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LASER SHOCK EFFECTS ON STRESSED STRUCTURAL MATERIALS - EXPERIMENTAL RESULTS

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ABSTRACT

The effects of intense single pulses of 1.06 μm radiation on structural composite materials have been investigated. Fluences in the 1000 - 3000 J/cm^2 range were delivered in single pulses with 20 ns pulse widths (FWHM) to thermal coupon and tensile bar type samples in vacuum. Materials studied included Kevlar/epoxy, fiberglass/epoxy, and graphite epoxy uniaxial composites in coated and uncoated conditions. Diagnostics were employed to assess energy partitioning in the interaction and stress wave histories in the material. Post-test sample examination and strength tests were conducted on the tensile bar samples. The diagnostics indicated that most of the beam energy goes into a very hot plasma (300,000 K) which drives a shock wave into the material. The shock wave has a peak amplitude of about 30-40 kbars and attenuates as it propagates through the sample. A synergistic damage effect was discovered wherein the sample fails in tension due to addition of the sample preload stress and the axial component of stress due to the shock wave reflected from the rear surface of the sample. Details of the beam energy partitioning and strength degradation in the samples will be presented.

INTRODUCTION

During the past several years, experimental laser effects research has been in progress to understand the effects of single short-wavelength laser pulses on composite materials in vacuum. In most cases, previous studies have been oriented toward understanding fundamental interactions in unstressed samples at low incident laser fluences (< 200 J/cm^2) and emphasized measurement of integrated response characteristics such as thermal coupling, impulse coupling, and effective heat of ablation, Q*. Recently, research was undertaken to extend some of these basic results to the more realistic case of laser interaction with stressed samples and, in particular, to look carefully at laser-induced shock enhancement of material damage. This paper summarizes results of an 18-month effort to develop an understanding of the damage and degradation of complex structural materials subjected to intense single laser pulses (20 ns, 1.06 μm) in vacuum while they are under preload stress conditions which might be typical of actual applications. (ref. 1) In the high fluence regime investigated, high plasma pressures generate stress waves which have damaging effects in addition to the simple removal of material by vaporization. Under certain conditions, laser induced stress wave components were found to add to the preload stresses to produce enhanced damage effects.

EXPERIMENTAL PROCEDURE

Three series of laser experiments were conducted in pursuit of an understanding of the effects produced by intense single pulses on composites. These included preliminary interaction investigations, shock wave pressure measurements in coupon tests, and preloaded tensile bar exposures. Materials investigated were Kevlar/epoxy, fiberglass/epoxy, and graphite/epoxy uniaxial structural composites. A crossply Kevlar/epoxy composite was also studied. Characteristics of the composites employed in the tests are presented in Table I. The first two test series utilized 1.5 cm x 1.5 cm x 1.5 mm square coupon targets while the last test series employed 30 cm x 1.5 cm x 1.5 mm tensile bars (with glued aluminum grip tabs) made from the same composite material.

In the initial tests, square coupon samples were exposed to assess plasma characteristics and damage phenomenology for the fluence range of interest. All samples were coated with a black absorbing coating (flat black Krylon paint) to provide uniform front surface boundary conditions from material to material. A 25 μm thick coating was adopted as a standard after a 12 μm layer was found to ablate away at 2800 J/cm^2 . In tests dedicated to pressure measurements, the square coupons were instrumented with quartz and manganin pressure transducers. In addition to the standard black coating condition, samples were exposed with an additional transparent overlay (shock enhancement coating) and with no coating at all. Tensile bar samples were exposed in subsequent tests with and without preload stress equivalent to 50-60 percent of ultimate strength. Fluence ranged from 2200 - 2600 J/cm^2 and all three coating conditions were studied.

Table I. Composite Test Sample Characteristics

Material*	Weight Percent Fiber	Volume Percent Fiber	Fiber Type	Fiber Orientation (degrees)
Kevlar/Epoxy	55-58	58	Dupont Kevlar 49 Aramid Yarn	0
Fiberglass/Epoxy	76-80	63	Owens Corning S-2 Glass P283A-X-1-750	0
Graphite/Epoxy	62-65	53	Hercules Type AS4-12K	0
Epoxy	0	0	None	0
Kevlar/Epoxy Crossply	55	56	Dupont Kevlar 49 Aramid Yarn	0-90-0-90-0
Material received 1.5 mm thick, 15 mm wide.				

