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**THIN-FILM ADHESION MEASUREMENT BY
LASER-INDUCED STRESS WAVES**

BY

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B.S., University of Science and Technology of China, 1994

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THESIS

**Submitted in partial fulfillment of the requirements
for the degree of Doctor of Philosophy in Theoretical and Applied Mechanics
in the Graduate College of the
University of Illinois at Urbana-Champaign, 2002**

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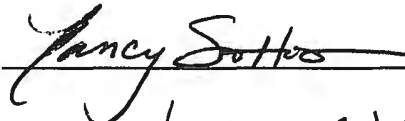
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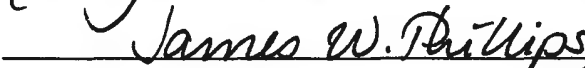
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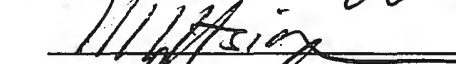
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ABSTRACT

Thin films are crucial components in a wide range of multilayer microelectronic and optical devices. They are also desirable candidates for micro-actuators in micro-electro-mechanical devices. The size scales and dissimilar nature of the constituents present great challenges regarding thermomechanical integrity and reliability. Interfacial adhesion is a critical parameter governing the mechanical behavior and reliability of a thin film on a substrate. Laser-spallation techniques have been developed to investigate the intrinsic strength of planar interfaces using laser-generated stress waves. In all the laser-spallation experiments reported in the literature, only normal pressure pulses were applied to the films. Consequently, the interfaces were subjected to only tensile (mode I) loading. In most commercial applications, however, thin-film interfaces fail under mixed-mode conditions. The goal of this dissertation is to develop the use of laser-generated stress waves to characterize intrinsic interfacial strength of thin films under mixed-mode conditions.

With a view towards optimal design for the mixed-mode loading experiment, a rigorous theoretical analysis of stress-wave generation, propagation, and attenuation is carried out followed by a systematic experimental study of tensile spallation. The spallation behavior of aluminum thin films is investigated as a function of substrate thickness, film thickness, laser energy, and various parameters governing the source. Two different substrate materials—single crystal silicon (100) and fused silica—are considered. Theoretical analysis provides meaningful insight into the nature of the wave propagation, while the experimental parametric study reveals important design variables for improving the tensile spallation test and developing the mixed-mode loading experiments. Of particular importance is that fused-

silica substrates, with their negative nonlinear elasticity, cause the compressive stress wave generated by the pulsing laser to evolve a decompression shock, which is critical for generation of the fast fall times needed for significant loading of thin-film interfaces.

Based on the theoretical and experimental parametric study results, the planar tensile sample geometry is modified to a triangular prism so that a high-strain-rate shear wave is generated (by mode conversion at an oblique surface) and then allowed to impinge upon the test film. By allowing the initial longitudinal wave to mode-convert to a shear wave and a secondary longitudinal wave at the oblique surface, mixed-mode loading of the thin film interface is realized. The film failure patterns observed under mixed-mode conditions are quite different from those observed in the tensile case. By measuring the out-of-plane displacement of the test film using an interferometer, interfacial strength under mixed-mode conditions is obtained and compared with the tensile values. The shear strength under mixed-mode conditions is slightly higher than the tensile strength for the same aluminum/fused silica interface. The mixed-mode loading experiment developed in this dissertation provides a new tool to explore a range of thin-film interfacial failure modes as well as the role of such extrinsic effects as residual stress, surface roughness, and imperfections.

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Chapter 1

Introduction

1.1 Background

Thin films are crucial components in a wide range of multilayer microelectronic and optical devices. They are also desirable candidates for micro-actuators in micro-electro-mechanical systems due to their high energy densities and strain capabilities. The size scales and dissimilar nature of the constituents present great challenges for thermomechanical integrity and reliability [1].¹ Large residual stresses induced in films during fabrication have a significant impact on mechanical properties and performance of the films. Under appropriate conditions the residual stress can cause the film to decohere, crack, or buckle away from the substrate. A particular challenge is the design and implementation of test procedures that measure thin-film interface properties.

Interfacial adhesion is a critical parameter governing the mechanical behavior and reliability of a thin film on a substrate. In any practical applications of thin films, the level of adhesion required for the thin film structure depends on the severity of the operating environment. Mittal [2, 3] classified adhesion into two categories: “basic adhesion” and “practical adhesion”. For an ideal planar interface, the basic adhesion strength corresponds to the summation of all atomic and molecular bonding forces acting between the adjoining material surfaces. In practice, however, the interfacial region can depend on many parameters,

¹Numbers in square brackets [] denote entries in the List of References, beginning on page 146.

such as substrate surface morphology, chemical interdiffusion, contamination, and defects [4, 5]. Thus, the measured force or work required to detach a film or coating from the substrate is defined as the practical adhesion. Testing interfacial adhesion strength typically involves applying external loads by some means to the surface of the film and generally results in a complex stress field at the interface because of the load-application mechanism, the dissimilar materials involved, and often large induced deformations.

Significant effort has been devoted to the development of test procedures for the measurement of thin-film adhesion, of which the most common are the scratch, peel, pull, blister, and indentation tests. The first quantitative use of the scratch test was reported by Heavens [6] and Weaver [7]. Later this method became a commonly used technique in characterizing the adhesion and mechanical durability of thin hard films, which usually have a high adhesion strength [8-13]. In the scratch test, a conical diamond indenter is driven into a film sample until a load drop occurs, indicating delamination. The scratch test is simple, but extracting a quantitative adhesion value has proven to be difficult [9]. Both the testing parameters (loading rates, stylus radius) and the sample parameters (film thickness and substrate hardness) influence the test results. Thus, the scratch test usually provides only a comparative measure of interfacial strength.

In the peel test, first used by Strong [14], an adhesive layer is bonded to the film surface and lifted off, potentially removing the film. In an alternative version of this test, another thicker layer is deposited on the thin film and the residual stress causes the layer to peel off the film [15]. Peel strength depends on many parameters, including film thickness and substrate compliance, and can involve substantial elastic-plastic deformation of the film [16]. The peel test is suitable only for films in which the adhesion to the substrate is weaker than to the adhesive layer. For cases where the adhesion is considerably higher, the pull test [9, 17, 18] is often used. In a pull test, a stud is attached to the film using a strong adhesive. The perpendicular force required to remove the stud is measured and then divided by the area of removed film to calculate the adhesive strength. A major limitation of the

pull test is that the maximum measurable strength cannot exceed the maximum bonding strength of available adhesives. In both the peel and pull tests the potential exists that the adhesive used in the attachment process to the thin film surface can penetrate the film and affect the film and interface. Another technique used for thin-film adhesion measurement is the blister test, in which a film layer is deposited on a rigid flat substrate with a center perforation. A pressurizing fluid is injected through the perforation creating a blister and causing a progressive debonding of the film/substrate interface at a critical pressure [19, 20]. Blister tests usually require difficult sample preparation procedures. Indentation-induced delamination has also been proposed for measuring film adhesion [21, 22]. As in the scratch test, a diamond indenter is used to initiate and propagate a delamination, which is then examined optically and related to the measured load and displacement. Indentation tests are difficult when applied to ductile or strongly adhering films because of the problem of initiating a delamination [23]. During all these tests—scratch, peel, pull, blister and indentation—the interface is subjected to very high stress levels and consequent inhomogeneous deformations. Large amounts of plastic deformation can result (especially in the peel test) and dominate the behavior during the test [24]. The stress fields are difficult to analyze and the resulting adhesion measurements tend to be qualitative and comparative.

Different from these methods, laser-spallation methods load the interface in a remote, non-contacting manner using laser-generated stress waves. This technique was first introduced by Vossen [25], who loaded the film using stress waves produced by short laser pulses as developed by Ready [26], Anderholm [27], Peercy et al. [28], Yang [29] and Fox [30]. This technique involves impinging a high-energy laser pulse (with a duration of nanoseconds) from a Q-switched laser onto a thin absorbing layer confined between a confining plate and the back surface of the substrate. Upon absorbing the laser energy, the sudden expansion of the confined layer generates a compressive stress wave directed towards the test film. The reflection of the compressive wave packet from the surface of the test film generates a tensile pulse, which leads to spallation of the test film. Yang [29] used an x-cut quartz crystal to

measure the stress at the front surface of the substrate.

Gupta and co-workers [31, 32] further developed Vossen and Yang's technique to measure thin-film interfacial strength. As in Yang's work [29], the stress impinging the interface was measured using a micro-electronic device with an x-cut piezoelectric crystal. In the later work of Gupta et al. [33-36], a laser-Doppler interferometer was introduced to measure the free-surface displacement and velocity, which were then related to interface stress. This technique was used to measure the tensile strength of a wide variety of metal/ceramic interfaces [33, 37], ceramic/ceramic interfaces [34], thermal-barrier coatings [38], ice/metal interfaces [39] and fiber/matrix interfaces [40]. More recently, Boustie et al. [41] used the laser-spallation technique to test adhesion of some metal/metal film interfaces with film thicknesses on the order of tens of micrometers. In their tests, upper and lower laser-intensity thresholds for spallation generation were determined experimentally and a traction range for debonding at the interface was determined numerically.

In all the laser spallation experiments reported in the literature, only normal pressure pulses were applied to the films. Consequently, the interfaces were subjected only to tensile (mode I) loading. In most commercial applications, however, thin-film interfaces fail under mixed-mode conditions [1]. Design of experiments to measure the interface debonding over a range of mode mixity relevant to practical problems is a significant challenge. The ultimate goal of this research is to develop an experimental technique to obtain a range of mixed-mode interfacial failure and quantitatively characterize the mixed-mode interfacial adhesion strength of thin films.

1.2 Overview of the Dissertation

This dissertation investigates the feasibility of using laser-generated stress waves to produce mixed-mode failure of thin-film interfaces and to characterize quantitatively the interfacial adhesion strength.

In Chapter 2, theoretical analysis is developed to gain insights of the wave-generation mechanism following a laser pulse deposition at the interface between two adjacent materials, the subsequent geometric attenuation due to finite YAG source size, the development of a decompression shock in a fused-silica substrate, and the associated shock attenuation. Results of the theoretical analysis are used to guide the subsequent experimental investigations.

In Chapter 3, a parametric study on the tensile laser spallation was carried out. The spallation behavior of aluminum thin films was investigated as a function of substrate thickness, film thickness, confining layer thickness, laser energy, and various parameters governing the source. Two different substrate materials—single crystal silicon (100) and fused silica—were studied. The parametric study results reveal important design parameters for improving the tensile spallation tests and developing the mixed-mode loading experiments.

In Chapter 4, the tensile spallation technique is successfully extended to the case of mixed-mode loading. The planar tensile sample geometry is modified to a triangular prism so that a high-strain-rate shear wave is generated (by mode conversion at an oblique surface) and then allowed to impinge upon the test film. By allowing the initial longitudinal wave to mode-convert to a shear wave and a secondary longitudinal wave at the oblique surface, mixed-mode loading of thin films is realized. Thin film failure patterns observed under mixed-mode and the corresponding interfacial strength measurements are compared with those obtained from the tensile experiments.

In Chapter 5, conclusions are reached and possible future research directions are discussed.

Chapter 2

Theory of Stress-Pulse Generation and Propagation

2.1 Introduction

In order to achieve an improved understanding of the wave-generation mechanism in the laser-spallation technique and to extend this technique successfully to mixed-mode loading of thin film interfaces, a rigorous theoretical analysis of the stress-wave generation, propagation, and attenuation is carried out.

Thermo-elastic calculations of the stress pulses generated by a thermal deposition of a YAG laser pulse at the interface between two adjacent media is provided. Calculations are based on one-dimensional plane wave assumptions. Solutions for both delta pulse deposition and Gaussian pulse deposition are obtained. Substrate displacements and substrate and interface stress are calculated using wave-propagation theory.

In practice, however, the YAG laser beam is of finite size. The stress wave will attenuate as it travels through the substrate and the plane-wave assumption remains valid only for a certain distance. Attenuation limits the thickness of the substrate used in the experiments. A theoretical analysis of stress wave attenuation with respect to the YAG beam size is carried out to determine the plane-wave regime and the maximum thickness of the substrate for the experiments.

The ultimate purpose of this project is to determine the interface strength of thin-film interfaces. Thus, sufficient stress must be reached at the interface in order to initiate interface failure. Due to the releasing of the stress at the test film surface, the interface stress is much lower than the stress in the substrate. Efficient loading of the interface requires a short fall time in the substrate stress. A possible solution to this problem is to use materials with nonlinear properties to develop a shock in the substrate. One such candidate of substrate materials is fused silica. With its unique negative nonlinear elasticity, fused silica softens under modest compression and helps to develop a shock in the substrate and enhances the loading at the film interface. The shock development mechanism in fused silica is thoroughly studied in the present work.

The theoretical analysis in this chapter provides meaningful insights into the nature of the wave propagation and useful guidelines for choosing the sample material and sample geometries for both tensile-mode and mixed-mode loading experiments.

2.2 Thermo-elastic Calculations

2.2.1 Mathematical Statement of the Problem

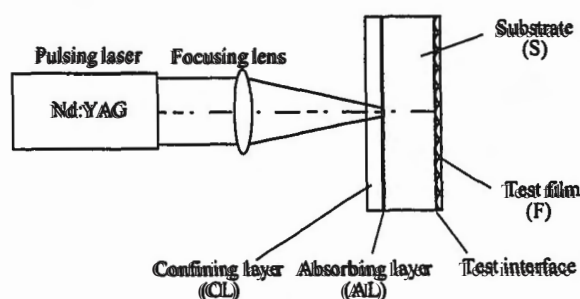


Figure 2.1: Schematic of the tensile laser-spallation technique.

A schematic of the tensile laser spallation experiment used both in the literature [31-40] and in the initial parametric studies of this work [42] is shown in Fig. 2.1. The thin film sample consists of (from left to right): a confining layer (CL), a thin energy absorbing

layer (AL), a substrate (S) and a test film (F). An infra-red, Nd:YAG laser pulse ($\lambda=1064$ nm) with a variable energy content between 1 and 110 mJ, and a rise time of order of 5 ns is incident on a metallic absorbing layer sandwiched between the confining layer and substrate. The energy-absorbing layer is chosen to be much thicker (typically 0.5 μm) than the critical penetration depth (on the order of tens of nanometers) of laser light at this wavelength. Under this consideration, it is assumed that the total laser energy is deposited at the interface of the confining layer and the energy-absorbing layer. A longitudinal stress wave of rise time comparable to that of the laser pulse is emitted from the metallic layer. The wave that propagates towards the film/substrate interface is then reflected back from the free film surface into a tensile wave, which then loads the test interface in tension.

The essential mechanics behind the generation of the compressive stress wave following the deposition of the thermal energy at an interface is described in the texts of Achenbach [43] and Graff [44]. It was also described by Gupta et al. [32].

For a short-duration laser pulse (within a few nanoseconds), the thermal diffusion distance (usually a few microns) in the absorbing layer is negligible compared with the radius of the laser heating spot (in millimeters). Thus the material behavior is completely governed by the amplitude of the laser energy impinging on it. Provided that over the central portion of the laser beam the incident energy density is flat, a one-dimensional planar wave idealization can be adopted as in the following derivations.

The one-dimensional heat-transfer equation in all relevant media is expressed as

$$\frac{\partial T_i}{\partial t} = D_i \frac{\partial^2 T_i}{\partial x^2}, \quad (2.1)$$

where subscript i refers to CL, AL, S. The front test film F is too far to be affected by the thermal effect. And D is the thermal diffusivity of each medium, equal to $k/\rho C$, where k is the thermal conductivity, ρ is the density, and C is the specific heat. The boundary

conditions at the interface of the confining layer and the absorbing layer are given by

$$q(t) = \begin{cases} k\left(\frac{\partial T}{\partial x}\right)_{\text{CL}} - k\left(\frac{\partial T}{\partial x}\right)_{\text{AAL}} & \text{for } t \leq \Delta t_i \\ 0 & \text{otherwise} \end{cases}, \quad (2.2)$$

where Δt_i is the duration of the YAG laser pulse, and $q(t)$ is the absorbed laser fluence. The associated one-dimensional elastic wave equation is

$$(\lambda + 2\mu)_i \frac{\partial^2 u_i}{\partial x^2} = \rho_i \frac{\partial^2 u_i}{\partial t^2} + (3\lambda + 2\mu)_i \alpha_i \frac{\partial T_i}{\partial x}, \quad (2.3)$$

where λ and μ are the Lamé moduli, u is the longitudinal displacement, and α is the coefficient of thermal expansion of the medium. The stress σ is calculated from the displacement and temperature gradients:

$$\sigma_i = (\lambda + 2\mu)_i \frac{\partial u_i}{\partial x} + (3\lambda + 2\mu)_i \alpha_i \Delta T_i. \quad (2.4)$$

At distances far from the thermal deposition region, the latter term vanishes.

2.2.2 Analytical Solution for Delta Laser Pulse Deposition

Equations (2.1-2.4) were solved by Gupta et al. [32] using one-dimensional finite element analysis. With closer observation, one may find that these equations can be solved analytically. The result is a closed-form expression for the stress pulse and displacement propagating away from the deposition zone. A system consisting of two juxtaposed isotropic thermo-elastic half-spaces is considered. The more complicated case of a thin film between two half spaces may be treated by a similar procedure. The anisotropic case is a trivial generalization since the governing equations will essentially take the same form with different coefficients [32].

A laser fluence of the form of $q(t) = \phi \delta(t)$ is deposited at the interface (Fig. 2.2) between

two half-spaces of two different materials perfectly bonded at $x = 0$.

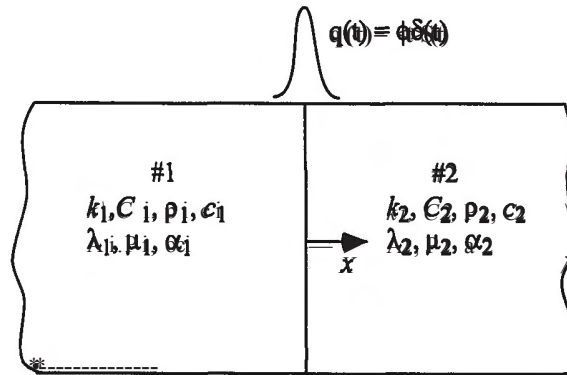


Figure 2.2: Schematic of two half-spaces of different materials.

The heat transfer problem is given by Eq. (2.1) with boundary conditions

$$T_1 = T_2, \quad (2.5)$$

$$q(t) = -k_1 \left(\frac{\partial T_1}{\partial x} \right)_{x=0} = -k_2 \left(\frac{\partial T_2}{\partial x} \right)_{x=0} = \phi \delta(t) \quad (2.6)$$

at $x = 0$. The solution is obtained using Laplace transformation (see Appendix A) as

$$T_1(x, t) = \frac{\phi}{\sqrt{k_1 \rho_1 C_1} + \sqrt{k_2 \rho_2 C_2}} \frac{\exp\left(-\frac{x^2}{4D_1 t}\right)}{\sqrt{\pi t}} H(t), \quad (2.7)$$

$$T_2(x, t) = \frac{\phi}{\sqrt{k_1 \rho_1 C_1} + \sqrt{k_2 \rho_2 C_2}} \frac{\exp\left(-\frac{x^2}{4D_2 t}\right)}{\sqrt{\pi t}} H(t). \quad (2.8)$$

The associated one-dimensional elastic wave problem is given by equation (2.3) with boundary conditions

$$u_1 = u_2, \quad (2.9)$$

$$(\lambda + 2\mu)_1 \frac{\partial u_1}{\partial x} + (3\lambda + 2\mu)_1 \alpha_1 \Delta T_1 = (\lambda + 2\mu)_2 \frac{\partial u_2}{\partial x} + (3\lambda + 2\mu)_2 \alpha_2 \Delta T_2. \quad (2.10)$$

Equations (2.9) and (2.10) are essentially the continuity conditions of displacement and traction at the interface. Since both the temperature gradient and the displacement gradient

are discontinuous across the interface, it is simpler to treat the problem with two continuous variables, namely material velocity and stress:

$$\rho_i \frac{\partial v}{\partial t} = \frac{\partial \sigma}{\partial x}, \quad (2.11)$$

$$\frac{\partial \sigma}{\partial t} = \left(\frac{\lambda + 2\mu}{A + 2\mu} \right)_i \frac{\partial v}{\partial x} - \alpha_i \left(\frac{3\lambda + 2\mu}{3A + 2\mu} \right)_i \frac{\partial T}{\partial t}, \quad (2.12)$$

where $i = 1$ when $x < 0$, $i = 2$ when $x > 0$, and boundary conditions at $x = 0$ are

$$v|_{x=0^-} = w|_{x=0^+}, \quad (2.13)$$

$$\sigma|_{x=0^-} = -\sigma|_{x=0^+}. \quad (2.14)$$

As the symbolic derivation is long and tedious (see Appendix A), only the final result is given here:

$$\begin{aligned} \alpha(x, t) = & K_1 \cdot H\left(t - \frac{x}{c_2}\right) \int_0^{t - \frac{x}{c_2}} \left\{ 1 - \frac{[x - c_2(t - t_s)]^2}{2D_1 t_s} \right\} \cdot \frac{e^{-\frac{[x - c_2(t - t_s)]^2}{4D_1 t_s}}}{t_s^{3/2}} dt_s \\ & + K_2 \cdot \int_{\max(0, t - \frac{x}{c_2})}^0 \left\{ 1 - \frac{[x - c_2(t - t_s)]^2}{2D_2 t_s} \right\} \cdot \frac{e^{-\frac{[x - c_2(t - t_s)]^2}{4D_2 t_s}}}{t_s^{3/2}} dt_s \\ & + K_3 \cdot H\left(t - \frac{x}{c_2}\right) \int_0^{t - \frac{x}{c_2}} \left\{ 1 - \frac{[x - c_2(t - t_s)]^2}{2D_2 t_s} \right\} \cdot \frac{e^{-\frac{[x - c_2(t - t_s)]^2}{4D_2 t_s}}}{t_s^{3/2}} dt_s, \end{aligned} \quad (2.15)$$

$$\begin{aligned} u(x, t) = & \frac{2K_1}{\rho_2 c_2} \cdot H\left(t - \frac{x}{c_2}\right) \int_0^{t - \frac{x}{c_2}} \frac{e^{-\frac{[x - c_2(t - t_s)]^2}{4D_1 t_s}}}{t_s^{1/2}} dt_s \\ & + \frac{2K_2}{\rho_2 c_2} \cdot \int_{\max(0, t - \frac{x}{c_2})}^0 \frac{e^{-\frac{[x - c_2(t - t_s)]^2}{4D_2 t_s}}}{t_s^{1/2}} dt_s \\ & + \frac{2K_3}{\rho_2 c_2} \cdot H\left(t - \frac{x}{c_2}\right) \int_0^{t - \frac{x}{c_2}} \frac{e^{-\frac{[x - c_2(t - t_s)]^2}{4D_2 t_s}}}{t_s^{1/2}} dt_s, \end{aligned} \quad (2.16)$$

where

$$\begin{aligned}
 K_1 &= \frac{\phi_{01}(3\lambda + 2\mu)_1}{2\sqrt{\pi}(\sqrt{k_1\rho_1 C_1} + \sqrt{k_2\rho_2 C_2})} \cdot \frac{\rho_2 c_2}{\rho_1 c_1 + \rho_2 c_2}, \\
 K_2 &= \frac{\phi_{02}(3\lambda + 2\mu)_2}{4\sqrt{\pi}(\sqrt{k_1\rho_1 C_1} + \sqrt{k_2\rho_2 C_2})}, \\
 K_3 &= \frac{\phi_{02}(3\lambda + 2\mu)_2}{4\sqrt{\pi}(\sqrt{k_1\rho_1 C_1} + \sqrt{k_2\rho_2 C_2})} \cdot \frac{\rho_1 c_1 - \rho_2 c_2}{\rho_1 c_1 + \rho_2 c_2}.
 \end{aligned} \tag{2.17}$$

In Eqs. (2.15) and (2.16), the first term is a wave that is launched by the temperature gradient in the region $x < 0$ that propagates to the detector at $x \gg 0$. The second term is a wave launched from the region $x > 0$ that propagates directly to the detector at $x \gg 0$. And the third term is a wave that is launched from the region $x > 0$ but propagates back towards the interface, then reflects and goes to the detector at $x \gg 0$.

