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**EFFECTS OF CW HIGH INTENSITY
LASER IRRADIATION ON CERAMIC
COMPOSITE RADOME MATERIALS**

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EFFECTS OF CW HIGH INTENSITY LASER IRRADIATION ON
CERAMIC COMPOSITE RADOME MATERIALS

Meyer, Fitzpatrick, and Whitcher

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ABSTRACT

Recently developed ceramic composite materials have been subjected to various CW high intensity irradiation and their responses were studied as a function of composition and processing parameters. The addition of high-purity silica, alumino-silicate and alumina fibers to slip-cast fused silica (SCFS) resulted in a moderate increase in flexural strength and elastic modulus but had no effect on the ablation rate. Under CW laser irradiation at $10.6 \mu\text{m}$ and an intensity of 2 kW/cm^2 , the fiber-reinforced fused silica samples failed by meltthrough with their penetration rates all being approximately 0.15 to 0.25 cm/sec. The damage area around the laser hole was melted into a glassy phase with the fibers completely dissolved.

Ablation rates of these composite materials were independent of fiber type and amount up to 8 kW/cm^2 and were density-dependent above 8 kW/cm^2 . The ablation rate of unreinforced fused silica was found to be equal to 0.5 mm/sec at 2 kW/cm^2 with or without a Mach 0.8 air flow across the sample.

The addition of ceramic fibers to SCFS did not degrade the laser response of the material but when a particular composite sample could not be made at a density level equivalent to that for SCFS, its penetration rate and ablation rate were greater and the materials would offer less ablation resistance.

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1. INTRODUCTION

A number of ceramic fiber-reinforced fused silica materials are being evaluated as an improved radome material with better rain erosion resistance to replace slip-cast fused silica (SCFS). Samples were prepared of slip-cast fused silica reinforced with three different ceramic fibers at several different levels of fiber content and exposed to laser irradiation. Unreinforced SCFS samples were also tested for a baseline curve. Thermal damage, either meltthrough time or ablation rate, was assessed as a function of sample composition and processing history.

2. EXPERIMENTAL PROCEDURE

Recent work by Meyer¹ has shown that adding small amounts of various ceramic fibers to SCFS yields a moderate increase in the flexural strength and elastic modulus over that for nominal SCFS. Additional samples were prepared for this program to evaluate the effects of laser irradiation on the reinforced SCFS. Three types of ceramic fibers were used: an alumina fiber, density 2.80 g/cc; an aluminosilicate fiber, density 2.56 g/cc; and a high-purity fused silica fiber, density 2.20 g/cc. The fibers were chopped in a one-gallon capacity blender until the L/d ratio was from 20 to 200. The chopped fibers were then dried and weighed and added to diluted fused silica slip in the appropriate amounts. Flat tiles, 15 cm by 15 cm, were slip cast on molds of US #1 Pottery plaster. As-cast thicknesses were from 1 to 2 cm. The samples were removed from the molds, dried at 90°C for 24 hours, and fired at 1225°C for 200 minutes.

Two separate types of laser test samples were prepared from the slip-cast tiles. For the meltthrough experiments the flat tiles were machined to a constant thickness to within 0.002 inch. For the ablation rate experiments small bars were machined to 3 mm by 3 mm by 75 mm long. Bulk densities of all samples were obtained using Archimedes' Principle. Flexural strengths were determined on an Instron Universal Testing Machine, Model #1115, using 4-point loading at a loading rate of 0.002 inch/minute. Modulus of elasticity data was obtained by measurement of the ultrasonic velocity in each material. A brief summary of these properties for all materials tested is shown in Table 1.

2.1 PENETRATION EXPERIMENTS

The time to melt through a sample of known thickness was measured and converted to a penetration rate. The laser used was continuous wave (CW) 2-kW CO₂ welding laser emitting radiation at 10.6 μ m. The beam was gaussian in cross section with two distinct major peaks. The time for burnthrough was measured by a timer which started when the laser beam burned through a thin metal strip in front of the sample and stopped when the beam burned through a similar metal strip behind the sample. There was no air movement across the face of the samples during the experiments.