* All materials procured from Quintus Enterprises, Inc.
Epoxy matrix is made from Epon 828 resin and Tonox 60/40 hardener.

A 6 mm diameter circular irradiance spot with a “flat-top” uniform fluence distribution was employed in all tests. A brief description of the target diagnostics employed follows.

<u>Plasma Microcalorimeters</u>

Two microcalorimeter disks were set up at a distance of 22.5 cm from the sample surface to sense the total time-integrated flux of reradiated energy and plasma kinetic energy at different angles relative to the target. Each disk was fabricated from aluminum and was 1.27 cm in diameter by 0.45 mm thick. The front surface was coated with flat black Krylon paint to provide absorption over a broad spectral range. The equilibrium temperature was sensed with a chromel/constantan foil thermocouple (RDF-20101-2) bonded to the rear surface of the disk. A reference thermocouple bonded to a massive portion of the disk holder was wired in opposition to the disk thermocouple to compensate chamber temperature drift. The disk was suspended from the holder with 0.3 mm diameter hypodermic tubing to minimize thermal conduction loss during equilibration. Filters employed to sort various spectral contributions to the energy received included a 3.0 mm thick quartz disk, a 2 µm thick mylar film, a 1.06 µm narrow band interference filter, and a 2.0 mm thick magnesium fluoride window. The disk calorimeters were adequate for sensing the radiation component of the energy, but were found to be inadequate in capturing the kinetic energy of the expanding plasma. A cone-shaped microcalorimeter was designed to trap plasma components more effectively in subsequent test series. The hollow cone was machined from aluminum and was suspended and instrumented in a manner similar to that of the disk microcalorimeter.

<u>Plasma Expansion Speed Sensor</u>

A very simple plasma probe consisting of a 1.2 cm diameter copper disk was placed at a distance of 22.5 cm from the target to measure the time of flight of the expanding plasma. The disk was wired directly to an oscilloscope input through a 50 Ohm coaxial cable which was terminated in 50 Ohms at both ends. Following an initial signal corresponding to plasma induced photoelectron emission, the probe output dropped rapidly and then rose sharply as the plasma front arrived at the probe surface.

<u>Plasma Emission Radiometer</u>

A radiometric measurement was performed for all laser tests using a u.v. enhanced silicon photodiode with broad band transmission filters for blue/green radiation. The photodiode assembly was located 1700 mm from the target plane along a line of view which was within a few degrees of normal incidence. The filter set consisted of a 2.0 optical density neutral density Wratten filter and 1.06 μm dielectric mirror followed by two interference filters centered at 450 nm and 500 nm, respectively. The collecting lens was a BK-7 plano-convex 52-mm-aperture lens with a 150-mm nominal effective focal length. The plasma brightness temperature was calculated from the measured detector power and filter characteristics assuming a uniform black-body radiator of 6 mm diameter.

<u>Plasma Emission Spectroscopy</u>

Since the plasma emission radiometry was employed to attempt to determine a plasma temperature, it was of interest to explore the validity of the black-body assumption used in the analysis. The black-body temperatures are actually brightness temperatures for the blue-green spectral region and represent lower bounds on the true temperatures. Time-integrated spectra were recorded in an attempt to provide some information on the nature of the emissions in this region of the spectrum. The survey spectra were recorded on Polaroid film with a Spex 3/4-meter Cherney-Turner spectrometer. Time-integrated, spatially-resolved spectra were recorded during the tests over the 230-500 nm spectral range. Strong continuum radiation was observed near the surface with ionized carbon lines appearing away from the surface. Because of the high temperature measured in the tests, it appears that x-ray spectroscopy would be more suitable for plasma characterization. The highest ionization level observed was CIV at 253 nm (upper level 55.8 eV).