By the change of variables, $x = c_2 t + z$, $t_2 = \xi$, and $z' = z c_2 / D_2$, $\xi' = \xi c_2^2 / D_2$, where ξ' is a nondimensional time at which a wave was launched to later arrive at the detector at position x and time t , the stress can be expressed using the nondimensionalized integrals as

$$\begin{aligned}
 \sigma(z') &= K_1 \cdot \frac{c_2}{\sqrt{D_2}} \cdot H(-z') \int_0^{-z'} \left\{ 1 - \frac{(z' + \xi')^2}{2\xi'} \cdot \frac{D_2 c_1^2}{D_1 c_2^2} \right\} \cdot \frac{e^{-\frac{(z' + \xi')^2}{4\xi'} \cdot \frac{D_2 c_1^2}{D_1 c_2^2}}}{\xi'^{3/2}} d\xi' \\
 &+ K_2 \cdot \frac{c_2}{\sqrt{D_2}} \cdot \int_{\max(0, -z')}^t \left\{ 1 - \frac{(z' + \xi')^2}{2\xi'} \right\} \cdot \frac{e^{-\frac{(z' + \xi')^2}{4\xi'}}}{\xi'^{3/2}} d\xi' \\
 &+ K_3 \cdot \frac{c_2}{\sqrt{D_2}} \cdot H(t - z') \cdot \int_0^{-z'} \left\{ 1 - \frac{(z' + \xi')^2}{2\xi'} \right\} \cdot \frac{e^{-\frac{(z' + \xi')^2}{4\xi'}}}{\xi'^{3/2}} d\xi',
 \end{aligned} \tag{2.18}$$

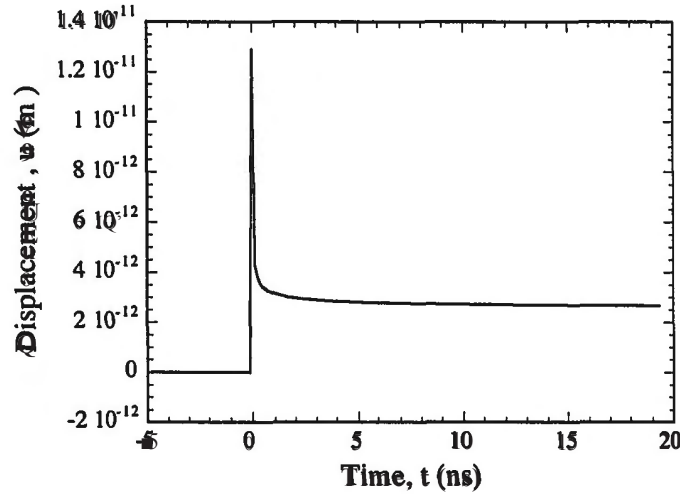


Figure 2.3: Displacement in the aluminum portion ($x > 0$) of a glass/aluminum sample due to the delta laser pulse deposition for a laser fluence $\phi = 1 \text{ J/m}^2$.

and the displacement is

$$\begin{aligned}
 u(z') = & 2K_1 \cdot \frac{\sqrt{D_2}}{\rho_2 c_2^2} \cdot H(-z') \int_0^{-z'} \frac{e^{-\frac{(z'+\xi')^2}{4\xi'}} \cdot \frac{D_2 c_2^2}{D_1 c_1^2}}{\xi'} d\xi' \\
 & + 2K_2 \cdot \frac{\sqrt{D_2}}{\rho_2 c_2^2} \cdot \int_{\max(0, -z')}^t \frac{e^{-\frac{(z'+\xi')^2}{4\xi'}}}{\xi'^{3/2}} d\xi' \\
 & + 2K_3 \cdot \frac{\sqrt{D_2}}{\rho_2 c_2^2} \cdot H(-z') \cdot \int_0^{-z'} \frac{e^{-\frac{(z'+\xi')^2}{4\xi'}}}{\xi'^{3/2}} d\xi',
 \end{aligned} \tag{2.20}$$

with K_1 , K_2 and K_3 as described in equation (2.17). These nondimensionalized integrals are easier to evaluate numerically. Figure 2.3 shows the displacement in the aluminum portion ($x > 0$) of a glass (material #1)/aluminum (material #2) sample following a delta pulse deposition at the two-material interface with $\phi = 1 \text{ J/m}^2$. The material properties for glass and aluminum together with those of silicon and fused silica are summarized in Table 2.1.

The displacement generated in the aluminum resembles a Heaviside function. After the laser pulse is completely deposited at the interface, the displacement remains at a constant value.

Table 2.1: Material properties.

Material	Glass [31]	Aluminum [31]	Fused silica*	Silicon [31]
ρ (g/cm ³)	2.5	2.71	2.2	2.3
C (J/kg · K)	750	896	670	729
k (J/ms · K)	1.4	204	1.4	138
λ (GPa)	23.3	58.5	15.9	62.0
μ (GPa)	29.6	26.0	30.8	41.0
c (m/s)	5378	6385	5940**	7910
α (K ⁻¹)	5.0×10^{-6}	8.4×10^{-6}	5.5×10^{-6}	7.6×10^{-6}

* from manufacturer

** measured in the Laboratory for Incoherent Ultrasound at UIUC

2.2.3 Asymptotic Solution for Delta Laser Pulse Deposition

The asymptotic solution for slow illumination is also derived. It is justified by recognizing that $\Delta t_i \gg c^2 j D$.

The Fourier transformation w.r.t. time of heat-transfer equation (2.1) yields

$$\hat{T}_{xx} = \frac{i\omega}{D} \hat{T}. \quad (2.21)$$

The solution of Eq. (2.21) can be written in the form

$$\hat{T} = \begin{cases} A_1 e^{\sqrt{\frac{i\omega}{D}} x} & \text{for } x \leq 0 \\ A_2 e^{-\sqrt{\frac{i\omega}{D}} x} & \text{for } x > 0 \end{cases} \quad (2.22)$$

with boundary conditions (2.5) and (2.6) to yield

$$\hat{T}|_{x=0^-} = \hat{T}|_{x=0^+} \quad (2.23)$$

$$(k\hat{T}_x)|_{x=0^-} = (k\hat{T}_x)|_{x=0^+} = 1 \quad (2.24)$$

Combination of Eqs. (2.22)-(2.24) provides the solution of temperature in the frequency

domain as

$$\hat{T}(x, \omega) = \frac{\phi}{\sqrt{k_1 \rho_1 C_1} + \sqrt{k_2 \rho_2 C_2}} \begin{cases} \frac{1}{\sqrt{w}} e^{\sqrt{\frac{i\omega}{D_1}} x} & \text{for } x \leq 0 \\ \frac{1}{\sqrt{w}} e^{-\sqrt{\frac{i\omega}{D_2}} x} & \text{for } x \geq 0 \end{cases}. \quad (2.25)$$

The solution for the displacement and stress is obtained from the wave equation (2.11) and (2.12) and using Fourier transformation w.r.t. time

$$\rho i \omega \hat{V} = \hat{\sigma}_{,x}, \quad (2.26)$$

$$i \omega \sigma = (\lambda + 2\mu) \hat{V}_{,x} = (3\lambda + 2\mu) \alpha(i\omega) \hat{T}. \quad (2.27)$$

Solving for $\hat{V}$ from (2.26) in terms of $\hat{\sigma}$ and substituting into (2.27), one obtains

$$\hat{\sigma}_{,xx} + \frac{w^2}{c^2} \hat{\sigma} + \frac{(3\lambda + 2\mu) \alpha w^2}{c^2} \hat{T} = 0. \quad (2.28)$$

The coefficients in (2.28) take different values of material 1 and 2 respectively for $x < 0$ and $x \geq 0$. The solution to Eq. (2.28) can be expressed in the form

$$\hat{\sigma} = \hat{\sigma}_g + \hat{\sigma}_p \quad (2.29)$$

where $\hat{\sigma}_g$ and $\hat{\sigma}_p$ are the general and particular solutions of Eq. (2.28), respectively. The particular solution is assumed to have the form

$$\hat{\sigma}_p = \begin{cases} f_1(\omega) e^{\sqrt{\frac{i\omega}{D_1}} x} & \text{for } x \leq 0 \\ f_2(\omega) e^{-\sqrt{\frac{i\omega}{D_2}} x} & \text{for } x \geq 0 \end{cases}. \quad (2.30)$$

Substitution of Eqs. (2.30) and (2.25) into (2.28) gives

$$f_1(\omega) = -\frac{(3\lambda + 2\mu)_1 \alpha_1 \phi}{\sqrt{k_1 \rho_1 C_1} + \sqrt{k_2 \rho_2 C_2}} \cdot \frac{\sqrt{i\omega}}{i\omega - \frac{c_1^2}{D_1}}, \quad (2.31)$$

$$f_2(\omega) = -\frac{(3\lambda + 2\mu)_2 \alpha_2 \phi}{\sqrt{k_1 \rho_1 C_1} + \sqrt{k_2 \rho_2 C_2}} \cdot \frac{\sqrt{i\omega}}{i\omega - \frac{c_2^2}{D_2}}. \quad (2.32)$$

The general solution of Eq. (2.28) takes the form

$$\hat{\sigma}_g = \begin{cases} A(b_j) e^{\frac{i\omega}{c_1} x} & \text{for } x \leq 0 \\ B(\omega) e^{-\frac{i\omega}{c_2} x} & \text{for } x > 0 \end{cases}. \quad (2.33)$$

Thus, the stress in the frequency domain is

$$\hat{\sigma}(x, \omega) = \begin{cases} A(b_j) e^{\frac{i\omega}{c_1} x} + f_1(\omega) e^{\sqrt{\frac{i\omega}{D_1}} x} & \text{for } x \leq 0 \\ B(b_j) e^{-\frac{i\omega}{c_2} x} + f_2(\omega) e^{\sqrt{\frac{i\omega}{D_2}} x} & \text{for } x > 0 \end{cases}. \quad (2.34)$$

Continuity conditions of stress and velocity at the interface (2.13) and (2.14) leads to

$$\hat{\sigma}|_{x=0-} = \hat{\sigma}|_{x=0+}, \quad (2.35)$$

$$\frac{\hat{\sigma}_{,x}}{\rho}|_{x=0-} = \frac{\hat{\sigma}_{,x}}{\rho}|_{x=0+}. \quad (2.36)$$

Substitution of (2.34) into (2.35) and (2.36) yields

$$A(\omega) = \frac{\rho_1 c_1}{\rho_1 c_1 + \rho_2 c_2} \cdot \frac{\phi}{\sqrt{k_1 \rho_1 C_1} + \sqrt{k_1 \rho_1 C_1}} \cdot \left\{ \left[\frac{\rho_2 c_2}{\rho_1 c_1} \cdot \frac{(3\lambda + 2\mu)_1 \alpha_1 \sqrt{D_1}}{i\omega \frac{D_1}{c_1^2} - c_1} + \frac{(3\lambda + 2\mu)_2 \alpha_2 \sqrt{D_2}}{i\omega \frac{D_2}{c_2^2} - c_2} \right] \right. \\ \left. + \left[(3\lambda + 2\mu)_1 \alpha_1 D_1 \cdot \frac{\sqrt{i\omega}}{i\omega D_1 - c_1^2} - (3\lambda + 2\mu)_2 \alpha_2 D_2 \cdot \frac{\sqrt{i\omega}}{i\omega D_2 - c_2^2} \right] \right\},$$

and

$$B(\omega) = \frac{p_2 c_2}{p_1 c_1 + p_2 c_2} \cdot \frac{\phi}{\sqrt{k_1 p_1 C_1} + \sqrt{k_2 p_2 C_2}} \cdot \left\{ \left[\frac{(3\lambda + 2\mu)_1 \alpha_1 \sqrt{D_1}}{i\omega \frac{D_1}{c_1} - c_1} + \frac{p_1 c_1}{p_2 c_2} \cdot \frac{(3\lambda + 2\mu)_2 \alpha_2 \sqrt{D_2}}{i\omega \frac{D_2}{c_2} - c_2} \right] \right. \\ \left. + \left[-(3\lambda + 2\mu)_1 \alpha_1 D_1 \cdot \frac{\sqrt{i\omega}}{i\omega D_1 - c_1^2} + (3\lambda + 2\mu)_2 \alpha_2 D_2 \cdot \frac{\sqrt{i\omega}}{i\omega D_2 - c_2^2} \right] \right\}.$$

The solution in the right half space ($x \geq 0$) is of particular interest because the signal will be picked up at $x > 0$. From the stress-displacement relation

$$\sigma = -p_2 c_2 \frac{\partial u}{\partial x}, \quad (2.37)$$

one obtains the Fourier-transformed displacement as

$$\hat{u} = -\frac{1}{p_2 c_2} \cdot \frac{\hat{\sigma}}{i\omega}. \quad (2.38)$$

Substitution of (2.34) into (2.38) and taking the limit $\omega \ll c^2/hD$ yield

$$\hat{u}(x, i\omega) = A_1 \frac{e^{-i\frac{\omega}{c_2}x}}{i\omega} + A_2 \frac{e^{-i\frac{\omega}{c_2}x}}{\sqrt{i\omega}} + A_3 \frac{e^{-\sqrt{i\omega D_2}x}}{\sqrt{i\omega}}, \quad (2.39)$$

where

$$A_1 = \frac{\phi}{(p_1 c_1 + p_2 c_2)(\sqrt{k_1 p_1 C_1} + \sqrt{k_2 p_2 C_2})} \left[\frac{(3\lambda + 2\mu)_1 \alpha_1 \sqrt{D_1}}{c_1} + \frac{(3\lambda + 2\mu)_2 \alpha_2 \sqrt{D_2}}{c_2} \cdot \frac{p_1 c_1}{p_2 c_2} \right] \\ A_2 = \frac{\phi}{(p_1 c_1 + p_2 c_2)(\sqrt{k_1 p_1 C_1} + \sqrt{k_2 p_2 C_2})} \left[-\frac{(3\lambda + 2\mu)_1 \alpha_1 D_1}{c_1^2} + \frac{(3\lambda + 2\mu)_2 \alpha_2 D_2}{c_2^2} \right] \quad (2.40) \\ A_3 = \frac{(3\lambda + 2\mu)_2 \alpha_2 D_2 \phi}{(p_2 c_2^3)(\sqrt{k_1 p_1 C_1} + \sqrt{k_2 p_2 C_2})}$$

In Eq. (2.39), the third term is exponentially small at large x (much larger than millimeters) and is unimportant. For small ω ($\omega \ll c^2/hD$), the second term is also much smaller than the first term. Thus only the first term counts for the late-time response for $x \gg 0$. By

taking the inverse Fourier transformation, the asymptotic solution of displacement for large x is obtained:

$$u(t) = A_i H\left(t - \frac{x}{c_2}\right), \quad (2.41)$$

which is indeed a Heaviside function. According to the material properties of glass and aluminum provided in Table 2.1, for $\phi = 1 \text{ J/m}^2$, $A_i = 2.67 \times 10^{-12} \text{ m}$ is calculated. This value is exactly the same as the constant value observed in Fig. 2.3. Thus the analytical solution in Section 2.2.2 is verified. Substitution of (2.41) back into (2.37) yields the asymptotic solution for stress,

$$\sigma(t) = -\rho_2 c_2 A_i \delta\left(t - \frac{x}{c_2}\right), \quad (2.42)$$

which is a delta function.

2.2.4 Practical Solution for Gaussian Pulse Deposition

In the above derivations, it is assumed that a laser energy of the form $q(t) = \phi \delta(t)$ is deposited at the interface of the two semi-infinite materials perfectly bonded at $x = 0$. However, the actual laser pulse is not a perfect delta function. As shown in Fig. 2.4, the actual laser pulse of energy 110 mJ is deposited within a period of 6–8 ns and is fitted to a Gaussian distribution

$$q(t) = q_{\max} \exp\left(-\frac{2t^2}{T^2}\right) \quad (2.43)$$

$$q(t) = q_{\max} \exp\left(-\frac{2t^2}{T^2}\right) \quad (2.44)$$

where $q_{\max} = 18.9 \text{ mJ/ns}$ and $T = 4.1 \text{ ns}$.

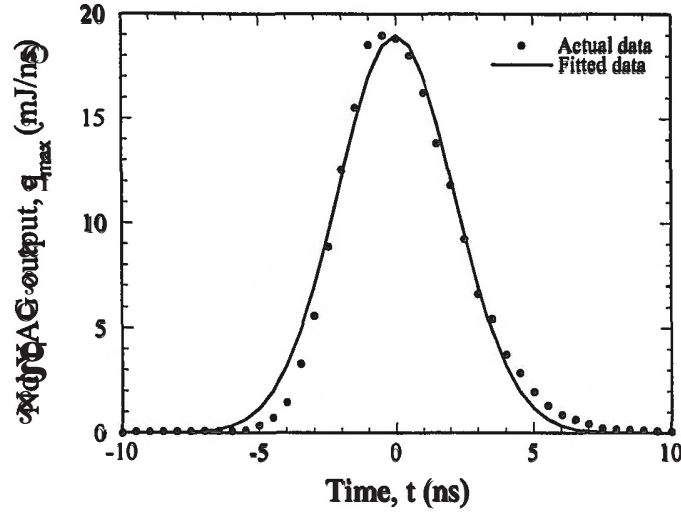


Figure 2.4: Nd:YAG laser pulse provided by manufacturer.

The actual late-time response in the detector is the convolution of the real energy distribution and the previous asymptotic solution, i.e.,

$$u^{\text{actual}} \equiv u^{\text{asymptotic}}(t) * \hat{q}(t), \quad (2.45)$$

$$\sigma^{\text{actual}} = -(\rho c)_{\text{sub}} \frac{\partial u^{\text{actual}}}{\partial t} = -(\rho c)_{\text{sub}} A_1 q(t). \quad (2.46)$$

Thus, one concludes that the stress pulse propagating away from the interface is a copy of and is proportional to the impinging laser fluence $q(t)$.

For a laser fluence of 110 mJ/mm^2 (full YAG laser energy deposited on a 1 mm^2 area), the displacement and stress in the substrate are predicted for the case of a glass (material #1)/aluminum (material #2) sample and plotted in Figs. 2.5 and 2.6, respectively. Under this laser fluence, the maximum displacement is around 195 nm and the maximum compressive stress is about 0.85 GPa . The amplitude of the displacement and stress strongly depends on the material properties of the two media. The shape of the stress pulse is determined by that of the deposited laser pulse.

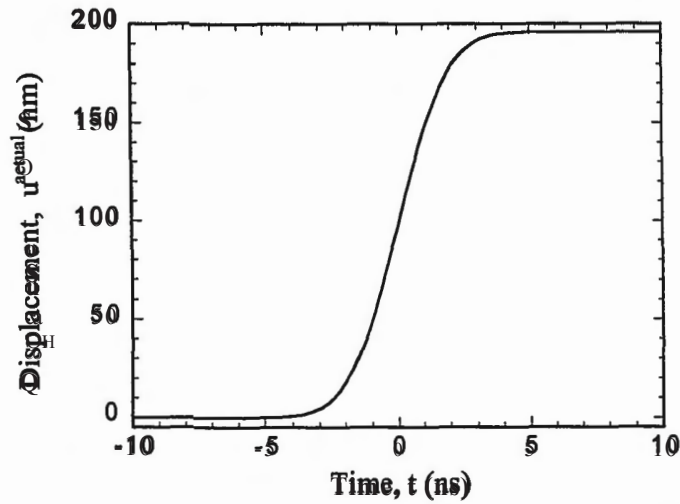


Figure 2.5: Predicted displacement in the aluminum portion ($x > 0$) of the glass/aluminum sample far from the laser deposition region for laser fluence of 110 mJ/mm^2 .

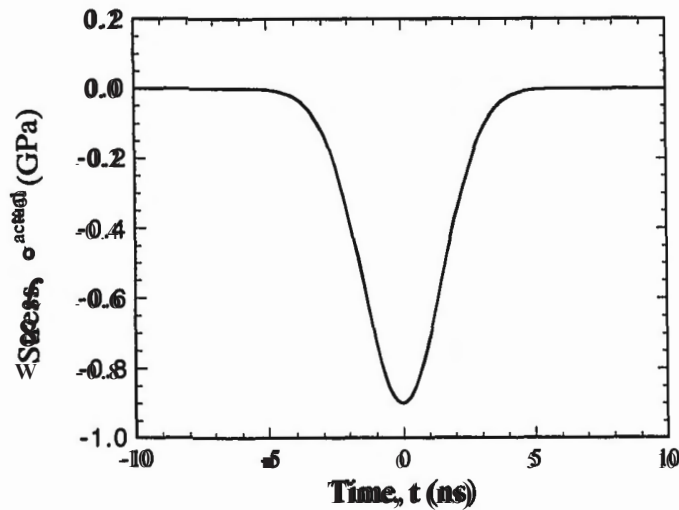


Figure 2.6: Predicted stress in the aluminum portion ($x > 0$) of the glass/aluminum sample far from the laser deposition region for laser fluence of 110 mJ/mm^2 .

Thin-Film Interface Stress

Previous solutions are based on the assumption of two semi-infinite media perfectly bonded at $x = 0$, with a laser pulse deposition at the interface. For the current analysis, the right half space serves as the substrate in the laser spallation experiment with a thin film of thickness h deposited at the far right end of the substrate. The stress at the film interface can be much less than the stress acting in the substrate because the nearby free surface releases some of the stress. The relation is calculated using elastic wave transmission and reflection coefficients (Appendix B). Here a simpler calculation is adopted, which is valid only for small film thickness h , that the stress is ρh times the acceleration of the free surface. It is also recognized that the displacement at the free surface is twice that of the wave in the substrate. Thus the stress at the film interface can be expressed in the form

$$\sigma_{\text{int}} = -2(\rho h)_{\text{film}} \frac{\partial^2 u_{\text{actual}}}{\partial t^2}. \quad (2.47)$$

Equation (2.47) is equivalent to Newton's second law—the interface stress is the mass density of the film times the outward acceleration of the center of mass of the film. This is the case regardless of the character of the propagation. That the measured surface displacement is a good approximation to the center of mass displacement follows from the known wave speeds in the film, the thickness of the film, and the measured time scales in the wave.

For a 1 μm aluminum film deposited at the right end of the glass (material #1)/aluminum (material #2) sample, with a laser fluence of 110 mJ/mm² (full YAG laser energy deposited on a 1 mm² area), the interface stress is calculated and plotted in Fig. 2.7. The maximum stress at the interface is about 120 MPa.

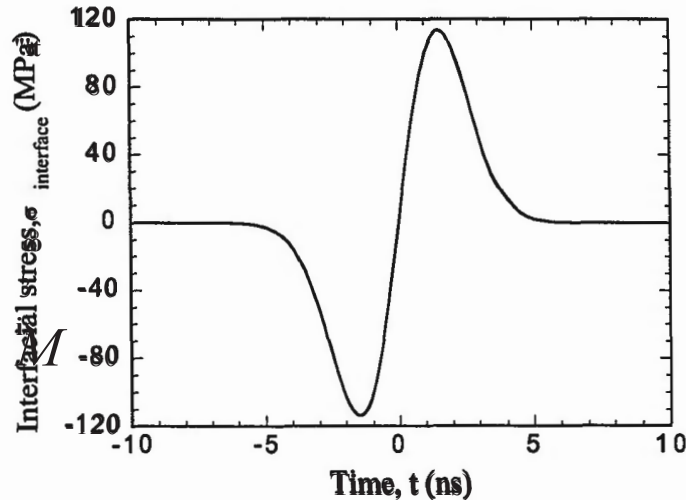


Figure 2.7: Predicted stress at the interface of a 1 μm thick aluminum film deposited at the right end ($x \gg 0$) of glass/aluminum sample for laser fluence of 110 mJ/mm^2 .

2.3 Stress-Wave Attenuation

The calculations in Section 2.2 are based on the one-dimensional plane wave assumption. In the actual experiment, however, geometric attenuation of the stress wave occurs due to the long travel distance and the finite laser beam size. Intrinsic attenuation is known to be weak in such materials as silicon and fused silica [45].

After the initial reports by Gupta et al. [32], the issue of geometric attenuation, and the related transition of the initial planar wave front to a spherical wave, were extensively discussed in the literature [46–48]. The issue appears to have been settled by numerical calculations in the report by Lev and Argon [47] in which it was concluded that the front remains essentially planar for a distance comparable to two or three times the radius of the laser spot. This conclusion was based on numerical observations of diminished peak stress amplitude at such distances. This conservative estimate may be compared with the standard acoustician's rule, that the near field of a planar acoustic source of diameter D extends a distance of $jD^2/4\lambda_e$ [49]. Even if one takes the ultrasonic elastic wavelength λ_e to be as large as 60 μm —corresponding to the length of the longitudinal disturbance generated by a 1.0 ns

laser pulse—a 1 mm diameter spot will generate a wave that remains approximately plane over a distance of 4 mm. For this reason, only modest losses due to geometric attenuation for substrates less than 4 mm is anticipated. In the present section, geometric attenuation is analyzed by considering a circular source region of diameter D as shown in Fig. 2.8.

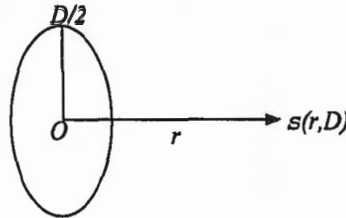


Figure 2.8: Stress generated by a finite-sized pancake YAG laser source.

Results in Section 2.2.4 indicate that the stress pulse in the source region generated by the current YAG laser has the same profile as the laser pulse and can be expressed using the Gaussian distribution

$$\sigma(t) = \sigma_{\max} \exp\left(-\frac{2t^2}{T^2}\right) \quad (2.48)$$

where $\sigma_{\max}$ is the maximum compressive stress generated at the source region and $T = 4.1$ ns is the half bandwidth of the Gaussian laser pulse. Thus, by Huygen's principle, the stress in an isotropic linear medium on axis at distance r from the source can be approximated by an integral over the entire source region as

$$\sigma(r,t;D) \equiv \frac{A}{B^2} \int_0^{2\pi} \int_0^{D/2} \frac{1}{\sqrt{r^2 + x^2}} \exp\left[-\frac{2(t - \frac{1}{c}\sqrt{r^2 + x^2})^2}{T^2}\right] x dx d\theta. \quad (2.49)$$

The stress at the test film interface is proportional to the time derivative of (2.49)

$$\sigma(r,t;D)_{if} = \frac{B}{D^2} \int_0^{2\pi} \int_0^{D/2} \frac{t - \frac{1}{c}\sqrt{r^2 + x^2}}{\sqrt{r^2 + x^2}} \exp\left[-\frac{2(t - \frac{1}{c}\sqrt{r^2 + x^2})^2}{T^2}\right] x dx d\theta, \quad (2.50)$$

where A and B are constants. These integrals can be evaluated analytically,

$$\alpha(r, t; D) \propto \frac{1}{D^2} \left\{ \operatorname{erf} \left[\frac{\sqrt{2} S \left(\frac{1}{4} \sqrt{r^2 + \frac{D^2}{4}} - \frac{t}{2} \right)}{T} \right] - \operatorname{erf} \left[\frac{\sqrt{2} \left(\frac{r}{2} - t \right)}{T} \right] \right\}, \quad (2.51)$$

and

$$\sigma(r, t; D)_{\text{interface}} \propto \frac{1}{D^2} \left\{ \exp \left[-\frac{2 \left(\frac{1}{c} \sqrt{r^2 + \frac{D^2}{4}} - t \right)^2}{T^2} \right] - \exp \left[-\frac{2 \left(\frac{r}{c} - t \right)^2}{T^2} \right] \right\}. \quad (2.52)$$

The relative maximum stress in the substrate and at the interface are evaluated for different source sizes D at different distances r and plotted in Figs. 2.9 and 2.10. The plots show that at fixed pulse laser energy, both the maximum substrate and interface stress decrease with increasing source diameter. For the same source diameter, greater attenuation occurs at longer distances. For a 1 mm diameter laser source, the maximum substrate stress begins to attenuate (quantified by a 10% drop) after about 2 mm and the maximum interface stress begins to attenuate after 3.5 mm. The maximum substrate stress attenuates faster than the maximum interface stress. Based on these analyses, the previous one-dimensional thermo-elastic analysis is seen to be valid for substrate thicknesses up to 3.5 mm.

