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Applications of Laser Generated Stress Pulses for Materials Characterization

A Thesis
Submitted to the Faculty
in partial fulfillment of the requirements for the
degree of

Doctor of Philosophy

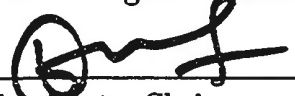
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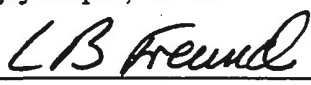
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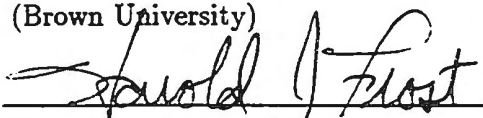
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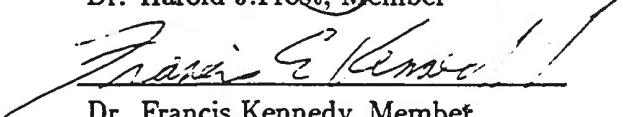
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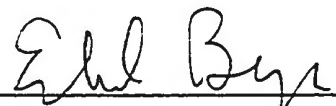
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Applications of Laser Generated Stress Pulses for Materials Characterization

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August 6, 1995

Abstract

Previously a laser spallation experiment was developed to measure the tensile strength of thin film interfaces. In this experiment, 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 interface stress is determined directly by recording the coating free-surface velocities using a Doppler interferometer. Since the interface separation occurs at a strain rate of almost 10^7sec^{-1} , the plastic deformation that usually accompanies the interface decohesion process is largely suppressed, and the measured tensile strength can be related to the intrinsic interface fracture energy by using a phenomenological atomic stress-separation relationship.

The aim of this thesis work was to extend the laser spallation technique to measure directly the interface fracture energy by implanting well-defined interfacial flaws. To this end, penny-shaped interfacial cracks were generated by encouraging buckling of the overlayer from selected spots on the substrate, by utilizing the intrinsic compressive stresses within the coating. The laser-produced shock wave was then used to load these flaws, and the critical stress amplitude responsible for their propagation was recorded. The key element of the work was to relate the measured free surface velocity to the local stress intensity factor for a well defined flaw, by using the concepts of dynamic fracture mechanics. This was implemented via a finite element simulation process. Besides acting as a reference to the strength measurements accomplished previously, this work now allows the measurement of the fracture energies of several metal/ceramic interfaces of interest to the microelectronic and composites industries.

In addition to the above, the potential of low-amplitude laser-generated stress pulses for non-destructive evaluation of flaws in composites and other engineering solids is also discussed.

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Introduction

The use of stress-waves produced by lasers and mechanical impact assemblies represents a unique tool for investigating the properties of materials, including the measurement of the elastic constants [1, 2], tensile strength [3], constitutive behavior under high strain rates [4], and dynamic fracture toughness [5]. In some cases it is the only tool that can be used to obtain information about material's internal structure and about its static and dynamic mechanical behavior. The major advantage of the stress wave techniques over those utilizing electro-magnetic waves is that the stress waves can propagate freely in the media whereas the electro-magnetic waves cannot propagate to a significant distance, for example in metals. Another advantage of the stress wave technique is that it allows direct mechanical testing of different systems using the tension supplied by the wave. For example, the tension stress supplied by the propagating wave can be used to measure the tensile strength of fiber/matrix interfaces in composites or those between thin films and substrates.

All applications of stress waves that involve material characterization may be divided into two groups: non-destructive and destructive applications. Non-destructive (NDE) applications use the low amplitude waves that do not produce any damage in the material. Typical examples of non-destructive applications include acoustic microscope, and mapping of internal microcracks through the vibration mode analysis. Destructive applications might involve tensile strength testing of different interfaces by a high amplitude tension pulse.

To this end, a previously developed laser spallation experiment was used. In this experiment the stress pulses are generated by the absorption of a laser pulse by thin film material. Such stress pulses have a very short duration, typically 17 *ns* in most of our experiments, and a very high amplitude, which could be made as high as 10 *GPa*. These parameters allow the laser spallation technique to test the strength of thin film interface in a well-characterized loading which otherwise is not possible by the other techniques.

The main aim of this thesis work was to extend the laser spallation technique devel-

oped previously to measure the strength of thin film interfaces to measure directly the interface fracture energies by implanting well-characterized interfacial flaws. To this end, two photolithography-based techniques were developed to generate interface microcracks. The stress pulse amplitude required to initiate the propagation of these microcracks was recorded *via* an optical interferometer and related to the local interface energy release rate by a dynamic wave-mechanics simulation. Development of this technique also allowed to explain very low strength values obtained previously for the interface between several metallic coatings and pyrolytic graphite substrate. The final aim of the thesis was to develop an experimental technique for studying the damage in laminated composite structures subjected to high amplitude stress pulses. Besides allowing for a structural design against such impacts, this work also led to the development of a strategy for *in situ* measurement of interface tensile strength in actual composite structures, by adapting the laser spallation experiment.

The presented thesis has the following structure. In Chapter 1 the development of the laser spallation technique is discussed. It includes the description of the previous model of the technique which was designed by Prof. V. Gupta at MIT (Section 1.2), and its subsequent development by his group at the Thayer School of Engineering, (Section 1.3). The stress pulse generation mechanism in solids by the laser pulse absorption process is considered in Section 1.4. The pulse generation has been optimized with respect to the choice of the laser absorbing film, constraining material, substrate material and laser fluence. Optimization of this process allowed generation of very short stress pulses with no long asymptotic tail, and discussed in Section 1.5. The stress field within the material was determined *via* an optical interferometer by recording the free-surface displacement. A special interferometer capable of measurements on rough surfaces was used for this purpose. The description of this interferometer is given in Section 1.6.

Chapter 2 discusses the measurement of the interface fracture toughness using the laser spallation experiment. The high strain rate loading in the spallation experiment largely suppresses the plastic deformation within the separating solids. The review of the literature on plastic deformation at high strain rates (Section 2.1) allowed making such an assumption for the material interfaces undertaken in this study. The idea behind the fracture toughness measurement was as follows. By means of photolithography methods, microcracks with well defined geometries were implanted at the interface of interest. These microcracks were then loaded by a high amplitude stress pulse. A numerical finite element program (described in Section 2.3) modeled the propagation of this stress pulse in the material and calculated the energy release rate at the microcrack tip during transient loading. The interface fracture energy was determined for the stress pulse profile recorded

at the threshold laser energy, (Section 2.4).

The interface toughness values obtained by the laser spallation technique were compared with those obtained independently by a spontaneous delamination technique, which is discussed in detail in Chapter 3, [6]. In this technique, a test coating of 100 Å to 200 Å thickness is deposited on the substrate of interest. This layer is then overlaid by a residually-stressed Nb coating (used as a loading device) and its thickness is controlled until the lower test layer buckles away from the substrate. By determining the intrinsic residual strain within the Nb layer in independent delamination experiments, the bilayer buckling morphology is quantified, and a measure of the interface fracture toughness is obtained. This technique has been successfully applied to several metal/sapphire interface systems.

Chapter 4 covers the application of the laser generated stress pulses in non-destructive testing. The primary purpose of this chapter was, however, the measurement of the fiber/matrix interface strength of carbon/carbon and carbon/polyimide composites. High amplitude stress pulses were used for this purpose. The debonding phenomenon in 2D carbon/carbon (C/C) and carbon-polyimide (C/P) laminates was studied by subjecting them to laser-produced nanosecond rise-time compressive stress pulses [7,8]. The compression pulse travels through the laminate normal to the plies and, upon reflection into a tension pulse from the sample's front surface, leads to fiber/matrix debonding either in the bundles within a ply or at the interply interface. The stress pulse amplitude responsible for initiating such damage was determined by recording the free surface velocity by an optical interferometer; on the continuum scale, this is referred to as the effective interply strength. By employing a special specimen geometry we were able to determine the on-set of such delaminations. The low amplitude stress pulses that have been sent through the thickness of the sample immediately after the propagation of high amplitude pulses helped to detect the induced damage in the material. The short stress pulses allowed as well measurement of the dispersion characteristics associated with the propagation of such stress pulses for both types of composite microstructures. The change in the shape of the low amplitude stress pulses that takes place during the propagation of the pulse in composites gave information necessary to determine the frequency dependence of such material characteristics as the averaged speed and attenuation of elastic waves. Using the interply strength as the local failure criterion, information on the dispersion characteristics allows the complete design of a composite component under impact conditions.

Chapter 1

Development of the Laser Spallation Experiment

1.1 Introduction

Several techniques exist in the literature for the determination of some average mechanical properties of the interface. References [9] and [10] contain critical evaluation of these methods and a bibliography on adhesion measurements respectively. The thrust of this chapter, however, is to provide a comprehensive review of developments in characterizing the tensile strength of thin film interfaces by a laser spallation technique.

In this technique [3,10,11] a high energy laser pulse 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. The typical result of the spallation is shown in Fig. 1.1. The feasibility of the spallation process was initially shown by Vossen [12] but no quantification was provided. This was first attempted by Gupta *et al.* [3,10], where the critical stress amplitude that accomplishes the removal of the coating was determined through a computer simulation of the process, which, in turn, was verified by means of a piezo-electric crystal probe that was capable of mapping out the profile of the stress wave generated by the laser pulse. This is briefly discussed in Section 1.2. More recently, the spallation experiment was modified [11,13–15], the interface strength was determined more accurately by recording the transient displacement and velocity of the coating free surface by a Doppler interferometer, and related to the interface stress using wave mechanics, albeit by assuming the density and the elastic modulus of the coating.

Two additional developments were made which increased the versatility of the tech-

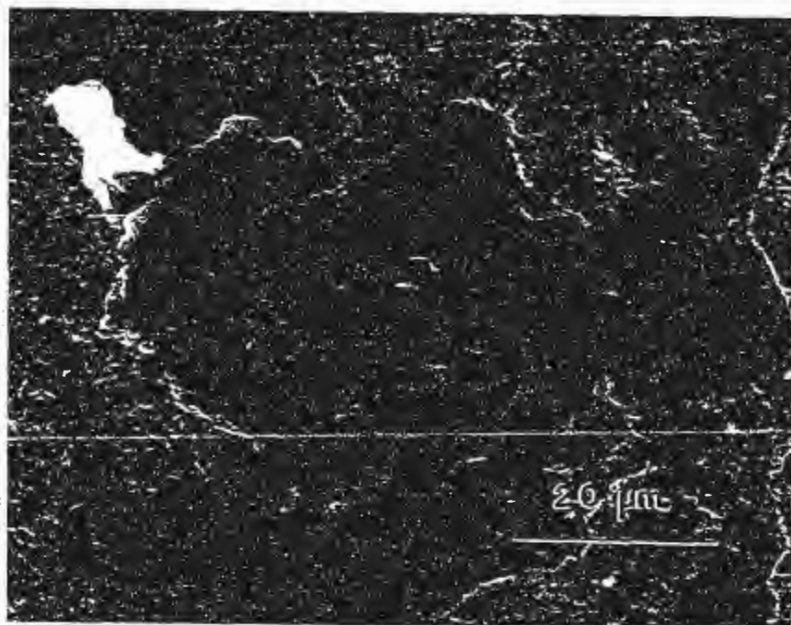


Figure 1.1: A micrograph showing a typical spallation crater. Here a 3 μm thick Nb coating is removed from an alumina substrate.

nique, allowing coatings with rough surfaces (*e.g.* thermal spray coatings) and those with thickness less than $0.1\ \mu\text{m}$ to be tested. The first development was that of producing sub-nanosecond rise-times stress pulses with amplitudes in excess of $3\ \text{GPa}$ in the substrate [14]. In contrast to pulse profiles assuming an asymptotic tail at about 5% to 10% of the of the peak stress, the new pulses show hitherto unreported, much sharper post-peak decays resulting in a zero-stress at about $17\ \text{ns}$. Since in the laser spallation experiment, the tensile stress at the interface results after reflection of the parent compressive stress pulse from the coating surface, a sharper rise-time and a sharper post-peak decay enhance the interface tensile stress in thin film interfaces, and as shown below, films $0.1\ \mu\text{m}$ in thickness could be separated from the substrate. The setup for producing such short stress pulses is given in Section 1.3 below.

The second development was that of an optical interferometer that allowed us to record the transient velocities of rough surfaces [16], which otherwise destroy the geometrical coherence of the reflected beam and make regular interferometry unusable. This setup is discussed in Section 1.6.

1.2 Previous Version of the Laser Spallation Experiment

The first successful laser spallation experiments were performed by Gupta *et al.* [3,10] at MIT in 1989. The experimental approach utilized a planar arrangement of a substrate and coating combination, as shown in Fig. 1.2. Details of the sample holder are given in Gupta *et al.* [3,10]. The collimated laser pulse is made to impinge on a $1\ \mu\text{m}$ 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 $1.06\ \mu\text{m}$). 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 pulse amplitude. A key element of the experiment was to determine the amplitude of the interface tensile stress at the threshold laser fluence causing interface failure.

Previously, the approach consisted of a three-part strategy. The first part was the

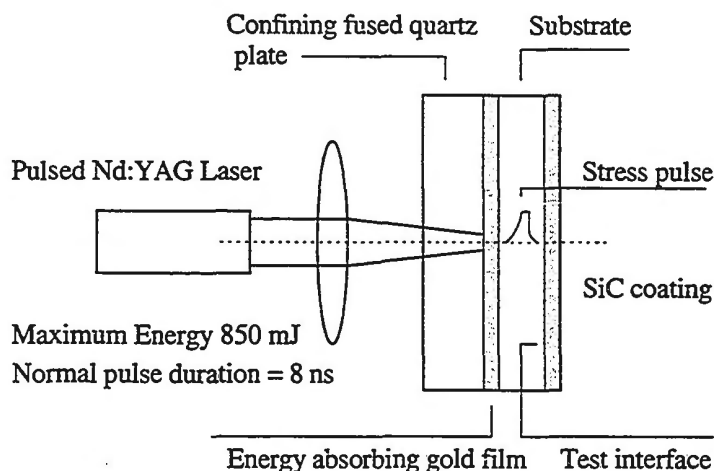


Figure 1.2: Schematic of the old version of the laser spallation experiment.

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 conditions of the computer simulations were experimentally achieved. This permitted verifying and fine tuning of the computer simulation. Finally, in the third part, actual spallation experiments were carried out for several thin film coating/substrate interfaces (*e.g.* SiC/C 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. The analysis of the post-spalled samples through in-depth Auger spectroscopy showed the failure to occur at the interface in the SiC coated pyrolytic graphite (PG) and Si wafer disks. The SiC/Si interface was varied by changing the deposition conditions of the SiC coating, and accordingly, interface strength values ranging from 3.7 to 10.5 GPa were determined. An average value of 7.2 GPa was determined for the SiC/PG interface.

1.3 Modified Version

A schematic of the modified setup is shown in Fig. 1.3. Two basic modifications from the previous version include the use of the optical interferometer to measure the velocity history of the free surface and the replacement of the fused quartz plate by a thin layer of water glass (sodium silicate) as a constraining layer [13,15]. The solid water glass layer was obtained by applying a thin layer of $\text{H}_2\text{SiO}_3 : (\text{H}_2\text{O})_x$ to the laser absorbing

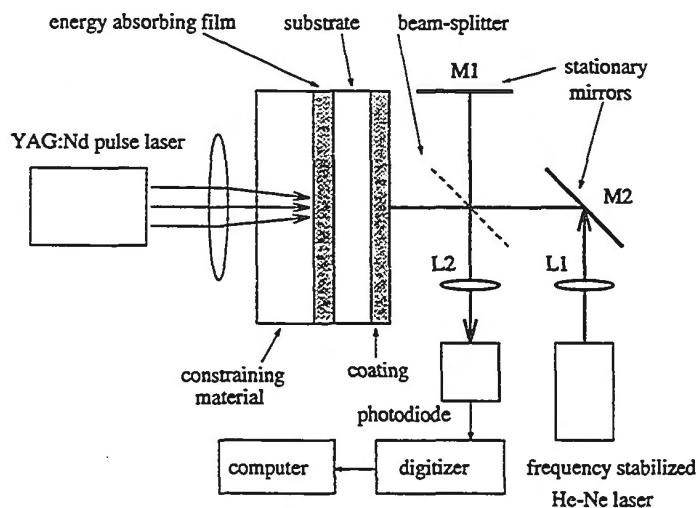


Figure 1.3: Schematic of the modified version of the laser spallation experiment with the displacement interferometer.

metal film; after several minutes exposure to the air, water evaporates from the water glass leaving behind a layer of SiO_2 that is transparent to the laser wavelength. The use of solid water glass is critical for the mechanism that allows producing sub-nanosecond rise-time stress pulse without asymptotic tail and is discussed in Section 1.5 below.

When the stress pulse is reflected from the free surface of the coating of the substrate, the free surface experiences a transient velocity which is proportional to the transient profile of the striking stress pulse. For specular surfaces, it is possible to measure both the free surface displacement and the velocity by using the standard laser Doppler displacement and velocity interferometers [17,18] which are used widely in plate impact research. Unfortunately, for many materials the roughness of the surface makes measurement of the velocity by standard Doppler techniques impossible. To tackle this problem, we used a new displacement interferometer which is capable of measuring the free surface velocities, as discussed in Section 1.6. In addition to the above, recording sub-nanosecond displacement and velocity fringes induced by sharp stress pulses (in many cases the rise-time was less than 1 ns) over a small area of only 7 mm² posed a certain challenge.

In a displacement interferometer (see Fig. 1.3), a beam splitter divides a He-Ne laser beam into two equal beams. The beam which is reflected from the stationary mirror (M1) is the reference beam, while the one reflected from the substrate's rear surface is the signal beam. When the stress wave arrives at the rear surface, the movement of the surface results in a change in the signal beam frequency through the Doppler effect and the change of the effective optical path length. Since the rear velocity $v(t)$ is much

less than the velocity of the light c , the Doppler shift in frequency $\nu(t)$ is given by [18]

$$\Delta\nu(t) = \frac{2v(t)}{\lambda_0}, \quad (1.1)$$

where λ_0 is the wavelength (632.8 nm) of the He-Ne laser. The photodiode output voltage amplitude $A(t)$ is recorded on a transient digitizer and related to the fringe count of the displacement interferometer as:

$$A(t) = \frac{A_{min} + A_{max}}{2} + \frac{A_{min} - A_{max}}{2} \sin \left(\frac{4\pi u(t)}{\lambda_0} + \phi_0 \right), \quad (1.2)$$

where A_{max} and A_{min} are the maximum and the minimum amplitudes, respectively, $u(t)$ is the free surface displacement, and ϕ_0 is the initial phase angle. Once $A(t)$ is measured by a digitizer, several numerical methods have been used to recover the displacement history of the surface. The displacement interferometer gives very precise data since one complete fringe record corresponds to a $\lambda_0/2$ displacement of the free surface. Since this displacement takes place within a very short time period (10-50 ns) and the rise time of the stress pulse is of the order of 1 ns, the experiment required a very fast acquisition system. The use of a Tektronix SCD1000 digitizer with a sampling rate of 200 GHz (frequency band 3 GHz), and a Newport 877 ultra-high speed photodetector with a rise time of less than 200 ps allowed us to record such fringes with a resolution of 0.3 ns.

In some cases it was required to measure only the profile of the surface velocity. The velocity interferometer was better suited for this purpose. The basic difference between the velocity interferometer (Fig. 1.4) and the displacement interferometer (Fig. 1.3) is that in the first one the signal and reference beams are essentially the same beam, but have different time delay δt . This makes the velocity interferometer less sensitive to the surface quality and surface displacement. The photodiode voltage output in this case is related to the velocity $v(t)$ of the surface as:

$$A(t) = \frac{A_{min} + A_{max}}{2} + \frac{A_{min} - A_{max}}{2} \sin \left(\frac{4\pi v(t) \delta t}{\lambda_0} + \phi_0 \right), \quad (1.3)$$

where δt is the time delay between the two laser beams. The above equation is valid as long as the characteristic time of the velocity variation is much longer than δt . Since the shortest characteristic time of the stress pulses generated in the spallation experiment is the rise time, which is in the range of 1 ns, the delay between two laser beams in the velocity interferometer expressed in the form of the length should be much less than 30 cm. If the surface velocity is relatively slow, e.g. when $v(t) \delta t \ll \lambda_0$, then the velocity interferometer works in the linear regime and Eq. 1.3 can be simplified as

$$A(t) \sim v(t). \quad (1.4)$$

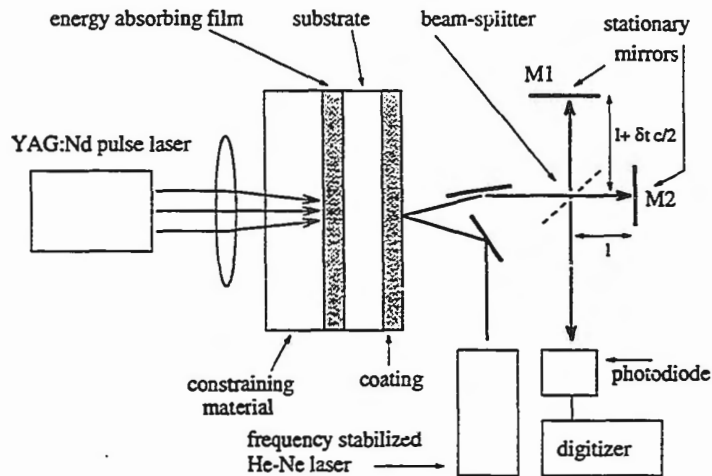


Figure 1.4: Schematic of the modified version of the laser spallation experiment along with the velocity interferometer.

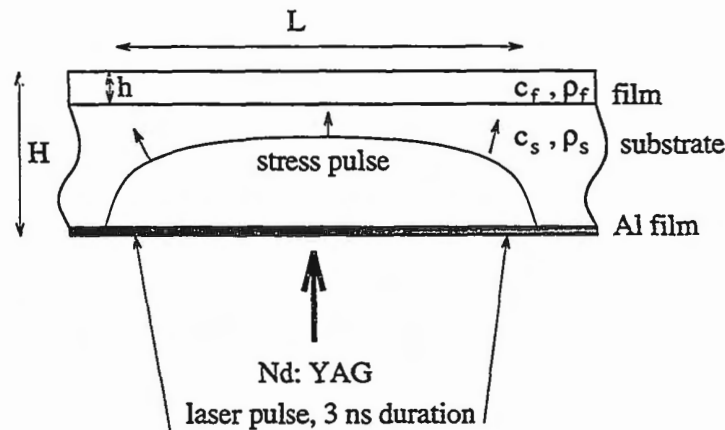


Figure 1.5: Schematic of the stress pulse generation.

A frequency stabilized He-Ne laser was used in both interferometers to obtain a clean fringe record, free from the beat frequency of the laser. The He-Ne laser beam was aligned and focused at the center of the sample's rear surface opposite to the spot illuminated by the Nd:YAG laser pulse. Stress pulses were produced over a spot 3 mm in diameter (L), see Fig. 1.5. In most of the experiments, the diameter of the laser heating spot was maintained at 3 mm, with a total sample thickness (H , Fig. 1.5) ranging from 0.8 mm to 1.2 mm. As shown by the finite element simulations of Nutt [19] for plate impact experiments, and by Yuan [20] for the propagation of laser-produced shock waves, such a geometry ensures a planar stress front to reach the free surface of a plate made out of homogeneous elastic material. This was also confirmed by doing interferometric mea-

surements on the rear surface of a Si wafer by changing the location of the He-Ne laser beam in subsequent experiments corresponding to the same laser energies [20].

It should however be pointed out that the attainment of such a planar condition is not really necessary in our experiments since the quantification of the threshold stress is achieved *via* a velocity interferometer that records the information from the center of the loaded area. The ratio of the illuminated spot size to the thickness of the film (L/h , Fig. 1.5) and the ratio of the substrate thickness to h (H/h , Fig. 1.5) both are of the order of 10^3 , therefore at scales close to the scale of thin film thickness, the stress wave propagation can be considered as one-dimensional even with a larger degree of planarity.

For a material with two Lamé elastic constants, λ and μ , and a constant density ρ , the longitudinal one-dimensional wave propagation is governed by the equation

$$\left(\frac{\lambda + 2\mu}{\rho} \right)_{f,s} \frac{\partial^2 u_{f,s}}{\partial x^2} = \frac{\partial^2 u_{f,s}}{\partial t^2} \quad (1.5)$$

or

$$c_{f,s}^2 \frac{\partial^2 u_{f,s}}{\partial x^2} = \frac{\partial^2 u_{f,s}}{\partial t^2}, \quad (1.6)$$

where $u_{f,s}$ is the displacement component in the x direction in the film and substrate. In the above, the subscripts s and f refer to the substrate and film respectively, and c is the longitudinal wave velocity. The stress (σ) in the x direction can be expressed in terms of the displacement gradients as

$$\sigma_{f,s}(x, t) = (2\mu + \lambda)_{f,s} \frac{\partial u_{f,s}}{\partial x}. \quad (1.7)$$

At the free surface of the film, the one-dimensional wave propagation normal to the free surface satisfies the boundary conditions

$$\sigma(x = 0, t) = 0 \quad (1.8)$$

and

$$v(x = 0, t) = 2v_f(t) = V_0(t), \quad (1.9)$$

where $V_0(t)$ is the measured velocity of the film free surface. In the modified experiment, two strategies were used to quantify the stress history at the interface, based on the data obtained by the Doppler techniques. In the first one, used by Yuan [20], the transient velocity was measured at the substrate's surface which had no test coating on its surface. The peak interface tensile strength generated at each level of the laser fluence was related to the maximum velocity of the free surface of the substrate 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 stresses at the interface were determined. The program simulated the one-dimensional propagation of the stress pulse in both the substrate and the film, and the reflection of the incoming and out-going stress pulses at the free surface and the interface. The above procedure resulted in a plot of laser fluence *vs.* the interface stress, which was numerically calculated based on the known materials properties, geometry, and on the interferometrically measured incoming stress pulse profile.

In the second strategy, the velocity was measured at the free surface of the film during each spallation test. In this case the recorded data were directly related to the actual stress pulse levels. The stress at the interface was determined based on the recorded free surface velocity in the following way.

Equation (1.6) of the one-dimensional wave propagation normal to the free surface of the film with boundary conditions (1.8) and (1.9) has the obvious solution

$$\begin{aligned} u(x, t) &= u_f\left(t + \frac{x}{c_f}\right) + u_f\left(t - \frac{x}{c_f}\right), \\ v(x, t) &= \frac{\partial u}{\partial t} = v_f\left(t + \frac{x}{c_f}\right) + v_f\left(t - \frac{x}{c_f}\right), \\ \sigma_f(x, t) &= \rho c_f^2 \frac{\partial u}{\partial x} = \rho c_f \left(v_f\left(t + \frac{x}{c_f}\right) - v_f\left(t - \frac{x}{c_f}\right) \right), \end{aligned}$$

where c_f is the longitudinal stress wave speed in the film, ρ is the film density, $u(x, t)$, $v(x, t)$ and $\sigma(x, t)$ are the particle displacement, velocity and the induced stress, respectively. Therefore the interface stress history can be easily determined as

$$\sigma(x = h, t) = \frac{1}{2} \rho c_f \left[V_0\left(t + \frac{h}{c_f}\right) - V_0\left(t - \frac{h}{c_f}\right) \right], \quad (1.10)$$

where h is the thickness of the film (Fig. 1.5). It is worth mentioning two advantages of this approach. First of all, unlike the previous procedure, the use of Eq. 1.10 eliminates the necessity of running the complicated computer code to calculate the interface stress. And second, the substrate properties are not required to determine the interface stress history.

1.4 Experimental Optimization of the Stress Pulse

The aim of this section is to discuss the optimization of the stress pulse generation conditions in the laser spallation technique by changing the type and thickness of the

laser-absorbing film, and its constraining medium. In addition, the influence of the substrate and laser fluence on the profile and amplitude of the stress pulse is also presented. This work was done in collaboration with Jun Yuan [11,14,15].

The modified laser spallation technique allows us to perform this optimization process experimentally since the generated stress pulse is detected at the rear surface of the coating or substrate. A competitive numerical model for obtaining the optimal conditions is very difficult to achieve as this requires modeling the multi-photon ionization process within the plasma, created at the film surface upon absorption of the laser energy. The ablating particles in the plasma produce a force normal to the material surface by momentum transfer. Acoustic waves formed by such processes differ markedly from those formed by the thermoelastic expansion of the solid or molten film. Since in the laser spallation experiment a combination of these processes occurs at the threshold laser fluences, a direct experimental optimization, as pursued here, is the only reliable way of studying such laser/material interactions. In addition, a column of liquid was used as an alternative to the solid confining medium in some experiments, which makes the modeling even more complicated.

The generation of stress pulses in solids by lasers has been pursued actively since 1963, starting with the study by White [21] of laser-produced ultrasounds. One of the main interests over the years has been to enhance the stress pulse amplitudes. The first and the simplest approach involved using high-power laser pulses. The second approach involved using constraining layers on the surface of the solids or laser-absorbing films, resulting in an amplitude enhancement through constrained plasma expansion [22,23]. O'Keefe and Skeen [24] used coatings of transparent, relatively volatile materials to produce an order of magnitude increase in the pulse amplitudes, and Fox [25] showed that a thin coat of black paint or drops of distilled water can produce results comparable to the constraining techniques. Yang [26] investigated the effect of various laser-absorbing metallic films on the amplitudes of the stress pulses produced in a piezo-electric quartz crystal. However, few researchers [27,28] discussed the effect of these on the stress pulse profile (defined by both the rise and fall times of the stress pulse). In our research results, presented here, we studied the changes in both the profile and amplitude of the stress pulses generated by varying the various experimental conditions in the laser spallation setup, including the type and thickness of the constraining material, and the laser-absorbing film. In addition, we also explored the effect of the laser fluence and the substrate material on the stress pulse.

All experiments for stress pulse optimization were performed using the modified laser

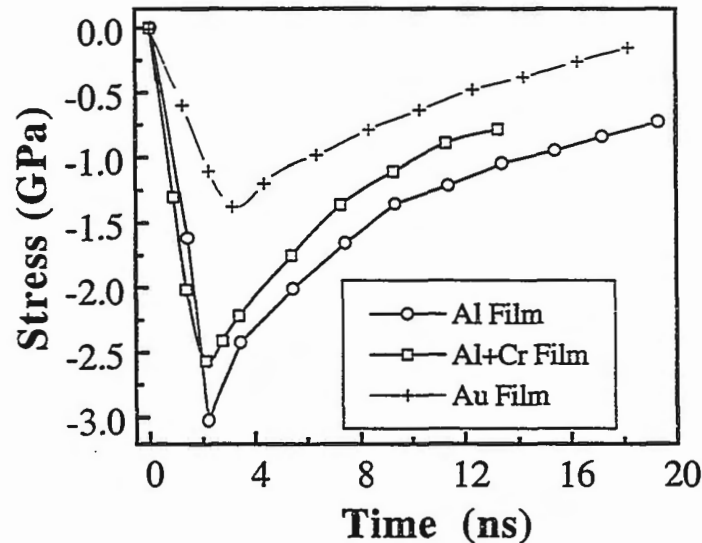


Figure 1.6: Influence of the type of laser-absorbing film on the stress pulse profile (laser fluence = $8.7 \times 10^4 \text{ J/m}^2$; constraining material: a fused quartz disk).

spallation setup discussed in detail in Section 1.3. Except when studying the effect of the substrate material itself, all experiments reported here were performed on substrates of *n*-type Si single-crystal wafers with a 25.4 mm diameter and a 1 mm thickness. Both (100) surfaces of the Si wafer were polished to an optical quality. The deposition of the laser-absorbing films was done by DC- and RF-magnetron sputter systems. The amplitudes and the profiles of the stress pulses were measured by monitoring the transient free-surface velocity of the Si surface by the Doppler displacement interferometer.

1.4.1 Influence of the Type of the Laser Absorbing Film

Figure 1.6 shows the profiles of stress pulses generated in the Si wafer with different laser absorbing films. Since each film has a different absorption coefficient at $1.06 \mu\text{m}$, for the purpose of comparison all experiments were performed at a laser fluence of $8.7 \times 10^4 \text{ J/m}^2$, measured before the laser pulse strikes the film. A fused quartz disc was used as the constraining material and lapped onto the film in a manner discussed in Gupta *et al.* [3, 10]. The candidates for the laser absorbing films were Au, Al, and Al+Cr. The bilayer of Al+Cr involved depositing a film of Al on the substrate, followed by a deposition of the Cr film. In this arrangement the Al film acts as a stress-generating film because of its high thermal expansion coefficient, while the Cr film maximizes the absorbed laser energy because of its low reflectivity to the $1.06 \mu\text{m}$ laser beam. The

thicknesses of Au, Al, and Cr films were chosen to be $0.5\ \mu\text{m}$, $0.5\ \mu\text{m}$, and $0.06\ \mu\text{m}$, respectively. Figure 1.6 shows that the Al and Al+Cr films produced higher amplitude and lower rise time stress pulses than the Au film. Specifically, the rise-times for the Au, Al, and Al+Cr films are $3\ \text{ns}$, $2.2\ \text{ns}$, and $2.1\ \text{ns}$ respectively, while the corresponding peak stresses, as measured from the interferometer, were $1.34\ \text{GPa}$, $2.5\ \text{GPa}$, and $3.0\ \text{GPa}$, respectively. The rise-time was defined as the time interval from the pulse start to the pulse maximum. The decay time of the pulse is controlled by the extent of the thermal pulse propagation within the film [3,10] which is controlled partly by the thermal diffusivities of the film and the constraining medium. Figure 1.6 shows similar decay times for all three cases. The low rise time for the Al film in comparison to the Au film is due to its lower heat capacity and lower reflectivity resulting in higher temperatures, as the thermal diffusivities of Al and Au are quite comparable. The thermal diffusivities for Al and Au are $0.982 \times 10^{-4}\ \text{m}^2/\text{sec}$ and $1.30 \times 10^{-4}\ \text{m}^2/\text{sec}$, respectively. Clearly, the rise time of the stress pulse is limited by the rate at which the heat energy is input, which in turn is controlled by the rise time of the laser pulse, being equal to about $1.5\ \text{ns}$ in our experiments. Although the rise time of $2.1\ \text{ns}$ for the Al+Cr film is marginally lower than $2.2\ \text{ns}$ obtained using the Al film alone, the amplitude of the stress pulse is about 20% lower. Therefore with respect to both the amplitude and rise time optimization, a layer of Al was chosen for the modified laser spallation experiment.

1.4.2 Influence of the Type of Constraining Material

The reproducibility of the results in the laser spallation experiment depends critically on the fused quartz/laser-absorbing film interface. Since the experimental assembly ensures only a hermetic contact between the two materials, it is imperative that the surfaces be flat in order to avoid trapping of interface air pockets. In order to remove the possibility of such pockets, the possibility was explored of using free standing columns of liquid, and thin layers of solid produced by applying liquid solution, which are transparent to the laser light. Interestingly, apart from achieving better reproducibility, both the stress pulse amplitudes and profiles were also optimized.

Three kinds of assembly were utilized to keep the constraining material in contact with the energy-absorbing film. When fused quartz was used as a constraining material, the assembly process described in Refs. [10] and [3] was used. The fused quartz was $3\ \text{mm}$ in thickness and $32\ \text{mm}$ in diameter.

As a second alternative, several liquids were also used as constraining materials. In these cases, a special liquid tank was designed and built. A schematic of the liquid tank

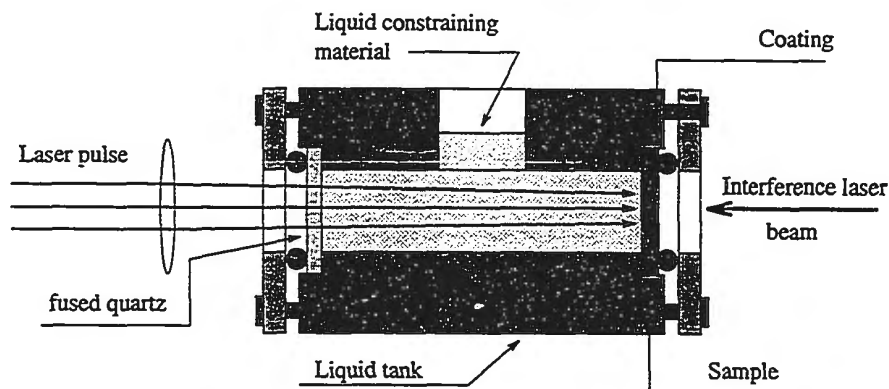


Figure 1.7: Schematic of the liquid tank used to generate the stress pulse.

is shown in Fig. 1.7. The liquid in the tank was held by a fused quartz wall and the surface of the sample was coated by the laser-absorbing film. The fused quartz also acts as a transparent window for the $1.06\ \mu\text{m}$ Nd:YAG laser pulse and allows it to reach the laser absorbing film through the liquid column. The O-rings prevent the liquid from leaking from the tank. The thicknesses of the liquid column and fused quartz were $25\ \text{mm}$ and $3\ \text{mm}$ respectively. As shown in Fig. 1.7, a $12\ \text{mm}$ diameter hole was provided on the upper part of the tank to release the air, otherwise the air pressure build-up upon evaporation of the liquid would break the substrate. The liquids studied included water, 2-propanol, and water glass ($\text{H}_2\text{SiO}_3 : (\text{H}_2\text{O})_x$).

Finally, a $0.5\ \mu\text{m}$ thick layer of solid water glass was used as a constraining medium. A thin layer of liquid water glass ($\text{H}_2\text{SiO}_3 : (\text{H}_2\text{O})_x$) was first applied to the energy-absorbing film; after several minutes exposure in the air, water evaporated from the water glass leaving behind a layer of SiO_2 film that is transparent to the laser wavelength. The solid water glass film fits exactly to the energy-absorbing film. The thickness of the solid film was controlled by the amount of liquid water glass. A laboratory procedure was adopted to yield a final solid layer thickness of approximately $5\ \mu\text{m}$. The thicker layer thickness lead to the longer stress pulse duration.

For all the constraining materials, Al film was used as the laser-absorbing film. The results at laser fluence of $8.7 \times 10^4\ \text{J}/\text{m}^2$ (measured at the Nd:YAG source) for $0.5\ \mu\text{m}$ thick Al film are presented in Fig. 1.8. Remarkably, a $0.5\ \mu\text{m}$ thick layer of solid water glass generates the strongest and sharpest stress pulse. Although the fused quartz plate produces the same amplitude of $3.0\ \text{GPa}$, the rise time of $3.05\ \text{ns}$ is almost 2 times that obtained by the layer of solid water glass. Furthermore, the stress pulse shows a much sharper decay in the case of solid water glass, a feature which is critical to increasing

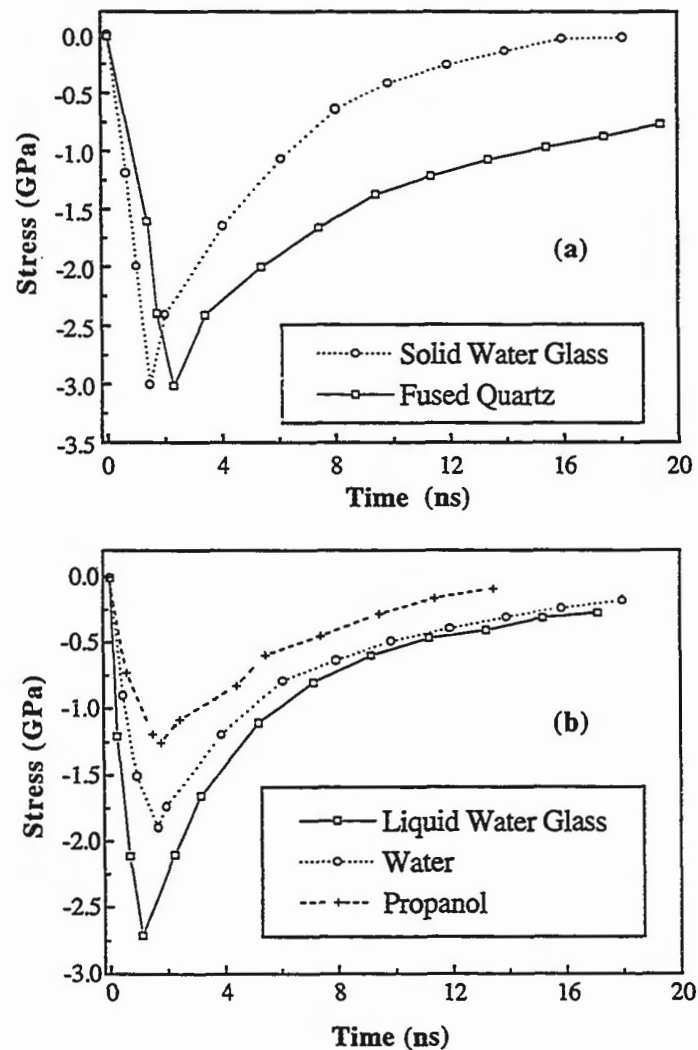


Figure 1.8: Influence of the type of constraining material on the stress pulse profile. All stress pulses were generated by a $0.5 \mu\text{m}$ thick Al film and at a laser fluence of $8.7 \times 10^4 \text{ J/m}^2$. (a) Using solid constraining media. (b) Using liquid constraining media.

the interface tensile stress. The shallow decay in the case of the fused quartz is due to a decrease in the effective thermal diffusivity and to the presence of fused quartz/substrate interface air pockets. As shown by Gupta *et al.* [3] such a decrease in the thermal diffusivity will result in a longer and more gradual tail of the stress pulse profile. In fact, earlier experiments by Vossen [12], where no constraining medium was provided, showed even slower decay times of almost 100 ns.

The most interesting feature of Fig. 1.8 is that the use of liquid water glass produces a stress pulse amplitude that is only 10% lower than that obtained by its solid counterparts. Interestingly, the rise time of 1.3 ns is lower compared to that of 3.0 ns obtained through the use of fused quartz. The same is true for the post-peak decay profile. In fact, compared to the fused quartz, both the rise and fall times are much sharper even for water (1.5 ns rise time) and for propanol (1.9 ns), albeit at lower amplitudes of 1.88 GPa for water and 1.2 GPa for propanol. This presumably occurs due to a better interface, free of air bubbles.

The process of stress pulse generation with liquids as constraining media involves reaching the nucleation condition through a non-equilibrium, super-heating of the liquid. For liquid water glass, both the amplitude and rise time of the pulse are almost the same as for its solid counterpart, because at very high strain rates, the liquid provides all the inertia and acts exactly like a solid. Therefore, for time intervals shorter than the laser pulse rise time, the liquid water glass acts like a solid with similar thermo-mechanical properties, thus leading to a similar rise time. Although a more quantitative understanding is required to select the most efficient liquid, nevertheless, the selection of various liquids based on a physical understanding given here indicates that a 0.5 μm thick layer of solid water glass provides the desirable profile for the modified laser spallation experiment.

