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George J. Dvorak (Ed.)

Inelastic Deformation of Composite Materials



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Inelastic Deformation of Composite Materials

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Measurement of Strength of Thin Film Interfaces by Laser Spallation Experiment

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Abstract

Laser Spallation Experiment is developed to measure the strength of planar interfaces between a substrate and a thin coating (.3 to 3 μ m). In this technique a laser pulse of a high enough energy and a pre-determined length is converted into a pressure pulse of a critical amplitude and width that is sent through the substrate toward the free surface with the coating. The reflected tensile wave from the free surface of the coating pries off the coating. The critical stress amplitude that accomplishes the removal of the coating is determined from a computer simulation process. The simulation itself is verified by means of a piezoelectric crystal probe that is capable of mapping out the profile of the stress pulse generated by the laser pulse. Interface strength values ranging from 3.7 to 10.53 GPa are determined for the Si/SiC system, whereas for the carbon/SiC system, an average value of 6.91 GPa is obtained. Furthermore, sufficient experimental evidences are provided to show the potential of the laser technique to determine the interface toughness, provided well characterizable flaws can be planted on the interface.

1 Introduction

It is now well recognized that considerable toughness in composites with brittle, but strong reinforcing fibers, can be achieved by controlled debonding of the fibers from the matrix to prevent premature fiber fracture. In composites with brittle matrices, such as ceramics and glass, this is often the only means of obtaining toughness (Evans 1989). In metal matrix composites, an additional problem of interface reaction between the matrix and the fiber is present, which

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sooner or later, leads to the failure of fiber. To neutralize this problem, it is now a standard practice to protect the fibers with a less reactive sacrificial coating (Gupta *et al.* 1989). This also permits controlled delamination of the coating from the fiber along the fiber-coating interface to prevent cracks external to the fiber or residual reaction products on the fiber from damaging the latter. Thus, it has been suggested (Gupta *et al.* 1989, Argon *et al.* 1989 *a*) that such fiber-coating interfaces with tailored strength and toughness properties can be used as mechanical fuses to decouple the undamaged fibers from their damaged surroundings to prevent catastrophic failure of the part. An important dimension of utilizing tailored properties of interfaces is the measurement of the tensile strength and intrinsic delamination toughness of the fiber-coating interface. How the intrinsic toughness can be determined from the spontaneous delamination from the substrates of stressed coatings, where the coating is under positive or negative residual stress, has been discussed earlier by us (Argon *et al.* 1989 *a* & *b*). Here, we discuss how the *tensile strength* of such fiber-coating interfaces can be measured by a laser spallation technique.

Although the development of the laser spallation experiment was motivated by our specific interest in engineering the interfaces in composites, the technique can be used to determine the strength of planar interfaces between coatings ($> .3 \mu\text{m}$ in thickness) and substrates of any material. Hence, it should be of considerable interest to researchers in the device and thermal spray industries, where the mechanical problems stemming from the interface failure are of critical importance.

Measurement of interface properties is not a new problem in the thin film and coating technology, and the literature is replete with practical techniques (Jacobsson 1976; Jacobsson and Kruse 1973; Chapman 1974; Chiang *et al.* 1981; Davutoglu and Aksay 1981; Chow *et al.* 1976) for the measurement of some average properties of thin films or coating interfaces, recently reviewed by Mittal (1978). But, none of the tests mentioned above measure the strength of interfaces. In order to measure the strength of interfaces between very thin coatings and substrates, a new laser spallation technique has been developed that had been initially introduced by Vossen (1978), utilizing shock waves produced by short laser pulses (Lang 1974; Fox 1974; Ready 1965; Anderholm 1970; Peercy *et al.* 1970). The technique involves impinging a high energy laser pulse (pulse width of nanosecond duration), from a Nd-YAG laser ($1.06 \mu\text{m}$ wavelength) onto a thin absorption layer on the back surface of the substrate to which the coating of interest is attached. The sudden expansion of the absorbing film generates a compressive shock wave directed towards the test coating/substrate interface. It is the reflection of the compressive wave packet from the surface of the test coating that gives rise to a tensile pulse and leads to the removal of the coating, if the amplitude is high enough. The technique was used earlier by Park (1986) to detach films from the substrates. In his experiments, the absorbing film was sandwiched by a transparent fused quartz disc in order to increase the amplitude of the generated stress pulse. In most of the previous investigations of this phenomenon, ample evidence for the

feasibility of the technique was provided, but insufficient details were given for the quantitative use of the approach as a research tool.

Hence, the technique in its previous form, prior to the present development, could be used at best only to provide a figure of merit of the interface. The thrust of the present research has been to develop the laser spallation experiment in a more definitive way capable of quantifying the interface strength. Furthermore, the goal here is to detach thin films ($0.3\text{--}2\text{ }\mu\text{m}$ thick) from various substrates in contrast to the experiments done by Vossen (1978) and Park (1976), wherein test coatings of $20\text{--}30\text{ }\mu\text{m}$ thickness were considered. In our investigation to be presented here, we demonstrate that by careful use of the technique accurate determinations of tensile strength of interfaces are possible.

II Strategy of Interface Strength Measurement

2.1 Experimental Arrangement

Since the actual interfaces of interest are between a cylindrical fiber and its coating, which are not readily accessible to measurement by the spallation technique, the experimental approach to be presented here utilizes a planar arrangement of a model substrate and coating combination which is shown in Fig. 1. The collimated laser pulse is made to impinge on a thin energy-absorbing film sandwiched between the back surface of the substrate of interest and a fused quartz confining plate, transparent to the laser wavelength ($1.06\text{ }\mu\text{m}$ in this experiment). The characteristics of the ideal energy absorbing film are: high absorptivity; a very small critical absorption depth (much smaller than the thickness of the film); a high melting temperature, high coefficient of thermal expansion, high elastic modulus, low thermal diffusivity, and finally a

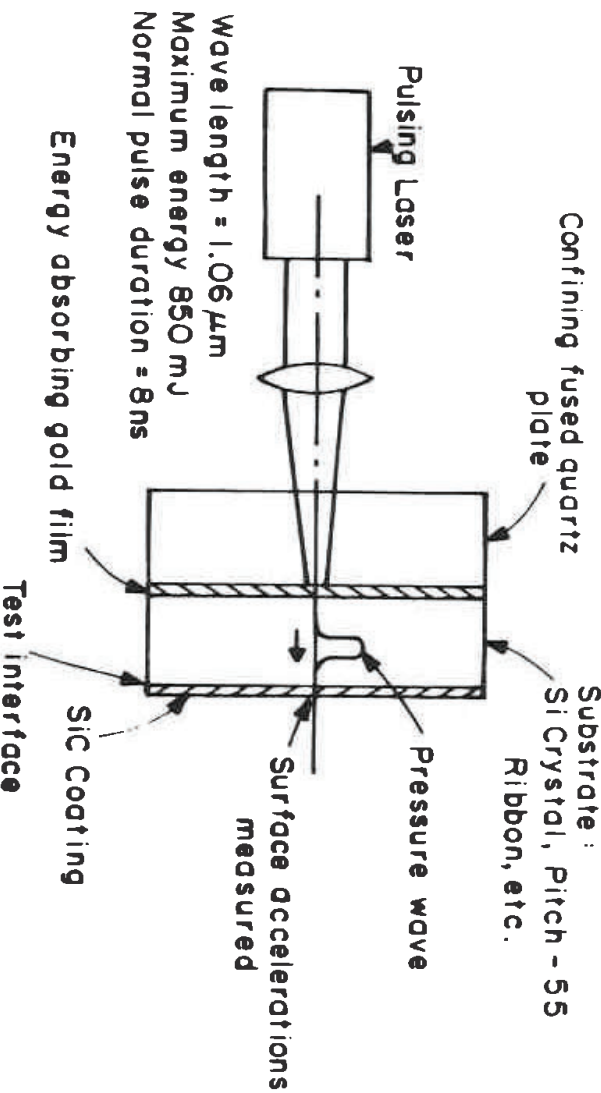


Figure 1. Schematic of the Laser Spallation Experiment.

thickness, λ , roughly equal to the test coating of interest applied to the front surface of the substrate. Based on a figure of merit analysis that captures the essence of the thermoelastic stress generation for various alternative energy absorbing materials, it was concluded that gold is the most effective material that maximizes the generated stress pulse amplitudes.

