500	1500		ation	1800	1800	
Specimen Number	H	2	10	14	19	Average	Std. Devi	2	11	12	15	Average	Std. Devi	e	7	6	13	Average	Std. Devi	4	8	17	23	Average	Std. Devi	20	21	Average

Table 4. Thermal Expansion of CaLa2S4 - Encl 4 Measured in Quartz Dilatometer

14145/5° 788889

Corrected Specimen Ilnit	Elongation	10 ⁻³ in./in.		50.2514 gm .2498 gm	ı	0.0	0.85	2.60	4.33	5.99	7.70	9.57	11.43	13.24	15.20	0.03	
Unit Elongation Correction for Dilatometer	Motion	10 ⁻³ in./in.		Initial weight: (Final weight: 60	ı	0.0	0.05	0.12	0.19	0.25	0.30	0.35	0.41	0.46	0.52	0.0	
Observed	Elongation	10 ⁻³ in./in.		in. n.		0.0	0.80	2.48	4.14	5.74	7.40	9.22	11.02	12.78	14.68	0.03	
Observed	lotal Elongation	10 ⁻³ in.		ength: 3.0119 ath: 3.0102 i		0.0	2.40	7.48	12.47	17.30	22.30	27.78	33.20	38.50	44.20	0.10	
	ures - 'F	Average		Initial l Final len		70	200	400	600	800	1000	1200	1400	1600	1800	70	
	nen Temperat	Bottom				70	200	400	600	800	1000	1200	1400	1600	1800	70	
•	Specie	Top		808		70	200	400	600	800	1000	1200	1400	1600	1800	70	
		en Time	0 HVT_A7_31	u, mvi-1/-1/ 4- Encl-4 MOCOSO1_60-27		9:08	9:25	9:40	9:55	10:10	10:18	10:28	10:40	10:51	11:11	7:45	
		Specim)CC-4114	CaLa ₂ Si Bin:					37								

Table 5. Thermal Expansion of CaLa2St - Encl 4 Measured in Quartz Dilatometer

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	Specia	en Tempera	tures - °F	Observed Total	Observed Unit	Unit Elongation Correction for Dilatometer	Corrected Spectmen Unit	
Specimen Time	Top	Bottom	Average	Liongarion 10 ⁻³ in.	Liongation 10 ⁻³ in./in.	10 ⁻³ in./in.	10 ⁻³ in./in.	
RHP-145, HV1-26 #2	2							
CaLa ₂ 54 - Encl 4 Run: NOCO521-64-2	9 BPR		Initial lei Final lengt	ngth: 3.0191 ch: 3.0189 ir	ז.	Initial weight: : Final weight: 52.	52.2260 gm .2237 gm	
8:45	70	70	70	0.0	0.0	0.0	0.0	
9:00	200	200	200	1.70	0.56	0.05	0.61	
9:18	400	400	400	6.97	2.31	0.11	2.42	
9:34	600	600	600	12.63	4.18	0.17	4.35	
9:45	800	800	800	17.07	5.65	0.23	5.88	
9:57	1000	1000	1000	23.57	7.81	0.32	8.13	
10:10	1200	1200	1200	29.07	9.63	0.39	10.02	
10:20	1400	1400	1400	32.47	10.75	0.48	11.23	
10:30	1600	1600	1600	38.67	12.81	0.57	13.38	
10:47	1800	1800	1800	44.39	14.70	0.65	15.35	
8:00	70	70	70	- 0.11	- 0.04	0.0	- 0.04	

Table 6. Enthalpy of CaLa2S4 Measured in the Adiabatic Calorimeter

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Y	11	Nbove	32 °F Ref		33.40	69.60	91.98	47.90	13.93	89.05	
Enthé	Btu	Nbove	85 °F Ref.		28.76	64.84	87.23	43.45	9.34	84.41	
Enthalpy		HS V-C -L	Btu/lb		29.60	65.17	86.76	44.02	10.28	83.81	
Final	Weight of	Sample	gm		16.2710	16.2475	15.9695	16.1824	16.1887	16.1305	
Initial	Weight of	Sample	шſ		16.2762	16.2710	16.2475	16.1804	16.1926	16.1824	
Initial	Sample	Temp	٩F		413	807	1059	602	193	1051	
Change	in Cup	Tump	40		4.000	8.796	11.509	5.926	1.375	11.23	
Final	Cup	Temu	نا 0		75.364	81.261	90.348	78.174	74.739	91.913	
Initial	Cup	Temp	e e		71.364	72.465	78.839	72.248	73.364	80.682	
			Run	NOC0215	120	121	122	120	119	121	
			Specimen		HC-22-2			HC-29-2			