1.4.3 Influence of the Thickness of the Laser Absorbing Film

The thickness of the laser-absorbing film also influences the characteristics of the stress pulse. Since the Al film, when constrained by a 5 μm layer of solid water glass, provides the optimal pulse profile, experiments were carried out with Al film thicknesses of 0.3 μm , 0.5 μm , and 1.0 μm ; all constrained by a 5 μm thick layer of solid water glass. Figure 1.9 shows the profiles of the stress pulses for different Al thicknesses, all obtained at a laser fluence of $8.7 \times 10^4 \text{ J/m}^2$. No trend, either for the rise time or for the amplitude of the stress pulse, was observed with the coating thickness. The highest amplitude (3.0 GPa) and lowest rise time (1.1 ns) were obtained with a 0.5 μm thick Al film. The

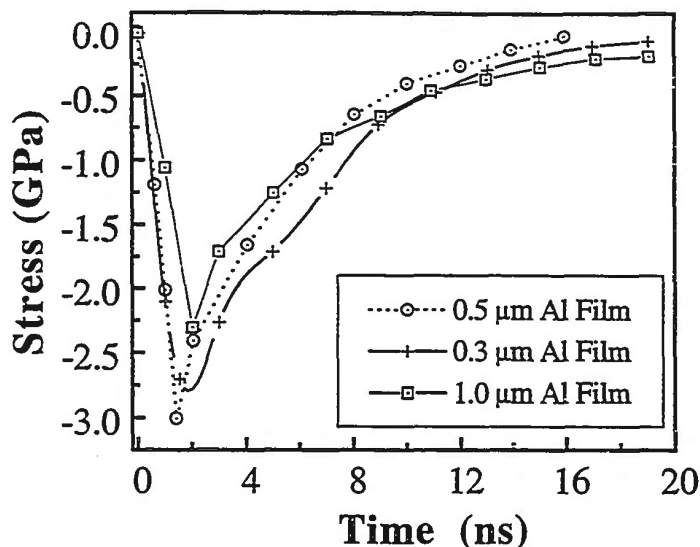


Figure 1.9: Influence of the thickness of the laser-absorbing film on the stress pulse profile. (Constraining medium: $5\text{ }\mu\text{m}$ thick layer of solid water glass; laser fluence $= 8.7 \times 10^4 \text{ J/m}^2$).

rise time/maximum amplitude pair of 2.2 ns , 2.3 GPa , was obtained with a $1.0\text{ }\mu\text{m}$ thick film, while the pair 1.6 ns , 2.7 GPa , resulted with a $0.3\text{ }\mu\text{m}$ thick film. The lower amplitudes at thicknesses less than $0.5\text{ }\mu\text{m}$ are due to a reduction in the total laser energy absorbed by the Al film. Clearly the penetration depth of the Nd:YAG laser wavelength of $1.06\text{ }\mu\text{m}$ should define the smallest thickness of the film for maximum pulse amplitudes. In conclusion, a $0.5\text{ }\mu\text{m}$ thick Al film confined by a $5\text{ }\mu\text{m}$ thick layer of solid water glass is used for the modified laser spallation experiment.

1.4.4 Influence of the Thickness of the Constraining Material

We also explored the optimal thickness for the solid water glass layer in order to maximize the pulse characteristics. A $0.5\text{ }\mu\text{m}$ thick Al film was used as the laser-absorbing material. Our experiments showed that the stress pulse does not change when the water glass thickness changed from $3\text{ }\mu\text{m}$ to $20\text{ }\mu\text{m}$, which were the thicknesses typically used in the experiment. Within this range in thickness, the solid water glass layer and the molten Al film are completely removed after the generation of the stress pulse. As shown by Gupta *et al.* [3], in the laser spallation setup, one compressive pulse is produced in each the substrate and the constraining medium. It is the reflection of the compressive pulse into a tension pulse from the free surface of the solid water glass that leads to its removal.

As explained in detail elsewhere [11], the removal of the Al and solid water glass occurs within 1.3 ns and all of the heat trapped within these layers is completely detached from the substrate. Consequently, the stress pulse within the substrate is free of any thermal tail and leads to a hitherto unreported stress pulse profile with 1.1 ns rise time, and with no post-peak decay tail. As shown in Fig. 1.8, the stress becomes close to zero at 17 ns, whereas for other constraining media, the tail of the pulse assumes an asymptotic behavior. This effect is analogous to the mechanism of stress pulse generation through an impact of a thin flyer plate in the plate impact experiment.

It was of interest to define the characteristics of the stress pulses in cases where the solid water glass layer remains intact. Additional experiments were performed with 5 μm and 1 mm thick solid water glass layers. Solid water glass 1 mm thick remained intact against the substrate upon microexplosion of the Al film. The results are presented in Figs. 1.10(a) and 1.10(b). The 1 mm layer produces a 30% higher amplitude than that produced by a 5 μm layer at low laser fluence levels, and much lower (60%) amplitudes at a higher laser fluence. The rise time of 1.2 ns with a 5 μm layer is much shorter in comparison with 2.0 ns for the 1 mm thick layer. The results indicate that different laser material interactions must occur in cases involving removed and intact constraining material. However, the simulation of these processes is outside the scope of this thesis work.

1.4.5 Influence of the Laser Fluence and Substrate Material

Within the range of laser fluences used in the experiment, we have found that a higher fluence results in an increased stress pulse amplitude. However, the effect of the laser fluence on the shape of the stress pulse is also important. For a given substrate, if the profile of the stress pulse remains similar (they can have different amplitudes) for different laser fluences, then one stress pulse profile could be used as an input into the computer program to calculate the transfer coefficients, and this can be used throughout the entire experiment. The transfer coefficient is defined as the ratio of the transient interface stress amplitude to the amplitude of the loading stress pulse. At the conditions when the stress pulse profile is insensitive to the amplitude of loading, the transfer coefficient is independent of the amplitude of loading as well, and it could be used to calculate the interface stress amplitude based only on the amplitude of the stress pulse. Otherwise, the computer program has to be run for every stress pulse shape, which can be time-consuming. Figure 1.11 presents the results for such experiments performed with 0.5 μm Al film and 5 μm solid water glass at increasing laser fluences. It shows that as the

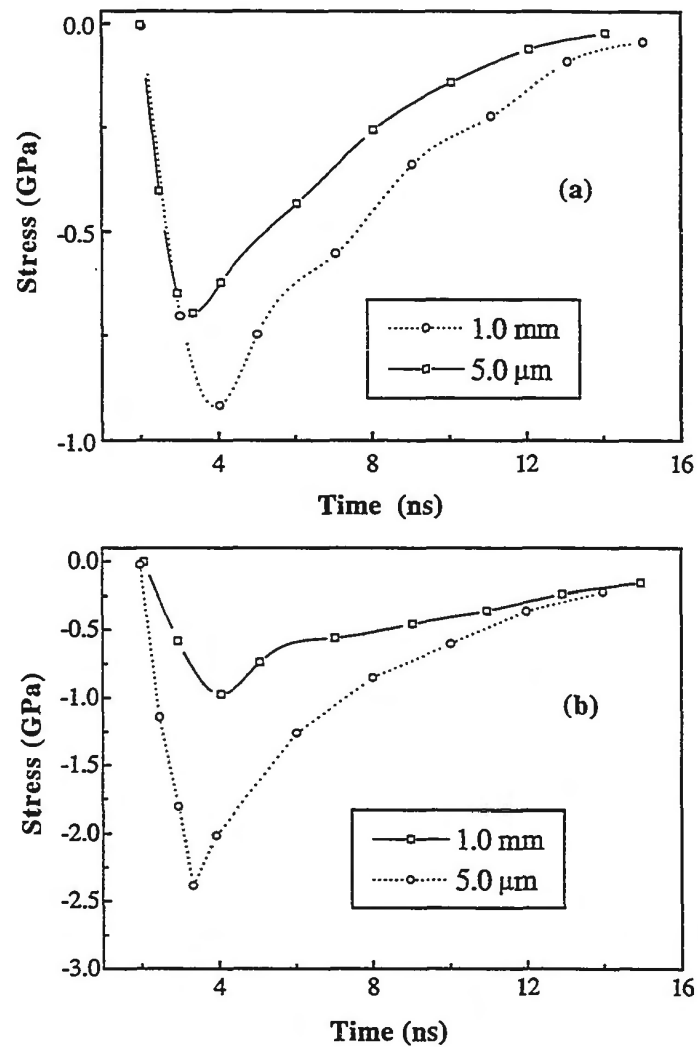


Figure 1.10: Influence of the thickness of the constraining material on the stress pulse profile. All experiments were performed with a 0.5 μm thick laser absorbing Al film. The laser fluence used to generate the stress profile in (a) was $1.1 \times 10^4 \text{ J/m}^2$ and for those in (b) was $4.2 \times 10^4 \text{ J/m}^2$.

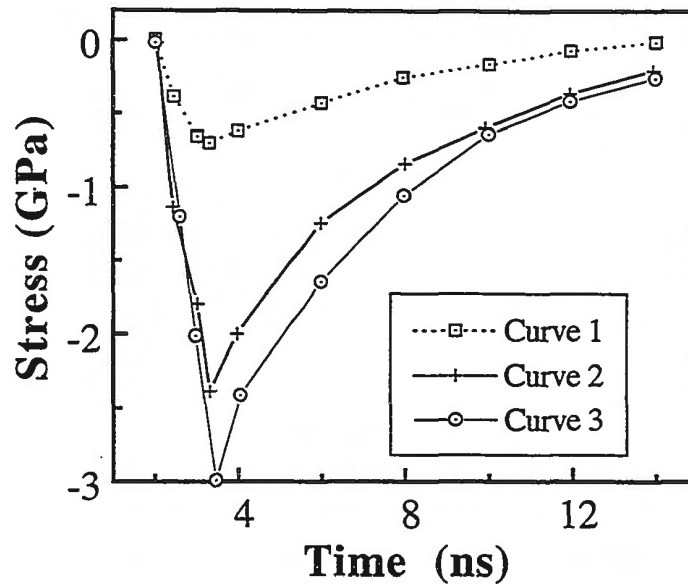


Figure 1.11: Influence of laser fluence on the stress pulse profile. Curve 1: $1.1 \times 10^4 \text{ J/m}^2$; Curve 2: $4.2 \times 10^4 \text{ J/m}^2$; Curve 3: $8.7 \times 10^4 \text{ J/m}^2$. All pulses were generated by using an Al film and a $5 \mu\text{m}$ thick layer of solid water glass.

fluence level is increased, the profile of the stress pulse is approximately retained, albeit with a different amplitude. Therefore, it is feasible to resort to the computer code only once for each substrate/coating system. The similarity in shape of the stress pulses implies the same mechanism for generation of the stress pulse within the fluence levels considered here. However, it should be noted here that the reproducibility of the stress pulse requires the careful control of all important parameters such as the thicknesses of the absorbing film and the the constraining material, the reflection of the film and so on. Due to the difficulty of controlling these parameters it was preferable to measure the free surface velocity at each measurement.

Figure 1.12 shows plots of the maximum compressive stress *vs.* the laser fluence for several different experimental conditions. Based on the results presented so far, it is apparent that such curves depend on the kind of substrate material, the kind and thickness of the energy absorbing material, and its constraining material. The amplitude of the stress pulse varies in different substrates even at the same laser fluence. As expected the profile of the stress pulse is also influenced significantly by a change of the substrate material. One such example is given in Fig. 1.13(a), which shows that the stress pulse generated in silicon substrate is much stronger (0.71 GPa) and sharper (1.2 ns rise time) than that in a pyrolytic graphite substrate with 0.05 GPa amplitude and 4.5 ns rise time.

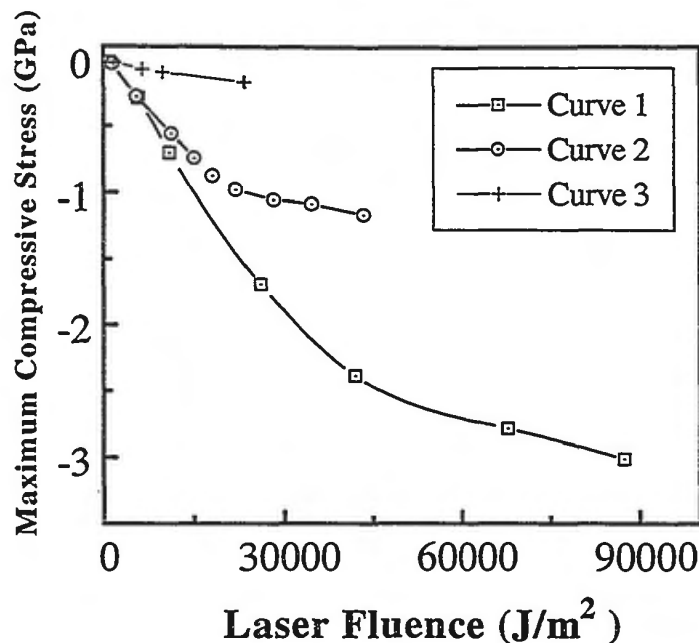


Figure 1.12: Plots of the maximum compressive stress *vs.* the laser fluence for several different experimental conditions. Curve 1: substrate: Si, energy absorbing medium: $0.5\ \mu\text{m}$ thick Al film, constraining material: $5\ \mu\text{m}$ thick solid water glass; Curve 2: substrate: polycrystalline Al_2O_3 , energy absorbing medium: $0.5\ \mu\text{m}$ thick Al film, constraining material: a fused quartz disc; Curve 3: substrate: pyrolytic graphite, energy absorbing medium: $0.5\ \mu\text{m}$ thick Al film, constraining material: $5\ \mu\text{m}$ thick solid water glass.

Even the post-peak decay is sharper for Si due to its higher thermal diffusivity. It will be of interest to see if a universal stress pulse shape can be obtained in normalized coordinates so that only one transfer function chart can suffice for all substrates in the laser spallation experiment. Normalizing the amplitude by using its maximum amplitude, and the time by using the rise-time, normalized profiles were obtained for different substrates. Unfortunately as shown in Fig. 1.13(b) the normalized shapes are not similar. Therefore the transfer coefficient charts which were used to determine the interface stress in first strategy (Yuan [20], Section 1.3) are associated with a given stress pulse profile and can be used only for a given system. The results presented in this section indicate that an experimental setup which consists of a Al film of $0.5\ \mu\text{m}$ as the energy-absorbing film and a layer of solid water glass several micrometers thick when used as the constraining material in the modified laser spallation experiment, gives the highest and the sharpest stress pulse. This has the best potential for spalling thin film interfaces. This setup is an improvement over an earlier version described in Gupta *et al.* [3], where a $1.0\ \mu\text{m}$ thick gold film, confined by a $2\ \text{mm}$ thick fused quartz was used.

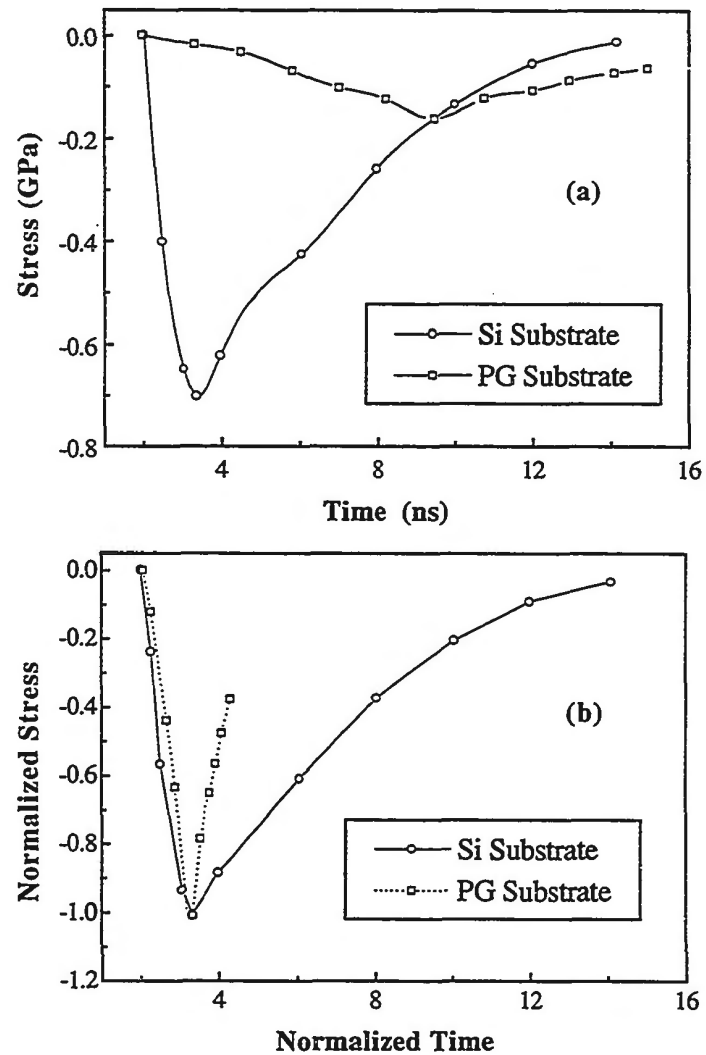


Figure 1.13: Influence of the substrate material on the stress pulse profile. (a) Actual stress pulses. (b) Normalized stress pulses.

In addition to the higher and sharper stress pulse profiles, there are several advantages of this setup over the old version. First, the contact between the energy-absorbing film and constraining medium is improved dramatically. In cases where a fused quartz is used as the constraining medium, even a very flat surface can lead to trapping of air gaps between the film/quartz interface due to small local roughness. The air gap reduces the amplitude of the stress pulse and results in a longer post-peak decay time. In addition, after the first shot, the thermal expansion and ablation of the energy-absorbing film and local microcracking of the fused quartz, causes additional air gaps near the ablated area. The replicability of the results in the modified setup is improved considerably due to the coherent, air bubble free interface. In addition to the elimination of air gaps, the experiment shows that the stress pulse is independent of the thickness of the constraining solid water glass provided the thickness is between $3\ \mu\text{m}$ and $20\ \mu\text{m}$. This modification makes the experiment much easier to handle.

For a given substrate, the stress pulse rise time is controlled predominantly by the rise time of the Nd:YAG laser pulse. In all of the experiments reported here, as expected, the stress pulse rise-time is relatively less sensitive to the experimental variables, remaining within $1\ \text{ns}$ to $3\ \text{ns}$. In contrast, the decay time or the fall time of the stress pulse is a strong function of the experimental variables. Fortunately, the interface tensile stress is far more sensitive to the decay time of the stress pulse than it is to the pulse's rise-time.

In the case of liquid constraining layers such as water and liquid water glass, the presence of both the constraining layer and evaporating liquid are demonstrated to be the cause of normal forces. Since such phenomena are difficult to simulate, the modified version has a big advantage over the old one, since it does not need to consider the generation mechanisms of the stress pulse by the pulsed laser. It is this feature that makes it possible to optimize the laser spallation experimentally.

Finally, modern acoustic applications require special ultrasonic sources to generate strong and short stress pulses for applications in interface strength measurements, measurement of elastic constants of materials through ultrasound, and non-destructive detection of sub-micron flaws. In the present experiment, it is demonstrated that narrower and stronger stress pulses can be produced by applying only a thin layer of water glass. Since water glass can be used simply on any kind of solid surface, it is feasible to use this method for *in situ* detection of flaws in the manufacturing industry. Without doubt, the results presented here should be useful in developing future laser ultrasonic sources.

The results of this section can be summarized as follows.

- Among the Al, Au and Al+Cr films, the Al film is most efficient for producing the strongest and the shortest rise-time stress pulses. Furthermore, a film thickness of $0.5\ \mu\text{m}$ maximizes the stress. Of the constraining materials, a layer of solid water glass of $5\ \mu\text{m}$ is demonstrated to be the best choice. Therefore, an experimental setup consisting of a $0.5\ \mu\text{m}$ thick Al energy-absorbing film, and a layer of solid water glass $5\ \mu\text{m}$ in thickness is chosen for the modified laser spallation technique.
- The optimized experimental setup has advantages over the old version with respect to having a coherent, air bubble free contact, resulting in a high repeatability of spallation results.
- The shape of the stress pulse for a given substrate is independent of the laser fluence.
- The shape of the stress pulse depends strongly on the kind of the substrate material.
- In the modified laser spallation experiment, the dominant mechanism for generating the stress pulse is through a plasma-induced momentum transfer process.

1.5 Nanosecond Rise-Time Stress Pulses with no Asymptotic Decay

In this section, we discuss the mechanics of generating transient stress pulses with no asymptotic post-peak decay. Apart from demonstrating a short rise time of $1.1\ \text{ns}$, and an amplitude in excess of $3\ \text{GPa}$, this pulse shows a remarkably sharp, post-peak decay resulting in zero stress at about $17\ \text{ns}$. This observation contrasts with all other reported cases of laser-produced stress pulses [3, 12, 23, 25], which show a gradual post-peak decay resulting in a long tail, assuming an asymptotic value at about 5% to 15% of the peak amplitude. As explained initially by Vossen *et al.* [12], and more quantitatively by Gupta *et al.* [10], the long tail results from the long penetration depth of the thermal pulse in the substrate. The use of a confining medium with higher thermal diffusivity than that of air helps decrease the penetration depth by providing an energy sink away from the direction of the substrate. Therefore, when air is used as a constraining medium for the laser-absorbing film, a long gradual tail reaching an asymptotic value of about 10% of the peak amplitude was observed at $100\ \text{ns}$ by Vossen *et al.* [12]. Using the old version discussed in Section 1.3, the stress pulse was produced by focusing a $2.5\ \text{ns}$ long Nd:YAG laser pulse onto a $1.0\ \mu\text{m}$ thick, laser-absorbing gold film, sandwiched

between a piezoelectric quartz substrate and a fused quartz plate. Both quartz plates were 25.4 mm in diameter and 2 mm in thickness. The stress pulses obtained by recording the piezoelectric current signal showed a sharper decay resulting in an asymptotic value of 15% of the peak stress at about 45 ns.

The new procedure involves [14] producing stress pulses by using a 0.5 μm thick Al laser-absorbing film sandwiched between a 5 μm thick layer of solid water glass, and an Si substrate (used here as an example) as described in Section 1.3. The stress pulse profiles were obtained by monitoring the transient free surface velocity of the Si surface by using a displacement interferometer. Figure 1.9 shows the stress pulse profile obtained for a laser fluence of $8.67 \times 10^4 \text{ J/m}^2$, measured at the Nd:YAG source. Apart from an amplitude increase, and a pulse rise-time of 1.1 ns, this profile displays a sharper post-peak decay with the stress finally decaying to zero in 17 ns. Interestingly, the layer of solid water glass spalls off from the Si surface along with the 0.5 μm thick Al film during the microexplosion of the laser-absorbing Al film.

Although a quantitative proof of the mechanism leading to such a stress profile is not attempted, a phenomenological model presented in Gupta *et al.* [14] provided a possible mechanism. As shown by Gupta *et al.* [3], in the laser spallation setup, one compressive pulse is produced in both the substrate and the constraining medium. It is the reflection of the compressive pulse into a tension pulse from the free surface of the solid water glass that leads to the removal of the Al film from the Si/Al interface. For a 5 μm thick layer of solid water glass and an Al film of 0.5 μm thickness, the tension pulse reaches the Al/Si interface in about 1.3 ns (assuming a longitudinal wave velocity of 8380 m/s in SiO_2 and 6400 m/s in Al). Since the laser heating pulse is 3 ns long, such a spallation process within 1.3 ns removes the heat source, resulting in the absence of a thermal tail in the generated stress pulse. Using the computer simulation developed in Gupta *et al.* [3], the spatial temperature distribution within the first 1.3 ns was determined in the vicinity of the Al/ SiO_2 , and of the Al/Si interface. The maximum temperature at the Al/Si interface was measured to be only 700 K, which decays to the ambient temperature within 0.5 μm of the Si substrate. Although such a temperature gradient and amplitude remain within the Si upon the removal of the heat source entrapped within the Al film, these are insufficient to produce any comparable stresses. Therefore, the decay tail of the stress pulse shown in Fig. 1.9 is a mechanical tail produced by the sudden impact of the Si substrate in the first 1.3 ns. In other words, the Si surface sees only a transient pressure loading in the first 1.3 ns, resulting from a pressure pulse in the Al film, produced through the laser-Al interaction and transmitted through the Al/Si interface. Figure 1.14 shows the stress history at the Al/Si interface in the first 1.4 ns as obtained from the simulation

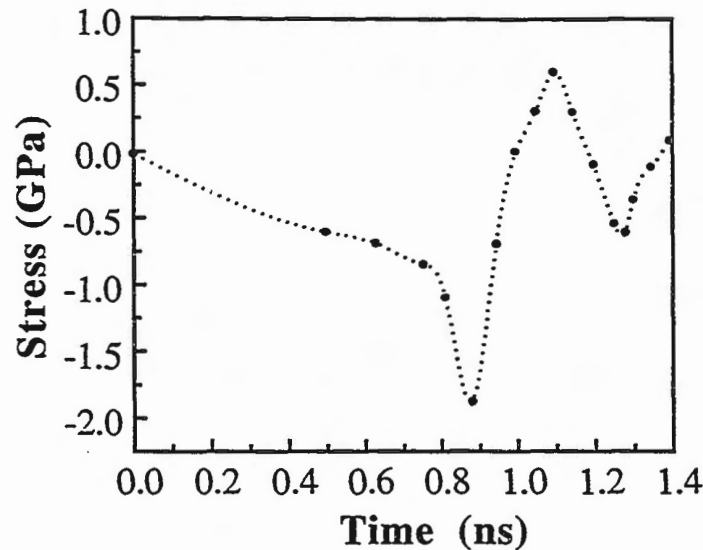


Figure 1.14: The Al/Si stress history at 1.4 ns as determined by the simulation given in Gupta *et al.* [3]

presented in Gupta *et al.* [3]. Remarkably, the first sizable tension at the Al/Si interface occurs at 1.3 ns. The chopped stress pulse now acts as a new boundary condition for the Si surface. Figure 1.8(b) shows stress pulse profiles obtained with the same setup but with different constraining media, including a 25 mm thick liquid column of water and liquid water glass. In all these cases, a typical asymptotic thermal tail at about 5% to 20% of the peak stress can be seen. No spallation of the Al film was observed in any of these setups. Interestingly, the chopping of the stress pulse tail within the Si is analogous to the mechanism of stress pulse generation through an impact of a thin flyer plate in the plate impact experiment.

1.6 Optical Interferometer for Non-Specular Surfaces

The stress pulse profile and amplitude are usually determined by measuring the free surface velocity of the substrate or thin film produced upon reflection of a stress pulse, by using a laser velocity or displacement interferometer. A good review of the various types of interferometers is given by Barker [18]. A major constraint with most interferometers is the availability of a specular moving surface, which preserves the geometric coherency of the reflected laser beam, and leads to interference fringes proportional to either the

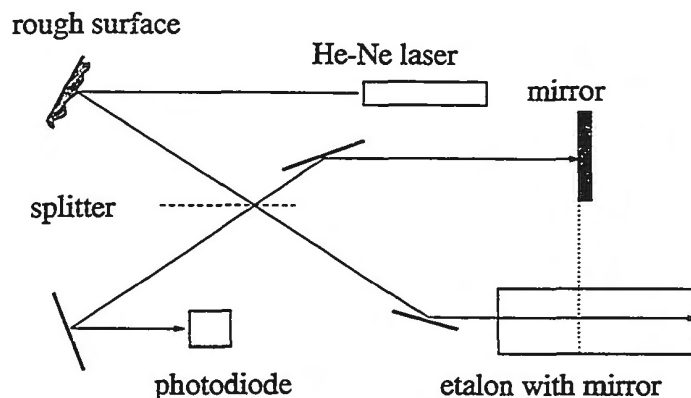


Figure 1.15: WAMI interferometer. The key part of this interferometer is the dielectric etalon. Both legs of the interferometer have the same apparent distance from the splitter to the apparent positions of the mirrors.

displacement or the velocity of the moving surface [29].

Although the incoherent reflected beam, produced by a diffuse or scattering surface, can be used to produce interference by placing the two mirrors in the Michelson interferometer equidistant from the photodiode, no velocity-related fringes will be produced due to a zero time delay (τ). This deficiency was removed by the design of a Wide-Angle Michelson Interferometer (WAMI) [30], shown in Fig. 1.15, capable of producing velocity-related fringes even when illuminated by an incoherent beam. This interferometer is actually a velocity interferometer. If the apparent mirror position is placed at the same distance from the beam splitter as the mirror on the other leg of the interferometer, geometrical coherence of the light beam is not required, and very good fringe contrast can be obtained. The same apparent distance allows to get interference from spatially non-coherent light, whereas the difference in the actual optical lengths allows the interference fringes to be related to the surface velocity. In this setup, an etalon is used to produce the virtual image of one of the mirrors. An alternative setup was used by Amery [31] to replace the etalon by a combination of two convex lens separated by the sum of their focal lengths. This setup is used to produce the virtual image, albeit with the same principle as the WAMI interferometer. The above setups allow one to study the properties of materials with non-uniform structure, including composites.

In our investigation, we measure the free surface velocities of diffuse surfaces by an interferometer utilizing the same principle as that of Amery [31] but with a considerably less complicated experimental setup, and still producing comparable quality fringe contrast. Furthermore, we show that the setup is really a variable displacement-velocity

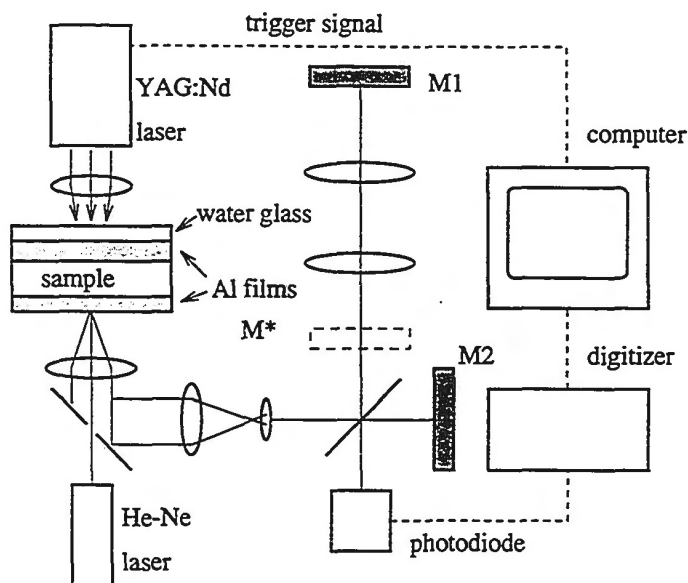


Figure 1.16: An optical interferometer. Both legs of the interferometer have the same apparent distance from the splitter to the positions of the mirrors. The setup to the left of the free surface is used to produce stress wave.

interferometer, depending upon the duration of the velocity signal in comparison to the delay time (τ) of the interferometer. Substrates of carbon-carbon composites and polycrystalline alumina with non-uniform microstructure on the scale of 5 to 10 μm , were considered. In addition, experiments were performed on Si crystal wafers with specular surfaces, with measured velocities of up to 800 m/s .

1.6.1 Optical Setup

Figure 1.16 shows the optical setup of the interferometer used to measure the free-surface velocities of both the diffuse and specular surfaces. The assembly on the top of the free surface of the sample is used to produce the stress pulse by a the Nd:YAG laser pulse. The stress pulse upon reflection from the free surface of the substrate subjects the free surface to a transient velocity. The beam from a frequency-stabilized He-Ne laser is brought to focus on the specimen surface by a convex lens. Light reflected from the diffuse surface is collected by the same lens and directed to a telescope, which condenses and collimates the beam. Next, a beam splitter directs the light to the two legs of the interferometer, and the returning beams are recombined at the Newport 877 photodiode with 200 ps rise time to produce the interference fringes. The fringes are recorded on a Tektronix SCD 1000 digitizer with a single-shot rise time of 5 ps . As discussed by Amery,

the key point of this optical setup is the second leg of the interferometer, which consists of two lenses (with focal length (f) equal to 50 *cm* each) and one mirror. It is possible to show [31] that the system of two lenses, which are separated by the sum of their focal lengths, images the mirror $M1$ onto the apparent position M^* (Fig. 1.16). In other words, it is possible to show that the laser beam which lost its spatial coherence upon reflection from the diffuse surface still can interfere with itself. A system of two lenses directs the mutually non-coherent parts of the beam from the first leg of the interferometer so that they fit exactly the same parts of the second beam (Fig. 1.17). As a result, each part of the geometrically non-coherent light interferes with itself and the loss of spatial coherence does not matter.

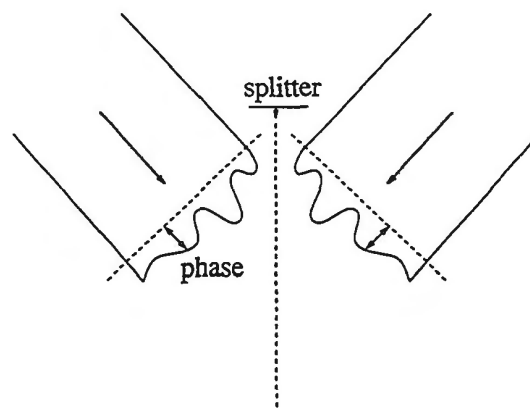


Figure 1.17: Interference of spatial non-coherent laser beam using the setup shown in Fig. 1.16.

The actual difference in optical path between the two optical legs of the interferometer is equal to $8f/c$ (which is about 13.3 *ns* in our case), and this difference determines the phase delay of the interferometer.

1.6.2 Interpretation of the Setup as a Displacement *vs.* Velocity Interferometer

To check the optical setup, we produced stress pulses in 25.5 *mm* diameter and 1 *mm* thick Si wafers with a Nd:YAG laser pulse in a manner discussed in previous sections. The (100) surface of the wafer provides a specular surface. The fringe record obtained directly is shown in Fig. 1.18(a) and that after removing the effect of the photodiode rise time in Fig. 1.18(b), and the velocity profile associated with it is given in Fig. 1.18(c). Figure 1.18(b) shows two similar sets of the interference fringes. The time difference

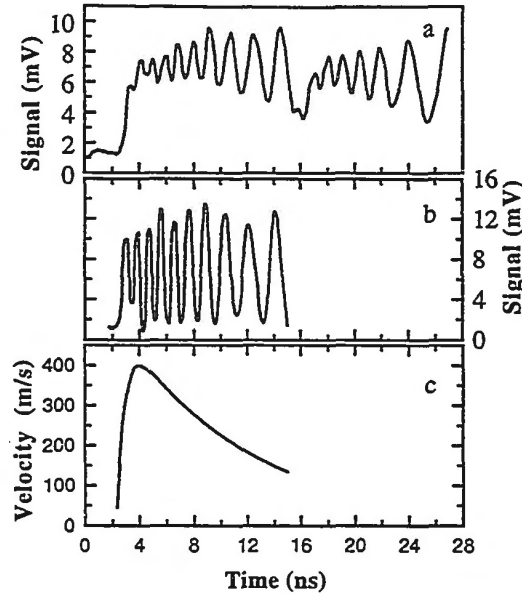


Figure 1.18: (a) Interference fringes obtained from a specular silicon surface. (b) The corrected fringe record without the effect of the photodiode rise time. (c) Velocity profile corresponding to the fringe record of (a).

between these sets is equal to $8f/c$, which in our experiment is 13.3 ns . Here f is the focal length of the lenses, and c is the velocity of light.

The signal of an ideal photo diode in a velocity interferometer is:

$$y(t) = A + B \cos [(u(t) - u(t - \tau))(4\pi/\lambda) + \varphi_0], \quad (1.11)$$

where $u(t)$ is the displacement of the moving surface, φ_0 is initial phase, λ is the laser wavelength, and A and B are constants. Before the arrival of the signal from the longer leg of the interferometer, $u(t - \tau)$ is constant, and the velocity interferometer within this period can be considered as a displacement interferometer. The time delay τ , which in our case is about 13.3 ns , is sufficient to get the information about the amplitude and the shape of the stress pulse. Clearly the fringes recorded after 13.3 ns in Fig. 1.18(a) provide the velocity information with the displacements averaged in some sense over 13.3 ns . The velocity profile of Fig. 1.18(c) was obtained from the displacement fringes obtained within the first 13.3 ns .

If the velocity rise-time is much longer than the delay length of the interferometer, then the fringes obtained can be treated as velocity fringes and related to the velocity $V(t)$ via Eq. 1.11. A more detailed discussion pertaining to this is given by Clifton [29]. In this case, the term $u(t) - u(t - \tau)$ in Eq. 1.11 can be replaced by $\tau V(t)$ to relate

the fringe frequency to the velocity. Therefore, for a given velocity profile the delay length can be changed to transform the interferometer from a velocity to a displacement one, and vice versa. Therefore, in one setup it is possible to combine the advantages of both a velocity and a displacement interferometer, that is, the high resolution of the displacement interferometer and the stability of the velocity interferometer.

The shape of the real fringe record as shown in Fig. 1.18(a) is different from that of the ideal signal, as given by Eq. 1.11. The signal of Fig. 1.18(a) shows the influence of the photodiode rise-time, as the measured velocity profile is close to the rise time of the photodiode. After removing the effect of the photodiode rise time, the fringes are reproduced in Fig. 1.18(b). The peak velocity corresponding to the fringe record is about 400 *m/sec*. The signal-to-noise ratio is determined by two factors. First, the power of the probe laser must be high enough compared to the electronic noise level. Second, at a given laser power, measured velocity can not be too high: there is a decrease of the signal-to-noise ratio with increasing measured velocity. Interestingly for the Si case, only a 5 *mW* laser and velocities as high as 800 *m/s* produce the high contrasting fringes of Fig. 1.18(a).

1.6.3 Experiments on Substrates with Diffuse Surfaces

The problem of insufficient He-Ne laser power becomes more important for diffuse surfaces. As a sample with a diffuse surface, we chose slightly polished alumina substrates. This polishing was not necessary for obtaining a good interference, but some degree of polishing was required to increase the total energy reflected from the sample. The roughness features of the polished alumina surface are within 5 to 10 μm , which lead to the scattering of the He-Ne laser beam upon reflection. In this case the power of the reflected beam is closer to the noise level of the photodiode. Figures 1.19(a) and (b), respectively, show the fringe record and the associated velocity profile from an alumina surface. The imperfect shape of these fringes is due to the influence of the noise. The peak velocity is equal to 150 *m/sec* with a rise time of 3 *ns*. As already discussed, this optical setup works as a displacement interferometer within the first 13.3 *ns*. At lower velocities this setup acts as the usual velocity interferometer.

Finally we consider an application involving two-dimensional, laminated, carbon-carbon composites. The stress wave is produced normal to the plane of the woven fabric. The aim was to measure the elastic constants by measuring the time delay between the generation of the Nd:YAG laser pulse and the start of the motion of the rear surface for a given laminate thickness. Carbon-carbon composites are difficult to polish to perfect

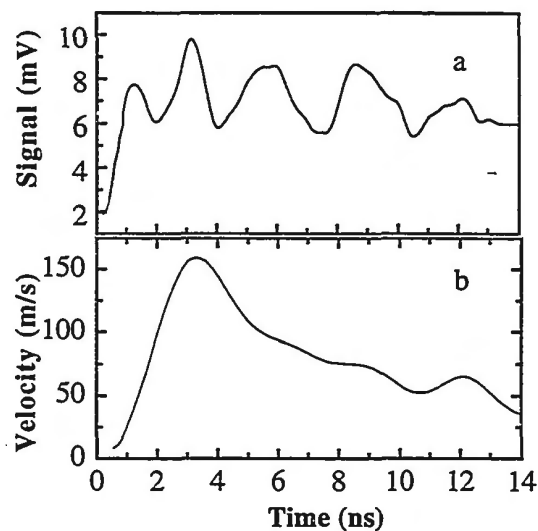


Figure 1.19: (a) Interference fringes from a non-specular polycrystalline alumina surface. (b) The velocity profile corresponding to (a).

optical quality and Fig. 4.1 (Section 4.4) shows a micrograph of the woven fabric with individual fibers. Figure 1.20 shows the fringe record for an experiment on carbon-carbon composites. In this experiment, the optical setup works as a velocity interferometer, as the delay time is much less than the characteristic time for the fringes. Although the stress wave characteristics at the front face of the sample are the same as those obtained from the Si free surface, a considerable dispersion of the wave occurs during its propagation within the 2 mm thickness of the composite plate. This is because the characteristic microstructural dimension (*e.g.*, the distance between the fabric planes) is almost 10 times the length of the stress pulse of about 54 μm .

1.6.4 Discussion of the Interferometer

We have discussed an optical setup which can be considered simultaneously as a displacement and a velocity interferometer depending on the duration of the velocity profile compared to the delay length of the interferometer. The setup combines the advantages of both types. It works as a displacement interferometer within the first period, $\tau = 8f/c$ and as a velocity interferometer with the same time delay τ . As discussed by Amery [31], there is no requirement for the spatial coherence of the reflected beam in this design. Using this optical setup, we measured the velocity profiles on rough alumina and carbon-carbon composite surfaces. In addition velocities as high as 400 m/sec were recorded on the specular Si surface.

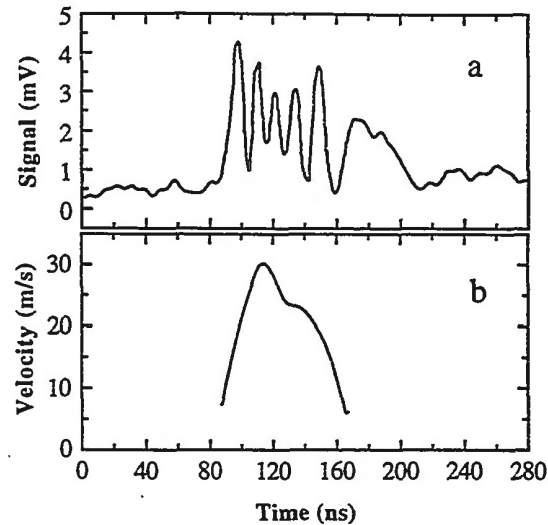


Figure 1.20: (a) Fringe record obtained from the rear surface of a carbon-carbon composite sample. (b) Velocity corresponds to the fringe record of (a).

Finally we discuss the issue of the determination of the point of velocity reversal from the fringe record of the displacement interferometer. As discussed earlier, this was achieved by Amery [31] by using differently polarized beams in the interferometer and by recording the fringe record on a series of photo-multiplier tubes. However, we determined this point through a careful visual inspection of the fringe record. In order 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, Fig. 1.21(b). Next, we used a linear velocity interferometer [18] 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 in the linear velocity-meter, the record shown in Fig. 1.21(a) corresponds to the velocity profile. It shows that the velocity of the surface increases first and reaches a peak value, followed by a gradual decline to zero. Since both curves in Fig. 1.21 peak at the same time, we consider our interpretation of the displacement fringe record to be correct.

A frequency stabilized He-Ne laser was used to obtain a clean fringe record in the Doppler setup. The ordinary laser, operating in the single transverse TEM-00 mode, usually has several active longitudinal modes with different frequencies. These longitudinal modes interfere to produce an output laser beam with the amplitude modulated by the beat frequency. The lowest beat frequency in a laser is given by $c/2L$, where c is the light velocity and L is the cavity length of the laser. Unfortunately, the beat frequency of

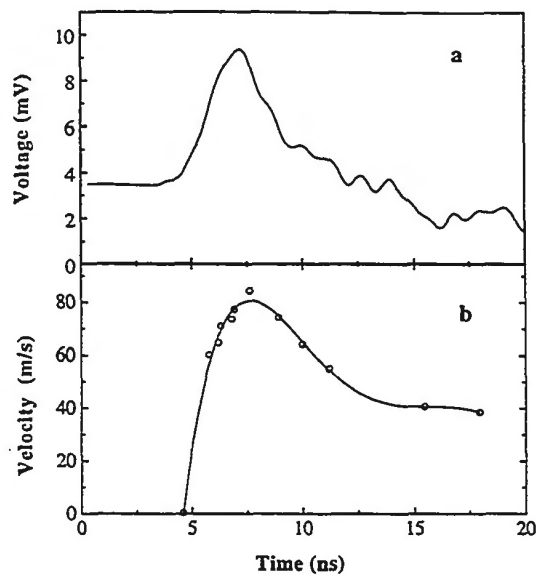


Figure 1.21: Similarity of the velocity profiles (b) as obtained through the displacement fringes, and (a) as obtained by a linear velocity interferometer.

the ordinary He-Ne laser (for example, 320 MHz) is within the range of the displacement fringe frequency. However, for recording slower and longer rise-time profiles, an ordinary He-Ne laser should suffice.

