

MEASUREMENT OF INTERFACE STRENGTH BY A LASER SPALLATION TECHNIQUE

V. GUPTA,[†] A. S. ARGON,[‡] D. M. PARKS and J. A. CORNIE

Massachusetts Institute of Technology, Cambridge, MA 02139, U.S.A.

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ABSTRACT

A LASER spallation experiment has been developed to measure the strength of planar interfaces between a substrate and a thin coating (in the thickness range of 0.3–3 μm). In this technique a laser pulse of a high enough energy and a pre-determined duration 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 of the process. The simulation itself is verified by means of a piezo-electric 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 10.5 GPa were determined for the Si/SiC system. For the interfaces between pyrolytic graphite and SiC coatings an average strength of 7.2 GPa was measured, while the corresponding interface strength between a Pitch-55 type ribbon with a fiber-like morphology and SiC coatings was found to be 0.23 GPa. Intrinsic strengths of SiC coatings and Si crystal were also determined using this technique. These were, on the average, 8.6 GPa for Si crystals and 11.9 GPa for a SiC coating. Furthermore, the potential of the laser technique to determine the interface toughness was also demonstrated, provided well-characterizable flaws can be planted on the interface.

1. INTRODUCTION

IT IS NOW widely 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 a possible interface reaction is often present between the matrix and the fiber, which can degrade the strength of the fiber and can lead to premature failure. 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) 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

[†] Present address: Thayer School of Engineering, Dartmouth College, Hanover, NH 03755, U.S.A.

[‡] Author to whom correspondence should be addressed.

catastrophic failure of the part. An important aspect of realizing tailored properties of interfaces is the measurement of the tensile strength and intrinsic delamination toughness of the fiber-coating interface. We have discussed earlier 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 (ARGON *et al.*, 1989). Here, we discuss how the *tensile strength* of such fiber-coating interfaces can be measured by a laser spallation technique.

Although the laser spallation experiment was specifically developed to probe interfaces of interest in composites, the technique can be used to determine the strength of planar interfaces between coatings ($> 0.3 \mu\text{m}$ in thickness) and substrates in general.

Measurement of interface properties is not new in the thin film and coating technology. 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, which have been reviewed by MITTAL (1978). However, none of the tests mentioned above provide an unambiguous measure of the strength of interfaces. Such interface strength between very thin coatings and substrates can be effectively measured by a laser spallation technique developed first by VOSSEN (1978), utilizing shock waves produced by short laser pulses (YANG, 1974; FOX, 1974; READY, 1965; ANDERHOLM, 1970; PEERCY *et al.*, 1970). The technique involves impinging a high energy laser pulse of *ca.* one nanosecond duration onto a thin, energy-absorbing film sandwiched between the back surface of the substrate to which the coating of interest is attached and a confining fused quartz plate. The sudden expansion of the absorbing film generates a compressive shock wave in the substrate directed towards the coating/substrate interface to be tested. The reflection of the compressive wave packet from the surface of the test coating gives rise to a tensile pulse that leads to the removal of the coating, if its amplitude is high enough. The technique was used earlier by PARK (1986) to detach films from substrates. 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. In the present research the laser spallation experiment has been developed in a more definitive way to permit actual interface strength measurements.

2. STRATEGY OF INTERFACE STRENGTH MEASUREMENT

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 ($1.06 \mu\text{m}$). The characteristics of the ideal energy absorbing film are that: it should have high absorptivity; a very small critical absorption depth (much smaller than the thickness of the film); a high melting temperature, high

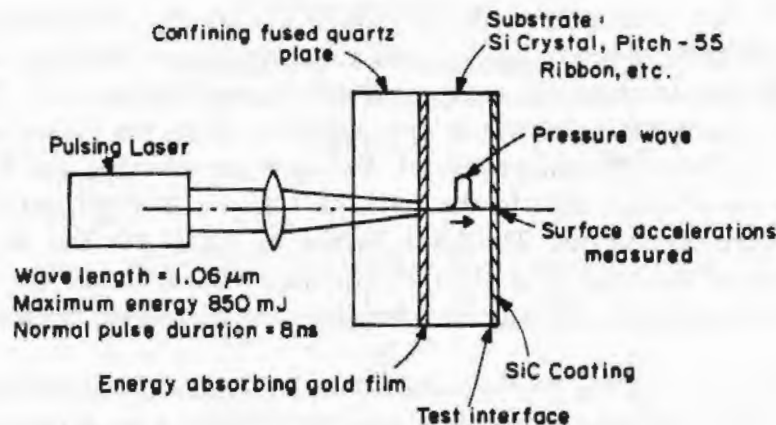


FIG. 1. Schematic view of the laser spallation experiment.

coefficient of thermal expansion, high elastic modulus, low thermal diffusivity, and finally a 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 for various alternative energy absorbing materials (GUPTA, 1990), it was concluded that gold is the most effective material that maximizes the generated stress pulse amplitudes. Experimental and numerical observations confirming these findings are presented below.

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 front 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 best done by laser Doppler interferometry, used widely in 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 $1\ \text{mm}^2$. 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, the quantitative evaluation of the measurements of the interface strength used here was based on a three part strategy.

The first part of the strategy has been the development of a finite element computer simulation code for the conversion of the laser light pulse into a pressure pulse. The simulation monitors the traverse of this pulse through any desired substrate of interest and provides the time history of the resulting tensile amplitude at the interface as the stress wave is reflected from the free surface of the coating of given properties and thickness. In the second part of the strategy, the pressure pulses have been measured in a micro-electronics 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 an X-cut piezo-electric (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 code. 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 acting across the interface that accomplishes this was determined from the computer program.

In what follows, we discuss first in Section 3 the computer simulation of the pressure pulses generated by the laser pulses. In Section 4, we present the experimental details of the microelectronics 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 Si/SiC and C/SiC interface systems for both pyrolytic graphite and Pitch-55 type ribbon substrates in Section 5. In Section 6, the basic results of the computer simulation are generalized to obtain interface stress charts for various coating/substrate systems. Finally, Section 7, provides a discussion of the approach and its potential.

3. 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 from left to right of: fused quartz confining plate (Q), the thin energy absorbing film (F) of thickness Λ , the substrate (S) and the coating (C) of interest of thickness Λ_c . 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 peak power of the laser was 3×10^8 watts. The spatial profile of the laser pulse was Gaussian in nature, while the temporal profile was a distorted Gaussian as shown in Fig. 3 (READY, 1971). The laser could be operated at either 2.5 or 8 ns nominal pulse widths with a maximum achievable energy of 800 mJ in a nominal laser beam diameter of 7 mm.

The critical penetration depth δ of laser light at this wavelength into the gold is only 20 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 was made that the energy is deposited entirely on the interface between the film and the fused quartz confining plate. The cross-sectional area of the laser beam incident on the energy absorbing film was of the order of $1\text{--}2\ \text{mm}^2$.

Additional simplifications used in the simulation included, ignoring temperature dependence in all the relevant physical properties, such as, thermal conductivity, coefficient of thermal expansion, specific heat, density, and elastic moduli. Such variations can, in principle, be taken into account in the finite element solution but

[†] The laser used was in the M.I.T. George Harrison Spectroscopy Laboratory.

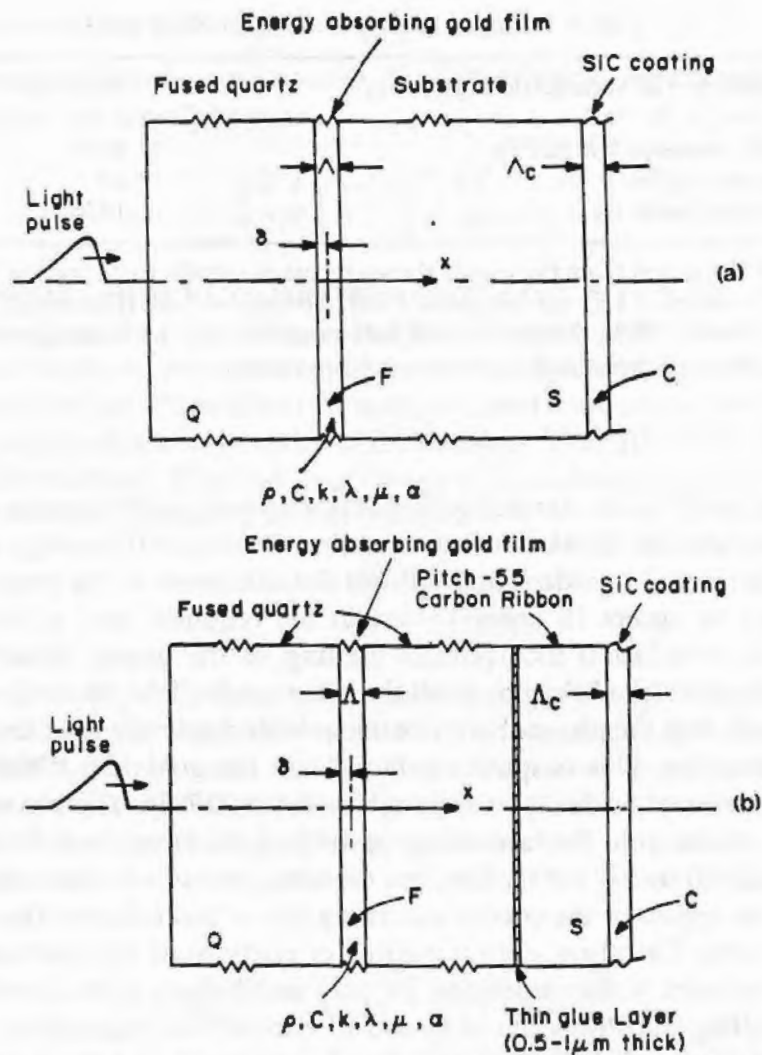


FIG. 2. (a) Configuration of the laser spallation probe with a homogeneous substrate. (b) Configuration for testing the Pitch-55 Ribbon/SiC system.

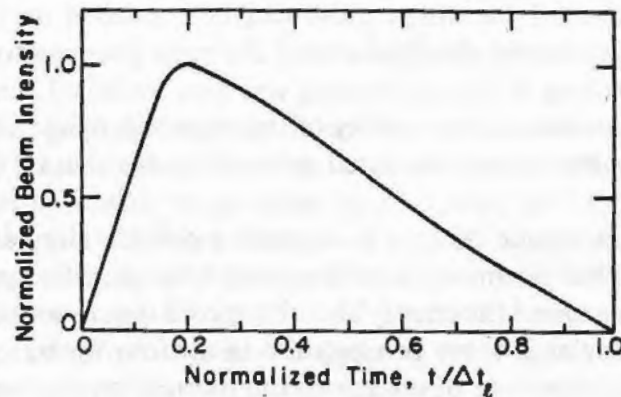


FIG. 3. Temporal profile of the laser pulse.

TABLE 1. *Physical properties of molten gold*

Coefficient of volumetric expansion, $\beta = 3\alpha$ (K^{-1})	7.05×10^{-6}
Bulk modulus,† K (GPa)	85.5
Density (g/cm^3)	17
Melting point (K)	1336

† Estimated from the trends shown by other metals. For example K_s/K_l for cesium = 1.5, potassium = 1.968, sodium = 2.04 (CHAMBERS and ROTHERY, 1969). Where K is the bulk modulus and the subscripts s and l refer to the solid and liquid phase, respectively.

were ignored in the initial simulation since they do not result in major effects. 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 was necessary to accept in order to obtain the required level of interface stress. Therefore, the simulation incorporates melting of the energy absorbing film but considers only solid-like behavior in all the other media. The effect of volume expansion associated with the phase change in the gold during melting of the film has been fully accounted for. This is quite significant for the gold film which undergoes a volume expansion of 5.1% upon melting (BRANDES, 1983). The physical properties used for the molten gold film are shown in Table 1 (LANDOLT and BORNSTEIN, 1971; CHAMBERS and ROTHERY, 1969). 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 gold 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 ns varies between 1 and 10 μm . This distance is negligible as compared to the radius of the laser heating spot of 1 mm. The absence of fluid-like flow of the molten material and small thermal diffusion distance suggest that the individual material points are unaware of 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 was also modelled using the one-dimensional stress wave equations. The validity of this planar propagation assumption will be verified later by the actual measured pressure pulse shapes to be discussed in Section 4 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.