2.2 ABLATION RATE EXPERIMENTS

Samples were submitted for determination of the one-dimensional rate of ablation utilizing a 30-kW CO₂ mixing laser.² The apparatus for measuring the ablation rates

1. MEYER, F. P. *Effects of Various Fiber Additions on the Properties of Slip-Cast Fused Silica*. Proceedings of the 15th Symposium on Electromagnetic Windows, Georgia Institute of Technology, Atlanta, Georgia, June 1980.
2. McCLEARY, R. C., WHITCHER, R. E., and BECKWITH, P. J. *A 30-kW CO₂ Mixing Laser*. MRL-R-751, AR-001-830, Materials Research Laboratories, Melbourne, Victoria, Australia, July 1979.

is shown schematically in Figure 1. Samples of fiber-reinforced SCFS were exposed to a series of laser power densities and their rate of ablation determined. The laser beam was passed through a beam integrator that produced a very good top hat cross section. At power densities of from 2 to 8 kW/cm² the beam was 6.25 mm by 6.25 mm, and at 10 to 18 kW/cm² the beam was approximately 4.50 mm by 4.50 mm. A wind tunnel was positioned so that an air flow of up to Mach 0.8 could be directed across the front face of the sample during testing.

Table 1. PROPERTIES AND LASER DAMAGE IN FIBER-REINFORCED SCFS COMPOSITE MATERIALS

Sample Composition	Bulk Density (g/cc)	Flexural Strength (psi)	Elastic Modulus (psi)	Penetration Rate (mm/sec)	Ablation Rate (mm/sec)
Slip-Cast Fused Silica	2.01	3490	$4.40 \cdot 10^6$	1.69	0.55
Fused Silica Fibers - 1 v/o	2.02	5200	3.73	1.58	0.55
5 v/o	2.02	7560	6.60	1.89	0.59
15 v/o	2.03	5310	4.59	1.89	0.80*
25 v/o	1.77	2670	--	2.66	0.73
Alumino-Silicate Fibers - 1 v/o	1.92	5330	4.33	--	0.73
5 v/o	1.94	6230	5.39	1.89	1.00
Alumina Fibers - 1 v/o	1.90	4430	3.73	--	0.62
5 v/o	1.68	1785	--	--	0.92
10 v/o	1.78	--	--	2.00	--
15 v/o	1.42	680	--	--	1.56

Note: All penetration rate and ablation rate data at 2 kW/cm² laser power.
All ablation tests done with Mach 0.8 air flow.

*Ablation rate sample density 1.94 g/cc.

