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Measurement of interface strength by the modified laser spallation technique. I. Experiment and simulation of the spallation process

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Part I of this paper presents the modified laser spallation technique for measuring the tensile strength of planar interfaces, using a Doppler displacement interferometer. In this technique, a laser-produced compressive stress pulse in the substrate, reflecting from the coating's free surface pulls the interface in tension and leads to its failure if the tensile amplitude is high enough. The current technique is an improvement over the previous one, since the interface stress is determined directly by recording the coating or substrate free-surface velocities using a laser displacement interferometer. The recorded surface velocity is related to the interface stress via an elastic wave equation simulation. The process of coating spallation is investigated, and the effect of the stress pulse profile and the coating and substrate characteristics on the interface tensile stress is studied using the simulation. Several interface stress charts are given for wider applicability of the modified technique.

I. INTRODUCTION

Interfaces between thin films and substrates exist in many fields and applications, including composite materials, tribology, and solid-state devices. In composites, the interface between a thin coating and a fiber is considered¹⁻³ for deflecting impinging matrix cracks. In the field of tribology, interfaces between various types of functional (magnetic, conducting, optical, and electrical), protective (thermal barrier, corrosion, and wear resistant), or decorative coatings and their underlying substrates are of interest. Finally, metal/ceramic interfaces are present in multi-layer devices and magnetic disk and head technology. In all of these applications the tensile strength of the interface is an important property that directly controls the interface decohesion process, and often controls the reliability of the coated components. Both control and measurement of the interface tensile strength are of importance in the above applications.

Measurement of interface properties is not a new problem in thin-film and coating technology, and the literature is replete with practical techniques⁴⁻⁹ for the measurement of some average properties of thin films or coating interfaces, recently reviewed by Mittal.¹⁰ However, none of the tests referenced above measure the strength of interfaces. In most of the reported cases,⁴⁻⁹ 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 to local deformation problems. The measurement of strength between a substrate and a thin coating presents particular difficulties in the application of stress by an inhomogeneous local deformation field, where premature failure elsewhere is likely before the desired interface can be probed.

A laser spallation experiment was developed recently

by one of us^{2,3} to determine the tensile strength of planar interfaces by overcoming the above inadequacies present in various test methods. In this technique a high-energy laser pulse of 3 ns duration from an Nd:YAG laser is converted into a pressure pulse of critical amplitude and width, and sent through the substrate toward the free surface of the coating. The reflected tensile pulse from the coating's free surface pries off the coating. In the prior work,^{2,3} the critical stress amplitude which results in the removal of the coating was determined through a computer simulation of the process, which in turn was verified by means of piezoelectric crystal probe that was capable of mapping out the profile of the stress wave generated by the laser pulse. Here we present a modified laser spallation technique to determine the tensile strength of planar interfaces, which improves upon the earlier version. In the modified version, the transient displacement and velocities of the coating free-surface are recorded by a laser Doppler technique, and related to the interface stress using wave mechanics, albeit by assuming the density and the elastic modulus of the coating. For the work presented here, the coating surface is always free. Therefore, henceforth, the use of the words "coating surface" implies that the surface is free.

Our results are presented in three companion articles. In this article (Part I), we furnish the details of the modified setup and model the process of spallation by considering the elastic wave mechanics associated with the development of the interface tensile stress. Specifically, for a given coating and set of substrate characteristics, the effect of the shape and amplitude of the stress pulse on the resulting interface stress is determined. In Part II we present the observed tensile strengths and failure mechanisms of interfaces between a pyrolytic graphite substrate and 3- μm -thick metallic coatings of Al, Nb, Cu, Cr, Sn, and Sb. In addition, the tensile strength of interfaces between Al_2O_3 substrates and coatings of diamond, Nb, and SnO_2 are also given. Finally, we discuss how the interface strength of an alumina/Nb interface can be tailored by depositing Cr and Sb interlayers of prescribed thicknesses.

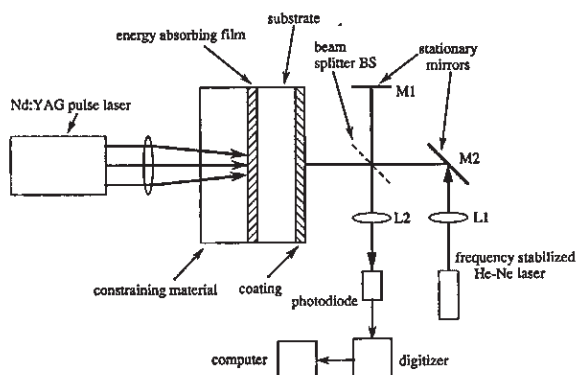


FIG. 1. Schematic of the laser Doppler displacement interferometer along with the laser spallation setup.

In Part III, the optimal profile and amplitude of the stress pulse generated in the modified laser setup is obtained experimentally. Stress pulses produced in an Si single-crystal substrate are investigated with different types and thicknesses of the laser-absorbing films, and constraining media. Furthermore, the effect of the substrate medium on the stress pulse is also determined. Finally, it is shown that for increasing laser fluences, the nature and shape of the pulse profile is retained, indicating similar laser pulse and material interactions within the fluence levels used in the laser spallation experiment.