3.2. Governing equations and solutions

The transient heat transfer equations in all relevant media were considered in one-dimensional form and are of the type

$$\left(\frac{\partial^2 T}{\partial x^2}\right)_i = \left(\frac{\rho C}{k}\right)_i \frac{\partial T_i}{\partial t}, \quad (1)$$

where the subscript i refers to the relevant medium of Q , F , or S (the test coating is too far removed from the internal heat absorbing interface to be thermally affected), x is the longitudinal coordinate measured from the energy absorbing interface between Q and F , in the direction of the substrate and, ρ , C , and k are respectively the density, specific heat, and thermal conductivity of the medium. The ratio $k/\rho C$ is the thermal diffusivity of the medium. Since all laser energy is considered deposited on the Q - F interface, the boundary condition at this interface is

$$q = \left(k \frac{\partial T}{\partial x}\right)_q - \left(k \frac{\partial T}{\partial x}\right)_f \quad (2)$$

during the duration Δt_i of the laser pulse, where q is the absorbed laser energy flux. After the laser flux is off, the boundary conditions between Q and F , and F and S require that both the temperature and heat flux are continuous across the interfaces. This is also the case for the F - S interface during the duration of the laser pulse, since all the energy is considered deposited on the Q - F interface.

The associated one-dimensional elastic wave equations are of the form (ACHENBACH, 1984)

$$(\lambda + 2\mu)_i \frac{\partial^2 u_i}{\partial x^2} = \rho_i \frac{\partial^2 u_i}{\partial t^2} + (3\lambda + 2\mu)_i \alpha_i \frac{\partial T_i}{\partial x}, \quad (3)$$

where λ and μ are the two Lamé elastic constants, u is the x -direction displacement, and α is the coefficient of linear thermal expansion for any of the four media i , i.e. Q , F , S , or C . According to the assumed idealization, displacements in the radial direction are vanishingly small. This should hold at the center region of the beam. The axial stress σ in the x -direction is then calculable from the displacement gradients, and is

$$\sigma_i = (2\mu + \lambda)_i \frac{\partial u_i}{\partial x} \quad (4)$$

in all of the media. The elastic wave boundary conditions require that both the displacements and the stresses are continuous at interfaces between various media.

The one-dimensional elastic wave equation (3) is valid for homogeneous isotropic media. However, there are certain substrates of current interest that show anisotropy in their elastic properties. Specifically, the Pitch-55 carbon ribbon and the pyrolytic graphite substrate used to model the Pitch-55 fiber are strongly orthotropic in their elastic properties. The governing one-dimensional wave equations in these media is presented in Appendix 1, and as might be expected, it is of the same form as Eqn (3) as

$$\frac{1}{\eta} \left[\frac{\partial^2 u}{\partial x^2} - \kappa \frac{\partial T}{\partial x} \right] = \rho \frac{\partial^2 u}{\partial t^2}, \quad (5)$$

where $1/\eta$ and κ/η are respectively the effective modulus E_{eff} and effective coefficient of expansion α_{eff} , which in turn depend on various anisotropic compliances. Thus, the same solution as the one for the isotropic case should be valid, albeit with different interpretation for the elastic constants. Explicit expressions are given in Appendix 1.

3.3. Governing equations in normalized form

Since solutions to the two equations are necessary in both the temporal and spatial domains to determine the maximum tensile stress at the interface between S and C for different pairs of S and C , it is useful to present these differential equations in a normalized form in terms of the properties and the thickness of the energy absorbing film. To accomplish this the following dimensionless variables were introduced:

$$\zeta = x/\Lambda; \quad \tau = t/t_0; \quad \theta = T/T_0; \quad \phi = u/u_0; \quad \Sigma = \sigma/\sigma_0, \quad (6)$$

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

$$t_0 = (\Lambda/c)_f; \quad T_0 = \frac{q\Delta t_l}{(C\rho\Lambda)_f}; \quad u_0 = \left(\frac{\Lambda\gamma}{\rho\delta c^2} \right)_f q\Delta t_l; \\ \sigma_0 = (\gamma/\delta)_f q\Delta t_l \quad \text{and} \quad \gamma = (3\lambda + 2\mu)(\alpha/\rho C), \quad (7a-e)$$

where in turn, Λ , c , C , ρ , δ , γ 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.

In the normalized coordinates, the basic governing equations for heat transfer in the various media become correspondingly

$$\frac{\partial^2 \theta_i}{\partial \zeta^2} = \frac{1}{\beta_i} \frac{\partial \theta_i}{\partial \tau}, \quad (8)$$

$$\beta_i = \left[\frac{k}{\rho C} \right]_i \left[\frac{1}{c\Lambda} \right]_f,$$

where, again, i stands for either Q , F , or S . The corresponding differential equation for the pressure wave then becomes

$$\frac{\partial^2 \phi}{\partial \zeta^2} = \frac{\partial^2 \phi}{\partial \tau^2} + \frac{\partial \theta}{\partial \zeta} \quad (9)$$

for the energy absorbing film and

$$\frac{\partial^2 \phi_i}{\partial \zeta^2} = H_i \frac{\partial^2 \phi_i}{\partial \tau^2} + K_i \frac{\partial \theta_i}{\partial \zeta} \quad (10)$$

for the other media, where i stands for Q , S , and C . The different physical properties of these media are represented in terms of the properties of the energy absorbing film through the material constants H_i and K_i , which are defined as

$$H_i = \left(\frac{c_f}{c_i} \right)^2; \quad K_i = \frac{(\gamma C/c)_i}{(\gamma C/c)_f}. \quad (11a,b)$$

Since the form of the wave equation for the orthotropic medium is the same as Eq. (3), the same normalization scheme can also be applied for Eq. (5).

These differential equations, i.e. (8)–(10) with the appropriate boundary conditions between the various media have been solved using a finite element approach with the ABAQUS (1988) code.

3.4. Discretization scheme and the solution strategy

The size of the elements for the heat transfer analysis were chosen in order to satisfy the numerical stability criteria relating the time increment to the element size via the thermal properties of the media (SALVADORI and BARON, 1961), i.e.

$$\Delta t > \left(\frac{\rho C}{6k} \right) (\Delta l)^2, \quad (12)$$

where k , ρ , and C are respectively the thermal conductivity, density, and the specific heat of the medium, Δt is the time increment used and Δl is the length of the element. Since the input laser flux lasts for 2.5 ns, temperature solutions are obtained for a total time period of 12 ns, within which the entire stress pulse of interest is generated in the substrate medium. Therefore, for the heat transfer analysis, the substrate and the confining quartz were discretized to depths corresponding to a diffusion distance of the thermal pulse corresponding to 120 ns. Solution of Eq. (3) does not entail any specific requirements on the element size. However, in order to resolve the details of the transient structure of the generated stress pulse, an element size that is a small fraction (0.01) of the stress wavelength was used. Since the generated stress wavelength is of the order of 5–10 μm , it is sufficient to model the substrate and the confining quartz to a depth of only 150 μm . In order to avoid internal reflections of the travelling wave by elements, the element size was kept constant within the same medium. This excludes the possibility of using the same element size for the heat transfer analysis, since such a choice would have made the problem too big to handle by the available computer system. The element size for the stress wave problem was chosen to be five times the one used for the heat transfer analysis and the mode numbers in the mesh used for the stress analysis were chosen to correspond precisely with the corresponding nodes in the heat transfer analysis mesh. Furthermore, the element types for the two analyses were chosen to produce compatible thermal and elastic strains.

The following effective constants were defined

$$E_{\text{eff}} = (\lambda + 2\mu); \quad \alpha_{\text{eff}} = \alpha \frac{(1 + \nu)}{(1 - \nu)} \quad (13a,b)$$

in order to write Eq. (3) in the same form as the standard one-dimensional plane stress wave equation, i.e.

$$E_{\text{eff}} \frac{\partial^2 u}{\partial x^2} = \rho \frac{\partial^2 u}{\partial t^2} + \alpha_{\text{eff}} E_{\text{eff}} \frac{\partial T}{\partial x}. \quad (14)$$

For orthotropic media, effective moduli E_{eff} and the coefficients of expansion α_{eff} have been defined in Appendix 1 in order to express the governing wave equation in the same form as Eq. (14). Hence, with these effective elastic constants, Eq. (3) has been solved by using the same ABAQUS code used to solve the standard plane stress wave equation.

Volume changes in the gold film upon melting were considered by defining an effective α by considering such volume changes to occur over a 1K range. The discrete increase in volume during melting had to be accommodated in a special way in the ABAQUS code that is described in Appendix 2.

3.5. Solution of the governing equations

Figures 4a and 4b show the traverse of pressure fronts both forward into S and backward into Q spaces for two different normalized times of 70 and 109 for the material constants H_q , K_q , and H_s and K_s (characteristic of a PE crystal substrate) are given in the figure. A more expanded figure of the pressure wave profile in the piezo-electric substrate is shown in Fig. 5 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_l 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 this interface toward the forward direction as a tension wave, because of the lower stiffness and much lower density of the fused quartz in comparison to the gold film. This was 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 "rebounding" effect should be favored in systems, in which the "flash" expansion of the energy absorbing packet cannot be significantly counteracted by the elastic wave tending to disperse the resulting misfit during the time while the laser pulse is on; i.e. systems in which

$$\left(\frac{k}{\Delta t_l C \rho c^2} \right)^{1/2} = \frac{k}{\Lambda_c C \rho c}, \quad (15)$$

i.e. the ratio of the average "thermal velocity" to the sound velocity is high. In Eq.

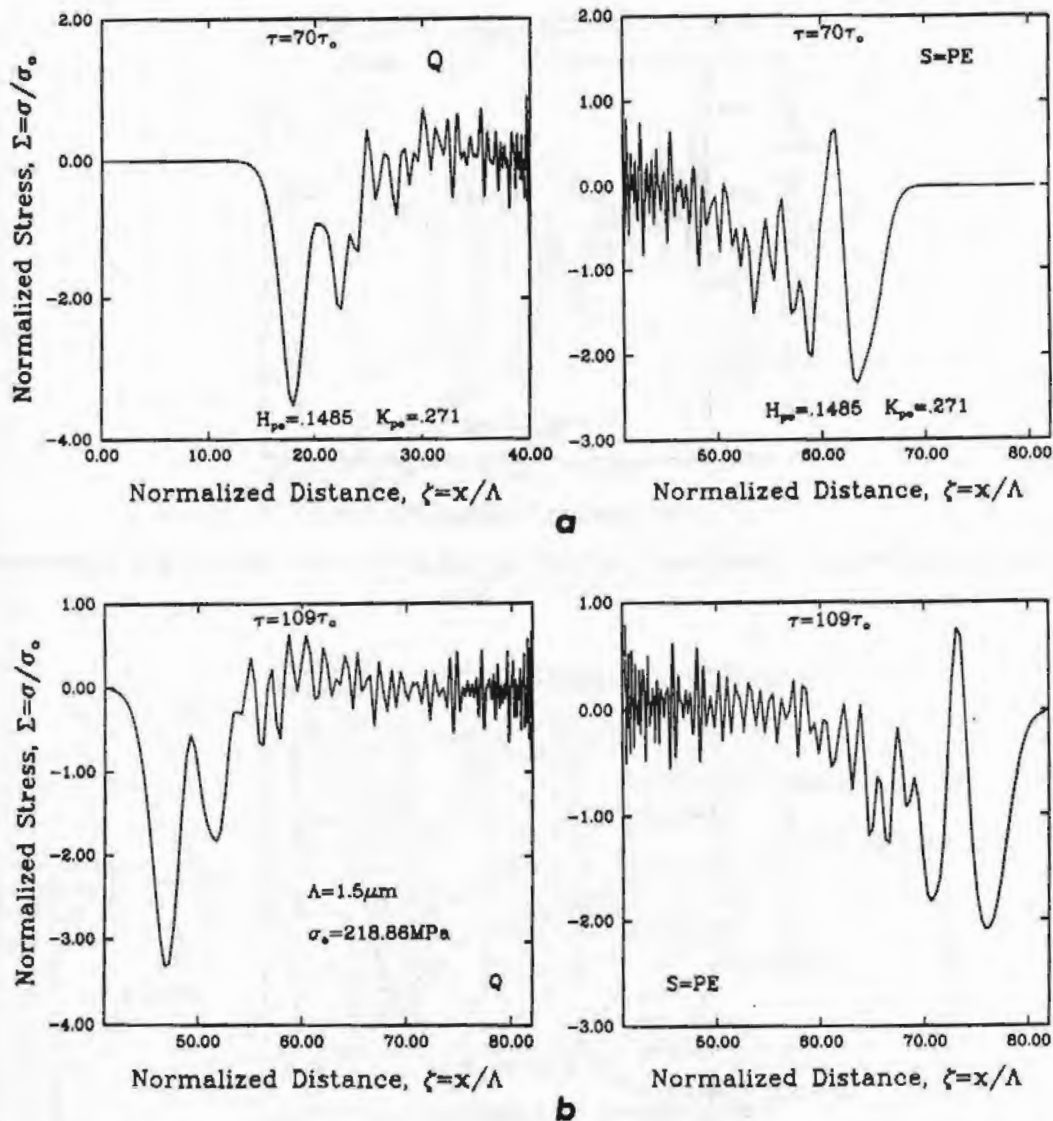


FIG. 4. (a), (b) Simulated propagation of the stress pulses in Q and S spaces for two stages of dimensionless time.