A key element of the experiment is to determine the amplitude of the tension wave that is formed by reflection of the pressure wave from the free surface of the coating attached to the rear surface of the substrate. The preferred method of accomplishing this is to measure the time rate of change of displacement of the free surface of the coating as the main compression pulse is reflected. This is usually done by the laser Doppler interferometry used widely in the plate impact research (Clifton, 1978). In the experiments to be reported here, however, only pulsing lasers of limited power were available, requiring to focus the beam over relatively small areas of roughly 1 mm^2 . This limits the planar portion of the pressure pulse to be reflected from the free surface of the test coating to a similarly small area, which makes the accurate measurement of the accelerations and decelerations of these small planar portions of the free surface rather difficult by the laser Doppler interferometry. In view of this, quantification of the measurements of the interface strength used here was based on a three part strategy.

The first part of the strategy was the development of a finite element computer simulation of the conversion of the laser light pulse into a pressure pulse. Next, the traverse of this pulse through any desired substrate of interest is monitored and finally the resulting amplitude and history of the tensile stress at the interface is determined as the stress wave is reflected from the free surface of the coating of given properties and thickness.

In the second part of our strategy, the pressure pulses were measured in a micro-electronic device in which the conditions of the computer simulation study were experimentally achieved. In this device, to be described below, the substrate and its test coating were replaced by a X-cut piezoelectric (PE) crystal equipped on its back face with an energy-absorbing gold film and on the front surface with a very thin gold electrode for signal pickup. The PE crystal with its attached fused quartz confining plate then became a pressure transducer operating in the short circuit mode, capable of pressure wave determination with a time resolution of 0.7 ns . The measured pressure signal profiles were then compared with those obtained with the computer simulation in which the substrate is appropriately given the elastic and thermal properties of the X-cut PE quartz crystal. This permits verifying and fine tuning of the computer simulation.

In the third part of the strategy, actual spallation experiments were carried out. The laser fluence necessary for the removal of the probed portion of the coating at the interface was recorded, and the tensile stress across the interface that accomplishes this was determined from the computer program.

In what follows, we discuss first in Section III the computer simulation of the laser-pulse-generated pressure pulses. In Section IV, we present the experimental details of the micro-electronic device for the measurement of

pressure pulses with the PE crystal probe. We then present the results of the pressure wave profiles that have been obtained. Results for the actual spallation experiment and the specific interface strength values are given for the SiC/Si and SiC/Carbon interface systems in Section V. In Section VI, the basic results of the computer simulation are given as generalized interface stress charts for various coating/substrate systems. Finally, Section VII provides a discussion of the approach and its potential.

III Simulation of the Stress Pulses (Part I of Strategy)

3.1 Statement of the Problem

Figure 2(a) shows schematically the configuration of the laser spallation probe consisting of, from left to right: the fused quartz confining plate (Q), the thin

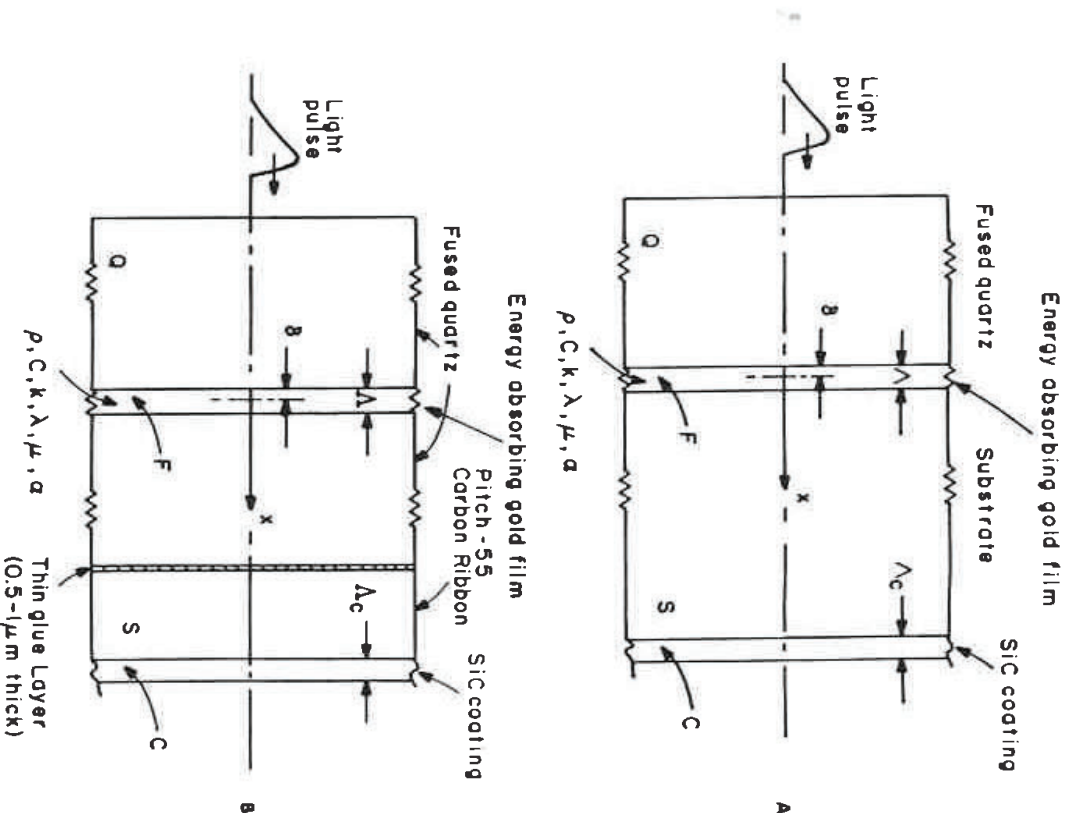


Figure 2. Configuration for testing (a) Si/SiC and (b) Pitch-55 Ribbon/SiC systems.

energy absorbing film (F) of thickness Λ , the substrate (S) and the coating (C) of interest. The laser pulse impinging on the energy absorbing film (gold) is generated by a Nd-YAG laser emitting at a wavelength of $1.06\ \mu\text{m}$. The laser used was in the MIT George Harrison Spectroscopy Laboratory. The peak power of the laser is 3×10^8 watts. The spatial profile of the laser pulse is Gaussian in nature. The temporal profile, however, is a distorted Gaussian (Ready 1971). The laser can be operated at $2.5\ \text{ns}$ or $8\ \text{ns}$ nominal pulse widths with a maximum achievable energy of $800\ \text{mJ}$ in a nominal laser beam diameter of $7\ \text{mm}$.

The critical penetration depth δ of laser light at this wavelength into the gold is only $20\ \text{nm}$ (Ready 1965). Since this is very much smaller than the thickness Λ ($\sim 1\ \mu\text{m}$) of the energy absorbing film, the simplifying assumption is made that the energy is deposited on the interface between the film and the fused quartz confining plate. The cross-sectional area of the laser beam incident on this interface is of the order of $1\text{--}2\ \text{mm}^2$.

Additional specifications in the simulation include: ignoring temperature dependence in all the relevant physical properties, such as thermal conductivity, coefficient of thermal expansion, specific heat, density, and elastic moduli. Such variation can, in principle, be taken into account in the finite element solution but were ignored in the initial simulation. Actual measurements and the simulation indicated that melting of the energy-absorbing film is undesirable since it broadens and diffuses the sharpness of the pressure wave, but is necessary to endure in order to obtain the required level of interface stress. Therefore, the simulation considers melting in the energy-absorbing film but only solid-like behavior in all the other media. The effect of volume changes associated with the phase change in the gold film is considered. This is quite significant for the gold film which undergoes a volume expansion of 5.1% (Smithells Metals Handbook 1983) on melting. The time constant for viscous flow relaxations in the narrow molten region of the energy absorbing film is much longer than the duration of the laser pulse. Therefore, even if melting of portions of the film occurs, it is adequate to account in the simulation for only the changes in the physical properties due to the melting transition, but to ignore all viscous flow relaxations. Furthermore, the thermal diffusion distance in most absorbing films during the laser heating time of $2.5\ \text{ns}$ varies between 1 to $10\ \mu\text{m}$. This distance is negligible as compared to the radius of the laser heating spot of $1\ \text{mm}$. The absence of fluid-like flow of the molten material and small thermal diffusion distance suggests that the individual material points are blind to their neighbors in the radial direction and their deformation is strictly governed by the amplitude of the laser energy impinging directly on them. Hence, the generation of the stress pulse can be modelled as a one-dimensional phenomenon.