II. PREVIOUS VERSION OF THE LASER SPALLATION EXPERIMENT

The experimental approach utilizes a planar arrangement of a substrate and coating combination, as shown in Fig. 1. Details of the sample holder are given in Refs. 2 and 3. The collimated laser pulse is made to impinge on a 1- μm -thick gold film that is sandwiched between the back surface of a substrate of interest and a fused quartz confining plate, transparent to the wavelength of the laser (Nd:YAG laser operated at 1.06 μm). Gold was chosen as the laser absorbing film. Absorption of the laser energy in the confined gold leads to a sudden expansion of the film which, due to the axial constraints of the assembly, leads to the generation of a compressive shock wave directed toward the test coating/substrate interface. A part of the compressive pulse is transmitted into the coating as the compression pulse strikes the interface. It is the reflection from the free surface of the coating of this compressive pulse into a tension pulse that leads to the removal of the coating, given a sufficiently high amplitude. A key element of the experiment was to determine the amplitude of the interface tensile stress at the threshold laser fluence causing the interface failure.

Previously, the approach consisted of a three-part strategy. The first part was the development of a finite element computer simulation of the conversion of the laser light pulse into a pressure pulse, and of the resulting history of tensile stress which develops at the interface as the wave is reflected from the free surface of the coating. In the

second part of the strategy, the pressure pulses were measured in a microelectronic, piezoelectric device in which the conditions of the computer simulation were experimentally achieved. This permitted verifying and fine tuning the computer simulation. Finally, in the third part of the strategy, actual spallation experiments were carried out for several thin coating/substrate interfaces (e.g., C/Si and SiC/Si interfaces). 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 accomplished this was determined from the computer program.

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. Although the detachment of ductile coatings from substrates which themselves can undergo plastic deformation under the transient stress is readily achieved, conversion of the threshold laser energy into interface strength requires careful consideration. In the old technique, this will require considering the change in the stress pulse characteristics due to the plastic deformations in the substrate and the coating in the computer simulation. Such effects, although ignored initially for interfaces involving brittle components, can be included but will involve largely unknown elastic and plastic properties of the coating and the substrate. In the modified laser spallation experiment discussed below, since the stress pulse is measured at the coating free surface, the effect of plastically deforming substrate and/or coating is obtained experimentally. Our results on ductile Nb coatings on Al_2O_3 substrates show one such application, and is discussed in Part II.

III. MODIFIED LASER SPALLATION EXPERIMENT

A. Strategy

The modified version employs the same assembly as shown in Fig. 1, but the experimental setup was modified so as to obtain a stress pulse with highest amplitude and smallest rise and fall times, for a given laser fluence. The details are presented in Part III. A 0.5- μm -thick Al laser absorbing film confined by a 5- μm -thick layer of solid water glass was used in the modified setup to obtain the generated stress pulses of high amplitude and short decay time. As before, the stress pulse is generated by a Q-switched Nd:YAG laser pulse of 3 ns duration. The substrate disks were either 12.7 or 25.4 mm in diameter and 1 mm thick. Typical test coating thickness ranged from 1 to 3 μm . The laser beam was focused onto a 3-mm-diam spot. The incident laser fluence was controlled by changing the energy of the laser beam while keeping the illuminated area constant.

When the stress pulse is reflected from the free surface of the coating or the substrate, the particle at the free surface experiences a transient velocity, which in turn is proportional to the transient profile of the striking stress pulse. In the modified version, the transient velocity is measured directly by a laser Doppler velocity interferom-

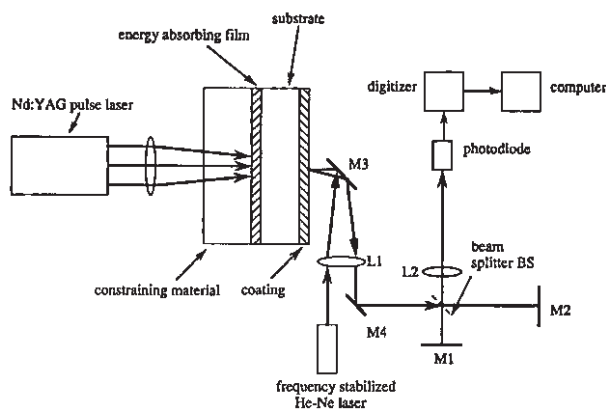


FIG. 2. Schematic of the laser Doppler velocity interferometer along with the laser spallation setup.

eter (see Fig. 2) and also more accurately, by recording the displacement history of the coating's or substrate's free surface by a Doppler displacement interferometer. The peak interface tensile stress generated at each level of the laser fluence is related to the maximum compressive stress at the free surface through a computer simulation. In this program, a stress pulse measured at the substrate's free surface (measured in separate experiments) was made to impinge at the interface from the substrate side, and the resulting peak tensile amplitudes of the normalized stresses at the interface and the coating's free surface are determined. We simply take the ratios of these calculated amplitudes and use them as transfer coefficients to convert the measured peak stress at the coating or the substrate free surface to the interface peak stress. Since the generated stress profile is similar for different laser fluence levels (see Part III), the simple approach of using transfer coefficients is very accurate provided the coating remains elastic. The above procedure results in a plot of laser fluence versus the interface stress. This information is then used to convert the threshold laser fluence causing coating spallation to the interface strength.