(15) above, Λ_c is the thickness of the coating to be blown-off, which governs the ideal duration of the laser pulse. 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 Eq. (15) 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} . Figure 6 shows the structure of the pressure pulse generated with carbon as the energy absorbing film, where the tension peak is absent.

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 experienced by the interface between a Si substrate and a SiC coating of $2 \mu\text{m}$ thickness, due to the pulse of Fig. 4, is shown in Fig. 7. The initial flat portion of the history prior to the main signal corresponds to the time lag in the arrival of the stress

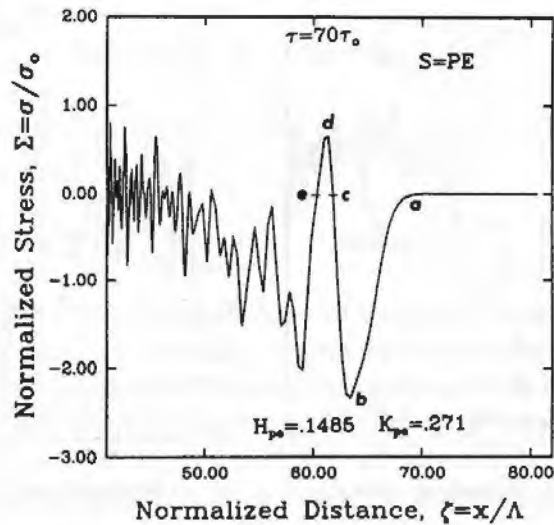


FIG. 5. Detailed structure of the simulated stress pulse in the piezo-electric substrate S at a normalized time of $\tau = 70\tau_0$.

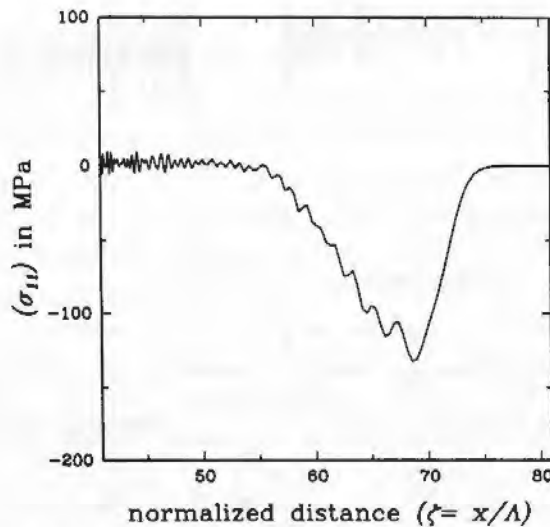


FIG. 6. Structure of the simulated stress pulse in the piezo-electric crystal with carbon as the energy absorbing film.

pulse at the interface. Thus, for a given threshold laser fluence resulting in spallations, the interface strength can be calculated from the amplitude of such plots.

4. EXPERIMENTAL STRESS PULSE MEASUREMENTS (PART II OF STRATEGY)

4.1. The micro-electronics device

A schematic view of the micro-electronics device with the X-cut piezo-electric (PE) crystal is shown in Fig. 8. The assembly, consisting of fused quartz plate (Q), energy absorbing (gold) film (F), the PE crystal, taking the place of a substrate, is shown encased in a bakelite housing. Gold films of $1\ \mu\text{m}$ thickness were chosen to completely

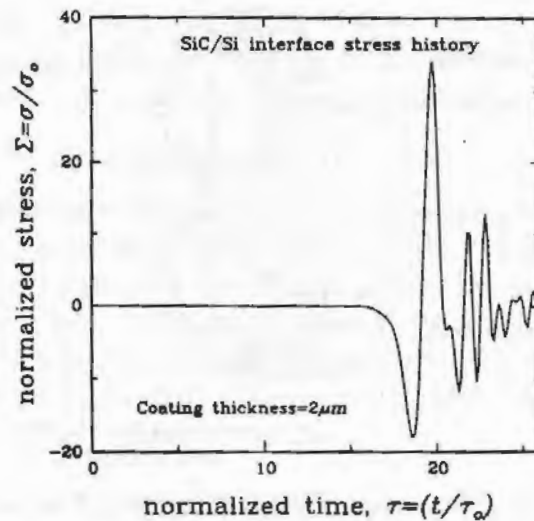


FIG. 7. Simulated stress history at the interface.

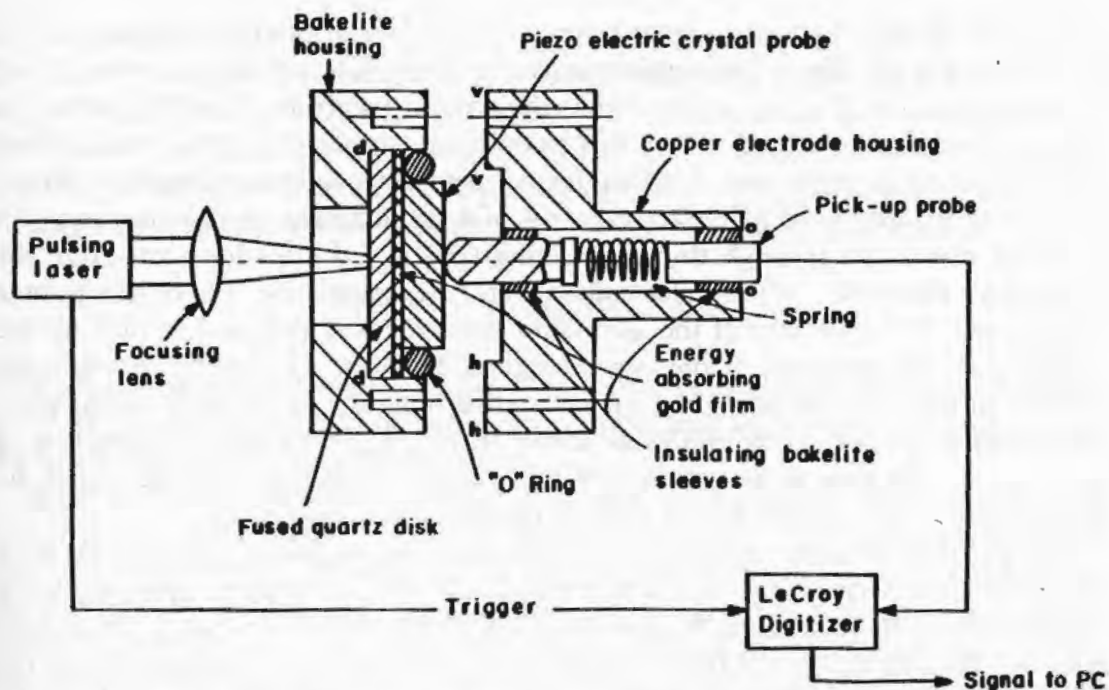


FIG. 8. A schematic view of the micro-electronics device with the X-cut piezo-electric crystal used to characterize the laser pulses.

absorb the laser fluence in order to avoid the well known radial cracking phenomena (VOLKOVA, 1967) in the substrate disk when direct penetration of laser energy occurs. The laser energy absorbing gold film also acts as a ground electrode for the piezo-electric crystal. A rubber "O" ring between the housing and the PE crystal radially compresses the latter when the copper electrode housing (C) is attached onto the bakelite housing. The radial pre-compression of the substrate disc helps to counteract the cracking of the substrate disc, subjected to the sudden impact of the generated

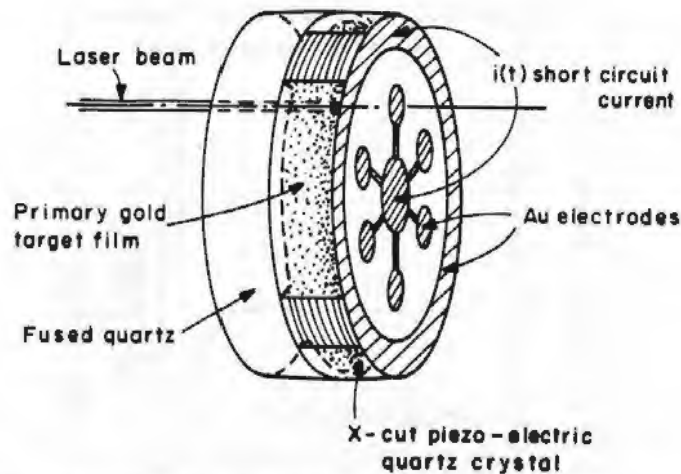


FIG. 9. Perspective view of the quartz gauge exhibiting the position of the laser beam with respect to the pickup electrodes.

stress pulse. Figure 9 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 electrode 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 μm wide) constitute the secondary electrode. These electrodes were deposited by photolithographic techniques.[†] The geometry of the secondary electrode was designed to pick up the one-dimensional structure of the current signal. This was ensured by focusing the laser beam to a 2–3 mm spot onto the laser absorbing film in line with one of the 2 mm diameter secondary electrodes as shown in Fig. 9. This assembly permitted 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 laser beam.

This assembly of fused quartz confining plate and piezo-electric quartz probe were then placed into a bakelite housing which was closed off by screwing a copper electrode housing onto it that contained the central pickup probe as shown in Fig. 8. The copper electrode housing makes electrical contact to the outer ground electrode ring on the rear face of the crystal. Thus, the whole copper enclosure acted as a ground electrode, while a specially constructed pickup probe with a copper leg, insulated from the copper housing acted as the central secondary electrode.

The short circuit current derived between the two electrodes was picked up via a shielded BNC plug fastened on top of the pick up probe and fed into a LeCroy high-speed digitizer by a BNC cable. The high speed LeCroy digitizer (1.3 GHz sampling rate) was needed to properly define the temporal shapes of the stress pulses with sub-nanosecond rise times. Since the experiments were performed in the isolated pulse mode, the digitizer was triggered by an electronic pulse that was fired just before the

[†] The photolithography was performed in the M.I.T. VLSI Center.

lasing of the optical pulse. The details of the micro-circuitry and various considerations in designing the micro-electronics device can be found elsewhere (GUPTA, 1990).

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 $50\ \Omega$ 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)

$$i(t) = \frac{fAc}{l} [\sigma_0(t) - \sigma_l(t)], \quad (16)$$

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, $\sigma_0(t)$ and $\sigma_l(t)$ are the amplitudes of the stress pulses on the ground electrode (impacted energy absorbing film) and front surface electrodes of the crystal, respectively. The term $\sigma_l(t)$ is to be interpreted as the amplitude that is incident upon the free surface, but is annulled instantly by an equal and opposite virtual amplitude in order to satisfy the free surface boundary condition. When the wave length of the stress packet is much smaller than the crystal thickness, Eq. (16) reduces to

$$i(t) = \frac{fAc}{l} \sigma_0(t) \quad \text{for } t < l/c, \quad (17)$$

which gives the time-dependent profile of the stress wave. Equation (17) predicts that the stress at the input electrode is directly proportional to the instantaneous current for times less than the wave transit time.

In the past, most researchers (YANG, 1974; FOX, 1974; READY, 1965; ANDERHOLM, 1970; PARK, 1986) have used the technique to measure the stresses at the input electrode of the crystal. However, if the thickness of the crystal is much greater than the width of the stress pulse, then two short circuit current signals corresponding to each electrode are obtained. While the stress is related to the current signal at the ground electrode through Eq. (17), the current signal measured when the stress pulse is reflected from the front electrode is twice that measured from the ground electrode. Because the laser fluences were sufficient to melt the input electrode, it became necessary to record the current signal emitted from the front electrode for more reliable measurement of the generated stress pulse.