The associated phenomenon of the pulse propagation in the substrate, and the eventual spalling of the test coating are also modelled using the one-dimensional stress wave equations. The validity of this assumption will be verified later by the actual measured pressure pulse shapes to be discussed in

Section IV below.

Ignoring the thermoelastic effect as being quite small, the simulation considers two related, but uncoupled phenomena of transient heat transfer, giving temperature distributions in and around the energy-absorbing gold film as a function of time, and the related transient elastic wave propagation in all four media of Q , F , S and C , that results from the time rate of change of the thermal misfit, induced by transient heating. Since the Pitch-55 carbon ribbon and the pyrolytic graphite substrate used to model the Pitch-55 fiber are orthotropic in their elastic properties, the anisotropic effects are also considered in the present formulation. The details of the governing equations and the solution technique can be found in Gupta (1990). Here, we discuss briefly the key features of the solution.

3.2 Solution of the Governing Equations

Figures 3 a & b show the traverse of pressure fronts in both the Q and S spaces for two different normalized times of 70 and 109 for the constants H_q , K_q and H_s and K_s (to be defined below) given in the figure. In Figure 3, the time t ; distance x , temperature T , displacement u , and the stress σ are normalized by introducing the variables:

$$\zeta = x/\Lambda; \quad \tau = t/t_0; \quad \theta = T/T_0; \quad \varphi = u/u_0; \quad \Sigma = \sigma/\sigma_0 \quad (1)$$

where Λ , t_0 , T_0 , u_0 and σ_0 are the fundamental units of length, time, temperature, displacement and stress respectively, and are defined as

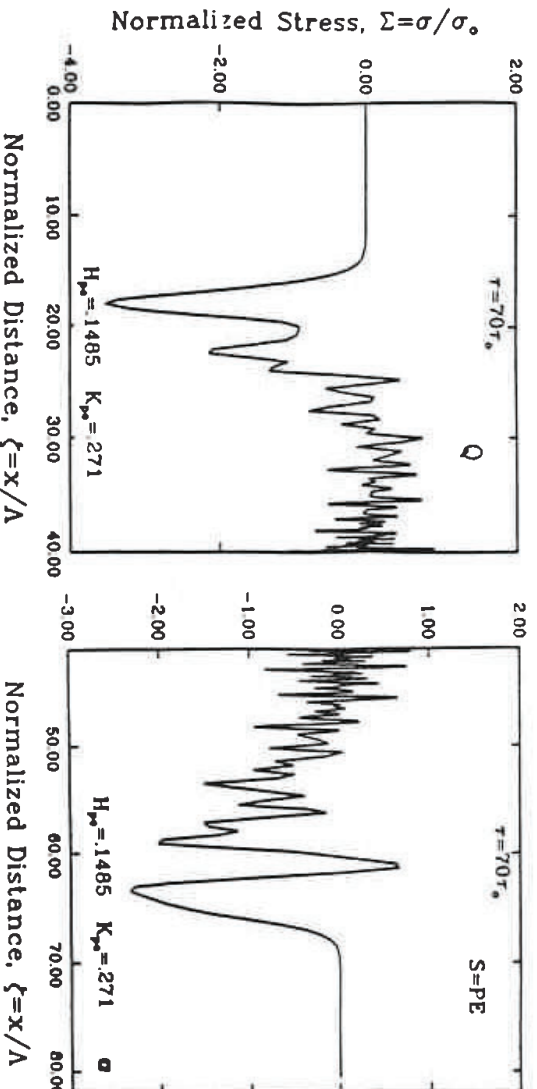


Figure 3. Generation and propagation of stress pulses in Q and S spaces.

$$t_o = (\mathcal{M}c)_f, T_o = \frac{q\Delta t_i}{(C\rho\Lambda)_f}; u_o = \left(\frac{\Lambda\gamma}{\rho\delta c^2} \right)_f q\Delta t_i \quad (2)$$

$$\sigma_o = \left(\frac{(2\mu + \lambda)\gamma}{\rho\delta c^2} \right)_f q\Delta t_i \quad \text{and} \quad \gamma = (3\lambda + 2\mu)(\alpha/\rho C)$$

where in turn, $\Lambda, c, C, \rho, \delta, \gamma$ are the thickness, the one-dimensional pressure wave velocity, the specific heat, the density, the characteristic penetration depth of light energy and the Grueneisen constant respectively, all for the energy-absorbing film, which is taken as the medium for the basis of normalization. Δt_i is duration of the laser pulse and q is the absorbed laser fluence. The parameters H and K enter the formulation when the different physical properties of the substrate and confining quartz media are represented in terms of the properties of the energy absorbing film as

$$H_i = \left(\frac{cf}{c_i} \right)^2; \quad K_i = \frac{(\gamma C/c)_i}{(\gamma C/c)_f} \quad (3)$$

In equations 2&3 above, the subscripts i and f refer to the substrate and the energy-absorbing film respectively.

A more expanded figure of the pressure wave profile in the piezoelectric substrate is shown in Fig. 4, which reveals, in addition to the expected main pressure wave abc , an initially unexpected tension wave cde immediately following the compression wave. This tension wave has been found in all simulations with metallic energy absorbing films and should have useful properties in providing additional tension across the test interface between S and C . It appears to result from a space heating effect. As the thermal front penetrates into the energy absorbing film away from the Q - F interface, the interior of the film undergoes a "flash" expansion during the time increment Δt_i of the short laser pulse, while the material in the forward direction still experiences no effect of the pulse. The pressure wave released from such interior slabs in the film and travelling back towards the Q - F interface will be partially reflected from the interface back into the film and in the forward direction as a tension wave, because of the lower modulus and much lower density of the fused quartz in comparison to those properties of the gold film. This was also confirmed by a test simulation where the elastic properties of the two media were matched at the Q - F interface, resulting in the absence of the tension peak. Furthermore, such a "rebouncing" condition should be favored in a system in which the "flash" expansion of the energy-absorbing packet cannot be significantly counteracted by the elastic wave tending to disperse the misfit

during the time, the laser pulse is on; i.e., systems in which

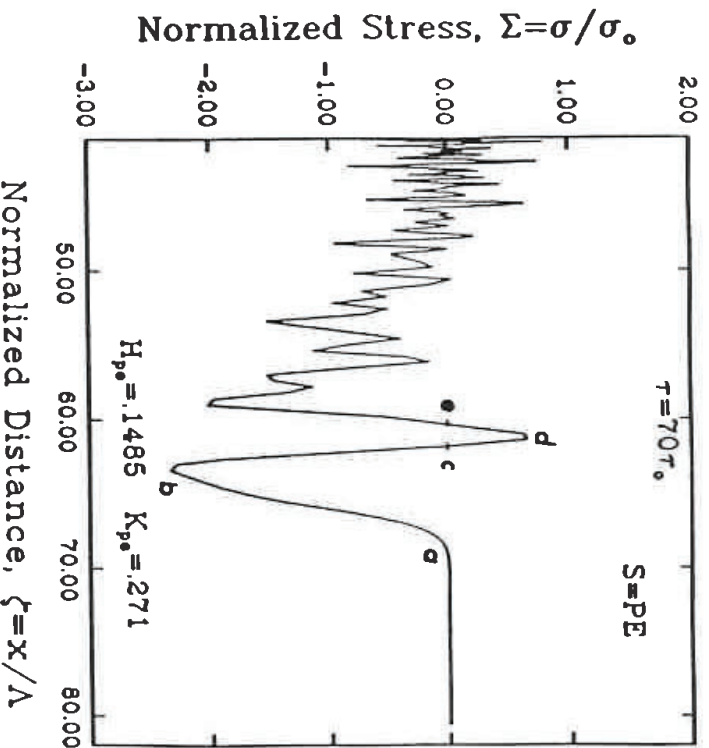


Figure 4. Detailed structure of the stress pulse in the piezoelectric substrate.

$$\left(\frac{k}{\Delta t C p c^2} \right)^{1/2} = \frac{k}{\Lambda_c C p c}, \quad (4)$$

the ratio of the average thermal velocity to the sound velocity is high. In eqn. (4) above, Λ_c is the thickness of the coating to be blown off and k is the thermal conductivity of the energy absorbing film. In systems with metallic energy-absorbing films for which the simulations have been performed, where the tension peak was found, the ratio

$$k/\Lambda_c C p c$$

in Eqn. (4) was found to be around 0.05-0.1, while for a carbon film which does not show the peak, the ratio is as low as 3×10^{-3} .