Quartz Pressure Gage Measurements

Nine of the laser tests employed quartz pressure gages for diagnosing shock wave transients. The gages were made by Valpy-Fisher and were all of the same design, consisting of a 7.0 mm diameter x-cut quartz disk with electrodes in a shorted guard ring configuration. The disk was 1.0 mm thick with 3.0 mm diameter central rear electrode (2.38 kbar/V, 175 ns write time). The guard ring electrode width was 2 mm which permits response close to that of a shunted guard ring gage for moderate stresses. For some tests, the standard black coating and the standard black coating with a transparent overlay coating (enhanced black coating) were applied directly to the front surface of the quartz gage (gold electrode). For other tests, the gages were coupled to the rear surface of the 1.5 mm thick samples with a thin silicon grease layer and light clamping pressure.

Manganin Pressure Gage Measurement

Four tests employed thin foil manganin pressure gages manufactured by Micro Measurements (No. LM-SS-125AD-100). The active area was a grid 3.2 mm x 3.2 mm and the manganin foil thickness was 4 μm. The foil was backed by a 25 μm thick fiberglass/epoxy layer. The gage was bonded (foil side in) to the sample with an approximately 50 μm thick epoxy layer. A grounded aluminum foil shield (25 μm thick) was bonded to the gage backing with an approximately 50 μm thick epoxy layer to block plasma associated noise. Finally, the foil was coated with the standard black paint coating.

SIGNIFICANT EXPERIMENTAL RESULTS

The laser tests described above have led to an improved understanding of the interaction and damage phenomenology associated with high fluence single pulse laser exposures of structural composite materials. Selected significant results are summarized in the following subsections.

Energy Partitioning

To provide a basis for future material response modeling, it is essential that the beam/target interaction physics be understood sufficiently for the fluence regime of interest to permit selection of appropriate models. Based on the plasma and target diagnostic results, a consistent picture has evolved for the simplest case of a surface absorber (standard black coating). A small amount of material is vaporized (~ 2 mg/cm^2 at 2500 J/cm^2) and raised to very high temperatures, 300,000 K, as determined by the radiometer measurement. The presence of triply ionized carbon (CIV) is consistent with temperatures near 10^5 K. The streak photography and plasma speed detector are consistent with a plasma expansion rate of 2.5 cm/μs at 2500 J/cm^2. Relating this value to plasma temperatures will require numerical calculations considering multiple ionization and two-dimensional expansion. An upper bound on temperature can be determined by noting the root mean square ion speed in the initial plasma which is assumed to be in local thermodynamic equilibrium at temperature, T,

$$v_i = \left[\frac{3kT}{m_i}\right]^{\frac{1}{2}} ,$$

where m_j is the ion mass (assumed to be C^{12}) and k is the Boltzmann constant. In the expansion, electrons transfer some of their energy to the ions. Therefore, the measured plasma front speed at large distance must be at least that given by v_j. If the measured speed is substituted into the above relationship for v_j, T is found to be 3.0 x 10^5 K. Fortuitous agreement is found with the radiometer result since lower temperatures will result in the same expansion rates when the expansion is correctly modeled. As an example, one-dimensional expansion of a singly-ionized plasma has been modeled and the plasma front speed is found to be three times the ion sound speed,

$$v = 3\left[\frac{kT_e}{m_i}\right]^{\frac{1}{2}}$$

If the measured expansion velocity is substituted into this relationship, T is found to be 1.0 x 10^5 K. It is concluded that the peak temperature is in the 100,000 - 300,000 K range. Better correlation of the data will require numerical modeling and refinement of the radiometric and spectroscopic measurements.

Microcalorimeter measurements were employed to assess the partitioning of the beam energy after the interaction. Data for typical tests with standard black coatings are presented in Table II for various filters. First it is noted that negligible reflection of the 1.06 μm laser radiation was observed in the tests. Thus, it is assumed that all beam energy ends up in the target, reradiation, or plasma energy. The thermal energy retained in the target was found to be 0.2 percent so it is clear that most of the energy resides in reradiation and plasma energy.