4.3. Measurement of stress pulses

While the temporal structure of the stress pulse is determinable from the measured short circuit current density from Eq. (17), the stress itself is related to the time rate of production of thermal misfit, which in turn, is proportional to the laser fluence in a

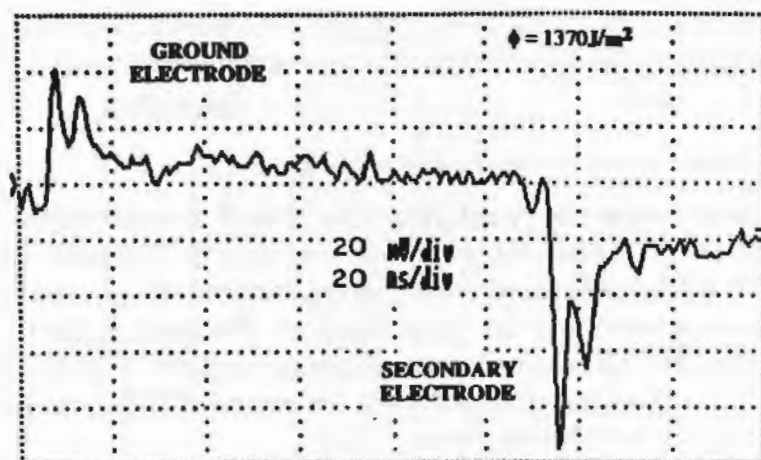


FIG. 10. A typical short circuit current trace as recorded by the PE crystal device.

pulse of given duration Δt_i . To determine this fluence, the total energy in a pulse was recorded and divided by the beam cross-section area, which was measured from the size of the discolored zone on an exposed (black) Polaroid photographic paper struck by the laser beam separately. A typical short circuit current trace recorded by the PE crystal device is shown in Fig. 10 for a laser fluence of 1370 J/m^2 (the current is recorded as the voltage drop across the 50Ω impedance of the digitizer). The trace shows, on the left, the signal generated by the original impingement of the laser pulse on the sandwiched gold film electrode between the PE crystal and the confining Q plate. After the stress pulse traverses through the PE crystal the pulse is reflected from the free surface bearing the secondary gold pickup electrode. Because of the reflection conditions from the free surface, where the stress reverses from compressive to tensile, the recorded pulse is twice as large as the impingement pulse, but otherwise has a nearly identical structure (GUPTA and EPSTEIN, 1990). For the laser pulse fluence in Fig. 10, the gold film has not melted but merely underwent a "flash" thermal expansion in the solid state. This has been verified by examining the struck portion of the gold film after the laser impingement.

The changes in the structure of the compressive stress pulse with increasing fluence are shown in Fig. 11. Traces A and B at the bottom of the figure for laser fluences of 1370 and 1413 J/m^2 are similar in shape and result from the flash expansion of the gold film without melting. The double peak structure of the pulse followed by a tail of a gradually decaying set of ringing oscillations will be discussed below. Above a critical laser influence, somewhere between 1413 and 2546 J/m^2 , the target area of the gold film melts and the typical trace corresponding to this condition broadens to the shape of the trace shown as C in the figure. The micrograph of Fig. 12 shows the tell-tale evidence of local melting for this case in the struck region of the gold film. Upon melting, the bulk modulus of the film drops to about half its value in the solid state and its coefficient of thermal expansion increases by a factor of two (CHAMBERS and ROTHERY, 1969). Also, there is a 5.1% increase in volume associated with the melting of the gold film (BRANDES, 1983). In addition, the considerably larger fluence of the incident energy makes the thermal quenching less effective. For all of these reasons,

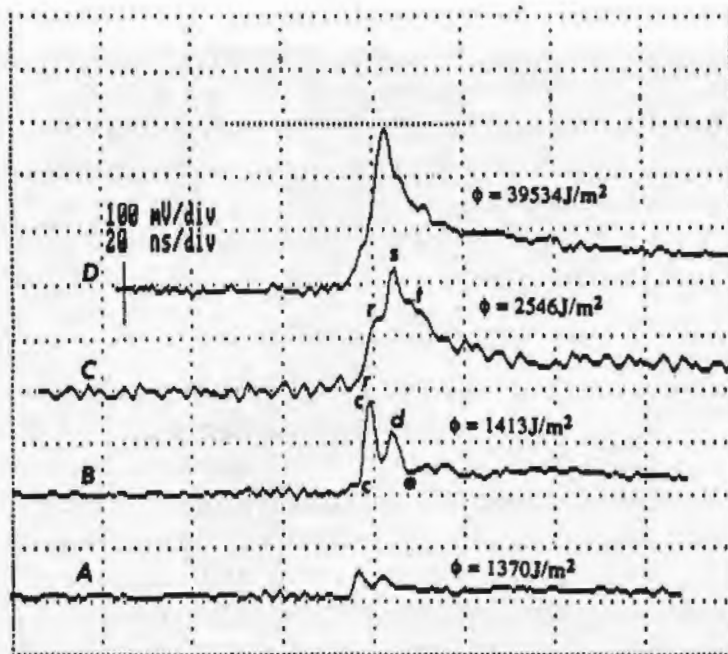


FIG. 11. Effect of the laser fluence on the structure of the stress pulse with gold as the laser energy absorbing film.

the structure of the pulse significantly alters and becomes much broader and there is a substantial increase in the amplitude of the stress pulses. The initial rising portions of the stress pulses B and C shown as *cc* and *rr* are nearly identical, since in this range in both cases, the gold film is still solid. When the film melts, corresponding to point *r* in the intermediate shelf of trace C the amplitude of the rest of the pulse *cde* of trace B is elevated to *rst* in trace C. The incident laser fluence for case C also makes the quenching time longer, so that the thermal misfit persists longer, as is clear from the above trace. Similar sequences of events showing the evolution of the structure of the stress pulse were also observed with platinum as the laser absorbing material.

At very high fluences, the film vaporizes, resulting in loss of opacity and in extensive ionization of the gold layer. The momentum imparted to the substrate by the ionized vapor depends on its level of ionization and the mode of interaction of this vapor with the laser pulse. These complex effects result in a saturation in the amplitude of the stress pulse with laser fluence (READY, 1971). At the stage of vaporization, the simulation discussed in Section 3 which considered the energy absorbing film as a perfectly opaque solid ceases to be valid, and also the effectiveness of the conversion of the light energy to pressure pulse is radically diminished. Clearly, the utility of the method is limited to solid-like, or confined melting behavior of the energy absorbing film. The decreasing effectiveness of the method at vaporization of the film is shown in Fig. 13, where the peak tensile stress has been calculated from Eq. (17). Figure 13 shows that while the gold film remains solid, the peak tensile stress increases sharply (ranges A–B). The stress rises less rapidly at an average rate of 2.76×10^{-2} MPa/(J/m²), as the film undergoes melting (range B–D). For very high fluences the peak tensile stress that can be attained reaches a saturation level of around 1.2 GPa

at fluences, of $4 \times 10^4 \text{ J/m}^2$. At these high fluences, the above mentioned complex effects set in, and the technique is no longer useful. Figure 13 also compares the relative effectiveness of two different energy absorbing films. For the same fluence, gold is clearly the most efficient film. Chromium reaches an early saturation of peak stress at a fluence of about $7 \times 10^3 \text{ J/m}^2$. Other less complete experimental observations with different laser absorbing films and effects such as the dependence of the stress pulse structure on the laser width have been given elsewhere (GUPTA, 1990).

4.4. Comparison of measurements with predictions

The simulation presented in Section 3 considered only a one-dimensional case of propagation of elastic waves due to the rate of change of thermal misfit produced by the laser pulse. That the central portion of the stress wave is nearly one dimensional is demonstrated in Fig. 14, 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 current profile due to the reflected wave by a factor of two above the input signal, the two profiles are quite similar in the main portion, but differ in the structure of their tails. (A slight reduction of the reflected wave at the secondary electrode in comparison with the impinging wave at the ground electrode is observable and is probably due to internal friction in the PE crystal.) This similarity is taken as evidence of planarity of the central portion of the main pulse.

Figure 15 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). For this and the previous evaluations of the stress pulses through the use of Eq. (17), the needed piezo-electric coefficient f is given in Table 2. In addition, Table 2 also gives the relevant values of the physical constants of the confining fused quartz disk that enter the computer simulation presented in Section 3. Figure 15 shows that the predicted profile of the stress pulse for an absorptivity of 0.18 for the gold film is very similar in broad outline to the profile measured by the PE device. A similar comparison of the predicted and the experimentally measured profiles with carbon (for an absorptivity of 0.23) as the laser energy absorbing film gave somewhat poorer agreement for the tail end of the temporal profile of the stress pulse. The physical properties of carbon film used in the numerical simulation are also listed in Table 2.

Figure 14, which shows the structure of the recorded pressure pulse for an energy absorbing gold film undergoing a flash expansion in the solid state, demonstrates the presence of two compression peaks with an intervening deep depression which, in the simulation of Fig. 5, appeared as a tension peak. The depression in Fig. 14 has remained in the compression region and has not gone into tension as the simulation predicts. The source of this discrepancy is not clear. In spite of this imperfection, however, there is sufficiently good agreement between the experimental measurement and numerical prediction to make the computer model a useful tool to evaluate experimental results on spallation to determine interface stress histories in arbitrary substrate/coating pairs of interest. The interface stress is determined from the amplitude of the calculated interface stress history plots of the type shown in Fig. 7.

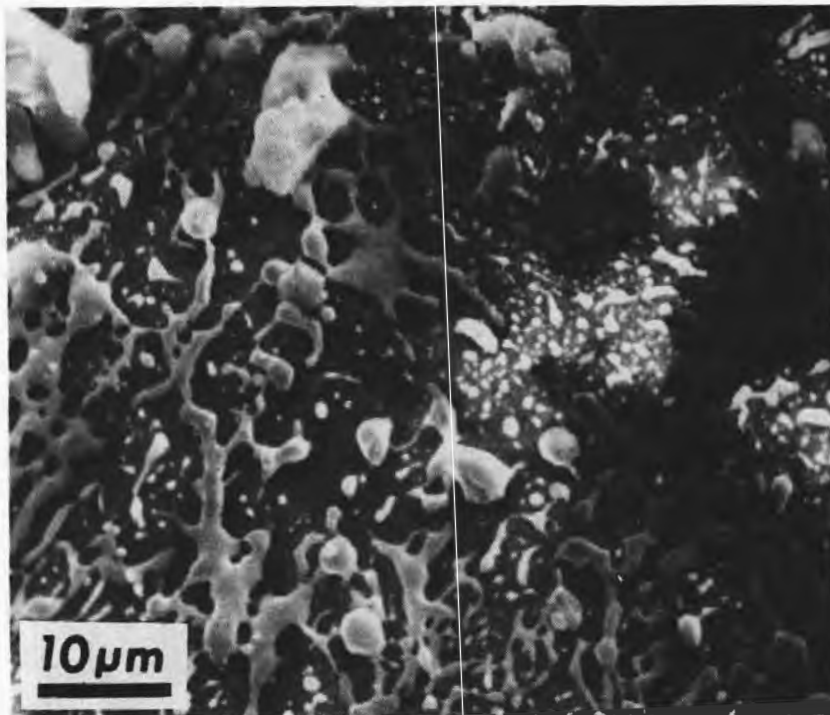


FIG. 12. Micrograph showing the evidence of local melting in the region of the gold film struck by the laser beam.

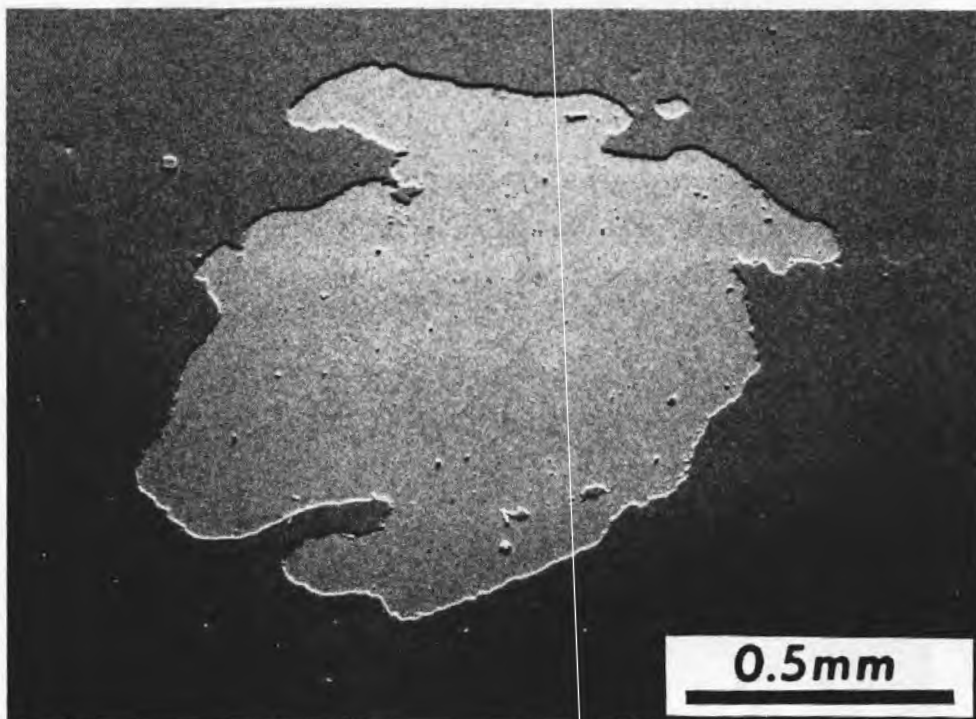


FIG. 16. Spalled spot of SiC coating from Si substrate.