B. Recording of the transient velocity profile and displacement history at the free surface

It is possible to measure both the free surface displacement and the velocity by using the standard laser Doppler displacement and velocity interferometers¹¹⁻¹³ which are used widely in the plate impact research. Since in our experiment, the stress pulses were sharp (rise time < 5 ns) and produced over a small area of 7 mm², recording of subnanosecond displacement and velocity fringes posed a significant challenge.

We used the displacement interferometer (Fig. 1) to obtain the stress pulse profiles. Since the useful duration of the stress pulses generated in the spallation experiment is about 20 ns, only about 10 displacement fringes are produced within this duration albeit with a minimum 0.5 ns rise time. The use of a Tektronix SCD 1000 digitizer with a transient rise time of 5 ps, and Newport's 877 ultrahigh speed photodetector with a rise time of 200 ps allows us to

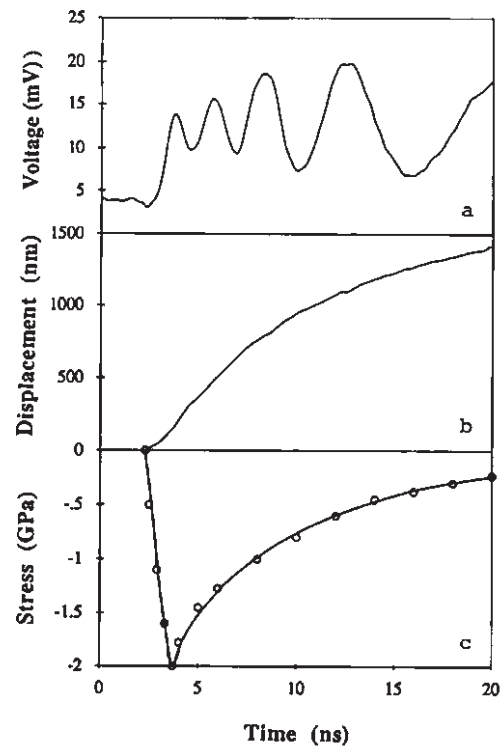


FIG. 3. (a) Photodiode voltage output corresponding to the single-crystal Si free surface obtained by the Doppler displacement interferometer. (b) Displacement as a function of time corresponding to the voltage record in (a). (c) Stress profile striking the coating free surface obtained from (b).

record such fringes with a resolution of 0.2 ns. Furthermore, our setup is capable of recording velocities to about 800 m/s. This resolution suffices for typical velocities obtained in our experiments. The process used for obtaining the stress pulse is presented in Fig. 3. Figure 3(a) shows the photodiode voltage output corresponding to the Si single-crystal free surface obtained by the Doppler displacement interferometer shown in Fig. 1. The photodiode output voltage amplitude $A(t)$ is recorded on a transient digitizer and related to the fringe count $f(t)$ as

$$A(t) = \frac{(A_{\max} + A_{\min})}{2} + \frac{(A_{\max} - A_{\min})}{2} \sin[2\pi f(t) + \delta], \quad (1)$$

where $A_{\max}$ and $A_{\min}$ are the maximum and minimum fringe amplitudes, respectively, and δ is a phase angle. Once $f(t)$ is determined from the above, the displacement $u(t)$ can be obtained as¹¹

$$u(t) = \lambda_0 f(t) / 2, \quad (2)$$

where λ_0 is the wavelength (632.8 nm) of the He-Ne laser. Figure 3(b) corresponds to the substrate free-surface displacement obtained from the fringe record of Fig. 3(a), and finally, the stress pulse profile generated by the pulsed laser obtained from Fig. 3(b) is shown in Fig. 3(c).

Since in our experiments we know the expected profile of the transient surface velocity (i.e., it increases monotonically to a peak value and then decays continuously to

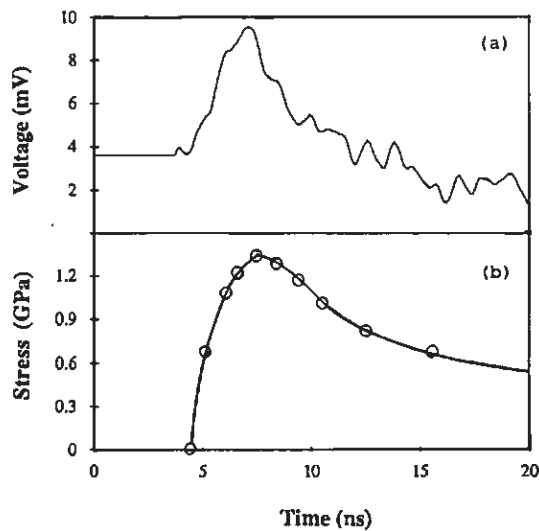


FIG. 4. (a) Photodiode voltage output in a velocity interferometer for Si substrate. (b) Velocity profile obtained from the displacement fringe record.

zero), it is not necessary to use the quadrature detector used by Amery¹⁴ to determine the point at which the velocity peaks. Since our setup can record fringes with high resolution, the point at which the velocity peaks, which corresponds to a change in the displacement fringe frequency, can be detected from the detailed digitized data. Even the most difficult case where the reversal (from deceleration to acceleration) information is contained within half a fringe can be resolved. To be certain of our interpretation of the displacement fringe record, we conducted a separate experiment in which the stress pulses were produced in a Si single-crystal substrate. We first obtained the displacement history of the Si surface, then differentiated it to obtain the velocity profile, as shown in Fig. 4(b). Next, we used a quasilinear velocity interferometer to determine the velocity of the free surface at the same laser fluence at which the displacement fringes were recorded. Since the velocity is approximately proportional to the photodiode output, the record shown in Fig. 4(a) corresponds to the velocity profile. Since both curves in Fig. 4 peak at the same time, our interpretation of the displacement fringe record is correct. A frequency stabilized He-Ne laser was used to obtain a clean fringe record in the Doppler experimental setup since the beat frequency of the ordinary He-Ne laser (for example, 320 MHz) is within the range of the displacement fringe frequency.