Table II. Typical Microcalorimeter Results for 2500 J/cm^2 Pulses on Standard Black Coatings

Shot No.	Filter	Passband (μm)	Fraction of Beam Energy per Steradian at 10° Incidence Angle	Calorimeter Type
4036	Fused Silica	0.2 - 3.5	0.004	Disk No. 2
7221	Magnesium fluoride	0.115 - 7.5	0.008	Disk No. 2
9322	None	All radiation + some particles	0.113	Disk No. 2
9322	None	All radiation + particles	0.392	Cone
7236	1.06 μm	$\Delta\lambda$ = 0.012	$<$ 0.001	Disk No. 2

The most significant microcalorimeter results turned out to be those for the cone and disk microcalorimeters under identical conditions (Shot 9322). The efficiency for collecting radiation was believed to be the same for both microcalorimeters; however, the sticking coefficient and trapping efficiency for particles striking the disk was not very high as indicated by almost a fourfold increase in measured energy per steradian for the cone. The energy trapped by the cone corresponds very closely to that expected if all the beam energy is distributed by a cosine law, $f = f_o \cos\theta$, where f_o is the fraction per steradian at normal incidence viewing angle (f_o = 0.32/steradian). If the disk is assumed to record only the radiation, then an independent check on the temperature can be made. Assuming all radiation is absorbed by the disk, a black body temperature of 250,000 K is calculated based on an effective radiating time of 40 ns. The actual radiation spectrum is time dependent and fairly complex at short wavelength, but this simple approximate result is consistent with the radiometer and plasma speed data. Numerical calculations will be required to achieve better correlations of the data. It was planned that broad-band filters would give additional data on spectral content; however, the amount of energy getting through the magnesium fluoride was so small that measurement errors distorted the results. The fraction of beam energy per steradian radiated within the pass band of this filter at 250,000 K was calculated to be less than 0.001 compared to a measured value of 0.008. It is conjectured that some of the energy absorbed in the filter was reradiated to the microcalorimeter.

Thus, it appears that for the 2500 J/cm^2 condition, < 30 percent of the energy is radiated and > 70 percent of the energy ends up as energy in the plasma. An estimate of the kinetic energy in the plasma can be made by assuming that all of the coating under the beam spot which is vaporized (2 mg/cm^2) ends up at the measured plasma speed (2.5 x 10^6 cm/sec). This results in 620 J/cm^2 which is only 25 percent of the 2500 J/cm^2 input. This mass is only about one fourth of the total measured mass loss, but the remaining mass comes from around the outside of the spot and has a wide distribution of terminal velocities which may be much less than 2.5 x 10^6 cm/sec. While this slower component accounts for some of the discrepancy, it is believed that there is another component of plasma energy that may be sensed by the cone microcalorimeter. It is estimated that about 150 eV per ion must be absorbed by the plasma during heating to 250,000 K (Z = 4). Because the expansion is sudden, not all of this potential energy is recovered in reradiation or plasma kinetic energy before expansion and in the low density expanded plasma, recombination has a low probability. Some of the recombination energy is recovered in the cone calorimeter and appears as heat. At 150 eV per ion, the ionization energy for 2.0 mg/cm^2 of carbon is 2400 J/cm^2 which is most of the 2500 J/cm^2 stored as potential energy initially. Some fraction of this energy plus the plasma kinetic energy should account for the total plasma energy sensed by the cone calorimeter. To fully separate the components of plasma energy, the ionization state of the expanded plasma would have to be measured.

Shock Pressure Levels

Results of the pressure gage measurements are summarized in Table III. The peak pressure at the front surface was found to be near 40 kbar for the standard black coating subjected to about 2500 J/cm^2 as measured with both quartz and manganin gages. The rise time recorded by the quartz gages was extremely fast (~ 4 ns) while the manganin gages which were farther from the surface exhibited about a 10 - 15 ns rise time. The quartz gage response was indicative of a plasma ignition occurring in the vapor flow created by the very early part of the laser pulse. A low level ablation pressure was sensed, followed by a small dip as flux to the surface was cut off at plasma ignition. A shock wave traveled back up the vapor flow, struck the surface, and was sensed by the quartz gage. These details are washed out by the time the stress wave reaches the depth of the manganin gage. The magnitude of the measured peak pressure is in order of magnitude agreement with estimates based on the measured temperatures. If it is assumed that peak plasma heating occurs in the vapor flow where the electron density reaches the critical value (plasma frequency equals laser frequency), then the electron pressure is 34 kbar and the total pressure (including ions) is 43 kbar assuming an ionization state of 4.