Chapter 2

Measurement of the Interface Fracture Toughness using the Laser Spallation Experiment

Since in the modified laser spallation experiment the interface is loaded at a strain rate of almost 10^7 sec^{-1} , the inelastic deformation that accompanies the decohesion process is largely avoided. The increase of the surface energy has to be close to the loss of the total energy of the stress pulse. Thus, the measured tensile strength data can be related to the interface fracture energy, albeit using a phenomenological stress-toughness relationship. To this end, it was shown by Gupta *et al.* [11] that the tensile strength obtained from the laser spallation experiment can be related to the interface fracture energies through a Griffith-type relationship, which in turn was derived by using the concept of universal bonding correlations. The relation of the peak strength to the intrinsic toughness takes either of following forms:

$$\sigma_{max}^2 = \frac{2E_0\gamma_{int}}{e^2h} \quad (2.1)$$

or

$$\frac{2\gamma_{int}}{e\sigma_{max}} = l. \quad (2.2)$$

Here σ_{max} is the peak strength measured by the spallation experiment, $2\gamma_{int}$ is the intrinsic toughness of the interface, e is the Napierian logarithm base, h is the average interatomic separation at the interface and l is a scaling parameter allowing universal form for the separation curve. Gupta *et al.* [11] showed that the interface toughness values obtained using the above equations were in good agreement (as shown in the Table 2-I) with experimentally obtained values, but only if interfaces were free of defects. However, for several metal/ceramic systems (Table 2-II), the measured strengths were far below the expected intrinsic strength. Clearly these values must be for flawed interfaces.

Table 2-I: Comparison of the interface toughness estimated from the spallation experiment with the values obtained from direct toughness measurements.

Material	Strength as measured by the spallation experiment, Pa	Intrinsic toughness value obtained from Eq. 2.1, J/m^2	Toughness obtained from independent experiment, J/m^2
C/SiC	6.9	3.0, [3]	3.3, [32]
Si/SiC	10.0	3.3, [3]	2.7, [32]
Silicon, cleavage	16	0.84, [3]	1.24, [33]

Table 2-II: Interface strength for some systems obtained by the laser spallation experiment.

Material	Sn/PG	Sb/PG	Cu/PG	Nb/Al ₂ O ₃	SnO ₂ /Al ₂ O ₃
Strength, MPa	18	5.9	15	41	280
reference	[13]	[13]	[13]	[13]	[34]

This suggests that the laser spallation experiment measures the actual strength of the interface which then approaches the intrinsic strength if the interface is free of flaws. This makes the measured value a suitable parameter for characterizing the interface. However the above values of toughness can be considered only as estimates.

To this end, we measured the fracture toughness directly by loading well-characterized interfacial flaws in the laser spallation assembly. Since the interface crack is loaded at a strain rate of approximately $10^7 sec^{-1}$, all inelastic deformation accompanying the crack initiation should be suppressed, thus yielding a fracture toughness value representative of the intrinsic interfacial energy. However, whether or not it is possible to completely ignore the plastic part of the decohesion energy at these strain rates is still an open question mostly due to the lack of the experimental data on dislocation motion at these conditions. In Section 2.1 a brief review of the data available in the literature on this subject is given.

Because of our prior experience, the Nb/Al₂O₃ interface system with different inter-layer (segregant) materials was used as a model system. The well-characterized cracks at the interface were implanted by using one of the two methods described in Section 2.2. After successfully implanting the crack at the interface, a compression pulse was gener-

ated in-line with the crack on the back surface of the sapphire disc by using the modified laser spallation assembly (Section 2.4). Using an interferometric technique, discussed in Section 1.6, we recorded the transient velocity of the coating free surface as the pulse interacted with the flaw and also with the free surface. The recorded critical velocity profile at which the crack initiated was related to the local dynamic J-Integral *via* a numerical program that simulated the dynamic initiation process of the artificially generated microcrack. This program was implemented by using the ABAQUSTM finite element package.

The overall experimental error in the laser spallation measurement of the interface stress stemming from the laser pulse variability, measurement of the ablated area, and recording of the fringes, *etc.* is estimated to be within 8%. All of the above strength data¹ were obtained as an average of 8 to 10 measurements, and the largest variation was limited to 10%.

2.1 Dynamic Deformation at High Strain Rates

Spallation experiments are characterized by the high strain rates which take place within very short time intervals. As mentioned in Chapter 1 the decay time and the amplitude of the stress pulse are the governing parameters in the spallation experiment. The decay time (Δt) is of the order of 17 ns. The stress pulse amplitude in the material can be determined by the free surface velocity. The characteristic amplitude of the velocity (v) in the spallation experiment is of the order of 50 m/s. The strain rate in the experiment can be estimated as

$$\dot{\epsilon} \simeq \frac{v}{c\Delta t}, \quad (2.3)$$

where c is the longitudinal wave velocity, which varies from 3000 to 7000 m/s for different materials. The above equation yields an estimate for the strain rate of the order of 10^6 s^{-1} . At the edges of flaws and microcracks, the stress concentration leads to the strain rate levels several orders of magnitudes higher. To perform any analysis, including the proposed numerical analysis of the dynamic fracture at the microcrack, a constitutive stress-strain relationship is required. However, at present, there is a substantial lack of experimental data of material properties at these strain rate levels. Most of the available data in the literature cover strain rate levels up to 10^5 s^{-1} .

The number of papers by Follansbee and Weertman [35–41] represent probably the

¹The raw experimental data obtained in this thesis work are available from archive of Prof. V. Gupta. The data are not included in Appendix due to the large volume of printing.

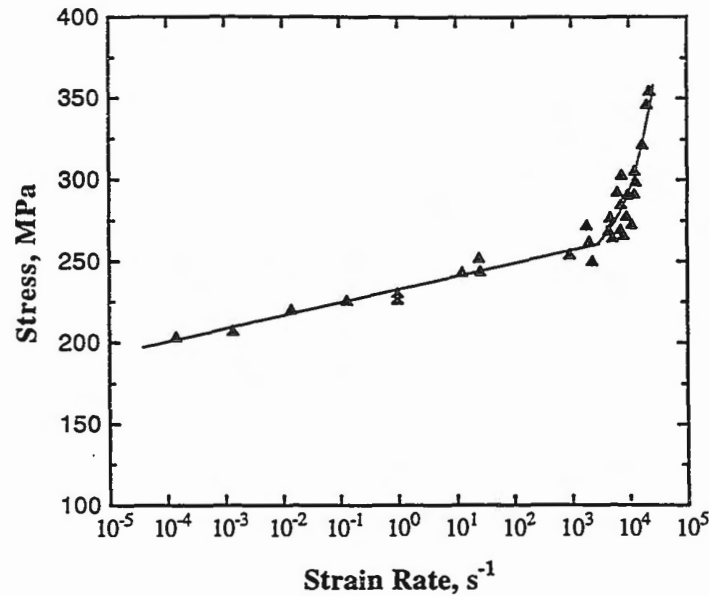


Figure 2.1: Strain-rate dependence of the flow stress of oxygen-free electronic copper at room temperature for a constant strain $\epsilon = 0.15$. Figure is taken from Regazzoni *et al.* [37].

most extensive review of the plastic deformation and dislocation dynamics at high strain rates. The short and incomplete review which follows below relies heavily on the material from these papers.

Many investigators of high strain rate deformation have shown that strain rate sensitivity in metals increases substantially at strain rate levels higher than 10^4 s^{-1} . At lower strain rates (and stresses), the yield stress sensitivity ($\partial \log \sigma / \partial \log \dot{\epsilon}$) is of the order of 0.01. Figure 2.1 (taken from [37]) illustrates the substantial increase in strain rate sensitivity obtained on pure polycrystalline copper at strain rates above 10^4 s^{-1} . Similar behavior was observed in other materials such as mild steel, tantalum [42], and aluminum single-crystals [43], *etc.*

Figure 2.1 shows that there are two regimes of dynamic plasticity and dislocation motion. At low stress levels the deformation is controlled by the thermal activation of dislocation motion, and general relationship between stress and strain rate at constant structure can be expressed in the following way [37]

$$\dot{\epsilon} = \dot{\epsilon}_0 \exp \left(-\frac{\Delta G}{kT} \right), \quad (2.4)$$

where $\dot{\epsilon}_0$ is the pre-exponential term, ΔG the activation energy, k the Boltzmann constant, and T the temperature. Frequently the free energy ΔG is expressed through the

phenomenological relationship [39, 44] as

$$\Delta G = g_0 \mu b^3 \left[1 - \left(\frac{\sigma}{\bar{M} \hat{\tau}} \right)^p \right]^q, \quad (2.5)$$

where σ is applied stress, $\hat{\tau}$ is the maximum dislocation glide resistance, $\bar{M}$ is the Taylor factor, g_0 is a normalized activation energy, μ is shear modulus, b is the Burgers vector, and p and q are the constants that characterize the obstacle profile.

The data on dynamic deformation obtained in impact experiments are available for the strain rates up to $10^5 s^{-1}$. For strain rates in the range between $10^3 - 10^5 s^{-1}$ (Fig. 2.1, [37]) Eq. 2.4 can no longer accurately represent the experimental data, and it is widely believed [35–41] that at this level of the stress the change in the deformation mechanism takes place. Dislocation motion becomes continuous, thermal activation of the trapped dislocations is no longer necessary for dislocation motion, and the strain rate (together with the dislocation velocity) is controlled by the dissipation mechanism such as energy dissipation by phonons, electrons, *etc.* This mechanism is sometimes referred to as drag-controlled dislocation mechanism.

The stress-strain rate relationship based on the experimental data (*e.g.* Fig. 2.1) in the drag-controlled regime at strain rates between $10^3 s^{-1}$ and $10^5 s^{-1}$ can be closely represented as [37]

$$\sigma = \sigma_b + \beta \dot{\epsilon}, \quad (2.6)$$

where σ_b and β are obtained experimentally. Strain rates in the spallation experiments are several orders of magnitude higher than the rates for which it is known that Eq. 2.6 is valid. Therefore to determine stress-strain rate relationship in our case we have to extrapolate this dependence on the basis of dislocation kinetics theory to the higher strain rate levels. Linear dependence of the flow stress on the strain rate has to have certain limitations at high strain rates, such as the following: the dislocation velocity cannot be higher than the Rayleigh speed for a given material (relativistic effect) and the shear stress cannot exceed the theoretical shear strength level. The well known equation relates the strain rate ($\dot{\epsilon}$) to the dislocation velocity (v) as [37]

$$\dot{\epsilon} = \rho_m b v / \bar{M}, \quad (2.7)$$

where b is the Burgers vector, ρ_m is the density of mobile dislocations, $\bar{M}$ is the Taylor factor. In case of the drag-controlled deformation and the ideal crystal (*e.g.* in the absence of obstacles) the velocity of the dislocation v can be determined by [37]

$$v = \sigma b / \bar{M} B, \quad (2.8)$$

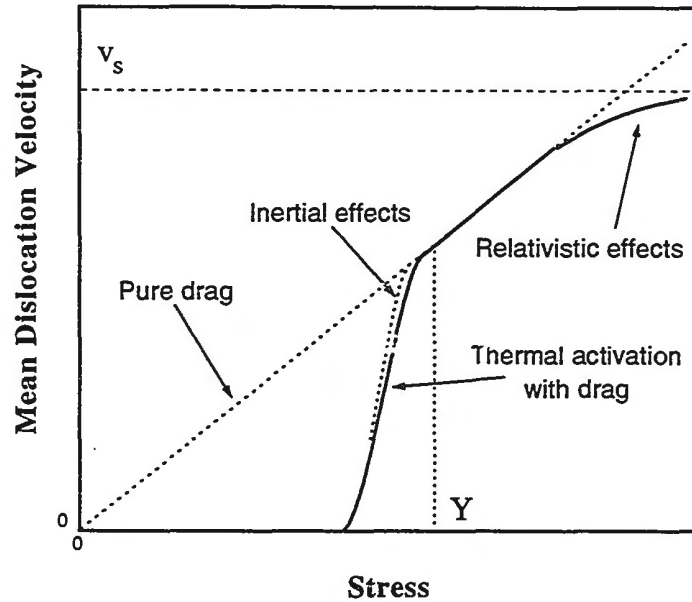


Figure 2.2: Typical deviations of the average dislocation velocity expected from inertial and relativistic effects. Figure is taken from Regazzoni *et al.* [37].

where B is the drag coefficient. As has been reviewed by Kocks *et al.* [44] the value of B for most metals lies in the range between 10^{-5} and $10^{-4} Pa \cdot s$.

Regazzoni *et al.* [37] presented a review of dislocation kinetics at high strain rates on the basis of the well known theory of dislocations. This model included superposition of drag-controlled and thermal activation-controlled deformation; description of the transition from one mechanism to the another, and a model of the drag-controlled dislocation motion in the presence of obstacles. Figure 2.2 illustrates qualitatively the dependence of the dislocation velocity on the applied stress, and the relativistic and inertial effects. Thermally activated kinetics of dislocation takes place at a stress level close to the threshold yield stress Y , and the yield stress is a weak function of the strain rate in this region. At higher stress levels, the strain rate dependence is close to the linear dependence. At higher stress the velocity reaches the relativistic limit of the Rayleigh speed. As was shown by Regazzoni *et al.* [37], relativistic effects start to limit dislocation velocity at a stress

$$\sigma > \bar{M}B/3b(\mu/\rho)^2, \quad (2.9)$$

where μ is the shear modulus. This equation yields a stress equal to 400 MPa for copper, which is easily achieved in spallation experiments.

In the model of dislocation motion under a viscous drag, developed by Frost and

Ashby [45], the authors came to a similar conclusion about the dependence of the flow stress on the strain rate as a combination of two mechanisms: the thermally activated motion and the drag-controlled motion. This model considered the dislocation motion through a regular field of point obstacles. At sufficiently high dislocation velocities, the deformation behavior is governed by the viscous drag; in the intermediate regime, the deformation is a result of the superposition of both mechanisms, in the same way as shown in Fig. 2.2.

According to the available data in the literature, the stress dependence of the mobile dislocation density remains poorly understood. Analysis by Follansbee and Weertman [46] has shown that at strain rates over 10^4 s^{-1} , only a fraction (10^{-3}) of the total dislocation density is mobile in many metals. As was suggested by Frost and Ashby [45] and by Regazzoni *et al.* [37], the stress dependence of the mobile dislocation density could be important at high strain rates and this dependence could be responsible for some inconsistencies between experimental data and the theoretical models at high strain levels.

Weertman and Follansbee [35] gave a treatment of plastic waves at high stress levels. It was shown that the description of plastic waves should be given in terms of the dislocation dynamics parameters rather than in terms of the parameters characterizing the plastic constitutive equations. The constitutive equations frequently used to model the plastic wave propagation are strongly history-dependent, and therefore, cannot describe fully all important internal parameters [35] in plastic wave propagation. In the same paper it was shown that “a weak-to-moderate strength plastic wave front becomes narrower and narrower as the shock pressure is increased. In the limit of strong shocks the width becomes of atomic dimensions” (Weertman, [36]). It has been argued that at high stress levels, when the wave front becomes comparable to atomic dimensions, the activation of dislocation sources in a plastic wave front is strongly suppressed. Thus “the deformation within the shock front must be entirely elastic, provided that the theoretical shear strength of the solid is not exceeded. If it is exceeded, an extremely narrow front can come into existence through the agency of a Smith dislocation interface” (Weertman, [36]).

Therefore in our spallation experiments it would be reasonable to expect the occurrence of Smith-type dislocation interfaces at the stress concentration locations, such as at the edges of the artificial flaws. The flow stress is close to the theoretical shear strength of the material τ_{tss} in these cases [36]

$$\tau_{tss} = \frac{k}{30} = \frac{E}{90(1 - 2\nu)}, \quad (2.10)$$

where k is the bulk modulus of the solid, E is the Young's modulus, and ν is Poisson's

ratio. The zone of plastic deformation r_p can be estimated as

$$r_p = \left(\frac{K}{\tau_{tss}} \right)^2 = \frac{\gamma E}{\tau_{tss}^2}, \quad (2.11)$$

where K is the stress intensity factor which is estimated as $\sqrt{\gamma E}$, and γ is the interface energy. For Nb, which was used in most of our experiments this estimation yields $r_p \approx 20 \text{ \AA}$. This estimation produced a value which can be used as the scale in the spallation experiments where the SSY (Small Scale Yielding) approximation is still valid. The small value of $20 \text{ \AA}$ in comparison to the film thicknesses of $1 - 3 \text{ }\mu\text{m}$ allows us to use the elastic theory in the numerical model of the dynamic fracture of the thin films (Section 2.3).

2.2 Techniques for Generation of Microcracks at the Film/Substrate Interface

Two photolithography techniques were used to create artificial flaws at interfaces. Specifically, the microcracks were generated at the interface between (0001) oriented sapphire substrates and a $1 \text{ }\mu\text{m}$ thick Nb layer. The thickness of the film was measured by a quartz crystal oscillator during the deposition with a 3% accuracy. This was confirmed by measurements using a thin film profilometer. The substrates were cleaned before the deposition of the film by the standard technique, which included a sequence of 5 minute ultrasound washings in trichloroethane, acetone, methanol, and de-ionized water.

2.2.1 First Technique

The first technique is illustrated schematically in Fig. 2.3. The $1 \text{ }\mu\text{m}$ thick Nb layer was deposited on the sapphire substrate by the DC-magnetron sputtering method which resulted in a film with low intrinsic residual compression. A $200 \text{ \AA}$ thin film of Al was deposited on the top of the Nb layer. A certain pattern of another Nb layer was deposited by the RF-magnetron sputtering method on top of this Al layer. This pattern was generated by the use of the photolithography technique. The photoresist covered all the surface of the Al film except a number of round spots $90 \text{ }\mu\text{m}$ in diameter (Fig. 2.3). This was followed by the deposition of another Nb layer but this time by the RF method, which resulted in much higher residual compressive strains. The measurement of the residual strain is discussed in Chapter 3. Deposition of the Nb film with high residual compressive strain on top of the patterned structure caused the generation of a microcrack at the interface (Fig. 2.3) located at the round spots which were free from the photoresist.

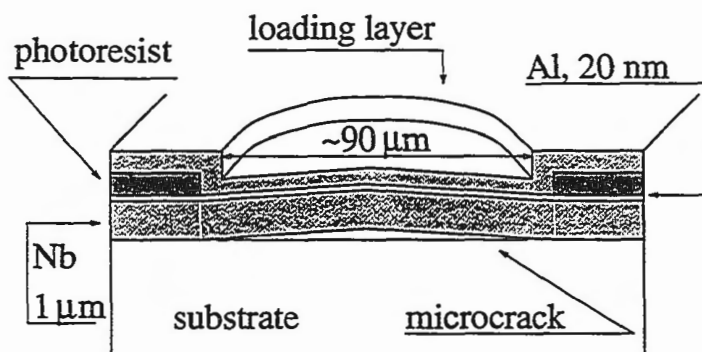


Figure 2.3: Microcrack at the film/substrate interface was obtained using the loading layer of Nb.

This is because the elastic strain energy entrapped within the total thickness of the Nb layers (both) is sufficient to overcome the interface fracture energy. The energy of the elastic compression stored in the Nb loading layer at places which were not free from the photoresist was essentially absorbed by the plastic deformation of the photoresist. Figure 2.4 shows significant buckling of the top loading Nb layer deposited on the top of the photoresist. The loading layer deposited on the Al film (Fig. 2.4) did not yet cause buckling of the lower structure from the substrate, as is shown by the nine white spots on the figure. Buckling was induced by an additional increase of the loading layer thickness at these spots.

As soon as there was enough energy to form the number of microcracks, the top several layers including the photoresist and the loading layer were easily removed by etching the sample in hydrochloric acid. Niobium and sapphire are stable in hydrochloric acid and therefore the dissolution of the Al inner-layer and the removal of the top layers was the only possible action of hydrochloric acid on the sample. Figure 2.5 shows the micrograph of the sample with several artificial flaws obtained after etching. Due to the optical effect, the apparent buckling amplitude of the flaw as observed under an optical microscope is quite different from the real one.

The photolithography technique used above to create the pattern of the Nb film is illustrated in Fig. 2.6, showing the exposure and the development of the positive photoresist, and the resulting etched film pattern. "Shipley Microposit S1813" positive photoresist was used in all the photolithographic procedures. The photolithography included the following sequence of steps: cleaning and drying of the substrate; spinning the photoresist first for 4 seconds at 400 RPM and then for 60 seconds at 4000 RPM; soft-baking the resist in an oven during 30 min at 120 °C; exposure of the samples using

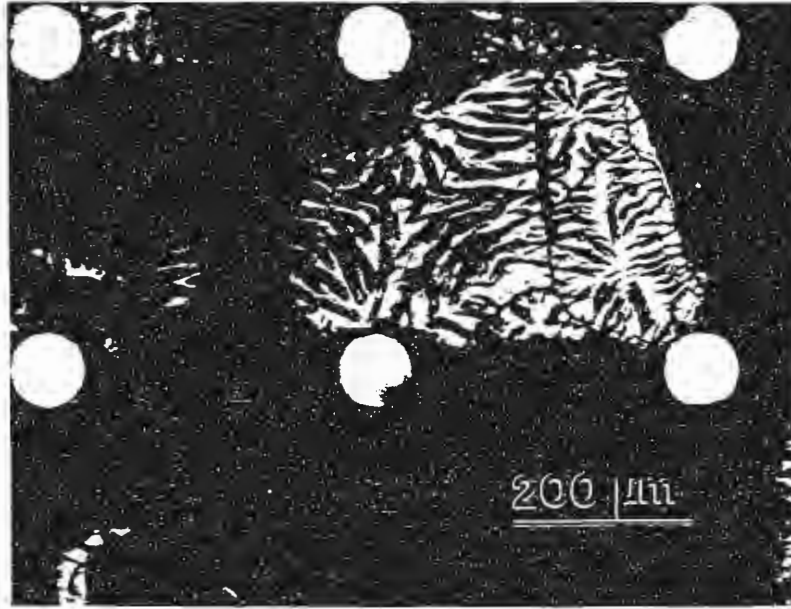


Figure 2.4: A micrograph showing the buckling behavior of the Nb loading layer deposited on the top of the photoresist.

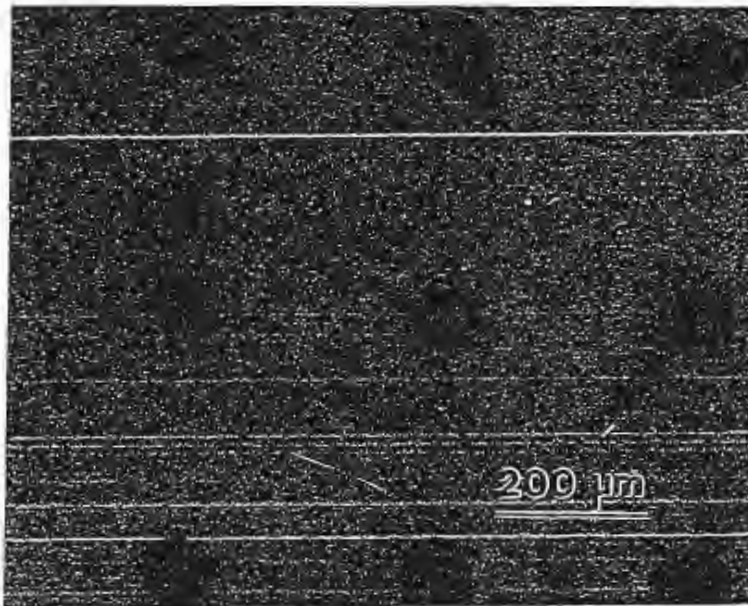


Figure 2.5: A micrograph of a sample with several round shaped microcracks induced by the loading layer in the state of residual compression. The sample was etched in hydrochloric acid. See text for the details of the process.

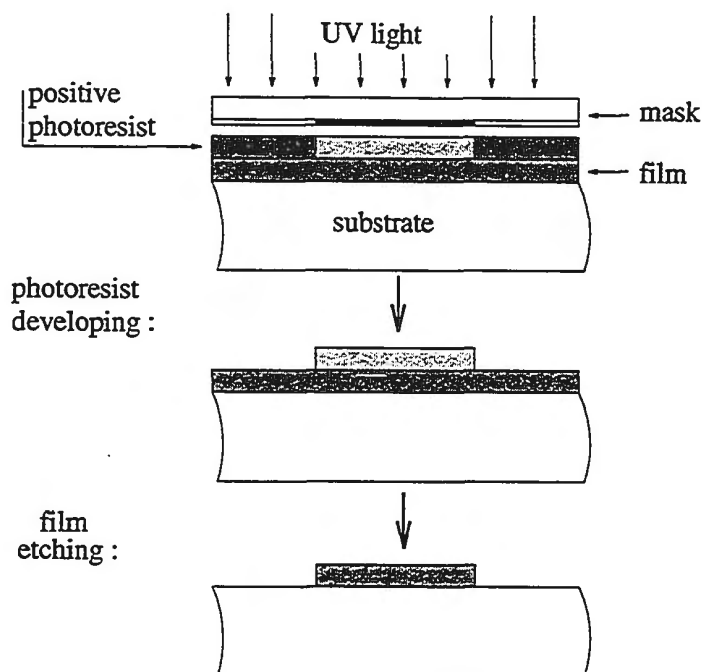


Figure 2.6: The generation of patterns in thin films using photolithography using positive photoresist.

a Karl Suss MJB 3 UV400 mask aligner for 12 seconds by an ultraviolet lamp with 15 mW/cm^2 power; developing the photoresist for 60 seconds and washing the samples with a de-ionized water; post-bake in the oven at 120°C for 30 min. Photographic film with a specially designed pattern was used as a mask in the process.

The Nb loading layer with high residual compressive strain was deposited by the RF-magnetron sputtering technique at the following experimental conditions: 3×10^{-6} Torr of the initial background pressure; 1×10^{-2} Torr of Ar pressure during sputtering; 250 W of RF-power; thickness of the target was equal to $1/8 \text{ in.}$ As discussed in detail in Section 3.3 niobium deposited at these conditions forms a film at 4×10^{-3} residual compressive strain. There we also discuss how the accumulated elastic energy can be used to determine the fracture interface energy for several metal/ceramic interfaces, (Chapter 3). However, for the purpose of the artificial flaw generation process discussed here, the residual stress was used here only to buckle the lower test layer from the substrate.

The geometry of the flaws and the stress pulse profile is an essential input to the numerical model of the fracture process at the thin film/substrate interface, discussed in Section 2.3 later. To this end the shape of the artificial microcrack was measured by the

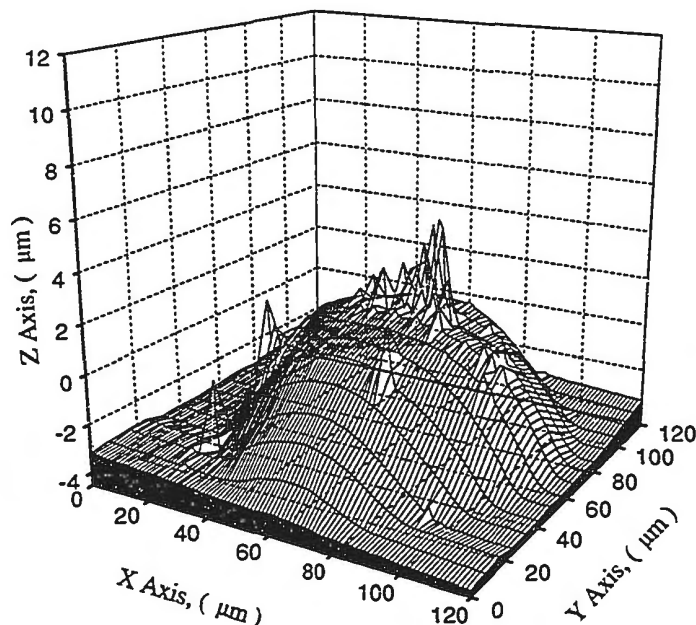


Figure 2.7: The profile of the buckled film as measured by a scanning force microscope. Buckling was induced by a Nb loading layer.

scanning force microscope² (SFM). The regular profilometers apply too strong a force on thin films which can disturb the measured profile of the buckled film. The measurement of the film profile was done in the non-contact mode, and the force acting on the film is induced mostly by the electrostatic interaction of the film with the microscope contact tip. Specifically, the force acting on the film by the scanning force microscope in this mode is negligibly small ($< 10^{-12}N$). It is five-to-six orders of magnitude less than the force acting upon the film by regular profilometer. The profile of the buckled film measured by SFM is shown in Fig. 2.7. The disturbances of the film profile which are seen in Fig. 2.7 are due to the presence of contamination and moisture on the surface of the film. The solid lines parallel to the X-axis on the figure represent the lines scanned by SFM. The shape of the buckled “cap” is obviously axi-symmetric and therefore the central line of the scanned profile has been taken as the input to the numerical model. Two lines that are nearest to the center of the “cap” are shown in Fig. 2.8. Despite its apparent outlook, the height of buckled cap is rather small in comparison to the spot diameter, the ratio of the height to the diameter is less than 5%. As shown in Section 2.3 later, the most important information about the buckled film shape is the profile of the

²I am thankful to Todd L. Hochwitz for helping me with the measurements on the scanning force microscope

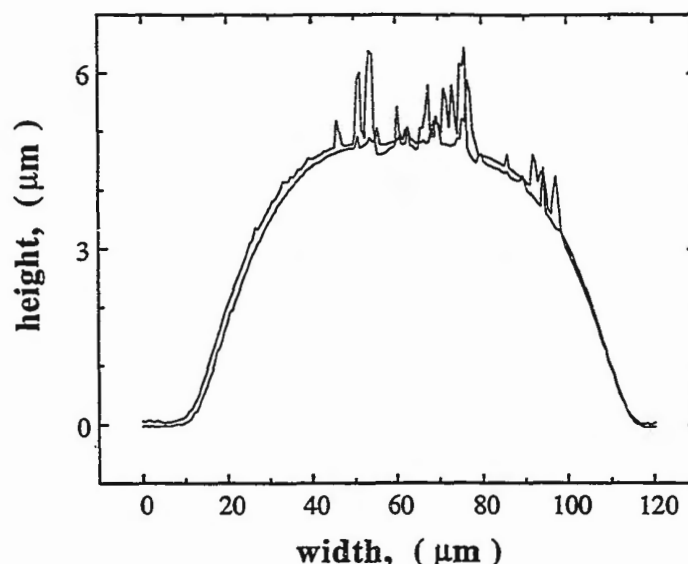


Figure 2.8: Two center lines of the buckled film as scanned by SFM.

film in the immediate vicinity of the crack tip. This profile is shown in Fig. 2.9.

The advantage of the technique is that pre-loading of the microcrack occurs naturally before dynamic loading. The method of microcrack generation discussed above in this section can be applied to many film/substrate pairs, although it cannot be applied to materials which will react with hydrochloric acid. In these cases, the problem might be solved by searching another combination of inner-layer (in the described case it was Al) and solvent (and hydrochloric acid). Other limitations to the technique are very strong interfaces. In these cases the elastic energy stored in the film could never achieve a level sufficient to force the film delamination at a given diameter of the spot which is free from the photoresist.

As discussed by Ortiz *et al.* [47], as soon as the critical thickness of the loading layer is greater than $\sim D\sqrt{\epsilon_0}$, the critical stress becomes higher than the residual stress and coating will not be able to buckle. Here the critical stress is defined as the stress which is required to buckle a free standing circular membrane, D is the diameter of the circular spot, and ϵ_0 is the residual strain in the film. In practical terms it means that in cases when the residual strain in the loading layer (for some reasons) was less than 4×10^{-3} , buckling was not always reproducible. The value of the residual strain in the loading layer is the critical parameter for the successful use of the technique. To eliminate the problems associated with the reproduction of the loading layer with high levels of the residual strain, a second technique was designed, and discussed next.

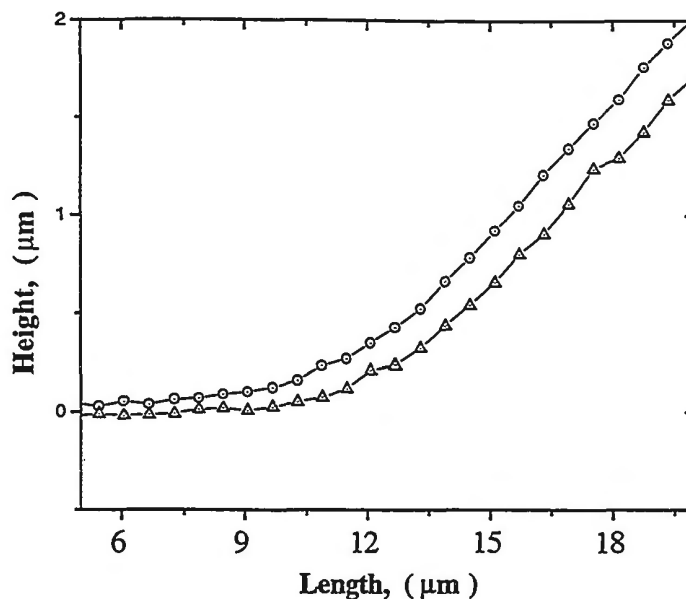


Figure 2.9: The profile of the buckled film close to the crack tip.

2.2.2 Second Technique

In the second technique, a high level of residual strain to induce buckling was not required. The delamination of the film from the surface of the substrate was achieved by chemical etching of a certain thin film microstructure (discussed below) produced by the photolithographic technique. The standard photolithography methods allowed deposition of a thin film structure shown in Fig. 2.10. To produce this kind of structure, a modification in the standard photolithography procedure was necessary.

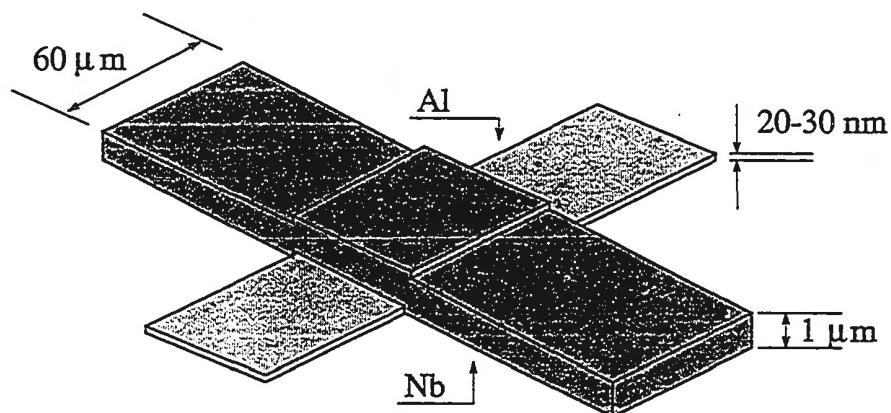


Figure 2.10: Cross-deposited strips of Al and Nb thin films on a sapphire substrate.

The lower strip of Al film (see Fig. 2.10) was obtained as a result of etching by using the regular photolithography technique, and is shown schematically in Fig. 2.6. The thickness of Al film was in the range of 200 to 300 Å. Next, a modified "lift-off" procedure was used. In the "lift-off" procedure, the film pattern was obtained not through the etching of the excessive parts of the film and conservation of the desired pattern underneath of the protective photoresist layer (Fig. 2.6), as done in the regular photolithography procedure, but instead, the desired pattern of the film was created first in the photoresist layer. The micrograph in Fig. 2.11 shows the strip of the aluminum film (white band) prepared by the standard technique, while the developed pattern in the photoresist lies on top of this structure. The next step was the deposition of a 1 μm thick Nb layer on top of the photoresist pattern. After deposition of the Nb film, the samples were immersed in an acetone ultrasound bath. The acetone dissolved the photoresist and the Nb layer was removed as well. The resulting structure is shown schematically in Fig. 2.10.

Structure was etched in an acid solution to dissolve the Al layer completely. This resulted in the generation of a microcrack beneath the Nb layer. A micrograph of such a microcrack is shown in Fig. 2.12. The ratio of the Al film to the Nb film thickness was small, less than 3%. Therefore the thickness step introduced in the Nb layer by the lower layer of Al could not significantly change the stress field distribution within the Nb film during dynamic loading, provided that the microcrack was statically pre-loaded. To provide pre-loading of the crack before transient loading was applied, the Nb film was deposited under conditions that resulted in entrapment of sufficient residual compressive strain. The analysis of the micrographs showed that under compressive stress the tips of the microcrack extended a little further during buckling than the steps induced by the lower Al layer. This can be clearly seen in an optical micrograph of the buckled film in Fig. 2.12. A more detail view at the tip under a scanning electron microscope is shown in Fig. 2.29, showing clearly that the crack tip is beyond the step created by the lower Al film.

The scanning force microscope was used to determine the profile of the buckled film for microcracks generated by both the first and the second techniques. Figure 2.13 shows the measured shape of the buckled film. The profile is a little different from the expected one-dimensional profile. This behavior could be explained by the same reason that explains general buckling, and it is due to the residual strain in the film. To relax residual strain in the direction normal to the direction of the strips, the film has to bend in the way shown in Figure 2.13, so as to satisfy the constraints of adhering substrate at the edges of the buckled film. A slice of the profile from the central region along the direction of the strip is shown in Fig. 2.14. This profile was used as an input to

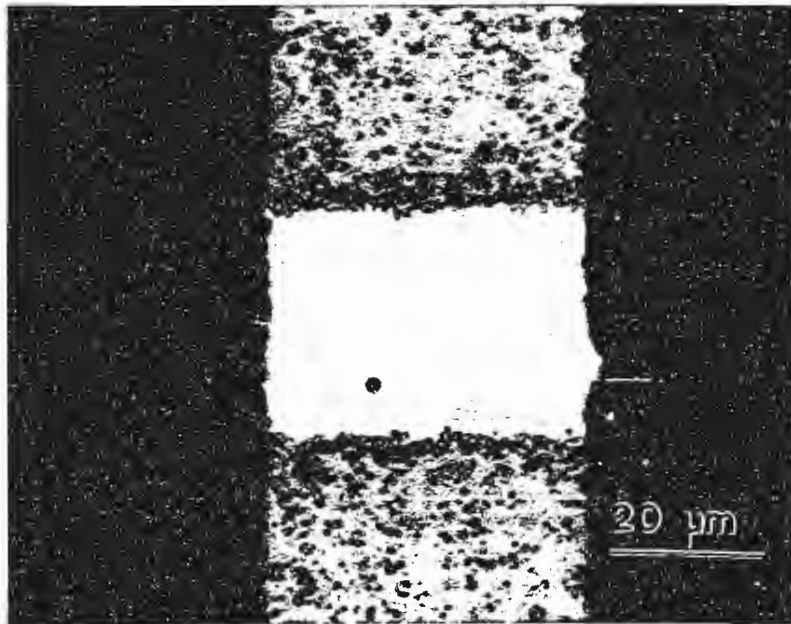


Figure 2.11: A micrograph showing the strip of an aluminum film (white band) prepared by the standard technique, awhile and the developed pattern in the photoresist lies on top of this structure.

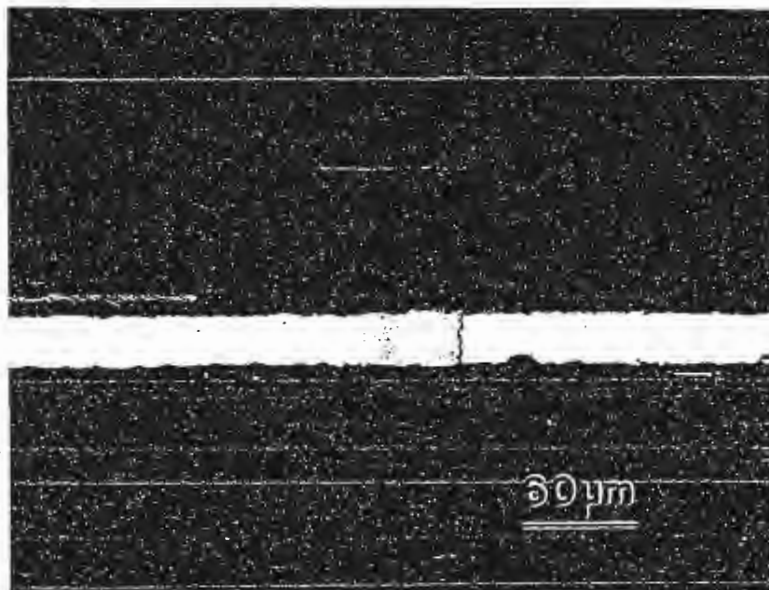


Figure 2.12: An optical micrograph of a microcrack obtained by the second technique.

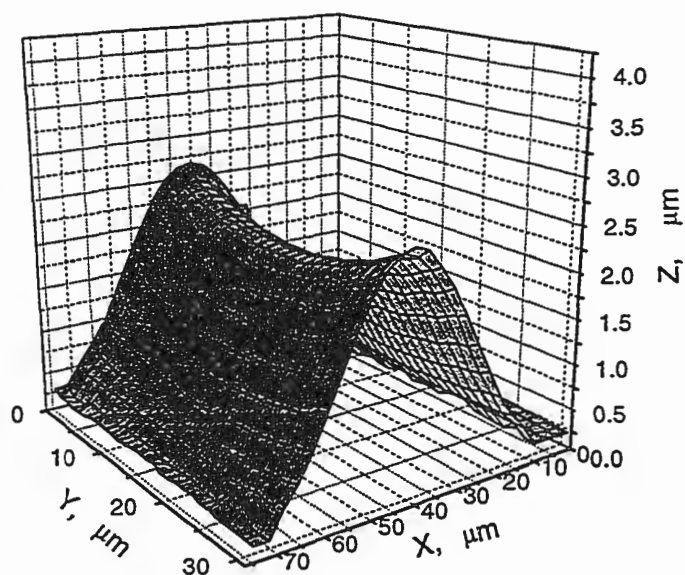


Figure 2.13: The shape of the buckled film as measured by a scanning force microscope. The microcrack was generated by using the second technique.

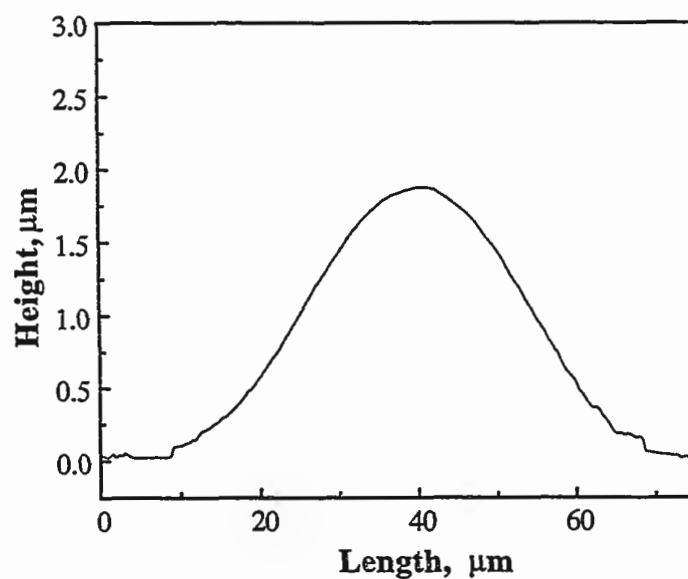


Figure 2.14: The profile of the buckled film that was used as an input in the numerical simulation of the fracture initiation process.

characterize the geometry of the film in the numerical simulations.