In the simulation, the propagation of the stress pulse was monitored in the substrate until it completed the tensile loading of the coating/substrate interface. The stress history of the Si/SiC interface due to the pulse of Fig. 4 and for a SiC coating of $1.5 \mu\text{m}$ thickness is shown in Fig. 5. Zero values prior to the main signal correspond to the time lag in the arrival of the stress pulse at the interface. Thus, for a given threshold laser fluence, the interface strength can be calculated from the amplitude of such plots.

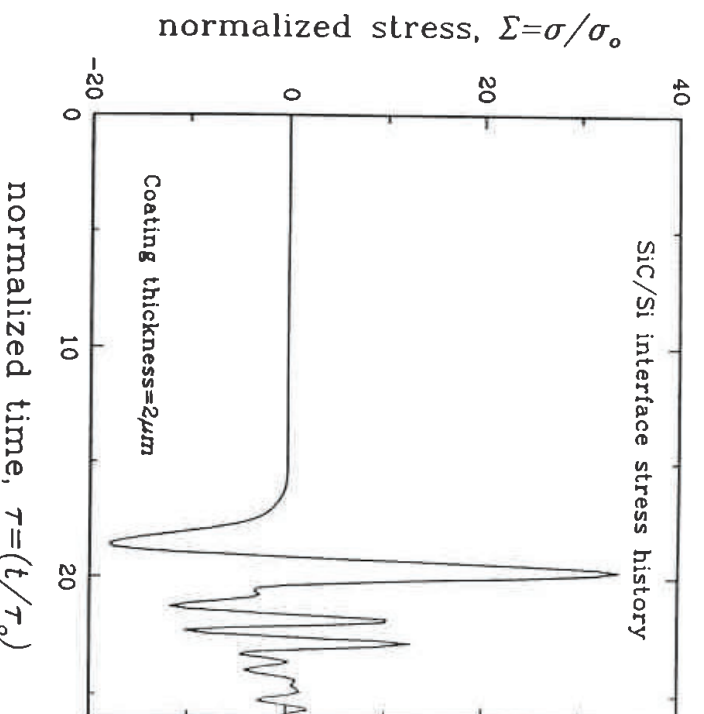


Figure 5. Stress history at the interface.

IV Experimental Stress Pulse Measurements (Part II of the Strategy)

4.1 The Micro-electronic Device

A schematic view of the micro-electronic device with the X-cut piezoelectric crystal is shown in Fig. 6. The assembly of fused quartz plate (Q), energy-absorbing(gold) film (F), and the PE crystal, taking the place of a substrate, is shown encased in a Bakelite housing. 1 μm thick gold films were chosen (Ditchburn 1963) to completely absorb the laser fluence in order to avoid the well-known radial cracking phenomenon (Volkova 1967) in the substrate disc due to its heating by the transmitted laser fluence. The laser-absorbing gold film also acts as a ground electrode for the piezoelectric crystal. A rubber "O" ring between the housing and the PE crystal radially compresses the latter when the copper electrode housing (C) is attached to the bakelite housing. The radial pre-compression of the substrate disc counteracts cracking of the substrate disc subjected to the sudden impact of the generated stress pulse. Figure 7 shows a perspective view of a typical sandwich element in the PE device. The PE device has a gold energy-absorbing film sandwiched between the PE crystal and the Q plate, acting also as the ground electrode of the PE device. As shown, this extends all the way to the front surface of the PE crystal, where it terminates as

an outer ring, to which the copper electrode housing makes contact. Six equally spaced round gold electrode tabs of 2 mm diameter communicating with a central round tab through thin conducting spokes ($25\text{ }\mu\text{m}$ wide) constitute the secondary electrode. These electrodes were deposited by photolithographic techniques. This geometry of the secondary electrode was designed to pick up the one-dimensional structure of the current signal. This was ensured by focussing the laser beam to a 2-3 mm spot on the laser-absorbing film in line with one of the 2 mm diameter secondary electrodes as shown in Fig. 7. This assembly permits as many as six separate measurements of the shapes of arriving pressure pulses by indexing a new tab into the line of sight of the collimated beam.

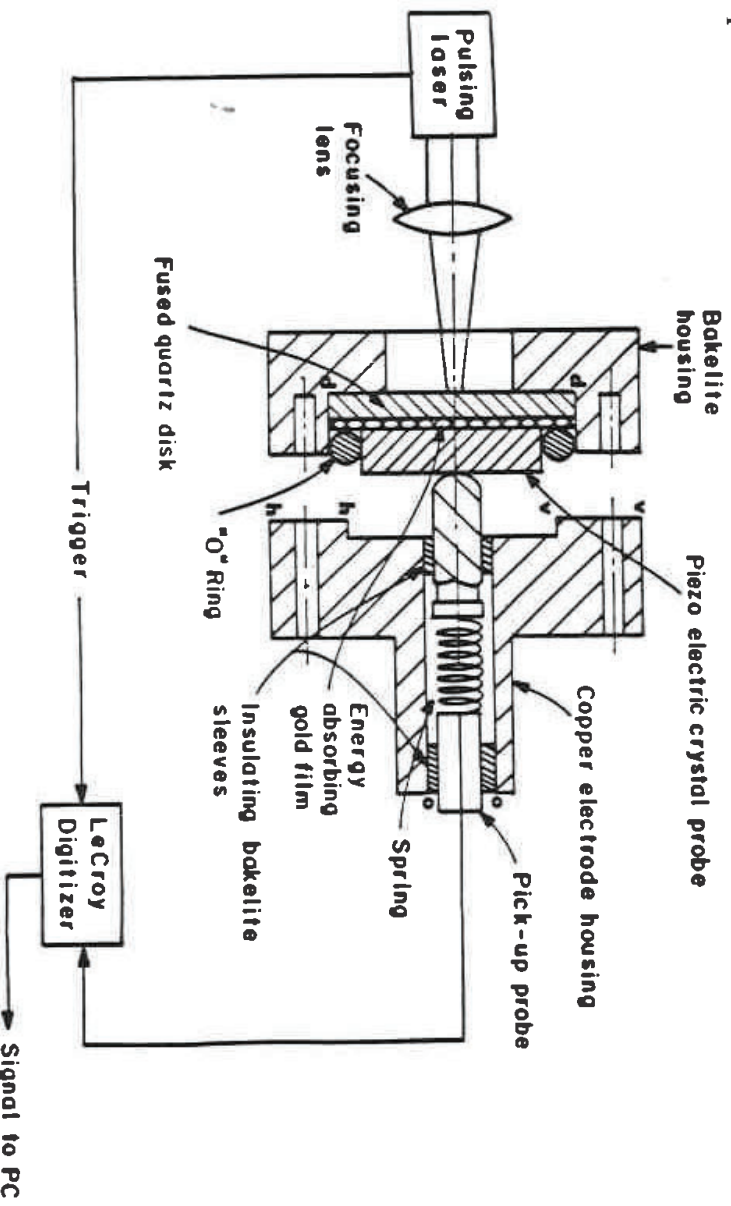


Figure 6. A schematic view of the microelectronic device.