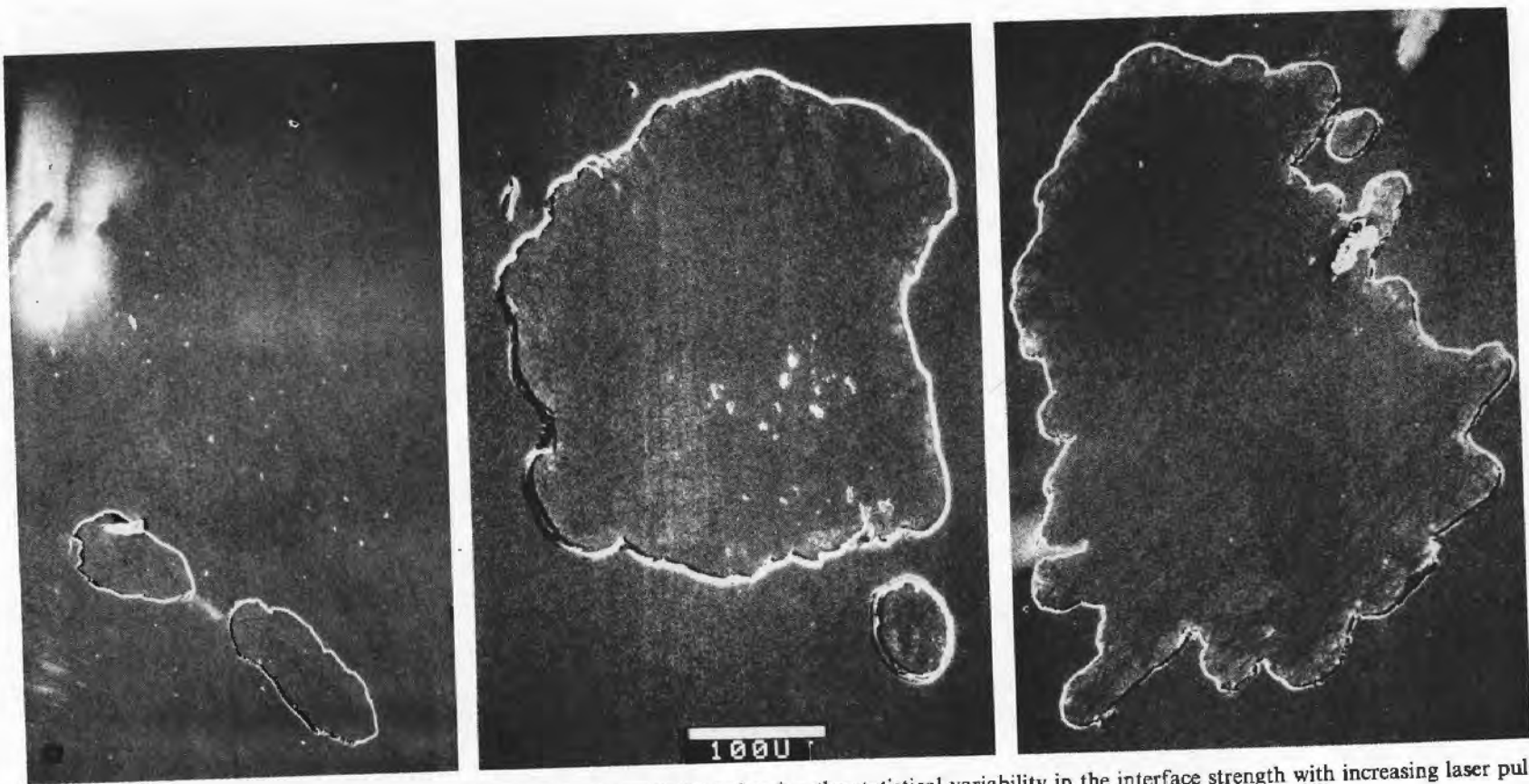


FIG. 17. (a)–(c) Sequence of spalled pattern of SiC coating from Si disks showing the statistical variability in the interface strength with increasing laser pulse fluences of 13,357; 13,833, and 14,265 J/m², respectively.

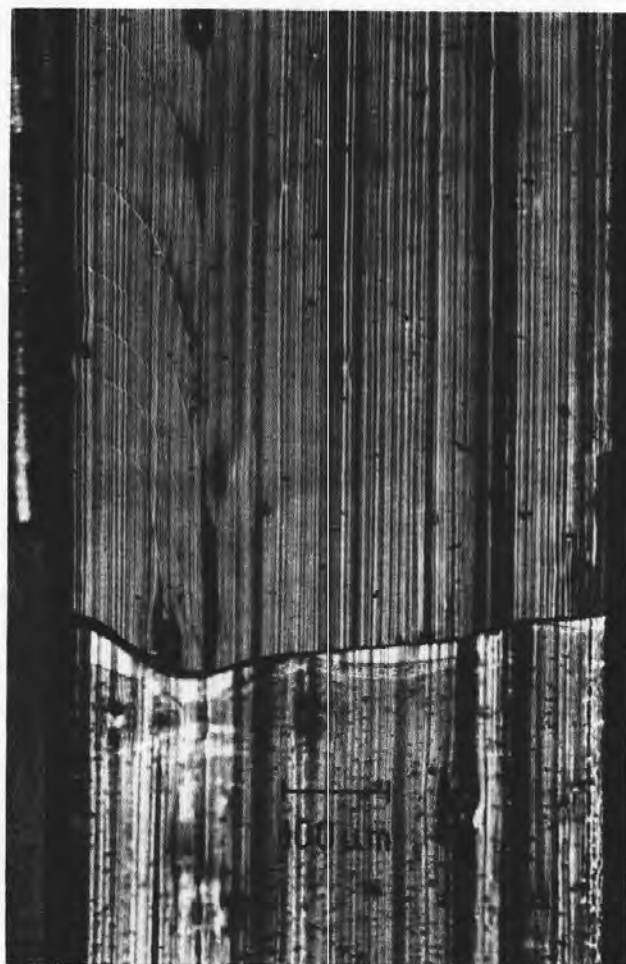


FIG. 19. High magnification view of the spalled spot of SiC coating from the Pitch-55 ribbon surface.

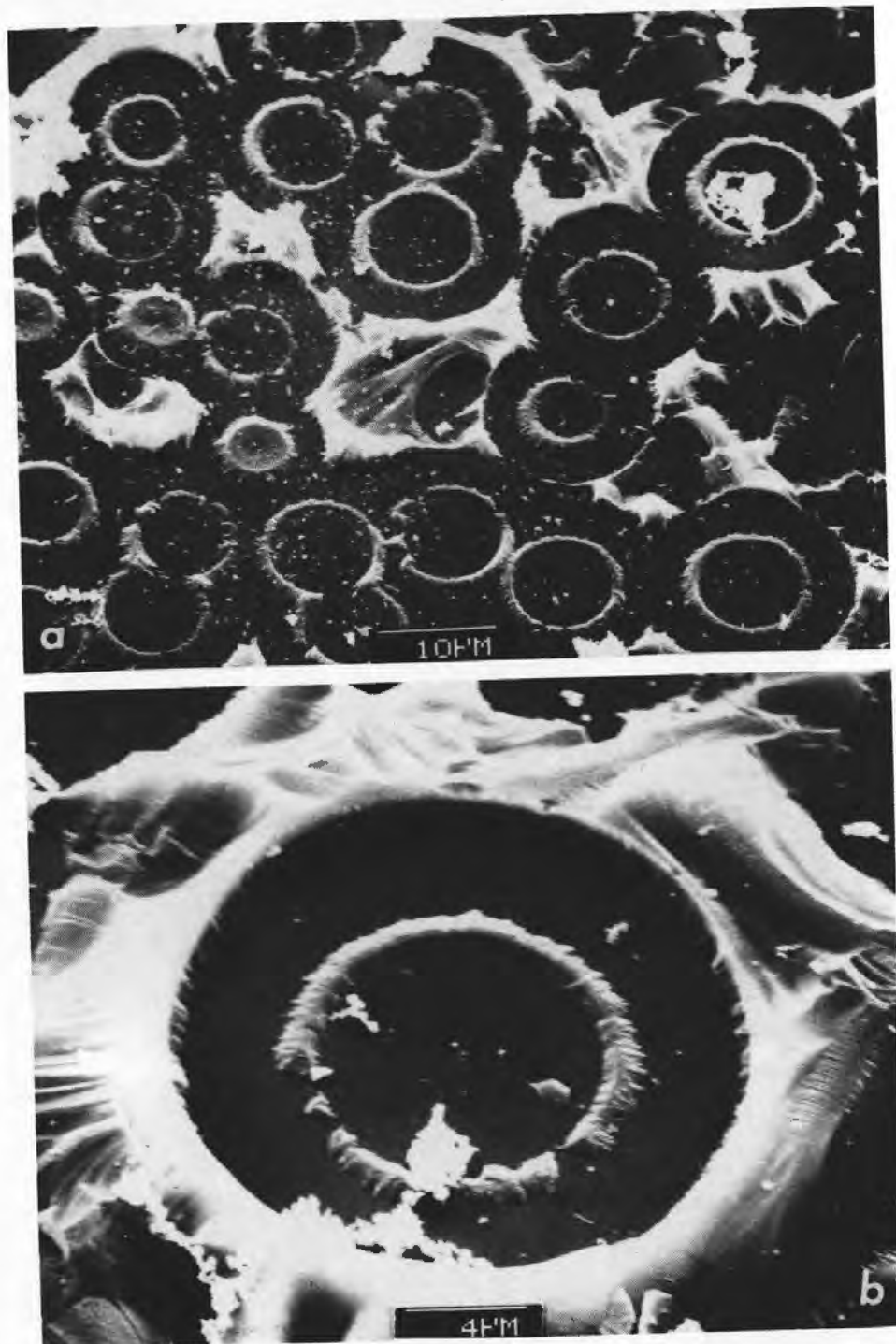


FIG. 24. Spalled crater patterns of SiC.

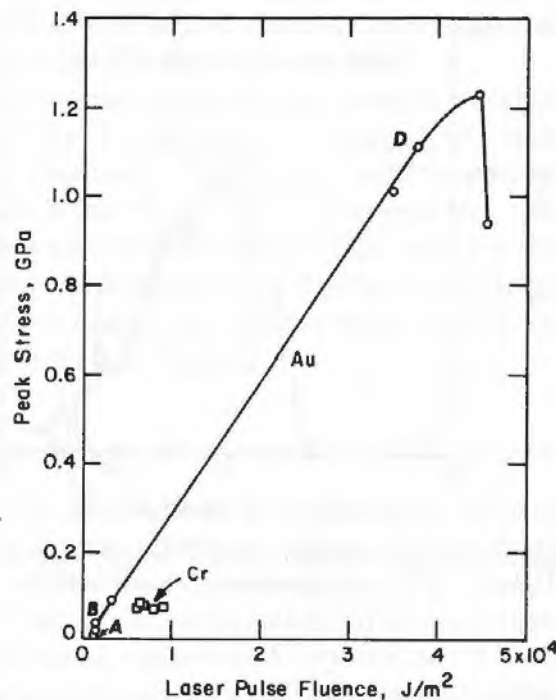


FIG. 13. Relative effectiveness of two energy absorbing films in generating the stress pulses.

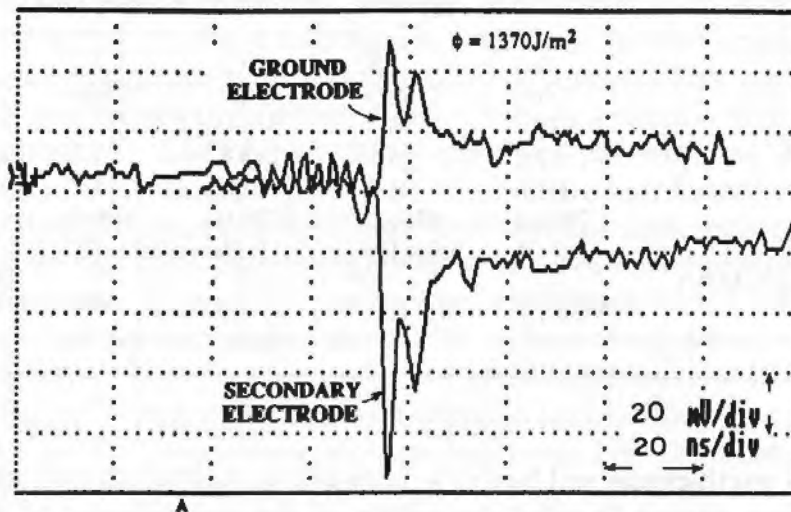


FIG. 14. Evidence of planarity of the travelling stress wave.