Since the stress pulse is produced over a 7 mm² area, it is important to align the He-Ne laser beam with the center of the laser ablated spot. In the Doppler setup, the He-Ne laser beam with an initial 0.6 mm diam was aligned and focused at the center of the sample's rear surface to a spot of about 632.8 nm diam (equal to the laser wavelength) by using a convex lens (L1) (see Fig. 1). This setup measures the peak stress amplitude even if the Nd:YAG laser pulse shows a spatial distribution of the energy. Next, we discuss how to obtain the interface strength through the recorded displacement and velocity profiles.

C. Determination of the interface strength via the wave mechanics simulation

The ratio of the diameter of the illuminated spot (stress pulse) to the thickness of the substrate was equal to 3. In this geometry the wave propagation within the substrate can be regarded as one dimensional, without any dispersive effects. For a material with the two Lamé elastic constants, λ and μ , and the constant density ρ , the wave equation in each medium can be represented as:

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

where c is the longitudinal wave velocity, u is the displacement in the x direction, t is the time, and i denotes the substrate or the coating. The axial stress in the x direction is calculated from the displacement gradients, and is expressed as

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

Three boundary conditions are required. The first two ensure the continuity of the displacements and stresses at the interface. The third one requires the stress at the coating free surface to be zero.

In order to simplify the numerical analysis and to explain the spallation process clearly, it is useful to present the equations for both the substrates and the film in a normalized form. To accomplish this, the following dimensionless variables for the coordinate (χ), displacement (Φ), time (τ), and stress (Σ), are introduced:

$$\chi = \frac{x}{x_0}, \quad \Phi = \frac{u}{x_0}, \quad \tau = \frac{t}{t_0}, \quad \Sigma = \frac{\sigma}{(\lambda + 2\mu)_s}, \quad (5)$$

where the subscript s refers to the substrate and x_0 , t_0 are, respectively, the fundamental units of displacement and time defined as $x_0 = l_p/50$ and $t_0 = \Lambda_c/c_s$. Where l_p , Λ_c , and c_s are the length of the stress pulse in the substrate, the thickness of the coating and the longitudinal wave velocity in the substrate, respectively.

In the normalized coordinates, the corresponding wave equation becomes

$$H_i \frac{\partial^2 \Phi_i}{\partial \chi^2} = \frac{\partial^2 \Phi_i}{\partial \tau^2}, \quad (6)$$

where the subscript i refers to both the substrate and the coating. In the above, the material constant H_i is defined as

$$H_i = (c_i)^2 / (c_s)^2 \quad (7)$$

and, correspondingly, the equation for the normalized stress becomes

$$\Sigma_i = \beta_i H_i \frac{\partial \Phi_i}{\partial \chi}, \quad \beta_i = \frac{\rho_i}{\rho_s}. \quad (8)$$

Thus, $H_i = \beta_i = 1.0$ in the substrate.

Equations (6)–(8) with the appropriate boundary conditions were solved by using a finite difference approach. The finite difference method was chosen because of its good stability and short processing time requirement.

To model the geometry, 15 000 nodes were used in the substrate. Various thicknesses of the coating were obtained by choosing the number of nodes (10–2500). The stress profile obtained from the free surface of a substrate or a coating (measured separately) is used as an input to the above equations. The experimentally measured stress profile is normalized to a unity peak compressive stress. The normalized displacement profile is obtained by integrating Eq. (8) with $H_i = \beta_i = 1$ (i.e., $i = s$), and by applying the following equation:

$$\Phi_i(x, t) = \Phi_i(t) c_s. \quad (9)$$

This displacement profile is represented by 15 000 nodes in the substrate. When expressed in normalized coordinates, the normalized displacement profile serves as an input to the set of Eqs. (6) and (8) with the appropriate values of H_i and β_i for the nodes representing the coating. Furthermore, the coating thickness was represented in normalized units of $l_p/50$, with l_p as the length of the stress pulse in the substrate.

Two transfer coefficients were defined to convert the stress at the free surface into the interface stress. Transfer coefficient I is the ratio of the peak tensile stress at the interface to the maximum compressive stress in the substrate, while transfer coefficient II is the ratio of the peak tensile stress at the interface to the maximum compressive stress at the free coating surface. Both transfer coefficients can be determined by obtaining appropriate values of the normalized stresses through the above simulation. When the stress profile is obtained experimentally from the free surface of the substrate in a separate experiment, the interface stress can be determined by multiplying the maximum compressive stress by the transfer coefficient I; otherwise, if the stress profile is obtained experimentally at the free coating surface, the interface stress can be determined by multiplying the maximum compressive stress by the transfer coefficient II. For wider applicability of the modified technique, the transfer coefficient charts for various values of β_i and H_i are given in Sec. IV B. A series of experiments with different laser fluences were performed in this way, resulting in a plot of the laser fluence versus the interface stress. Finally, the interface strength is determined by finding the interface strength at the threshold laser fluence causing coating spallation. Applications of the modified laser spallation experiment are given in Part II. Next, we numerically study the mechanics of interface stress buildup.