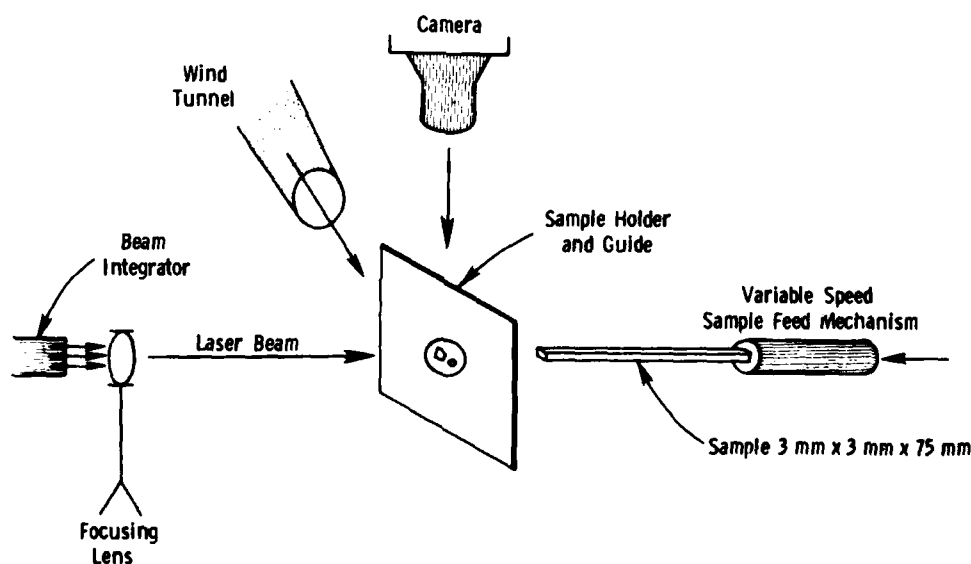


Figure 1. Apparatus for ablation rate measurement on the 30-kW laser facility.

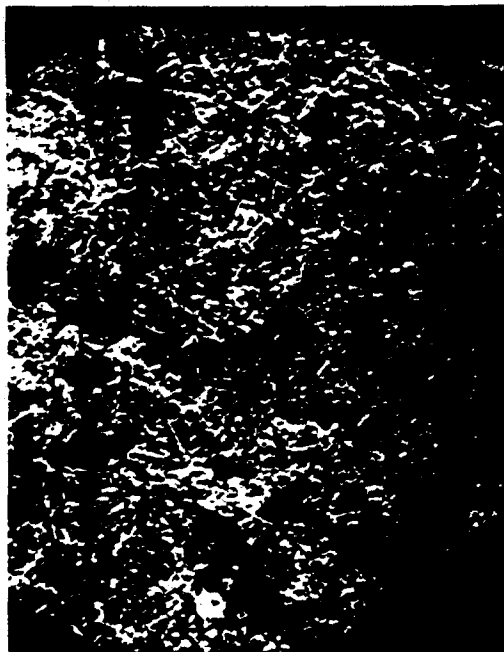
3. RESULTS AND DISCUSSION

A typical microstructure for the fiber-reinforced slip-cast fused silica materials is shown in Figure 2.

3.1 PENETRATION EXPERIMENTS

After exposure to the 2 kW/cm^2 laser beam, all the fiber-reinforced SCFS samples failed by meltthrough. A clean hole defined by the beam was made in each sample. Because of the gaussian cross section of the laser beam, a conical-shaped hole (referred to as the damage cone) was always produced. Several samples were sliced through the damage cone and photomicrographs were taken in and near the damaged areas. Figure 3 shows the area adjacent to the damage cone in a 10 v/o alumina fiber composite. The right-hand side of Figure 3a shows the glassy surface produced when the composite material melted. Figure 3d clearly indicates that the material has melted to form a clear glassy phase with no porosity. No fibers are visible in this region as they have melted also. A transition area exists around the damage cone in which the energy from the laser beam has begun to affect the microstructure of the material. Figure 3c depicts this area in which fibers have begun to dissolve and large pores have begun to form. Closer examination of a similar transition area in a fused silica fiber-reinforced SCFS, Figure 4, reveals that the fibers are beginning to dissolve into the matrix and lose their fiber identity.

Penetration rate for each sample tested at 2 kW/cm^2 is given in Table 1. In general, the penetration rates are higher for the lower density materials. Of particular interest are the fused silica fiber-reinforced SCFS composites. The addition of up to 15 v/o fused silica fibers increased the penetration rate by only 12%. Due to the experimental setup, it was difficult to obtain an exact time for penetration.



Mag. 200X



Mag. 500X

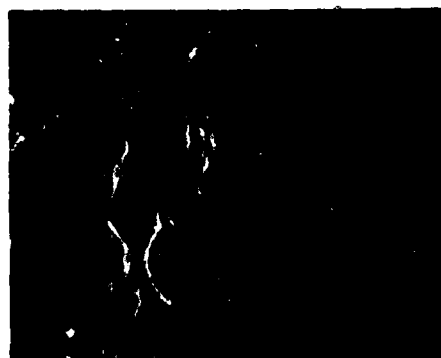
Figure 2. Fused silica fiber-reinforced fused silica - fiber content 25 v/o, fiber diameter $10 \mu\text{m}$.