2.3 Numerical Simulation of the Microcrack Initiation Process

2.3.1 Description of the Numerical Model

To extract useful information from the data obtained in the spallation experiment a numerical model of the fracture initiation process at the microcrack tip during dynamic loading was developed. The geometry of the model was consistent with the shape of the microcrack obtained by SFM, as discussed in the previous section. The experimentally measured free surface velocity allowed definition of the loading conditions in the model. The purpose of numerical modeling was to determine the transient energy release rate history around the crack tip during the initiation of the fracture. A computer code based on a finite element package was used to calculate the time-dependent J-Integral. The J-Integral is a contour integral that gives the value of the energy release rate at fracture initiation. The code was not intended to model the propagation of the interface crack, which in turn is a more complicated problem and is amenable to much more sophisticated numerical methods that use “moving mesh” finite-element procedures [48].

The finite element software package ABAQUSTM developed by Hibbitt, Karlson & Sorensen, Inc. was used in the numerical analysis. The package allows application of already developed procedures (including dynamic) to the stress-deformation analysis. The major advantage of using ABAQUS was that it was not required to determine the matrices for individual elements of the material, to form mass and stiffness matrices of the system and to run through the full set of standard finite element procedures. ABAQUS has already taken care of that part of the numerical calculations. However, this advantage of the package leads to certain disadvantages: ABAQUS does not permit application of special boundary conditions (discussed below) and the fine tuning of the numerical model. For example, it does not allow to relate in one equation the displacement of the node and velocity of the same node. This relationship, as it will be shown below, is critical to defining a special boundary condition which allows for running the program in a reasonably short time (~ 12 hours). To this end FORTRAN program was added to the ABAQUS code. Another shortcoming of the package was the absence of the effective mesh generator. As a consequence of this, 80% of the typical ABAQUS source code is a mesh generating code. The ABAQUS source code and Fortran “patch” code are both

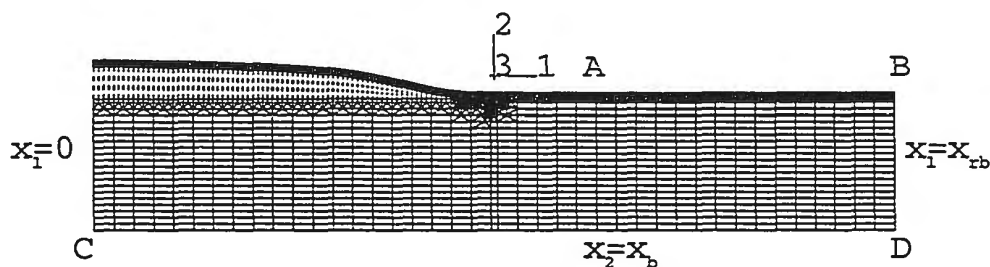


Figure 2.15: The finite element mesh created to model the axis-symmetric microcrack.

included in Appendix A.

The program models the geometries of the microcracks created by both the first and second techniques described previously in Section 2.2, and therefore, the code was designed to work in both axis-symmetric and two-dimensional geometries. The model describes the propagation of elastic stress waves in two-dimensional space. The switch from the axis-symmetric case to the two-dimensional case was done by simply redefining the type of the finite elements used in the code. The mesh and the numerical procedures were otherwise the same in both cases. The axis-symmetric microcrack (Fig. 2.8) was modeled by the finite element mesh shown in Fig. 2.15. The mesh close to the crack tip was generated based on the data shown in Fig. 2.9, and its detail near the crack tip are shown in Fig. 2.16.

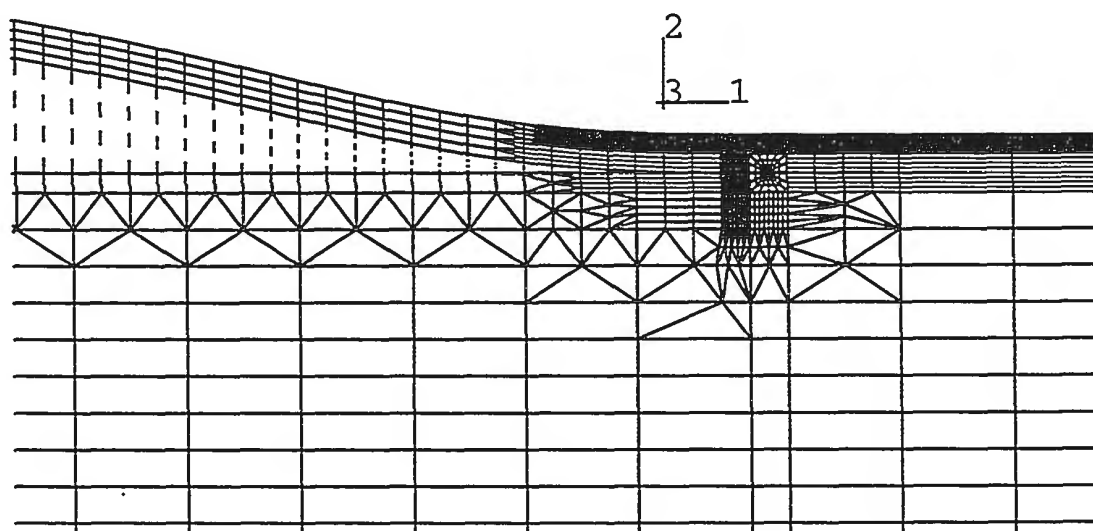


Figure 2.16: The finite element mesh in the vicinity of the crack tip.

To effectively utilize the “J-INTEGRAL” procedure of ABAQUS, a focused mesh near the crack tip was used, Fig. 2.16. This part of the mesh consisted of the second-order 8-node elements (CAX8R-type in the axi-symmetric case, and CPE8R-type in the plane strain case) with the midside nodes moved to the quarter-point positions. It allowed modeling to some degree the singularities of the solution close to the crack tip. Five contours encircling the crack tip were defined, with each contour surrounding a set of 16 elements at the crack tip. The rest of the finite element mesh is shown in Fig. 2.15. The 20 horizontal rows and 42 vertical columns of finite elements were created in the substrate. Most of the elements used in modeling were defined as 4-node bilinear elements with reduced integration, which were CAX4R and CPE4R types in the axi-symmetric and plane strain cases, respectively. Reduced integration uses a lower-order integration to form the element stiffness matrices.

The following assumptions were made in the numerical model. In accordance with the arguments presented in the previous Section 2.1, the material deformation was limited to the elastic regime since as discussed before SSY (small-scale-yielding) conditions are likely to prevail under the high rate of loading in our experiments. Thus, the following wave mechanics equations were used to model the stress wave propagation in both the substrate and the film

$$\frac{\partial \sigma_{ij}}{\partial x_j} = \rho \frac{\partial^2 u_i}{\partial t^2}, \quad (2.12)$$

$$\sigma_{ij} = C_{ijkl} \varepsilon_{kl}, \quad (2.13)$$

where σ_{ij} is the stress tensor, ε_{kl} the strain, and u_i the displacement field in the material. Further, ρ and C_{ijkl} are the material density and the elastic stiffness constants, respectively. The film density and elastic properties were taken as those of the polycrystalline bulk metal. However, for single-crystal films, or for polycrystalline films with a strong crystallographic texture, the anisotropy of the elastic properties must be taken into consideration [49]. For example, the biaxial elastic modulus of a cubic single-crystal film is given by

$$M(001) = C_{11} + C_{12} - \frac{2C_{12}^2}{C_{11}}, \quad (2.14)$$

for a film with the (001) cube plane parallel to the substrate, and

$$M(111) = \frac{6C_{44}(C_{11} + 2C_{12})}{C_{11} + 2C_{12} + 4C_{44}}, \quad (2.15)$$

for a film with (111) plane along the substrate [49]. By using the single crystal stiffness coefficients of the Nb equal to $C_{11} = 246.5 \text{ GPA}$, $C_{12} = 134.5 \text{ GPA}$ and

$C_{44} = 28.73 \text{ GPa}$ [50], these equations give 234 and 141 GPa as the effective elastic constants for (001) and (111) orientations, respectively. The biaxial elastic modulus of the Nb film calculated using the isotropic elastic values ($E_{is} = 104.9$ and $\nu = 0.397$, [51]) equals 174 GPa. The difference in the assumed elastic modulus of the film will not influence the amplitude of the interface stress significantly, provided the thickness of the film is much smaller than the length of the stress pulse. The thickness of the film in our experiment was usually maintained at $1 \mu\text{m}$, while the stress pulse length was in the range of 100 to $200 \mu\text{m}$, depending on the stress wave velocity in the substrate. At these conditions, the interface stress component normal to the free surface can be calculated as

$$\sigma_{22} = \rho h \frac{\partial V}{\partial t}, \quad (2.16)$$

where ρ is the density of the film, h is the thickness of the film and V is the free surface velocity. The stress depends only on the mass of the film, and not on the elastic or plastic properties of the film. Thus, the same stress will develop between the substrate and an even liquid layer, as long as the mass of the liquid is the same. Equation 2.16 is simply obtained from the Newton's second law, and does not require any assumptions of elasticity.

However for measurements that involve calculation of the stored elastic energy due to the residual strain in the film, the knowledge of the exact elastic coefficients is essential. The interface toughness value measured by Spontaneous Delamination Technique (discussed later in Chapter 3) is proportional to the elastic modulus, and therefore the inaccuracy in elastic modulus determines the accuracy of the measurement. Fortunately, the growth of the Nb films with epitaxial or strong texture requires elevated substrate temperatures. The Nb films used in our experiments (throughout this work) were always deposited at the room temperature. As shown by Datta *et al.*, [222] is the preferred orientation of the Nb films deposited by a RF sputtering technique with zero substrate bias voltage [52]. Sürgers *et al.* [53] showed that the texture of the Nb film decreases towards lower substrate temperature during the deposition. They also showed that the transition from a columnar growth structure to a fine grain structure occurs at a substrate temperature T_s of about 300°C . At lower temperatures, the grain size becomes less than $150 \text{ \AA}$. Due to the lack of experimental data on the texture, and elastic anisotropy of deposited Nb film, the elastic properties were assumed isotropic throughout our work here. The determination of the exact elastic anisotropy of the deposited Nb film for different technological conditions was not pursued in this thesis.

The total thickness of the substrate used in the calculation was equal to $20 \mu\text{m}$. The

width of the mesh assembly was taken equal to the diameter of the buckled area; in the axi-symmetric case, it was $120\ \mu m$. Because of the symmetry of the buckled film, only half of the system was modeled. This determined the boundary conditions for all the nodes on the left boundary as

$$u_1|_{x_1=0} = 0. \quad (2.17)$$

A similar boundary condition was used for the nodes on the right boundary; it was based on the assumption of plane wave propagation normal to the surface and it neglected the influence of all crack tip phenomena on the motion of the right boundary (rb), thus

$$u_1|_{x_1=x_{rb}} = 0 \quad (2.18)$$

was maintained for nodes on the right boundary. On the free surface of the film no boundary conditions were prescribed. Additional mesh refinement was performed to model the film and the area of the substrate close to the crack tip. This area is also shown in Fig. 2.16. To simulate the mechanical interaction of the film with the substrate, interface elements were introduced. These elements are shown by dotted lines in Fig. 2.15. The primary function of these elements was to model the film/substrate interactions without allowing the meshes of the film and the substrate to inter-penetrate each other during the deformation caused by the pulse loading. Mesh refinement included also 3-node linear elements as well. The thickness of the film was maintained at $1\ \mu m$ in all simulations.

To model 20 ns of stress wave propagation without allowing the first reflected wave from the free surface and the interface to arrive at the crack tip after reflection from the substrate's lower boundary would require modeling $100\ \mu m$ of the substrate thickness. Unfortunately, given the nature of the problem here, discretization of such a large space will make the execution of the program almost impossible. To this end, a numerical "trick" was applied to decrease the size of the mesh and the required computer time. It is evident that all phenomena of interest in the model are taking place close to the crack tip while the rest of the calculation simply integrate the regular wave equation, Eq. 2.12. These additional calculations however take the major part of the time. The stress pulse incoming to the free surface (AB, Fig 2.15) represents a one-dimensional displacement field

$$u_{+2}(x, t) = f(x_2 - ct), \quad (2.19)$$

while $u_1(x_i, t) = u_3(x_i, t) = 0$. Here x_2 denotes the direction normal to the free surface, and x_1, x_3 are the directions along this surface. The stress pulse reflected from the free surface is also mostly a one-dimensional stress field with the exception of some non-planar

disturbances caused near the area around the crack tip. Assuming that this disturbance from the plane front is negligibly small at some distance from the interface, the reflected wave can be also considered as a plane wave. At some distance from the film/substrate interface the virtual boundary with “non-reflective” boundary condition was introduced. This “non-reflective” boundary was placed at 20 μm away from the interface. The “non-reflective” boundary condition is

$$\left. \frac{\partial u_2}{\partial t} - c \frac{\partial u_2}{\partial x_2} \right|_{x_2=x_b} = 2V(t), \quad (2.20)$$

which then allows the specification of the amplitude of the incoming stress pulse according to

$$\left. \frac{\partial u_{+2}(x, t)}{\partial t} \right|_{x_2=x_b} = V(t), \quad (2.21)$$

and transmits through all the out-coming plane waves propagating normal to the boundary without any reflection. The function $2V(t)$ is the velocity of the free surface as measured by the interferometer. The out-coming plane wave (from the free surface (AB) towards the lower boundary(CD), as shown in Fig. 2.15) which does not propagate normal to the boundary are partially reflected back into the mesh. However, as will be shown later, the influence of these reflected waves on the crack tip energy release rate is negligibly small.

To impose equation 2.20 as the boundary condition it is necessary to relate the nodal displacement to the nodal velocity in one equation. As mentioned earlier, ABAQUS does have this option which includes in one procedure the velocity and the displacement of the same node. ABAQUS allows, however, use of a “BOUNDARY” option where node displacements can be specified explicitly by a user-defined Fortran subroutine, “DISP”. This subroutine was modified to scan the ABAQUS results file for the nodal velocities, which were required to calculate the displacements.

The energy release rate around the crack tip was determined by means of an ABAQUS procedure. This procedure was designed to calculate the J-Integral around the crack tip in the case of static loading according to the fracture mechanics definition of the integral, which has the following form

$$J_s = \int_{\Gamma} \left(U n_1 - \sigma_{ij} n_j \frac{\partial u_i}{\partial x_1} \right) d\Gamma, \quad (2.22)$$

where $U = \frac{1}{2} \sigma_{ij} \varepsilon_{ij}$ is the elastic energy density, u_i is the displacement, Γ is a small contour that surrounds the crack tip, and n_j is the vector normal to this contour. However, as is well known from the literature [54, 55], the application of the J-Integral concept to

the cases of transient loading and propagating cracks has certain limitations. The energy release rate at the crack tip under dynamic conditions can be determined by a relationship which is different from the relationship given by Eq. 2.22, [54]:

$$J_d = \lim_{\Gamma \rightarrow 0} \int_{\Gamma} \left((U + T)n_1 - \sigma_{ij} n_j \frac{\partial u_i}{\partial x_1} \right) d\Gamma, \quad (2.23)$$

where

$$T = \frac{1}{2} \rho \frac{\partial u_i}{\partial t} \frac{\partial u_i}{\partial t}$$

is the kinetic energy density. Equation 2.23 gives the correct value for the energy release rate in the general case of dynamic loading only if the limit $\Gamma \rightarrow 0$ holds. The integral given by Eq. 2.23 is path-independent only under the stationary crack propagation condition. Thus, in the case of transient loading of the stationary crack, the integral is not path-independent. However, it is evident that as soon as the integration contour Γ approaches the crack tip the integral becomes increasingly path-independent [55].

The velocity of particles in the vicinity of the stationary crack tip decreases as the square root of the distance to the tip, and therefore the kinetic term in Eq. 2.23 does not represent a singular term in the equation. The ratio of the kinetic energy density to the elastic energy density vanishes as the square of the distance to the crack tip. It can be estimated as

$$\frac{T}{U} \sim \frac{\rho}{E} \frac{V^2(t, x)}{\varepsilon^2(t, x)} = \frac{1}{c^2} \left(\frac{\partial u / \partial t}{\partial u / \partial x} \right)^2 \sim \left(\frac{r}{c\Delta t} \right)^2, \quad (2.24)$$

where c is the stress wave velocity, Δt the characteristic time of transient loading, and r is the distance from the tip to the contour. Characteristic values of these parameters in the spallation experiment are as follows: $\Delta t \sim 20 \text{ ns}$, $c \sim 5 \text{ } \mu\text{m/ns}$, $r \sim 1 \text{ } \mu\text{m}$. This yields a ratio T/U close to 10^{-4} , and therefore in our case we can neglect the contribution of the kinetic energy in Eq. 2.23 and use the expression from Eq. 2.22 to numerically calculate the energy release rate. From a physical point of view, the condition

$$\frac{r}{c\Delta t} \ll 1 \quad (2.25)$$

means that the stress changes slowly enough to allow quasi-static consideration of the stress field around the crack tip. The J-Integral calculated by the ABAQUS built-in procedure can now be used to determine the energy release rate for transient loading which takes place in our experiments.

In total, 2355 elements and 3727 nodes were generated in the model. This represented a relatively large-size model for the dynamic analysis. One increment of the analysis

performed by RISC-6000 processors took approximately 2 minutes of computer time. However, to model approximately 20 *ns* of the stress wave propagation in real time, it was necessary to run several thousand increments. The time in the model processed in one increment is determined by the type of the dynamic analysis, by the stability of the numerical model and by the size of the model. Direct integration of the system was used in the calculations. The method of integration used in the ABAQUS is called the “Hilber-Hughes-Taylor operator”. The Hilber-Hughes-Taylor operator utilizes the implicit method of solving the nonlinear dynamic equations at each increment which makes the calculations numerically expensive.

Implicit methods of integration of hyperbolic equations are supposed to be unconditionally stable with respect to the choice of time increment. However this stability may be violated by the introduction of boundary conditions and by certain types of constraints imposed on the mesh. The the unconditional numeric stability of the model is reduced to the following conditional stability: the numerical model will be stable only if the following expression is valid (“Courant” condition)

$$\frac{c\Delta t}{\Delta x} < 1. \quad (2.26)$$

Here c is the wave velocity, Δt is the increment time and Δx is the size of the element which participates in the boundary condition. If the chosen time increment is longer than $\Delta x/c$, then some eigenvalues of the stiffness matrix become close to 1 and lead to an instability in the numerical solution. By trial-and-error, it was determined that the critical time increment in our model was of the order of 0.1 *ns*. The accuracy of the solution was an additional parameter that had to be taken into consideration while choosing the time increment. The increment was chosen equivalent to one/tenth of the mesh size used to calculate the J-Integral. A value of 0.02 *ns* was selected as the time increment, which optimized both the accuracy and the stability. Thus 20 *ns* of stress propagation modeling required at least 500 increments; which took approximately 12 hours of the computation time.

2.3.2 Results of the ABAQUS Finite Element Model

In this section the numerical results obtained from the ABAQUS dynamic model are discussed. The results are given for a chosen pulse profile. The stress wave is initiated at the lower boundary (CD) of the mesh, Fig. 2.15. As was discussed in the previous Section 2.3.1, the condition defined by Eq. 2.20 at the lower boundary allows formulation of the pulse propagating from the bottom of the mesh toward the free surface and

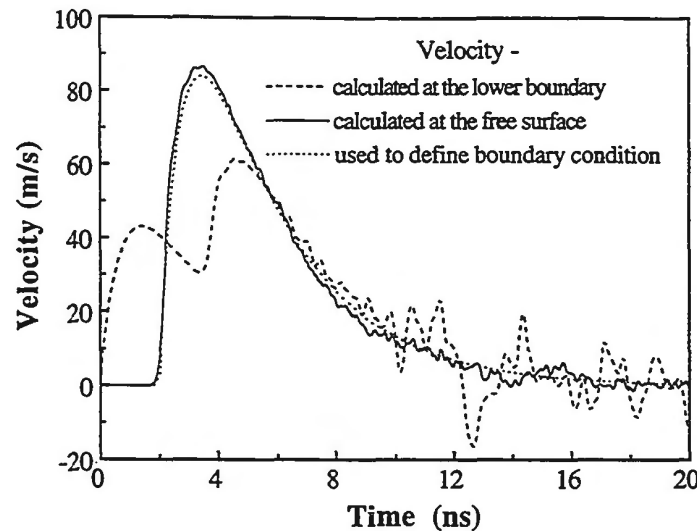


Figure 2.17: Velocities calculated at the free surface and the boundary. Dotted line represents the velocity used to define the a “non-reflective” boundary condition.

transmission of the wave coming towards the lower boundary (CD) without significant reflection.

Figure 2.17 shows the velocity history calculated at different locations of the mesh. The dotted line is the velocity that was used to define the “non-reflective” boundary condition. The velocity of the stress pulse propagating towards the free surface (upper boundary, (AB), see Fig. 2.15) is equal to half of that amplitude. The amplitude of the velocity at any point within the material is the sum of the incoming and the outgoing stress pulses. The pulse outgoing from the free surface is equivalent to the incoming pulse, but is delayed in time. The solid line in Fig. 2.17 is the free surface velocity which is, according to the free surface boundary condition, twice that of the incoming pulse velocity. The good correlation between the dotted and solid lines in Fig. 2.17 indicates that numerical model behaves in accordance with the analytical predictions. Correlation supports the above mentioned assumption that all outgoing waves are transmitted through the lower boundary (CD) without a significant reflection. Otherwise it would lead to a substantial disturbance of the free surface velocity (solid line) from the expected velocity (dotted line). The dashed line in Fig. 2.17 represents the velocity at the lower boundary. The influence of both incoming and outgoing pulses is evident in this case. Pulses are separated approximately by a 4 ns time delay which is the wave flight time from the boundary (CD) to the free surface (AB) and back.

The primary purpose of the numerical analysis was to determine the transient energy

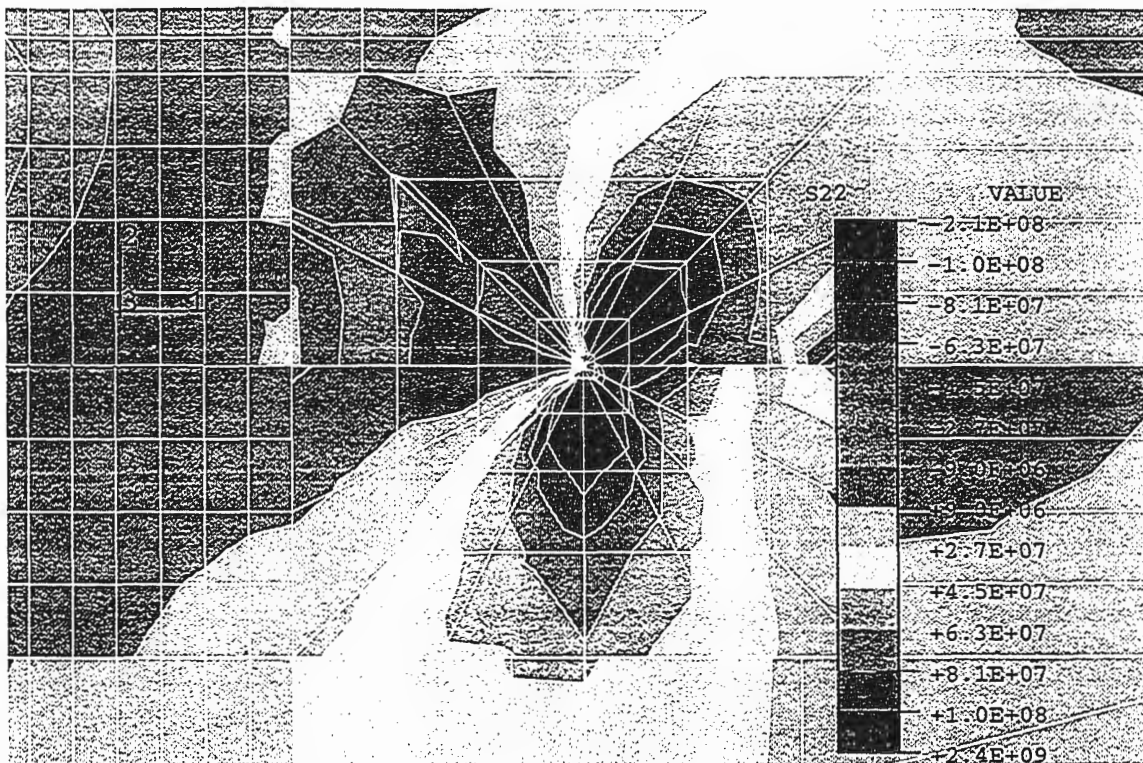


Figure 2.18: A contour plot of the σ_{22} stress component in the vicinity of the microcrack tip.

release rate or the J-Integral. Figure 2.18 represents a contour plot of the σ_{22} component of the stress tensor in the vicinity of the crack tip. The contours of the stress centered at the crack tip indicate crack-type stress singularity. The stress field in Fig. 2.18 corresponds to the 506th increment of the program and to 20 ns of total accumulated time. Figure 2.19 shows the transient J-Integral calculated at four contours closest to the crack tip. The numerical error in the determination of the J-Integral values by finite element methods is usually estimated as the difference between integral values calculated on different contours. Figure 2.19 shows that the error in the J-Integral due to the mesh discreteness and numerical effects is small, and usually less than 5%. The modulation of the J-Integral transient profile is caused by the complex dynamic motion of the thin film. The impulse of the substrate surface motion induces the Lamb-type waves in the free-standing film. Figure 2.20 shows both the original and the displaced meshes of the model. The nodal positions in the displaced mesh are plotted according to the following ABAQUS convention

$$u_d = u_i + M d,$$

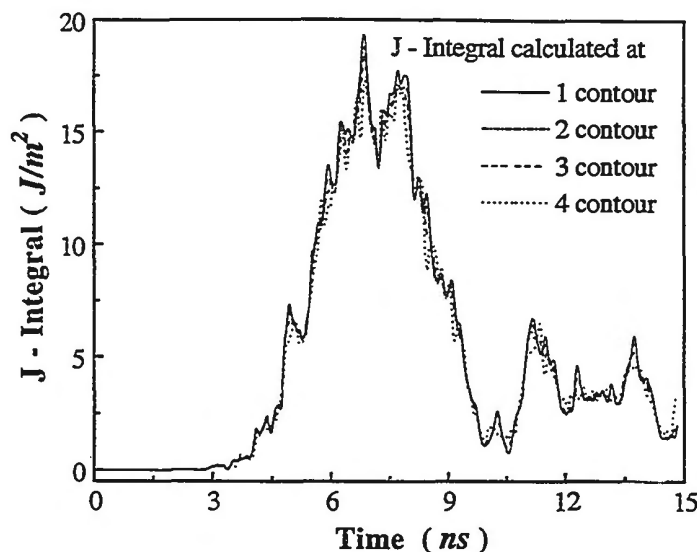


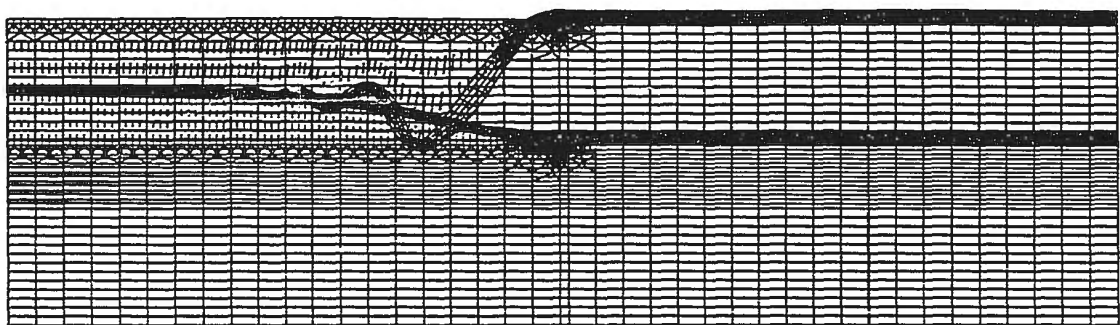
Figure 2.19: J-Integral time history. Four contours are used to calculate the integral.

where u_d is the position of the displaced node, u_i is the initial coordinate of the node, d is the displacement of the node and M is the magnification factor. The magnification factor M is used in ABAQUS post-processing to display graphically the displaced mesh, the displaced mesh looks rather distorted from its real shape. The generated Lamb-type waves in the film are shown in Fig 2.20. The group velocity of the lower mode of the antisymmetric Lamb wave vary according to [56]

$$v_g = 2 \left(\frac{E}{3\rho(1-\nu^2)} \right)^{\frac{1}{4}} \sqrt{\omega d}, \quad (2.27)$$

where E is Young's modulus, ρ is the density, ω is the frequency of the wave, ν is the Poisson's ratio, and d is the thickness of the film. In our case ω can be estimated as $2\pi/\Delta t$, where Δt is the stress pulse duration. At small enough ratios of the film thickness to the stress pulse duration, the group velocity of the antisymmetric Lamb waves can be much smaller than any bulk wave velocity, which is of the order of $\sqrt{E/\rho}$. Assuming that Δt equals to 15 ns, Eq. 2.27 yields in our case a Lamb wave velocity of 1.8 $\mu\text{m}/\text{ns}$ for a 1 μm thick Nb film. This estimation is consistent with the numerical results shown in Fig. 2.20, where during the first 20 ns the disturbance in the Nb film has propagated approximately 36 μm from the crack tip.

The low velocity of the Lamb waves in comparison to the bulk velocities leads to several conclusions. First of all, the low velocity of the deformation propagation along the film localizes the stress field in the film close to the edge. The J-Integral assumes



2
 3 1
 DISPLACEMENT MAGNIFICATION FACTOR =32 ORIGINAL MESH DISPLACED MESH
 TIME COMPLETED IN THIS STEP 2.000E-08 TOTAL ACCUMULATED TIME 1.00

Figure 2.20: The original and displaced meshes after 20 ns.

its maximum value 5 ns after the transient tension pulse has reached the crack surface. During this time the deformation was able to propagate only $9 \mu m$ ($=1.8 \mu m/ns \times 5ns$) into the free standing film as measured from the crack tip. Thus, the local shape of the buckled film very close to the crack tip is critical to the model. From that perspective it is evident that such parameters of the model as the type of the buckled shape (axi-symmetric or one-dimensional) and the size of the buckled film are not important geometrical parameters in the experiment.

To accommodate the total displacement caused by the substrate motion the stress waves in the film should have higher amplitude than that in the substrate, provided that the velocity of stress wave propagation in the film is smaller than that in the substrate. The localization of the stress field in the film results in increasing values of the stress in the film, as was shown in Fig. 2.21. The red and blue regions in Fig. 2.21 correspond to the regions under tension and compression, respectively. The details of the displaced mesh after 10.56 ns of wave propagation are shown in Fig 2.22. Comparison of Fig. 2.16 with Fig. 2.22 shows significant bending of the film, which is a characteristic of the antisymmetric Lamb waves.

The J-Integral was calculated for different maximum velocity amplitudes at the same relative time profile. The dependence of the maximum J-Integral value on the maximum transient velocity amplitude is shown in Fig. 2.23. The data is plotted on a double-logarithmic scale. At small velocities, the J-Integral is proportional to the square of the

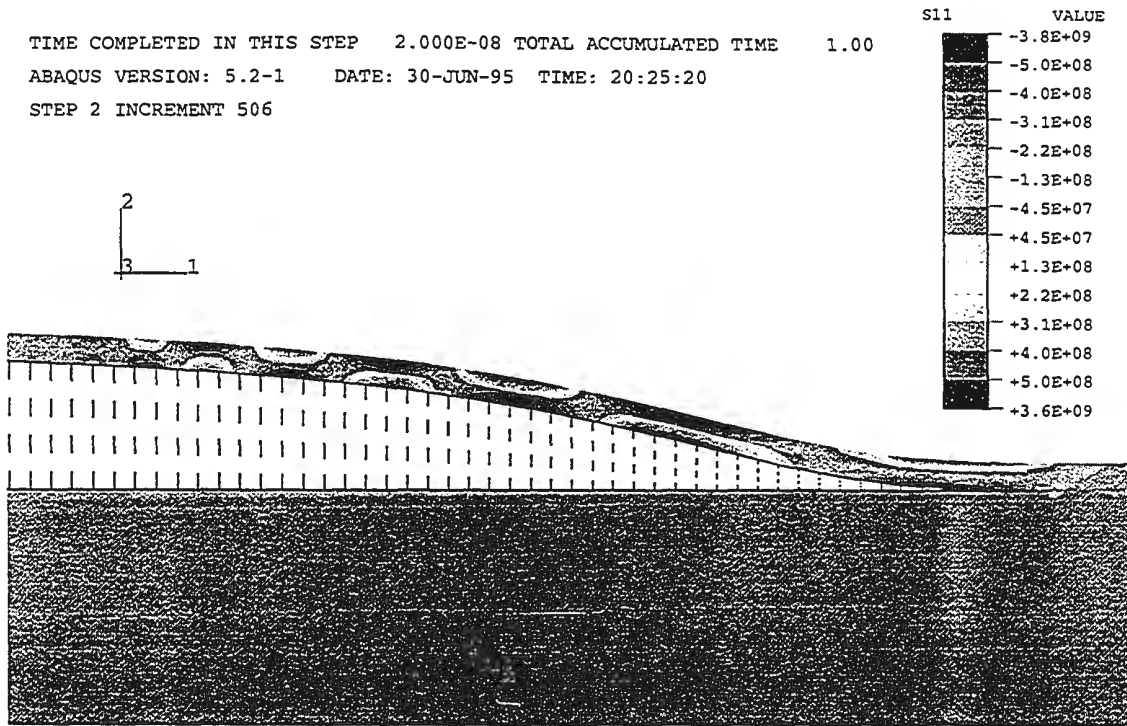


Figure 2.21: Contour plot of σ_{11} stress component. Scale is determined by the thickness of the film, which is $1 \mu m$.

velocity amplitude. This relationship follows directly from

$$J \sim \frac{K^2}{E} \sim (\sigma\sqrt{a})^2 \sim \sigma^2 \sim V^2,$$

where a is the effective dynamic crack size and K is the stress intensity factor. a depends on the effective stress wave velocity (c) through the following relationship $a \sim c\Delta t$, where Δt is the stress pulse characteristic time. Effective stress wave velocity in this case should be close to the Lamb wave velocity, which, as shown by Eq. 2.27, is not a constant but rather a strong function of the pulse duration (Δt) itself. Nevertheless, in the limit of low particle velocities when the mesh geometry remains practically the same, the effective crack size (a) is not a function of the particle velocity. However, at higher particle velocities, multiple collisions between the film and the substrate occur and this makes the model geometrically non-linear, and hence impossible to analyze by using analytical approach. Our numerical results (Fig. 2.23) in this regime indicate that the J-Integral is approximately proportional to $\sim V^4$. Figures similar to Fig. 2.23 can be used as calibrated charts to transfer the experimentally obtained free surface velocity data using an interferometer into the local critical energy release rate values.

DISPLACEMENT MAGNIFICATION FACTOR = 1.00
 TIME COMPLETED IN THIS STEP 1.056E-08 TOTAL ACCUMULATED TIME 1.00
 ABAQUS VERSION: 5.2-1 DATE: 09-MAR-95 TIME: 13:02:23 STEP 2 INCREMENT 600

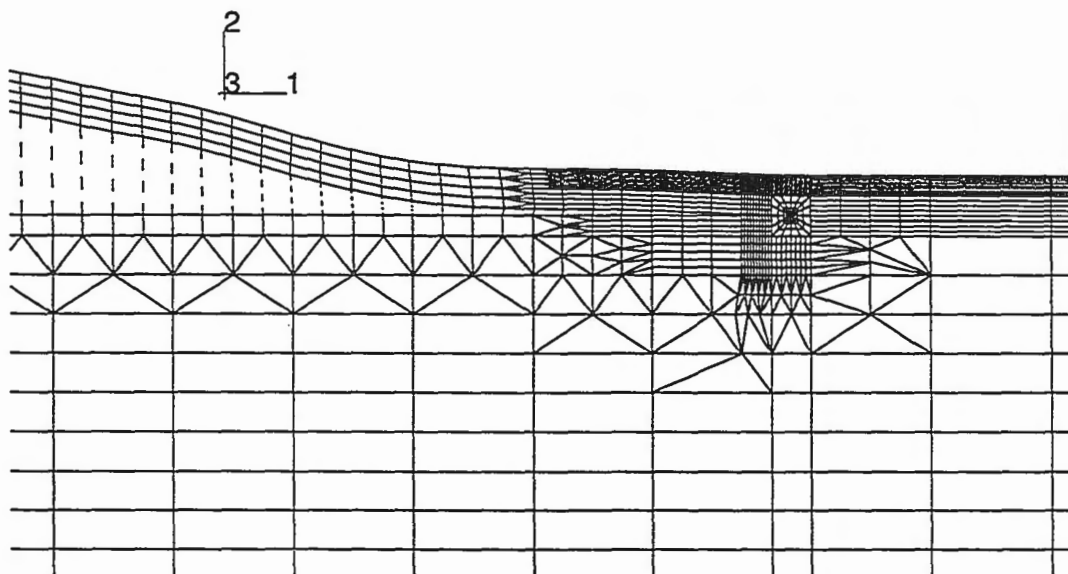


Figure 2.22: A picture showing the displaced mesh after 10.56 ns. Here the displacement magnification factor equals 1.

2.4 Experimental Results

As discussed earlier, the artificially generated interface microcracks were loaded by transient stress pulse generated in the laser spallation assembly. The purpose of the experiment was to determine the critical level of the free surface velocity that caused the onset of crack propagation. The velocity data obtained by the laser interferometric technique served as an input to the numerical model that calculated the transient energy release rate (or J-Integral) at the microcrack tip.

The systems tested in this work were chosen because of our prior experience with these systems, and also from our desire to compare the results of dynamic testing with those obtained by an independent spontaneous delamination experiment (Chapter 3). In both techniques (dynamic loading and spontaneous delamination), a test coating 100 Å to 200 Å in thickness of Al, Cu, Sb was deposited on (0001) oriented sapphire substrate disks. This layer was then overlaid by a Nb layer of 1 μm thickness. The ratio of test-layer thickness to the thickness of Nb was negligibly small. Therefore in both techniques, the lower test-layer/sapphire interface was of interest and it was assumed that its tough-

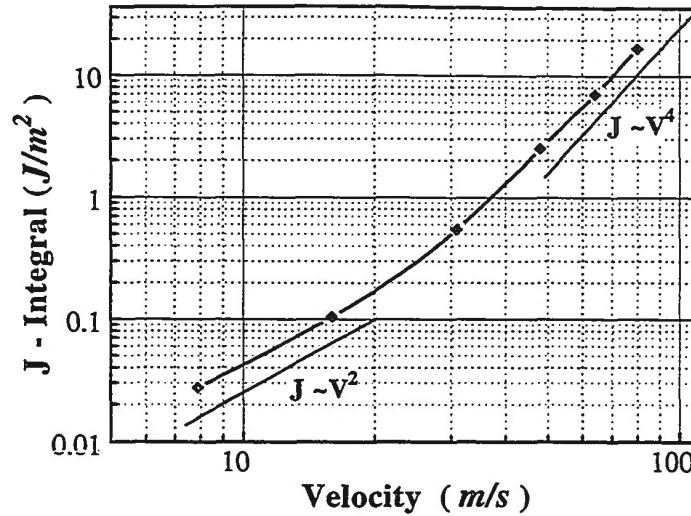


Figure 2.23: J-Integral dependence on the free surface velocity amplitude.

ness is determined by the local sapphire/testing-layer interfacial structure, while its the loading stress is determined by the stress distribution within the overlaying Nb layer and the sapphire substrate as well. The intrinsic strain in Nb films was different in the two cases. In the spontaneous delamination technique the built-in stresses were high enough to induce delamination along the interface (Chapter 3). In the case of dynamic loading the Nb overlayer of much lower intrinsic stress was used to avoid delamination but yet ensuring the same geometry of the microcracks as in the other technique. The Nb films with high level of residual compressive strains were deposited by the RF-magnetron deposition technique. On the other hand, the DC-magnetron deposited films were characterized by much lower residual strains and were used for dynamic loading.

Determination of the critical stress level required for crack initiation was performed by the viewing the microcracks using an optical microscope. Optical microscopy proved quite sensitive to detect delaminations on the scale of the microcracks under consideration. For instance, the micrograph in Fig. 2.5 shows round shaped microcracks with diameter of approximately $100 \mu\text{m}$ and height of $5 \mu\text{m}$. The clear and distinct image of the microcrack is obtainable due to the optical effect created by the curvature of the buckled film. Assuming a spherical shape of the buckled cap, it is possible to estimate the curvature (C) of the film as

$$C = \frac{1}{R} = \frac{h}{2d^2}, \quad (2.28)$$

where R is the radius of the curvature, h is the height of the buckled film, and d is the diameter of the buckled area. This estimation gives 4 mm for the radius of curvature.

The buckled film with a 4 mm radius of curvature acts as a negative lens with a 2 mm focal length. The film forms a strong lens, which is clearly visible under the optical microscope, Fig. 2.5. The propagation of the interface cracks is equally easy to detect by an optical microscope.

The interface crack propagated over different distances depending on the level of the applied transient stress. The initial crack shown in Fig. 2.12 can grow to the size shown in Fig. 2.24(a) for a lower stress level, and to the size shown in Fig. 2.24(b), for higher stress amplitudes.

It is well known in the thin film deposition technology [57] that films deposited by sputtering and e-beam deposition methods have a columnar microstructure. During deposition the grains of the film grow in columns from the substrate surface to the free surface. The experiments have shown that the intergranular interfaces of the film attract a propagating interface crack. As a result, the buckled film under dynamic loading in many cases spalls off from the substrate. Fig. 2.25 shows the deflection of the crack from the substrate/film interface to an intergranular interface. When the deflections occur at both sides of the buckled film, the buckled film spalls off from the substrate. This is shown in Fig. 2.26 which shows an electron microscope image of the spalled film. The investigation of the spalled off areas by optical and electron microscopy, along with the use of X-ray analysis have shown that the microcrack propagates along the interface between the lower test-layer and the substrate.