This assembly of fused and piezo-electric quartz is then encapsuled by a copper electrode housing by lapping it onto the edges of the bakelite housing so that the legs vv and hh of the copper piece (see Fig. 6) touch the outer ground electrode ring on the rear face of the crystal. Thus, the whole copper enclosure acts as a ground electrode. A specially constructed pickup probe with a copper leg, insulated from the copper piece, is then inserted via the opening oo , so that the copper leg touches the central secondary electrode. The short circuit current derived between the two electrodes is picked up via a BNC plug fastened on top of the pick-up probe and fed into a Lecroy high speed digitizer by a BNC cable. A high speed Lecroy digitizer (1.3 GHz sampling rate) is needed to record the stress pulses with sub-nanosecond rise times. Since the experiment is performed in the single shot mode, the digitizer is triggered by an electronic pulse that is fired just before the lasing of the optical pulse. The details of the micro-circuitry and various considerations in designing the micro-electronic device can be found in Gupta (1990).

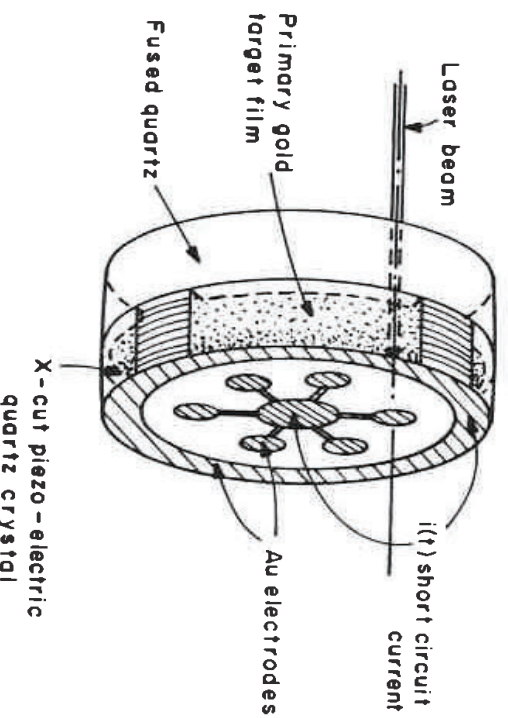


Figure 7. Perspective view of the micro-electronics device.

4.2 Pressure Pulses in the Piezo-electric Crystals

The procedure of using the X-cut PE quartz crystal to record ultra-short duration high amplitude pulses follows the technique described by Graham *et al.* 1965. The measurement is made in the short circuit mode, between the ground electrode that is struck by the laser beam and the front electrode from which the pressure wave is reflected, through an external circuit incorporating the digitizer with a 50Ω impedance. The short circuit current $i(t)$ obtained from the crystal as the stress wave propagates in the crystal is given by (Graham *et al.* 1965)

$$i(t) = \frac{fAc}{l} [\sigma_o(t) - \sigma_l(t)] \quad (5)$$

where A is the effective cross-sectional area of the laser beam, l is the thickness of the crystal, c is the velocity of the planar pressure wave, f is the polarization coefficient of the crystal and, $\sigma_o(t)$ and $\sigma_l(t)$ are the amplitudes of the stress pulses on the ground (impacted energy-absorbing film) and front surface electrodes of the crystal. The term $\sigma_l(t)$ is to be interpreted as the amplitude that hits the free surface, but is annulled instantly by an equal and opposite virtual amplitude in order to satisfy the free surface boundary condition. In the absence of such interpretations, which were apparently not explained by (Graham *et al.* 1965), equation (5) could be misleading as the term $\sigma_l(t)$ is always zero. Nevertheless, if the wavelength of the stress packet is smaller than the crystal thickness, equation (5) reduces to

$$i(t) = \frac{fAc}{l} \sigma_o(t) \text{ for } t < l/c \quad (6)$$

which gives the time dependent profile of the stress wave. Equation (6) predicts that the stress at the input electrode is directly proportional to the instantaneous current for times less than the wave transit time.

Most of the researchers (Lang 1974; Fox 1974; Ready 1965; Anderholm 1970; Park 1986) in the past have used the above equation to measure the stresses at the input electrode of the crystal. However, if the thickness of the crystal is greater than the width of the stress pulse, then two short-circuit current signals corresponding to each electrode are obtained. The stress is related to the current signal at the ground electrode via equation (6), but interestingly, the current signal output when the pulse hits the rear electrode is exactly twice that obtained from the ground electrode. This is explained in details by Gupta and Epstein (1990), where the physical phenomenon described by the above equations is elucidated. Because the laser fluences were sufficient to melt a part of the input electrode, it became necessary to record the current signal emitted from the rear electrode for more reliable measurement of the generated stress pulse.

4.4 Comparison of Measurements with Predictions

The simulation presented in Section III considered only a one-dimensional case of propagation of elastic waves due to the laser-pulse-induced, rate-of-change-of-thermal-expansion misfit. That this is close to reality is demonstrated in Fig. 8, where the input current profile resulting from the impingement of the laser beam on the ground electrode is plotted above the current profile emitted from the secondary electrode due to the first reflection of the compressive wave. Apart from the expected amplification of the reflected current by a factor of 2 above the

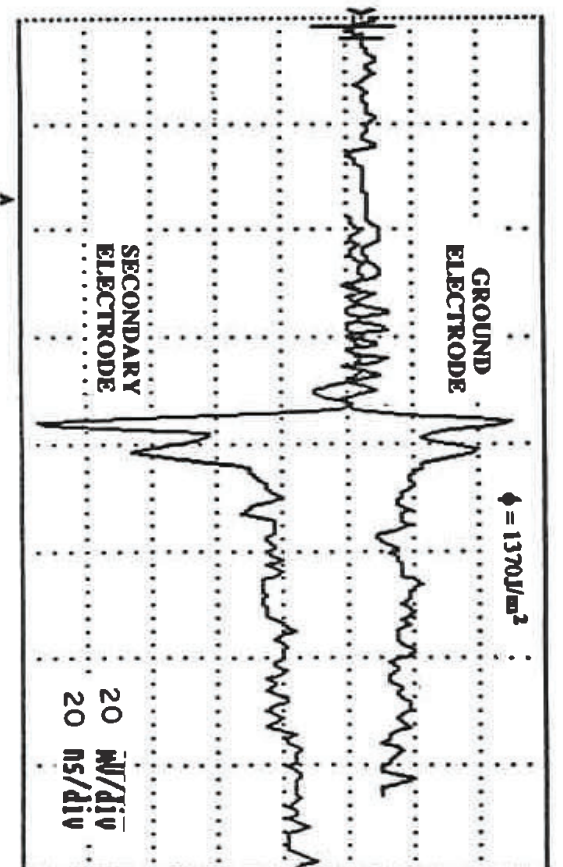


Figure 8. Evidence of the planarity of the travelling stress wave.

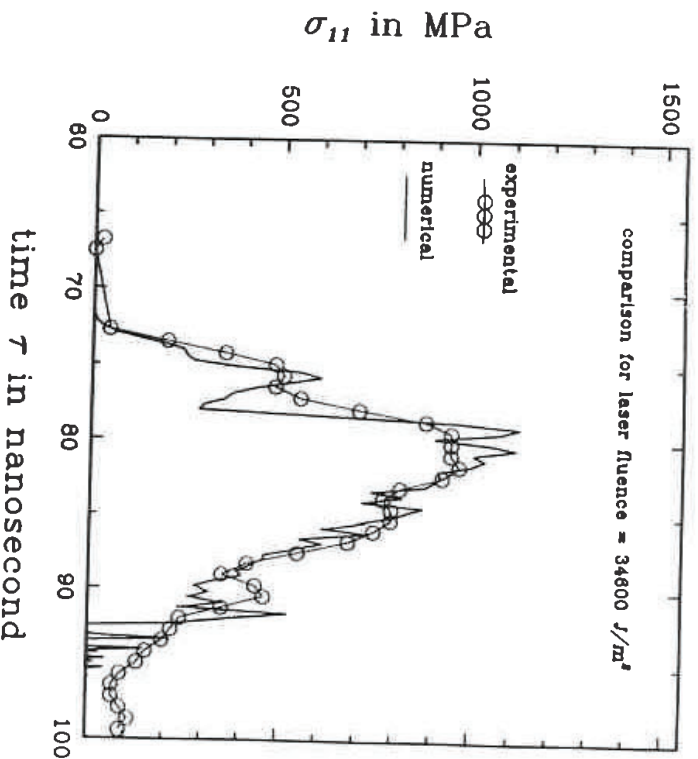


Figure 9. Comparison of the predicted and measured profiles of the stress pulse.

input signal, the two profiles are quite similar in the main portion, but differ in the structure of their tails. This similarity is taken as evidence of planarity of the main pulse.