5. SPALLATION EXPERIMENTS (PART III OF STRATEGY)

5.1. Choices of systems

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, to mimic surface morphologies of Pitch-55 fibers, and Pitch-55 (P-55) type carbon ribbons of 600 μm

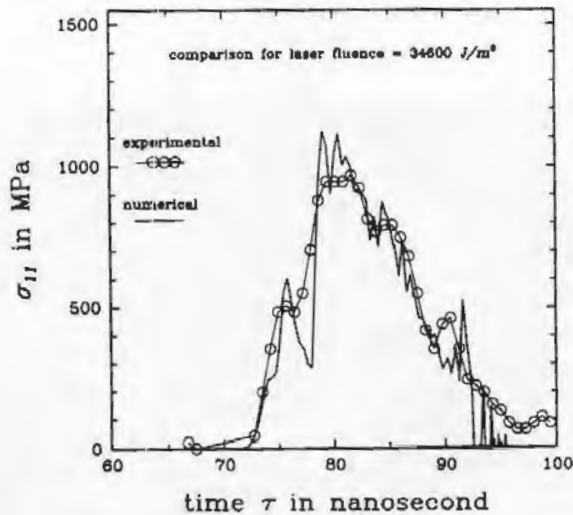


FIG. 15. Comparison of the simulated and measured profiles of the input stress pulse for a fluence of 34,600 J/m² for gold as the laser energy absorbing film.

TABLE 2. *Relevant physical constants for fused quartz, gold, and piezo-electric quartz crystal, silicon crystal, and carbon, entering the computer simulation*

	Silicon	Fused quartz	Gold film	Carbon film	Piezo-electric quartz
ρ (g/cm ³)	2.3	1.8	19.3	2.2	1.8
C (J/Kg K)	729.0	1130.0	130.0	1800.0	1130.0
k (J/ms K)	138.0	1.7	316.0	3.2	1.7
λ (GPa)	62.0	84.3	145.8	7.5	84.3
μ (GPa)	41.0	21.1	27.8	11.2	21.1
c (m/s)	7910.0	8380.0	3230.0	3690.0	8380.0
γ	1.21	5.8×10^{-2}	0.36	4.3×10^{-2}	0.58
f [(coulombs/m ²)/MPa]	—	—	—	—	21.5×10^{-7}
α (K ⁻¹)	7.6×10^{-6}	4×10^{-7}	14.1×10^{-6}	3.8×10^{-6}	4×10^{-6}
ϵ (absorptivity)	—	—	(0.18)	(0.23)	—

width and 35 μm thickness and having a meso-phase morphology very similar to the Pitch-55 carbon fibers.† Detailed description of the morphologies of the Pitch-55 ribbon and the PG can be found elsewhere (GUPTA and ARGON, 1991 and GUPTA *et al.*, 1989 respectively). In all these cases, the coatings to be removed were amorphous SiC coatings prepared by the plasma assisted chemical vapor deposition technique.‡ The coatings had thicknesses in the range of 1–3 μm . They were deposited in a nearly stress free manner by maintaining the substrates at a certain temperature to prevent the entrapment of significant concentration of hydrogen, that otherwise would have

† These 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. Schultz.

‡ We are grateful to Mr B. Chambers for the preparation of the SiC coatings.

resulted in high residual compressive stresses and premature delamination in a manner described in detail by us earlier (ARGON *et al.*, 1989).

The Si/SiC, PG/SiC interface systems were tested in an experimental configuration of the type shown in Fig. 2a, whereas for the P-55/SiC system, the configuration shown in Fig. 2b was employed. The coating could be successfully spalled off in all cases with appropriate levels of laser pulse energies. The threshold laser energy at which spallation occurs was recorded using a light power meter. Since the amplitude of the stress pulse should depend upon the absorbed laser fluence, the diameter of the laser beam was recorded on exposed photographic film as described above for the tabulation of the threshold laser fluence.

5.2. Experimental observations and discussion of results for various interfaces

Si/SiC coating interface. Figure 16 shows a micrograph of the spot from where the SiC coating has spalled off in one of several different samples that were tested. The laser fluence necessary to achieve this spallation was 7388 J/m^2 . The interface strength corresponding to this laser fluence as calculated from the computer simulation is 5.29 GPa. The relevant physical constants of Si crystals and SiC coatings that were used in the computer simulation are given in Table 2. The thickness of this amorphous SiC coating of low modulus, deposited at low ion beam energy was $1.5 \mu\text{m}$. 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. It is well known (WEIBULL, 1939; MCCLINTOCK and ARGON, 1966) that brittle fracture shows considerable scatter due to the statistical variability of strength. This variability in any given sample was investigated further. Figure 17 shows a sequence of micrographs of a spalled pattern of a low modulus SiC coating of $3 \mu\text{m}$ thickness. The laser was focussed onto a 2 mm diameter spot. At an intermediate laser fluence of $13,357 \text{ J/m}^2$ ($\sigma = 10.19 \text{ GPa}$), the coating came off only from scattered weak spots at the interface as shown in Fig. 17a. Thus, the coating remained intact over a large part (about 90%) of the stressed area. As the laser fluence was increased to $13,833 \text{ J/m}^2$ ($\sigma = 10.55 \text{ GPa}$), the spalled area increased to about 50% of the stressed area as shown in Fig. 17b. With a further increase of laser fluence to $14,265 \text{ J/m}^2$ ($\sigma = 10.88 \text{ GPa}$), the coating spalled off almost uniformly from the interface over the entire stressed area, as shown in Fig. 17c. The sequence of events depicted in Figs 17a–c indicates that for the Si/SiC interface, the strength varies from 10.19 to 10.88 GPa. This variability in strength of only 7% is surprisingly low for a brittle interface.

Examination, at higher magnification, of the edges of spalled spots such as that in Fig. 17 showed them to be sharp, abruptly dropping down to a clean flat surface, suggesting that the SiC coating had come off uniformly from the substrate at the interface. To verify this, Auger electron spectroscopy (AES) was performed on the spalled spot to determine the path of the fracture. The AES of the surface of the spalled spot and on the coating gave nearly identical element profiles. The strong signal of carbon from the spalled spot is interpreted to be due to either the slight penetration of carbon into the top layers of the Si crystal from the SiC or due to carbon contamination from the atmosphere while the spalled sample was kept in the

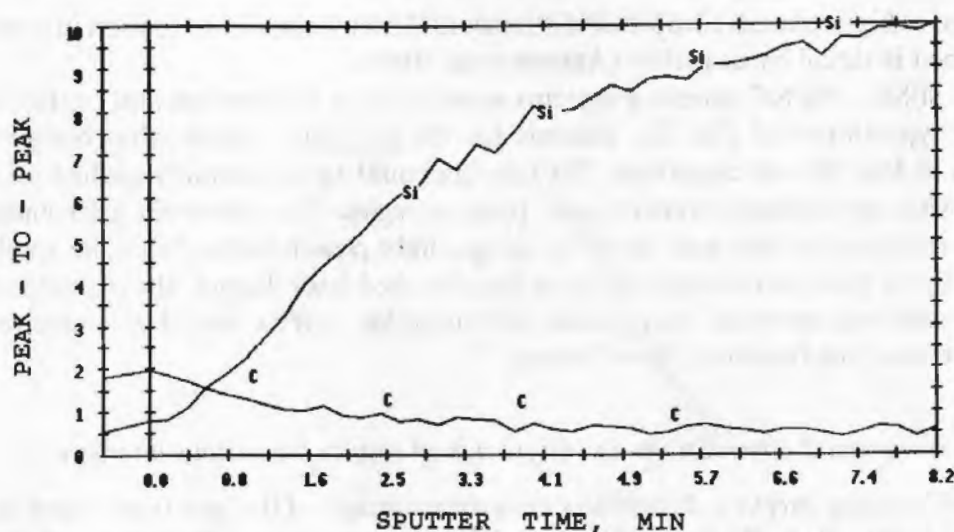


FIG. 18. Elemental composition profile as obtained by AES on the spalled Si/SiC interface during the sputtering process.

laboratory. Partial confirmation for this has come from additional AES measurements from the spalled spot by sputtering the surface for 8.2 min and thereby progressively removing atomic layers from the spalled surface. Figure 18 shows the element composition obtained at various levels beneath the surface during the sputtering process. This clearly shows that the concentration of carbon falls off steeply with the sputter time (and hence beneath the surface) and that the concentration of silicon increases to almost 100%. If there were any substantial concentration of SiC on the surface, the element composition curves for both carbon and Si should have remained constant or, at least, varied much less rapidly with sputter time due to the fixed stoichiometry of the SiC coating.

Table 3 shows the threshold laser fluence and the interface strength values calculated from the simulation program for various Si/SiC pairs. The variation in the interface

TABLE 3. *Calculated interface strengths for Si/SiC system*

Sample	Variation in threshold laser fluence (J/m ²)	Coating thickness (μm)	Average tensile strength (GPa)	Variation in strength (GPa)
A	11,628–12,494	1.68	9.2	8.87–9.53
B	13,357–14,625	2.5	10.53	10.19–10.88
C	7388	1.5	5.29	—
D	5257	1.5	3.70	—
E	6973	2.4	5.86	—
F	40,590	0.3	8.20	—
G†	65,410	2.4	10.60	—
H†	38,705	2.1	6.30	—

† Calculated with carbon as the laser absorbing film.

strength given in the table is determined as explained before. Table 3 indicates that it is possible to successfully delaminate coatings of thickness ranging from 0.3 to 3 μm .

In summary, the actual interface strength values for the various Si/SiC systems produced by using different deposition parameters was found to vary from 3.7 to 10.88 GPa. While such a variation may appear disappointing, this degree of freedom to change interface strength is exactly what is required to carry out the controlled interface delamination in composites as outlined in Section 1.

Finally, we note that by comparing the results of the measurements on the PE quartz with those on Si substrates that comparable laser fluences produce much more intense stress pulses in the latter than in the former. This is largely a result of the much larger pulse conversion efficiency in Si over PE quartz—measured primarily by the much larger Grueneisen constant of Si in comparison to that of PE quartz.

PG/SiC coating interface. The appearance of the spot from where the SiC coatings of 2.1 μm thickness, spalled-off from the PG surface was similar to that for Si crystals as shown in Fig. 16. The PG discs were *ca.* 1.7 mm thick. The edges of the spalled pattern showed strong alignment with the edges of the graphitic planes that terminate perpendicular to the surface. Both the stiffness and the strength of PG across these planes is quite low. This characteristic was found to affect the SiC coating. For this system the average interface strength was found to be 3.68 GPa. These calculations also include the anisotropic character of the PG substrate. The elastic properties for PG that enter the simulation are given in Table 4. Since the surface analytical 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 2.5–10 nm) compared remarkably well with the thickness of the deposited SiC coatings. The measured interface strengths between PG and SiC coatings are given in Table 5. The range in strengths measured in the same way as described before for the Si/SiC case is also indicated in the table. Table 5 also gives information on the reproducibility of results. Thus, very similar lower threshold laser fluences for samples A and B were required for removing identical SiC coatings from PG substrates of different thicknesses (substrate A was 1.7 mm while substrate B was 2.1 mm thick). We note that as in the case of the delamination experiments of ARGON *et al.* (1989) the recorded interface strength values for the C/SiC interface is remarkably

TABLE 4. *Relevant physical constants for Pitch-55 ribbon, glue and pyrolytic graphite entering the computer simulation*

	Pitch-55 ribbon	Glue	Pyrolytic graphite
ρ (g/cm ³)	2.0	1.3	2.2
$C^\dagger$ (J/Kg K)	—	—	1674
$k^\dagger$ (J/ms K)	—	—	2.42
$E_{\text{eff}}^\dagger$ (GPa)	593	4.04	44
$\alpha_{\text{eff}}^{\dagger\dagger}$ (K ⁻¹)	—	—	25.2×10^{-6}

[†] Blank entries in the column indicate that the values are not required in the computer simulation.

[‡] Defined in Appendix 1.

TABLE 5. *Calculated interface strengths for PG/SiC system*

Sample	Variation in threshold laser fluence (J/m ²)	Coating thickness (μm)	Average tensile strength (GPa)	Variation in strength (GPa)
A	14,268–17,294	2.51	6.91	6.25–7.58
B	14,397–19,716	2.51	7.48	6.31–8.65
C†	43,889	2.1	3.68	—

† Calculated with carbon as the laser absorbing film.

close to that obtained for the Si/SiC interface. This result that SiC should bond to Si and C in a closely similar way should not be entirely unexpected.