The peak front surface pressure in the case where a transparent overlay coating was added to the black coating (enhanced black coating) was believed to be much greater than in the standard black coating case. Unfortunately, very few enhanced coating data were obtained in the tests. The result with the quartz gage (> 80 kbar) was out of the linear range of the gage and only one manganin gage record (50 kbar) was obtained. Based on the extent of damage to the quartz gage and previous experience with the overlay technology, it is conjectured that 80 - 100 kbar was achieved in the quartz gage case. The lower value for the manganin gage case may have resulted from greater attenuation in the gage package.

TABLE III. Summary of Pressure Gage Results

Gage	Shot No.	Fluence (J/cm^2)	Material	Coating[c]	Surface	Peak Pressure (kbar)	Measured Shock Speed (cm/μsec)
Q-1	7222	2336	Quartz	SB	Front	38.0	--
Q-2	7223	2575	Quartz	SB	Front	(48.0)[a]	--
Q-3	7224	2746	Quartz	EB	Front	$\gtrsim$ 80.0[b]	--
Q-6	7227	2186	Fiberglass/epoxy	SB	Rear	5.7	0.36
Q-7	7228	2168	Graphite/epoxy	SB	Rear	8.6	0.33
Q-8	7229	2189	Kevlar/epoxy (crossply)	SB	Rear	8.9	0.30
Q-9	7231	2325	Fiberglass/epoxy	UN	Rear	4.3	0.33
M-1	7235	2414	Kevlar/epoxy	SB	Front	39.0	--
M-2	7236	2675	Kevlar/epoxy	SB	Front	38.0	--
M-3	7237	2811	Kevlar/epoxy	EB	Front	50.0	--

(a) Smoothed peak.

(b) Off-scale in nonlinear regime.

(c) SB = Standard Black Coating, EB = Enhanced Black Coating, UN = Uncoated.

Rear surface pressure transients were recorded in four tests with quartz gages bonded to the rear surface of samples with a thin layer of silicone grease. In all cases, pressures were within the linear range of the gages. For the standard black coatings, the peak rear surface pressures (as sensed in the quartz) ranged from 5.7 to 8.9 kbar for the three materials. These values must be corrected for the acoustic impedance mismatch at the interface to determine the peak stress in the sample material which would appear at that point if the sample were infinitely thick. A detailed simulation was performed by NRL for the Kevlar case and excellent agreement was achieved with the measured transmitted pulse shape. The ratio of sample stress to peak quartz stress was found to be 0.6 in the calculation.

One material (fiberglass/epoxy) was subjected to the same test conditions with no coating on the front surface. The distribution of the beam energy within the material led to a 25 percent lower peak pressure on the rear surface, but the duration of the pressure pulse was probably considerably longer in the uncoated case (higher total impulse). The gage record was limited to 175 ns by transit time through the quartz and, therefore, duration of the pressure pulse was not measured.

Tensile Strength Reduction

Examination of the post-test strength results for the Kevlar/epoxy showed that those samples having the standard black (SB) coating sustained the least degradation of local tensile strength (0 to 30 percent). There were not sufficient data to speculate whether the loaded or unloaded condition experienced the greater effect on tensile strength. The other two conditions, uncoated and coated with enhanced black (EB) coating, both showed significant reductions in local tensile strength. The strength of uncoated, unloaded samples under the laser spot decreased 75 to 100 percent from the unshocked range. The damage in the enhanced coated samples was primarily severing of the fibers under the irradiated region. In the unloaded samples, this damage was confined to the irradiated spot, but in the loaded samples, additional damage in the form of longitudinal cracking extended along the gage length. This extended damage may be attributed to the energy release associated with severing the stressed fibers. For the EB coating case, the loaded condition definitely showed the largest decrease in tensile strength (100 percent). One sample ruptured during laser exposure. The unloaded samples exhibited local tensile strength loss of 50 to 70 percent.

The uncoated fiberglass/epoxy samples all showed a significant decrease in tensile strength. The uncoated, loaded samples had a residual tensile strength at or just above the preload stress (100 percent loss of strength under laser spot). In one case, the sample failed during laser exposure i.e., below the preload stress. The SB coated samples all showed higher tensile strengths than the uncoated samples. Local strength loss ranged from 10 to 90 percent.