2.4 Shock Development in Fused Silica

Thermo-elastic calculations in Section 2.2 revealed that the compressive stress pulse is a copy of, and proportional to, the deposited laser pulse at the interface (Eq. 2.46). The amplitude of that stress is determined by the material properties of the two adjacent media. In many cases, the pulses that are generated have compressive stress amplitudes of the order of giga-pascals, corresponding to strains ϵ_{max} of the order of 1%. Such a pulse can be expected to evolve a weak shock in times proportional to the time scales of the laser

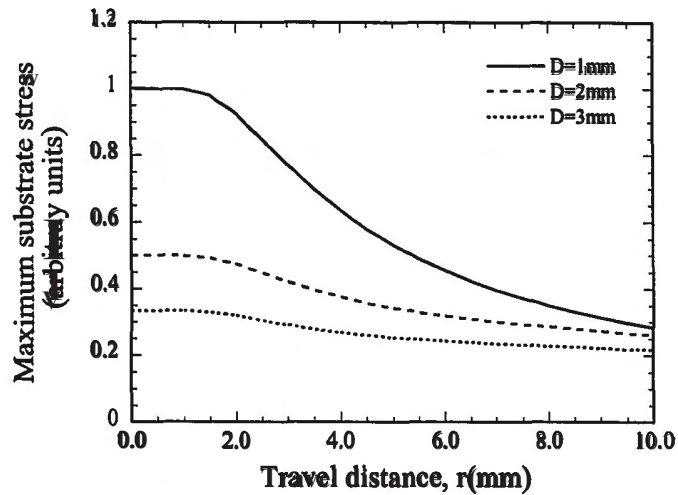


Figure 2.9: Attenuation of maximum substrate stress w.r.t. distance r and source diameter D . Data were normalized by the maximum stress for $D = 1$ mm at $r = 0$.

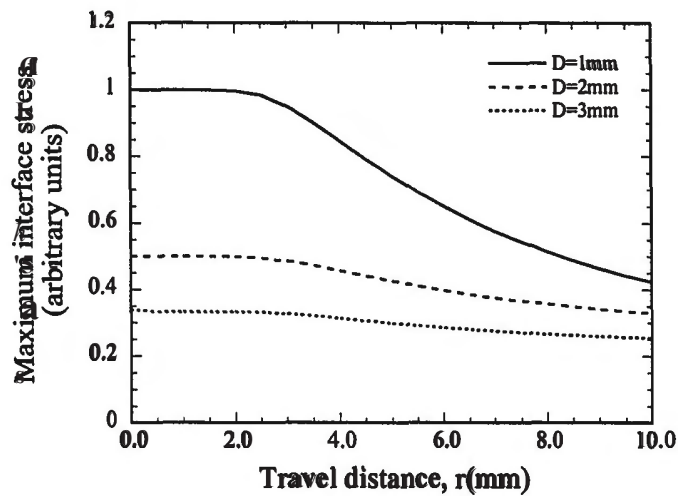


Figure 2.10: Attenuation of maximum interface stress w.r.t. distance r and source diameter D . Data were normalized by the maximum stress for $D = 1$ mm at $r = 0$.

pulse and inversely proportional to ϵ_{max} . The generation of a shock and in particular the precise time and distance also depend on the nonlinear parameters of the medium and on the competing effects of absorption and attenuation. Because the test film interface is loaded by the difference between the stress at one time and the stress at a time $2h/c$ earlier (due to reflection from the free surface), the development of a shock with an extremely rapid rise (or fall) time will significantly affect that load. The 4.1 ns rise time pulse generated by the laser is inefficient for the purpose of spalling a one micron film. For such a film, the interface load is related to the pulse's stress change over a time difference of 0.33 ns (0.66 ns for shear waves). The stress difference on the interface is therefore substantially less than the peak stress developed in a 4.1 ns pulse. If a shock develops with a sub-nanosecond rise (or fall) time, however, the peak stress on the interface can be equal to the peak stress in the pulse. Therefore, materials with nonlinear properties that enhance shock development are desirable for studying the interface properties using the laser spallation technique.

Because fused silica has the unusual property of softening under modest compression, the development of weak shocks in fused silica has received considerable attention in the literature. The basic characteristics of compressive wave shapes in fused silica, including a ramp-wave front up to ~ 4 GPa (40 kbar) was first reported by Wackerle [50]. He concluded that fused silica behaves as a nonlinear elastic solid up to the phase change at 9.8 GPa. Later, Barker and Hollenbach [51] demonstrated the existence of rarefaction shocks in fused silica (as opposed to the more common compression shocks found in other materials). They refined Wackerle's compressive wave data in the low-stress region and confirmed Wackerle's assertion of elasticity by determining that the release stress-strain path followed in a rarefaction wave coincided with the compressive stress-strain path, which determines the compressive wave shape. Barker and Hollenbach [51] also fitted the compressive wave data into a fourth-order stress-strain relation

$$\sigma = \alpha \epsilon + \beta \epsilon^2 + \gamma \epsilon^3 + \zeta \epsilon^4, \quad (2.53)$$

where σ is the stress in GPa, $\alpha = 77.6$ GPa, $\beta = -415.9$ GPa, $\gamma = 3034.0$ GPa and $\zeta = -6926.0$ GPa. Equation (2.53) is valid for stresses up to the phase transition at about 9.8 GPa. The stress-strain relation in Eq. (2.53) is plotted in Fig. 2.11a for stresses up to 9.8 GPa. By taking the derivative of Eq. (2.53) with respect to ϵ , one obtains the relation between the tangent modulus and stress (or strain), which is plotted in Fig. 2.11b. Under compressive stress below 4 GPa (corresponding to a strain of 6.5%), the tangent modulus decreases or "softens" with increasing stress. For stresses above 4 GPa, the tangent modulus increases ("hardens") with increasing stress. In the low stress region, fused silica has the potential for developing a shock.

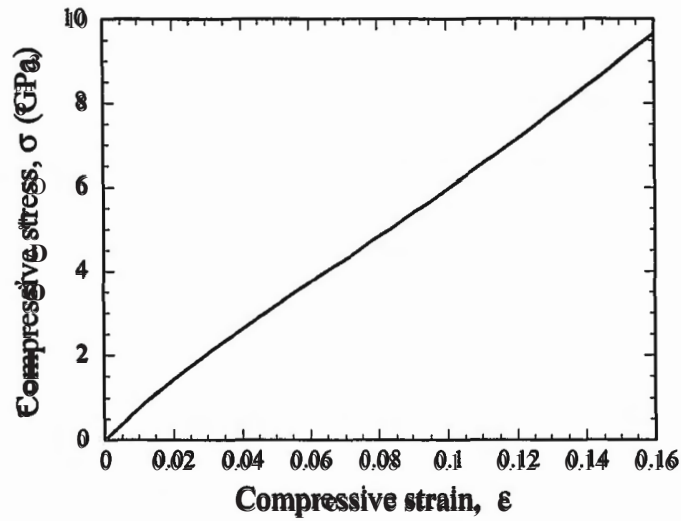
Standard wave propagation theory in nonlinear elastic solids [49] is used to analyze the shock development problem in fused silica. First, the wave motion is considered in the reference frame (X, t) . The material velocity and stress are expressed as $v(X, t)$, and $\sigma(X, t)$. The governing equations are

$$\rho_0 \frac{\partial v}{\partial t} = \frac{\partial \sigma}{\partial X} \quad (\text{balance of momentum}), \quad (2.54)$$

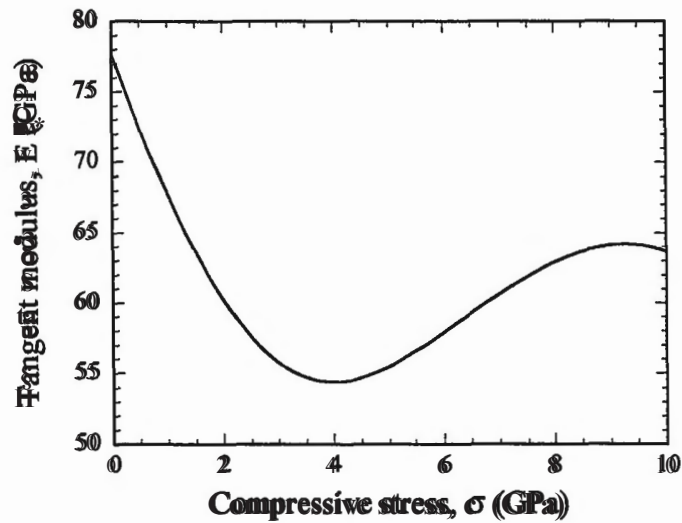
$$\frac{\partial \epsilon}{\partial t} = \frac{\partial v}{\partial X} \quad (\text{conservation of material}). \quad (2.55)$$

Assuming a constitutive relation of the form

$$\sigma = \sigma(\epsilon), \quad (2.56)$$



(a)



(b)

Figure 2.11: Nonlinear elastic properties of fused silica: (a) stress-strain relation; (b) tangent modulus as a function of stress.

and that v is a single valued function of σ , one may write

$$\frac{\partial v}{\partial t} = \frac{\partial v}{\partial \sigma} \frac{\partial \sigma}{\partial t}; \quad \frac{\partial v}{\partial X} = \frac{\partial v}{\partial \sigma} \frac{\partial \sigma}{\partial X} \quad (2.57)$$

and Eqs. (2.54) and (2.55) become

$$\rho_0 \frac{\partial v}{\partial \sigma} \frac{\partial \sigma}{\partial t} - \frac{\partial \sigma}{\partial X} = 0, \quad (2.58)$$

$$\frac{\partial \epsilon}{\partial \sigma} \frac{\partial \sigma}{\partial t} - \frac{\partial v}{\partial \sigma} \frac{\partial \sigma}{\partial X} = 0. \quad (2.59)$$

Solving equations (2.58) and (2.59) leads to

$$\rho_0 \left(\frac{\partial v}{\partial \sigma} \right)^2 = \frac{\partial \epsilon}{\partial \sigma}. \quad (2.60)$$

For a given function of $\sigma(\epsilon)$, $\partial \epsilon / \partial \sigma$ is fixed function of σ ; thus Eq. (2.60) has two solutions for $v = v(\sigma)$. Once the root is chosen, a speed of propagation of characteristics can be identified (from 2.58)

$$c(\sigma) = \frac{1}{\rho_0 \frac{\partial v}{\partial \sigma}} = \frac{1}{\sqrt{\rho_0 \frac{\partial \epsilon}{\partial \sigma}}} = \sqrt{\frac{1}{\rho_0} \frac{\partial \sigma}{\partial \epsilon}}. \quad (2.61)$$

The time it takes the stress wave to propagate through a distance L is given by

$$t_L = \frac{L}{c(\sigma)} = \frac{L \sqrt{\rho_0}}{\sqrt{\frac{\partial \sigma}{\partial \epsilon}}}. \quad (2.62)$$

For a fused silica substrate with the stress-strain relation provided by Barker and Hollenbach

[51] (Eqs. 2.61, 2.62), the speed of the propagation of characteristics is

$$\begin{aligned}
 c(\sigma) &= \sqrt{\frac{1}{\rho_0}(\alpha + 2\beta\epsilon)} \quad (2\text{nd-order approximation}) \\
 &= \sqrt{\frac{\alpha}{\rho_0}\left(1 + \frac{2\beta\sigma}{\alpha^2}\right)} \quad (1\text{st-order approximation}) \\
 &= \sqrt{\frac{\alpha}{\rho_0}}\left(1 + \frac{\beta\sigma}{\alpha^2}\right) \quad \left((1 + a)^{1/2} \approx 1 + a/2\right) \\
 &= c_0\left(1 + \frac{\beta\sigma}{\alpha^2}\right) \quad (2.63)
 \end{aligned}$$

where $c_0 = \sqrt{\alpha/\rho_0}$ coincides with the low-amplitude dilatational sound velocity in fused silica, 5.9391 mm/ μ sec. The time/travel-distance ratio is given by

$$\frac{t_L}{L} = \frac{1}{c_0}\left(1 - \frac{\beta\sigma}{\alpha^2}\right). \quad (2.64)$$

Equations (2.63) and (2.64) reveal that in the low stress region, the speed of the stress wave as well as the time/travel-distance ratio are linear functions of the compressive stress. Higher amplitude stress has a lower travel speed, and thus higher time/travel-distance value, i.e., the higher amplitude compressive stress travels slower. In the actual laser spallation experiments, the stress pulse generated at the absorbing layer interface is a compressive Gaussian stress pulse. When it travels through a fused silica substrate, the Gaussian stress pulse will be deformed according to the relation given in Eq. (2.64). The low stress at the front of the pulse travels most quickly. The peak stress, initially a few nanoseconds behind the front of the pulse, will be retarded relative to the front of the pulse. The rising part of the compression pulse will be stretched and eventually evolve into a linear ramp. The falling part of the stress pulse evolves in a very different manner. The tail of the initial pulse propagates more quickly than the peak. After certain time, the tail overtakes the peak and a discontinuity in stress (and therefore in material velocity) develops. The time it takes for the tail of the stress pulse to overtake the peak, t_{sh} , can be estimated using the material

properties apparent in the slope of the ramp in Eq. (2.64), the fall time of the laser pulse T , and the peak stress of the Gaussian stress pulse σ_p as

$$t_{sh} = \frac{T\alpha^2}{\beta\sigma_p} \quad (2.65)$$

The deformed profile of an initial Gaussian stress pulse at any location x before it forms the shock is given by the stress—time—of—arrival relation

$$t = t_0 + \frac{x}{c_0} \left(1 - \frac{\beta\sigma}{\alpha^2}\right), \quad \text{for } x < c_0 t_{sh} \quad (2.66)$$

where t_0 is related to stress σ by Eq. (2.67), i.e.,

$$\sigma(t_0) = \sigma_{max} \exp\left(-\frac{2t_0^2}{T^2}\right), \quad (2.67)$$

where σ_{max} is the peak compressive stress.

Equation 2.65 indicates that the time for a stress pulse to evolve into a shock is directly proportional to the fall time of the laser pulse and inversely proportional to the amplitude of the stress. The laser to be used in the experiments has a fixed fall time of 4.1 ns. Thus, the higher the amplitude of the compressive stress, the shorter it takes the stress to evolve into a shock. From the values of T , α and β provided earlier, the estimated time for a shock to develop for a 1.0 GPa, 2.0 GPa, and 3.0 GPa stress pulse are 58 ns, 28 ns and 19 ns, respectively. Fig. 2.12 shows the evolution process of a 1.0 GPa, 2.0 GPa, and 3.0 GPa stress pulse over time. As expected in fused silica, an initial Gaussian stress pulse deforms with time. A shock is developed for all three stress pulses at a time slightly earlier than that predicted by Eq. 2.65. Stress pulses with higher peak values developed a shock in a shorter time.

In Fig. 2.13, the initial Gaussian stress pulses with peak values of 1.0 GPa, 2.0 GPa and 3.0 GPa are plotted together in (a), and the deformed stress pulses after traveling 20

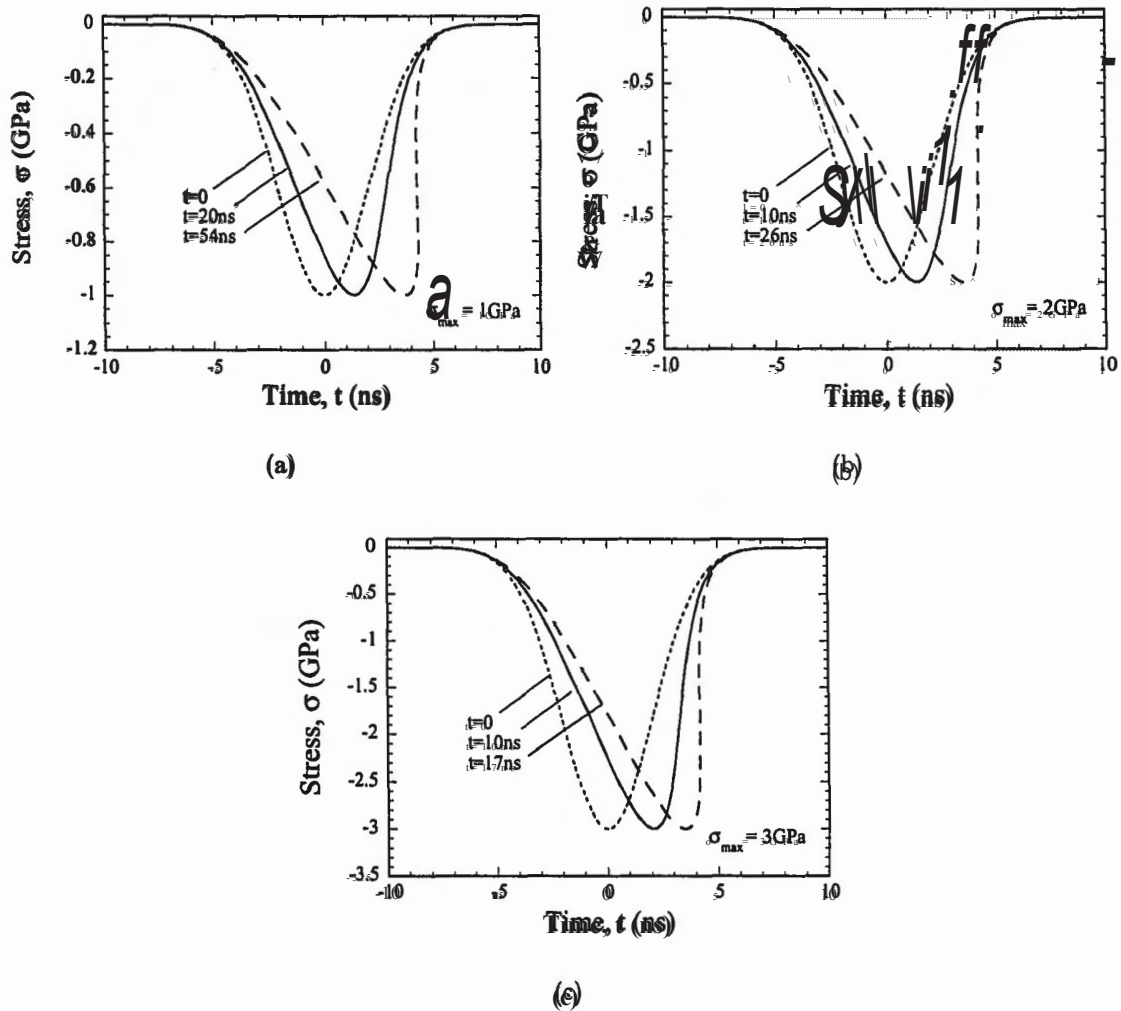
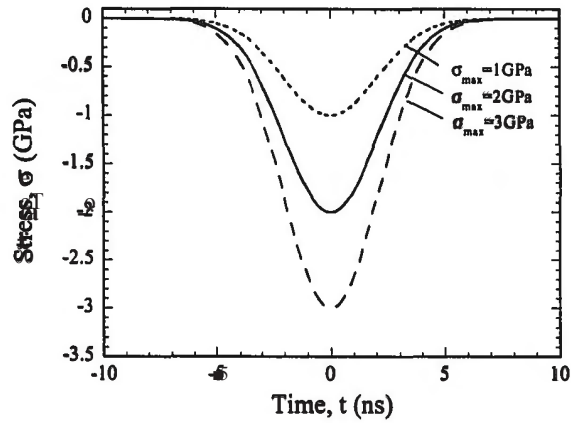
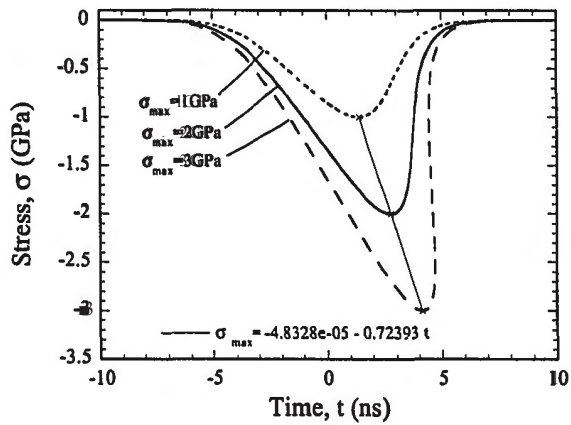


Figure 2.12: Evolution of the Gaussian stress pulse in fused silica with time: (a) 1.0 GPa peak stress; (b) 2.0 GPa peak stress; (c) 3.0 GPa peak stress.



(a)



(b)

Figure 2.13: Evolution of the Gaussian stress pulse in fused silica with time: (a) the initial Gaussian stress pulse of 1.0 GPa, 2.0 GPa, 3.0 GPa peak value; (b) the stress pulse in (a) after traveling 20 ns in the substrate.

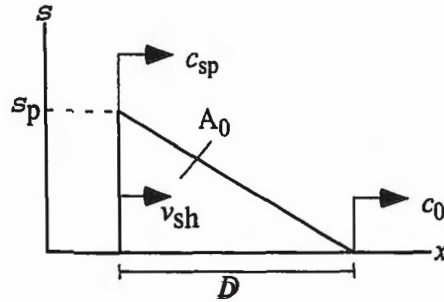


Figure 2.14: Schematic of the shock motion

ns in the media are plotted in (b). The peak stresses from the three stress pulses are fitted into a linear equation. The slope of the linear fit, -0.724 GPa/ns , is exactly predicted from Eq. (2.64).

After the shock is formed, the stress profile will continue to evolve according to the equal-area rule [52]. The evolution process can be explained using the simplified model shown in Fig. 2.14. The total area under the stress profile is A_0 , the duration of the ramp is D , and the peak stress at an arbitrary moment is σ_p . Once a discontinuity has developed at the trailing edge of the profile, it will move with the speed equal to the average of the wave speeds behind and ahead of the shock [43]:

$$\begin{aligned} v_{sh} &= \frac{v_{sh+} + v_{sh-}}{2} \\ &= c_0 \left(1 + \frac{\beta \sigma_p}{2\alpha^2} \right). \end{aligned} \quad (2.68)$$

Thus, the shock will move more slowly than the front of the linear ramp (zero stress). The ramp part will continue to be stretched and D will increase with time. According to the equal-area rule, the area A_0 will remain constant and thus

$$D = \frac{2A_0}{\sigma_p}, \quad (2.69)$$

$$\frac{\partial D}{\partial t} = -\frac{c_0 \beta \sigma_p}{2\alpha^2}; \quad (2.70)$$

Substituting (2.69) into (2.70), one obtains

$$\frac{\partial \sigma_p}{\partial t} = \frac{c_0 \beta}{4A_0 \alpha^2} \sigma^3. \quad (2.71)$$

The solutions of Eq. (2.71) takes the form

$$\sigma_p = \frac{1}{\sqrt{C_0 - \frac{c_0 \beta}{2A_0 \alpha^2} t}} \quad (2.72)$$

where C_0 is a constant. Application of the initial condition

$$\sigma_p = \sigma_{p0} \quad \text{at } t = t_{sh}, \quad (2.73)$$

where σ_{p0} is the peak stress at the time the shock forms, yields

$$C_0 = \sigma_{p0}^{-2} + \frac{c_0 \beta}{2A_0 \alpha^2} t_{sh}. \quad (2.74)$$

Substituting the value of C_0 into (2.72) and simplifying, one obtains

$$\sigma_p = \frac{\sigma_{p0}}{\sqrt{1 - \frac{c_0 \beta}{2A_0 \alpha^2} (t - t_{sh}) \sigma_{p0}^2}}. \quad (2.75)$$

Therefore

$$A_0 = \frac{1}{2} D \sigma_{p0} \approx \frac{1}{2} (2c_0 T) \sigma_{p0} = c_0 T \sigma_{p0}. \quad (2.76)$$

Substituting (2.76) and (2.65) into (2.75), one finally obtains

$$\sigma_p = \frac{\sigma_{p0}}{\sqrt{3/2 - \frac{\beta \sigma_{p0}}{2T\alpha^2}t}}, \quad \text{for } t > t_{sh}. \quad (2.77)$$

For fixed substrate thickness L ,

$$\sigma_p = \frac{\sigma_{p0}}{\sqrt{3/2 - \frac{\beta \sigma_{p0} L}{2c_0 T \alpha^2}}}. \quad (2.78)$$

The above equation is valid only after the shock has formed. In order to guarantee shock development before the stress travels through distance L , σ_{p0} must satisfy

$$\sigma_{p0} \geq \sigma_{p0}^{\min} \equiv \frac{c_0 T \alpha^2}{\beta L}. \quad (2.79)$$

For a 1.5 mm thick fused silica substrate $\sigma_{p0}^{\min} = 0.234$ GPa and for a 3 mm thick fused silica substrate, $\sigma_{p0}^{\min} = 0.116$ GPa. In Fig. 2.15, the values of the attenuation factor σ_p/σ_{p0} and σ_p are plotted as functions of σ_{p0} for two substrate thicknesses, 1.5 mm and 3 mm, respectively. As expected from Eq. (2.78), for the same initial peak stress σ_{p0} , greater attenuation occurred in the thicker substrate. For the same substrate thickness, L , greater attenuation occurs for larger initial peak stress σ_{p0} . The remaining peak stress σ_p , however, is still greater for greater initial peak stress σ_{p0} because $\sigma_p \propto (\sigma_{p0})^{1/2}$, for large σ_{p0} .