Figure 2.27 shows the results of an X-ray microanalysis that was done on the sample before (a) and after (b) spallation of a 1 μm thick Nb/Cr bi-layer from the sapphire substrate. The Nb film was deposited on top of the 100 Å thick test layer of Cr. X-ray measurements used a Si(Li) detector of a Link eXL system which was installed in a Zeiss DSM962 electron microscope. The X-ray spectrum of the sample before spallation (Fig. 2.27(a)) shows two strong peaks that correspond to K_α lines of Nb (2.166 KeV) and Al (1.487 KeV). The small peak corresponds to the K_α line of Cr (5.414 KeV). The X-ray analysis detected in this case a large quantity of Nb (1 μm thick film) and Al (sapphire substrate), and indicated only a small amount of Cr, which is due to the presence of a 100 Å thick layer of Cr underneath the Nb film. Dotted line (b) in Fig. 2.27 is the X-ray spectrum measured at the same location of the sample but after the Nb/Cr bi-layer has been spalled off. The spectrum in this case contains a strong Al peak which is caused by the sapphire substrate. The spectrum does not have a visible Cr peak. Further, the micrograph shown in Fig. 2.26 and other micrographs taken under higher resolution did not show any trace of the coating material on the surface of the sapphire. Thus, the

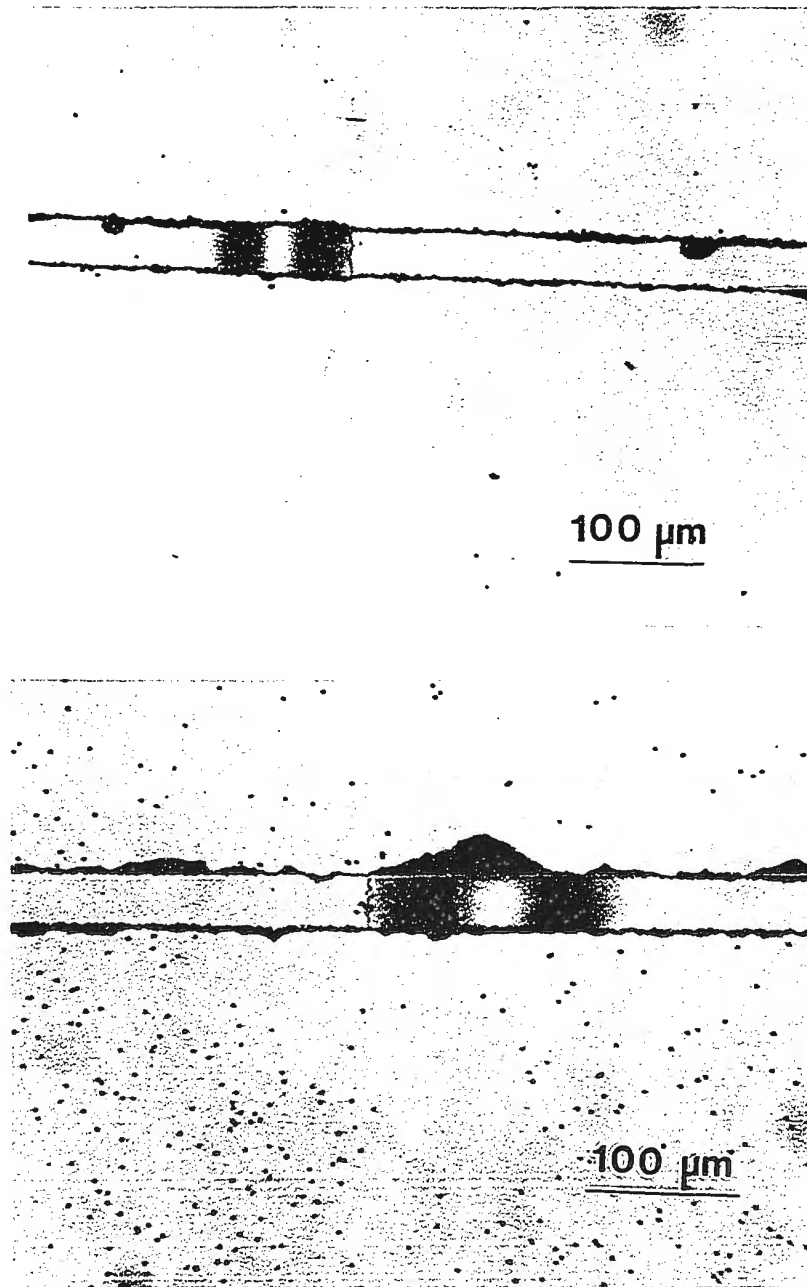


Figure 2.24: Propagation of an interface crack under a low (a) and high (b) amplitude of transient loading.

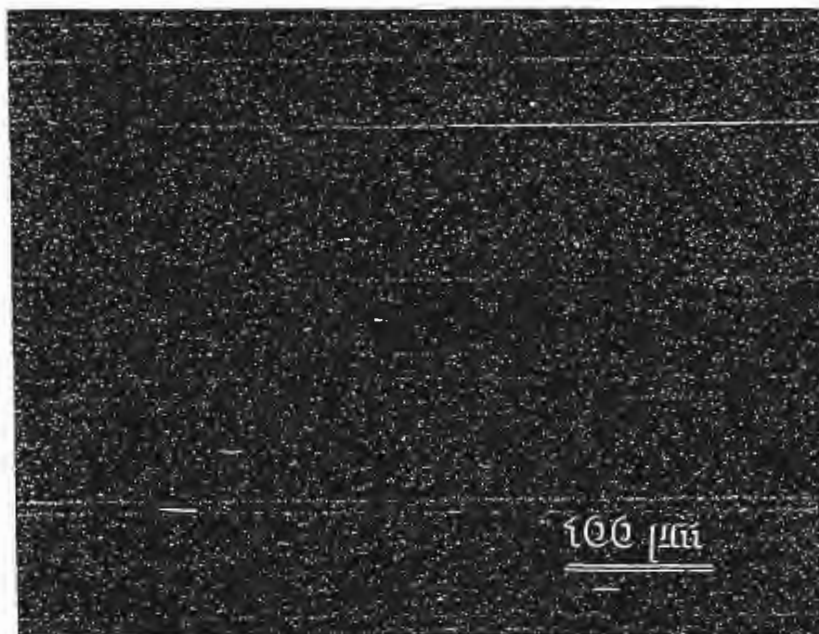


Figure 2.25: Deflection of the crack from the film/substrate interface to the intergranular interface.

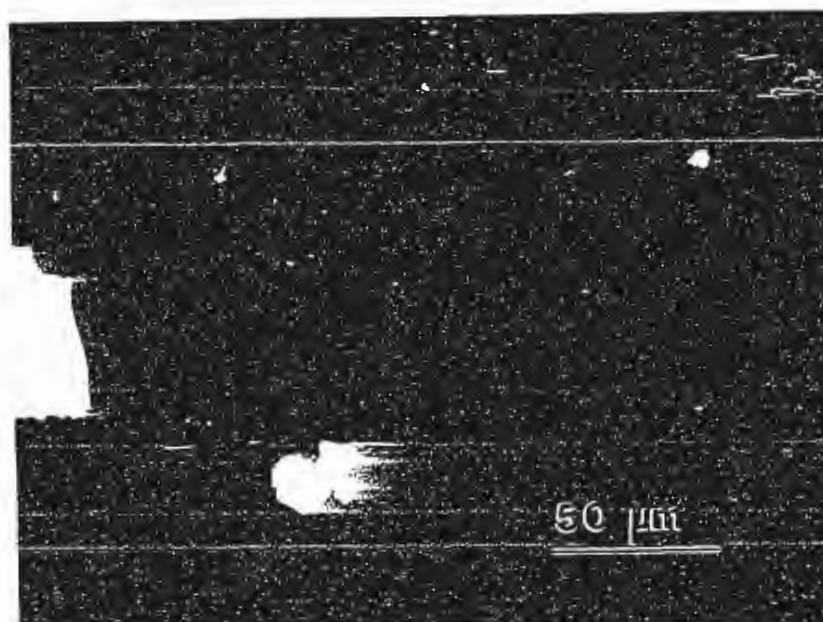


Figure 2.26: A scanning electron micrograph showing a spalled off Nb coating. The initial length of the buckled "bridge" was equal to 50 μm .

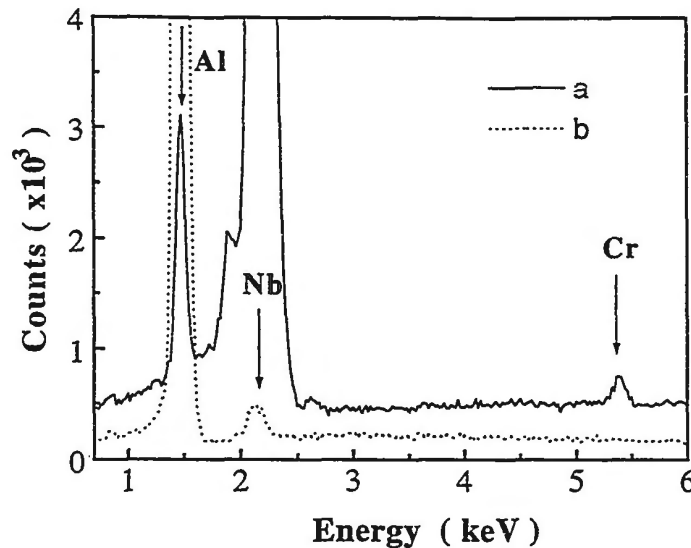


Figure 2.27: X-ray microanalysis spectra of Nb/Cr/sapphire system measured before (a) and after (b) spallation.

crack propagation occurs exactly at the test layer/substrate interface. Furthermore, no visual traces of plastic deformation either in the form of void growth or plastic tearing at the spalled edges were detected. Figure 2.28 shows a high resolution electron micrograph of the edge of the spalled film. The micrograph shows more or less a brittle-type fracture within the film without any plastic tearing. The surface of the substrate is also free of any metal. The shadowed area underneath the film shown by an arrow indicates the presence of an interface crack. The brittle-type fracture confirms the assumption made previously, when discussing the numerical model, that the plastic part of the interface toughness can be largely neglected in our experiments. One must however take this conclusion as one made on simplistic continuum arguments. The plasticity effects at the crack tip must be determined by local nucleation of dislocations within the coating from the crack tip. The details of the dislocation dynamics can only be obtained by a detail TEM analysis of the failed Nb coating. This was not pursued here.

Calculation of the interface toughness was done for each interfacial system based on the critical velocity history that lead to initiation of interface crack from its initial position. The initial position of the interface crack obtained by the second micro-lithography technique (Section 2.2.2) is shown by an electron micrograph in Fig. 2.29. These images were taken by using an electron beam that was inclined at 20° to the surface, giving a good side-view of the tested structures. The initial position of the interface crack tip extends a little further from its original position at the step, which is also visible in Fig. 2.29

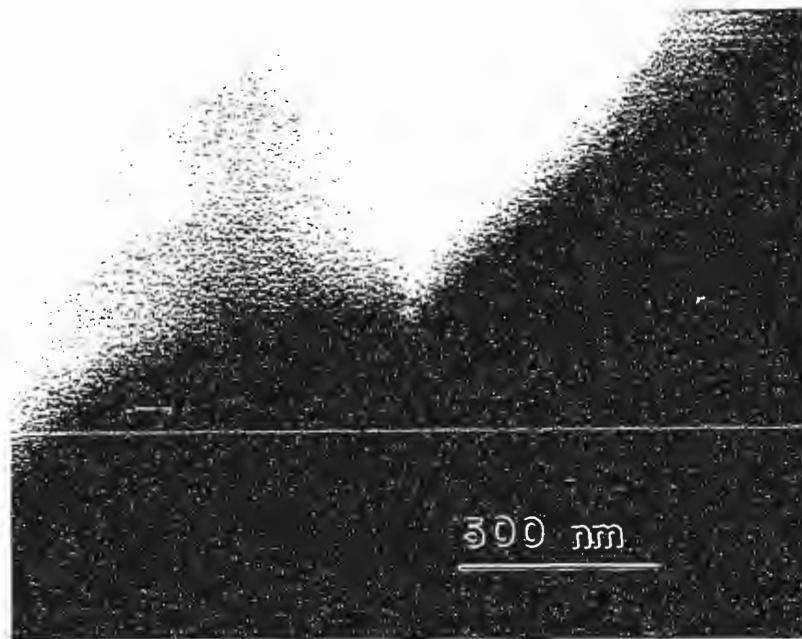


Figure 2.28: An electron micrograph of the film edge. The film/substrate interface crack is shown by an arrow. Electron beam was inclined at 20° to the sample's surface.

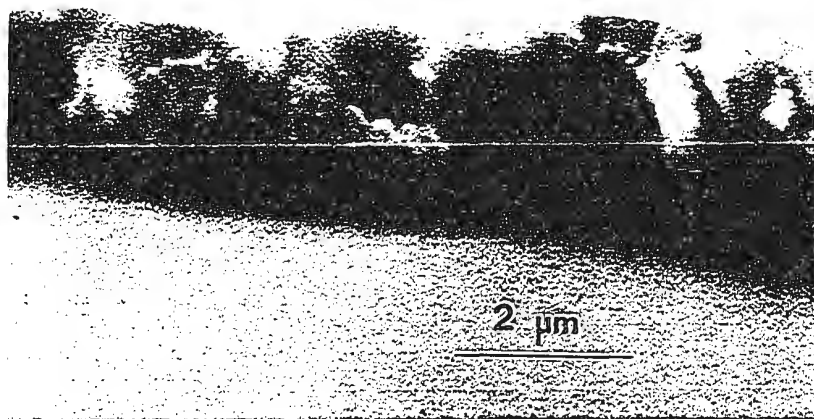


Figure 2.29: A side view of the film structure as viewed under an electron microscope. The electron beam was inclined at angle of 20° to the surface. The step is shown by an arrow.

and is shown by an arrow. The propagation of the crack from its initial position of the step occurred during the preparation of the structure itself and was caused by the residual stresses in the film. This stress provided a creep-type pre-loading of the crack, which is the standard fracture mechanics method to obtain an atomically sharp crack tip prior to mechanical loading. The subsequent dynamic loading caused an additional propagation of the crack tip. A side view of the crack tip after a successful dynamic loading is shown in Fig. 2.30. The fracture takes place along the film/substrate interface. The overall fracture pattern in the figure is fairly consistent with brittle fracture.

Fracture toughness was calculated for conditions of critical loading, specified in turn by the measured free surface velocity. The velocity was measured at places located far away from the tested microstructures, so the disturbances in the velocity field created by the crack tip did not influence the measurements. The stress amplitude required to start the crack propagation was measured to be significantly less than the amplitude required to cause the spallation of the film without any microcracks. This could be explained in the following way: to spall a film without the presence of any artificial interface cracks, the stress level should be as high as the stress needed either to overcome the ideal tensile stress (the case of flaw-free interfaces, Table 2-I) or to be high enough to start a number of interfacial cracks from sub-micron size flaws (Table 2-II). Due to relatively low surface velocity, the interference fringe record contains just a few fringes, Fig. 2.31(a). Recovery of the free surface velocity was done by using Eq. 1.11 through the closest fit of the expected velocity profile ($V(t) = C[e^{-t/A} - e^{-t/B}]$) to the measured fringe record, Fig. 2.31(b).

The critical velocity was determined in the above described way for three metal/sapphire systems containing test layers of Al, Cr, and Sb on sapphire substrate disks. The transient J-integral was calculated using the finite element ABAQUS program for these cases. The corresponding transient J-Integral profiles are shown in Fig. 2.32. The calculated energy release profiles for the systems are not similar to each other and the peaks were reached at different time instants. This is due to the different degree of the geometrical nonlinearity at different levels of loading. Stronger the load, a larger part of the film gets involved in the motion, and at later times, the stress intensity factor at the crack tip reaches a maximum. It should be noted again that the energy release rate data are very sensitive to the velocity, which is proportional up to the fourth power of the velocity. This might lead to relatively significant errors in calculations. Usually acceptable 10% accuracy of the velocity determination in the previous spallation experiments could lead to 50% error in the J-Integral calculations at high velocities. Nevertheless, the interface toughness value obtained for three systems were given in the Table 3-I.

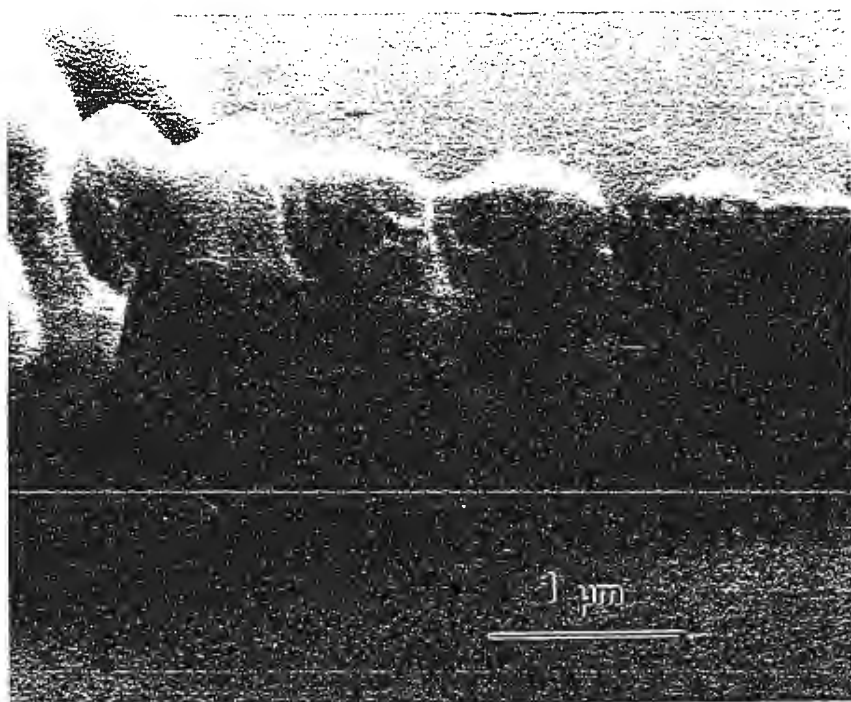


Figure 2.30: An electron micrograph of the crack-tip after fracture initiation by a transient stress pulse.

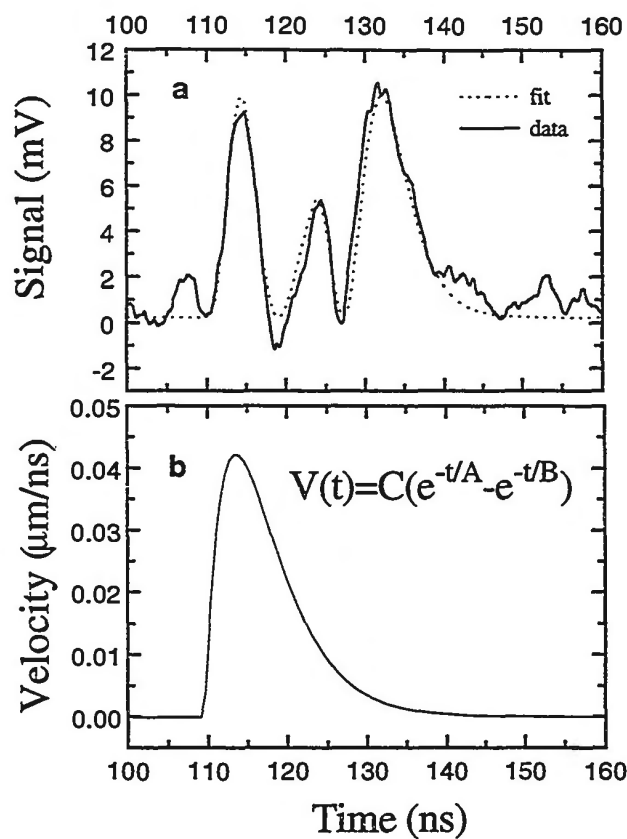


Figure 2.31: The closest fit of the expected fringe record (dotted line, (a)) to the measured fringe record (solid line, (a)) based on the variations of the expected velocity (b) with parameters A , B , and C .

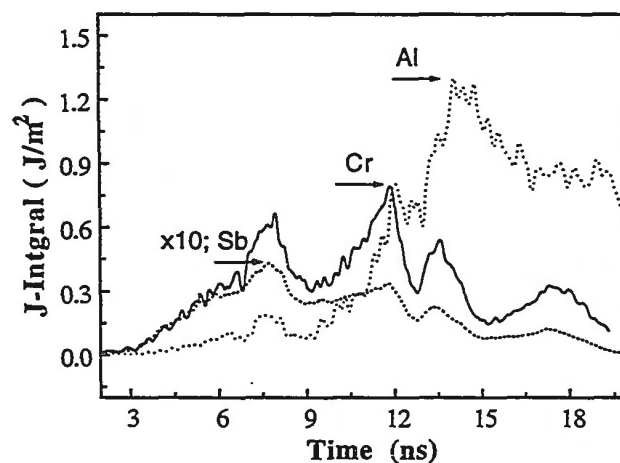


Figure 2.32: Transient J-Integral profiles corresponding to the critical dynamic loading for interfaces between sapphire substrate and coatings of Al, Sb, and Cr.

The interface energy values for the above systems obtained by the spallation technique may be compared to the values obtained independently by a spontaneous delamination experiment, as shown in Table 3-I (Chapter 3). The Al,Cr/sapphire interface energy values obtained in these experiments are close to each other. However, the value for the Sb/sapphire interface energy obtained by the spallation experiment is much lower. This may be explained by the lower role of plasticity at high strain rate loading.

The comparison of the independently obtained interface energy values obtained from the spallation experiment data shows the consistency of the results and proves that the previously made assumption of the negligible role of the plastic deformation in spallation experiments. In spite of the microscopic scale ($\sim 1 \mu m$) of the model, high strain rate loading makes it possible to apply a SSY (small-scale-yielding) approach to the spallation of the film.

Besides using the above technique to measure the fracture toughness of interface, it can be also used to understand the rather low strength values for certain metal/PG interfaces recorded previously, see Table 2-II. Assuming a flawless interface, an estimation of the interface toughness for these systems using Eq. 2.1 gives $7 \times 10^{-6} J/m^2$, $2 \times 10^{-6} J/m^2$, $27 \times 10^{-6} J/m^2$ for Cu, Sb and Sn/PG interfaces, respectively [11]. These toughness values are unreasonably small. Therefore, as mentioned earlier, these small values may be due to the presence of interfacial flaws. The toughness values ($2\gamma_{int}$) cannot be estimated in the general case using the standard fracture mechanics relationship for static loading as

$$2\gamma_{int} \sim \frac{K^2}{E} \sim \frac{\sigma^2 2a}{E}, \quad (2.29)$$

where K is the stress intensity factor, E is the Young's modulus, σ is the interface stress, and $2a$ is the flaw size. Thus it was of interest to see if reasonably sized interface microcrack would explain the low strength values for these systems. To this end, the numerical model simulated dynamic loading of a flawed Cu/PG interface with negligibly small initial crack opening, Fig. 2.33. Calculations were done for two thicknesses of the Cu film (h , Fig. 2.33), 1 and 3 μm . The duration (Δt) and the stress pulse profile were measured experimentally [11]. The purpose of the analysis was to determine the influence of the possible flaw size ($2a$) on the toughness at a given interface stress history. Also it was interesting to understand the dependence of the interface toughness on the film thickness.

Figure 2.34 shows the time history of the J-Integral for different flaw sizes. The stress pulse profile and the maximum of the transient interface stress ($\sigma = 15 MPa$) far away from the crack tip were chosen to be the same for all flaw sizes. Figure 2.35 shows the

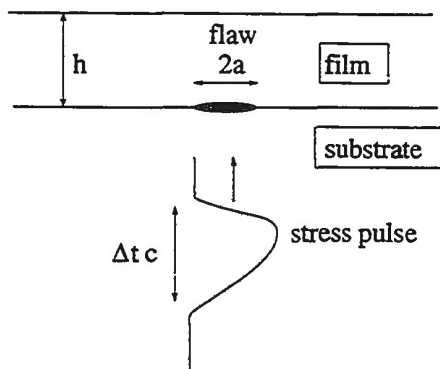


Figure 2.33: Schematic of the flawed film/substrate interface system used in the numerical modeling of the metal/PG systems.

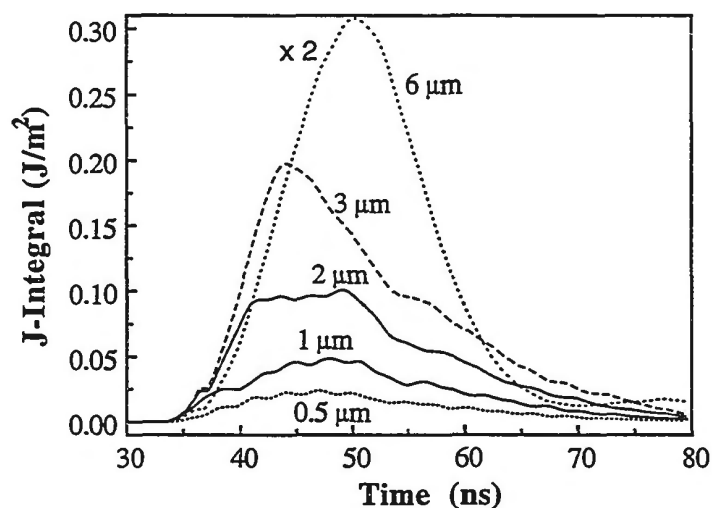


Figure 2.34: The J-Integral time history for different flaw sizes. Thickness of the Cu film for these calculations was fixed at $3 \mu\text{m}$.

dependence of the J-Integral maximum on the flaw size for the two film thicknesses, 1 and $3 \mu\text{m}$. In the case when a is much smaller than both h and Δtc , the numerical solution for dynamic loading should be very close to the solution for static loading of the interface flaw. This explains why at small flaw sizes, the J-Integral is proportional to a as

$$J = \frac{\sigma^2 a}{E_{eff}}, \quad (2.30)$$

where E_{eff} is the “effective” Young’s modulus of the Cu/PG interface system. E_{eff} was calculated to be equal to 4.3 GPa (Fig. 2.35), which lies between the Young’s modulus of Cu (120 GPa) and PG (2.7 GPa , E_2 , softest direction which is normal to the free surface). It is easy to note that the “effective” Young’s modulus is much closer to the

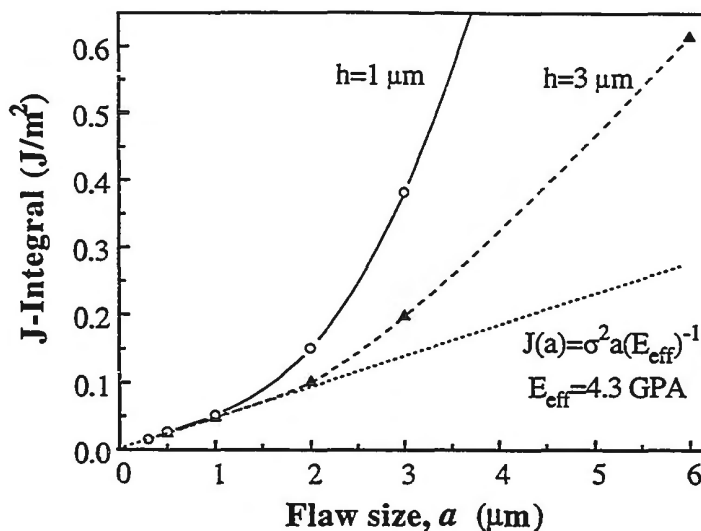


Figure 2.35: The dependence of the maximum J-Integral on the flaw size.

modulus of the softer material (PG), indicating the fact that the most elastic energy around the crack tip is stored in the more compliant material. The length of the stress pulse ($\Delta t c \sim 40 \mu\text{m}$) in PG substrate was several times bigger than the film thickness ($1\text{-}3 \mu\text{m}$) and the flaw size (ranged up to $6 \mu\text{m}$). This simplified the model, allowing elimination of the transient wave effects in the J-Integral time dependence.

As soon as the flaw size becomes bigger than the thickness of the film, the J-Integral is no longer proportional to a , Fig. 2.35. Therefore, for the interface systems when the average size of the intrinsic flaws is bigger than the thickness of the film, it should be expected that the laser spallation experiment will yield small interface strength values. The interface toughness value of those systems has to be numerically calculated based on the knowledge of the interface flaw population. The calculated value might be much bigger than the toughness estimated by Eq. 2.30.

Chapter 3

A New Technique to Measure the Toughness of Thin Film Interfaces

3.1 Introduction

In recent years there has been an active interest in determining the mechanical properties of thin film interfaces appearing in composites, microelectronic devices, tribological, and other thin film applications. Although sophisticated methods [11,13] have been developed to obtain the intrinsic tensile strength of the interface (if the interface is free of flaws), there are many others which provide a measure of the total interface fracture energy (G) including plastic deformation within the coating and/or substrate that occurs during the interface separation process. Accordingly, the measured values depend upon the load phase angle Ψ [58], and are of engineering importance. A challenge, however, is to measure the intrinsic toughness of the interface, because it scales the entire $G - \Psi$ curve, and allows a more fundamental representation of the interface. In this strategy, the determination of the plastic deformation and its dependence on the actual load phase-angle becomes a part of the overall boundary value problem, of which the interface is a part. An indirect strategy for estimating the intrinsic toughness involves obtaining the intrinsic tensile strength by the laser spallation experiment [13], and relating it to the intrinsic toughness through an assumed interface stress-separation relationship [11]. The laser spallation experiment was used as well for direct measurements of the interface fracture toughness, as has been discussed previously in Chapter 2. Because the interface decohesion occurs at a strain rate of almost 10^7 sec^{-1} , the plastic deformation within the separated components is largely minimized, and a measure of the intrinsic strength

is obtained, provided the interface is free of flaws.

An alternative direct approach is to use the residual stress within the coating itself to cause its separation from the underlying substrate. Because of the local character of the loading, the plastic dissipation during delamination is minimized, and the measured values approach that of the intrinsic toughness of the interface. The effective use of the naturally occurring residual stress in thin films to cause delaminations from substrates, and their quantification to obtain the interfacial energy, was first shown by Argon *et al.* [59] on SiC/Si and SiC/C systems. Specifically, for plasma-assisted CVD SiC coatings deposited on Si wafers and Pitch-55 carbon fibers, toughness values of 5.5 J/m^2 and 5.4 J/m^2 were obtained, respectively. If the residual stress in the deposits is high enough (usually in the *GPa*-range) to cause delamination under tensile stress or buckling under compressive stress, then the theoretical treatment given in Argon *et al.* [59] and Hutchinson and Suo [60] can be used to obtain the interface toughness.

In several thin film systems, however, including metallic coatings on ceramic substrates, the residual stress is too low (50 *MPa* to 100 *MPa*) to allow the spontaneous delamination phenomenon described above. A new technique based on residual stress-induced controlled film delaminations has been developed, and presented here, to measure the fracture toughness of such interfaces. A test coating of 100 Å to 400 Å thickness is deposited on the substrate of interest. This coating is overlaid by a residually-stressed Nb coating (used as a loading device) using sputter deposition. Since the Nb coating is under compressive stress, its thickness is controlled until the lower coating buckles away from the substrate, i.e., at the interface whose energy is of interest. Since the thickness of the test coating is typically two orders of magnitude smaller than that of the Nb layer, the elastic strain energy entrapped within it can be neglected in comparison to that within the Nb film. Thus, it is only necessary to characterize the stress state in the Nb, which in turn, is obtained by measuring the intrinsic misfit strain from independent delamination experiments. The new technique is demonstrated on interfaces between metallic coatings of Cr, Sb, Nb, Cu, and Al and substrates of sapphire with an (0001) orientation. Recently, an article by Bagchi *et al.* [61] appeared in the literature, which essentially uses the same concept to determine the interfacial energies. However, as discussed in the discussion section of this chapter, the procedure adopted here is quite different.

3.2 Experimental Procedure and Observations

All test coatings and the Nb loading-layer were deposited by a sputtering system, which consists of two 50.8 mm diameter magnetron sputtering guns, a 1000-watt DC power supply, a 600-watt RF power supply with a matching network, and a backspattering gun for cleaning the substrate surface prior to the deposition. Depositions were performed in a cryo-pumped system with an ultimate base pressure of 2×10^{-7} Torr. The background argon pressure was 10 mTorr. The sapphire substrates were cleaned chemically with trichloroethane, acetone, and methanol, rinsed with de-ionized water, and dried with ultra-pure N₂ gas, followed by backspattering cleaning in the sputter deposition chamber for 20 minutes. Metallic films of Cr, Sb, Nb, Cu, and Al 400 Å-thick were deposited on the (0001) plane of sapphire, followed by a prescribed thickness of Nb coating at ambient conditions, using the second sputtering gun. This procedure avoids any chemical reactions between the Nb and the test coating, as shown by the reaction-free Cr/Nb and Sb/Nb interfaces observed using a high-resolution transmission electron microscope [11, 62]. Furthermore, the residual compressive stress within the Nb was high enough to cause the buckling of all test coatings from their respective substrate discs. The deposition rate and the thickness of the films were monitored by an *in-situ* quartz monitor. The accuracy of the thickness measurement was checked by comparing the quartz sensor data with those obtained by the profilometer. The discrepancy of data was within 3 %. Depending upon the test coating, the critical thickness of the Nb layer that resulted in the buckling of the bilayer ranged from 0.3 μm to 1 μm , (Table 3-I). The accuracy of the critical thickness

Table 3-I: Toughness of Several Metal/Sapphire Interfaces as Obtained from the Spontaneous Delamination Technique

Interface Systems	Critical Nb Film Thickness (μm)	Interface Toughness (J/m^2)	Bending Contribution to G_0 in %	Dynamic J-Integral (J/m^2)
Al/sapphire	1.0 ± 0.05	1.03 ± 0.05	0.5	1.3
Sb/sapphire	0.3 ± 0.03	0.31 ± 0.03	0.2	0.04
Nb/sapphire	0.85 ± 0.05	0.88 ± 0.05	0.6	
Cu/sapphire	0.90 ± 0.05	0.93 ± 0.05	0.5	
Cr/sapphire	0.91 ± 0.05	0.94 ± 0.05	0.5	0.8

measurement was determined by the used procedure of the incremental film deposition and was different in each case, as shown in Table 3-I. Typical buckled morphologies of Sb/Nb and Cu/Nb bilayers are shown in Figs. 3.1(a) and (b), respectively. Except

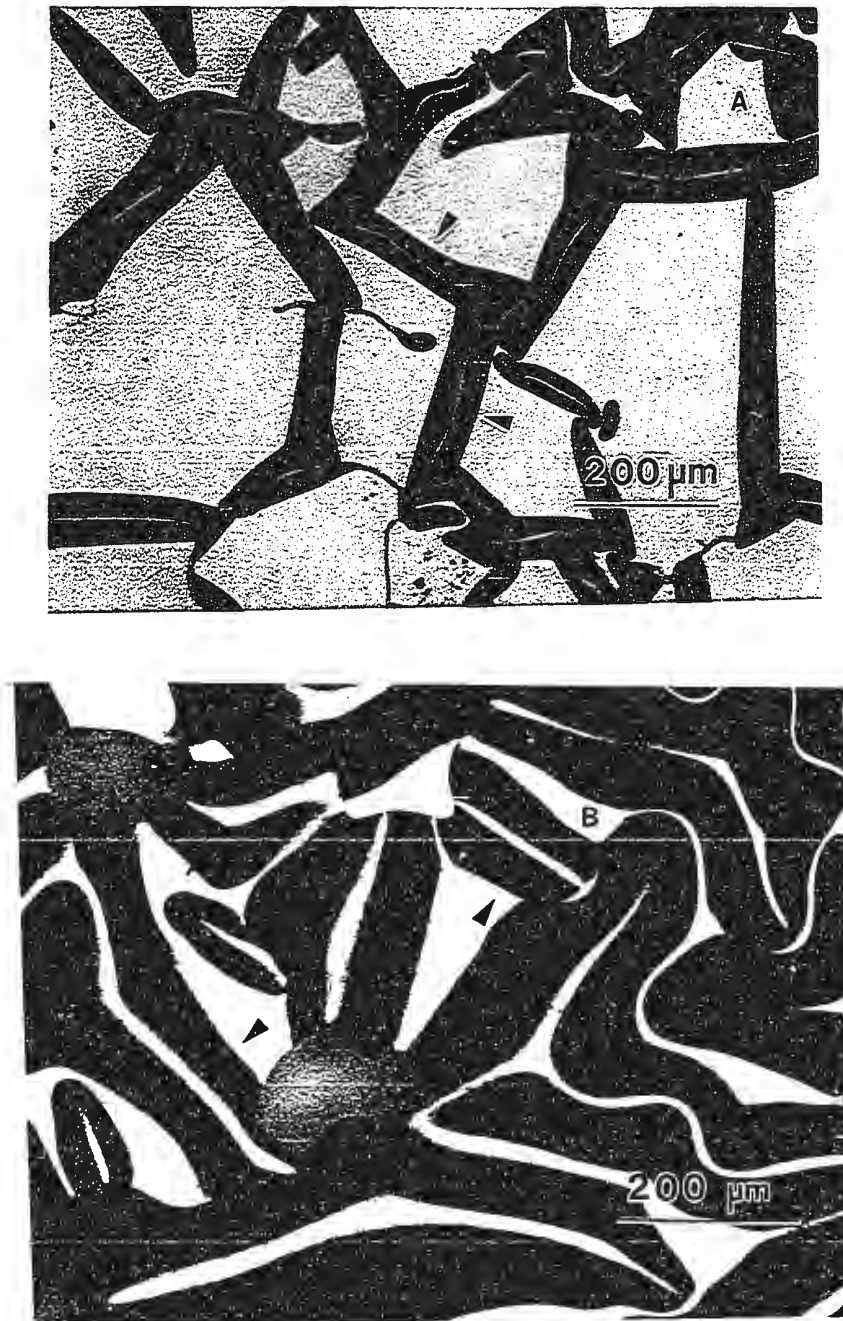


Figure 3.1: A micrograph showing typical buckled morphologies in Sb/Nb (a) and Cu/Nb (b) bilayers.

for the amplitude of the buckled wave front, all bilayers studied here showed similar morphologies. In all cases, the Nb/test coating interface was intact upon delamination of the test coating from the substrate. Because of the transparent nature of the sapphire, it was relatively easy to confirm the buckling of the lower test layer by simply viewing the assembly from the free sapphire surface.

3.3 Calculation of the Interfacial Energies

Quantification of the above mechanism requires: (a) determination of the residual stress in the Nb layer, which then allows us to estimate the strain energy in the bilayer at the critical Nb thickness, and (b) the determination of the strain energy entrapped within the buckled bilayer so that the difference of it from that calculated in (a) provides a value for the interface fracture toughness. The strain energy within the test coating can be neglected since its thickness is almost two orders of magnitude smaller than that for the Nb layer. This was also confirmed through independent experiments where, by reducing the thickness of the test layer to 200 Å, no change in the critical Nb layer thickness was observed.

(a) *Stress in the Nb coating:* The intrinsic strain in the Nb coating was determined from independent delamination experiments, where a Nb coating was delaminated from a sapphire substrate. The as-deposited Nb coatings are under biaxial compression. By using photolithographic techniques, Nb coatings were deposited in the form of strips 100 μm wide, and with a rectangular cross-section as shown in Fig. 3.2.

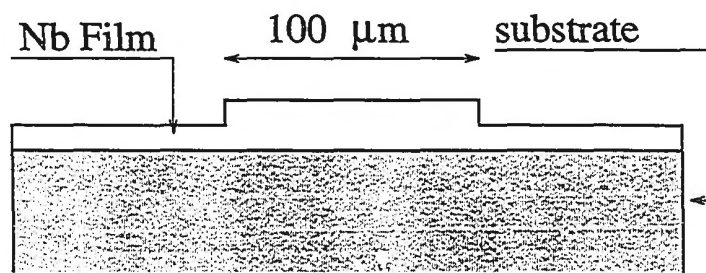


Figure 3.2: A schematic of the Nb coating pattern obtained using photolithographic techniques in order to induce a one-dimensional buckle front.

The total thickness of the coating at its crests was controlled to the critical thickness needed for delamination, whereas at the troughs, the strip thickness was maintained at only one-half the critical thickness. As shown in Fig. 3.3 this geometry forces the Nb coating to buckle only along its width direction, while remaining attached to the substrate

at the troughs. Due to this buckling, the residual strain is completely relieved along the strip width, whereas it remains at its original level along its length. By measuring the one-dimensional post-buckled profile using a mechanical profilometer (Fig. 3.4) or more accurately by an atomic force microscope, the intrinsic strain ε_0 can be computed from

$$\varepsilon_r = \varepsilon_p + (1 + \nu)\varepsilon_0. \quad (3.1)$$

Here ε_p is the residual strain in the post-buckled film along its width direction, and ε_r is the strain that is released upon buckling. This strain can be simply calculated by taking the difference of the initial unbuckled length $2L$ of the strip (Fig. 3.4) from its relaxed length upon buckling as

$$\varepsilon_r = \frac{1}{2L} \left[\int_{-L}^L \sqrt{1 + y'^2(x)} - 1 \right] \simeq \frac{1}{4L} \int_{-L}^L y'^2(x) dx, \quad (3.2)$$

where $y(x)$ is a polynomial curve fit to the buckled profile in Fig. 3.4 and the prime on y indicates its differential. If the buckled length $2L$ is much greater than the thickness of the coating, then essentially all the strain along the width direction is relaxed and Eqs. 3.1 & 3.2 give

$$\varepsilon_0 = \left(\frac{1}{1 + \nu} \right) \frac{1}{4L} \int_{-L}^L y'^2(x) dx. \quad (3.3)$$

An average value of 4.1×10^{-3} was obtained for the intrinsic strain in Nb over five delamination experiments. All measured values fell between 3.5×10^{-3} and 4.5×10^{-3} . Once ε_0 is obtained, the strain energy G_i entrapped within a thickness t of the Nb coating can be readily computed from

$$G_i = t \frac{E\varepsilon_0^2}{1 - \nu}, \quad (3.4)$$

where E is the Young's modulus, and ν is the Poisson's ratio for the Nb film.

(b) *Estimation of the strain energy in the post-buckled shape:* A closer look at micrographs of the buckled bilayers shows a non-unique buckle wavelength despite the same intrinsic strain. The presence of this diverse wavelength-set on the same micrograph (same intrinsic strain) in a telephone-chord-like buckled morphology does not follow the predictions of a recent theory [47] dealing with the stability of such buckle fronts. For the systems studied here, it may be necessary to consider the inelasticity in such formalisms to explain these observations. For the purpose of the discussion here, however, these issues are not relevant. It is only necessary to find locations on the micrograph where the coating has buckled in a one-dimensional morphology with a straight edge, as marked with arrows in Fig. 3.1. For all the systems shown in Table 3-I, we were able to find such regions in the buckled bilayer morphology. Interestingly, in some cases, only straight

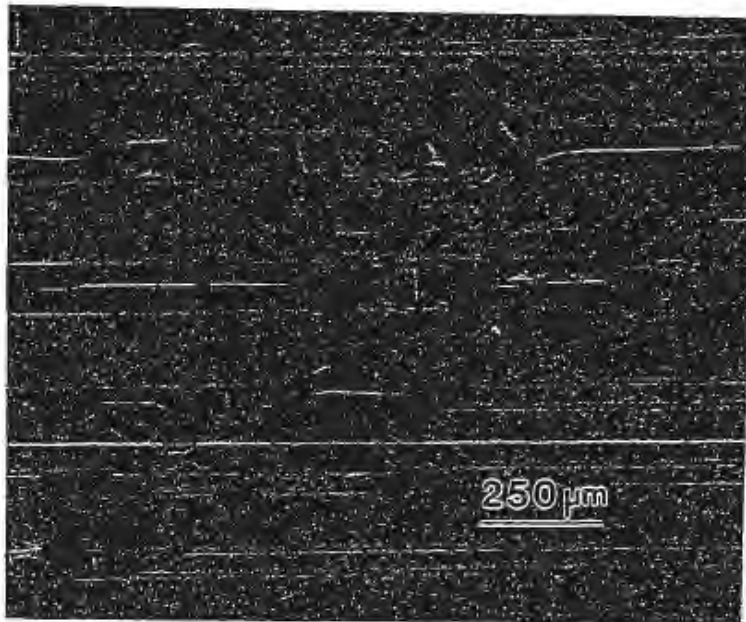


Figure 3.3: A micrograph showing one-dimensional buckled morphologies in an Nb coating deposited using photolithographic techniques.