Figure 9 shows a direct comparison of the predicted and measured profiles of the input stress pulse for a fluence of $34,600 \text{ J/m}^2$ (with distinct melting behavior of the gold film). Figure 9 shows that the predicted profile of the stress pulse for an emissivity of 0.18 for the gold film is clearly similar in broad outline to the profile measured by the *PE* device. The amplitudes of the experimentally recorded pulse and the predicted ones are remarkably similar. This is very encouraging for the simulation, as the ultimate aim in this exercise is to predict the stress amplitudes at the substrate/coating interface. Figure 9, which shows the structure of the recorded pressure pulses for an absorbing gold film, demonstrates the presence of two compression peaks with an intervening gold film, depression which in the simulation of Fig. 4 appeared as a tension peak. The depression in Fig. 9 has remained in the compression region and has not gone into tension as the simulation predicts. This currently remains a discrepancy that must be resolved. This, however, is of little interest in the present strategy as the laser fluence required to spall the test coatings is sufficiently high to cause the melting of the gold film. Therefore, the computer model can be used with sufficient confidence to predict the interface stress histories in other substrate/coating pairs of interest to researchers in the thin film area. The interface stress is determined from the amplitude of the interface stress history plots of the type shown in Fig. 5.

V Spallation Experiments

The spallation technique was applied to three substrates consisting of Si single-crystal wafers with (100) plane surfaces, Pyrolytic graphite (PG) platelets with the principal axis of layer normals lying in the plane of the platelet, and Pitch-55 (P-55) type carbon ribbons of $600\mu\text{m}$ width and $35\mu\text{m}$ thickness having a meso-phase morphology very similar to the Pitch-55 carbon fibers. Detailed description of the morphology of the Pitch-55 ribbon can be found in Gupta and Argon (1990) and for PG in Gupta *et al.* (1988). In all these cases, the coatings to be removed were amorphous SiC coatings deposited by the plasma-assisted chemical vapor deposition technique. The coatings had a thickness of about $1\text{--}3\mu\text{m}$. They were deposited in a nearly stress free manner by maintaining the substrates at a certain temperature to prevent the entrapment of a significant concentration of hydrogen, which otherwise would have resulted in high residual compressive stresses and premature delamination in a manner described in detail by us earlier (Argon *et al.* 1988).

The Si/SiC, PG/SiC interface systems were tested in an experimental configuration of the type shown in Fig. 2 (a), whereas for the P-55/SiC system, the setup of Fig. 2 (b) was employed. The coating could be successfully spalled off in all cases with appropriate levels of laser pulse energy. The threshold laser energy at which spallation occurs was recorded using a light power meter. Since

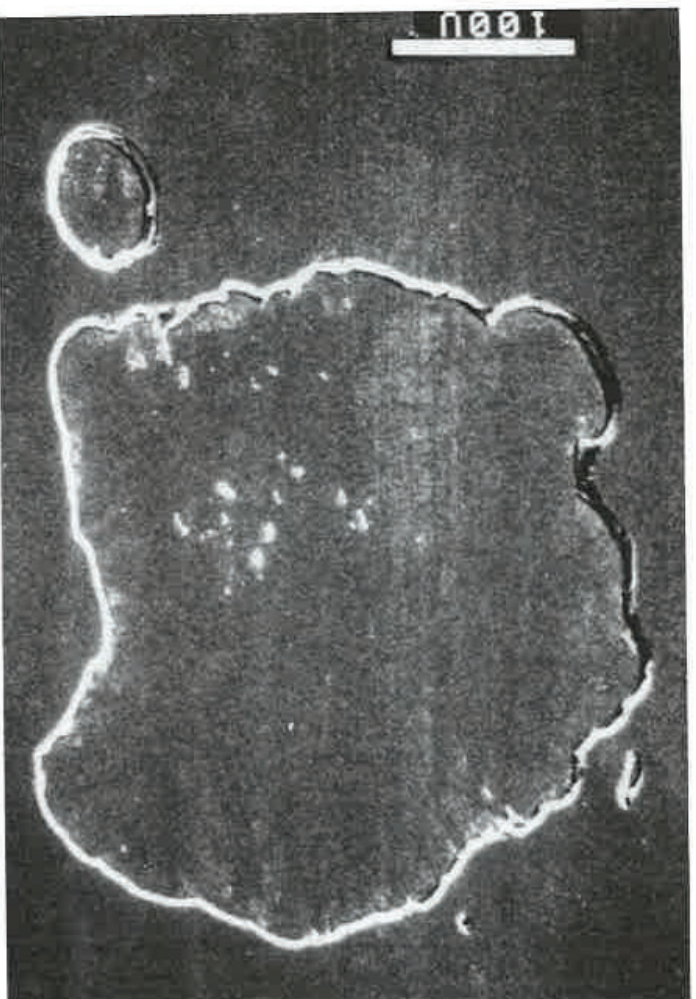


Figure 10. Spalled spot of SiC coating from Si single crystal.

¹These exploratory Pitch-55 ribbons were specially prepared and furnished to us by the textile Fibers Department of the DuPont Company for which we are grateful to Dr. E. M. Schulz.

the amplitude of the stress pulse should depend upon the absorbed laser fluence, the diameter of the laser beam was recorded on photographic film for the tabulation of the threshold laser fluence, as mentioned above.

(a) *Si/SiC interface*: Figure 10 shows a micrograph of the spot from which the SiC coating has spalled off. The laser fluence necessary to achieve this spallation is $7,388 \text{ J/m}^2$. The interface strength corresponding to this laser fluence as calculated from the computer simulation is 5.29 GPa. The thickness of the amorphous SiC coating of low modulus, deposited at low ion beam energy, was $1.5 \text{ }\mu\text{m}$. The dark portions of the micrographs are the regions where the SiC coating still adheres to the Si substrate. The islands of SiC within the spalled spots are due to the statistical variability of the interface strength over the delaminated spot. The interface strength varies from 10.19 GPa to 10.88 GPa, which is surprisingly low (only 7%) for a brittle interface.

The clean flat structure of the interface shows that the SiC coating has come off uniformly from the substrate at the interface. In order to verify this, Auger electron spectroscopy (AES) was performed on the spalled spot to determine the location of the failure. The irregular pattern at the rim of the spalled spot is due to the partial delamination and breaking of the coating at the circumference of the spalled coating. It was possible to successfully delaminate coatings of thicknesses ranging from 0.3 to $3 \text{ }\mu\text{m}$.

Finally, the actual interface strength values for the various SiC/Si systems produced by using different deposition parameters (of the SiC coating) vary from 3.7 GPa to 10.88 GPa. While such a variation may appear disappointing, this degree of freedom is exactly what is required to carry out the interface delamination scheme in composites as outlined in Section I. The interface strength between the carbon and the SiC interface is of interest in such an endeavor. These measurements are discussed next.

(b) *PG/SiC coating interface*: Figure 11 shows a spot from which the SiC coating of $2.1 \text{ }\mu\text{m}$ thickness is spalled off from the PG surface. The substrate disc was 1.69 mm thick. Interestingly, the spalled pattern is elongated along the edges of the graphitic planes that terminate perpendicular to the surface. This is also the stiffest direction in that plane. For this case the interface strength is determined to be 3.68 GPa. These calculations also include the anisotropic character of the PG substrate. Since the surface techniques available were not able to distinguish the carbon of the SiC from that of the PG substrate, the depth of the crater as determined by a mechanical profilometer (to an accuracy of 2.5 to 10 nm) compared remarkably well with the thickness of the deposited SiC coatings, thereby confirming the failure at the interface. Interface strength values ranging from 3.4 to 7.48 GPa were obtained for different SiC coatings.