2.5 Conclusions

In this chapter, a one-dimensional thermo-elastic analysis was performed to predict the stress generated both in the substrate and at the film interface following thermal deposition of a YAG laser pulse. Both analytical and asymptotic solutions due to a delta laser pulse deposition as well as the practical solution due to the actual Gaussian laser pulse deposition were derived. The stress profile in the substrate was determined by the profile of the depo-

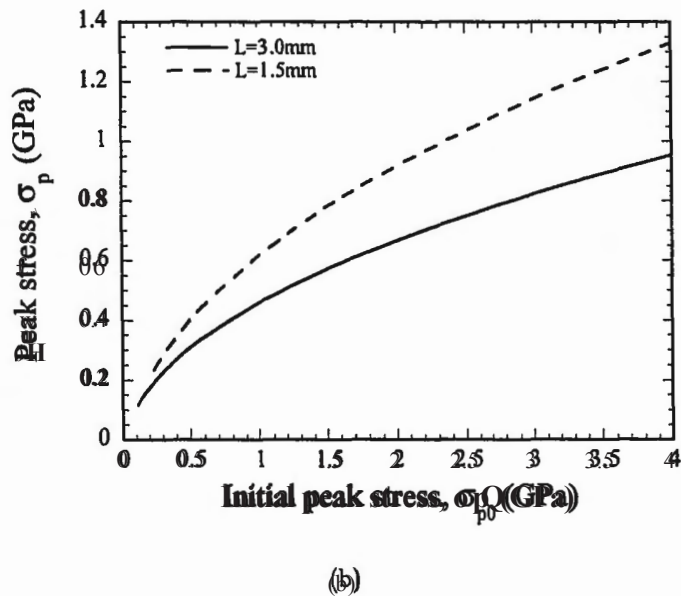
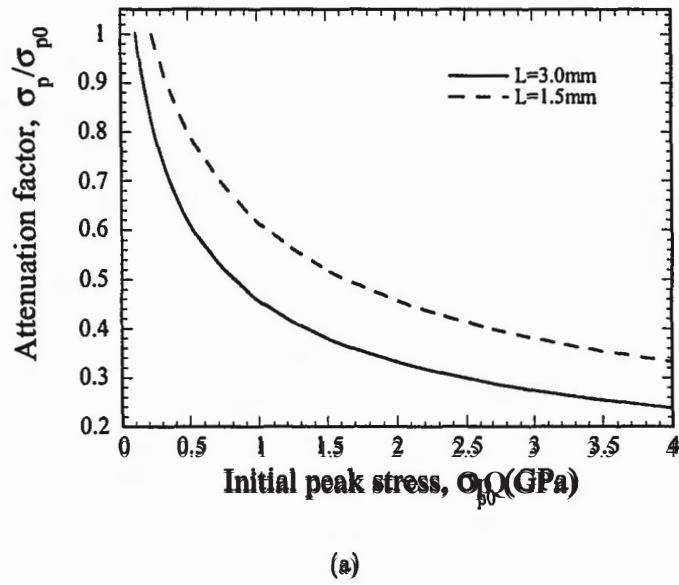


Figure 2.15: Shock attenuation in fused silica: (a) attenuation factor as a function of initial peak stress and substrate thickness; (b) remaining peak stress as a function of initial peak stress and substrate thickness.

sited laser pulse. For the stress at a thin-film interface, both simplified stress calculations based on Newton's second law and the exact calculations (Appendix B) using wave transmission and reflection theories were provided. For thin test films, both solutions gave the same results. When the test film is thick, however, the exact solution must be employed. Although the actual source mechanism in experiments is much more complicated than the thermo-elastic approximation (involving melting or ablation), these simplified calculations serve as a lower-bound prediction for the expected magnitudes of displacement and stress.

In experiments, the laser beam size is finite, and the sample geometry is more complicated. Significant stress attenuation may occur due to the finite source size. Based on the stress expression obtained from the one-dimensional analysis, further theoretical analysis for the stress wave attenuation with respect to the YAG beam size and the wave travel distance was carried out on a circular pancake YAG source. The wave attenuation was strongly dependent on the YAG beam size and travel distance. Greater attenuation occurs for smaller YAG beam size and longer travel distance.

For fused silica, which behaves as a nonlinear elastic solid up to the phase transition at 9.8 GPa, the initial Gaussian stress pulse generated at the absorbing layer evolves into a shock after traveling a short distance in the fused-silica substrate. The mechanism of shock development is analyzed using wave-propagation theory in nonlinear elastic solids and the nonlinear stress-strain relation obtained by Barker and Hollenbach [51]. It is found that the wave velocity is an approximately linear function of compressive stress. Higher stress has a slower traveling speed. Stress pulses with higher peak values evolve into a shock in a shorter time, i.e., after traveling a shorter distance. The substrate stress has a very short fall time due to the shock, which could greatly enhance interface loading and subsequent film failure. However, after the shock has formed, significant attenuation in the peak stress occurs as the stress travels through longer distances. The attenuation associated with the shock wave is even greater than that associated with geometric attenuation.

In designing the laser spallation experiment, both geometric attenuation and shock

attenuation should be considered. Hence, these theoretical analyses provide useful guidance in choosing the appropriate sample geometry and YAG spot size for the experimental investigation to follow.

Chapter 3

Tensile Spallation Experiment

3.1 Introduction

Several practical thin-film adhesion measurement techniques, including the scratch, peel, pull, blister and indentation tests, were summarized in Chapter 1. For each of these tests the interface is subjected to high stress levels and consequent inhomogeneous deformations. Large amounts of plastic deformation can result (especially in the peel test) and dominate the behavior during the test [16, 24]. The stress fields are difficult to analyze and the resulting adhesion measurements tend to be qualitative and comparative. Both the intrinsic work of adhesion and work of plastic dissipation control thin-film interfacial failure strength. The relationship between the macroscopic interfacial toughness (the total work of fracture) and the work of adhesion is an integral part of recent theoretical models of thin-film failure [53]. Experiments capable of characterizing the intrinsic interfacial strength, without any contribution from plastic dissipation, are therefore critical to obtaining a fundamental understanding of thin-film failure modes.

Different from above described methods, laser-spallation methods load the interface in a remote, non-contacting manner using laser-generated stress waves. This technique was first introduced by Vossen [25] and further developed by Gupta et al. [31-40]. The major advantage of the laser spallation technique over its counterparts is its ability to provide a fundamental measure of the interfacial strength, because interface decohesion is usually

achieved at a relatively high strain rate, during which all inelastic dislocation-related processes are essentially suppressed, and the measured strength is intrinsic to the interfacial structure.

In all the experiments reported to date, only normal-pressure pulses were applied to the films. Consequently, the interfaces were subjected only to tensile (mode I) loading. In most commercial applications, however, thin-film interfaces fail under mixed-mode conditions [1]. The design of experiments to measure the interface debonding over a range of mode mixity relevant to practical problems is a significant challenge. With the goal of developing an experimental technique for mixed-mode loading, this project starts with an investigation of the tensile laser-spallation technique. Although this technique has been reported on extensively in the literature, past work has largely focused on evaluating film adhesion, with questions relating to optimal experiment design given less emphasis. The effect of laser fluence was investigated by Vossen [25], Yang [29], and Gupta et al. [31]; the effect of absorbing layer thickness was reported by Yang [29]. Although the effect of sharpness and shape of the stress pulse, film properties, and film thickness were also studied by Yuan and Gupta [35], their investigation was mainly based on numerical simulation. In order to achieve a better understanding of the mechanics of thin film spallation, it was judged appropriate to investigate systematically the effects of film thickness, substrate thickness, and laser fluence. In this chapter, a series of spallation experiments for aluminum thin films on both (100) silicon and fused silica substrates of varying thickness are reported. The results reveal important design parameters for improving the tensile spallation experiments and developing the mixed-mode loading experiments.

3.2 Experimental Procedure

3.2.1 Experimental Setup

Figure 2.1 shows a schematic of the tensile laser spallation experiment. The mechanism of the stress-wave generation and propagation from the absorbing layer to the thin-film interface was described in Section 2.1. The nominal diameter of the YAG laser beam used in the current project is 3.5 mm. In order to increase the laser fluence, the laser beam is focused onto a 1-2 mm diameter spot on the energy-absorbing layer. The laser energy is increased until a longitudinal wave is generated with an amplitude sufficient to fail the film/substrate interface. Interferometric measurements of out-of-plane displacement are made at the surface of the test film. From displacement measurements at the free surface, the stress history at the interface can be inferred using standard wave mechanics [54] and the maximum stress acting on the interface can then be calculated.

A detailed schematic of the Michelson interferometer is shown in Fig. 3.1. A single, linearly polarized light beam of wavelength $\lambda_0 = 514.5$ nm from an argon laser is incident upon a beam splitting cube (BS). One beam is focused to a sharp point ($\sim 60 \mu\text{m}$) on the test film using an objective lens (L2), while the second beam is focused on a stationary mirror (M2). The two beams are recombined in the beam splitter and then imaged onto the active area of a photodiode detector. The signal from the detector is acquired using a digitizing oscilloscope. Because the stress pulse in the experiment has a rise time of less than a few nanoseconds, fringes must be resolved with sub-nanosecond resolution. Such signals have been successfully measured in plate-impact research [55-58], shock-wave studies [51], and previous spallation experiments [31-36]. An ultra-high speed photodetector was selected with a rise time less than 300 ps. A high-resolution LeCroy oscilloscope with a bandwidth of 1 GHz and a sample rate of 8 G sample/sec was used to record the interferometric fringes. Typical noise levels are less than 1% of a fringe.

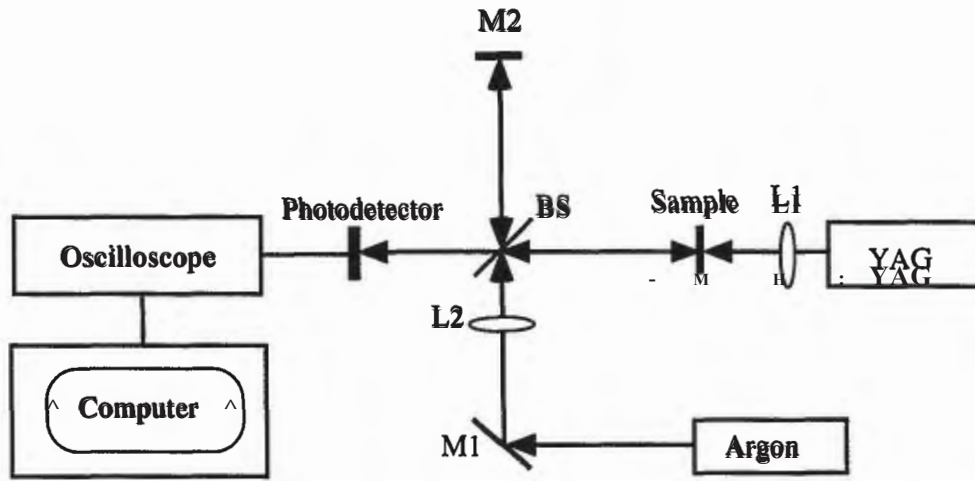


Figure 3.1: Schematic of laser-spallation experiment and interferometric displacement measurement.

3.2.2 Measuring Principles

Any movement of the sample surface causes a change in the signal frequency due to the Doppler effect. The Doppler frequency deviation for laser light of wavelength λ_0 reflected normally from a moving surface with velocity $V(t)$ is given by the Doppler shift equation as

$$\nu(t) = \frac{2V(t)}{\lambda_0}. \quad (3.1)$$

At the interferometer alignment of maximum sensitivity (the so-called $\pi/2$ point), the light intensity on the photodiode detector is related to the fringe count, $n(t)$, as

$$I(t) = \frac{I_{\max} + I_{\min}}{2} + \frac{I_{\max} - I_{\min}}{2} \sin(2\pi n(t) + \phi), \quad (3.2)$$

where $I_{\max}$ and $I_{\min}$ are the maximum and minimum intensities of the interference fringes and ϕ is the phase angle. The surface displacement is obtained in terms of the fringe count by integrating Eq. (3.1) [51]:

$$n(t) = \frac{\lambda_0 \nu(t)}{2}. \quad (3.3)$$

The fringe count is determined from the output of the photodiode detector (Eq. 3.2) and the displacement is calculated using Eq. (3.3). One complete fringe shift corresponds to a displacement of $\lambda_0/2$ (257 nm). The resolution of this type of interferometer is limited by the noise arising from fluctuations in the laser intensity and fluctuations in the environment (i.e., temperature). Once the free-surface displacement is obtained from the interferometric measurements, for small film thickness h , the compressive stress propagating from the absorbing layer towards the substrate is calculated using simple 1-D wave mechanics:

$$\sigma_{\text{sub}} = -\frac{1}{2}(\rho c)_{\text{sub}} \frac{\partial u}{\partial t}, \quad (3.4)$$

The interface stress is calculated using

$$\sigma_{\text{int}} = -(\rho h)_{\text{film}} \frac{\partial^2 u}{\partial t^2}, \quad (3.5)$$

as described in Chapter 2. For thick films, a more detailed analysis using wave transmission and reflection coefficients must be carried out to calculate stress accurately. Derivation of thick-film stress equations is provided in Appendix B.

3.2.3 Sample Fabrication

The thin-film sample used for the spallation experiments is shown schematically in Fig. 3.2. For these experiments, an aluminum film was used for both the absorbing layer and the test film. For all the samples, waterglass was chosen as the confining material based on the work of previous researchers [33] and on an extensive test comparison in our own lab between water, glass, and waterglass.

In preliminary experiments, water was applied with a squirt bottle to the back surface of the sample. The interferometric signal induced by the laser was enhanced slightly by the water, but the water dripped and evaporated too quickly, and the results were not consistent.

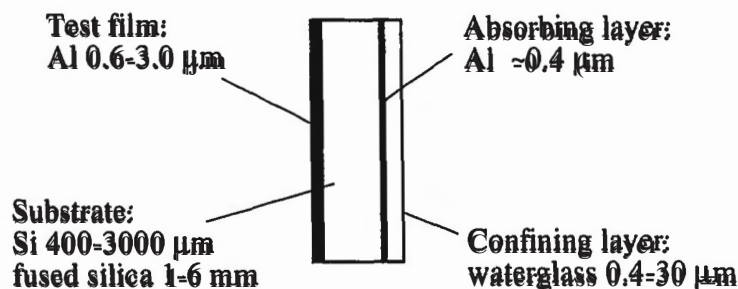


Figure 3.2: Schematic of thin film sample for spallation experiment.

Next, an ordinary glass slide was glued onto the backside of the sample. A large increase in the interferometric signal was observed, but the thickness of the glue layer tended to vary from sample to sample. When the glue layer was too thick, the signal decreased significantly. Finally, a layer of solid waterglass was tried as the confining material. The layer of waterglass was obtained by applying a layer of liquid waterglass ($\text{H}_2\text{SiO}_3 \cdot (\text{H}_2\text{O})_x$). After a few minutes' exposure to air, the water evaporates, leaving behind a layer of solid waterglass (SiO_2). Waterglass was found to provide consistent behavior and strong stress pulses with minimal expense.

Both fused silica and single-side-polished single crystal silicon (100) were investigated as candidates for the substrate material to be used in the mixed-mode experiments. Aluminum films were either evaporated or sputtered onto both sides of the sample. For the silicon substrate samples, the aluminum layer on the rough side of the substrate was used as an absorbing layer, while the aluminum film on the polished surface served as the test film. For all the samples, the absorbing layer was $0.4\text{--}1.0\ \mu\text{m}$ thick and the test film was $0.6\text{--}3\ \mu\text{m}$ thick.

Two film-deposition systems were adopted. In early experiments, the ATC2000 sputtering system in the Frederick Seitz Materials Research Lab was used for aluminum film deposition. The main chamber pressure was held at $4\text{--}8 \times 10^{-7}$ Torr and the sputtering rate was typically $1.5\text{--}2\ \text{\AA}/\text{s}$. The sputtered film usually has a good quality in terms of reflectivity and uniformity. Due to the low deposition rate, depositing a film thickness of a few microns

as required in this project became very costly in terms of the time and target material.

As a result, a thermal evaporator was used to deposit films for the majority of the experiments. This system provides a large range of deposition rates by adjusting the current going through the evaporating basket. For all the films tested in this thesis, the deposition rate was held between 10-15 Å/s. During deposition, the bell jar vacuum was controlled at $2-5 \times 10^{-6}$ Torr.

After deposition of the aluminum film, a thin layer of waterglass was applied onto the back surface of the absorbing aluminum layer as shown in Fig. 3.2. In order to generate high enough stress levels, different substrate thicknesses required different optimal thickness of waterglass. For waterglass layers 8 μm or greater, the waterglass solution was painted onto the sample using a swab. For waterglass thicknesses below 8 μm, the thin layer was obtained using a standard photolithography spinner (Headway Research Inc., Garland, Texas). For spin casting, the film thickness was governed by the spinner speed and the viscosity of the solution. The viscosity of the solution was adjusted by diluting the as-received waterglass solution to the desired extent.

3.3 Qualitative Damage Observations

The thin-film samples described in Section 3.2.3 were tested using the experimental apparatus described in Fig. 3.1. Prior to making any quantitative displacement measurements, a series of qualitative experiments were carried out to assess the influence of YAG laser fluence, thickness of the test film, and thickness of the substrate, on spallation pattern and damage to the test film.

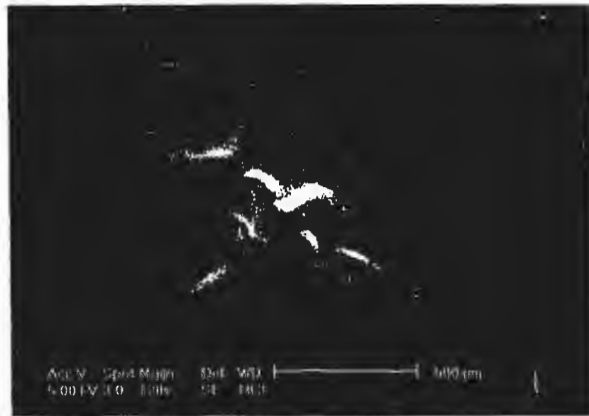
3.3.1 Spallation Patterns

The spallation patterns observed are strongly affected by the substrate material, film and substrate thickness, as well as the laser fluence. Typical damage patterns observed in 1.0 μm

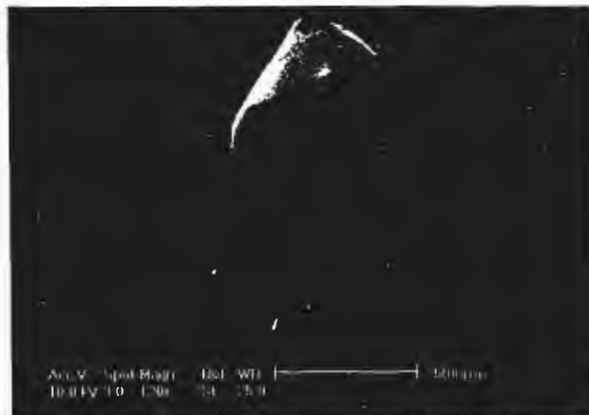
thick aluminum films deposited on 600 μm thick silicon substrates are shown in Fig. 3.3. In Fig. 3.3a, the aluminum film was folded and lifted from the substrate, but did not come off completely. In Fig. 3.3b, the aluminum film was spalled off from the substrate while leaving the substrate intact. In Fig. 3.3c, not only is the aluminum film completely spalled off the substrate, there is also a deep crater generated in the silicon substrate. The failure pattern in Fig. 3.3a typically occurs for thinner films or at lower laser fluence, while the pattern shown in Fig. 3.3c is usually observed for thicker films or at higher laser fluence.

In order to estimate the damage generated in the sample, a Sloan DekTak³ST stylus surface profilometer was used to scan across the surface of the damaged spot. Figures 3.4a and 3.4b show two other pictures of damaged aluminum film on silicon substrate similar to Figs. 3.3b and 3.3c. The height distribution along the dashed line in each figure was measured using the profilometer. The scanned data are plotted in Figs. 3.4c and 3.4d. The profilometer trace clearly shows the extensive damage caused in the silicon substrate. Damage patterns similar to Fig. 3.3a are not suitable for examination with the profilometer because the lifted film tends to be torn and pushed away by the stylus. Since the primary interest of this project is to measure the interface adhesion between film and substrate, substrate damage (Fig. 3.4b) is not desired.

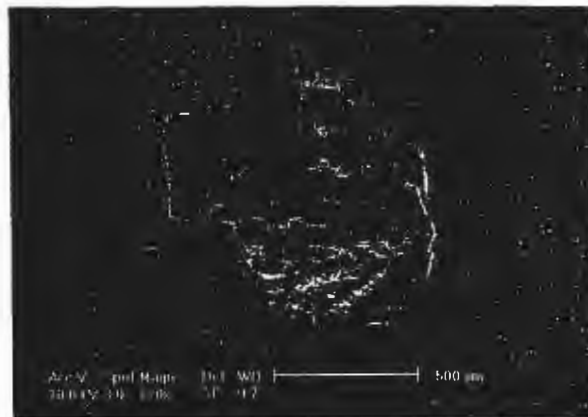
To investigate further the damage to the substrate, samples with no test film were fabricated. Two distinct damage patterns were observed on the bare silicon substrate (Fig. 3.5) with a thickness of 600 μm . In the first pattern, a crater formed in the substrate while in the second, a donut-like damage zone was observed. From the associated height profiles, the crater depth generated at full laser power was between 80 and 120 μm . Also, the height profile in Fig. 3.4d associated with the donut-like damage, shows that the central area of the donut region was elevated from the original substrate height and may indicate a deep crack under that region. While these damage patterns are typical for silicon substrates, they are not desired in the laser-spallation experiments. The severe damage in the substrate may cause the film to come off before the interface fails.



(a)



(b)



(c)

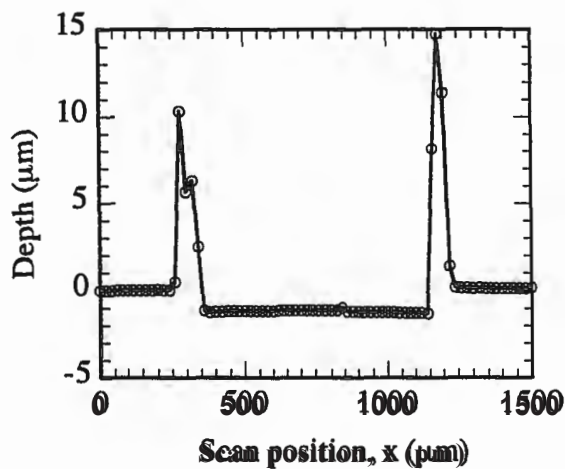
Figure 3.3: Damage observed for 1.0 μm thick aluminum film deposited on 600 μm thick silicon substrate: (a) film lift-off; (b) film spallation, no substrate damage; (c) substrate damage plus film spallation.



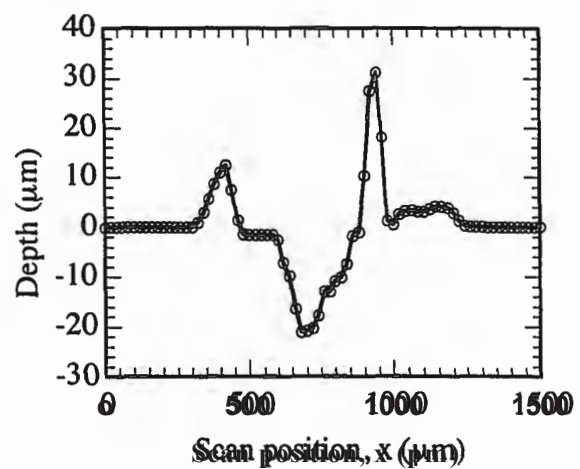
(a)



(b)

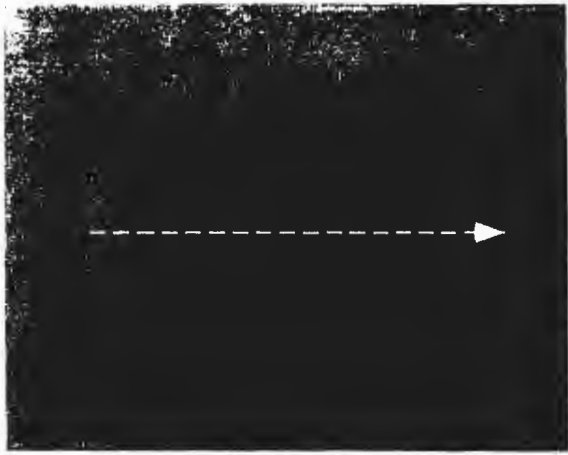


(c)

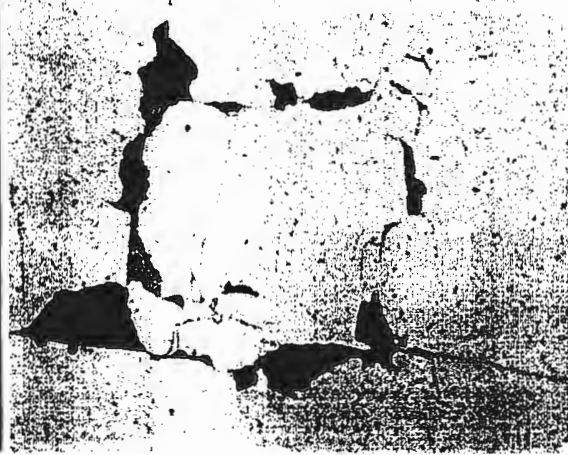


(d)

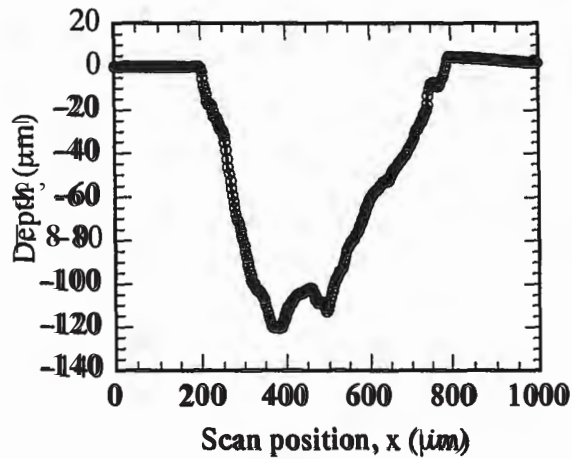
Figure 3.4: Damage observed for 1.2 μm thick aluminum film deposited on 600 μm thick silicon substrate: (a) no substrate damage, only film spallation; (b) substrate damage plus film spallation; (c) height profile corresponding to (a); (d) height profile corresponding to (b).