Pitch-55 ribbon/SiC coating interface. This experiment was carried out by employing the test configuration of Fig. 2b. Due to the presence of inhomogeneities and the low (transverse) strength of these ribbons the fracture paths were observed to be predominantly within the ribbon. However, in some cases, apparent fracture along the interface was also observed. Figure 19 shows a micrograph of an edge of a spalled coating. The coating is intact on the lower part of the micrograph and is removed from the upper part. For this sample (A), an average delamination strength of 0.24 GPa was obtained. These strength determinations on the Pitch-55 ribbons with SiC coatings include the anisotropic character of the ribbon material. The elastic properties for the ribbon and the thin glue layer that enter the simulation are given in Table 4. Some Pitch-55 ribbons were also tested without a coating in order to determine the transverse strength of the ribbon. The result, given as measurement D in Table 6, is 0.26 GPa, and is, within experimental error, the same as two results for A and B. In all the above cases, the location of the fracture path was probed by comparing the coating thickness to the depth of the spalled spot determined from the high precision profilometer reading. The fact that the strength results from these tests are significantly lower than those obtained from the PG/SiC is indicative that the fracture in all probability followed along planes close to the interface but within the ribbon. Thus, the measured delamination strength is apparently governed by the transverse strength of the ribbon. This is compatible with the known morphology of these ribbons (GUPTA

TABLE 6. *Calculated interface strengths for Pitch-55 ribbon SiC system*

Sample	Threshold laser fluence (J/m ²)	Coating thickness (μm)	Average tensile strength (GPa)
A	15,176	2.51	0.24
B	13,014	4.28	0.22
D†	16,441	2.1	0.26
C†	12,582	4.64	0.13

† Fracture observed in the ribbon.

and ARGON, 1991). In view of these results on the ribbon experiments, the PG/SiC system appears to be better suited for the determination of the C/SiC interfaces. Nevertheless, the results on the Pitch-55 ribbon samples indicate that ribbons of this morphology are self-protected for purposes of crack arrest.

6. INTERFACE STRESS CHARTS

6.1. Re-scaling of interface stresses

While the more precise determination of interface strengths from the laser pulse measurements will require specific simulations with the computer code, it is of interest to provide close estimates based on the present simulations by generalizing the results into interface stress charts. While it is not possible to provide charts for all systems over wide ranges of properties it is, nevertheless possible to give useful results for three substrates over relatively wide ranges of H_s and K_s values. A more approximate approach for obtaining the stresses for other substrate-coating interfaces (with arbitrary values of H_s and K_s for the substrates) is presented in Section 6.5 below.

Since the stress pulse generated in the substrate is independent of the test coating on its front surface, the interface stress $\tilde{\sigma}_i$ can be re-scaled by the use of an acoustic transmission coefficient B

$$\tilde{\sigma}_i = \sigma_0/B, \quad (18)$$

where σ_0 is the normalized stress defined above in Section 3.3 and B is the acoustic transmission coefficient at the substrate/test coating interface defined as

$$B = \frac{2\sqrt{(E_c\rho_c)}}{\sqrt{(E_s\rho_s)} + \sqrt{(E_c\rho_c)}}. \quad (19)$$

In Eq. (19) 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 re-scaling, it is possible to determine the stress at the interface between a substrate and a coating of *any* material provided the elastic properties of the two media are known in order to calculate the acoustic transmission coefficient B . Although the interface stress charts presented below are generated for specific material pairs, these can be used for other substrates with the same H_s and K_s values.

6.2. Interface stress chart based on the Si/SiC system

Figure 20a shows the interface stress chart depicting the history of the stress developed at the interface for a given normalization stress of $\sigma_0 = 218$ MPa, generated by a gold film, for various thicknesses of the SiC coating on a Si crystal substrate. For this pair $H_s = 0.148$ and $K_s = 0.271$. The laser fluences used to generate these stress amplitudes are in the range that produce no melting of the gold film. Because the interface stress is normalized with respect to the properties of the gold film, in cases with *no melting* of the energy absorbing film, the interface stress chart of Fig. 20a is valid for any other absorbing film/substrate combination with similar H_s and

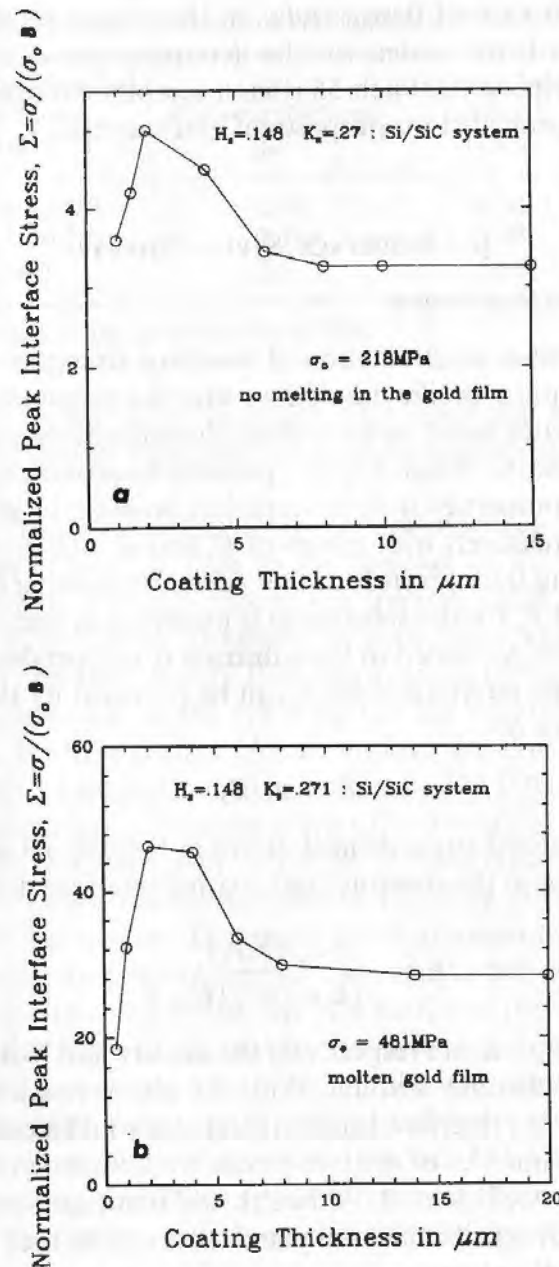


FIG. 20. Interface stress charts for a Si/SiC interface: (a) for fluence levels causing no melting of the gold film, (b) for fluence levels causing melting of the gold film.

K_s values. In such cases, the conditions for simultaneous arrival of the initial tension pulse and the *peak* reflected tension wave (from the initial compressive pulse) at the interface can be achieved for a certain critical thickness of the test coating. For the case shown in Fig. 20a this occurs for a coating thickness of 2 μm. For coating thickness above the critical level, the interface stress drops to a plateau level corresponding to a thickness equal to almost half the critical thickness for peak stress generation. For the present case, this corresponds to about 6 μm where a plateau stress is reached. As expected for coatings below the critical thickness, the stress at the interface decreases monotonically with decreasing coating thickness.

The generalized chart of Fig. 20a is useful only in systems with low interface strengths, wherein, the spallation of the interface can be achieved at laser fluences causing *no melting* of the gold film. However, for many systems (for e.g. the current Si/SiC system) it becomes necessary to increase the laser fluence to levels far above that, that will cause melting of the gold film. Figure 20b shows the interface stress chart that includes the effect of melting in the gold film. In this case however, the interface stress chart is valid *only* with gold as the laser absorbing film. This restriction arises because the specific properties of the molten gold film (such as, volumetric expansion on melting) need to be incorporated in the simulation program. Moreover, the coefficients of the governing differential equation (10) are now a function of time (e.g. the coefficient of expansion and the bulk modulus are taken to be different for the solid and the melt regime). For uniformity, the interface stress in Fig. 20b is normalized with respect to the properties of the *solid gold* film. The normalized peak interface stress is increased almost five fold as compared to the case with no melting. The critical thickness of the coating that maximizes the interface stress remains equal to $2\text{ }\mu\text{m}$, i.e. as for the case of no melting. Similarly, for coatings that are below and above the critical thickness, the interface stress drops from the value corresponding to the critical thickness as before. Since the stress pulse broadens on melting of the gold film, the coating thickness at which the interface stress levels off is also slightly increased to about $8\text{--}9\text{ }\mu\text{m}$. It must be emphasized that the interface stress chart of Fig. 20b can be used only if gold is used as the laser energy absorbing film, where, however, the choice of the substrates is not restricted. This is not considered to be a sacrifice since gold is the most efficient film for stress wave production.

6.3. Interface stress chart for the PG/SiC system

Since PG can be obtained in bulk form, the experimental configuration of Fig. 2a was employed. Figure 21 shows the interface stress chart when there is melting of the

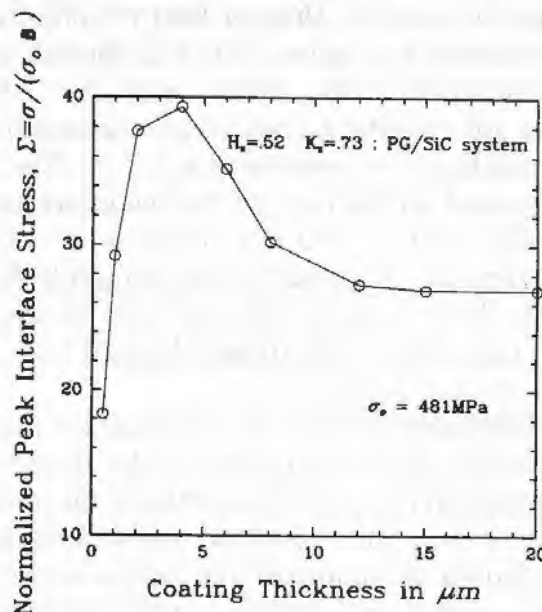


FIG. 21. Interface stress chart for the PG/SiC interface for fluence levels causing melting of the gold film.

gold film. In this case, the peak normalized stress reaches a value as high as 39 (18.75 GPa). This is about 18% higher than the stress generated in the Si/SiC system for the same absorbed laser fluence. Such high interface stress values suggest that the laser spallation technique could be employed for systems with very high interface strength. Parenthetically, it is clear that the laser spallation technique can also be used to determine the intrinsic cohesive strength of brittle homogeneous solids free of significant plastic dissipation. All the basic features of the Si/SiC system are also reproduced in this case of PG. However, the critical thickness for this system where peak stress is achieved is about 4 μm . The anisotropic elastic properties of the PG were accounted for in the simulation. Since the governing differential equation for orthotropic media is of the same form as the one for isotropic media, it is still possible to define the H_s and K_s values. For this case $H_s = 0.52$ and $K_s = 0.73$. The elastic properties used in the computer program were given in Table 4. It must be noted again that the same restrictions apply to Fig. 21 as those for Figs 20a and 20b, i.e. the energy absorbing film must be gold, but that the substrates are otherwise not restricted as long as they have similar H_s and K_s values.

6.4. Interface stress chart for polymer/coating systems

Interfaces between polymeric materials and hard inorganic media have been of considerable interest to the computer device industry. Many initial attempts to measure the toughness and strength of interfaces were stimulated by such interests (DAVUTOGLU and AKSAY, 1981; CHOW *et al.*, 1976; ARAVAS *et al.*, 1989). While in most of these instances the substrate will be a hard solid such as Si or some other semi-conductor crystal, and the coating a thin polymer film, the strength of the interface can be probed by considering the polymer as the substrate and the hard crystal as the coating. Here we consider as a first approximation a (non-dissipative) polymer substrate with a SiC coating. Then the laser spallation technique can be used to measure the interface strength as before. Figure 22 shows the interface stress chart for a typical polymer substrate/coating system with $H_s = 2.51$ and $K_s = 3.59$. No melting behavior of the gold coating is considered in generating this interface stress chart. The critical coating thickness considered is 1.5 μm . The same restrictions and degrees of freedom discussed for the two previous cases are applicable here too, i.e. the energy absorbing film must be gold but otherwise the choice for the substrate is free—provided its properties are described by constants H_s and K_s in the same range. It must be noted here, however, that inelastic dissipation in all polymeric media is expected to be high. Hence, the stresses obtained from the chart will be over-estimates.

The critical coating thickness of most of the interface pairs discussed above is around 2 μm . This indicates that the structure of the stress wave generated in the substrate is strongly dependent on the characteristics of the laser pulse and the energy absorbing film. The utility of the above interface stress charts is that they will permit the use of the laser spallation technique without an associated computer simulation. A more approximate scheme for obtaining the stress at the interface between coatings and substrates of arbitrary H_s and K_s values is discussed below.