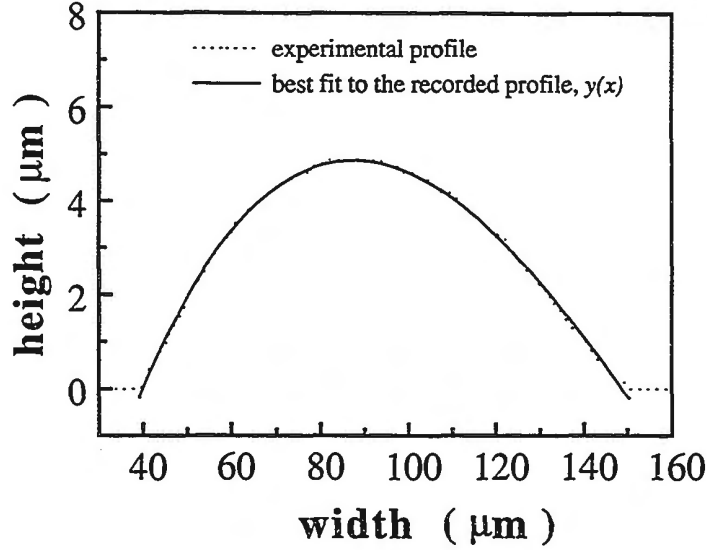


Figure 3.4: A one-dimensional post-buckled profile of the Nb film obtained from a mechanical profilometer. The solid line represents a polynomial fit to the measured profile (dashed curve).

edge buckle fronts were observed, as shown in Fig. 3.5 for the Sb/Nb system. Assuming that the strain in the direction of buckling is completely relieved, the unrelaxed strain energy on account of the remaining residual stress in the other direction is given by

$$G_f = \frac{1}{2} t E \epsilon_0^2. \quad (3.5)$$

Neglecting the strain energy in the test layer, the energy release rate G_0 for the interface can be readily obtained by subtracting Eq. 3.5 from Eq. 3.4 as

$$G_0 = G_i - G_f = \frac{1 + \nu}{2(1 - \nu)} t E \epsilon_0^2. \quad (3.6)$$

In the above we have neglected the strain energy in the post-buckled shape due to the bending of the bilayer. When this effect is considered, Eq. 3.6 takes the following form [60]

$$G_0 = \frac{(1 - \nu^2)t}{2E} (\sigma - \sigma_c)(\sigma + 3\sigma_c), \quad (3.7)$$

where σ is the residual compressive stress in the film prior to the buckling, and σ_c is the classical buckling stress of a very wide plate of length $2L$ and thickness t when clamped along its edges. σ_c is given by

$$\sigma_c = \frac{\pi^2}{12} \frac{E}{1 - \nu^2} \left(\frac{t}{L} \right)^2. \quad (3.8)$$

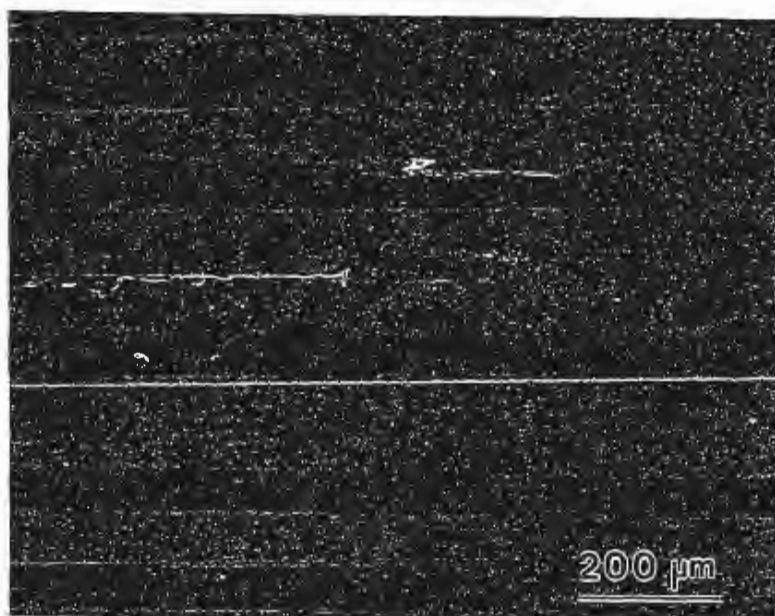


Figure 3.5: Naturally occurring straight edge one-dimensional buckled profiles in an Sb/Nb bilayer.

The values of interface toughness calculated by using the above Eq. 3.8 are given in Table 3-I. These values were calculated by assuming $\nu = 0.38$, $E=103 \text{ GPa}$, and an average measured intrinsic strain of 4×10^{-3} . The bending contribution is usually less than 1 % of the total strain energy released and as shown in Table 3-I, this can be safely neglected.

3.4 Discussion

This technique is different in several aspects from that proposed by Bagchi *et al.* [61]. First, delamination here occurs under compressive residual stress, whereas they use the tensile residual stress to control delamination. Second, the residual stress within the loading layer (Nb) is obtained here by measuring the intrinsic strain rather than the wafer-curvature technique, which in turn introduces additional inaccuracies on account of the error in reading the coating thickness. This inaccuracy can potentially be removed from their technique if a measure of the intrinsic strain in the coating is obtained by looking for delaminated strips that lie flat on the substrate and have undergone a decrement in their lengths upon relief of the residual tensile stress. Such observations were made by Argon *et al.* [59] in annealed SiC coatings that were under residual tension. Third, it appears from our results that there is neither a need for introducing any interface crack-starter layer in our experiments, nor a need for patterning the coatings using photolithography to induce one-dimensional buckling. Instead, the natural buckled morphologies always present themselves as one-dimensional blisters at certain locations.

The spontaneous delamination technique now provides an additional measure of the interface toughness independent from the laser spallation technique, albeit under static-load conditions. The toughness values of several systems, including Al, Sb and Cr/sapphire (Table 3-I) were experimentally measured by both these techniques. The toughness values obtained for the Al/Al₂O₃ and Cr/Al₂O₃ systems are surprisingly close to each other. Since the plastic part of the toughness for the steady-state loading and high strain rate loading should be very different, it means that in both cases the plastic part should be small and the measured values must be quite close to the intrinsic interface energy for these systems. However, the very small value of interface toughness for Sb/Al₂O₃ in comparison to that obtained by the spontaneous delamination experiment is puzzling and cannot be explained at this moment.

3.5 Conclusions

The following procedure should be used to estimate the interfacial energies using the new technique presented here.

(a) Estimate the intrinsic strain in the Nb loading-layer by applying Eq. 3.3 to its one-dimensional buckled morphology, as obtained by independent experiments. Since this value depends upon the specific deposition conditions, this should be measured rather than choosing the value given here.

(b) Next, deposit a 200 Å-to-400 Å-thick layer of the test coating on the substrate, followed by a controlled deposition of the Nb layer until the bilayer buckles. This may require several attempts. Look for a straight edge buckle front on the micrograph and determine its profile using an atomic force microscope or simply by a mechanical profilometer.

(c) Finally, by assuming the Young's modulus E and the Poisson's ratio ν for the coating material, determine the interface toughness using Eq. 3.6.

(d) Users are also encouraged to devise their own loading coatings that are likely to entrap larger residual stress.

Chapter 4

Mechanisms and Quantification of Spalling Failure in Laminated Composites Under Shock Loading

4.1 Introduction

The initial development of the laser spallation experiment discussed earlier was motivated by the desire to measure the tensile strength of interface between fibers and their protective coatings [3,59]. However, most developments to date, including that discussed in previous chapters, are limited to testing of only planar interfacial geometries in laser spallation assembly. Such planar geometries limit testing of real composite interfaces, where residual stresses on account of fiber geometries and composite processing steps could be important. Here we explore the possibility of actual composite microstructure testing by using the laser spallation experiment.

To this end, 2D woven carbon/carbon (C/C) and carbon/polyimide (C/P) composites were subjected to compressive stress pulses by impinging a Nd:YAG laser pulse on the back surface of a laminate sample, and made to propagate normal to the plies. The compressive stress pulse propagates through the composite sample and upon reflection into a tensile pulse from the sample's front surface, spalls the material at the interply interface that is nearest to the free surface, given a sufficiently high amplitude. The profile of the stress pulse which causes spallation was measured by recording the free-surface velocity by a wide-angle interferometer ([16] and section 1.6 in Chapter 1) fringes even from a geometrically incoherent light, as expected after reflecting from a rough composite surface. Here and throughout the chapter, the word "spall" is used to indicate that the material is completely removed or flies off from the parent material. Since the stress wave

in our experiments is generated only over a part (3 mm-diameter area) of the composite plate, a higher amplitude of the input compression pulse is necessary to completely remove the material since the nucleated sub-surface crack has to break through the overlaying fiber weaves at the edges of the stressed area. Certainly, the sub-surface crack should nucleate at much lower stress amplitudes (or laser energies) and this is taken here as the local failure criterion that initiates a debond in an otherwise undamaged material. As discussed in detail later, the damage starts in the form of fiber/matrix delaminations either in the bundles within a ply or at the interply interface. Sometimes the failure locus enveloped both the above sites. Thus it is possible to measure the interface strength between fibers and matrix as that between fibers and coatings if the recorded free-surface velocity can be related to the local interface stress by a computer simulation of the type discussed in Chapter 2. Here, the simulation should include the actual geometry of the fibers, composite microstructure, and residual stress. This was not pursued in this thesis work. Instead, we focused on a failure mechanisms and resistance of C/C and C/P laminates under high strain rate loading. The rest of the chapter is oriented towards this goal and discusses the failure criteria, and dispersion and attenuation characteristics that are associated with the propagation of such high rate stress pulses through the composite microstructure. Obtaining such an information suffices to design composite structures against high rate impacts.

Although the failure on the microscale is in the form of matrix and fiber/matrix interface delaminations, the stress pulse amplitude responsible for such damage on the continuum scale is referred to here as the effective interply strength. Since by employing a special specimen geometry we could determine the onset of such damage, its characterization in terms of an effective interply strength should be acceptable. This was achieved by patterning the free surface of the sample in a square grid, with grooved edges which apparently do not lead to any significant stress concentrations (as discussed later). By keeping the grid area smaller than the laser heating spot, the square coupon could be completely separated at the threshold stress amplitude with no additional energy required to break the fiber bundles. This provides a direct measurement of the effective interply tensile strength. Henceforth we will drop the word "effective" and use interply strength to quantify the onset of damage.

The initiation of the damage discussed above can be characterized using a stress criterion, independent of the pulse width. However, the subsequent growth of such initial debonds under additional stress pulses should be dependent on both the amplitude and duration of the stress pulses, especially when the debond lengths are too large to permit a quasi-static loading of the faces by disallowing several traverses of the stress wave along

the crack surfaces. There is a vast literature on this subject dealing with the dynamic growth of flaws of various sizes under stress wave loading (*e.g.*, see Curran *et al.* [4]). Although the growth of delaminations under subsequent loading is of interest in its own right, here we shall limit ourselves to the initiation of damage only since in many practical situations the subsequent or already existing static compressive, shear or tensile stresses can lead to failure through their inplane growth [63].

For the C/P composites with no pre-existing flaws, the recorded stress pulse amplitude at the threshold laser energy corresponds to the interply tensile strength. However, for the C/C composites with processing-induced material discontinuities, the measured strength reflects the far field stress required to grow the microstructural flaws. Because of the relatively small sizes of the flaws, one can essentially consider the measured value as the strength of a flawed continuum.

In the strategy adopted here, the first step in the design of a composite structure under impact loading is to determine the critical stress amplitude which nucleates a sub-surface crack under a uniform local stress. When these measurements are related to the composite microstructure, guidelines can be provided to the manufacturers for developing more impact-tolerant composites. This information is provided here for the C/C and C/P composites with identical weave architecture but with largely different matrices.

The above information is however insufficient to design the thickness of a composite part that may be subjected to an impact. To this end, it is necessary to know how the composite microstructure will change the amplitude and the profile of the stress pulse as it reaches the free surface to load the component. This information is best given in terms of the dispersion and attenuation characteristics of the stress pulse (of a given profile) as it propagates through the composite microstructure. These were obtained for each laminate-type experimentally, by propagating stress pulses of well-known profiles (obtained in separate experiments on Si crystals) with much lower amplitudes than those needed for spallation. Since the stress pulse duration is comparable to the pulse propagation time through the interply spacing, significant pulse distortion occurs due to multiple reflections from interply interfaces and scattering from material inhomogeneities in the form of fiber bundle curvatures and material discontinuities such as voids and cracks (only in C/C). The resulting distortion in both the peak amplitude and the profile of the pulse were recorded at the sample's free surface for different thicknesses of the sample by the wide-angle interferometer. As discussed in detail later, both the real and the complex part of the wave number were obtained by taking the ratios of the Fourier transforms

of the input and the free surface signals, which in turn, provides the dispersion and attenuation characteristics associated with the propagation of various frequencies inherent in the input stress pulse. Since the transient stress pulses have nanosecond rise times, the attenuation and dispersion associated with high frequencies (10-100 *MHz*-range) were obtained.

The above now suffices to address the main thesis of the chapter. There are few other phenomena that can be addressed within the current framework. For example, the above procedure provides a measure of the dilatational wave velocity through the material, and leads to the determination of an effective transverse modulus for the laminate from independently obtained values of the composite density. Such values are of interest in the micro-mechanical modeling of laminates. Further, since the stress pulse duration is quite small, low amplitude stress pulses can be used as a non-destructive evaluation (NDE) tool. We show this potential by using them to detect the onset of spalling damage in laminates.

The immediate utility of our study can be realized while considering the development of light-weight armor structures where dynamic stress waves can result due to an impact of a projectile. Delaminations in structural components (wings, nose cones and future composite turbine blades) of airplanes and the space shuttle upon impact from flying objects is a well known problem. In C/C composites, dynamic stresses can result through a sudden heating of the inner surface of the composite when used in exhaust cones and heat-shielding tiles in the space shuttle and rockets. Thus, an understanding of their behavior under dynamic loads is warranted for optimizing performances in current applications and for developing more impact-tolerant microstructures.

The chapter is organized in the following way. First we discuss the experimental setup to produce stress pulses and the interferometric technique to record them. This is followed by observations of the propagation characteristics of low amplitude stress pulses to obtain dispersion profiles, followed by the characterization of damage and the associated interply tensile strength. The above discussion-format allows us to demonstrate how low amplitude short stress pulses can be used to identify the initiation of interply damage, which is important in defining the interply strength.

4.2 Background Literature

The study of composite laminates under shock loading has been the subject of investigation for over two decades. The most recent studies [64] deal with the damage on the

front impact surface caused by the local tensile stresses, which in turn, depend upon the geometry and velocity of the impacting body. The damage is usually in the form of radial cracks emanating from the impact point. Although this is an important study in its own right, it is of little interest to the thesis advanced in this chapter. Early experiments on the fracture of 3D-carbon-carbon composites under dynamic loading were performed by Goeke and McClintock [65]. The samples were loaded using square stress pulses of approximately 0.2 to 2.0 μs in duration, and were generated along the fiber bundles threading the woven fabrics through the impact of flyer plates propelled by a magnetic field. Both the impacted and the rear surfaces of the sample showed damage in the form of several fiber/matrix debondings and failure of stress-pulse aligned threading bundles. The amplitude and duration of the stress pulses responsible for such failures were estimated numerically by converting the flyer plate velocities. The stress level causing such failures at both the impacted and the rear surface were dependent on the pulse widths. The purpose of the study was to provide insights into the local shock-related damage and its dependence on the pulse widths rather than to provide a general framework of design against shock failures. Contrary to this study, here we consider 2D laminates, and subject them to a well-characterized temporally-fixed stress pulse profiles.

The work of Gilath *et al.* [66] is directly related to the current research, as they too used laser-produced shock waves to study spalling damage in 2D C/C composites. They related the free-surface spallation damage to the laser energy densities with no direct measurements of the stress responsible for damage. Unfortunately, this does not allow for any proper quantification of the local stress state or the damage in the form of interply delaminations and fracture of overlaying fiber bundles. Because of the excessive energies necessary to completely spall the material, the distinction between the failure initiation and actual spall was ignored. In contrast to their and other's works, the work presented here provides a better characterization of the stress pulse amplitudes responsible for material damage by using interferometric techniques. Further, since the rise-time (1-2 ns) and duration (17-20 ns) of the generated stress pulse is comparable in spatial extent to that of the fiber bundles, it is possible to invoke a local material response with such pulses rather than a continuum one, as obtained in earlier studies with microsecond-long stress waves. Finally, the measurement of the dispersion and attenuation profiles provides the all-important information that is needed to use the sample-specific information on damage for designing large composite structures, albeit with the same local microstructure.

4.3 Experimental Setup

The basic setup discussed in previous chapters was used to produce a stress pulse normal to the plane of the laminate. In all experiments, the diameter of the laser heating spot was maintained at 3 *mm*, with a total sample thickness ranging from 0.8 *mm* to 1.2 *mm*. As shown by the finite element simulations of Nutt [19], for the plate impact experiments, and by Yuan [20] for the propagation of laser-produced shock waves, such a geometry ensures a planar stress front reaching the free surface of a plate made out of a homogeneous elastic material. It was already pointed out previously that the attainment of such a planar condition is not really necessary in our experiments since the quantification of the threshold stress is achieved via a velocity interferometer that records the information from the center of the loaded area. Nevertheless, maintaining such a condition over the entire loaded area results in the complete spallation of the square coupon at the threshold laser energy. In very few instances, a small debond initiating from the center of the coupon would get arrested without the formation of a complete spall. To capture such events, and hence to accurately determine the threshold laser energies, after loading by a large amplitude stress pulse, the same area was loaded by a low amplitude stress pulse operating in a non-destructive mode. As discussed later, comparison of the dispersion profiles of such pulses from those obtained from the virgin material readily led to the detection of any damage. If no damage was observed, the laser energy was increased and a new virgin spot of the sample was loaded in order to avoid any fatigue effects from previous loadings.

A key element of the experiment was to determine the amplitude of the interply tensile stress corresponding to the threshold laser fluence. The transient free surface velocity $V(t)$ is related to the stress pulse $\sigma(t)$ striking the laminate free-surface via the acoustic impedance ρc_L of the material as

$$\sigma(t) = \frac{1}{2} \rho c_L V(t), \quad (4.1)$$

where ρ is the density of the material, and c_L is the velocity of the longitudinal waves. The transient velocity was measured by using a wide-angle interferometer that is insensitive to the reflection quality of the surface. A schematic of this interferometer is shown in Fig. 1.16 and the details are provided in Pronin and Gupta [16] and as well in Section 1.6 of the present thesis.

The voltage output $S(t)$ of the photodiode is related to the surface displacement $u(t)$ at any instant t by the relationship given in Eq. 1.11. Since the delay time between the two paths of the interferometer ($\tau = 13.3$ *ns* in our setup) is much smaller than

the total duration of the stress pulse, replacing $u(t) - u(t - \tau)$ by $\tau V(t)$ leads to a direct measurement of $V(t)$ from the recorded fringe record via Eq. 1.11. Combining this information with the independently measured values of ρ and c_L , the stress pulse profile striking the laminate's free surface could be obtained through Eq. 4.1.

When the stress pulse amplitude is sufficiently high, several displacement fringes can be obtained. For low stress amplitudes however, as used for obtaining the dispersion profiles, the total displacement of the laminate free surface is less than $0.2 \mu m$, leading only to a fraction of an optical fringe. Under such conditions, the interferometer of Fig. 1.16 works in the linear regime with the signal $S(t)$ proportional to $\delta u = u(t) - u(t - \tau)$. If the delay length of the interferometer τ is chosen to be much smaller than the stress pulse duration, the signal $S(t)$ is proportional to the velocity, and therefore to the incoming stress pulse. Although the use of the interferometer in the linear regime provides an accurate measure of the temporal profile of the free-surface velocity, determination of its absolute magnitude becomes largely inaccurate. However, this is sufficient for obtaining dispersion profiles where it is only important to determine the relative changes in the amplitude and the profile of the pulse as it propagates through the microstructure. For obtaining the spall data where the amplitude of the peak stress is important, the interferometer was used in its regular form. A typical stress pulse profile generated in a Si substrate, and determined via the recorded free surface velocity by the optical interferometer is shown in Fig. 1.18. The pulse has a rise time of approximately $1.5 ns$, and a total duration of only $17 ns$. Since this profile is largely controlled by the laser pulse characteristics, it was used as the input pulse profile while studying the dispersion characteristics in both the C/C and C/P laminates.

4.4 Material Description and Sample Preparation

Two dimensional laminated C/C and C/P composites were used in this study. The C/C panels were obtained from C-CAT¹ and were made from a T-300 carbon fiber, 8 harness (H) satin weave prepreg and densified with a phenolic resin. These panels had undergone three densification/carbonization ($800^\circ C$) cycles and a final heat treatment at $1700^\circ C$. Even with these densification cycles, both interply and transverse bundle cracks remain in C/C laminates as shown in an cross-sectional view of the material in Fig. 4.1(a). The polymer laminates used were "Fiberite 966C" obtained from ICI Fiberite². The polymer composites were made by impregnating the 8H satin weave made from carbon fibers with

¹Carbon-Carbon Advanced Technologies, 7445 E. Lancaster Ave., Fort Worth, Texas 76112.

²ICI Fiberite, 4300 Jackson Street, Reenville, Texas 75401.

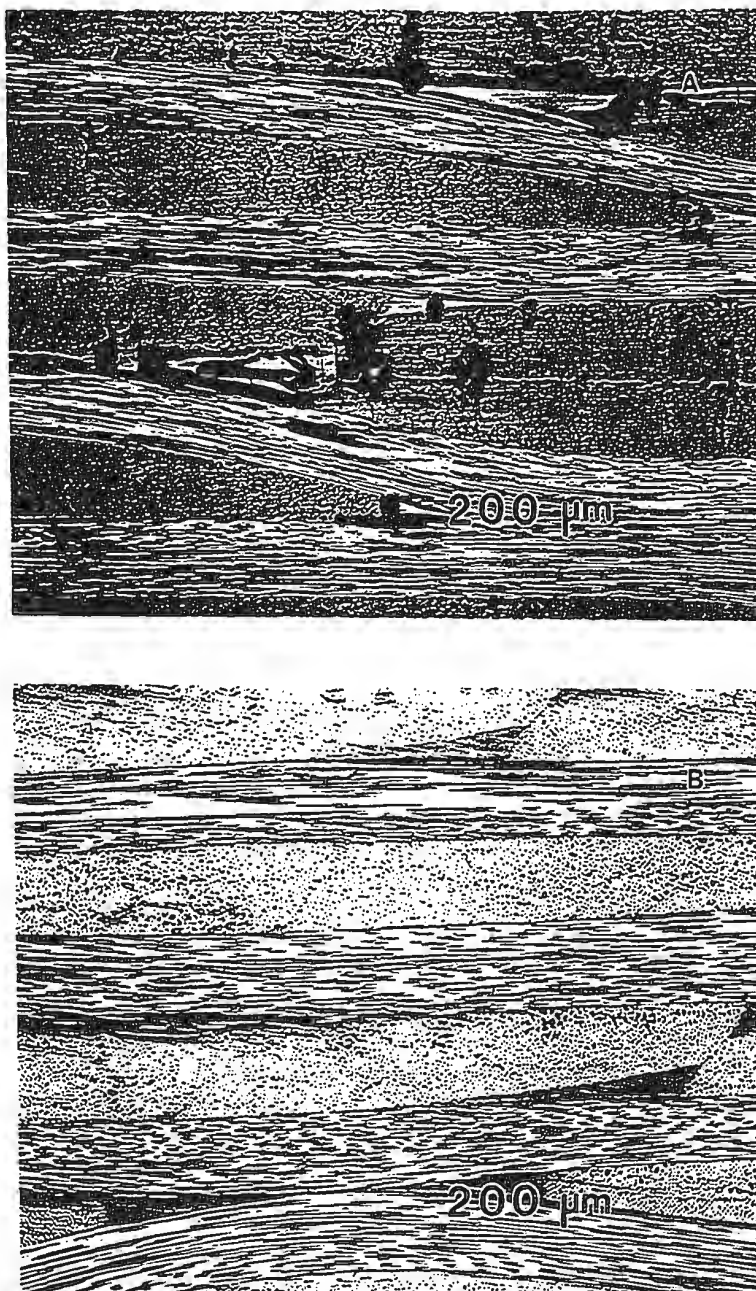


Figure 4.1: (a) A cross-section of a C/C composite showing the presence of interply delamination cracks along with other material discontinuities, such as voids and transverse bundle cracks. (b) A cross-sectional micrograph of a C/P laminate showing excellent infiltration of the matrix, leaving absolutely no cracks and voids. However, the distribution of the crimp on account of the weave geometry leads to matrix- and fiber-rich pockets. For both laminates, the pulse propagation direction was normal to the plies.

the polyimide resin (966C). In contrast to the C/C samples, the C/P laminates showed no voids or cracks due to excellent infiltration of the matrix material, Fig. 4.1(b). However, due to the weave architecture and the autoclave process, the C/P composite showed non-uniform microstructure in the form of fiber- and matrix-rich regions. Each composite panel consisted of 24 plies stacked in the 0° - 0° orientation. Besides the difference in the matrix materials, these two materials represent the two extreme microstructural cases, with and without matrix discontinuities.

For both steps (spalling and dispersion), the composite samples were machined into square plates with sides of 12 mm length, and a total thickness of 1.1 mm normal to the ply direction. Since the stress pulses were generated normal to the plies, a 0.5 μm thick aluminum film was deposited on the back surface. To improve the reflectivity of the He-Ne laser beam even while using the wide-angle interferometer, a 200 Å thick Al reflecting coating was deposited on the laminate's front surface. Since this thickness of 200 Å is much smaller than the length of the stress pulse, the reflecting stress pulse is insensitive to the presence of the Al film, while aiding in obtaining displacement fringes. Both films were deposited by sputtering. Finally, the aluminum film on the sample's back surface was covered by a 5 to 10 μm thick layer of solid water glass which serves as the constraining medium. The layer of solid water glass was obtained by applying a hydrated solution of sodium silicate.

4.5 Experimental Determination of the Dispersion and Attenuation Curves

The stress pulses with low amplitudes were generated normal to the plies by focusing the laser beam onto a 3 mm diameter area. The corresponding laser fluence did not cause any ablation of the material. For these experiments no constraining layer of water glass was used. Since the stress pulse profile of the just generated pulse is largely controlled by the profile of the laser heating pulse, we consider the temporal stress profile of Fig. 1.18 as an input signal at the back surface of composite samples. Figures 4.2 and 4.3 represent the free-surface velocity profiles obtained via the linear interferometer for the C/P and C/C laminates respectively, after the pulse traverses through a total laminate thickness of approximately 0.55 mm (C/P) and 1.1 mm (C/C). As discussed earlier, the amplitude of the signals in Figs. 4.2 and 4.3 is proportional to the velocity. Since for the study in this section it is only necessary to obtain the stress pulse profiles, it is not necessary to obtain the actual amplitudes. To ensure that the stress pulse remains planar over the

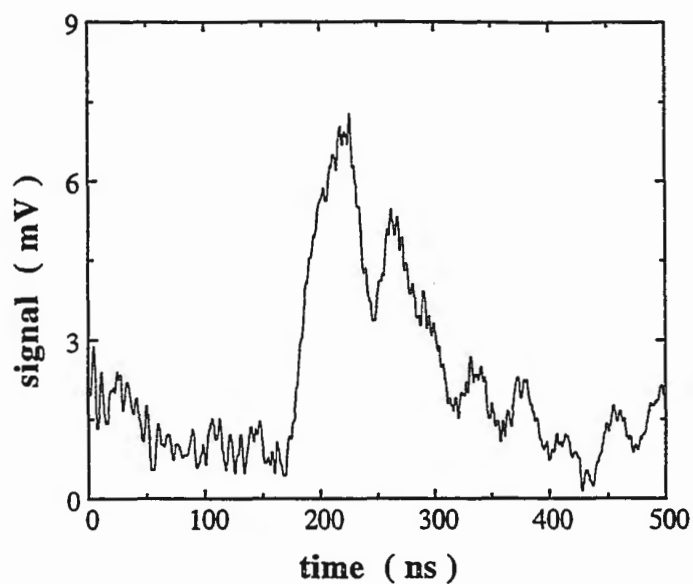


Figure 4.2: The velocity profile measured at the free surface of a 0.55 *mm* thick C/P sample.

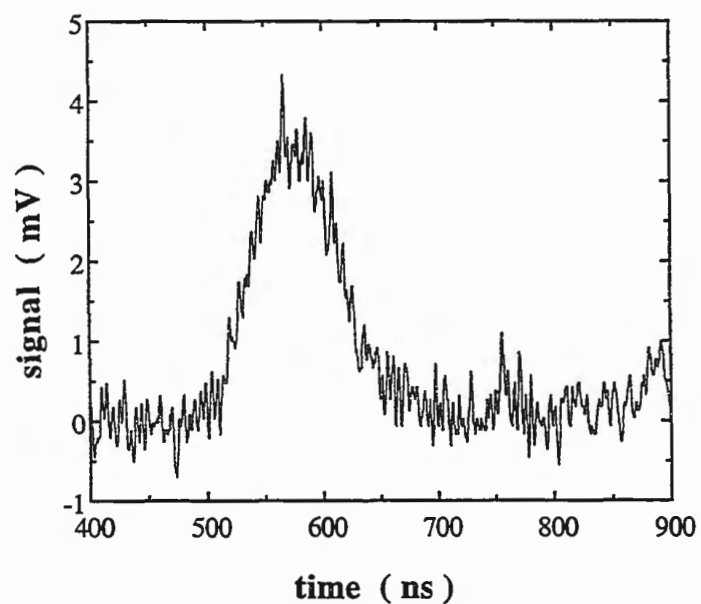


Figure 4.3: Velocity history at the C/C composite surface obtained by a linear interferometer upon impact from a low amplitude stress pulse. The total thickness of the sample was 1.1 *mm*. This profile was used to determine the complex wave number.

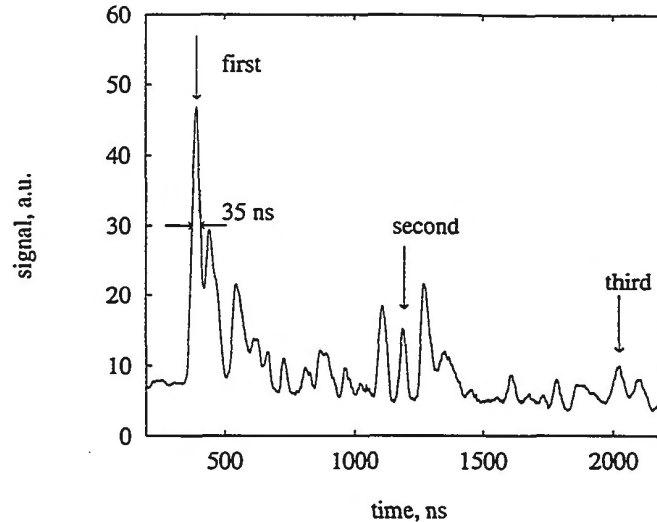


Figure 4.4: Velocity history at the C/P composite surface obtained by a linear interferometer upon impact from a low amplitude stress pulse. The effect of interply reflections is indicated by the nature of the profile. The arrows in the inset show the first and subsequent arrival of the dilatational wave to the free surface.

central free-surface region from where the interferometric signal is recorded, the ratio of the diameter of the stress generation area (or the laser heating spot) to the total travel distance was maintained at 3 in all the experiments (Nutt [19], Yuan [20]).

The propagation of the stress pulse in the C/P composite is accompanied by multiple reflections from the matrix/fiber-weave interface, resulting in a significant broadening of the pulse upon arrival at the second free surface, Fig. 4.2. These reflections become significant due to the large difference in the acoustic impedances of the polymer matrix and the fiber-weave. As shown in Fig. 4.2, this results in a pulse envelope of 200 ns in duration after propagation through approximately 8 interply interfaces, while still remaining planar over the central 2 mm diameter region. Figure 4.2 clearly shows the saw-tooth features arising due to the reflection from the interply interfaces. Interestingly, for the C/C case with little difference in the acoustic impedances of the matrix and the fiber bundles, the saw tooth structure is absent, Fig. 4.3.

In order to study the influence of the C/C and C/P microstructures on the pulse propagation characteristics, the free surface velocities were monitored for long times so that the subsequent arrivals of the stress pulse at the free surface could be recorded. Figures 4.4 and 4.5 represent this information on C/P and C/C composites, respectively.

Time was measured from the moment the stress pulse was generated at the back

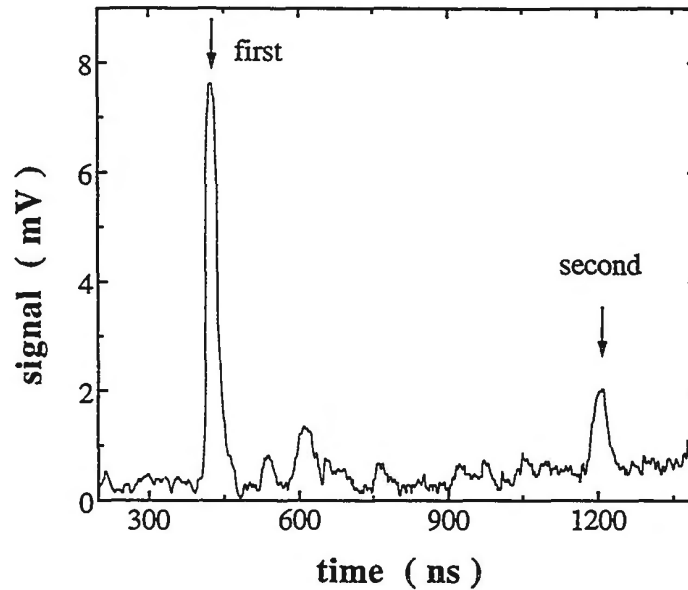


Figure 4.5: The recorded velocity history at the C/C composite surface using the wide-angle interferometer. The pulse propagates through a total sample thickness of 1.1 mm normal to the plies. The arrows in the inset show the first and second arrival of the dilatational wave at the free surface.

surface. Figure 4.4 indicates that there is no significant scattering of the pulse upon interaction with the individual fibers as soon as the pulse length becomes equal to 35 ns. This corresponds to a spatial extent of approximately 90 μm when related with the independently measured stress wave velocity of 2600 m/s transverse to the plies. Clearly this is much larger than the individual fiber diameter of 6 μm . Thus, an upper frequency $f_{up} = 1/\tau = 30$ MHz can be used to determine the effective transverse-ply elastic properties of the C/P composites. In contrast to the C/P case, the C/C profiles (Fig. 4.5) have no saw-tooth structure and due to the presence of interply voids and cracks, the second arrival of the stress pulse is characterized by a significantly lower amplitude, compared to the C/P case in Fig. 4.4. At the present time no quantitative model that predicts the output stress pulse profile for a given microstructure and an input profile can be provided. However, as discussed below, it is possible to obtain the fundamental dispersion and attenuation profiles for the composite microstructures by making use of the input and the recorded free-surface velocity profiles.

The dispersion and the attenuation factors for the C/C and C/P laminate microstructures can be obtained by assuming the profile of Fig. 1.18 as the input stress profile (on the sample's back surface), along with the measured profiles in Figs. 4.2 and 4.3. The

transient input stress pulse $\sigma(x_0, t)$ at x_0 can be written in terms of its Fourier components $F(\omega)$ as

$$\sigma(x_0, t) = \int_{-\infty}^{+\infty} F(\omega) e^{i(k(\omega)x_0 - \omega t)} d\omega, \quad (4.2)$$

where $k(\omega)$ is the complex wave number, ω and t are the frequency and time respectively. The recorded pulse profiles of Fig. 4.2 or 4.3 at any other location $x = x_1$ can be written similarly by replacing $\sigma(x_0, t)$ by $\sigma(x_1, t)$, and x_0 by x_1 in Eq. (4.2). If k is written as $k_1 + i k_2$, then the real part k_1 is related to the wave velocity through the frequency ω as

$$k_1(\omega) = \frac{\omega}{c_L} \quad (4.3)$$

whereas, the complex part k_2 when multiplied by $(x_1 - x_0)$ provides a measure of the attenuation in nepers. The aim here is to determine both c_L and k_2 experimentally. This was obtained by taking the ratios of the inverse Fourier transforms of Eq. 4.2, and that of a similar equation representing the profile at any other location (e.g., the profile in Fig. 4.2 or 4.3) giving

$$\exp(i k(\omega)(x_1 - x_0)) = \frac{\int_{-\infty}^{+\infty} \sigma(x_1, t) e^{-i\omega t} dt}{\int_{-\infty}^{+\infty} \sigma(x_0, t) e^{-i\omega t} dt}. \quad (4.4)$$

The right hand side of the above equation can be numerically evaluated and expressed as

$$A(\omega) e^{iB(\omega)}, \quad (4.5)$$

assuming that both $A(\omega)$ and $B(\omega)$ are real functions. Comparison of the above with the left hand side of Eq. 4.4 readily provides the information on k_1 and k_2 as

$$\frac{\omega}{c_L}(x_1 - x_0) = B(\omega) \quad (4.6)$$

$$k_2(x_1 - x_0) = \log A(\omega). \quad (4.7)$$

Figure 4.6(a) shows the dilatational wave speed as a function of frequency obtained via Eq. 4.6. Surprisingly, the wave speed is fairly insensitive to the frequency, and the medium can be considered as non-dispersive. Figure 4.6(b) provides the value of k_2 or the attenuation factor in *nepers/cm* for the C/P laminate by using the profiles in Fig. 4.2 and Fig. 1.18.

As expected, the velocity profiles obtained after different propagation distances of 0.55 mm and 1.1 mm yielded almost the same attenuation factor. This suggests that the attenuation is solely due to the microstructure and the pulse remains planar upon arrival at the free surface. The hump in Fig. 4.6(b) at 27 MHz corresponds directly to

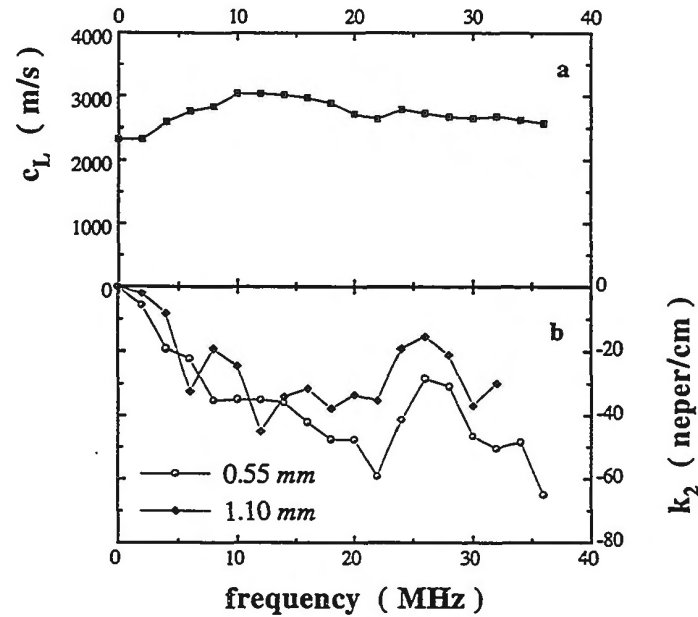


Figure 4.6: Data showing the dispersion characteristics for the C/P laminate with local microstructure given in Fig. 4.1(b). The imaginary part of the wave number k gives the velocity of the dilatational wave, presented as a function of frequency in (a). The attenuation for different frequencies in the C/P microstructure is shown in (b).

the interply reflection. Taking the wave velocity as 2800 m/s , the frequency of 27 MHz corresponds to a spatial distance of $104 \mu\text{m}$, which in turn, is equal to the average ply thickness.

Due to the absence of multireflection effects in C/C composites, our results indicate that the wave propagation normal to the plies can be considered as nondispersive, with an average wave speed $c_L = 2590 \text{ m/s}$. Figure 4.7 provides the information on attenuation of different harmonics obtained by using Figs. 1.18 and 4.3.

The information on the stress wave velocity c_L and decay k_2 can be used to determine the exact profile of the stress pulse that the free surface will experience for a given input of the stress pulse profile, and a thickness of the composite laminate. This final profile can be used to determine both the peak amplitude and its critical location from the free surface. Failure in the form of interply delamination will occur when the peak amplitude is equal to the interply strength, the measurement of which we discuss next.

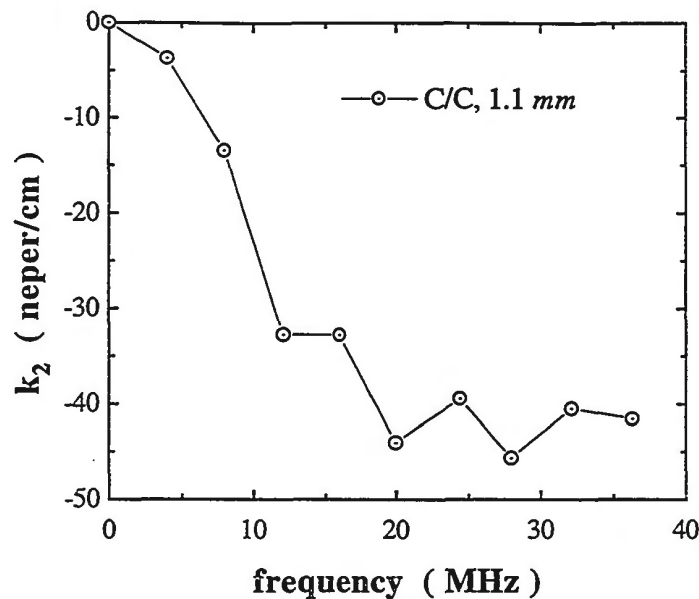


Figure 4.7: Data showing the attenuation of different frequencies for the C/C microstructure given in Fig. 4.1(a).

4.6 Measurements of the Interply Tensile Strength

In these sets of experiments, the laminates were subjected to successively higher laser energies until free-surface spallation was observed. At such laser energies, the aluminum film melts, and as discussed in Gupta *et al.* [14] the overlayer of solid water glass is completely removed. This requires the laser heating spot to be changed after each sub-threshold loading. This procedure also removes any fatigue effects on the measured values. In all the experiments, the diameter of the laser heating spot was maintained at 3 mm, with a total sample thickness ranging from 0.8 mm to 1.2 mm. As discussed before, this geometry ensures a planar stress front reaching the free surface over a 2 mm diameter area.

4.6.1 Failure Mechanisms

Since the stress pulse is produced only over a small area of the laminate surface, un-grooved samples required much higher threshold laser energies for complete spalling of the material, as the failure involved rupturing of the overlaying fiber bundles along the circumference of the loaded area. Figure 4.8 shows one such failure in the C/C sample at a threshold laser fluence of $7.8 \times 10^4 \text{ J/m}^2$, measured at the laser source. Similar failures

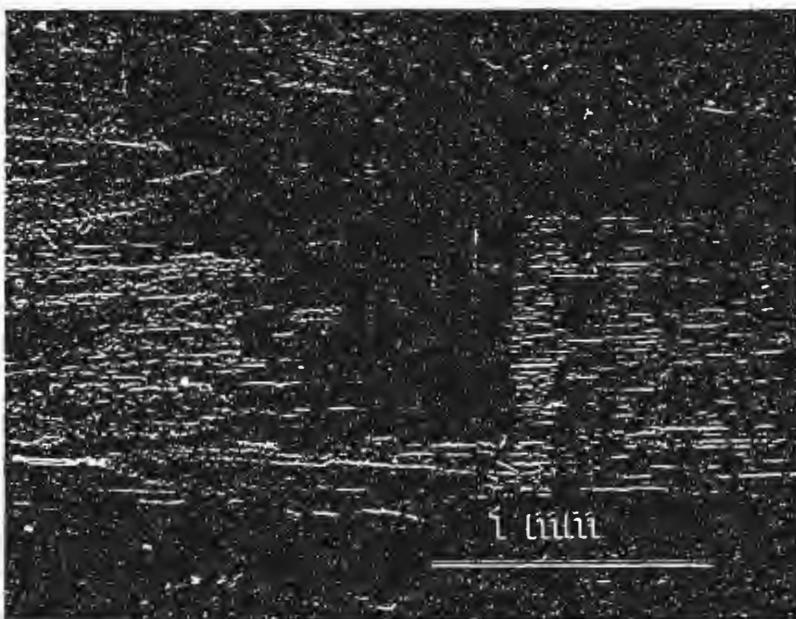


Figure 4.8: A micrograph depicting a free surface spalling event in a C/C sample where no surface grooves were provided. Since the stress is produced only over part of the surface, the sub-surface delamination has to break through the overlaying fiber bundles to cause complete spallation. The tearing of the fiber bundles at the edges can be clearly seen in the figure.

were reported by Gilath *et al.* [66]. It is clear that the nucleus of the spallation failure, possibly in the form of sub-surface fiber/matrix debonding, occurs much before the spallation is observed. In order to determine the limiting interply property, which can also be taken as the failure criterion for describing the onset of spallation shown in Fig. 4.8, the laminate free surface was patterned with 2 mm square areas, with 0.6 mm-deep edge grooves, as shown in Fig. 4.9. Since the square coupon dimensions are smaller than the stress pulse area, at the threshold laser energy the square coupon from the sample's back surface was completely removed at the interply interface, as shown in Fig. 4.9 for a C/P sample. An identical continuum-scale failure was replicated in C/C samples with the same groove geometry.