IV. NUMERICAL STUDY OF THE SPALLATION PROCESS

A. Mechanics of interface stress buildup

The discussion so far does not give a detailed understanding of the coating spallation process. A full understanding can be reached only through a computer simulation of the stress wave profiles as a function of time in the vicinity of the interface. In addition, in the laser spallation experiment, it is desirable to maximize the interface stress for a given laser fluence and an interface system in order to

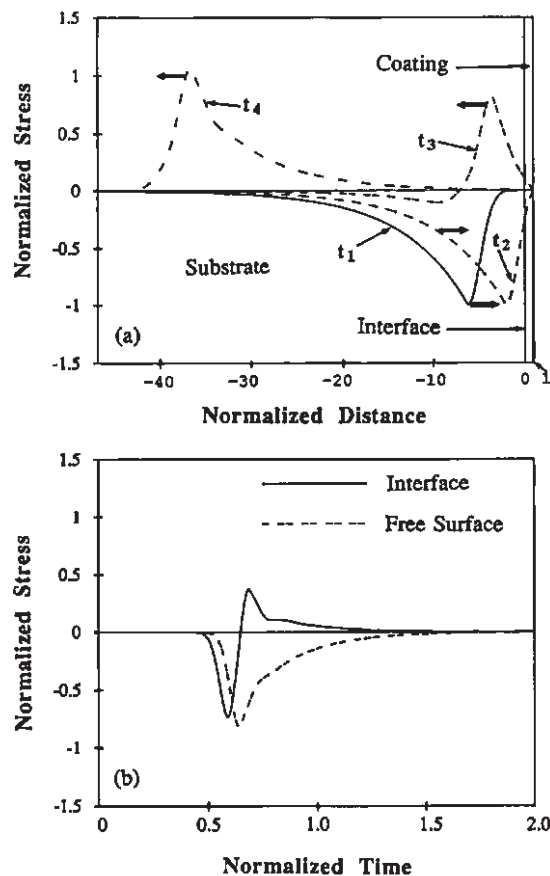


FIG. 5. Process of the spallation for $H_i=0.5$ and $\beta_i=1.0$. (a) Several snapshots of the normalized stress pulses as they propagate through the coating and the substrate. (b) The normalized stress histories at the interface and at the free coating surface as functions of time.

spall coatings from interfaces with high tensile strengths. To achieve this goal, the sensitivity of the peak interface stress to the properties and thickness of the test coating, properties of the substrate, and amplitude and sharpness of the laser-produced stress pulse was explored.

Figure 5(a) shows several snapshots of the normalized stress pulses as they propagate through the coating and the substrate, while Fig. 5(b) shows the normalized stress histories at the interface, and that striking the free coating surface, both as functions of time. Figure 5 corresponds to a case of a softer coating (relative to the substrate) with normalized parameters, $H_i=0.5$ and $\beta_i=1.0$. In this simulation, the shape of the normalized stress pulses used are similar to those obtained experimentally [see Fig. 3(c)], in contrast to the work of Nutt.¹⁵ The normalized incident stress is assigned a unit maximum compressive stress for convenience. A positive value of the stress corresponds to a tensile stress. Here, the term “stress striking the free coating surface” is used to indicate that the stress is calculated through the velocity of the free surface even though the stress at the free surface is always zero.

In Fig. 5(a), the stress profile at t_1 is an input to the computer program, while the stress profiles at t_4 result after the reflection from the interface. The arrows indicate the propagation direction of the stress waves. The first re-

flexion occurs at the interface with a tensile amplitude for the case $H_i=0.5$. Consequently, the transmitted compression stress in the coating is lower. The corresponding simulation for stiffer coating ($H_i=2.0$; $\beta_i=1.0$) yielded a transmitted compressive pulse in the coating which has a higher amplitude than the incident pulse. At time t_2 , the wave front reaches the free coating surface. When the maximum compressive stress is reflected from the free surface, the tensile stress appears. It can be seen that even though the stress near the free surface already becomes positive at t_3 the tail part of the stress profile still remains compressive, suggesting that a sharper post-peak decay will enhance the interface tensile stress.

The most important feature to note in Fig. 5 is that the stresses striking the free coating surface have similar shapes to the incident pulse profile, although with different amplitudes. This was also seen experimentally. The details are given in Part II. This supports the procedure for obtaining the interface strength discussed in Sec. III C, where the ratio of the highest compressive stress striking the coating surface to the highest interface tensile stress was used as the transfer coefficient II to convert the experimentally measured free surface stress to the interface stress. As expected the transfer coefficients are strongly influenced by the properties of the materials and are discussed next.