(c) *Ribbon/SiC Coating Interface*: Due to the presence of inhomogeneities and the weak (transverse) strength of these ribbons across lamellae, failure was predominantly observed to be within the ribbon. However, in some cases, failure at the interface was also observed. Figure 11 shows a high magnification view of an edge of a spalled coating. The coating is intact on the left of the micrograph and is removed from the right side. For this sample, an average interface strength value of .240 GPa was obtained. These calculations also

include the anisotropic character of the ribbon material. A part of the same ribbon was tested without the coating in order to determine the transverse strength of the ribbon. A value of 0.26 GPa was obtained. As expected, this value is higher than the interface strength observed on the same ribbon/coating system. Interface strength values ranging from 0.22 to 0.24 GPa were obtained



Figure 11. Spalled spot of SiC coating from the Pitch-55 ribbon.

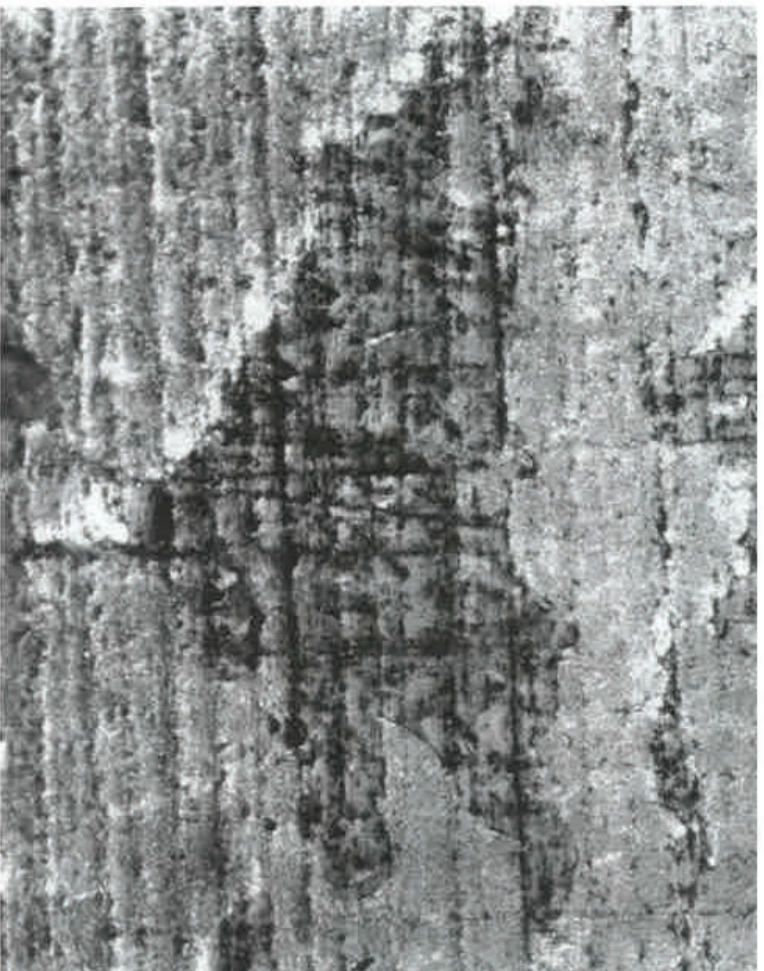


Figure 12. Spalled spot of SiC coating from PG substrate.

for the Pitch-55 ribbon/SiC system. In all the above cases, the location of the failure was confirmed by matching the coating thickness to the depth of the spalled spot determined from a high-precision profilometer. The interface strength determined from this test is significantly lower than that obtained from the PG/SiC system. This is probably due to the inhomogeneities on the ribbon surface (see Gupta 1990 for the structure of the ribbon), which act as sizable interface flaws and lead to lower strength values. In view of the poor structural integrity of the ribbon material, the PG/SiC system appears to be a better candidate for determining the properties of the Pitch-55 fiber/SiC interface. Nevertheless, the values obtained from both the systems are greater than the desired level of interface strength of 200 MPa for interface delamination in the SiC coated Pitch-55 fiber/aluminum matrix composites (Gupta 1990). Thus, it is necessary to impair the interface strength by implanting embrittling agents at the interface during the coating deposition process. This could be achieved by planting a few atomic layers of either Na, Sb or As. Recently, Rice and Wang (1989) have theoretically explored the effect of such foreign agents on the interface toughness. Similar effects need to be experimentally achieved in the present work in an attempt to bring down the interface strength levels.

Since the interface is loaded by a stress pulse that is external to the material system, the laser spallation experiment is capable of determining the interface strength for any thin film interface. To encourage the wide applicability of the laser spallation experiment, the results of the computer code, which has been verified by the piezo-electric probe, are furnished as generalized interface stress charts so that researchers interested in using the technique do not have to reproduce the computer code. These results should make such strength measurements possible in most systems of interest to workers on composite materials.

VI Interface Stress Charts

The intent of this section is to present generalized interface stress charts for various substrate/coating pairs so that researchers interested in using this technique do not have to reproduce the computer model. It is not possible to give results for exhaustive sets of substrates. Here, we give results for four substrates only, spanning wide ranges of H_s and K_s values. An empirical scheme for obtaining the stresses at other substrates (with arbitrary values of H_s and K_s values)/coating interfaces can be found in Gupta (1990). Since the stress pulse generated in the substrate is independent of the test coating on its front surface, the interface stress can be further normalized as

$$\tilde{\sigma}_i = \sigma_o/T \quad (7)$$

where σ_o is the normalized stress, defined before in Section 3.2 and T is the acoustic transmission coefficient at the substrate/test coating interface, defined as

$$T = \frac{2\sqrt{E_c\rho_c}}{\sqrt{E_s\rho_s} + \sqrt{E_c\rho_c}} \quad (8)$$

where ρ_c , E_c and ρ_s , E_s are respectively the density and Young's modulus of the test coating and the substrate medium. With the above normalization, it is possible to determine the stress at the interface between a substrate and coating of *any* material provided the elastic properties of the two media are known *a priori* in order to calculate the acoustic transmission coefficient T . A typical interface stress chart is shown in Fig. 13. This chart can be used for any substrate with the same H_s and K_s values. A catalogue of interface stress charts valid for other systems can be found in Gupta (1990). Thus, the threshold laser fluence determined from the experiment can now be converted into the actual strength values via the interface stress charts over a wide range of substrate/coating systems of interest to researchers in the device and thermal spray industries.

Due to the absence of any gripping effects, the spallation experiment also provides a good measure of the intrinsic strength of thin coatings and single crystals. The results of such measurements can be found in Gupta *et al.* 1990.

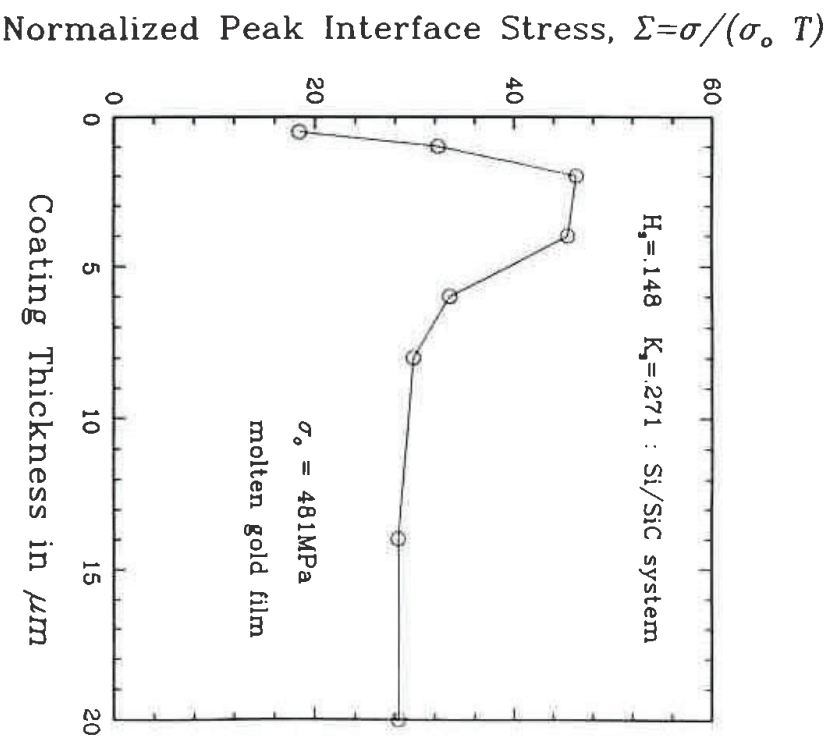


Figure 13. A typical interface stress chart.