(a)



(b)



(c)



(d)

Figure 3. Effects of CW high intensity laser irradiation on 10 v/o alumina fiber-reinforced SCFS.

- a. Cross-sectional view, damage cone on right, Mag. 50X
- b. Unaffected area, Mag. 500X
- c. Transitional area, Mag. 1000X
- d. Glassy area in damage cone, Mag. 1000X

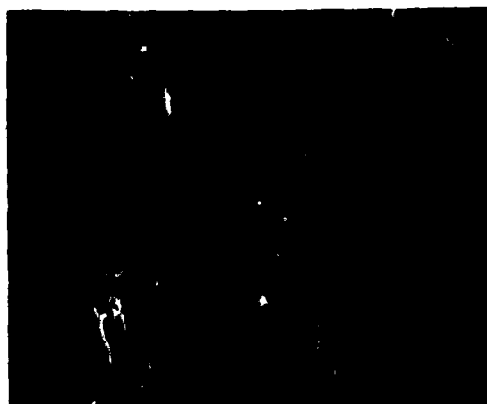


Figure 4. Effects of CW high intensity laser irradiation on fused silica fiber-reinforced SCFS. Mag. 2000X

Because of the gaussian beam profile only a very small pinhole was produced when the beam first burned through the sample. If the metal timing strip was offset slightly, the laser beam would miss it at the exact time of penetration and an additional fraction of a second would be needed before the beam was large enough to irradiate the metal strip. The 12% increase in penetration rate can therefore be considered as within experimental error. An increase in penetration rate of 57% seen in the sample containing 25 v/o fused silica fibers is definitely attributed to the lower density of this material.

3.2 ABLATION RATE EXPERIMENTS

All the ablation rate data reported herein utilized a Mach 0.8 air flow across the face of the sample during testing. Figure 5 shows the ablation rate as a function of power density level for the fused silica fiber-reinforced SCFS samples. Up to a power density level of approximately 8 kW/cm² there is little difference in the