B. Influence of various variables on the interface stress: Interface transfer coefficients

1. Effect of the material properties

Figures 6(a), 7(a), and 8(a) give the values of transfer coefficient I with different coating thicknesses. The higher transfer coefficient I means that the higher interface stress can be achieved for a given incident stress profile. Furthermore Figs. 6(b), 7(b), and 8(b) show the values of transfer coefficient II as a function of the normalized coating thickness. Each interface system is characterized by the material parameters H_i and β_i . In Figs. 6–8, $H_i=1$ corresponds to the stress within the substrate at different depths from the free surface. It should be noted that the normalized coating thickness is of identical scale to that of Fig. 5(a), which means that 1.0 unit of the normalized coating thickness represents the coating thickness in Fig. 5(a).

The interface transfer coefficient charts should be used in the following way. For a measured substrate pulse profile, the length of the pulse should be scaled to fit the profile of the pulse shown in Fig. 5(a). If this can be achieved then the length-scaling parameter when divided by 50 serves as the fundamental unit of length, to be used to represent the coating thickness in normalized units. For the normalized coating thickness, the transfer coefficients can be read from Figs. 6–8. However, if no scaling parameter can be found to fit the measured stress profile of Fig. 5(a), the transfer coefficients given here cannot be used. Instead, the set of equations should be solved for the new pulse profile.

It is impossible to provide the transfer function coefficients for an exhaustive set of the interface systems characterized by the parameters H_i and β_i . Here we provide

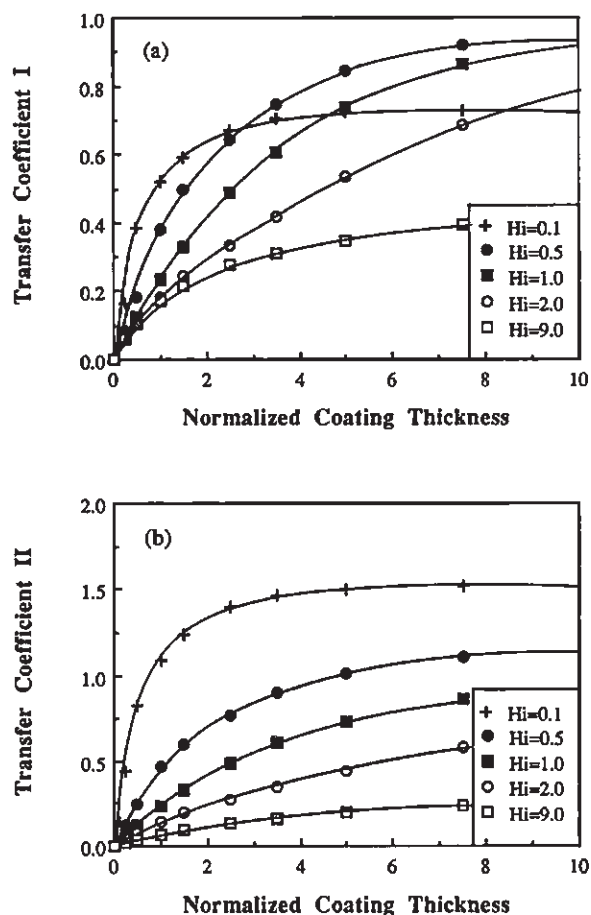


FIG. 6. Influence of the coating and substrate characteristics on the interface stress for $\beta_i=1.0$. (a) Transfer coefficient I. (b) Transfer coefficient II.

charts for three values of β_i equal to 0.2, 1.0, and 5.0, with H_i varying from 0.1 to 9. This combination of H_i and β_i covers a wide range of stiff and soft coatings relative to the substrate material. First, consider the case with $\beta_i=1$, as shown in Fig. 6. Generally, the transfer coefficients increase with the coating thickness since the increased coating thickness minimizes the destructive interference between the initial compressive pulse and the reflected tensile pulse at the interface.

For transfer coefficient II, all values of H_i reveal an increase in transfer coefficient II as H_i declines. The declining H_i values correspond to an increasingly softer coating, which results in a transmitted amplitude lower than the incident pulse impinging at the interface from the substrate side, and consequently to a higher transfer coefficient II. Within the thickness of interest (actual coating thicknesses in our experiments are less than 2.5 of the normalized coating thickness), the difference in transfer coefficient II for the various H_i values is enormous: the transfer coefficient II equals 1.1 for $H_i=0.1$, whereas it is only 0.05 for $H_i=9.0$ at 1.0 of the normalized coating thickness. This can be explained physically as above since the higher H_i values (stiffer coating relative to the substrate) give the higher compressive stress pulse striking the free coating surface. Interestingly, the value of transfer coefficient II is