Although the planarity of the stress pulse reaching the free surface is not critical for quantifying the recorded velocity fringes, this condition ensured complete separation of the square coupons at the threshold laser fluence in most experiments. Since no edge flaws were introduced during the grooving process (confirmed by observations under a scanning electron microscope), we do not expect any significant stress concentrations at the edges either due to the presence of flaws or the geometry of the grooves. The experimental observations seem to support this contention, as the entire square coupon would spall off at the threshold laser energy. Further, at the sub-threshold stress amplitude that was only 5 % lower than the critical one, no cracks were observed to nucleate from the groove edges. Interestingly, in very few instances, an initial debond would occur at the center and be arrested before the formation of a complete spall. To capture such events, and hence to accurately determine the threshold laser energies, after loading by a large amplitude stress pulse, the same area was loaded by a low amplitude stress pulse operating in a non-destructive mode. The dispersed profile of such a pulse was then compared with that obtained from the virgin material, and from the same spot, before loading it with a large amplitude stress pulse. Figure 4.10(a) shows a typical free surface velocity profile obtained from a virgin spot whereas the velocity profile of Fig. 4.10(b) corresponds to a sample subjected to a threshold laser energy but without the formation of a spall.

The presence of the damaged area forced the low amplitude stress pulse to propagate around the damage and led to a delay of about 150 ns in Fig. 4.10(b). This delay can be used to estimate the size of the damaged area, which in this case should not be less than 1 mm ($2 \times \text{delaytime} \times c_L$). Sometimes the occurrence of such damage resulted in a total disappearance of the free surface profile. Without fail, the absence of any damage was reflected in almost no visual change in the profile of Fig. 4.10(a). Besides helping us to determine the threshold laser fluence, the above also suggests the potential of short stress pulses as a NDE tool, if an acceptable base signal can be established.

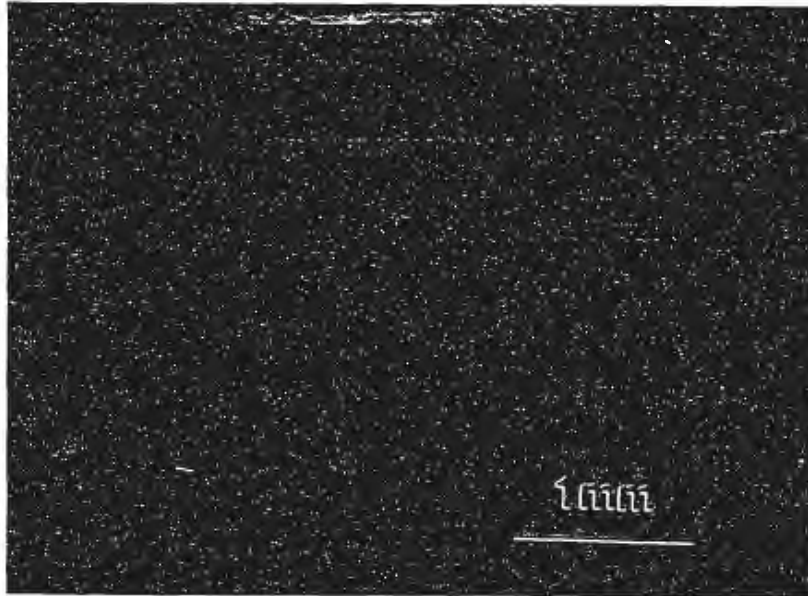


Figure 4.9: A micrograph showing spallation in a central square coupon when the stress pulse was generated on the back surface and in line with the central coupon. The failure was at the interply interface since no tearing of the fibers within the bundles was observed on the two separated surfaces. The above micrograph is for the C/P laminate. Identical failures were observed in C/C samples with the same square coupon geometry and groove depths.

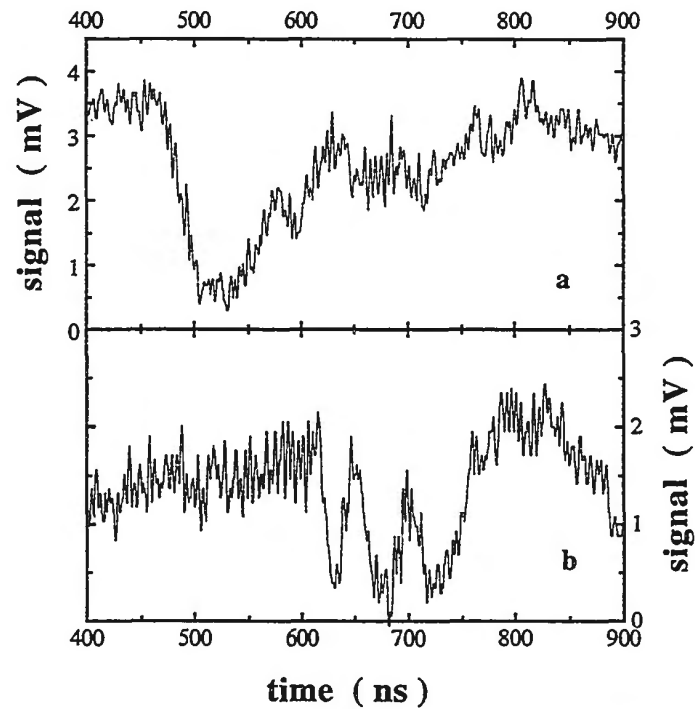


Figure 4.10: An example showing the use of low amplitude stress pulses as an NDE tool to detect damage caused by a high amplitude stress pulse. The profile (a) shows the relative intensity at the photodiode which is proportional to the free-surface velocity, obtained from a virgin sample when subjected to a low amplitude stress pulse. Profile (b) shows a signal obtained under similar conditions but after sub-spallation damage by a high amplitude stress pulse.

To determine the mechanism of damage initiation, the shock wave was generated near the edge of an ungrooved sample. After each increment of loading the sample's edge was viewed under an optical microscope to observe the nucleation of cracks. Figure 4.11 shows that cracks initiate in the bundle within a ply and then curl up to the interply interface. In many cases cracking was directly at the interply interface. We were able to capture both types of failure in our experiments. Although the matrix at the interply interface bonds fibers in two different bundles (and possibly in orthogonal directions), as opposed to that of parallel fibers within the same bundle, it should be pointed out that for an out-of-plane pulse loading both sites are equally prone to fracture. At the local scale, the failure locus is an envelope of matrix and fiber/matrix interface delaminations, as shown in a high magnification view of the initiated crack in Fig. 4.12. The choice of the initiation site in C/P composite is governed by the availability of matrix-rich pockets as shown in Fig. 4.1(b) and by the final stress distribution due to the transient loading. The tensile stress builds up from zero at the free surface to a maximum at the above locations with the exact distribution depending upon the profile of the stress pulse, the elastic constants of the material, and microstructural inhomogeneities.

Figure 4.13 depicts a spalled C/P surface showing both the warp and fill bundles. This failure must have started at the interply interface, since if it did start within the bundle then one must see fibers rupturing in either the fill or the warp yarns. However, when the interply region is pulled apart by the transient pulse, due to the variability in the microstructure, some fibers from both the fill and the warp yarns are pulled, giving thereby a final non-planar fracture surface as shown in Fig. 4.13. Figure 4.14 shows a failure which initiated inside a bundle. The initiated crack propagates entirely through this bundle and similarly oriented adjacent bundles to form the final spall. Accordingly, the fracture surface in Fig. 4.14 has all fibers aligned in one direction. The choice of the failure in Fig. 4.13 *vs.* the one in Fig. 4.14 is governed by the location of the probed area. If the 2 mm diameter stressed area is near the crimp, then the geometry of the fiber weave forces the crack to be trapped at the interply interface since otherwise, a larger energy-consuming process of fiber breakage would be necessary. The failure surface in Fig. 4.13 supports this explanation. On the other hand, if the probed area is between the two crimped regions, then the initiated intra-bundle crack can propagate without hindrance from the transverse bundles, leading to the failure surface shown in Fig. 4.14. For an 8 harness (H) weave where there is a large distance between the two adjoining crimps this process should be preferred. It also appears that this deformation should take less effective interply stress as compared to the one near the crimps. Thus, in a plane weave with crimps every alternate fiber bundle should be more efficient against spalling

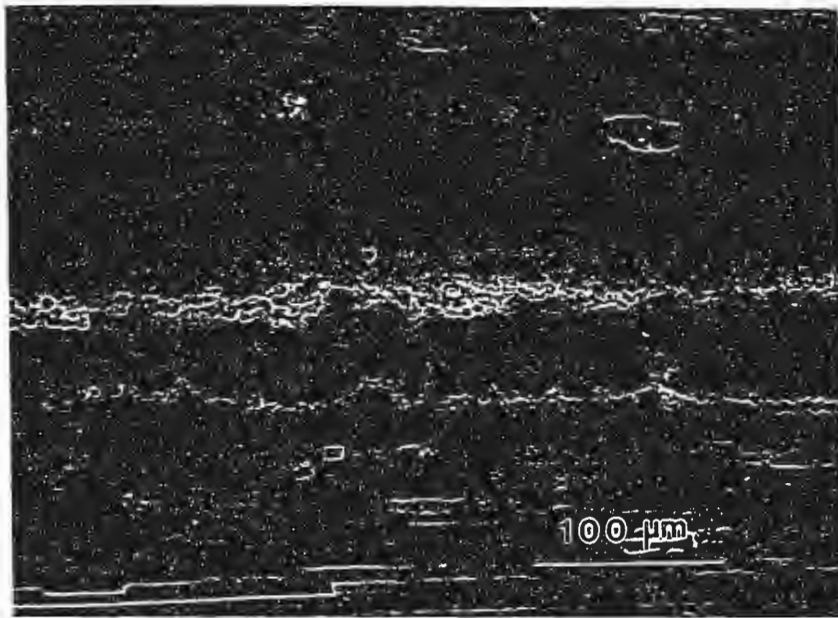


Figure 4.11: A micrograph showing the edge view of a shock-loaded C/P sample. The initiation of an intra-bundle crack and its curling towards the interply interface can be clearly seen.

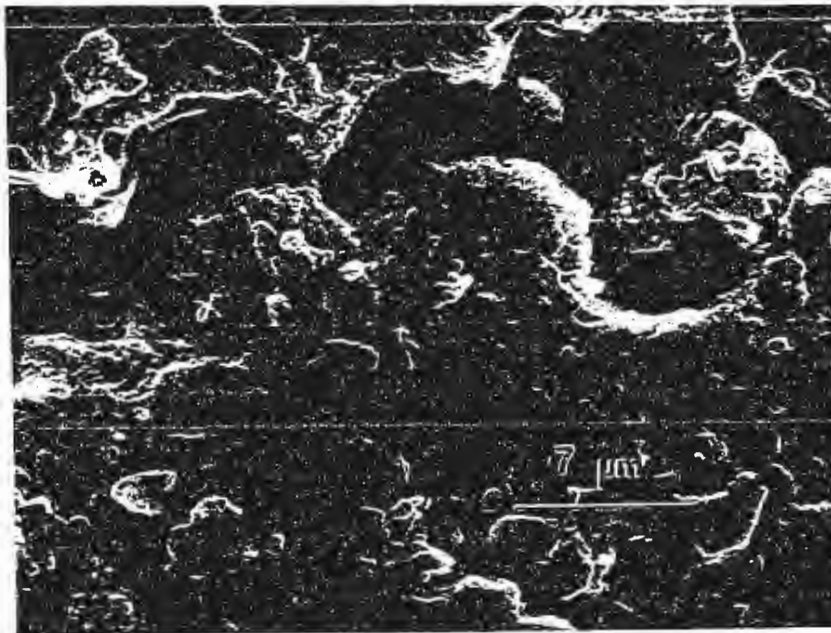


Figure 4.12: A high magnification view of the intra-bundle crack in the C/P sample showing the local failure to be through the matrix and the fiber-matrix interface.

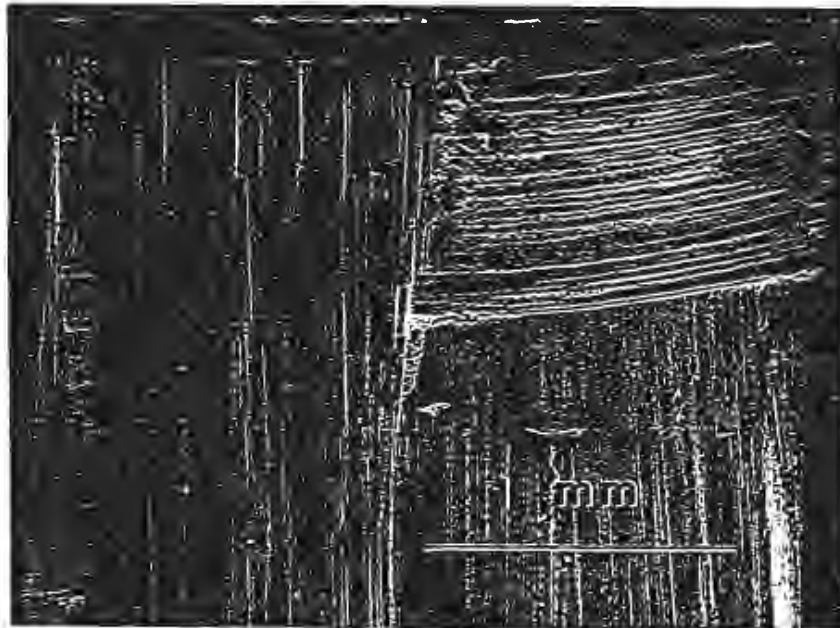


Figure 4.13: A micrograph showing both the warp and fill yarns on the spalled surface of a C/P laminate.

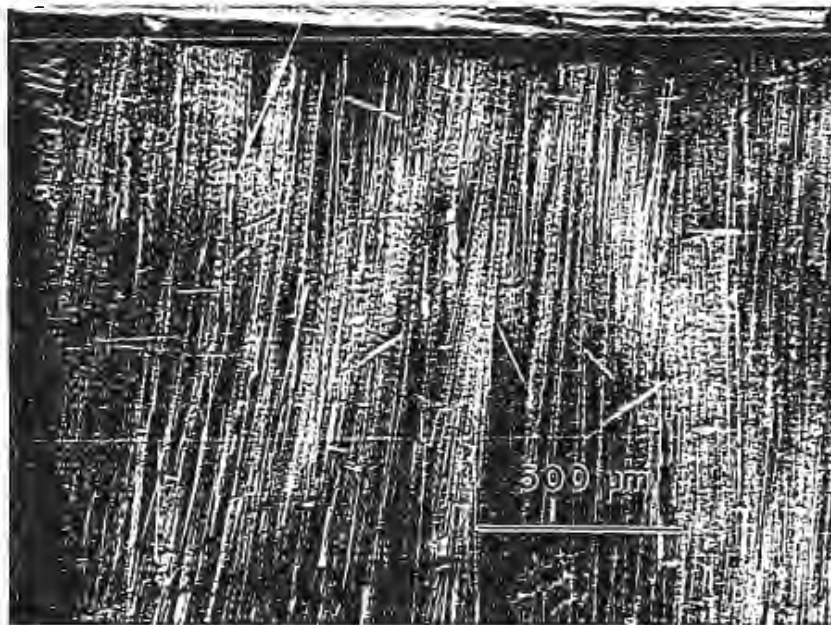


Figure 4.14: A micrograph showing spallation within parallel fiber bundles as indicated by the similarly oriented fibers on the fractured surface.

failures as compared to an 8 H or a 5 H weave composite.

Figure 4.15 shows the influence of matrix-rich pockets on the final failure. This failure is obviously at the interply interface but as this region was pulled by the stress pulse, the matrix from the matrix-rich pocket beneath the interply interface was uprooted from the bundle under it, and led to the formation of a dimple on the fractured surface. The underlying fiber bundle from where the matrix delaminated can also be seen in Fig. 4.15.

All the above failures at the local scale were at the fiber/matrix interface. Figure 4.16(a) shows one such failure in a C/P sample.

The barren matrix-free surface of the spalled spot is also evident in Fig. 4.12. The failure in the C/C samples was almost identical to that shown in Fig. 4.16(b) except that the failure initiation site was entirely governed by the distribution of pre-existing matrix cracks. Also the porous nature of the matrix and the weak fiber/matrix interface is more prominent in C/C samples. As discussed below, this leads to much lower values of the interply strength.

4.6.2 Measurement of the Failure Stress

In the light of the above failure mechanisms we define the local continuum-scale stress responsible for above damage as the effective interply tensile strength. The dotted lines in Fig. 4.17 show a typical fringe record obtained from a C/C sample surface by using the Doppler setup of Fig. 1.16. In these experiments the stress level is substantially higher compared to those employed for obtaining the dispersion profiles, and therefore, the signal is no longer proportional to the velocity but related to the transient displacement of the surface through Eq. 1.11. Because of the scattering of the He-Ne laser pulse from the fibers, the fringe record is not sharp (despite the presence of the Al film) and contains noise, which requires a careful interpretation of the recorded fringe data in order to obtain the amplitude of the stress pulse. Obviously the signal-to-noise ratio can be improved by using a higher power frequency-stabilized He-Ne laser but the requirements for the frequency stability limits their availability in the low power regimes only. The latter requirement is necessary since the mode frequency of the laser is quite close to the frequency of our signals. Anyway, with careful interpretation of the as-recorded data we were able to obtain the critical amplitudes of the stress pulses using the standard interferometry methods discussed in Section 1.6. However, in order to gain confidence in our interpretation of the fringe record, an alternative scheme was also used. The strategy involved determining the profile of low amplitude stress pulses using the linear

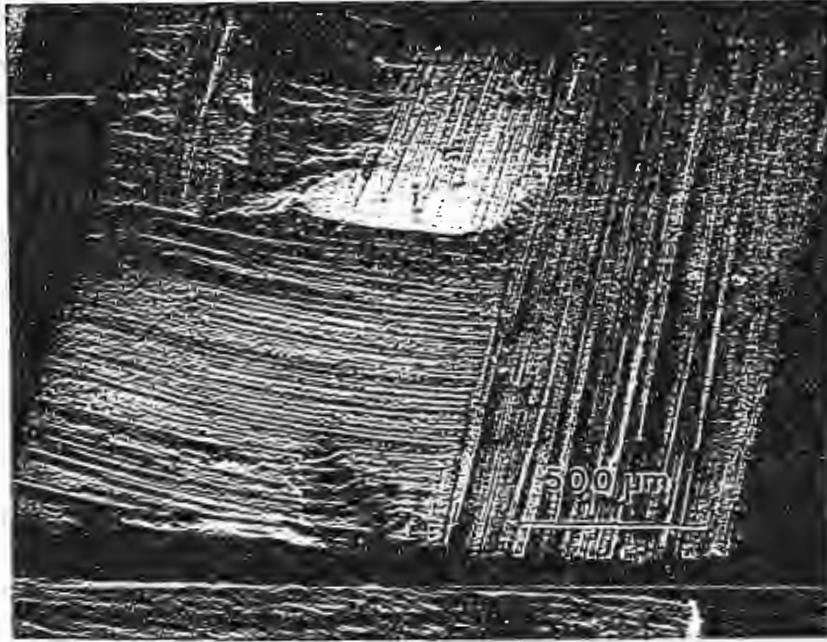


Figure 4.15: The influence of matrix-rich pockets on the spalled surface. A dimple corresponding to the matrix failure from the fiber bundle directly beneath the fractured surface can be clearly seen.

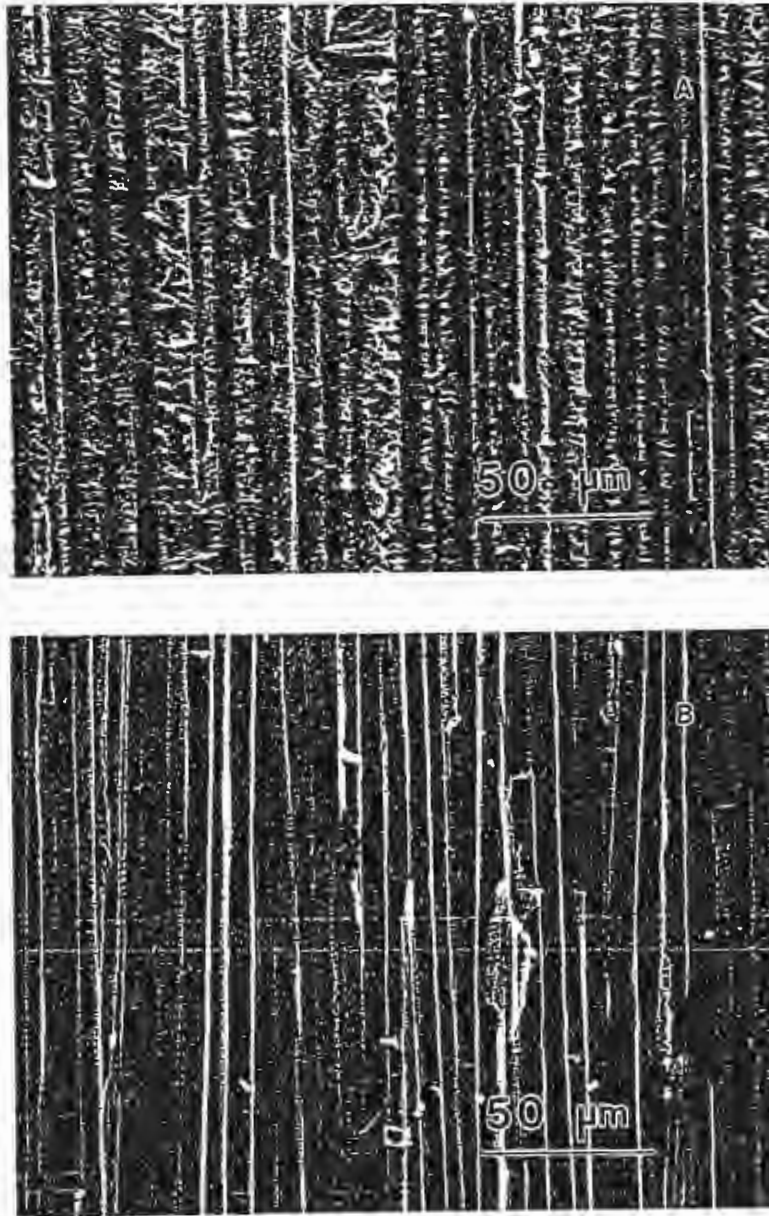


Figure 4.16: A high magnification view of the local fiber/matrix interface failures in C/C and C/P laminates. In C/P composites (a) the failure occurred both at the fiber/matrix interface and within the matrix, whereas in C/C laminates (b) the failure was predominantly at the fiber/matrix interface.

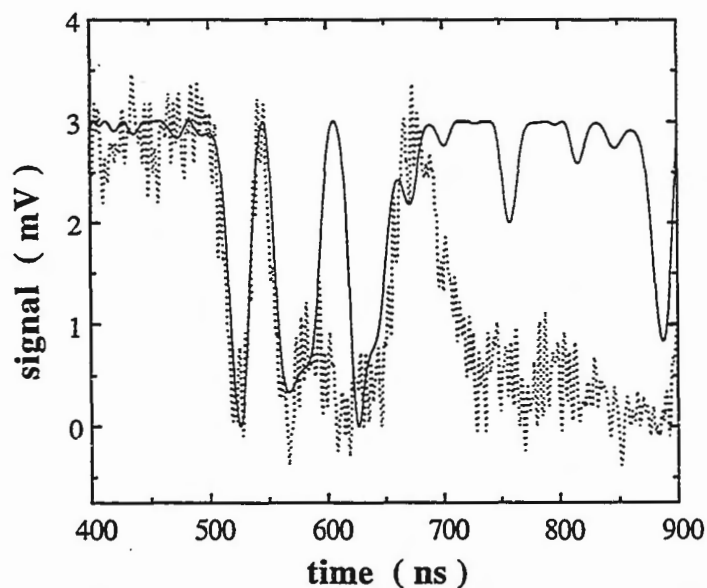


Figure 4.17: In order to relate the interference fringes to the velocity profile in high velocity experiments, the profile of the stress pulse as recorded in low velocity measurements (Fig. 4.3) was used, and the Doppler fringes that would occur for this profile are then computed (solid line in figure) and fitted to the directly obtained fringes (dotted line in figure) for the high velocity measurements, by adjusting the amplitude of the low amplitude stress pulse.

interferometer after propagating them through the same spot. One such profile is shown in Fig. 4.3. Assuming this to be the velocity profile (in normalized coordinates, with the pulse amplitude normalized by the peak stress) even at high stress levels, the amplitude of this signal was varied and the expected fringe record that would result upon reflection of such profile was calculated, and compared with what was obtained directly by an interferometer. The amplitude of the velocity profile was adjusted until a good match between the calculated and the measured fringe record was obtained. The dotted line in Fig. 4.17 represents the measured fringe record for the stress pulse causing spallation, whereas the solid line is the calculated profile. This fit was obtained by assuming an amplitude of 100 *m/s*. The discrepancy between these two curves at later times is due to the occurrence of the spallation and the disturbance of the velocity profile as a result of that. This procedure was also adopted for the C/P samples, and for both materials, the amplitude of the stress pulses from this method compared to within 5 to 7 % of those obtained from the direct analysis of the as-recorded fringes. The latter method can however be used without any advance expertise of the user.

For the one-dimensional planar wave propagation here, the recorded free surface velocities $V(t)$ were converted to stress amplitudes by using Eq. (4.1). The longitudinal wave velocities c_L for C/C and C/P laminates were taken from Section 4.5 be 2590 *m/s* and 2700 *m/s*, respectively. A material density ρ of 1.54 *gm/cc* for the C/P material, and a value of 1.67 *gm/cc* for the C/C composite was obtained from independent experiments. This amplitude information along with the profiles in Figs. 4.2 (for C/P) and 4.3 (for C/C) completely defines the stress pulse characteristics striking the free surface. For these stress profiles and above wave velocities, the peak tensile stress should occur at a distance of approximately 130 μm and 200 μm from the free surface in C/C and C/P samples, respectively. These values are fairly consistent with the measured spall-depths by a mechanical profilometer.

Figure 4.18 shows the measured interply tensile strengths for the C/C and C/P laminates. The strength values for the C/P composite ranged from 150 *MPa* to 230 *MPa*. This large variation should be related to the non-uniform distribution of the matrix-rich pockets, the position of the probed area close or far from the crimped region, and the location (interply *vs.* the intra-bundle) where the stress peaks. The average strength value of 200 *MPa* for the C/P laminate is much greater than the value of 60 *MPa*, obtained for the C/C laminates. This is due to the pre-existing interply cracks and voids in the C/C samples. As shown in Fig. 4.1(a), the scale of these discontinuities ranges from individual fiber dimensions to as large as 0.4 *mm*. A high magnification view of the failed C/C sample surface (Fig. 4.16(b)) shows the absence of any matrix on the fibers, suggesting

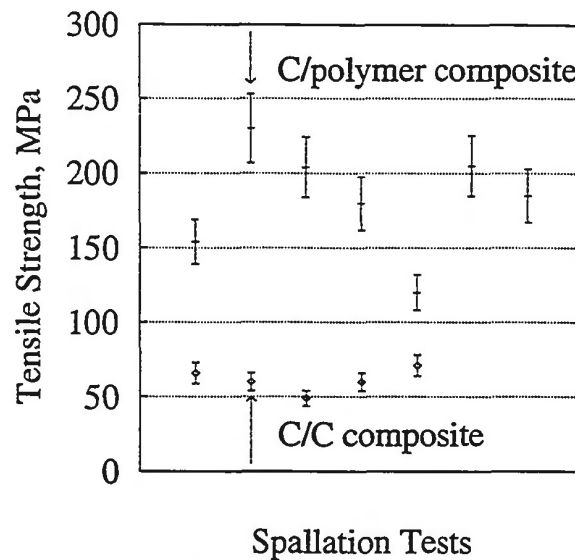


Figure 4.18: Interply strength data for both C/C and C/P laminates as measured by the spallation experiment.

thereby a weak fiber/matrix interface. The presence of these cracks in C/C composite also suggests that the measured values in the range 60 MPa should be considered only as an overall average stress required to grow the microstructural flaws. Fortunately, the size of such flaws is quite small compared with the size of the stress pulse and thus conditions of quasi-static loading of the crack are readily achieved. For such cases, the initiation of such cracks should be largely independent of the width of the stress pulse. Nevertheless a more rigorous parameter which describes the growth of such pre-existing cracks should be the dynamic energy release rate, which should be determined by relating the measured stress amplitude to the pre-existing crack size. At the present time such an analysis is not available.

The small variation in the measured tensile strength values in C/C samples is due to the uniform distribution of the processing-induced fiber/matrix cracks. We note that the measured strength values for both types of laminates are higher than the values in the range of 4-5 MPa (C/C) and 50 MPa (C/P) obtained from direct tension tests [67]. In such tests the sample surfaces are glued to a loading adapter plate and pulled directly in a tensile test by an MTS machine. Since the loaded area is far greater than the one used in the spallation experiment, the longest crack governs the peak value in the direct tension test, whereas such a crack need not be present over the stressed area in the spallation.

4.7 Conclusions

Spalling failures in 2D laminated carbon/carbon and carbon/epoxy composites were studied by loading them with laser-produced stress waves. Depending upon the location of the shock-loaded area, the fiber/matrix delaminations were observed either at the interply interface or within the fiber bundle. In several samples the failure was observed at both sites. The onset of delamination was characterized in terms of effective interply tensile strength, measured in turn by an optical interferometer. Although the local stress-state at the fiber/matrix interface is largely unknown, the interferometer provides an accurate measure of the overall stress pulse profile responsible for damage. Average tensile strength values of 60 *MPa* and 200 *MPa* were obtained for the C/C and C/P laminates, respectively. It was suggested that these values control the initiation of shock damage and should be taken as the local failure criterion for spalling failures.

The above measurements were complemented by determining the attenuation and dispersion characteristics associated with the propagation of stress pulses in the C/C and C/P microstructures. Using this information it becomes possible to design the thickness of a composite structure against impact-induced spalling failures, by assuming the interply strength as the criterion. Also, for a given thickness of the structure, a design value for the interply strength can be provided for accommodating a given impact.

Interestingly, the dispersion results in C/C laminates suggest that the short stress pulses can be potentially used for non-destructive evaluation (NDE) of small flaws in these materials. However, a quantitative prediction of the flaw size and density from the measured free-surface velocity profiles would require a numerical model capable of predicting the distortion in the initial profile as it interacts with the flaw population. In the absence of such models, the pulses can be used as a quality control tool, provided a base signal from an acceptable quality material sample is available. This strategy was used here to spot the onset of delaminations.

Chapter 5

Summary and Suggestion for a Future Work

In this thesis, laser generated stress pulses were used for various materials characterization purposes. First of all, the high amplitude stress pulses were used for the interface tensile strength measurement of various interface systems using the laser spallation technique, which itself was substantially modified from the previous version. Specifically, the process of the stress pulse generation was investigated and optimized. The influence of the constraining material, substrate and absorbing material on the stress pulse profile was studied. It was shown that the replacement of the solid fused quartz plate used as a constraining material by a very thin layer of solidified water glass allowed generation of stress pulses with similar amplitudes but with much shorter duration. In addition, a new interferometer which is insensitive to the surface roughness of the substrate was developed. This interferometer allowed testing of other engineering composites and ceramics. The interferometrically-measured free surface velocity allowed a direct determination of the interface stress.

The main aim of the thesis was to extend the laser spallation technique to measure directly the interface fracture energy by implanting well-defined interfacial flaws. Two techniques were designed to create these microcracks. The sequence of deposition and photolithographic steps was used to artificially generate the axi-symmetric and one-dimensional microcracks. The characteristic size of these structures was approximately $50\text{ }\mu\text{m}$. The shape of these structures was measured by scanning force microscope. The loading of the artificially created microcracks by laser-generated stress pulses was modeled by a 2-D finite element program. This program modeled the propagation of the stress waves, loading of the microcracks and calculated the J-Integral values at the crack tip. The maximum value of the J-Integral at the critical loading was taken as a measure

of the interface toughness. Based on certain theoretical considerations and experimental observations, the assumption of small-scale-yielding was made in the model. However, what part of the measured interface toughness value is influenced by local nucleation of dislocation type processes remains an open question. The investigation of this problem is suggested as a next step in the development of the laser spallation technique. It should include experimental investigation of the high strain rate plasticity in engineering solids and TEM work on dislocation dynamics at the crack tip. The finite element program was also used to explain small interface strength values obtained previously for interfaces between metallic coatings and PG substrates. These small values were explained by the presence of flaws at the interface. The presence of the soft PG substrate substantially increases the J-Integral value at the microcrack tip, leading to extremely small strength values. As a suggestion for future work, several model experiments with various densities of artificial microcracks might be performed. Microcracks should be created and loaded with low-amplitude (in NDE-mode) stress pulses. This would provide valuable experimental information for fine tuning of several theoretical models that exists in the non-destructive evaluation literature.

The interface toughness values measured using the laser generated stress pulses were compared with the toughness values obtained independently, by using a spontaneous delamination technique. In this technique, the elastic energy of the residual compression in the film was used to determine an interface toughness value. The toughness values obtained by the two techniques were encouragingly close to each other, which lead to the conclusion that both techniques must be giving values close to the intrinsic devoid of any large plastic deformation.

Finally, the laser spallation assembly was adopted to study the failure mechanisms and resistance of 2-D laminated C/C and C/P woven composites. Both the failure interface stress, and attenuation and dispersion characteristics associated with the propagation of stress pulses were experimentally obtained. This allows for a complete design of laminated composite structure for impact.

One of the important results of the last work was that it was possible to debond the local fiber/matrix interface by using a sharp stress pulse in actual composite microstructure. For future it was suggested that by adopting the dynamic wave mechanics simulation discussed in Chapter 2, one may relate the critical stress amplitude to the local interface stress by considering the fiber geometry and microstructural details, along with the residual stresses, if any. This, as I believe, will be an important continuation that would allow development of high performance composites.

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Appendix A

Listing of Fortran Codes and ABAQUS Program Used to Model the Transient Loading of the Microcrack

Following listing represents the ABAQUS source code of the program which models the propagation of the stress pulse in the substrate and in the vicinity of the microcrack.

**

```
WAVE PROPAGATION
*PREPRINT,ECHO=NO,HISTORY=NO,MODEL=NO
*****DEFINITION OF POINTS
*****
**F*****G*****H*****K
*****
*****
**L*****B***-2d-****C*****|*****M
*****t*****
**O*****-a-*****A*****|*****
*****h*****
**N*****D***-2d-****E*****|*****Q
*****
*****
*****-w-*****
*****
**P*****T*****R*****S
*****
**a-crack length, d-halfsize of central region, t-film thickness
**h-substrate thickness,w-width - ALL IN FILE node.lst
*****
*****
***FIRST ROW
*NODE,INPUT=node.lst
```

```

*NSET,NSET=F
6000
*NSET,NSET=G
6080
*NSET,NSET=G2
7080
*NSET,NSET=H
7000
*NSET,NSET=H3
8000
*NSET,NSET=K
8080
*****
*NSET,NSET=L
6800
*NSET,NSET=B1
6880
*NSET,NSET=B2
7880
*NSET,NSET=C2
7800
*NSET,NSET=C3
8800
*NSET,NSET=M
8880
*****
*NSET,NSET=L5
10000
*NSET,NSET=B5
10080
*NSET,NSET=B
1290
*NSET,NSET=C
1210
*NSET,NSET=C4
9900
*NSET,NSET=M4
9980
*****
*NSET,NSET=O
10400
*NSET,NSET=U5
10480
*NSET,NSET=U
1330
*NSET,NSET=AA
5330
*NSET,NSET=XX
1170
*NSET,NSET=Y2
9500
*NSET,NSET=Y3
9580
*****
*NSET,NSET=O6
11000
*NSET,NSET=U6
11080
*NSET,NSET=W
1000
*NSET,NSET=A
5000
*NS ET,NSET=X

```

```

1160
*NSET,NSET=Y1
9400
*NSET,NSET=Y4
9480
*****
*NSET,NSET=N
11400
*NSET,NSET=D6
11480
*NSET,NSET=D
1040
*NSET,NSET=E
1120
*NSET,NSET=E4
9000
*NSET,NSET=Q
9080
*****
*NSET,NSET=N7
12000
*NSET,NSET=D7
12040
*NSET,NSET=D8
15000
*NSET,NSET=E8
15008
*NSET,NSET=E9
16000
*NSET,NSET=Q9
16040
*****
*NSET,NSET=P
14500
*NSET,NSET=T
14540
*NSET,NSET=T8
15500
*NSET,NSET=R
15508
*NSET,NSET=R9
18500
*NSET,NSET=S
18540
*****
*****
*****NODE-GENERATION-IN-CENRAL-REGION*****
*****
*NFILL,NSET=CLEFTBOT
W,D,4,10
*NFILL,NSET=CBOTTOM
D,E,8,10
*NFILL,NSET=CRIGHBOT
E,X,4,10
*NFILL,NSET=CRIGHTUP
XX,C,4,10
*NSET,NSET=CRIGHT
CRIGHBOT,CRIGHTUP
*NFILL,NSET=CUP
C,B,8,10
*NFILL,NSET=CLEFTUP
B,U,4,10
*NSET,GENERATE,NSET=CUPT

```

```

1220,1280,10
*NSET,GENERATE,NSET=CBOTTOT
1040,1110,10
*NSET,NSET=CBOUNDAR
CLEFTBOT,CBOTTOM,CRIGHTBOT,CRIGHTUP,CUP,CLEFTUP
*NFILL,NSET=TIP
A,AA,33,10
*NFILL,SINGULAR=2,NSET=CENTER
CBOUNDAR,TIP,10,400
*NSET,GENERATE,NSET=CRACKUP
1570,5170,400
*NSET,GENERATE,NSET=CRACKDN
1560,5160,400
*****
*****ELEMENT GENERATION IN CENTRAL REGION*
*****
*ELEMENT,TYPE=CAX8R
**CPE8R,CAX8R
1,1000,1020,1820,1800,1010,1420,1810,1400
*ELGEN,ELSET=CELDOWN
1,8,20,1,5,800,40,1
*ELCOPY,OLD SET=CELDOWN,NEW SET=CELUP,SHIFT NODES=170,ELEMENT SHIFT=500
*****
*****NEXT REGION -"1" OR "LFGB"*****
*****
*NSET,NSET=UP1,GENERATE
6000,6080,1
*NSET,NSET=BOTTOM1,GENERATE
6800,6880,1
*NSET,NSET=BOTTOM1T,GENERATE
6800,6880,1
*NFILL,NSET=NSET1
UP1,BOTTOM1,8,100
*NSET,GENERATE,NSET=LEFT1
6000,6800,100
*NSET,GENERATE,NSET=RIGHT1
6080,6880,100
*****
*****ELEMENT GENERATION IN "1" REGION*****
*****
*ELEMENT,TYPE=CAX4R
**CAX4R,CPE4R
2000,6400,6401,6001,6000
*ELGEN,ELSET=ELSET1A
2000,71,1,1,2,400,80,1
*ELEMENT,TYPE=CAX3,ELSET=ELSET1B
**2070,6070,6471,6071
**2150,6870,6471,6070
**2230,6870,6871,6471
*****
2310,6071,6272,6072
2390,6071,6471,6272
2470,6471,6472,6272
****
2550,6471,6672,6472
2630,6471,6871,6672
2071,6871,6872,6672
****
2151,6072,6173,6073
2231,6072,6272,6173
2311,6272,6273,6173
2391,6272,6373,6273
2471,6272,6472,6373

```

```

2551,6472,6473,6373
2631,6472,6573,6473
2072,6472,6672,6573
2152,6672,6673,6573
2232,6672,6773,6673
2312,6672,6872,6773
2392,6872,6873,6773
*ELEMENT,TYPE=CAX4R
2073,6173,6174,6074,6073
*ELGEN,ELSET=ELSET1C
2073,6,1,1,8,100,80,1
*****
*****NODE GENERATION IN "2" OR GHCB REGION**
*****
*NFILL,NSET=UP2
H,G2,8,10
*NFILL,NSET=BOTTOM2
C2,B2,8,10
*NFILL,NSET=NSET2
UP2,BOTTOM2,8,100
*NSET,GENERATE,NSET=BOTTOM2T
7810,7870,10
*NSET,GENERATE,NSET=RIGHT2
7000,7800,100
*NSET,GENERATE,NSET=LEFT2
7080,7880,100
*****
***ELEMENT GENERATION IN "2" REGION*****
*****
*ELEMENT,TYPE=CAX4R
3000,7110,7100,7000,7010
*ELGEN,ELSET=ELSET2
3000,8,10,1,8,100,10,1
*****
*****NODE GENERATION IN "3" OR CHKM REGION*****
*****
*NFILL,NSET=UP3
H3,K,80,1
*NFILL,NSET=BOTTOM3
C3,M,80,1
*NFILL,NSET=NSET3
UP3,BOTTOM3,8,100
*NSET,GENERATE,NSET=LEFT3
8000,8800,100
*NSET,GENERATE,NSET=RIGHT3
8080,8880,100
*****
***ELEMENT GENERATION IN "3" REGION*****
*****
*ELEMENT,TYPE=CAX4R
**CPE4R
4000,8100,8101,8001,8000
*ELGEN,ELSET=ELSET3A
4000,4,1,1,8,100,80,1
*ELEMENT,TYPE=CAX4R
**CPE4R
4004,8104,8108,8008,8004
*ELGEN,ELSET=ELSET3B
4004,19,4,1,8,100,80,1
*ELSET,ELSET=ELSET3
ELSET3A,ELSET3B
*****
*****NODE GENERATION IN "4" OR ECMQ REGION*****