Table 8. Thermal Conductivity of CaLa2S4 - Encl 6 using Comparative Rod Apparatus with TM-Silica References

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Specimen and Time	Mean Temperature of Specimen •F	Thermal Conductivity of Specimen - ks Btu-in./hr-ft ² -f	ΔT through Specimen •F	Heat Flux through Specimen Btu/hr-ft ²	Heat Flux through Upper Reference Btu/hr-ft ²	Heat Flux through Middle Reference Btu/hr-ft ²	Heat Flux through Lower Reference Btu/hr-ft ²
RHP-155-HV1-30 #2; Run: NOC0746-16-4 Run: 1 Density: 4.602 g/	2-1 1 ^cm³		Initial th Final thic	uickness: 0. :kness: 0.5(.5050 in. I 353 in. F	nitial weight: 29. inal weight: 29.7	7989 gm 25 gm
8/20/84							
2:00 P.M.	249	60.6	19.00	566	639	534	596
2:30	249	9.07	19.00	565	639	533	596
8/21/85							
5:10 A.M.	433	10.23	26.63	893	1003	846	935
5:40	433	10.29	26.64	668	1004	854	934

Thermal Conductivity of CaLa₂S4 - Encl 6 using Comparative Rod Apparatus with TM-Silica References Table 9.

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Specimen and Time	Mean Temperature of Specimen	Thermal Conductivity of Specimen - ks Btu-in./hr-ft ² - ^e F	ΔT through Specimen •F	Heat Flux through Specimen Btu/hr-ft	Heat Flux through Upper Reference Btu/hr-ft ²	Hcat Flux through Middle Reference Btu/hr-ft ²	Heat Flux through Lower Reference Btu/hr-ft ²
RHP-207-HV1-41 #32 Run: NOC0746-16-4 Run: 1 Density: 4.608 g/v	E.		Initial th Final thic	nickness: 0.5	.5058 in. Ir 057 in. Fi	itial weight: 29. .nal weight: 29.76	,7898 gm 337 gm
8/20/84							
2:00 P.M.	169	9.77	16.99	543	639	534	596
2:30	169	9.73	17.03	542	639	533	596
8/21/85							
5:10 A.M.	316	9.82	26.74	829	1003	846	935
5:40	316	9.90	26.75	866	1004	854	934
10:45	787	13.49	39.17	1728	1988	1687	1962
11:15	788	13.57	39.13	1736	1989	1696	1963

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Table 10. Thermal Conductivity of CaLa₂S, - Encl 6 using Comparative Rod Apparatus with TM-Silica^References

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Specimen and Time	Mean Temperature of Specimen •F	Thermal Conductivity of Specimen - ks Btu-in./hr-ft ² -f	ΔT through Specimen •F	Heat Flux through Specimen Btu/hr-ft ²	Heat Flux through Upper Reference Btu/hr-ft ²	Hcat Flux through Middle Reference Btu/hr-ft ²	Heat Flux through Lower Reference Btu/hr-ft ²
RHP-155-HV1-30 #22 Run: NOC0746-19-4 Run: 2 Density: 4.570 g/c	1. Ĕ		Initial Final th	thickness: ickness: 0.	0.5053 in. 5054 in.	Initial weight: Final weight: 29	29.7425 gm).7222 gm
8/28/84							
9:30 A.M.	497	10.25	35.42	1611	1355	1121	1288
10:00	497	10.28	35.40	1192	1356	1121	1290

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Table 11. Thermal Conductivity of CaLa₂S₄ - Encl 6 using Comparative Rod Apparatus with TM-Silica References