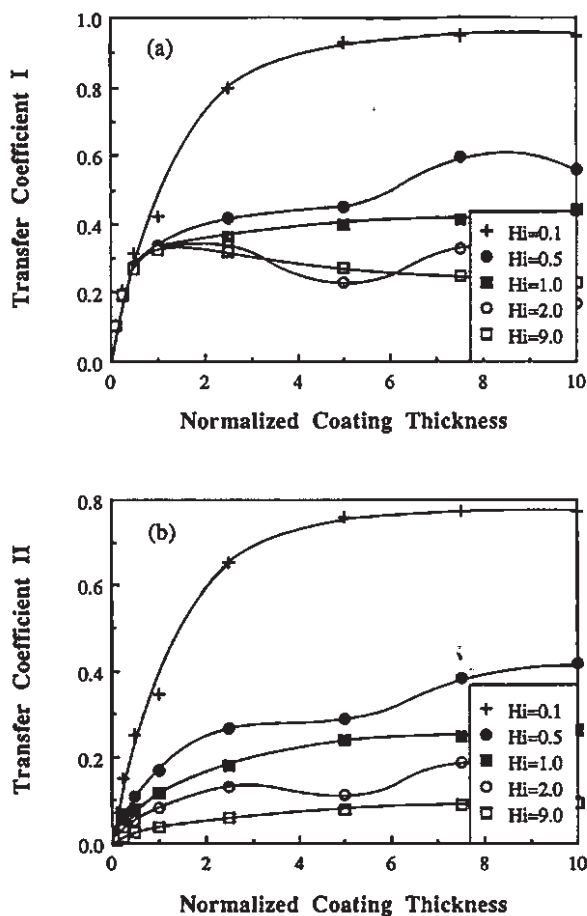


FIG. 7. Influence of the coating and substrate characteristics on the interface stress for $\beta_i = 5.0$. (a) Transfer coefficient I. (b) Transfer coefficient II.

larger than 1.0 when H_i is equal to and larger than 0.5 for some ranges of the coating thickness. Therefore, for such combinations of elastic and geometric properties, the stress at the interface can be higher than the peak compressive stress amplitude striking the free surface.

When β_i is not equal to 1, i.e., the coating and the substrate do not have the same densities, the results are quite different. The transfer coefficients corresponding to the two cases, $\beta_i = 5.0$ and 0.2, are given in Figs. 7 and 8, respectively. The profile of the generated stress pulse in the given substrate remains similar (except for its amplitude) within the range of the laser fluences used in the experiment, therefore the same chart can be used at different laser fluence levels, although for the same normalized pulse profile here, as shown in Fig. 5.

2. Effect of the sharpness and shape of the stress pulse

The peak tensile stress is also affected by the rise and fall times of the substrate stress pulse. As discussed above, the interface stress reduces as the thickness of the coating decreases. For applications to thin coating interfaces (with coating thickness less than $0.1 \mu\text{m}$), it is of interest to see if the interface stress can be enhanced by shortening the

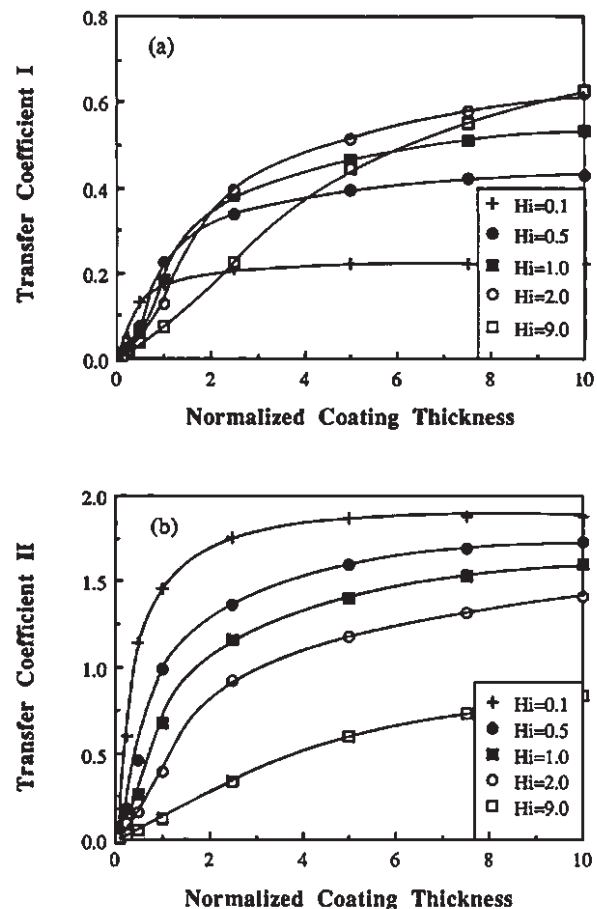


FIG. 8. Influence of the coating and substrate characteristics on the interface stress for $\beta_i = 0.2$. (a) Transfer coefficient I. (b) Transfer coefficient II.

stress pulse rise and fall times. This is studied in this section, and the details of how an optimal pulse profile can be obtained experimentally are discussed in Part III.

For an investigation of the effect of the sharpness and shape of the stress pulse, we considered a series of special stress waves, which are shown in Fig. 9(a), and determined the values of transfer coefficient I as a function of the normalized coating thickness. Stress pulse 1 is the same as that used above. Stress pulse 2 has a sharper post-peak decay, while stress pulse 3 has identical rise and decay times to that of a typical Nd:YAG laser heating pulse. The comparison of the interface stress generated by pulses 1, 2, and 3 reveal that the effect of the post-peak decay time, whereas the effect of the rise time should be clear by comparing the stresses due to the pulses 3 and 4, which have the same fall time, but very different rise times.