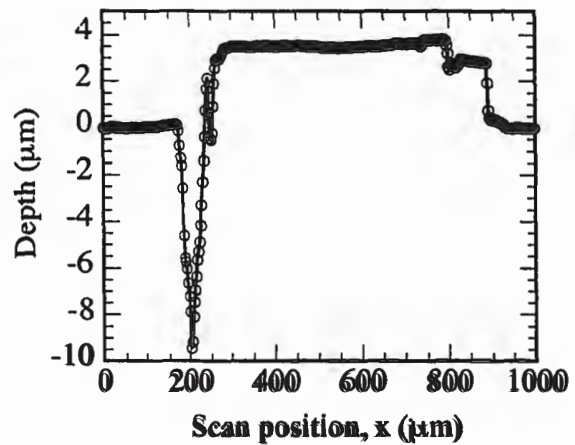
(a)



(b)



(c)



(d)

Figure 3.5: Damage patterns on bare silicon substrate (600 μm): (a) crater-shaped damage; (b) donut-shaped damage; (c) height profile corresponding to (a); (d) height profile corresponding to (b).

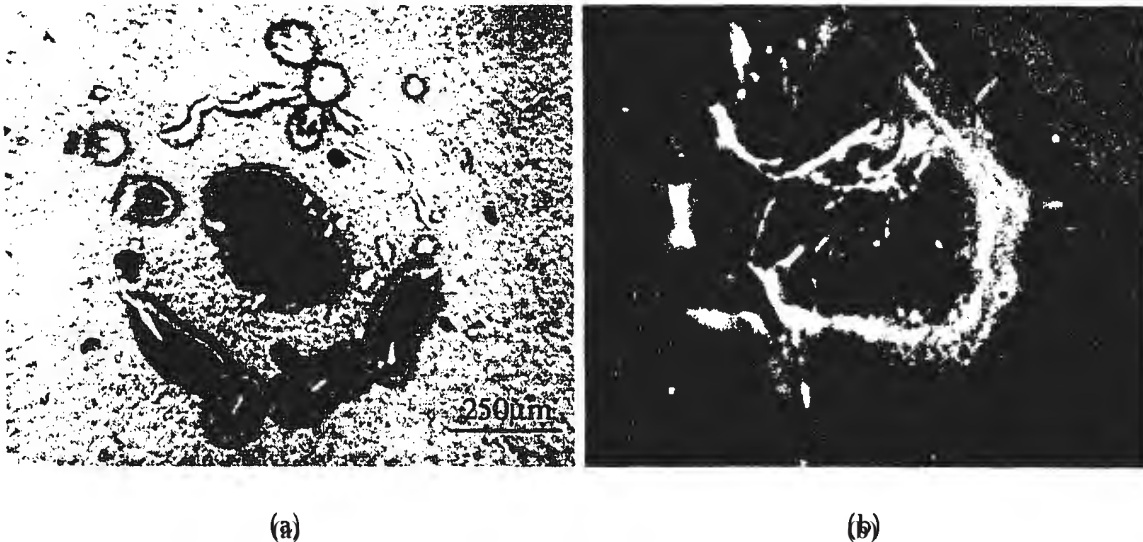


Figure 3.6: Typical damage patterns for a 1.9 μm thick aluminum film deposited on a 3000 μm thick fused-silica substrate sample: (a) lower laser fluence (0.07 J/mm²); (b) higher laser fluence (0.140 J/mm²). The diameter of the laser spot is about 1 mm.

Typical damage patterns for a 1.9 μm aluminum film on 3000 μm thick fused silica are shown in Fig. 3.6. Patterns like that in Fig. 3.6a typically appear at lower laser fluence (0.07 J/mm²) while patterns like that in Fig. 3.6b are representative of higher laser fluence (0.140 J/mm²). In fused-silica substrate samples, no substrate damage was observed.

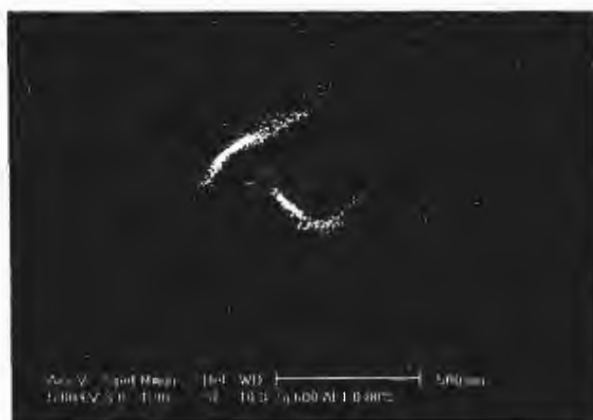
3.3.2 Effect of Laser Fluence on Film Spallation

The effect of laser fluence was studied on a sample with test film thickness of 1.0 μm , silicon substrate thickness of 600 μm and absorbing layer thickness of 1.0 μm . The YAG laser fluence was slowly ramped up from 0.056 J/mm² to 0.084 J/mm², 0.112 J/mm², and 0.140 J/mm² on different spots. Damage was observed for fluence greater than 0.084 J/mm². Figure 3.7 contains scanning electron microscope (SEM) pictures showing the development of damage patterns with increasing laser fluence. More damage was observed with higher laser fluence.

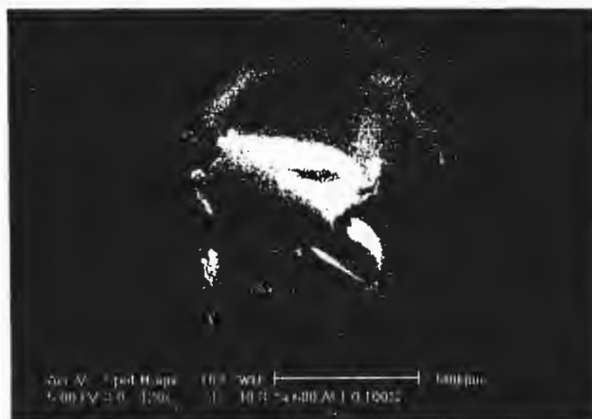
The effect of laser fluence was also studied for aluminum/fused silica samples. Figure 3.8 shows the SEM pictures showing the damage development on a 1.0 μm thick test film on



(a)



(b)



(c)

Figure 3.7: Effect of laser fluence on the damage of a $1.0\ \mu\text{m}$ thick aluminum film deposited on a $600\ \mu\text{m}$ thick silicon substrate: (a) laser fluence at $0.084\ \text{J}/\text{mm}^2$; (b) laser fluence at $0.112\ \text{J}/\text{mm}^2$; (c) laser fluence at $0.140\ \text{J}/\text{mm}^2$.

1500 μm thick fused-silica substrate. Laser fluence was increased from 0.056 J/mm² to 0.084 J/mm², 0.112 J/mm², and 0.140 J/mm². Damage was also observed for laser fluence greater than 0.084 J/mm². More damage was generated at higher laser fluence.

3.3.3 Effect of Test Film Thickness on Film Spallation

The effect of test film thickness was first investigated for samples with a constant silicon substrate thickness of 400 μm , aluminum absorbing layer of 0.6 μm , and waterglass thickness of ~ 20 μm . Three different test film thicknesses—0.4 μm , 1.4 μm and 2.0 μm —were studied at full YAG power. Spallation was observed at the test film for all three samples (Fig. 3.9). The extent of the damage strongly depends on the film thickness. The spallation patterns of the film in Fig. 3.9 indicate that for the same substrate thickness, same absorbing layer, and same YAG laser power, the extent of damage is larger for thicker films. This result is consistent with expectations from Eq. (3.5), i.e., a thicker film (larger h) has larger interface stress.

The effect of film thickness is also examined on a 1500 μm thick fused-silica substrate with two different film thicknesses, 0.3 μm and 0.9 μm . The damage patterns of the films at two laser fluences 0.084 J/mm² and 0.140 J/mm² are shown in Figs. 3.10 and 3.11. For both film thicknesses, more damage occurred at higher laser fluence. But at the same laser fluence, the two films show quite different damage patterns. On the 0.3 μm thick film, damage is developed as many straight-sided blisters, while the area between those straight-sided blisters remains attached to the substrate. In the 0.9 μm thick film, the blistered area is much larger with distinct "finger-like" regions. The number of fingers increases with increasing laser fluence. A possible explanation is that for the thicker test films, higher interface stress is generated; thus a larger area is delaminated and lifted off. For the same film thickness at higher laser fluence, more energy is stored in the film, resulting in more blistering.

Residual stress in the films was determined using wafer curvature measurements (Appendix C). The residual stress in the test films was slightly tensile, but very low for all thicknesses



(a)



(b)



(c)

Figure 3.8: Effect of laser fluence on the damage of a $1.0\ \mu\text{m}$ thick aluminum film deposited on a $1500\ \mu\text{m}$ thick fused silica substrate: (a) laser fluence at $0.084\ \text{J}/\text{mm}^2$; (b) laser fluence at $0.112\ \text{J}/\text{mm}^2$; (c) laser fluence at $0.140\ \text{J}/\text{mm}^2$.



(a)



(b)



(c)

Figure 3.9: Effect of test film thickness for aluminum film deposited on a 400 μm silicon sample:aluminum (a) aluminum film thickness = 0.4 μm ; (b) aluminum film thickness = 1.4 μm ; (c) aluminum film thickness = 2.0 μm .

(60–150 MPa). Thus the changes in damage patterns were solely due to the tensile stress generated at the interface. However, the observation of blister-like failure is different from the cracking failure that is usually associated with brittle films under residual tension [59].

3.3.4 Effect of Substrate Thickness on Film Spallation

To study the effect of substrate thickness on film failure, samples were fabricated using silicon wafers of thickness 400 μm , 500 μm , 700 μm , and 3000 μm while keeping the absorbing layer thickness at 1.0 μm . Several different test film thicknesses—0.6 μm , 1.0 μm , 1.5 μm , and 3.0 μm —were studied sequentially for each of the different substrate thicknesses. The results are summarized in Table 3.1. At full YAG power, spallation always occurred for the thinnest 0.6 μm film on the 400 μm and 500 μm substrates. No spallation was observed for the 0.6 μm film on the 700 μm and 3000 μm substrate. For the thicker 1.0 μm film, spallation occurred for all of the 400 μm and 500 μm substrate samples, for only a few samples with 700 μm substrate, and for no samples with a 3000 μm substrate. After increasing the film thickness to 1.5 μm , spallation was observed for all 400 μm , 500 μm , and 700 μm substrate samples but not for the 3000 μm substrate. Finally, for a film thickness of 3.0 μm , some spallation appeared on the 3000 μm sample.

Three different thicknesses of the fused-silica substrate samples—1500 μm , 3000 μm and 6000 μm —were also investigated. For an aluminum test film thickness of 1.0 μm , film spallation was observed for the 1500 μm fused-silica substrate at all laser fluences greater than 0.056 J/mm². No damage was observed on the 3000 μm and 6000 μm samples regardless of fluence. When a thicker test film of 1.8 μm was used, huge amounts of film damage were observed on the 1500 μm sample at very low YAG power. A tiny area of film failed on the 3000 μm sample under full YAG power (laser fluence of 0.140 J/mm²), while no damage was observed in the test film deposited on the 6000 μm substrate, even at full power.

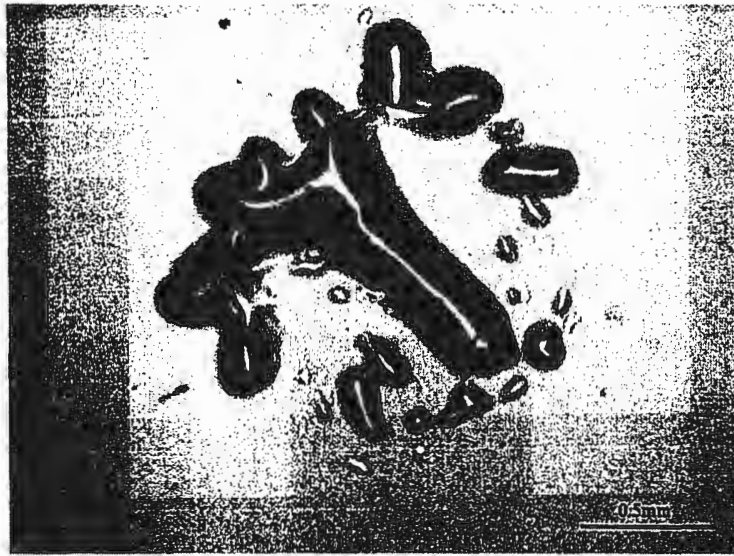


(a)

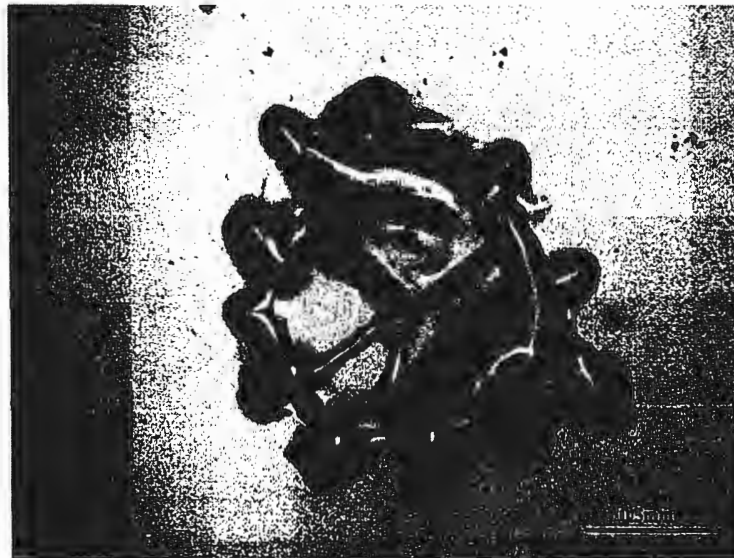


(b)

Figure 3.10: Damage development for a 0.3 μm thick aluminum film deposited on 1500 μm thick fused silica substrate: (a) laser fluence at 0.084 J/mm²; (b) laser fluence at 0.140 J/mm².



(a)



(b)

Figure 3.11: Damage development for a 0.9 μm thick aluminum film deposited on 1500 μm thick fused silica substrate: (a) laser fluence at 0.084 J/mm^2 ; (b) laser fluence at 0.140 J/mm^2 .

Table 3.1: Effect of aluminum film/silicon substrate thickness on spallation.

Test film thickness	Substrate thickness			
	400 μm	500 μm	700 μm	3000 μm
0.6 μm	Yes	Yes	No	No
1.0 μm	Yes	Yes	Yes	No
1.5 μm	Yes	Yes	Yes	No
3.0 μm	—	—	—	Yes

Note: Yes means some spallation is observed; No means no spallation at all.

3.3.5 Observation of Backside Ablation

Besides the damage generated on the test film, damage patterns were also observed on the backside of the substrates. During the experiment, the energy from the YAG laser pulse causes melting, vaporization and volume expansion of the absorbing layer. The expansion of the aluminum absorbing layer is limited by the confining layer and substrate, and thus generates a high-amplitude stress pulse. As a result, a deep crater is left on the substrate in the area exposed by the YAG laser pulse. The ablation patterns are strongly affected by YAG power level and the confining layer (waterglass) thickness. Figure 3.12 contains six optical photographs of craters on the fused silica substrates with three different waterglass confining layer thicknesses—30 μm , 10 μm and 4 μm —at two YAG laser powers. For the samples with 30 μm and 10 μm thick waterglass confining layers, many parallel lines were left on the fused silica substrates. More lines are associated with a thicker confining layer and higher YAG power. For the samples with a 4 μm thick waterglass layer, no lines were observed.

The depth of the crater is measured using the Sloan DekTak³ST stylus surface profilometer mentioned earlier. The depth profile along a line across the crater shown in Fig. 3.12d is plotted in Fig. 3.13. The profilometer trace shows that more material is ablated from the substrate near the edge of the YAG laser pulse with a maximum depth of about 1.2 μm . The central portion of the crater is flatter, with an average depth about 0.2 μm . The physical process that generates these craters and parallel lines is unknown. It is nevertheless clear

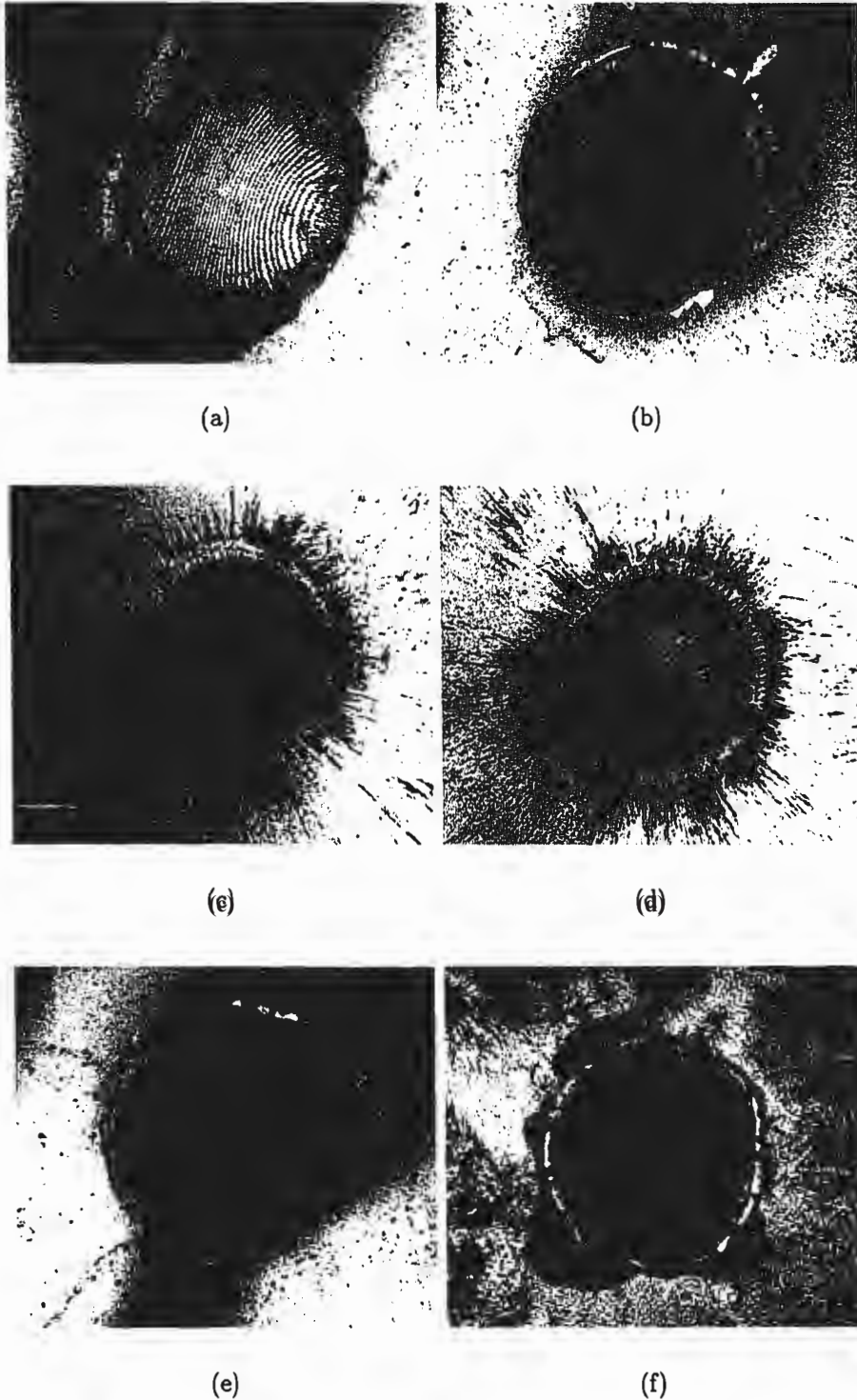


Figure 3.12: Backside craters on fused silica substrates: (a, b) 30 μm thick waterglass layer; (c, d) 10 μm thick waterglass layer; (e, f) 4 μm thick waterglass layer. (a, c, e) high YAG power; (b, d, f) low YAG power.

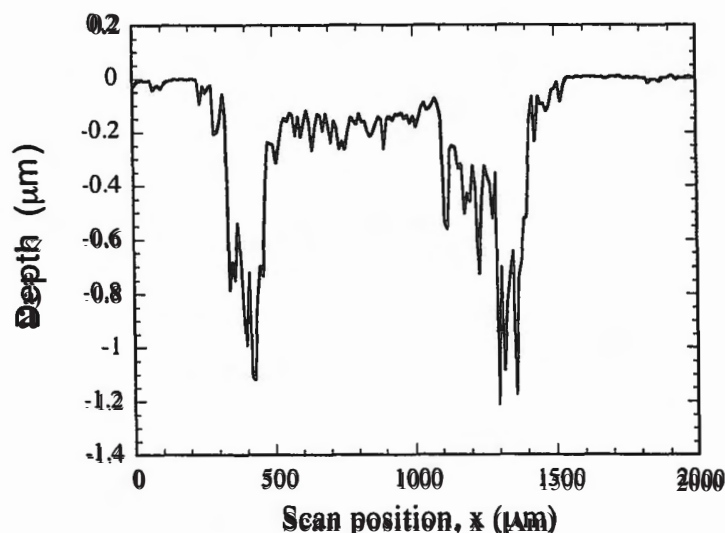


Figure 3.13: Crater depth associated with Fig. 3.12d.

that the ablation pattern with more parallel lines and higher depth is always associated with greater waterglass thickness and more test film damage.

The observations of the backside craters on the substrate provide evidence that the actual wave-generation mechanism following the deposition of a YAG laser pulse at the absorbing layer is indeed much more complicated than the thermal-elastic calculation assumed in Chapter 2.

3.3.6 Discussion

For both fused-silica and silicon substrate samples, higher YAG laser fluence leads to greater amounts of damage to the film. Thicker test films and thinner substrates also lead to greater damage. The effect of substrate thickness indicates that there may be strong stress attenuation with respect to substrate thickness. The damage observations also reveal that at high laser fluence, there is a large amount of damage in the silicon substrate due to cleavage. Since the ultimate goal of this project is to test the adhesion between the film and substrate, substrate cleavage is undesirable. In the fused-silica substrate samples, however, no substrate damage was observed, indicating that fused silica may be a better substrate

material to study the mechanics of the laser-spallation test.

3.4 Quantitative Interferometric Measurement

Quantitative measurements of displacement, substrate stress, and interface stress were obtained using a Michelson interferometer, as shown in Fig. 3.1. A typical interferometric signal measured for a $1.0\text{ }\mu\text{m}$ thick aluminum film deposited on a $600\text{ }\mu\text{m}$ thick silicon substrate is shown in Fig. 3.14a. From Eqs. (3.2) and (3.3), the displacement profile of the film free surface (Fig. 3.14b) is determined from the fringe data (Fig. 3.14a). As discussed in Section 3.2.2, the compressive substrate stress (Fig. 3.14c) and the stress at the test film interface after reflecting from the free surface (Fig. 3.14d) are calculated from Eqs. (3.4) and (3.5), respectively. In the data for silicon substrate samples shown in Fig. 3.14, five fringes developed within 15 ns, corresponding to a maximum displacement of 1400 nm. The maximum compressive substrate stress of 1.7 GPa was achieved shortly after the wave arrival and the maximum tensile interface stress of 60 MPa occurred slightly later. The long fall time in Fig. 3.14c leads to only modest tensile loading of the test film interface. Despite the differences between the laser pulses, thicknesses of the film, substrate, and confining layer, the magnitudes of the stress in the silicon substrate fall in the same range as those reported by Yuan and Gupta [35].