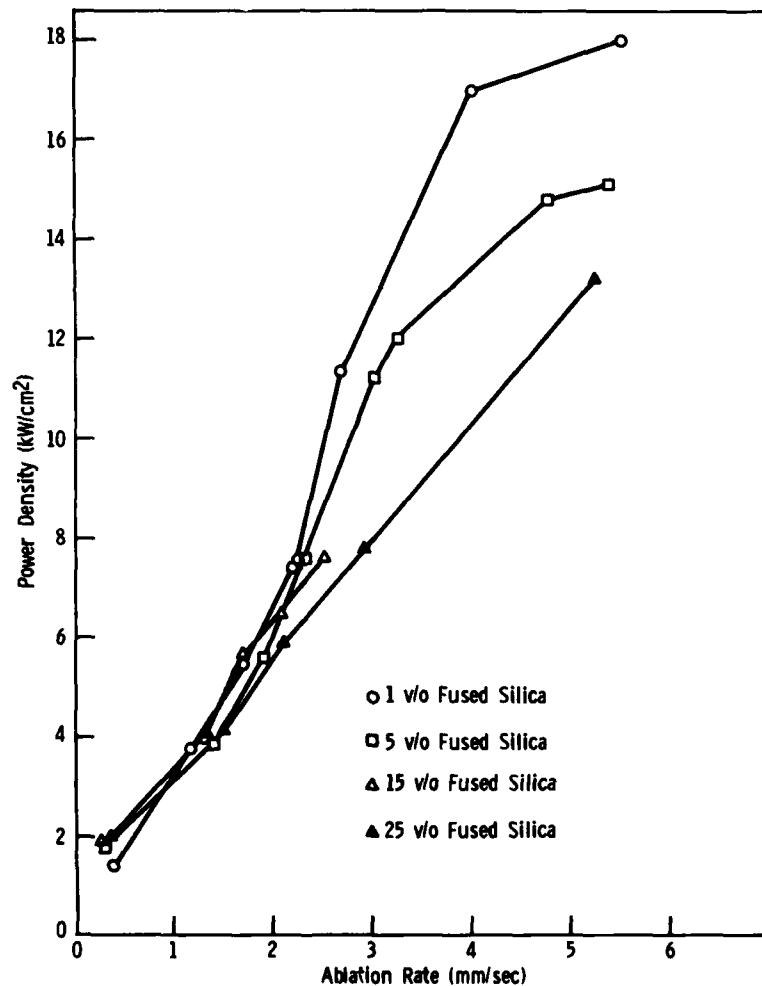


Figure 5. Ablation rate versus power density at Mach 0.8 for fused silica fiber-reinforced fused silica composites.

ablation rates of these composites. Even the low density sample containing 25 v/o fibers did not have a significantly higher ablation rate than the other more dense composites. At higher power density levels the samples were completely overmatched by the laser beam. Ablation, particularly at Mach 0.8, is difficult to measure and the spread in the data for samples containing 1 v/o and 5 v/o fibers, both having very similar densities, must be caused by the accuracy of the experiment. These samples, however, did have higher ablation rates than the lower density sample containing 25 v/o fibers.

Figure 6 shows the ablation rates versus power density level for three SCFS composite materials each containing 5 v/o of a different fiber. Theory would predict that the more refractory alumina and alumino-silicate fiber composites would have lower ablation rates than fused silica fiber composites at a given power density. This is not the case, however, as the data shows just the opposite to be true. The differences in ablation rates are attributed to the differences in densities of the samples and not to the type of fiber that has been added. This fact is further illustrated in Figure 7 where three samples of different composition yet very similar density level are shown to have almost identical ablation rates.

Figure 8 shows the ablation rate as a function of power density level for SCFS at Mach 0.8. The material had a density of 2.01 g/cc. The data shown here agrees quite nicely with a similar curve presented by Viechnicki et al.⁷

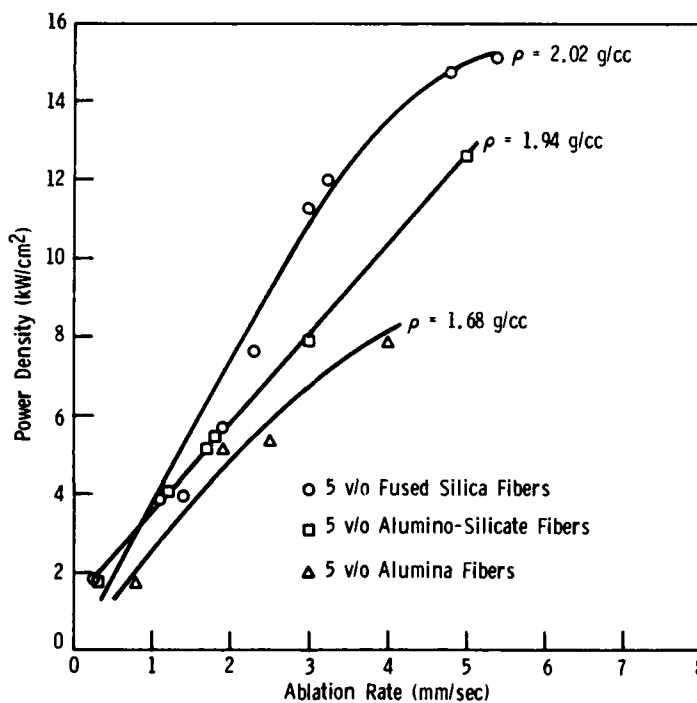


Figure 6. Ablation rate versus power density at Mach 0.8 for fused silica composites reinforced with 5 v/o ceramic fibers.