These results suggest that a coating protects the underlying composite from the direct, thermal effects of the irradiation. In the uncoated samples, these direct effects cause vaporization and removal of epoxy, fiber breakage, longitudinal and transverse splitting, and thermal damage. In the SB coated samples, the damage can be caused only by the stress wave. There was no significant amount of fiber breakage or loss of epoxy matrix material in this case. Some longitudinal splitting was visible on the back surface. This type of damage does not decrease the tensile strength to the degree that the thermal effects do. However, the EB coating, having the thin transparent layer that provides an enhancement of the magnitude of the shock wave, increases the stress wave effects to the point where the mechanically induced damage significantly reduces the residual strength. This result was seen only in the Kevlar/epoxy samples since EB coated fiberglass/epoxy samples were not investigated. The graphite/epoxy tensile results showed no definitive effects of laser damage on tensile strength. All the samples were uncoated, but the graphite fibers withstood the direct thermal effects of the irradiation, and effectively protected the epoxy. There was some visible evidence of back surface cracking, but this had little effect on the tensile properties.

While extensive rear surface spallation is expected in shock loading of unbacked materials, the breaking of the fibers on the rear surface of the Kevlar samples that had enhanced coatings is believed to be a new result. This fiber failure is believed to be a synergistic effect caused by addition of the preload stress to the axial component of the laser induced shock wave after reflection from the rear surface. Based on a simple analysis of the stress components and on the measured stress wave attenuation through 1.5 mm samples, the threshold front surface normal stress for fiber tensile failure at the rear surface was found to be

$$\sigma_n \approx 10\,\sigma_o \left(1 - \frac{\sigma_p}{\sigma_o}\right)$$

where σ_o is the ultimate axial tensile stress and σ_o is the preload stress. For 50 percent preload, the threshold front surface normal stresses (pressures) for Kevlar/, fiberglass/, and graphite/epoxy are estimated to be 60, 80, and 90 kbar, respectively. Since enhanced coatings were used only on Kevlar, this was the only material to be subjected to pressures > 50 kbar and to fail in this mode.

It is believed that all three materials can be failed at the 2500 J/cm^2 level, if an appropriate enhancement coating is used. The results would, of course, be representative of damage occurring at higher fluences with normal service coatings.

CONCLUSIONS

Based on the investigations summarized above, the following conclusions can be drawn:

- Short (20 ns) single pulses of 1.06 μm radiation produce damaging shock waves in structural composites at 2500 J/cm^2.
- For an opaque coating, a 200,000-300,000 K plasma is formed which exerts a 40 kbar pressure pulse on the front surface which produces primarily delamination and cracking damage.
- For this case, < 30 percent of the beam energy is reradiated and almost all of the remaining energy resides in the expanding plasma.
- Uncoated samples of translucent composites exhibit lower peak shock pressures but extensive internal thermal damage.
- Opaque coatings with transparent overlays provide higher peak pressures which caused severe damage in Kevlar/epoxy in the tests.
- A synergistic effect was observed in Kevlar wherein the axial component of the reflected shock wave added to the preload stress to break fibers near the rear surface of the sample.
- Significant tensile strength reductions due to laser shocking were measured for Kevlar/epoxy and fiberglass/ epoxy, while graphite/epoxy exhibited no significant tensile strength reductions for the conditions studied.
- Extensive internal cracking was observed in all materials.

- Void closure was observed in uncoated Kevlar/epoxy and fiberglass/epoxy near the irradiated surface.

Because of the many parameters affecting the interaction, these results represent a first step in understanding shock damage effects. Additional research is needed to provide a better understanding of energy deposition in uncoated materials, shock wave attenuation, pressures generated by enhancement coatings, fluence and pulse width dependence of the laser effects, plasma expansion dynamics, and strength degradation at higher input stress levels.

REFERENCES

(1) C. T. Walters, and A. H. Clauer, "Laser Shock Effects on Stressed Structural Materials", Battelle Columbus Division Final Report to Naval Research Laboratory, Washington, D. C. (July 21, 1986).