Interferometric data are shown in Fig. 3.15 for a $1.0\text{ }\mu\text{m}$ thick aluminum film deposited on a $3000\text{ }\mu\text{m}$ thick fused silica substrate. Figure 3.15a contains the raw fringe data. The other figures show the results from processing those data, namely the associated displacement and stresses. The fringe data in Fig. 3.15 are significantly different from those of the silicon substrate. The fringes develop over 20 to 30 ns in fused silica, whereas in silicon, the full waveform was confined to a duration of about 10 ns comparable to the duration of the YAG pulse. In silicon samples, the spacing between successive fringes grows with time; in fused silica the fringes become more tightly spaced. Possibly most telling is the occurrence of

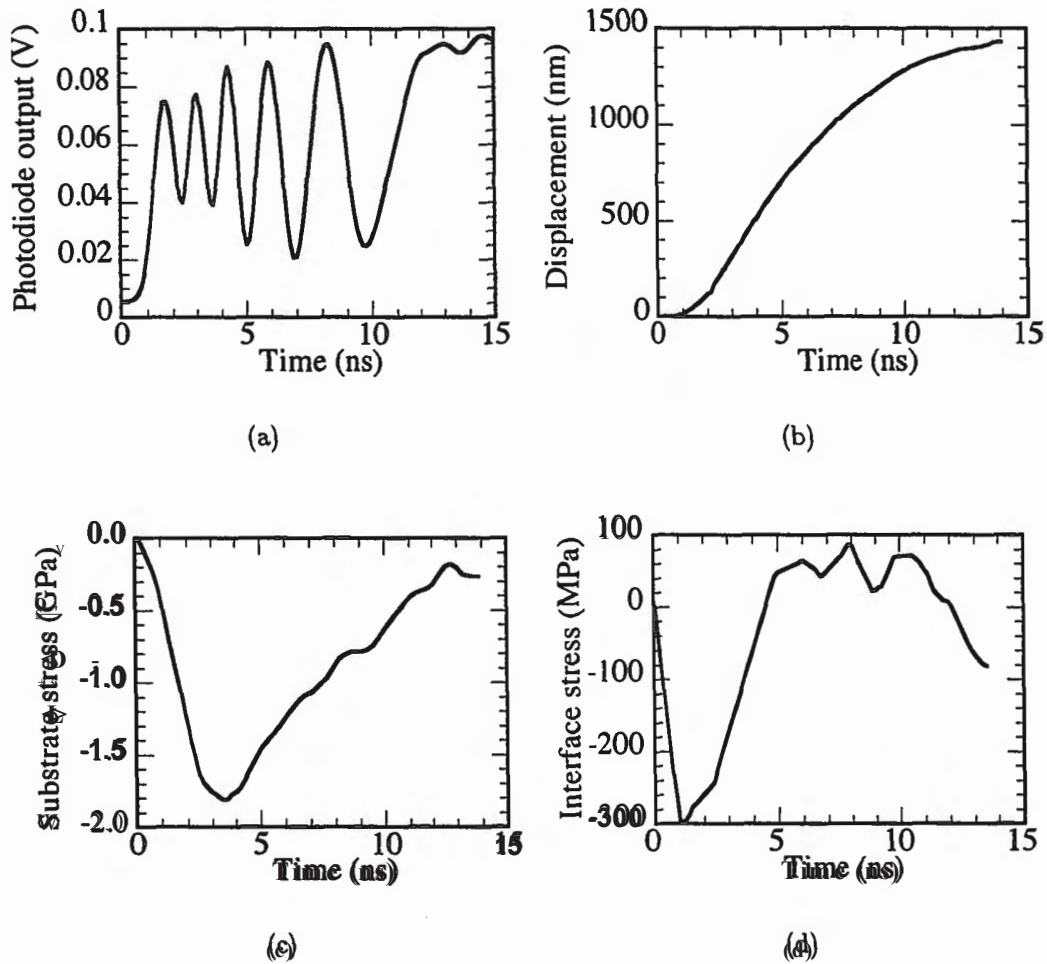


Figure 3.14: A typical data processing cycle for a 1.0 μm aluminum film deposited on a 600 μm thick silicon substrate (laser fluence of 0.140 J/mm²): (a) interferometric fringe pattern; (b) displacement profile; (c) substrate stress profile; (d) interface stress profile.

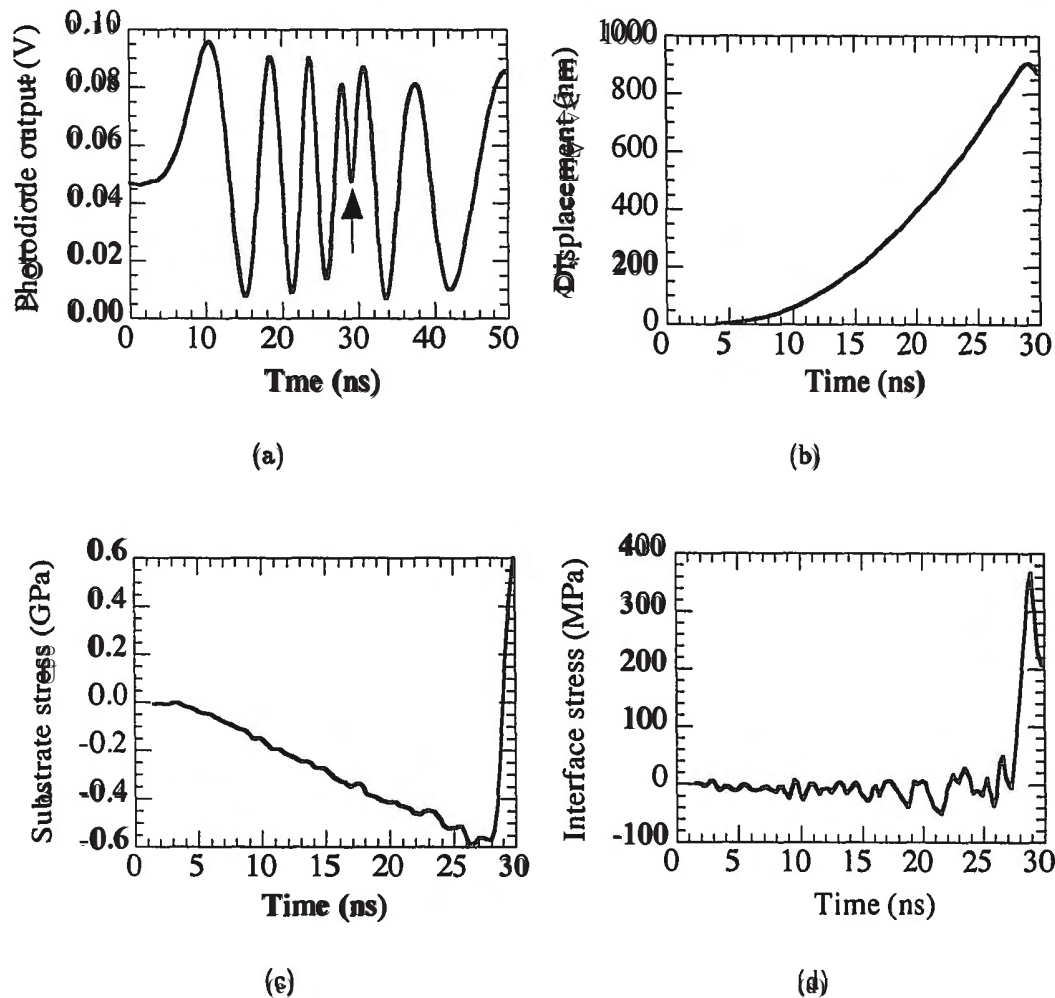


Figure 3.15: Typical data profile for a 1.0 μm thick aluminum film deposited on a 3000 μm thick fused silica substrate (laser fluence of 0.140 J/mm²): (a) interferometric fringe pattern, arrow points to turning point; (b) displacement profile; (c) substrate stress profile; (d) interface stress profile.

a turning point (marked by an arrow in Fig. 3.15a), where the material velocity changes direction in the middle of a fringe.

That there is a turning point here is supported by theoretical considerations. Fused silica has significant nonlinear elastic properties, softening at compressive strains up to 5% and stiffening at higher strains. This behavior is well documented by studies of shocks from the impact of flyer plates [29]. The theory of the development and subsequent evolution of such "weak shocks" may be found in standard references, *e.g.*, [52] and is summarized in Chapter 2. As expected from the shock analysis, the initial Gaussian stress pulse evolves into a shock long before it reaches the test film. The rising part of the initial compression pulse thus evolves into a linear ramp, a classic acceleration wave. This ramp is precisely what is seen in Fig. 3.15c. The slope of that ramp (in units of stress per time/thickness) is a material parameter and is the same as that reported by Barker and Hollenbach [51]. The falling part of the stress pulse evolves in a very different manner. The tail of the initial pulse propagates more quickly than does the peak. It overtakes the peak in a time which may be calculated using the material properties apparent in the slope of the ramp. A discontinuity in stress (and therefore also in material velocity) develops when the tail overtakes the peak. That sufficient time has occurred in the present case for a decompression shock to develop is evident. After traveling through the substrate, the rising front edge of the pulse has stretched from about 4.1 ns to 20 ns or 30 ns. The falling tail-end of the pulse has compressed by the same amount. An initial fall time of a few nanoseconds has therefore been reduced to nearly zero and occurs long before the pulse arrives at the test film. The experimental picture is complicated somewhat by the presence of a tensile wave, reflected from the back of the confining layer. The surface properties of that confining layer are not well controlled. Assuming a plausible longitudinal wave speed in the confining layer, the original compressive pulse is followed, about 30 ns later, by a tensile pulse of comparable magnitude. Being tensile, it propagates faster than the peak compressive stress, and faster than the low stress tail of the main pulse. It eventually overtakes the compressive pulse and linear dissipative mechanisms govern the

final thickness of the shock. In Fig. 3.15, the shock is followed by a regime in which the velocity has changed sign indicating that the tensile pulse has overtaken the compression pulse. The decompression shock is especially beneficial for tensile thin film tests, where the optimal compressive pulse ought to have a rapid fall time [36].

Using these data acquisition and processing procedures, a large number of tests were performed on both silicon and fused silica samples. The effects of laser power, substrate thickness, film thickness and confining layer thickness were investigated and the results are summarized below.

3.4.1 Effect of Laser Fluence

The effect of laser fluence was studied on a sample with a test film thickness of $1.1\ \mu\text{m}$, fused-silica substrate thickness of $3000\ \mu\text{m}$, absorbing layer thickness of $0.4\ \mu\text{m}$ and confining layer thickness of $\sim 20\ \mu\text{m}$. The YAG laser fluence was stepped from $0.056\ \text{J}/\text{mm}^2$ to $0.140\ \text{J}/\text{mm}^2$ as the YAG laser was moved to different positions on the sample. Fig. 3.16 shows the development of stress with increasing laser power. As laser fluence increases, the maximum compressive substrate stress also increases. At $0.056\ \text{J}/\text{mm}^2$ laser fluence, the maximum substrate stress of $0.28\ \text{GPa}$ is achieved in a rise time of $18\ \text{ns}$; while for $0.140\ \text{J}/\text{mm}^2$ laser fluence a maximum substrate stress of $0.6\ \text{GPa}$ is obtained in $28.5\ \text{ns}$. The higher maximum stress always occurs at later times and is approximately proportional to the laser fluence. The shock profiles in Fig. 3.16 are consistent with the nonlinear behavior of fused silica. The slope of the ramp is fitted by a linear function. The average slope of the stress ramps, $-0.026\ \text{GPa}/\text{ns}$, is close to the theoretical value, $-0.028\ \text{GPa}/\text{ns}$, obtained using Barker's material parameters [51]. From these stress profiles, an effective wave speed can be extracted as a function of stress. The higher compressive stresses arrive later because higher compression means slower waves. The corresponding tensile interface stress can be estimated from Fig. 3.16 as described previously. The maximum interface stresses are $200\ \text{MPa}$, $100\ \text{MPa}$, $350\ \text{MPa}$ and $400\ \text{MPa}$ for $0.056\ \text{J}/\text{mm}^2$, $0.084\ \text{J}/\text{mm}^2$, $0.112\ \text{J}/\text{mm}^2$ and $0.140\ \text{J}/\text{mm}^2$ laser

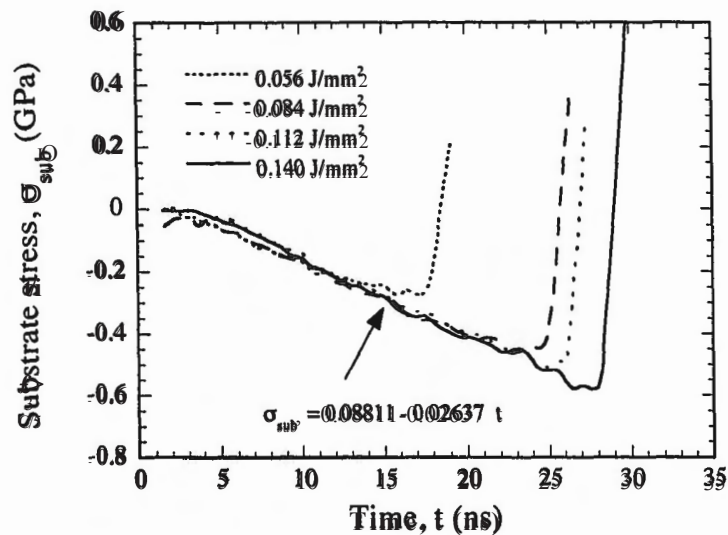


Figure 3.16: Effect of laser fluence for a 1.1 μm thick aluminum film deposited on a 3000 μm thick fused silica substrate with 0.4 μm thick absorbing layer.

fluence, respectively. These numbers are obtained by numerical differentiation of the data in Fig. 3.16 and are somewhat imprecise. Overall, the maximum interface stress increases with increasing laser fluence.

The effect of laser fluence was also studied on a 600 μm thick silicon wafer with a 1.1 μm thick test film, 0.4 μm absorbing layer and confining layer of $\sim 20 \mu\text{m}$. As YAG laser fluence increased from 0.056 J/mm² to 0.140 J/mm², the maximum compressive substrate stress increased from 0.7 GPa to 2.3 GPa, and in all cases occurred at approximately the same time after the wave's initial arrival, about 3.5 ns, comparable to the rise time of the laser pulse. The corresponding maximum tensile interface stress also increased from 60 MPa to 100 MPa. Significant damage occurred to the silicon substrate for laser fluence greater than 0.084 J/mm². Although the maximum substrate stress and maximum interface stress increase as laser fluence increases for both fused silica and silicon substrate samples, the data are quite different for the two kinds of samples. The maximum substrate stress and interface stress for the silicon samples were achieved in a very short time, usually within 5 ns after the wave arrival. But for the fused-silica substrate, the rise of the maximum substrate stress and interface stress was slower (from 15 to 30 ns). These differences are due to the development

Table 3.2: Effect of test film thickness on $\sigma_{\text{sub}}^{\text{max}}$ and $\sigma_{\text{int}}^{\text{max}}$ for fused silica samples at maximum YAG power (0.140 J/mm²).

Film thickness	1.1 μm	1.45 μm	1.9 μm
$\sigma_{\text{sub}}^{\text{max}}(\text{GPa})$	-0.6	-0.7	-0.8
$\sigma_{\text{int}}^{\text{max}}(\text{MPa})$	400	600	1000

of a decompression shock in the fused-silica substrate. In addition, the shock is responsible for the much greater interface stress in fused silica samples than in the silicon samples.

3.4.2 Effect of Film Thickness

The effect of test film thickness was studied for 3000 μm thick fused-silica substrate samples. Three film thicknesses—1.1 μm , 1.45 μm and 1.90 μm —were tested at full laser power (0.140 J/mm²). The absorbing layer thicknesses varied between 0.4 μm and 0.54 μm for these samples and was shown to have little effect. Previous experiments by Yang [29] also indicated that small variations in absorbing layer thickness have little impact on the generated stress. The results for these tests are summarized in Table 3.2. The maximum interface stress was found to be greater for thicker films. The large increase in interface stress for the 1.9 μm film, however, may be due to imperfect stress pulse generation or imprecision in the measurement. A small increase in the maximum substrate stress is also measured for increasing film thickness. This anomalous increase is possibly due to effects of excessive film damage for thicker films or variability in the stress pulse generation mechanism due to variations in the waterglass thickness.

3.4.3 Effect of Substrate Thickness

The effect of substrate thickness was investigated for both fused silica and silicon substrate samples with a 1.1 μm thick test film and 0.4 μm thick absorbing layer. The resulting maximum substrate stress and maximum interface stress values are summarized in Table 3.3 for three thicknesses of fused silica (1500 μm , 3000 μm and 6000 μm) and in Table 3.4 for

three thicknesses of Si (400 μm , 600 μm and 3000 μm). The interferometric data are similar to Fig. 3.14a for each thickness of Si. Fringe patterns for fused silica are similar to Fig. 3.15a. A decompression shock was observed for all three thicknesses of the fused silica. For both types of samples, the maximum substrate stress and maximum interface stress were found to be lower for greater substrate thickness. This observation is consistent with the theoretical analysis of the wave attenuation performed in section 2.3.

Table 3.3: Effect of substrate thickness on $\sigma_{\text{sub}}^{\text{max}}$ and $\sigma_{\text{int}}^{\text{max}}$ on silicon samples with a 1.1 μm thick aluminum film at maximum YAG power (0.140 J/mm²).

Substrate thickness	400 μm	600 μm	3000 μm
$\sigma_{\text{sub}}^{\text{max}}$	-1.7	-1.4	-0.55
$\sigma_{\text{int}}^{\text{max}}$	100	90	75

Table 3.4: Effect of substrate thickness on $\sigma_{\text{sub}}^{\text{max}}$ and $\sigma_{\text{int}}^{\text{max}}$ on fused silica samples with a 1.1 μm thick aluminum film at maximum YAG power (0.140 J/mm²).

Substrate thickness	1500 μm	3000 μm	6000 μm
$\sigma_{\text{sub}}^{\text{max}}$	-1.2	-0.5	-0.3
$\sigma_{\text{int}}^{\text{max}}$	370	350	50

3.4.4 Effect of Confining Layer Thickness

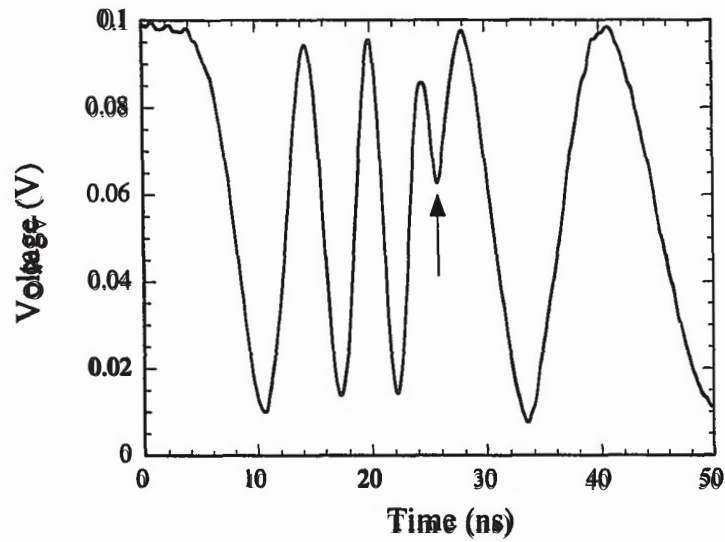
As the confining material, waterglass plays an important role in controlling the stress wave generation. When a YAG laser pulse reaches the absorbing layer, the sudden expansion of the absorbing layer produces a compressive stress pulse. The confining layer limits the expansion of the absorbing layer, generating a much higher stress pulse. The waterglass thickness greatly affects the amplitude of the generated stress pulse in the substrate. Parametric experiments on fused silica samples showed that only for a certain range of waterglass thickness can a strong enough shock be developed and propagated to the thin-film interface. Since the development of the shock in fused silica can greatly enhance the interface failure, it is desirable to obtain a well defined shock wave. If the waterglass is too thin, no shock

is detected either because no shock is developed at all or because the shock is so weak that it attenuated before reaching the film interface. Little or no damage occurs on the film. In order to develop a high amplitude shock to fail the thin-film interface and accurately estimate interfacial strength, different substrate thickness require different optimal confining layer thickness.

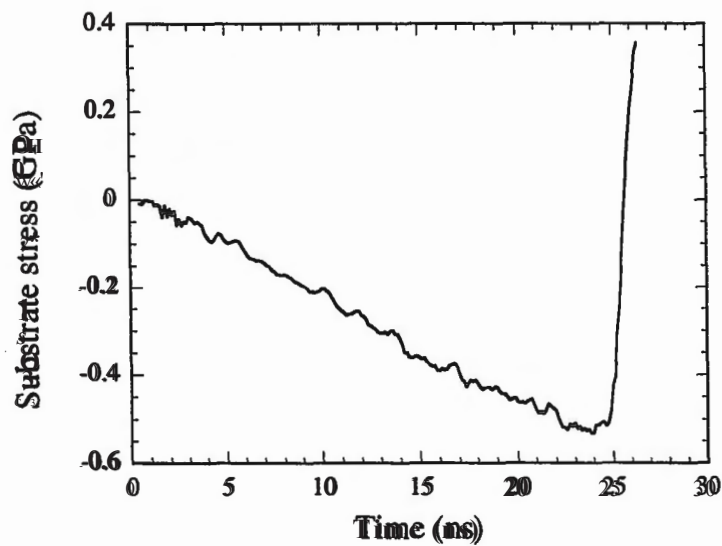
For a 3000 μm thick fused-silica substrate, a waterglass thickness greater than 8 μm is needed to develop a shock with high enough amplitude to fail the interface. Thicker waterglass results in higher substrate and interface stress, and greater film damage. For waterglass thickness less than 8 μm , no shock was measured and little damage was observed on the test film. Figures 3.17 and 3.18 contain interferometric data obtained for a 1.8 μm thick aluminum film deposited on a 3000 μm thick fused-silica substrate with waterglass thickness of 30 μm and 8 μm , respectively.

For the sample with a 30 μm thick waterglass layer, the fringe turning point occurs 26 ns after the wave arrival. More than 3 full fringes were developed before the fringes turned and a maximum compressive stress of 0.54 GPa was reached just prior to this point. For the sample with a 8 μm thick waterglass layer, the turning point occurred much earlier, about 13 ns after the initial wave arrival. Only one full fringe developed before the turning point and the maximum compressive stress was much lower, about -0.28 GPa. These data are consistent with estimates from fused silica material properties, lower stress travels faster and arrives earlier. The 1.8 μm thick aluminum film failed on the sample with 30 μm thick waterglass, while no damage occurred in the sample with 8 μm thick waterglass. Hence, thick waterglass is desired for generation of high stress and film failure.

For a 1500 μm thick fused-silica sample, no fringe turning point was observed for the same range of waterglass thickness ($\geq 8 \mu\text{m}$), but significant damage occurred in the test film. A typical interferometric signal obtained for a 0.5 μm aluminum film deposited on a 1500 μm fused-silica sample with 8 μm thick waterglass at 80% of the full YAG power is shown in Fig. 3.19. Within 40ns, 10 fringes developed corresponding to a total displacement

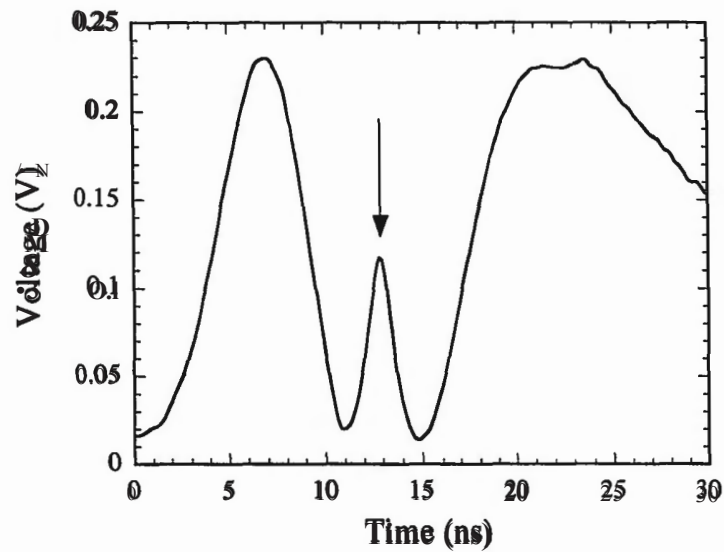


(a)

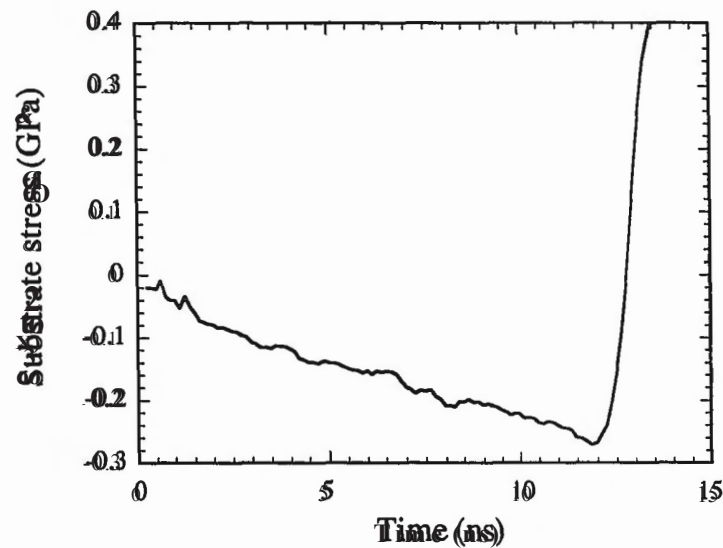


(b)

Figure 3.17: Interferometric signal obtained for a $1.8 \mu\text{m}$ thick aluminum film deposited on a $3000 \mu\text{m}$ thick fused-silica substrate with $30 \mu\text{m}$ thick waterglass (0.1440 J/mm^2): (a) photodiode output; (b) substrate stress. Arrow points to the position where fringes suddenly changed direction.



(a)



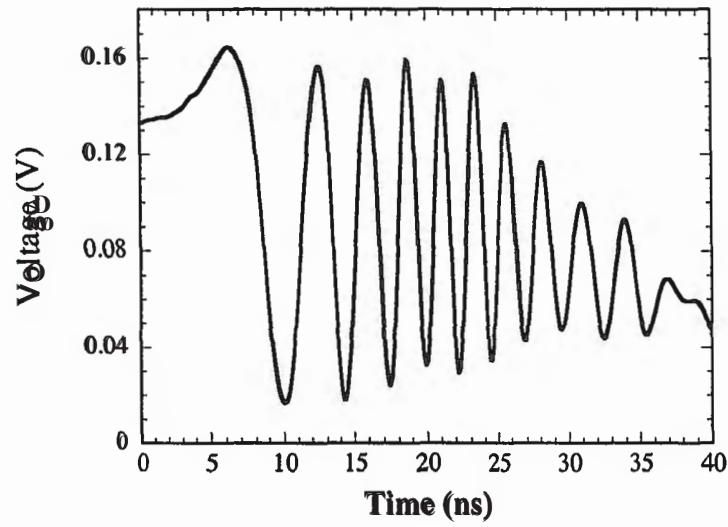
(b)

Figure 3.18: Interferometric signal obtained for a $1.8\text{ }\mu\text{m}$ thick aluminum film deposited on a $3000\text{ }\mu\text{m}$ thick fused-silica substrate with $8\text{ }\mu\text{m}$ thick waterglass (0.140 J/mm^2): (a) photodiode output; (b) substrate stress. Arrow points to the position where fringes suddenly changed direction.