3. VIECHNICKI, D. J., MEYER, F. P., and PETSCHKE, C. *Response of Fused Silica and Silicon Nitride to HEL Irradiation*. Army Materials and Mechanics Research Center, AMMRC TR 78-31, July 1978.

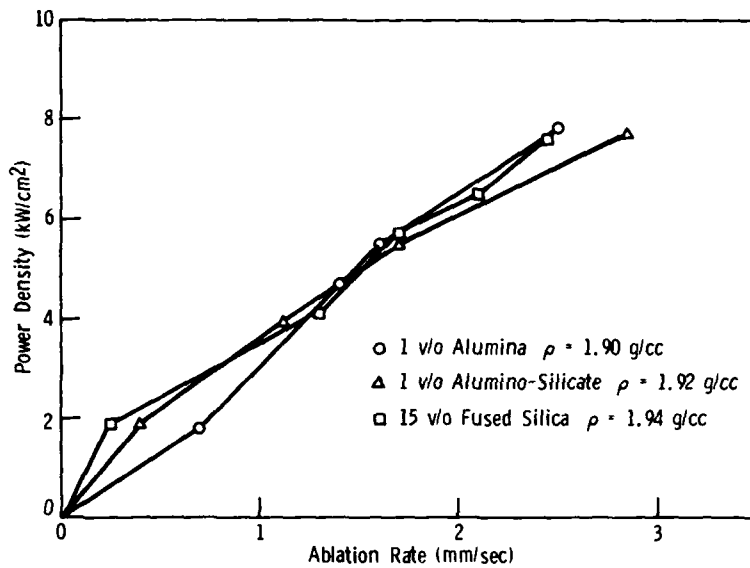


Figure 7. Ablation rate versus power density at Mach 0.8 for three different fused silica composites at similar density levels.

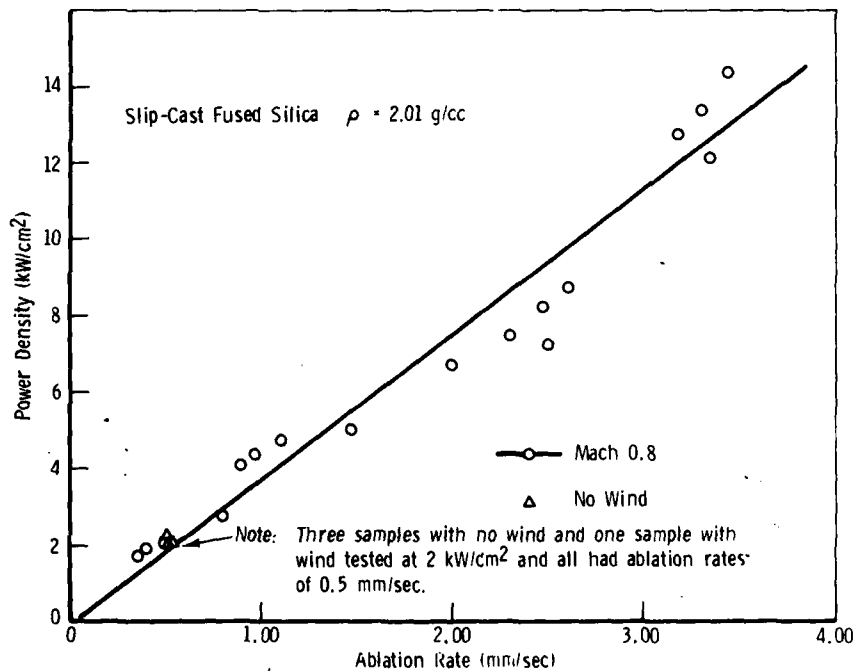


Figure 8. Ablation rate versus laser power density for slip-cast fused silica.