Figures 9(b) and 9(c) show the change in transfer coefficient I with normalized coating thickness for different stress pulses at $H_i = 0.1$ and $H_i = 5.0$ for $\beta_i = 1$. The transfer coefficient I is used because it is actually the normalized interface stress for a given stress profile. The results in Fig. 9 indicate that the post-peak decay times significantly influence transfer coefficient I or the interface stress. The enhancement depends on the coating thickness and the H_i ,

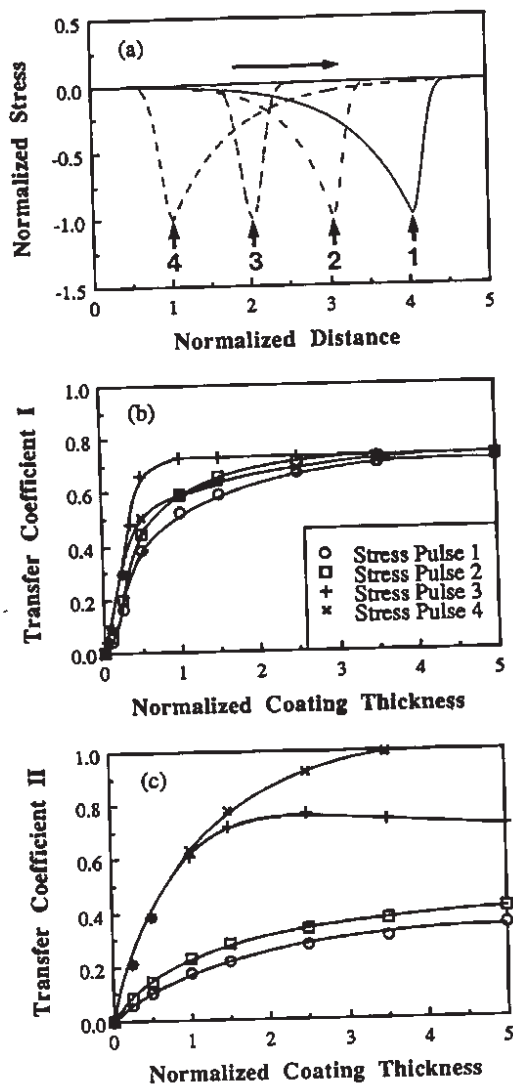


FIG. 9. Effect of the sharpness and shape of the stress pulse on the interface stress. (a) The specially designed stress waves. (b) Transfer coefficient I for $H_i=0.1$ and $\beta_i=1.0$. (c) Transfer coefficient I for $H_i=5.0$ and $\beta_i=1.0$.

value. Curves for stress pulse 1 and 2 in Fig. 9(b) show that even a small alteration in the sharpness of the fall side can create an enhancement of up to 10% of the normalized interface stress.

The interface stress due to pulses 3 and 4 shows no increase at high H_i value and a small increase at low H_i values for thin coatings. Therefore, a sharper post-peak fall time of the stress profile is more significant in enhancing the interface stress for thin coatings than a shorter rise time pulse.

V. DISCUSSION AND CONCLUSIONS

We have described a modified laser spallation experiment to measure the tensile strength of thin-film interfaces. The current technique is an improvement over an earlier version since the interface tensile strength is determined directly by using a Doppler displacement interferometer.

Since the transient velocity can be measured at either the free substrate surface or the free coating surface, it is also possible to decide which free surface should be used for the most efficient usage of the Doppler interferometers. In case the maximum amplitude at the free substrate surface is beyond the highest resolution of the Doppler displacement interferometer, the signal can be picked up at the free coating surface for softer coatings. Similarly, for stiff coatings, when the coating's free-surface velocity is beyond the resolution limit of the interferometer, the substrate's free surface can be used to record the velocity. Similar strategies can be used when the velocities are lower than the sensitivity limit of the interferometer. In addition, the free surface of the substrate is chosen if it has good reflective properties. For systems involving rough substrates and coatings (e.g., polycrystalline alumina), the standard Doppler displacement interferometer cannot be used to record the free-surface velocity profiles. For such cases, a WAMI¹¹ interferometer can be used to obtain the free-surface velocities.

In the modified version, the stress striking the free surface can be determined experimentally without considering how the stress is generated through the complicated, nonlinear, laser-material interactions, especially when liquid columns are used as the constraining media (see Part III). This unique feature allows us to find the effect of the experimental conditions on stress pulse shape and amplitude, which (as shown in Part III) leads to hitherto unreported stress pulse profiles with 1.14 ns rise times and with no post-peak decay tail. The stress becomes zero at 17–20 ns.

The numerical simulation of the wave mechanics associated with the building up of the tensile interface stress indicate that despite stress pulse length in considerable excess of the coating thickness (20–30 times!), the profile of the transient pulse remarkably generates sufficient interface tensile stress to produce spallation. Indeed, as shown in Part II, the technique has been used successfully in several substrate/coating systems with a maximum coating thickness of only 3 μm .

As discussed in Sec. IV, the properties and the thickness of the substrate/coating system, and the amplitude and sharpness of the stress pulse generated by the laser pulse determine the amplitude of the interface tensile stress, which can be determined through the transfer coefficient I in Figs. 6(a), 7(a), and 8(a). Finally, it was found that the interface tensile stress is less sensitive to the rise time of the substrate stress pulse and very sensitive to the post-peak decay characteristics of the pulse. As shown by our experimental pulse optimization results in Part III, it is possible to change the post-peak decay significantly while the stress pulse rise time is almost fixed by the rise time of the Nd:YAG laser pulse.

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