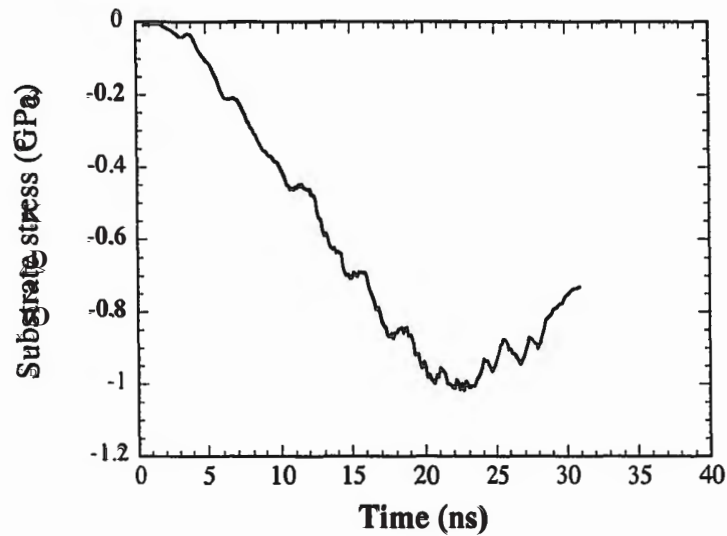
of 2.57 μm . Significant film damage was also observed. In the first 20 ns, the fringe spacing becomes tighter and tighter, indicating acceleration in surface velocity. Without any direction change after 20 ns, the fringe spacing becomes larger indicating a deceleration in surface velocity. The leading ramp ($t < 20$ ns in figure) in the substrate stress profile is consistent with the discussion of the initial effect of nonlinear elasticity for fused silica substrate (see Section 2.4). Because there is no sudden change of velocity (turning point), and no actual shock, there is a slow long fall time of the apparent substrate stress. Although the maximum substrate stress is high, 1.1 GPa, the resulting interface stress is quite low (~ 20 MPa for a half micron thick test film). A possible explanation for the discrepancy between extensive damage observation and low interface stress is that the fringe data is not reflecting the actual movement of the interface. After the test film delaminated from the substrate, the film keeps moving forward, which results in the large number of fringes and no turning point. Because the interface stress is proportional to the slope of the tail-end of the substrate stress wave, the fringe data associated with extensive film failure are not reliable for extracting the interface strength. The actual interface stress could be much higher than the measured value. The estimated substrate stress, however, is accurate until delaminating.

For the same confining layer thickness, experiments were also performed at reduced YAG powers for films thicker than 0.5 μm . YAG power of 40% (0.056 J/mm²) or below was required to avoid extensive film damage. When operated at such a low power, the reduced fringe count causes increased error in data processing. Reducing the waterglass thickness is a better strategy for accurate thin-film interface strength characterization.

In order to find the optimal waterglass thickness for 1500 μm thick fused silica substrates, a systematic study of waterglass thickness was carried out. The photolithography spinner described in Section 3.2.3 was used to spin-cast the waterglass layer at different thicknesses. Spin-casting conditions were optimized by controlling the spinner speed and viscosity of the waterglass solution. The viscosity was controlled by diluting the as received waterglass solution. The solution was deposited through a glass dropper onto the fused silica substrate,



(a)



(b)

Figure 3.19: Interferometric signal obtained for a $0.5\ \mu\text{m}$ thick aluminum film deposited on a $1500\ \mu\text{m}$ thick fused-silica substrate with $2\ \mu\text{m}$ thick waterglass ($0.112\ \text{J}/\text{mm}^2$): (a) photodiode output; (b) substrate stress.

initially spinning at 300 rpm. This slow spin speed is necessary to coat the central area of the specimen uniformly. After the central area was saturated with solution, the specimen rotation was manually increased to a higher speed for the remainder of the 2 minutes total spin time. Table 3.5 shows the relationship between the spinner speed and final waterglass thickness for different waterglass solutions.

Extensive experiments w.r.t waterglass thickness revealed that a thickness of $0.4\ \mu\text{m}$, obtained at a spin speed of 3000rpm for 1:1 diluted waterglass solution, is optimal. Figure 3.20 shows the interferometric data for a $0.5\ \mu\text{m}$ thick aluminum film deposited on a $1500\ \mu\text{m}$ fused silica substrate with waterglass thickness of $0.4\ \mu\text{m}$. The fringe data show a clear turning point at 22 ns. Almost 6 fringes developed before the turning point. A maximum compressive stress of 1.2 GPa was achieved in the substrate. For a $0.5\ \mu\text{m}$ thick aluminum film, a small amount of film failure was observed at the full YAG power and no failure was observed at 80% of the YAG power.

Thinner waterglass layers were also studied. Figure 3.21 shows the interferometric data and the associated substrate stress for a 1.5 mm thick fused-silica sample with a $0.25\ \mu\text{m}$ thick waterglass layer. The fringe data still show a clear turning point, but at a much earlier time, 13 ns after the initial wave arrival with a much lower maximum compressive stress, 0.52 GPa.

Thus, a thicker confining layer generates higher substrate stress and greater film damage. Higher maximum substrate stress arrives later than lower maximum stress. This observation is the same for both $3000\ \mu\text{m}$ and $1500\ \mu\text{m}$ thick fused silica samples, but with a different optimal waterglass thickness range.

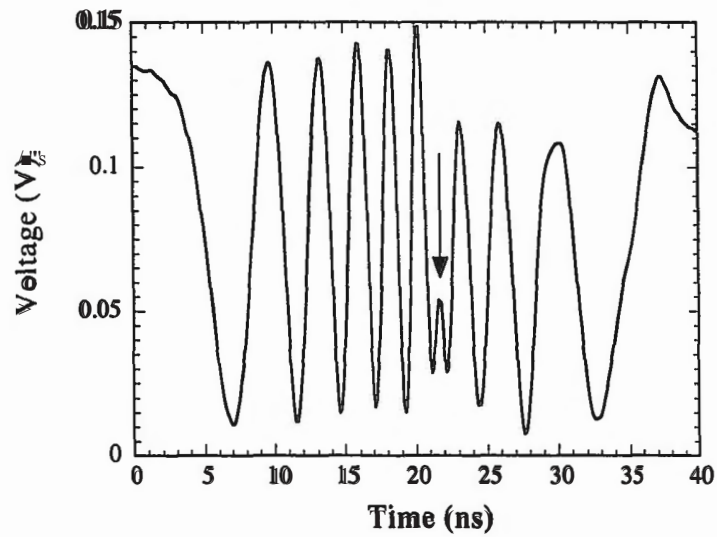
Table 3.5: Waterglass thickness as a function of spinner rpm and dilution.

Waterglass: water ratio	1000 rpm	2000 rpm	3000 rpm	4000 rpm	5000 rpm
2:0	8 μm	6 μm	4 μm	—	2 μm
2:1	1.88 μm	—	1.0 μm	—	0.8 μm
2:2	0.97 μm	0.54 μm	0.4 μm	0.33 μm	0.25 μm

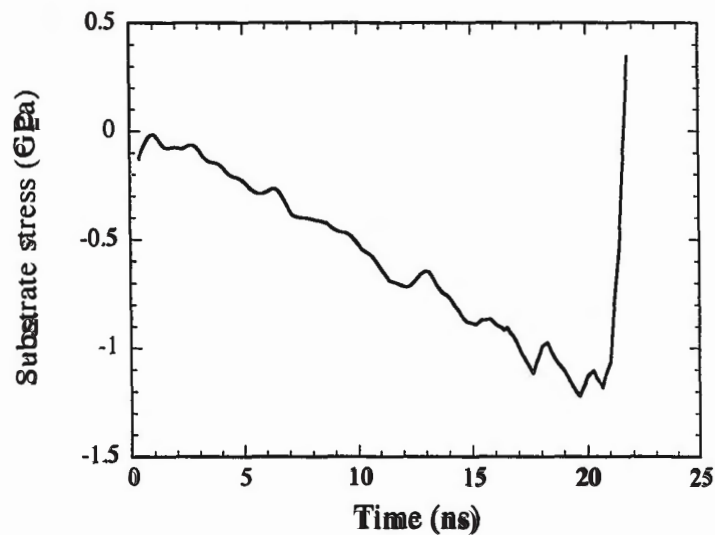
3.4.5 Interface Strength of Aluminum/Fused Silica Interface

The interface strength of aluminum/fused silica interface was measured on both 1500 μm and 3000 μm fused silica substrates with different film thickness. The YAG power was gradually stepped up until a small amount of damage was initiated at the film interface. For the 3000 μm fused-silica substrate, an aluminum test film of 1.8 μm thickness was used to characterize the interfacial strength. Film failure initiated at full laser power (0.140 J/mm²). The maximum substrate and interface stress for tests performed at eleven different spots on the same sample are summarized in Table 3.6. Since this film thickness is relatively thick, the maximum substrate stress and interface stress were calculated using both simplified equations (2.46), (2.47) and the exact solution (B.17), (B.18). At film failure, the maximum compressive substrate stress is about 0.57 GPa. The maximum interface stress is about 481 MPa from simplified calculations and 439 MPa from the exact solution. The interface stress obtained from the exact solution is 8.7% lower than the simplified calculations, but within the range of experimental error. The substrate stress at failure calculated using both simplified and exact calculations was the same. For a test film thickness of 1.8 μm or lower, the simplified calculations are valid for both the substrate and interface stress calculations. To show the consistency of these experiments, substrate stress profiles for the 11 tests are plotted together in Fig. 3.22.

Interface strength of the aluminum/fused silica interface was also measured on a 1500 μm thick fused-silica substrate for two aluminum film thicknesses, 0.5 μm and 1.8 μm . For the 0.5 μm thick film, most failure initiated at full laser power (0.140 J/mm²). For the 1.8 μm thick film, failure initiated at a lower laser fluence of 0.084 J/mm². The maximum substrate stress and maximum interface stress calculated using the simplified equations (2.46) and (2.47) for 5 tests performed at different spots are summarized in Tables 3.7 and 3.8 respectively. For the 0.5 μm thick film, the interface failed at an average maximum substrate stress of -1.12 GPa and interface stress of 497 MPa. For the 1.8 μm thick film, the interface

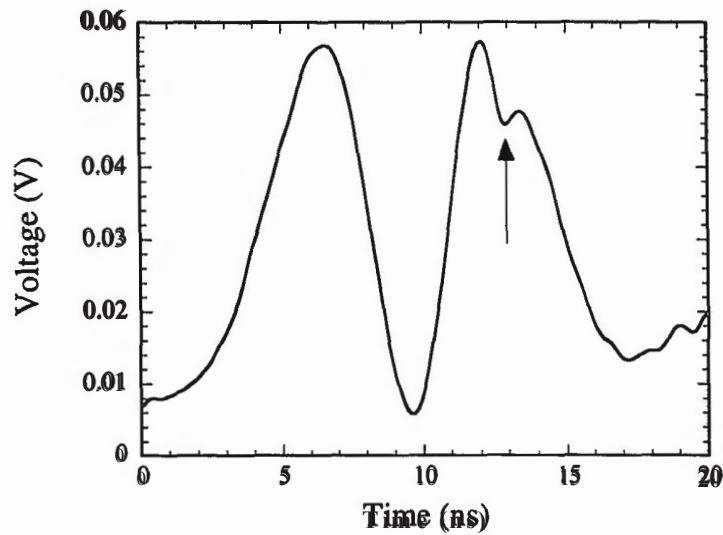


(a)

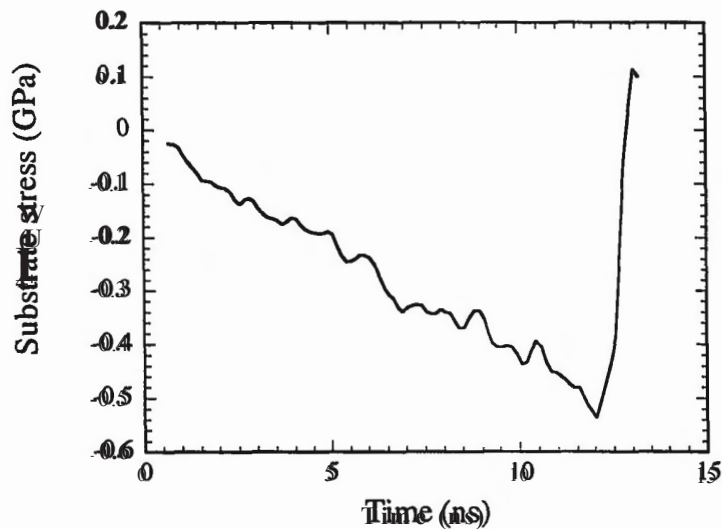


(b)

Figure 3.20: Interferometric signal obtained for a $0.5 \mu\text{m}$ thick aluminum film deposited on a $1500 \mu\text{m}$ thick fused-silica substrate with $0.4 \mu\text{m}$ thick waterglass (laser fluence of 0.140 J/mm^2): (a) photodiode output; (b) substrate stress. Arrow points to the position where fringes suddenly changed direction.



(a)



(b)

Figure 3.21: Interferometric signal obtained for a $0.5 \mu\text{m}$ thick aluminum film deposited on a $1500 \mu\text{m}$ thick fused-silica substrate with $0.25 \mu\text{m}$ thick waterglass (laser fluence of $0.140 \text{ J}/\text{mm}^2$): (a) photodiode output; (b) substrate stress. Arrow points to the position where fringes suddenly changed direction.

Table 3.6: Interface strength of a 1.8 μm thick aluminum film deposited on a 3000 μm thick fused silica substrate.

Test No.	Laser fluence (J/mm ²)	Substrate stress ^{1,2} (GPa)	Interface stress ¹ (MPa)	Interface stress ² (MPa)
1	0.140	-0.61	435	351
2		-0.55	460	377
3		-0.54	492	483
4		-0.57	462	408
5		-0.55	480	508
6		-0.59	478	440
7		-0.53	470	409
8		-0.58	532	503
9		-0.60	511	474
10		-0.54	505	500
11		-0.52	462	399
Range	0.140	-(0.53 - 0.61)	435 - 532	351 - 508
Average	0.140	-0.57 $\pm$ 0.03	481 $\pm$ 28	439 $\pm$ 54

¹Calculated using the simplified equations 2.46 and 2.47 valid for small film thickness

²Calculated using the simplified equations B.17 and B.18 for large film thickness

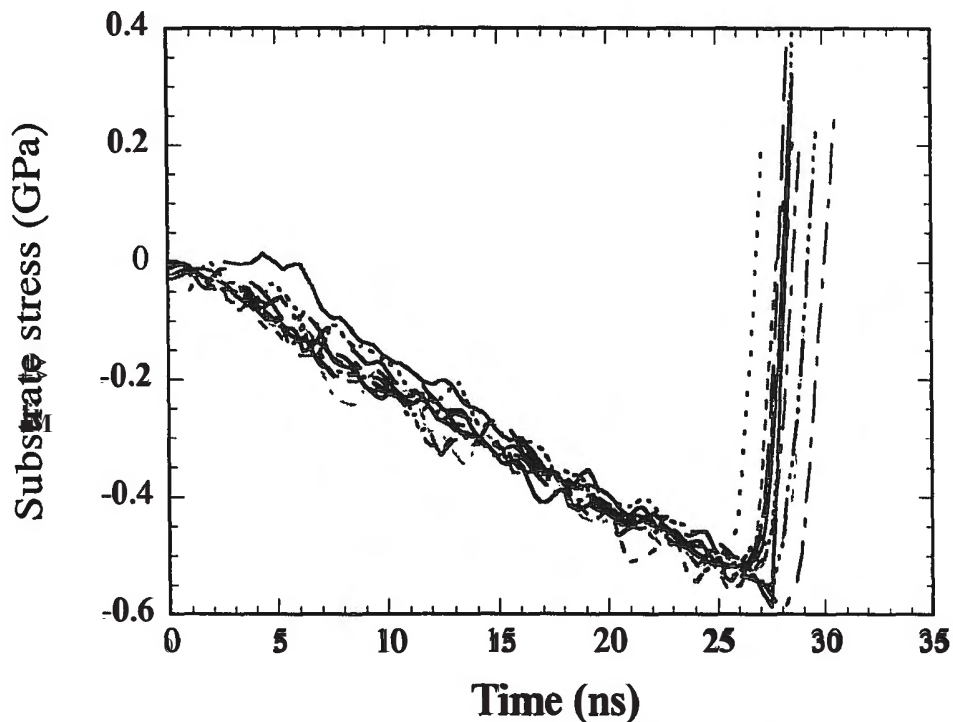


Figure 3.22: Substrate stress profile for a 1.8 μm thick aluminum film deposited on a 3000 μm thick fused silica substrate.

Table 3.7: Interface strength of a 0.5 μm thick aluminum film deposited on a 1500 μm thick fused-silica substrate.

Test No.	Laser fluence (J/mm ²)	Substrate stress (GPa)	Interface stress (MPa)
1	0.140	-1.1	583
2		-1.1	502
3		-1.2	420
4		-1.1	547
5		-1.1	435
Range	0.140	-(1.1 ~ 1.2)	420 ~ 583
Average	0.140	-1.12 $\pm$ 0.045	497 $\pm$ 70

Table 3.8: Interface strength of a 1.8 μm thick aluminum film deposited on a 1500 μm thick fused-silica substrate.

Test No.	Laser fluence (J/mm ²)	Substrate stress (GPa)	Interface stress (MPa)
1	0.084	-0.55	429
2		-0.62	474
3		-0.55	525
4		-0.61	568
5		-0.55	465
Range	0.084	-(0.55 ~ 0.62)	429 ~ 568
Average	0.084	-0.58 $\pm$ 0.04	492 $\pm$ 55

failed at an average maximum substrate stress of -0.58 GPa and interface stress of 492 MPa. Tables 3.6, 3.7 and 3.8 indicate that for different film thicknesses and different substrate thicknesses, the interface strength of aluminum/fused silica is approximately the same, 481 $\pm$ 28 MPa for 1.8 μm film on 3000 μm substrate, 497 $\pm$ 70 MPa for 0.5 μm film on 1500 μm substrate and 492 $\pm$ 55 MPa for 1.8 μm film on 1500 μm substrate. For the same film thickness on different substrate thickness, the maximum substrate and interface stress are the same, but lower laser fluence is required for the thinner substrate. This result is probably due to the reduced stress wave attenuation in the thinner substrates. For different film thicknesses on the same substrate thickness, thicker films require less laser fluence, and consequently lower substrate stress to fail. This result is consistent with the predictions from Eqs. (2.46) and (2.47), higher stress is generated in thicker films.

3.5 Conclusions

For both fused silica and silicon substrate samples, the maximum stress in the substrate and the maximum stress at the interface increased with increasing YAG laser fluence. For fused-silica substrate samples with test film thickness in the range of 1.1 μm and 1.9 μm , the maximum interface stress also increased with increasing film thickness. Hence, thicker films lead to higher interface stress and fail more easily. For both types of samples, the maximum substrate stress and maximum interface stress decreased with increasing substrate thickness probably due to geometric attenuation caused by the finite size of the YAG beam. In the case of the fused-silica substrate, there is also attenuation due to the shock evolution. A thicker confining layer helps to generate a higher compressive stress in the substrate and greater damage on the test film. In the experiments, several factors including YAG spot size, film thickness, substrate thickness, and confining layer thickness must be coordinated.

Observations of damage revealed both substrate and film failure in silicon substrate samples but only film failure in fused-silica substrate samples. Quantitative interferometric measurements confirmed the difference in stress development for the two kinds of sample. In fused silica substrates, the compressive pulse evolved into a decompression shock. The existence of a shock created a large stress at the film interface, promoting failure. The development of the shock is due to the nonlinear elastic properties of fused silica [51]. This shock mechanism is advantageous in the design of laser-spallation tests of thin-film interface adhesion. Moreover, fused silica is an ideal material for studying the mechanics of the spallation test in that it is intrinsically strong and no substrate damage occurs before the interfacial failure. Although shock development in fused silica was demonstrated in the 1970s by Barker and Hollenbach using a gas-gun facility, the usefulness of such shocks in applications to laser spallation has not been reported.

Chapter 4

Mixed-mode Loading Experiments

4.1 Introduction

For all the experiments reported in Chapter 3, thin-film interfaces were subjected only to tensile (mode I) loading. In most commercial applications, however, thin-film interfaces fail under mixed-mode conditions [1]. Design of experiments to measure the interface debonding over a range of mode mixity relevant to practical problems is a significant challenge. Several test methods for characterizing mixed-mode interfacial fracture of a bilayer have been proposed in literature, such as mixed-mode double cantilever beam [60], double cantilever drilled compression [1], mixed-mode flexure [61-66], and Brazil-nut specimen [67]. Most of these test methods involve sandwich configurations in which a film is deposited onto two substrates and subsequently bonded together before adhesion measurement. The bonding process requires diffusion either at relatively high homologous temperatures or under applied pressure, and often leads to changes at the interface and in the microstructure of the film. Thus, the bonding process plays a role in the nature and the magnitude of the energy-dissipative mechanisms. Measurement using these sandwiches is even more problematic for thin-film interfaces produced at relatively low temperatures (e.g., by sputtering or evaporation) due to difficulty loading the film in a controlled manner at the relevant phase angle. Since the film is sandwiched between two substrate materials, it is also difficult to examine the damage pattern resulting from interface failure.

In this chapter, a new test method is developed for studying mixed-mode interfacial failure of thin films using laser-generated stress waves. With this technique, the interfacial adhesion of a surface film deposited on a substrate is measured in the as-processed condition, which differs significantly from the sandwich specimens used in the literature.

4.2 Design of Mixed-mode Experiment

4.2.1 Sample Geometry

Mixed-mode loading is accomplished by modifying the sample geometry so that a high-strain-rate shear wave is generated by mode conversion at an oblique surface and then allowed to impinge upon the test film. A diagram describing the generation of high-amplitude, short-duration shear waves is shown in Fig. 4.1. Similar to the tensile spallation experiment, the sample consists of a transparent confining layer, a thin energy absorbing layer, a substrate and a test film. In order to generate a high amplitude shear wave, the planar substrate in the tensile spallation is replaced with a triangular prism. An infrared Nd:YAG pulse ($\lambda = 1064 \text{ nm}$) with a variable energy content between 1 and 110 mJ is incident on the absorbing layer at point C. The shape of the laser pulse fits well to a Gaussian distribution of the form in Eq. (2.44). The energy-absorbing layer is chosen to be much thicker than the critical penetration depth of laser light at this wavelength. Under this consideration, the laser energy is deposited at the interface of the confining layer and the energy-absorbing layer without penetrating the substrate. The nominal diameter of the YAG laser beam is 3.5 mm. Again, in order to increase the laser fluence, the laser beam is focused to a 1 mm diameter spot on the energy-absorbing layer.

When the laser pulse reaches the absorbing layer, a longitudinal compressive stress wave, L_1 , of rise time comparable to that of the laser pulse is emitted from the absorbing layer. The wave that propagates into the triangular prism is reflected at the oblique surface, and a major fraction of its energy — the exact amount depends on details of the angle θ_1 and

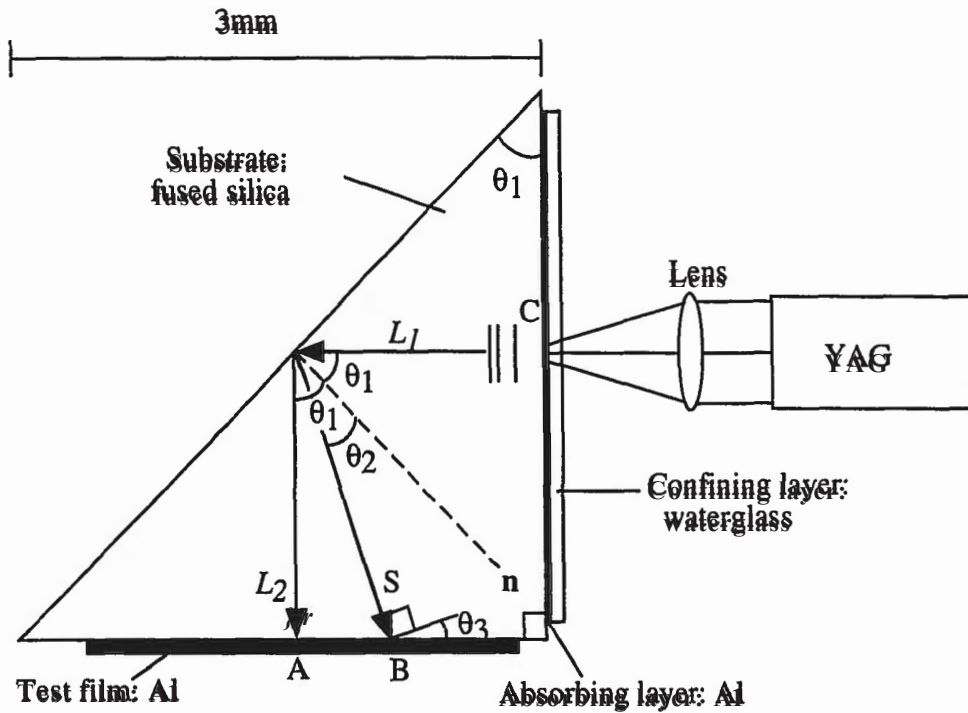


Figure 4.1: Configuration for laser generated shear waves.

the material elastic constants—is mode-converted into a shear wave, S . A smaller amount reflects as a longitudinal wave, L_2 . The two reflected waves are then incident —propagation direction depends on the precise choice of θ_1 — upon the test film surface at points B and A, respectively.