VII Discussion

The tensile strength of the interfaces between two media in many instances determines the overall strength of a heterogeneous solid. For example, considerations of initiation of cavities on interfaces of particles is of interest in understanding the initial phases of the ductile fracture in metals and alloys. Similar considerations abound in composite materials where the tensile strength of interfaces between reinforcing fibers and the surrounding matrix, or between the fiber and a protective coating is often of major importance. The measurements of such strengths in macroscopic experiments has been attempted by many investigators but is not free of problems. In most of the reported cases, part of the interface is subjected to high stresses in an inhomogeneous deformation field. The magnitudes of these stresses need to be determined through the solutions of complex boundary value problems of elastic and plastic deformation. While these attempts have often given operationally useful answers, the factor of uncertainty in them has been considerable because of the inadequacy of the solution of the local deformation problems. The measurement of strength between a substrate and a thin coating presents particular difficulties in the application of the stress by an inhomogeneous local deformation field where premature failure elsewhere is likely, before the desired interface can be probed.

It is in these latter cases that the laser spallation technique is an attractive alternative, provided that the interface to be probed can be obtained in planar form. If the coating to be pried off is of thickness A_c , the required stress pulses with widths of roughly the same magnitude as the coating thickness can be achieved by a laser pulse of duration Δt_l ,

$$\Delta t_l \cong A_c^2 \rho C / k \quad (9)$$

where ρ , C , and k are the density, specific heat and thermal conductivity of the energy-absorbing film in which the primary thermal misfit is being generated. Thus, for an energy-absorbing film of Au, the required laser pulse duration to pry off a coating of $1 \mu m$ thickness is about $8 ns$. This is readily achievable with many pulse laser systems. The three-part strategy which we have outlined here furnishes an operationally attractive means of measurement of the strength of planar interfaces that can be made part of a laser spallation probe. In this, the fundamental tool is the computer code which permits a reliable means of simulating the generation and propagation of elastic waves in the substrate. The basic results of this code, which have been broadly verified by the piezo-electric probe for various material pairs, are provided in the form of interface stress charts presented in Section VI. These results should make such strength measurements possible in most systems of interest to workers on composite materials by using the interpolation scheme furnished in Section 6.4.

Tensile strength of interfaces between amorphous SiC coatings and

substrates of Pyrolytic graphite, Pitch-55 carbon ribbon and Silicon single crystal discs were explored in the present investigation. The average interface strength for the SiC/Si pair determined from the experiment ranges from 3.7 GPa to 10.88 GPa, depending upon the deposition conditions for the SiC coating. Average interface strength values ranging from 3.68 to 7.48 GPa for the PG/SiC system and 0.22 to 0.26 GPa for the Pitch-55 fiber/SiC system were obtained.

Since the tensile stress causing the interface delamination is built up as a result of the reflection of the main compressive pulse from the free surface of the coating, it is necessary that the length of the initial compressive pulse is of the same order as the thickness of the SiC coating. This will ensure building of sufficient tensile stress at the interface to cause interface failure. It was found that SiC coatings of thicknesses 0.3 to 3 μm could be readily removed from substrates. In order to spall even thinner coatings from the substrate, it will become necessary to use picosecond laser pulses in place of nanosecond ones as employed in the present investigation. Results of the numerical exercise in Section III indicate other interesting possibilities to delaminate very thin coatings ($<1\ \mu\text{m}$ thick) from substrates, that are of considerable interest to researchers in the semiconductor and device industry.

While the interface tensile strength is an important quantity required to determine the critical initiation conditions in interface separation, in flawed interfaces or in cases when the propagation of an existing crack along the interface is the determining factor, the most relevant quantity required is the fracture toughness K_{Ic} or the critical energy release rate G_c for the propagation of a crack along the interface. A measure of this can also be obtained with the laser spallation method, if well characterizable and reproducible flaws can be

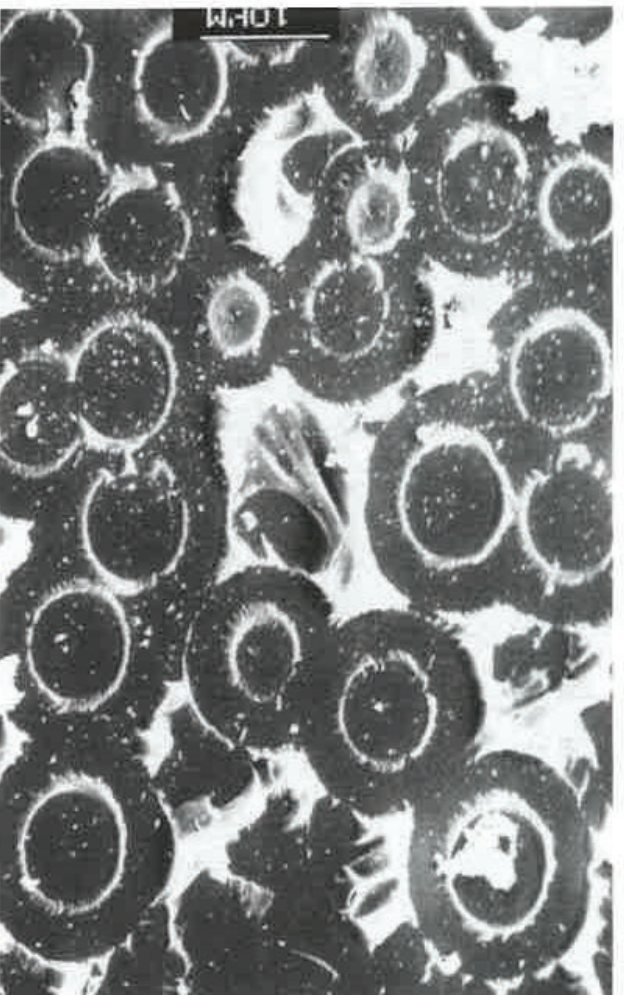


Figure 14. Spalled pattern on a contaminated interface.

placed on the interface, so that the cracks emanating from these can be propagated under a critical reflected tensile stress pulse that can provide the required G_c or produce the K_{IC} . In a preliminary experiment where some local interface contamination of an undetermined nature had apparently occurred during the deposition of the SiC coating, flaws of a potentially interesting type were implanted on the interface. When this specimen was subjected to a typical laser pulse, the coating spalled off independently from individual spots as shown in Fig. 14. Clearly, under the condition of the reflected tensile stress pulse, which probes the interface, a central crack is formed in the area of the interface flaw where a much reduced level of interface strength must have been present. After the crack had propagated radially outward a certain distance, the effective ratio of the crack tip stress intensity factors K_I and K_{II} had apparently undergone almost two full reversals of sign before the delamination is complete. This has resulted first in an excursion of the crack away from the interface into the coating, to be followed by a sharp reversal returning the crack back to the interface, only to be followed by another reversal putting the crack into the coating again where it continued to propagate to final fracture. Evidently, two separate effects: the bi-material nature of the field in which the crack propagates (Rice, 1988), and the dynamic nature of the crack propagation process (Freund, 1976) produce important changes in the driving forces that propagate the crack must be better understood, before an interpretation of the phenomenon can be attempted to extract a precise value of the interface fracture toughness out of the experiment. Nevertheless, it is clear that the potential for the measurement of the interface toughness is present in the laser spallation experiment.

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