3.3 COMPARISON OF PENETRATION VERSUS ABLATION

In both this test program and the one by Viechnicki et al., a well-defined top hat beam was used. This is one of the major reasons for the lower ablation rates shown in Table I. The penetration experiments used a gaussian beam and the time of penetration was determined when the first pinhole of light emerged through the backside of the sample, the area of the entrance hole in the sample being many hundreds of times that of the exit pinhole. Approximately one-third of the volume of material was removed as would have been removed had the full diameter of the beam burned completely through the sample. Also in the penetration studies the laser beam was much smaller than the sample and because of the low thermal conductivity of SCFS the heat from the beam would be more concentrated and would tend to increase the penetration rates. Using a Mach 0.8 air flow across the sample would seem to help dissipate some of the heat from the beam and reduce the ablation rate. This was not the case, at least at 2 kW/cm² power density. Three samples of SCFS were irradiated in the ablation test apparatus without any air flow. An average ablation rate of 0.5 mm/sec was obtained which is identical for the SCFS samples tested with Mach 0.8 wind.

4. CONCLUSIONS

1. The failure mechanism for SCFS and all the fiber-reinforced SCFS composites is melt through with approximately 33% of the material in the path of the beam removed by vaporization.
2. For all the fiber-reinforced SCFS composites tested, the laser beam melted each material, regardless of fiber type or amount, into a glassy phase in which no fibers were visible.
3. At 2 kW/cm² SCFS composites containing from 0 to 15 v/o fused silica fibers showed no difference in their penetration rates. A sample containing 25 v/o fibers and having a density of 1.77 g/cc showed a 57% increase in penetration rate.
4. Ablation rates for power density levels of from 2 to 8 kW/cm² of SCFS composite materials are virtually unaffected by fiber type or amount and are also independent of density.
5. At power density levels of from 10 to 18 kW/cm², the ablation rates for fiber-reinforced SCFS were also independent of fiber type and amount but were a function of bulk density, the lower density samples having a higher ablation rate.
6. At 2 kW/cm² the ablation rate of unreinforced SCFS is approximately 0.5 mm/sec, both with a Mach 0.8 air flow and without.
7. In general, it can be concluded that the addition of ceramic fibers to SCFS did not in itself degrade the laser response of any of the materials. However, if a particular fiber-reinforced SCFS composite is to be considered a viable candidate to replace SCFS, it must be fabricated with a density level very similar to that of SCFS in order to give equal thermal response.

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illus-table, D/A Project 1L162105AH84,
AMCMS Code 612105.H840011

Recently developed ceramic composite materials have been subjected to various CW high intensity irradiation and their responses were studied as a function of composition and processing parameters. The addition of high-purity silica, alumina-silicate and alumina fibers to slip-cast fused silica (SCFS) resulted in a moderate increase in flexural strength and elastic modulus but had no effect on the ablation rate. Under CW laser irradiation at 10.6 μm and an intensity of 2 kW/cm², the fiber-reinforced fused silica samples failed by meltthrough with their penetration rates all being approximately 0.15 to 0.25 cm/sec. The damage area around the laser hole was melted into a glassy phase with the fibers completely dissolved. Ablation rates of these composite materials were independent of fiber type and amount up to 8 kW/cm² and were density-dependent above 8 kW/cm². The ablation rate of unreinforced fused silica was found to be equal to 0.5 mm/sec at 2 kW/cm² with or without a Mach 0.8 air flow across the sample. The addition of ceramic fibers to SCFS did not degrade the laser response of the material but when a particular composite sample could not be made at a density level equivalent to that for SCFS, its penetration rate and ablation rate were greater and the materials would offer less ablation resistance.

Amy Materials and Mechanics Research Center,
Watertown, Massachusetts 02172
EFFECTS OF CW HIGH INTENSITY LASER IRRADIATION
ON CERAMIC COMPOSITE RADOME MATERIALS -
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Russell E. Whitcher
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