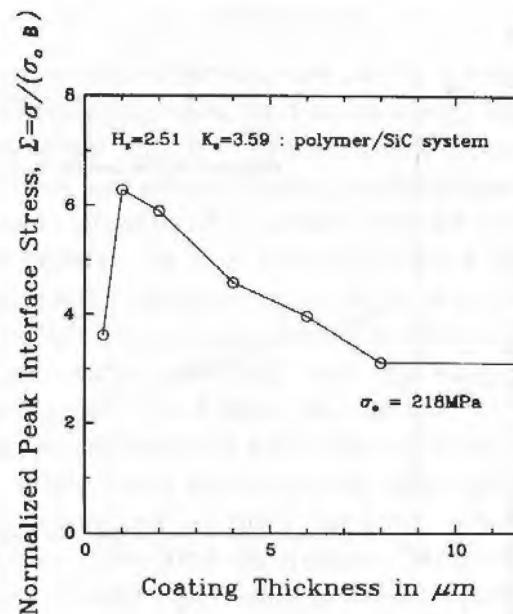


FIG. 22. Interface stress chart for a polymer/SiC interface for fluence causing no melting of the gold film.

6.5. Approximate method for determining the interface stress for substrate/coating systems with arbitrary H_s and K_s values

When the interface stress is re-scaled to incorporate the properties of the test coating, the interface stress charts become valid for *any* test coating that is attached to a substrate having the same H_s and K_s values as the ones used for constructing the respective charts. This, however, still restricts the use of interface stress charts into narrow ranges. In this section, a scheme is presented that permits the determination of the approximate stress at the interface between a coating and substrate of arbitrary H_s and K_s values. Table 7 shows the interface stress as a function of several H_s and K_s values for different thicknesses of the coating as obtained from the interface stress charts. We note that the interface stress decreases with an increase in the H_s and K_s values. This is to be expected because, as indicated by Eq. (11a), an increase in the H_s value is associated with the lowering of the substrate stiffness. Therefore, substrates with increasing H_s values should reduce the constraint on the expanding gold film.

TABLE 7. Normalized interface stress as a function of H_s and K_s values

H_s	K_s	Normalized interface stress coating thickness		
		1 μm	2 μm	4 μm
0.148	0.27	32.30	46.26	45.43
0.52	0.73	26.26	33.89	35.35
2.51	3.59	21.00	19.76	14.82

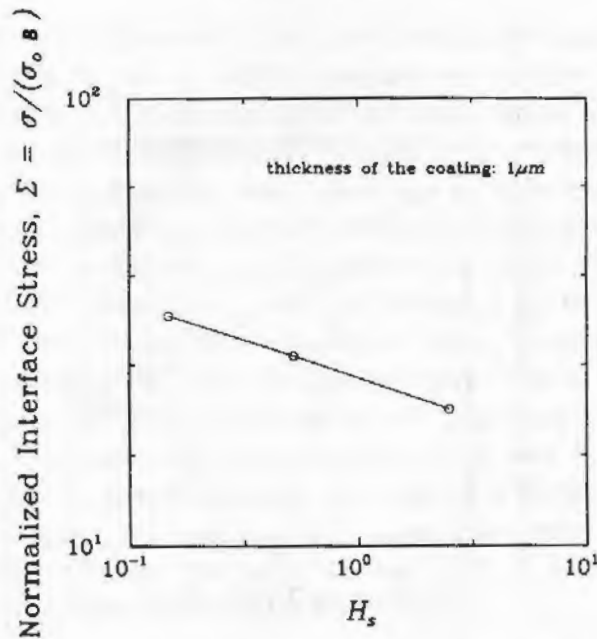


FIG. 23. Dependence of normalized interface stress on the substrate function H_s .

This leads to a better local accommodation of the imposed misfit and thus, to a reduction of the generated stress amplitude in the substrate and hence, also, at the substrate-coating interface. This observation also suggests that the relationship between H_s and the interface stress cannot be linear, because, the stress must asymptotically tend to zero as the substrate takes on the properties of a vacuum. The interface stress will reach a maximum value in the limit when the substrate becomes rigid or the H_s value approaches zero. Based on such phenomenological observations, that suggest a power-law dependence between the interface stress and H_s , the validity of this was explored by a plot on logarithmic coordinates. This is shown in Fig. 23 for a coating of 1 μm thickness, and can be stated analytically as

$$\Sigma = AH_s^n, \quad (20)$$

where an empirical fit provides $A = 23.6$ and $n = -0.17$, respectively.

Since each substrate is also characterized by a K_s value, every value of H_s in Fig. 23 is associated with a corresponding value of K_s . Although the interface stress may depend also on K_s values, for a given value of H_s , the variation in the K_s values appears to be quite restricted. It can be noted from Table 7 that an increase in the K_s value is associated with a corresponding increase in the H_s value. Although these two parameters are not entirely dependent, a physical relationship between them must exist, restricting the possible range of values of K_s once H_s is fixed. Thus, within such limits, the interface stress for substrates with arbitrary H_s (and associated K_s) values can be determined from the plots of the type shown in Fig. 23. While such approximate answers can be useful in rank ordering different systems, more precise determinations of the interface strength will always require the use of the computer code.

7. DISCUSSION

The tensile strength of 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 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 measurement 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 developed 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 (ARGON *et al.*, 1975). 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 these latter cases for which 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 Λ_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_i ,

$$\Delta t_i \cong \Lambda_c^2 \rho C / k, \quad (21)$$

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 $1 \mu\text{m}$ thickness is about 8 ns. This is readily achievable with many pulsed 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 piezoelectric probe for various material pairs have been provided in the form of interface stress charts presented in Section 6. These results should make such strength measurements possible to a reasonable level of precision in most systems of interest to workers on composite materials by using the interpolation scheme furnished in Section 6.5.

Tensile strength of interfaces between amorphous SiC coatings and substrates of pyrolytic graphite, Pitch-55 carbon ribbon and silicon single crystal disks were explored in the present investigation. The average interface strength for the Si/SiC pair determined from the experiment ranges from 3.7 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 ribbon/SiC system were obtained. In the latter case this result was most likely governed by fractures in the Pitch-55 ribbon. 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-up of sufficient tensile stress at the interface to cause interface fracture. It was found that SiC coatings of thicknesses 0.3–3 μm could be readily removed from substrates. In order to spall even thinner coatings from substrates, it will become necessary to use picosecond laser pulses in place of nanosecond ones as employed in the present investigation. Results of the numerical study in Section 3 indicate other interesting possibilities to delaminate very thin coatings ($<1 \mu\text{m}$ thick) from substrates, that should have interesting applications to computer-chip systems.

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 whenever the propagation of an existing crack along the interface is the determining factor, the most relevant quantity that is required is the fracture toughness K_{Ic} or the critical energy release rate G_{Ic} for the propagation of a crack along the interface. A measure of this can apparently also be obtained with the laser spallation method, if well characterizable and reproducible flaws can be deliberately 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_{Ic} or produce the K_{Ic} . In a preliminary experiment where some local interface contamination of an undetermined nature had 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. 24a. Figure 24b shows an enlarged view of one of these craters. Clearly, under the condition of the reflected tensile stress pulse, which probes the interface, a central crack was formed in the area of the interface flaw where a much reduced level of interface strength must have been present. After the crack has propagated radially outward a certain distance, the effective ratio of the crack tip stress intensity factors K_{I} and K_{II} has apparently undergone almost two full reversals of sign before the delamination was 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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APPENDIX 1. GOVERNING WAVE EQUATION FOR A GENERAL ORTHOTROPIC MATERIAL

If α_1 , α_2 , and α_3 are the coefficients of thermal expansion in the 1, 2, and 3 directions respectively, then using Hookes' law, total strain components can be expressed as

$$\begin{aligned}\epsilon_1^T &= a_{11}\sigma_1 + a_{12}\sigma_2 + a_{13}\sigma_3 + \alpha_1 T, \\ \epsilon_2^T &= a_{12}\sigma_1 + a_{22}\sigma_2 + a_{23}\sigma_3 + \alpha_2 T, \\ \epsilon_3^T &= a_{13}\sigma_1 + a_{23}\sigma_2 + a_{33}\sigma_3 + \alpha_3 T,\end{aligned}\tag{A1.1}$$

where a_{ij} are the elastic compliances. Using $\epsilon_2^T = \epsilon_3^T = 0$. (In conjunction with the physical state of the uniaxial straining of the material as described in Section 3) to eliminate σ_2 and σ_3 from Eq. (A1.1) we get

$$\epsilon_1^T = \kappa T + \sigma_1 \eta,\tag{A1.2}$$

where

$$\kappa = \left[\alpha_1 - \left(\frac{a_{13}}{a_{33}} \right) \alpha_3 + \beta' \left(\left(\frac{a_{23}}{a_{33}} \right) \alpha_3 - \alpha_2 \right) \right],$$

$$\eta = \left[a_{11} - \left(\frac{a_{13}}{a_{33}} \right) a_{31} - \beta' \left(\left(\frac{a_{23}}{a_{33}} \right) a_{31} + a_{21} \right) \right]$$

and

$$\beta' = \frac{[a_{12} - (a_{13}/a_{33})a_{32}]}{[a_{22} - (a_{23}/a_{33})a_{32}]}.$$

Using Eqs (A1.1) and (A1.2), the dynamic equilibrium condition reduces to the desired elastic wave equation

$$\frac{1}{\eta} \left[\frac{\partial^2 u}{\partial x^2} - \kappa \frac{\partial T}{\partial x} \right] = \rho \frac{\partial^2 u}{\partial t^2}. \quad (\text{A1.3})$$

For the isotropic case, the elastic wave equation (A1.3) for the anisotropic case reduces to

$$(\lambda + 2\mu)_i \frac{\partial^2 u_i}{\partial x^2} = \rho_i \frac{\partial^2 u_i}{\partial t^2} + (3\lambda + 2\mu)_i \alpha_i \frac{\partial T_i}{\partial x}. \quad (\text{A1.4})$$

Both PG and carbon ribbon show isotropy in the 1-2 plane. The anisotropic constants for these cases reduce to

$$\eta = \frac{1}{E_1} [1 - \nu_{31}^2 + \beta'(\nu_{12} + \nu_{31}^2)], \quad (\text{A1.5})$$

$$\kappa = \alpha_1 + \alpha_3 [\beta'(1 + \nu_{31})], \quad (\text{A1.6})$$

where

$$\beta' = \frac{(\nu_{12} + \nu_{31}^2)}{(1 - \nu_{31}^2)} \quad (\text{A1.7})$$

and E_1 and ν_{ij} are respectively the modulus and Poisson's ratio of the media. The further useful abbreviations of E_{eff} and α_{eff} are defined as follows:

$$E_{\text{eff}} = 1/\eta, \quad (\text{A1.8})$$

$$\alpha_{\text{eff}} = \kappa/\eta. \quad (\text{A1.9})$$

The values of the latter used in the computer simulation for PG and the carbon ribbon are given in Table 4.

APPENDIX 2. MODELLING OF THERMAL EXPANSION DUE TO MELTING IN THE PRESSURE PULSE SIMULATION

For incorporation into the ABAQUS code the volumetric thermal expansion strain due to a phase change such as melting can be written as

$$\varepsilon_{\text{th}}(T) = \begin{cases} \alpha_s(T - T^I) & (T_m > T > T^I) \\ \alpha_s(T_m - T^I) + \Delta\varepsilon_f + \alpha_L(T - T_m) & (T \geq T_m^+), \end{cases} \quad (\text{A2.1})$$

where α_s , α_L are respectively the thermal expansion coefficients in the solid and the molten states, T_m is the melting temperature, T^I is the initial temperature and $\Delta\varepsilon_f$ is the volumetric

thermal strain of fusion, and the subscripts + and - are used to specify small deviation from the base quantities. In the ABAQUS, code thermal strain is defined as

$$\varepsilon_{th}(T) = \alpha^{AB}(T - T^0) - \alpha(T^I)(T^I - T^0), \quad (\text{A2.2})$$

where T^I and T^0 are the initial and the reference temperature respectively and α^{AB} is the expansion coefficient used as an input to the program. Using $T^I = T^0$, the ABAQUS thermal strain reduces to

$$\varepsilon_{th}(T) = \alpha^{AB}(T - T^I). \quad (\text{A2.3})$$

The physical thermal strain can now be modelled using the ABAQUS code by defining an equivalent $\alpha^{AB}(T)$ by equating equations (A2.1) and (A2.3) as

$$\alpha^{AB} = \begin{cases} \alpha_s & (T_m > T > T^I) \\ \frac{[\alpha_s(T_m - T^I) + \Delta\varepsilon_f] + \alpha_L(T - T_m)}{[T - T^I]} & (T \geq T_m). \end{cases} \quad (\text{A2.4})$$

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