```

```

*****
*NFILL,NSET=UP4
C4,M4,80,1
*NFILL,NSET=M4UP
Y2,Y3,80,1
*NSET,GENERATE,NSET=MIDL4UP
9500,9580,1
*NFILL,NSET=M4DN
Y1,Y4,80,1
*NSET,GENERATE,NSET=MIDL4DN
9400,9480,1
*NFILL,NSET=BOTTOM4T
E4,Q,80,1
*NSET,GENERATE,NSET=BOTTOM4
9000,9080,4
*NFILL,NSET=NSET4DN
BOTTOM4T,M4DN,4,100
*NFILL,NSET=NSET4UP
M4UP,UP4,4,100
*NSET,GENERATE,NSET=LEFT4UP
9500,9900,100
*NSET,GENERATE,NSET=LEFT4DN
9000,9400,100
*NSET,GENERATE,NSET=RIGHT4UP
9580,9980,100
*NSET,GENERATE,NSET=RIGHT4DN
9080,9480,100
*NSET,NSET=LEFT4
LEFT4UP,LEFT4DN
*NSET,NSET=RIGHT4
RIGHT4UP,RIGHT4DN
*****
*****ELEMENT GENERATION IN "4" OR ECMQ REGION*****
*****
*ELEMENT,TYPE=CAX4R
**CPE4R
5000,9000,9001,9101,9100
*ELGEN,ELSET=ELS4DNA
5000,4,1,1,4,100,80,1
*ELEMENT,TYPE=CAX4R
**CPE4R
5004,9004,9008,9108,9104
*ELGEN,ELSET=ELS4DNB
5004,19,4,4,4,100,80,1
*ELSET,ELSET=ELSET4DN
ELS4DNB,ELS4DNA
*ELCOPY,NEW SET=ELSET4UP,OLD SET=ELSET4DN,SHIFT NODES=500,ELEMENT SHIFT=320
*****
*****NODE GENERATION IN "5" OR OLBQ REGION*****
*****
*NSET,GENERATE,NSET=UP5
10000,10080,1
*NSET,GENERATE,NSET=BOTTOM5
10400,10480,1
*NSET,GENERATE,NSET=UP5T
10000,10080,1
*NFILL,NSET=NSET5
UP5,BOTTOM5,4,100
*NSET,GENERATE,NSET=LEFT5
10000,10400,100
*NSET,GENERATE,NSET=RIGHT5
10080,10480,100
*****

```

```

*****ELEMENT GENERATION IN "5" REGION*****
*****
*ELEMENT,TYPE=CAX4R
**CPE4R
6000,10200,10201,10001,10000
*ELGEN,ELSET=ELSET5A
6000,71,1,1,2,200,80,1
*ELEMENT,ELSET=ELSET5B,TYPE=CAX3
**6070,10070,10271,10071
**6150,10070,10470,10271
**6230,10470,10471,10271
*****
6310,10071,10172,10072
6071,10071,10271,10172
6151,10271,10272,10172
6231,10271,10372,10272
6311,10271,10471,10372
2632,10471,10472,10372
*ELEMENT,TYPE=CAX4R
6072,10172,10173,10073,10072
*ELGEN,ELSET=ELSET5C
6072,7,1,1,4,100,80,1
*****
*****NODE GENERATION IN "6" OR NOUD REGION*****
*****
*NFILL,NSET=UP6
06,U6,80,1
*NFILL,NSET=BOTTOM6
N,D6,80,1
*NFILL,NSET=NSET6
UP6,BOTTOM6,4,100
*NSET,GENERATE,NSET=BOTTOM6T
11400,11472,4
*NSET,GENERATE,NSET=LEFT6
11000,11400,100
*NSET,GENERATE,NSET=RIGHT6
11080,11480,100
*****
*****ELEMENT GENERATION IN "6" REGION*****
*****
*ELEMENT,TYPE=CAX4R
**CPE4R
7000,11400,11401,11001,11000
*ELGEN,ELSET=ELSET6A
7000,72,1,1,1,1,1,1
*ELEMENT,TYPE=CAX3,ELSET=ELSET6B
7072,11072,11472,11273
7152,11072,11273,11073
7232,11472,11473,11273
7312,11473,11474,11374
7313,11473,11374,11273
7233,11374,11274,11273
7153,11273,11274,11174
7073,11273,11174,11073
7090,11073,11174,11074
*ELEMENT,TYPE=CAX4R
7074,11174,11175,11075,11074
*ELGEN,ELSET=ELSET6C
7074,5,1,1,4,100,80,1
*****
*****FLAW FREE SPACE ELEMENTS*****
*****
*ELEMENT,TYPE=INTER2

```

```

20000,11000,11001,10400,10401
*ELGEN,ELSET=FLAW1
20000,79,1,1,1,1,1,1
*ELEMENT,TYPE=INTER2
21000,1000,1400,1330,1730
*ELGEN,ELSET=FLAW2
21000,10,400,1,1,1,1,1
*****
*****NODE GENERATION IN "7" OR PNDT REGION*****
*****
*NFILL,NSET=UP7
N7,D7,20,2
*NFILL,NSET=BOTTOM7
P,T,20,2
*NFILL,NSET=NSET7
UP7,BOTTOM7,20,125
*NSET,GENERATE,NSET=UP7T
12000,12036,2
*NSET,GENERATE,NSET=LEFT7
12000,14500,125
*NSET,GENERATE,NSET=RIGHT7
12040,14540,125
*****
*****NODE GENERATION IN "8" OR TDER REGION*****
*****
*NSET,NSET=UP8
D8,E8
*NSET,NSET=BOTTOM8
T8,R
*NFILL,NSET=NSET8
UP8,BOTTOM8,20,25
*NSET,GENERATE,NSET=LEFT8
15000,15500,25
*NSET,GENERATE,NSET=RIGHT8
15008,15508,25
*****
*****NODE GENERATION IN "9" OR REQS REGION*****
*****
*NFILL,NSET=UP9
E9,Q9,20,2
*NFILL,NSET=BOTTOM9
R9,S,20,2
*NFILL,NSET=NSET9
UP9,BOTTOM9,20,125
*NSET,GENERATE,NSET=LEFT9
16000,18500,125
*NSET,GENERATE,NSET=RIGHT9
16040,18540,125
*****
*****CONNECTION OF DIFFERENT REGIONS*****
*****
*MPC
TIE,LEFT2,RIGHT1
*MPC
TIE,RIGHT2,LEFT3
*MPC
TIE,CLEFTUP,RIGHT5
*MPC
TIE,CRIGHTUP,LEFT4UP
*MPC

```

```

TIE,CRIGHTBOT,LEFT4DN
*MPC
TIE,CLEFTBOT,RIGHT6
*MPC
TIE,LEFT8,RIGHT7
*MPC
TIE,RIGHT8,LEFT9
*****
*****TIE BOTTOMS*****
*****
*MPC
**TIE,BOTTOM1,UP5
TIE,UP5T,BOTTOM1T
*MPC
**TIE,BOTTOM2,CUP
TIE,BOTTOM2T,CUPT
*MPC
TIE,BOTTOM3,UP4
*MPC
TIE,CRACKUP,CRACKDN
*MPC
TIE,MIDL4UP,MIDL4DN
*MPC
**TIE,BOTTOM6,UP7
TIE,BOTTOM6T,UP7T
*MPC
TIE,BOTTOM4,UP9
*****
*****ADDITIONAL MESH REFINEMENT*****
*****
*NSSET,GENERATE,NSET=RLFTU1
6079,6879,100
*NCOPY,OLD SET=RLFTU1,NEW SET=RLFTUP1,CHANGE NUMBER=13921,SHIFT
0,0,0
0,0,0
*MPC
TIE,RLFTUP1,RLFTU1
*****
*NSSET,GENERATE,NSET=RLFTU2
10179,10479,100
*NCOPY,OLD SET=RLFTU2,NEW SET=RLFTUP2,CHANGE NUMBER=10721,SHIFT
0,0,0
0,0,0
*MPC
TIE,RLFTUP2,RLFTU2
*****
*NSSET,GENERATE,NSET=RRGHTU1
6080,6880,100
*NCOPY,OLD SET=RRGHTU1,NEW SET=RRGHTUP1,CHANGE NUMBER=13970,SHIFT
0,0,0
0,0,0
*MPC
TIE,RRGHTUP1,RRGHTU1
*****
*NSSET,GENERATE,NSET=RRGHTU2
10180,10480,100
*NCOPY,OLD SET=RRGHTU2,NEW SET=RRGHTUP2,CHANGE NUMBER=10770,SHIFT
0,0,0
0,0,0
*MPC
TIE,RRGHTUP2,RRGHTU2
*****

```

```

*NSSET,NSET=RRGHTUP
RRGHTUP1,RRGHTUP2
*NSSET,NSET=RLFTUP
RLFTUP1,RLFTUP2
*****
*NFILL
RLFTUP,RRGHTUP,10,5
*****
*ELEMENT,TYPE=CAX4R
22000,20100,20105,20005,20000
*ELGEN,ELSET=FILMREF
22000,10,5,1,12,100,10,1
*****
*NSSET,NSET=SERV1
11479
*NCOPY,OLD SET=SERV1,NEW SET=SERV,CHANGE NUMBER=10921,SHIFT
0,0,0
0,0,0
*****
*NSSET,NSET=RLFTD
11079,11179,11279,11379
*NCOPY,OLD SET=RLFTD,NEW SET=RLFTDN,CHANGE NUMBER=10921,SHIFT
0,0,0
0,0,0
*MPC
TIE,RLFTDN,RLFTD
*NSSET,NSET=RLFTDNS
RLFTDN,SERV
*****
*NSSET,GENERATE,NSET=RRGHTD
11080,11480,100
*NCOPY,OLD SET=RRGHTD,NEW SET=RRGHTDN,CHANGE NUMBER=10970,SHIFT
0,0,0
0,0,0
*MPC
TIE,RRGHTD,RRGHTDN
*****
*NFILL
RLFTDNS,RRGHTDN,10,5
*****
*ELEMENT,TYPE=CAX4R
23000,22100,22105,22005,22000
*ELGEN,ELSET=SUBREF
23000,10,5,1,4,100,10,1
*****
*ELEMENT,TYPE=INTER2
24000,22000,22005,21200,21205
*ELGEN,ELSET=INTERREF
24000,10,5,1,1,1,1,1
*****
*****ADDITIONAL MESH REFINEMENT IN REGION -RIGHT-TOP**
*****
*NSSET,NSET=SERV2
12038
*NCOPY,OLD SET=SERV2,NEW SET=LFTTPRF,CHANGE NUMBER=12962,SHIFT
0,0,0
0,0,0
*NSSET,NSET=SERV3
12040
*NCOPY,OLD SET=SERV3,NEW SET=RGHTPRF,CHANGE NUMBER=13040,SHIFT
0,0,0
0,0,0
*NSSET,NSET=SERV4

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12163
*NCOPY,OLD SET=SERV4,NEW SET=LFTDNRF,CHANGE NUMBER=13337,SHIFT
0,0,0
0,0,0
*NSET,NSET=SERV5
12165
*NCOPY,OLD SET=SERV5,NEW SET=RGHDNRF,CHANGE NUMBER=13415,SHIFT
0,0,0
0,0,0
*NFILL
LFTTPRF,RGHTPRF,4,20
*NFILL
LFTDNRF,RGHDNRF,4,20
*NSET,NSET=TPMDL
25060
*NFILL,NSET=TPRGH
TPMDL,RGHTPRF,10,2
*NSET,NSET=DNMDL
25560
*NFILL,NSET=DNRGH
DNMDL,RGHDNRF,10,2
*NSET,NSET=TPREF
25000,25020,25040
TPRGH
*NSET,NSET=DNREF
25500,25520,25540
DNRGH
*NFILL
TPREF,DNREF,5,100
*****
*ELEMENT,TYPE=CAX4R
25000,25100,25120,25020,25000
*ELGEN,ELSET=REF7A
25000,3,20,1,5,100,3,1
*ELEMENT,TYPE=CAX4R
25500,25160,25162,25062,25060
*ELGEN,ELSET=REF7B
25500,10,2,1,5,100,10,1
*****
*****REFINMENT IN THE TOP OF 8 REGION*****
*****
*NCOPY,OLD SET=D8,NSET=D8REF,CHANGE NUMBER=11000,SHIFT
0,0,0
0,0,0
*NCOPY,OLD SET=E8,NSET=E8REF,CHANGE NUMBER=11000,SHIFT
0,0,0
0,0,0
*NSET,NSET=DD8
15025
*NCOPY,OLD SET=DD8,NSET=DD8REF,CHANGE NUMBER=11020,SHIFT
0,0,0
0,0,0
*NSET,NSET=ED8
15033
*NCOPY,OLD SET=ED8,NSET=ED8REF,CHANGE NUMBER=11020,SHIFT
0,0,0
0,0,0
*NFILL,NSET=TOP8REF
D8REF,E8REF,8,1
*NFILL,NSET=DN8REF
DD8REF,ED8REF,8,1
*NFILL,NSET=REF8
TOP8REF,DN8REF,5,9

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*ELEMENT,TYPE=CAX4R
26000,26009,26010,26001,26000
*ELGEN,ELSET=TOP8RF
26000,8,1,1,5,9,8,1
*****
*****ELEMENT GENERATION IN "7" REGION*****
*****
*ELEMENT,TYPE=CAX4R
**CPE4R
8040,12375,12377,12252,12250
*ELGEN,ELSET=ELSET7A
8040,18,2,1,1,125,20,1
*ELEMENT,TYPE=CAX4R
8060,12500,12502,12377,12375
*ELGEN,ELSET=ELSET7B
8060,19,2,1,1,125,20,1
*ELEMENT,TYPE=CAX4R
8080,12625,12627,12502,12500
*ELGEN,ELSET=ELSET7C
8080,20,2,1,16,125,20,1
*****
*NSET,NSET=SERV50
12125
*NCOPY,OLD SET=SERV50,NEW SET=SERV51,SHIFT,CHANGE NUMBER=27875
0,0,0
0,0,0
*NSET,NSET=SERV52
12161
*NCOPY,OLD SET=SERV52,NEW SET=SERV53,SHIFT,CHANGE NUMBER=27911
0,0,0
0,0,0
*NFILL
SERV51,SERV53,72,1
*NSET,GENERATE,NSET=SERV54
12125,12161,1
*NSET,GENERATE,NSET=SERV55
40000,40072,2
*MPC
TIE,SERV55,SERV54
*****
*ELEMENT,TYPE=CAX3
30000,40000,11401,11400
*NSET,GENERATE,NSET=SERV10
12125,12163,2
*NSET,GENERATE,NSET=SERV11
12127,12165,2
*NFILL
SERV10,SERV11,2,1
*ELGEN,ELSET=ELSET7D
30000,36,2,1,1,1,1,1
*ELEMENT,TYPE=CAX3
30040,40000,40002,11401
*ELGEN,ELSET=ELSET7DA
30040,36,2,1,1,1,1,1
*ELEMENT,TYPE=CAX3
40000,11401,40002,11402
*ELGEN,ELSET=ELSET7DB
40000,36,2,1,1,1,1,1
*****
*ELEMENT,TYPE=CAX3
30100,12250,12252,12126
*ELGEN,ELSET=ELSET7E
30100,18,2,1,1,1,1,1

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*****
*ELEMENT,TYPE=CAX3
30200,12250,12126,12125
*ELGEN,ELSET=ELSET7F
30200,18,2,1,1,1,1,1
*****
*ELEMENT,TYPE=CAX3
30300,12252,12127,12126
*ELGEN,ELSET=ELSET7G
30300,18,2,1,1,1,1,1
*****
*****
*NSET,NSET=SERV14
12161
*NSET,NSET=SERV15
12163
*NCOPY,OLD SET=SERV15,NEW SET=SERV15A,CHANGE NUMBER=38,SHIFT
0,0,0
0,0,0
*NFILL
SERV14,SERV15A,4,10
*****
*NSET,NSET=SERV16
11473
*NSET,NSET=SERV17
12171
*NCOPY,OLD SET=SERV17,NEW SET=SERV17A,SHIFT,CHANGE NUMBER=-678
0,0,0
0,0,0
*NFILL
SERV16,SERV17A,2,10
*NSET,NSET=SERV16B
11474
*NSET,NSET=SERV17B
12181
*NCOPY,OLD SET=SERV17B,NEW SET=SERV17BA,SHIFT,CHANGE NUMBER=-687
0,0,0
0,0,0
*NFILL
SERV16B,SERV17BA,2,10
*****
*NSET,NSET=SERV18
11475
*NSET,NSET=SERV19
12191
*NCOPY,OLD SET=SERV19,NEW SET=SERV19A,CHANGE NUMBER=-686,SHIFT
0,0,0
0,0,0
*NFILL
SERV18,SERV19A,3,10
*****
*NGEN
12286,12290,1
*****
*ELEMENT,TYPE=CAX3,ELSET=ELSET7J
30600,12161,11483,12036
30601,12036,11483,11473
30602,11473,11483,11474
30603,11474,11483,11484
30604,11484,11483,12181
30900,12181,11483,12171
30605,12171,11483,12161
*****

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30606,12181,12191,11495
30607,11495,11484,12181
30608,11484,11495,11485
30609,11484,11485,11474
30610,11474,11485,11475
*****
30611,11475,25100,25000
30612,11475,11485,25100
30613,11485,25200,25100
30614,11485,11495,25200
30615,11495,25300,25200
30616,11495,12191,25300
30617,12191,25400,25300
30618,12191,25500,25400
*****
30619,12161,12286,12171
30620,12286,12287,12171
30621,12171,12287,12181
30622,12181,12287,12191
30623,12191,12287,12288
30624,12288,25500,12191
*****
30625,12286,12411,12287
30626,12287,12413,12288
30627,12411,12413,12287
*****
*****
*NSSET,NSSET=SERV22
15025,15033
*NSSET,NSSET=SERV23
15050,15058
*NCOPY,OLD SET=SERV23,NEW SET=SERV23A,SHIFT,CHANGE NUMBER=-1
0,0,0
0,0,0
*NFILL
SERV22,SERV23A,2,12
*****
*NSSET,NSSET=SERV24
12289
*NSSET,NSSET=SERV25
15050
*NCOPY,OLD SET=SERV25,NEW SET=SERV25A,SHIFT,CHANGE NUMBER=-2711
0,0,0
0,0,0
*NFILL
SERV24,SERV25A,5,10
*****
*NSSET,NSSET=SERV26
12309
*NSSET,NSSET=SERV27
25560
*NCOPY,OLD SET=SERV27,NEW SET=SERV27A,SHIFT,CHANGE NUMBER=-13191
0,0,0
0,0,0
*NFILL
SERV26,SERV27A,2,30
*****
*NSSET,NSSET=SERV28
12339
*NSSET,NSSET=SERV29
15037
*NCOPY,OLD SET=SERV29,NEW SET=SERV29A,SHIFT,CHANGE NUMBER=-2693
0,0,0

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0,0,0
*NFILL
SERV28,SERV29A,5,1
*****
*NSET,NSET=SERV30
12289
*NSET,NSET=SERV31
12540
*NCOPY,OLD SET=SERV31,NEW SET=SERV31A,SHIFT,CHANGE NUMBER=-243
0,0,0
0,0,0
*NFILL
SERV30,SERV31A,2,4
*****
*ELEMENT,TYPE=CA3,ELSET=ELSET7K
30630,25500,12288,25520
30631,25520,12288,12289
30632,25520,12289,25540
30633,25540,12289,12339
30634,12289,12309,12339
30635,25540,12339,25560
*****
30636,12288,12413,12289
30637,12289,12413,12293
30638,12289,12293,12309
*****
30639,12309,12293,12319
30640,12293,12415,12319
30641,12415,12329,12319
30642,12415,12290,12329
*****
30643,12413,12538,12293
30644,12293,12538,12540
30645,12540,12415,12293
*****
30646,25560,12339,25562
30647,25562,12339,12340
30648,12340,25564,25562
30649,25564,12340,25566
30650,25566,12340,12341
30651,12341,25568,25566
30652,25568,12341,25570
30653,25570,12341,12342
30654,12342,25572,25570
30655,25572,12342,25574
30656,25574,12342,12343
30657,12343,25576,25574
30658,25576,12343,25578
30659,25578,12343,15037
30660,15037,15025,25578
*****
30661,12339,12309,12340
30662,12340,12309,12319
30663,12319,12341,12340
30664,12341,12319,12329
30665,12329,12342,12341
30666,12329,12343,12342
30667,12329,15050,15037
30668,12329,15037,12343
*****
*ELSET,ELSET=ELSET7
ELSET7A,ELSET7B,ELSET7C,ELSET7D,ELSET7DA
ELSET7E,ELSET7F,ELSET7G,ELSET7DB

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ELSET7J,ELSET7K
*****
*****ELEMENT GENERATION IN "8" REGION*****
*****
*ELEMENT,TYPE=CAX4R
**CPE4R
13000,15100,15108,15083,15075
*ELGEN,ELSET=ELSET8A
13000,1,1,1,17,25,10,1
*****
*NGEN
15050,15058,4
*NGEN
15037,15045,2
*****
*ELEMENT,TYPE=CAX3,ELSET=ELSET8B
30700,15025,15037,26046
30701,26046,15037,15039
30702,15039,26047,26046
30703,15039,26048,26047
30704,15039,15041,26048
30705,15041,26049,26048
30706,15041,26050,26049
30707,15041,15043,26050
30708,26050,15043,26051
30709,26051,15043,26052
30710,26052,15043,15045
30711,15045,26053,26052
*****
30712,15037,15050,15039
30713,15039,15050,15054
30714,15054,15041,15039
30715,15041,15054,15043
30716,15043,15054,15058
30717,15058,15045,15043
*****
30718,15050,15075,15054
30719,15054,15075,15083
30720,15083,15058,15054
*****
*ELSET,ELSET=ELSET8
ELSET8A,ELSET8B
*****
*****ELEMENT GENERATION IN "9" REGION*****
*****
*ELEMENT,TYPE=CAX4R
**CPE4R
14000,16127,16129,16004,16002
*ELGEN,ELSET=ELSET9A
14000,19,2,1,3,125,20,1
*ELEMENT,TYPE=CAX4R
14070,16500,16502,16377,16375
*ELGEN,ELSET=ELSET9B
14070,20,2,1,17,125,20,1
*****
*NGEN
16125,16127,1
16250,16252,1
*ELEMENT,TYPE=CAX3,ELSET=ELSET9C
30800,16250,16375,16251
30801,16251,16375,16377
30802,16377,16252,16251
*****

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30803,16251,16252,16127
30804,16127,16126,16251
30805,16251,16126,15045
30806,15045,16250,16251
30807,15045,16126,16125
*****
*NSET,NSET=SERV40
9002
*NCOPY,OLD SET=SERV40,NEW SET=SERV41,CHANGE NUMBER=20998,SHIFT
0,0,0
0,0,0
*NSET,NSET=SERV42
16126
*NCOPY,OLD SET=SERV42,NEW SET=SERV42A,CHANGE NUMBER=13904,SHIFT
0,0,0
0,0,0
*NFILL
SERV41,SERV42A,3,10
*****
*ELEMENT,TYPE=CAX3,ELSET=ELSET9D
30810,26017,9001,26008
30811,26017,26026,30010
30812,30010,9001,26017
30813,9001,30010,9002
*****
30814,26026,26035,30010
30815,30010,26035,30020
30816,30020,26035,26044
30817,26044,16125,30020
30818,30020,16125,16126
*****
30819,30020,16126,16127
30820,16127,30010,30020
30821,30010,16127,9003
30822,9003,16127,16002
30823,9003,9002,30010
*****
*ELSET,ELSET=ELSET9
ELSET9A,ELSET9B,ELSET9C,ELSET9D
*MPC
TIE,SERV41,SERV40
*****
*****CONNECTION OF THE REFINED AREAS*****
*****IN 7 AND 8 REGIONS*****
*****
*NSET,NSET=UP7REFA
11476,11477,11478
22400,22405,22410,22415,22420,22425,22430
22435,22440,22445,22450
*NSET,GENERATE,NSET=UP7REFB
25000,25060,20
25062,25080,2
*MPC
TIE,UP7REFB,UP7REFA
*NSET,NSET=SERV70
11479
*NSET,NSET=SERV71
22400
*MPC
TIE,SERV70,SERV71
*****
*NSET,NSET=UP8REFA

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1050,1060,1070,1080,1090,1100,1110
16000
*NSET,NSET=UP8REFB
26001,26002,26003,26004
26005,26006,26007,26008
*MPC
TIE,UP8REFB,UP8REFA
*MPC
TIE,D8REF,D7
*****
*NSET,GENERATE,NSET=RGHT7REF
25180,25580,100
*NSET,GENERATE,NSET=LFT8REF
26009,26045,9
*MPC
TIE,LFT8REF,RGHT7REF
*NSET,NSET=D7DR
25580
*NSET,NSET=D7DRD
12165
*MPC
TIE,D7DR,D7DRD
*****
*****TIE FIXING*****
*****
*NSET,NSET=TIE1
22450
*NSET,NSET=TIE2
12040
*MPC
TIE,TIE1,TIE2
*****
*ELSET,ELSET=FLAW
FLAW1,FLAW2,INTERREF
*INTERFACE,ELSET=FLAW,NAME=FLAW
*SURFACE CONTACT
2.e-11,0.
**,SOFTENED
**1.e-9,1.e6
*****
*****PLANE STREIN CONDITION*****
*****
*NSET,NSET=LEFT
LEFT1,LEFT5,LEFT6,LEFT7
*NSET,NSET=RIGHT
RIGHT3,RIGHT4,RIGHT9
*****
*NSET,NSET=BOTTOM
BOTTOM7,BOTTOM8,BOTTOM9
*****
*****NODE SETS FOR AMPLITUDE OPTION*****
*****
*NSET,NSET=BOT1,GENERATE
14500,14540,2
18500,18540,2
*NSET,NSET=BOT2,GENERATE
14375,14415,2
18375,18415,2
*****
*****MATERIAL PROPERTIES*****
*****
*****FILM AND SUBSTRATE ELSETES**
*ELSET,ELSET=FILM

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ELSET1A,ELSET1B,ELSET1C
ELSET2,ELSET3
ELSET5A,ELSET5B,ELSET5C
ELSET4UP,CELUP,FILMREF
*ELSET,ELSET=SUBSTRAT
ELSET6A,ELSET6B,ELSET6C,ELSET4DN,CELDOWN,ELSET7
ELSET8,ELSET9
SUBREF,REF7A,REF7B, TOP8RF
*****FILM*****
*SOLID SECTION,ELSET=FILM,MATERIAL=FILM
*MATERIAL,NAME=FILM
*ELASTIC,TYPE=ISOTROPIC
1.049E11,0.397
**1.298E11,0.343
**PLASTIC
**1.e6,0
**3e8,0.01
*DENSITY
8.57E3
**8.96E3
**DAMPING
**BETA=1E-10
*****SUBSTRATE*****
**for Al2O3: 10^11 dynes/cm^2; C11=49.6;C33=50.2;C44=20.6
**C12=10.9;C13=4.8;C14=3.8
**S11=2.18;S33=2.02;S44=5.04;S12=-.50;S13=-0.16;S14=-0.49
***DENSITY=3.99(ALPHA)
**LAMBDA=C12;LAMBDA+2*MJU=C11
**E=(C11-C12)*(C11+2*C12)/(C11+C12);n=LAMBDA/(2*(LAMBDA+MJU))
**n=C12/(C11+C12)
*SOLID SECTION,ELSET=SUBSTRAT,MATERIAL=SUBSTRAT
*MATERIAL,NAME=SUBSTRAT
*ELASTIC,TYPE=ISOTROPIC
4.56E11,0.18
**2.7e9,0.3
**ELASTIC,TYPE=ANISOTROPIC
*497.6E9,162.6E9,497.6E9,117.2E9,117.2E9,501.9E9,0.,0.
*0.,167.5E9,0.,0.,0.,22.9E9,147.2E9,-22.9E9
*22.9E9,0.,0.,0.,147.2E9
*DENSITY
3.9E3
**2.2E3
**DAMPING
**BETA=1E-10
*****
*****c=sqrt((E/r)*(1-n)/((1+n)*(1-2*n)))=11149.5***for PG=1285.3
*****dx=20.3e-6/n(n=20)=1.015e-6*****tx=dx/c=9.10357e-11***for PG=3.9678e-10; 7.8967e-10
*****
**C {L}=SQRT(C11/R)
*****
**AMPLITUDE,VALUE=R,INPUT=VEL.AMP,NAME=VELOC
*****
*****PROCEDURE*****
*****
**LET'S DEFINE INITIALLY STRESSED ELEMENTS*****
*****
*ELSET,ELSET=STRESSED,GENERATE
3000,3003
3010,3013
3020,3023
3030,3033
3040,3043
3050,3053

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3060,3063
3070,3073
*****
501,504
541,544
581,584
621,624
661,664
*****
4000,4022
4080,4102
4160,4182
4240,4262
4320,4342
4400,4422
4480,4502
4560,4582
*****
5320,5323
5400,5403
5480,5483
5560,5563
*****
5324,5396,4
5404,5476,4
5484,5556,4
5564,5636,4
*STEP
*STATIC
**INITIAL CONDITIONS,TYPE=STRESS
**STRESSED,1e7,0,1e7,0
**STEP
**STATIC
*BOUNDARY
RIGHT,1
LEFT,1
*RESTART,WRITE,FREQUENCY=100
*END STEP
*STEP,INC=40000
*DYNAMIC
**HAFTOL=10e-1
**,ALPHA=-0.2
**NOHAF
5e-11,3.E-8,1E-15,5e-10
*BOUNDARY
RIGHT,1
LEFT,1
*RESTART,WRITE,FREQUENCY=100
*****
*****
*BOUNDARY,USER SUB
BOT1,2
*****
*****J-INTEGRAL CALCULATION*****
*****
*J-INTEGRAL,CONTOUR=5,OUTPUT=BOTH
0,1
TIP
*NODE FILE,NSET=BOT2
V
U
*ELSET,ELSET=ELREP
5392,5312

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*EL FILE,ELSET=ELREP
S
*NSET,NSET=REPORT
14500,14375,6000,1330,1000,11000,8080
*NODE FILE,NSET=REPORT
V
U
*NODE PRINT,NSET=BOT1,FREQUENCY=0
V
U
*EL PRINT,ELSET=ELSET1A,FREQUENCY=0
S22
*END STEP
*USER SUBROUTINE
SUBROUTINE DISP(U,KSTEP,KINC,TIME,NODE,JDOF)
INCLUDE 'ABA_PARAM.INC'
DIMENSION U(3),TIME(2)
INTEGER KK,KC, NNODE,JDOF,CHECK
REAL*8 V2,U2,AMPL,tt,tx,V1,U1,A
COMMON /CHECK/CHECK,/R/VR1(50),/UR1/UR1(50)
COMMON /VR2/VR2(50),/UR2/UR2(50)
COMMON /B/VB(50),/UB/UB(50),/TTIME/TTIME,/TB/TB

tx=9.10357e-11
c tx=7.8967e-10
c      file data is f3.dat
VAR=-1.0
A=3.7431
c A=8.7431
A=A*1E-9
B=3.9050
c B=8.9050
B=B*1E-9
C=0.65488
C=C*1000.
C=C*15.
AMPL=C*(DEXP(-TIME(1)/A)-DEXP(-TIME(1)/B))*VAR

IF(CHECK.NE.KINC)THEN

IF(NODE.EQ.14500)THEN
c      print*
c      print*,'bef:', 'VELOCITY=',AMPL,'node=',NODE
c      print*,'bef:', 'TIME=',TIME(1)
c      print*,'bef:', 'U(1)=',U(1),', ', 'U(2)=',U(2),', ', 'U(3)=',U(3)
c      print*,'bef:', 'UB(1)=',UB(1),', ', 'VB(1)=',VB(1)
c      print*,'bef:', 'UR2(1)=',UR2(1),', ', 'VR2(1)=',VR2(1)
c      print*,'bef:', 'UR1(1)=',UR1(1),', ', 'VR1(1)=',VR1(1)
TTIME=TB
ENDIF

TB=TIME(1)
tt=TB-TTIME
IF(tt.LE.0.)GO TO 25
C      or go to 20

IF(NODE.EQ.14500)THEN
OPEN(UNIT=14,FILE='/tmp/alex/v2',ACCESS='DIRECT',RECL=100)
OPEN(UNIT=15,FILE='/tmp/alex/u2',ACCESS='DIRECT',RECL=100)
DO 10 K=1,49
UR1(K)=UB(K)

```

```

VR1(K)=VB(K)
10      CONTINUE

WRITE(*,*)'INCREMENT IN ABAQUS=',KINC,'JDOF=',JDOF
c      CALL SYSTEM('sleep 2')
CALL SYSTEM('/tmp/alex/vel')
ELSE
GO TO 20
ENDIF
20      CONTINUE

C in JNODE(K) should be numbers 14500-14538,15500-15507
c 18500-18540

IF(NODE.GE.14500.AND.NODE.LE.14540)THEN
KK=(NODE-14499+1)/2
ELSEIF(NODE.GE.18500.AND.NODE.LE.18540)THEN
KK=(NODE-18456)/2
ELSE
GO TO 300
ENDIF

READ(UNIT=14,REC=KK)V2
VR2(KK)=V2
READ(UNIT=15,REC=KK)U2
UR2(KK)=U2

25      CONTINUE

TB=TIME(1)
tt=TB-TTIME
U(2)=AMPL+(UR2(KK)-UR1(KK))/tx+(VR2(KK)-VR1(KK))*(tt/tx)
VB(KK)=U(2)
IF(tt.NE.0.)THEN
U(3)=(U(2)-VR1(KK))/tt
ELSE
U(3)=0.
ENDIF
U(1)=UR1(KK)+VR1(KK)*tt
UB(KK)=U(1)

IF(NODE.EQ.18540)THEN
CLOSE(UNIT=14,STATUS='KEEP')
CLOSE(UNIT=15,STATUS='KEEP')
CHECK=KINC
ENDIF

ELSE
GO TO 30
30      CONTINUE
ENDIF

IF(CHECK.NE.KINC)GO TO 200
IF(NODE.GE.14500.AND.NODE.LE.14540)THEN
KK=(NODE-14499+1)/2
ELSEIF(NODE.GE.18500.AND.NODE.LE.18540)THEN
KK=(NODE-18456)/2
ELSE
GO TO 300
ENDIF

TB=TIME(1)
tt=TB-TTIME

```

```

U(2)=AMPL+(UR2(KK)-UR1(KK))/tx+(VR2(KK)-VR1(KK))*(tt/tx)
VB(KK)=U(2)
IF(tt.NE.0.)THEN
U(3)=(U(2)-VR1(KK))/tt
ELSE
U(3)=0.
ENDIF
U(1)=UR1(KK)+VR1(KK)*tt
UB(KK)=U(1)

200      CONTINUE

c      IF(NODE.EQ.18540)THEN
c      print*
c      print*,'bef:', 'VELOCITY=', AMPL, 'node=', NODE
c      print*,'bef:', 'KK=', KK
c      print*,'bef:', 'TIME=', TIME(1)
c      print*,'bef: ', 'U(1)=', U(1), ' ', 'U(2)=', U(2), ' ', 'U(3)=', U(3)
c      print*,'bef: ', 'UB(KK)=', UB(KK), ' ', 'VB(KK)=', VB(KK)
c      print*,'bef: ', 'UR2(KK)=', UR2(KK), ' ', 'VR2(KK)=', VR2(KK)
c      print*,'bef: ', 'UR1(KK)=', UR1(KK), ' ', 'VR1(KK)=', VR1(KK)
c      ENDIF

300      CONTINUE
RETURN
END

```

This Fortran program has been used to generate list of the mesh coordinates according to the geometry of the microcracks measured by SFM.

program node c

```

:   program calculates the node list

c           ALL DATA IN "SI" (meters, seconds, kg/m^3, ...)
c
c   "t"      is the film thickness
c           t=1.e-6
c   "d"      is the size of the central region
c           d=2.e-7
c d=5.e-7
c   "h"      is the substrate thickness
h=-20.e-6
c   "a"      is the crack length
c           a=5.e-7
c a=60.e-6
c   "w"      is the system width
w=20.e-6
c w=120.e-6
c
c   10 FORMAT(I6,A,E10.4,A,E10.4)
c
c           OPEN(UNIT=20,FILE="node.1st")
c
c           DO 30 I=0,80
c             dd=defl(a,I)
dd=defl1(a,I)
dd=0.
c             x=((a-d)/80)*I
c             WRITE(UNIT=20,FMT=10)6000+I*1,"",x,"",dd+t
c             WRITE(UNIT=20,FMT=10)6800+I*1,"",x,"",dd+d
c             WRITE(UNIT=20,FMT=10)10000+I*1,"",x,"",dd+d
c             WRITE(UNIT=20,FMT=10)10400+I*1,"",x,"",dd
30      CONTINUE

c
c           WRITE(UNIT=20,FMT=10)7080,"",(a-d),"",t
c           WRITE(UNIT=20,FMT=10)7000,"",a+d,"",t
c           WRITE(UNIT=20,FMT=10)8000,"",a+d,"",t
c           WRITE(UNIT=20,FMT=10)8080,"",w,"",t
c
c           WRITE(UNIT=20,FMT=10)7880,"",(a-d),"",d
c           WRITE(UNIT=20,FMT=10)7800,"",a+d,"",d
c           WRITE(UNIT=20,FMT=10)8800,"",a+d,"",d
c           WRITE(UNIT=20,FMT=10)8880,"",w,"",d
c
c           WRITE(UNIT=20,FMT=10)1290,"",(a-d),"",d
c           WRITE(UNIT=20,FMT=10)1210,"",a+d,"",d
c           WRITE(UNIT=20,FMT=10)9900,"",a+d,"",d
c           WRITE(UNIT=20,FMT=10)9980,"",w,"",d
c
c           WRITE(UNIT=20,FMT=10)1330,"",(a-d),"",0.
c           WRITE(UNIT=20,FMT=10)5330,"",a,"",0.
c           WRITE(UNIT=20,FMT=10)1170,"",a+d,"",0.
c           WRITE(UNIT=20,FMT=10)9500,"",a+d,"",0.
c           WRITE(UNIT=20,FMT=10)9580,"",w,"",0.
c
c           WRITE(UNIT=20,FMT=10)11000,"",0.,"",0.
c           WRITE(UNIT=20,FMT=10)11080,"",(a-d),"",0.

```

```

WRITE(UNIT=20,FMT=10)1000,"",(a-d),"",0.
WRITE(UNIT=20,FMT=10)5000,"",a,"",0.
WRITE(UNIT=20,FMT=10)1160,"",(a+d),"",0.
WRITE(UNIT=20,FMT=10)9400,"",(a+d),"",0.
WRITE(UNIT=20,FMT=10)9480,"",w,"",0.

WRITE(UNIT=20,FMT=10)11400,"",0.,",",(-1.)*d
WRITE(UNIT=20,FMT=10)11480,"",(a-d),"",(-1.)*d
WRITE(UNIT=20,FMT=10)1040,"",(a-d),"",(-1.)*d
WRITE(UNIT=20,FMT=10)1120,"",(a+d),"",(-1.)*d
WRITE(UNIT=20,FMT=10)9000,"",(a+d),"",(-1.)*d
WRITE(UNIT=20,FMT=10)9080,"",w,"",(-1.)*d

WRITE(UNIT=20,FMT=10)12000,"",0.,",",(-1.)*d
WRITE(UNIT=20,FMT=10)12040,"",(a-d),"",(-1.)*d
WRITE(UNIT=20,FMT=10)15000,"",(a-d),"",(-1.)*d
WRITE(UNIT=20,FMT=10)15008,"",(a+d),"",(-1.)*d
WRITE(UNIT=20,FMT=10)16000,"",(a+d),"",(-1.)*d
WRITE(UNIT=20,FMT=10)16040,"",w,"",(-1.)*d

WRITE(UNIT=20,FMT=10)14500,"",0.,",",h
WRITE(UNIT=20,FMT=10)14540,"",(a-d),"",h
WRITE(UNIT=20,FMT=10)15500,"",(a-d),"",h
WRITE(UNIT=20,FMT=10)15508,"",(a+d),"",h
WRITE(UNIT=20,FMT=10)18500,"",(a+d),"",h
WRITE(UNIT=20,FMT=10)18540,"",w,"",h

STOP
END

REAL FUNCTION defl(a,node)
REAL a,xx
INTEGER node
s=a*1.e6/80
xx=(80-node)*s
b=0.00296
c=0.00089
a1=-0.0122
b1=0.00156
c1=0.00016
corr=1.
aa=0.0
defl=corr*(1.e-6)*(aa*xx+b*xx**2+c*xx**3)/
c (1.+a1*xx+b1*xx**2+c1*xx**3)
END

REAL FUNCTION defl1(a,node)
real a,xx
integer node
s=a*1.e6/80
xx=(80-node)*s
a2=0.0074315
a3=-6.785e-4
a4=5.8251e-5
a5=-2.9732e-6
a6=8.3166e-8
a7=-1.3107e-9
a8=1.1089e-11
a9=-3.9375e-14
y=a2*xx**2+a3*xx**3+a4*xx**4+a5*xx**5+a6*xx**6
y=y+a7*xx**7+a8*xx**8+a9*xx**9
defl1=y*1.e-6

```

The following Fortran program scanned ABAQUS results file to determine the velocities of the nodes at different time increments and allowed to define non-reflecting boundary condition in the substrate.

PROGRAM VELOCITY c

```

PROGRAM scans the result file to
c determine the velocities of the nodes
IMPLICIT REAL*8 (A-H,O-Z)
INTEGER NUMBER,IOS,KK,NNODE,NRU,JUNIT
DOUBLE PRECISION ARRAY,U2(50),V2(50),AMPL,U,A
DOUBLE PRECISION U1(50),V1(50)
DIMENSION ARRAY(513), JRRAY(2,513),LRUNIT(2,1)
EQUIVALENCE (ARRAY(1),JRRAY(1,1))
REAL*8 TIME
CHARACTER*80 FNAME
COMMON /JOBPAR/FNAME
FNAME='/tmp/alex/ppp'

OPEN(UNIT=15,FILE='/tmp/alex/v2',ACCESS='DIRECT',RECL=100)
OPEN(UNIT=17,FILE='/tmp/alex/u2',ACCESS='DIRECT',RECL=100)
OPEN(UNIT=16,FILE='/tmp/alex/inc')
NRU=1
LRUNIT(1,1)=8
LRUNIT(2,1)=2
CALL INITPF(NRU,LRUNIT,LOUTF)
JUNIT=8
CALL DBRNU(JUNIT)
DO 100 KOUNTER = 1, 9999999
CALL DBFILE(0,ARRAY,JRCD)
IF (JRCD.NE.0) GO TO 110
KEY = JRRAY(1,2)

IF (KEY.EQ.2000) THEN
KINGS=JRRAY(1,9)
TIME=ARRAY(4)
ENDIF

IF(KEY.EQ.102) THEN
NNODE=JRRAY(1,3)
VEL=ARRAY(5)
IF(NNODE.GE.14375.AND.NNODE.LE.14415)THEN
KK=(NNODE-14373)/2

ELSEIF(NNODE.GE.18375.AND.NNODE.LE.18415)THEN
KK=(NNODE-18331)/2
ELSE
GO TO 100
ENDIF
V2(KK)=VEL
ENDIF

IF(KEY.EQ.101) THEN
NNODE=JRRAY(1,3)
U=ARRAY(5)
IF(NNODE.GE.14375.AND.NNODE.LE.14415)THEN
KK=(NNODE-14373)/2

ELSEIF(NNODE.GE.18375.AND.NNODE.LE.18415)THEN
KK=(NNODE-18331)/2
ELSE
GO TO 100
ENDIF

```

```

U2(KK)=U
ENDIF

100 CONTINUE
110 CONTINUE

DO 200 K=1,42
WRITE(UNIT=15,REC=K)V2(K)
WRITE(UNIT=17,REC=K)U2(K)
200 CONTINUE

WRITE(16,*)KINCS
c WRITE(*,*)'INCREMENT NUMBER=',KINCS
CLOSE(UNIT=8, STATUS = 'KEEP')
CLOSE(UNIT=15,STATUS = 'KEEP')
CLOSE(UNIT=17,STATUS = 'KEEP')
CLOSE(UNIT=16,STATUS='KEEP')
STOP
END

c

```