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Flux Heat Flux Heat Flux bugh through through iference Middle Reference Lower Reference ir-ft ² Btu/hr-ft ²	Initial weight: 29.7837 gm Final weight: 29.7837 gm		55 1121 1288	56 1121 1290	30 2332 2676	32 2333 2676		- 692 786
Heat thro Upper Re Btu/h	3.5057 in. 5058 in.		135	135	263	263		I
Heat Flux through Specimen Btu/hr-ft ²	nickness: (;kness: 0.!		1147	1146	2384	2384		706
∆T through Specimen •F	Initial th Final thic		35.16	35.21	52.79	52.75		21.04
Thermal Conductivity of Specimen - ks Btu-in./hr-ft ² - ^e F			9.81	9.95	13.81	13.82		10.26
Mean Temperature of Specimen °F	c and		344	344	1031	1033		259
Specimen and Time	RHP-207-HVI-41 #32 Run: NOC0746-19-4 Run: 2 Density: 4.603 g/	8/28/85	9:30 A.M.	10:00	2:00 P.M.	2:30	8/29/85	6:00 A.M.

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Table 12. Summary of CaLa2S4 Mechanical and Thermal Data

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51	aLa2S4	ZuZ	ZnSe
70° Flexural Strength (psi)	7240	9300	6200
1000° Flexural Strength (psi)	7230	13600	7700
70° Flexural Modulus (10 ⁶ psi)	13.6	10.6	10.6
1000° Flexural Modulus (10 ⁶ psi)	13.7	6.7	7.6
Thermal Expansion to 1500 °F (in./in. x 10 ⁻³)	12.4	6.72	7.30
Specific Heat at 500 °F (Btu/lb-°F)	0.088	0.120	0.090
Thermal Conductivity at 500 °F (Btu-in./hr-ft ² - ^o F)	10.5	67	55
Density (gm/cc)	4.61	4.07	5.24
Sonic Velocity (in./µsec)	0.201	0.212	0.174

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APPENDIX A

FLEXURAL LOAD - DEFLECTION CURVES







1.260 × 3.750 m. Jun ... Stress | Schort Rates 10'000 PS / PD . 20 , 002 5 E = 2998.0 5 = 2598.0 (4.1×13.3) = 13.760 Her 1441 1444 -/4-85 Temperaturei 622 Kingelit had black 8.195 4. 7 ů Lettered Stopin Ofraction. RI Run World LA Sur : 454/ or) , 9. 48x1. 32 Logisting Direction. Beatmen fio. Meterials Deter 2 21 Ete 13.74P MA LUNDAL DECENTION DECENTION 15001. 812 J 10.21.05. ; A-5



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APPENDIX B

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SENSITIVITY STUDIES

Sensitivity Studies

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The sensitivity studies in Reference 1 were conducted on Pyroceram Dusing nominal properties available prior to the data set developed under that effort. The analysis was conducted on a radome structure at aerodynamic heating condition somewhat exceeding those anticipated for the IR windows for which the materials under this effort were evaluated. The analysis was conducted using state of the art aerodynamic heating codes, the Asthma code for indepth heating analysis and SAAS-III body of revelation finite element structural analysis. The material properties were modeled as elastic with equal tensile and compressive moduli which varied as a function of temperature.

Prior to initiating the testing effort, the peak stressed volumes and areas were calculated. These were comparable to the tensile specimens used in that effort. Note that the volume and surface areas are about the same as would be involved in the ID surface of an IR Dome despite the much larger size of the full radome as shown in Figure 1.

The sensitivity studies were conducted by varying the properties listed in Table 1 one at a time. As can be seen the most sensitive parameters in terms of stress generation were the stiffnesses and thermal expansion.



ERROR INTRODUCED	PARAMETER	ERROR IN Back face Temp. Rise	ERROR IN Max Theta Stress
5%	VELOCITY	10%	9%
5%	THERMAL CONDUCTIVITY	1.2%	1.2%
5%	THICKNESS	1.5%	1.5%
100%	EMISIVITY	0.1%	0.1%
1/8"	STRAIN GAUGE LOCATION		6%
1/8	THERMO COUPLE LOCATION	8.5%	
			6.2%
			3%
- 15% BASELINE	MODULUS		- 15%
+ 11 % BASELINE	MODULUS		+11%
+ 11 % CONSTANT F (T)	MODULUS		+4%
LINEAR/2 nd SLOPE	EXPANSION		-3%
- 30% LINEAR/2 nd SLOPE	EXPANSION		- 34 %
- 40% LINEAR/2 nd SLOPE	EXPANSION		- 47 %
- 50% LINEAR/2 nd SLOPE	EXPANSION		- 52 %

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