Figure 4.1 shows the angles θ_1 and θ_2 between the normal to the oblique surface, n , and the propagation directions of the longitudinal and shear waves; θ_3 is the angle between the shear wave displacement (displacement is normal to the propagation direction of the shear wave) and the test film; $\theta_3 = \theta_1 - \theta_2$. The displacement amplitudes of the incident longitudinal, reflected longitudinal and shear waves are denoted with u_{L_1} , u_{L_2} and u_S . According to standard wave propagation theory [44], the amplitude ratios are expressed as

$$\frac{u_{L_2}}{u_{L_1}} = \frac{\sin 2\theta_1 \sin 2\theta_2 - k^2 \cos^2 2\theta_2}{\sin 2\theta_1 \sin 2\theta_2 + k^2 \cos^2 2\theta_2}, \quad (4.1)$$

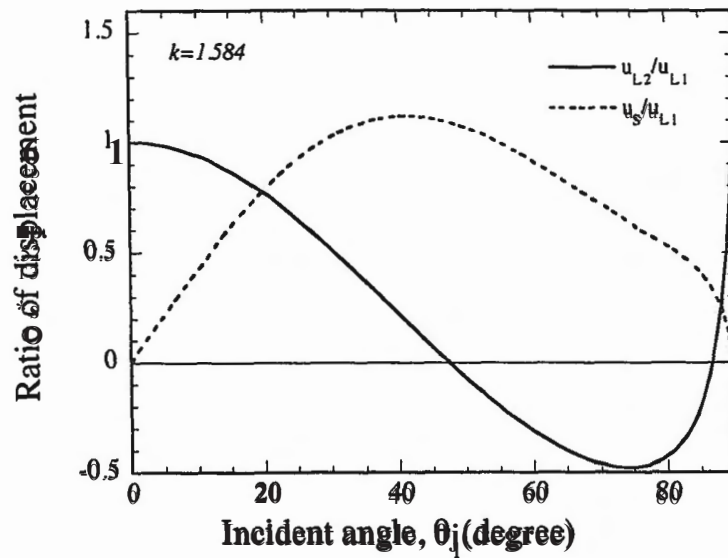


Figure 4.2: Ratio of wave displacement amplitudes as function of incident angle θ_i .

$$\frac{u_S}{u_{L1}} = \frac{2k \sin 2\theta_1 \cos 2\theta_2}{\sin 2\theta_1 \sin 2\theta_2 + k^2 \cos^2 2\theta_2}, \quad (4.2)$$

where $k = c_L/c_S$ is the ratio of longitudinal and shear wave speeds, and θ_2 is given by Snell's law

$$\frac{\sin \theta_1}{\sin \theta_2} = k. \quad (4.3)$$

Based on the parametric study of the tensile spallation, fused silica (GE type 124) is chosen as the substrate material, waterglass as the confining layer, and aluminum as both absorbing material and test film. The longitudinal wave speed for the substrate is $c_L = (5.9407 \pm 0.0004) \times 10^3$ m/s as measured on a prism using waves traveling between the parallel surfaces. A shear wave speed of $c_S = 3.75 \times 10^3$ m/s is taken from the manufacturer's reference provided by Integrated Optics Service Co., as is the density $\rho = 2.2 \times 10^3$ kg/m³. For this specific material, $k \approx 1.584$.

In Fig. 4.2, the displacement amplitude ratios (4.1) and (4.2) are plotted as a function of incident angle θ_i . At an incident angle $\theta_i = 45^\circ$, the coefficient of mode conversion from incident longitudinal wave L_1 to shear wave S is nearly at a maximum, i.e., most of the

energy of the incident longitudinal wave, L_1 , is mode-converted into the shear wave, S . Thus, for our purposes, the convenient specimen angle $\theta_1 = 45^\circ$ is also optimal for the generation of high amplitude shear waves. From Eq. (4.3), the angle of the reflected shear wave is found to be $\theta_2 = 26.5^\circ$, so $\theta_3 = 18.5^\circ$. In this configuration, the reflected longitudinal wave will be incident at point A normal to the test film surface and all of the amplitude of the longitudinal displacement will contribute to the measured out-of-plane motion at point A. The corresponding shear wave arrives at the test film surface at point B, where only one part of the shear wave displacement contributes to the out-of-plane motion. The distance between points A and B is determined by the size of the sample and specific incident position of the YAG laser. Based on the previous analytical analysis of wave attenuation w.r.t. substrate thickness, the lengths of the orthogonal sides of the triangular cross-section in Fig. 4.1 are chosen to be 3 mm. Assuming the YAG beam is incident at the center of the 3 mm wide absorbing layer surface, the distance between points A and B is found to be approximately 0.5 mm. However, for a 1 mm diameter YAG spot, the diameter of the wave front of the longitudinal wave will also be 1 mm (centered at point A) and the diameter of the wave front of the shear wave will be slightly larger, about 1.33 mm (centered at point B). Thus within region AB, the signals from the reflected longitudinal and shear waves are overlapped, although they arrive at different times.

Actually when shear wave S reaches the test film surface at point B, it will be mode-converted again into a shear wave S' and a longitudinal wave L_3 as shown in Fig. 4.3. The incident angles of waves S' and L_3 are θ_3 and θ_4 , respectively. From Snell's law,

$$\frac{\sin \theta_4}{\sin \theta_3} = k, \quad (4.4)$$

$\theta_4 \approx 30.17^\circ$. The out-of-plane and in-plane displacements at point B are denoted $u_{\perp}$ and $u_{\parallel}$, respectively. For an incident shear wave S with displacement amplitude u_S , the out-of-plane

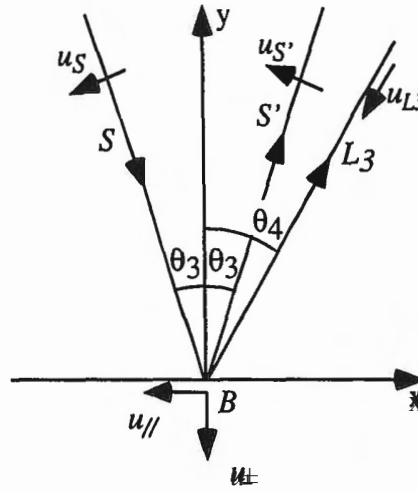


Figure 4.3: Mode conversion at point B.

displacement at point B is obtained from basic wave-propagation theory [44],

$$u_{\perp} \equiv u_s \beta, \quad (4.5)$$

where

$$\beta = \frac{k \sin 4\theta_3 \cos \theta_4 + 2 \sin 2\theta_3 \sin 2\theta_4 \sin \theta_3}{\sin 2\theta_3 \sin 2\theta_4 + k^2 \cos^2 2\theta_2}; \quad (4.6)$$

The ratio between the in-plane and out-of-plane displacement components at point B is

$$\frac{u_{\parallel}}{u_{\perp}} = \frac{k \sin 4\theta_3 \sin \theta_1 + 2k^2 \cos^2 2\theta_3 \cos \theta_3}{k \sin 4\theta_3 \cos \theta_4 + 2 \sin 2\theta_4 \sin 2\theta_3 \sin \theta_3} \equiv \gamma. \quad (4.7)$$

For the current geometry, $\beta \approx 0.778$ and $\gamma \approx 2.30$.

4.2.2 Measuring Principles

Similar to the case of tensile spallation, a Michelson interferometer is used to detect the out-of-plane motion of the test film surface. The stresses acting in the substrate and at the interface are inferred from the motion of the test film using wave mechanics. Any out-of-

plane motion of the sample surface causes a change in signal frequency due to the Doppler effect. A full fringe corresponds to half wavelength ($\lambda_0 = 257 \text{ nm}$) out-of-plane movement. By focusing the argon detection beam at a point between A and B (Fig. 4.1), a longitudinal wave, L_2 and a shear wave, S , will be detected. The normal displacement in the substrate u_{L_2} can be obtained directly from the out-of-plane interferometric measurements at point A. The shear displacement amplitude u_S can then be calculated from using the mode conversion coefficients in Eqs. (4.1) and (4.2):

$$\frac{u_S}{u_{L_2}} = \frac{2k \sin 2\theta_1 \cos 2\theta_2}{\sin 2\theta_1 \sin 2\theta_2 - k^2 \cos^2 2\theta_2}. \quad (4.8)$$

Alternatively, the value of u_S can also be obtained from the shear part of the interferometric measurement at point B. Ideally, these two methods should give the same results. Note that in the experiment, the wave displacements u_3 and u_{L_2} in the substrate are not measured directly; rather, the out-of-plane displacement of the film, u_1 (Eq. 4.7) and $2u_{L_2}$ are actually measured. Once the displacements are obtained, the corresponding stresses in the substrate and at the interface are calculated:

$$\sigma_{\text{sub}}^{L_2} = -(\rho c_L)_{\text{sub}} \frac{\partial u_{L_2}}{\partial t}, \quad (4.9)$$

$$\tau_{\text{sub}}^S = -(\rho c_S)_{\text{sub}} \frac{\partial u_S}{\partial t}, \quad (4.10)$$

$$\sigma_{\text{int}}^{L_2} = -2(\rho h)_{\text{film}} \frac{\partial^2 u_{L_2}}{\partial t^2}, \quad (4.11)$$

$$\tau_{\text{int}}^S = -(\rho h)_{\text{film}} \frac{\partial^2 u_{\parallel}}{\partial t^2} = -(\rho h)_{\text{film}} \frac{\partial^2 u_S \beta \gamma}{\partial t^2}, \quad (4.12)$$

$$\sigma_{\text{int}}^S = -(\rho h)_{\text{film}}^v \frac{\partial^2 u_{\perp}}{\partial t^2} = -(\rho h)_{\text{film}} \frac{\partial^2 u_S \beta}{\partial t^2}, \quad (4.13)$$

where h is the test film thickness, $\sigma_{\text{sub}}^{L_2}$ and τ_{sub}^S are the normal and shear stress amplitudes of the waves in the substrate, $\sigma_{\text{int}}^{L_2}$ is the interface tensile stress induced by the longitudinal wave L_2 at point A (Fig. 4.1), τ_{int}^S is the interface shear stress induced by the shear wave S at point B, and σ_{int}^S is the interface tensile stress induced by the shear wave S at point B.

(Fig. 4.1).

Equations (4.12) and (4.13) indicate that both normal and shear stresses exist at point B and thus the film interface in this region is actually under mixed-mode loading. For the current sample dimensions, however, the shear stress is much higher than the normal stress ($\tau_{int}^S/\sigma_{int}^S = u_{||}/u_{\perp} = \gamma = 2.30$). In order to obtain a pure shear-mode loading, the sample shown in Fig. 4.1 needs to be cut perpendicular to the propagation direction of shear wave S around point B (Fig. 4.4a). Alternatively, θ_y could be chosen to be greater (Fig. 4.4b). In Fig. 4.4b, $\theta_2 = \pi/2 - \theta_1$, Snell's law (Eq. 4.3) yields

$$\frac{\sin \theta_1}{\sin(\frac{\pi}{2} - \theta_1)} = k. \quad (4.14)$$

Solving for θ_1 , one obtains

$$\theta_1 = \arctan(k) = 57.7^\circ. \quad (4.15)$$

In the pure shear case, however, the Michelson interferometer will no longer be able to detect any signal at point B, the pure shear displacement at point B can be inferred by measuring the signal at point A and then using the mode conversion coefficient in Eq. (4.8) or possibly can be measured directly using an alternate interferometric technique.

4.3 Experimental Procedure

4.3.1 Experimental Setup

The Michelson interferometer used to measure the displacement on the test film is similar to that used in the tensile tests (Fig. 4.5). Two identical short-focal-distance lenses L2 and L3 are placed 20 mm away from the sample and stationary mirror in order to obtain a small focal spot ($\sim 10 \mu\text{m}$). In the tensile-spallation experiment, every point within the argon

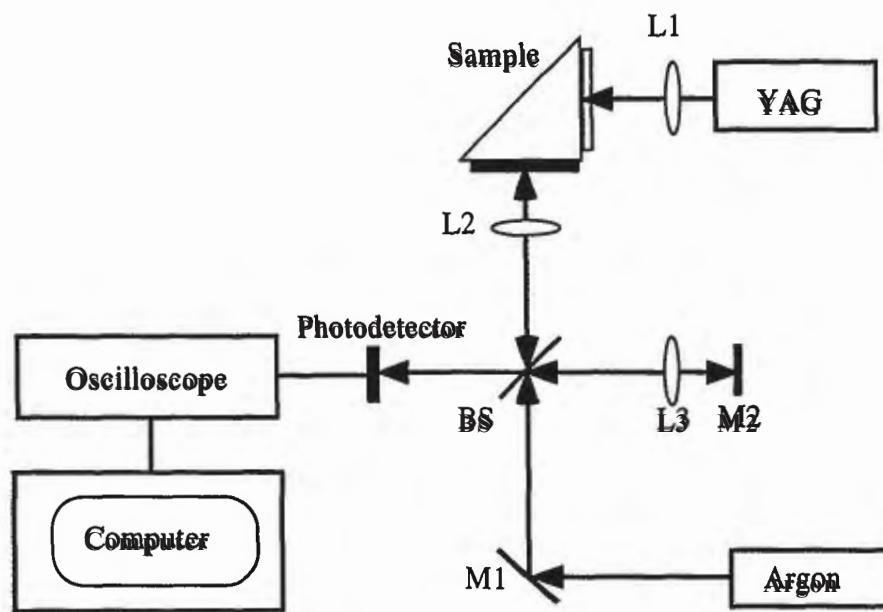


Figure 4.5: Schematic of the mixed-mode loading experiment and interferometric displacement measurement.

detection spot is in the same phase of motion; thus the spot size of the Argon beam is not critical. In the mixed-mode test, however, each point within the detection window has a different arrival time, so a smaller argon detection spot will better resolve the fastest feature in the stress wave.

When using the interferometer to measure out-of-plane displacement, the Argon beam can be focused anywhere between points A and B and the interferometer will detect both the longitudinal wave L_2 and shear waves S . The arrival time difference for the two waves depends on where the YAG is aimed, but is over 100 ns. Previous results obtained on a 3 mm thick fused silica plate sample [42] indicate the maximum stress is achieved within 30 ns after the initial wave arrival. By the time the shear wave arrives in the current experiment, the maximum of the longitudinal stress wave has already passed and the tail of that stress is almost negligible. Therefore, the important parts of the interferometric signals due to the longitudinal and shear waves are expected to be distinguishable.

4.3.2 Sample Fabrication and Alignment

A schematic of the prismatic sample used for the mixed-mode loading experiment is shown in Fig. 4.6. The length of the prism is chosen to be 15 mm based on the translation extent of the mounting stage. When preparing the sample, the aluminum test film and absorbing layer are vapor-deposited at the top and bottom sections of the two orthogonal surfaces of the prism, each with an area of 6 mm × 3 mm. In the middle section of the sample, two windows of 3 mm × 3 mm are left for the purpose of alignment. A thin layer of aluminum film is also deposited on the center portion of the oblique surface in the middle window. The vacuum rate of the evaporator is controlled at 4.0×10^{-6} Torr and the deposition rate is held between 10 and 15 Å/sec. The final thickness for the absorbing layer is controlled at 0.4 μm while the test film thickness is varied between experiments. After the aluminum is deposited, a thin waterglass layer of uniform thickness is deposited on the absorbing layer surface with a standard photolithography spinner as described in Chapter 3.

In order to hold the sample flat during the film and waterglass deposition, a silicone elastomer, PDMS (polydimethylsiloxane), is used to make individual molds for holding the prisms. Figure 4.7 shows the cross sections of two molds created for the current experiments. The mold in Fig. 4.7a is used to hold the prism when evaporating aluminum onto the central window of the oblique surface. The mold in Fig. 4.7b holds the prism while evaporating aluminum (both test film and absorbing layer) onto the top and bottom sections of the two orthogonal surfaces of the prism and also for spin casting the waterglass confining layer. High-temperature copper tape is used to mask any areas on the prism where the film is not desired.

Waterglass Deposition

The use of PDMS molds for spinning waterglass is essential. The mold holds the sample flat during the spinning and provides a good seal between the mold and the test film surface,

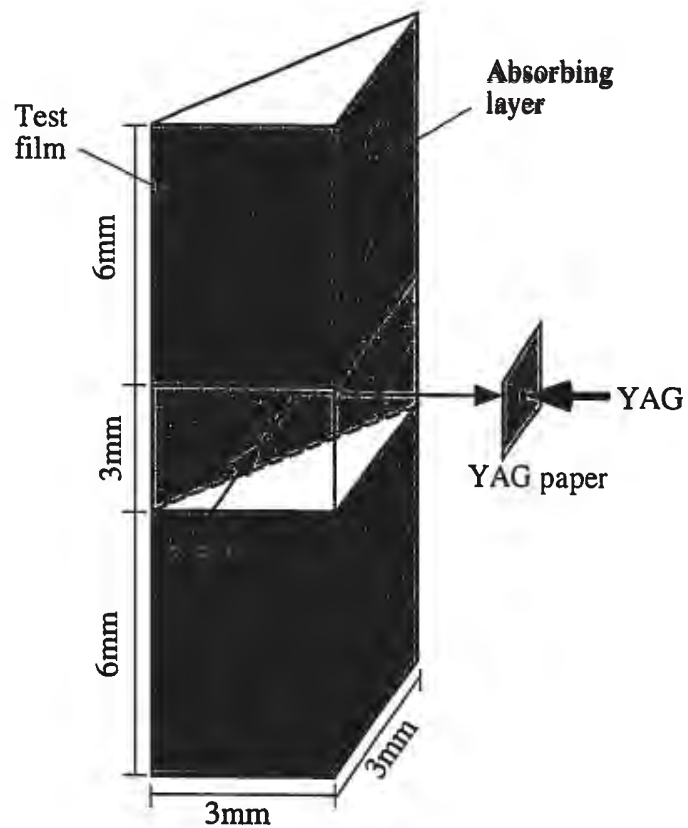
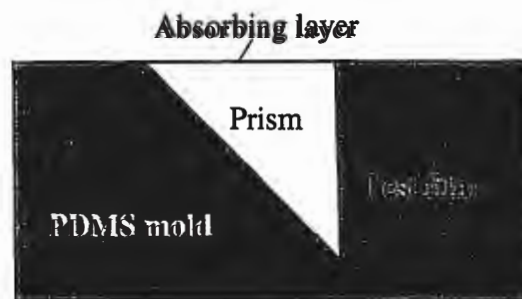


Figure 4.6: Sample schematic for mixed-mode experiment.



(a)



(b)

Figure 4.7: Configurations of the PDMS molds for aluminum film deposition and waterglass spinning.

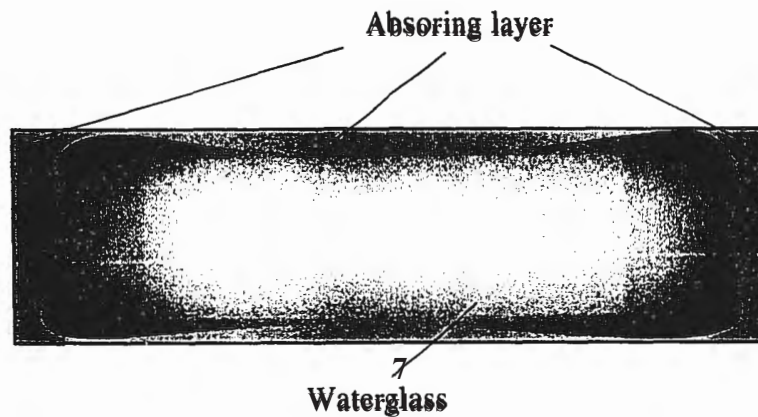


Figure 4.8: Edge effect in the waterglass spinning for the prism sample.

preventing waterglass from leaking to the test film side. The most important role of the mold, however, is to provide a large, flat area adjacent to prism surface. Spin casting always produces an edge effect, i.e., the spin cast layer is uniform in the central region, but increases in thickness near the edge. For the 1×1 inch plate specimen used in the tensile spallation experiment, the edge effect is only significant at the four corners. Most of the central area has a uniform waterglass thickness. For the triangular prism used in the mixed-mode testing, the surface area of the absorbing layer to be coated is $15 \text{ mm} \times 3 \text{ mm}$. The width of the area (3 mm) is much smaller than the length (15 mm) and the edge effect becomes a serious problem. The waterglass solution may not extend to the edge and corners of the prism as illustrated in Fig. 4.8, or the waterglass layer thickness in the coated area may not be uniform. Any variation in waterglass thickness is not desirable because it affects the consistency of the wave-generation mechanism. As a result, the same YAG power will generate a different amplitude of stress wave at different test spots. Also, excessively thick waterglass near the edge tends to delaminate the absorbing layer from the prism before testing.

In order to obtain a uniform waterglass layer over the whole absorbing layer surface, the prism sample must be placed in a mold such that the surface of the prism is perfectly flush with the surface of the mold. Moreover, the surface characteristics of the mold must be suitable for spinning. The PDMS mold easily satisfied the first requirement as shown in

Fig. 4.7. However, waterglass does not adhere to the hydrophobic PDMS mold, preventing spin coating. To solve this problem, the PDMS molds are illuminated by a UV light for 5-10 minutes. The UV illumination oxidizes any organic molecules on the PDMS surface and makes the PDMS more hydrophilic. After UV processing, the prism sample is placed into the mold (Fig. 4.7) for waterglass spinning. Finally, a uniform waterglass layer is obtained that covers the whole area of the absorbing layer surface.

Sample Alignment

For the mixed-mode configuration, alignment of the YAG and Argon laser beams is challenging. As pictured in Fig. 4.6, the Argon laser is incident normal to the center of the window on the test film surface and reflected out of the window on the absorbing layer surface. A YAG sensitive paper is then placed in front of the window on the absorbing layer surface. By carefully adjusting the position of the sample and YAG laser, the reflected Argon laser and incident YAG laser can be well aligned on the YAG paper. Because the longitudinal waves L_1 and L_2 follow the same path as the Argon laser beam, this alignment method provides a good match between the arrival position of the longitudinal wave L_2 and the Argon detection spot. In order to align the arrival position of the shear wave S and the Argon laser, one needs only to move the Argon laser horizontally by a theoretically known distance (0.5 mm for the current geometry). Since moving the Argon laser can complicate the alignment, the sample is translated instead in the opposite direction for the same distance. After the alignment is completed, the sample is moved vertically to different spots for multiple tests. The current geometry allows up to 4 tests per sample.

4.4 Experimental Results

4.4.1 Qualitative Damage Observations

Significant damage is observed in the mixed-mode loading experiment. Figure 4.9 shows two optical photographs of a $1.0\text{ }\mu\text{m}$ thick damaged film near point B. That the film has failed in shear is clear. The film damage and failure mode are significantly different than observed for the tensile film spallation in Chapter 3. In the shear case, the failed film is wrinkled and torn in sections, while under tensile loading the film was lifted off the surface in a blister like fashion (Fig. 3.6). The direction of the wrinkling in Fig. 4.9 provides additional evidence that the failure is caused by a shear wave.

Similar to the tensile case, the amount and extent of the damage depends on the applied laser power, the test film thickness, and the waterglass confining layer thicknesses. More wrinkling and tearing occur at higher laser power in thicker films with thicker waterglass layers. Figures 4.10 and 4.11 contain photographs of the damage observed on a $1.2\text{ }\mu\text{m}$ film with a $4\text{ }\mu\text{m}$ and $6\text{ }\mu\text{m}$ thick waterglass confining layer, respectively. For the same sample, more damage was observed at higher laser fluence. And for the same film thickness at the same laser fluence, more damage was observed in the samples with a thicker waterglass layer.

4.4.2 Quantitative Interferometric Measurement

Interferometric measurements were carried out on samples with an aluminum test film thicknesses between 0.5 and $1.0\text{ }\mu\text{m}$. A typical interferometric signal at point A is shown in Fig. 4.12. Based on the material properties provided earlier and the travel distance of 3 mm , the arrival of the longitudinal wave L_2 is expected at about 585 ns . The arrival of the shear wave S at point A is expected at about 710 ns . Each arrival time is referenced to the trigger time of the Q-switch of the YAG laser ($t = 0$). The interferometric signal in

$$\frac{3.0\text{mm}}{15.9\text{mm}/\mu\text{s}} + 80\text{ns delay in the YAG}$$

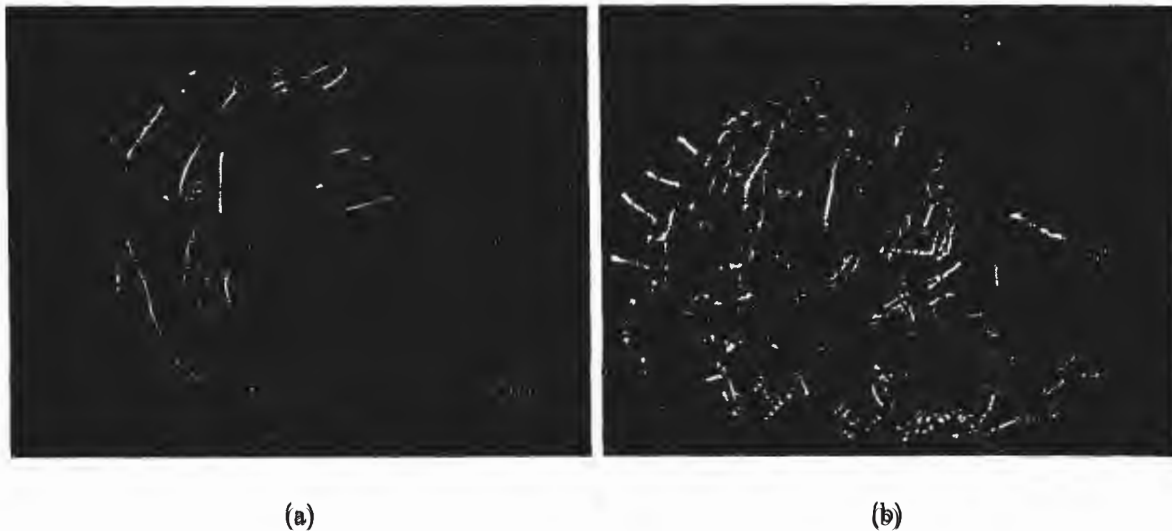


Figure 4.9: Typical failure patterns observed on a 1 μm aluminum film in the mixed-mode loading experiment: (a) at low laser power (0.086 J/mm²); (b) at high laser power (0.140 J/mm²). The horizontal direction in the microphotograph here corresponds to the direction A→B of Fig. 2. B is at the center of the damage and A is to the left.

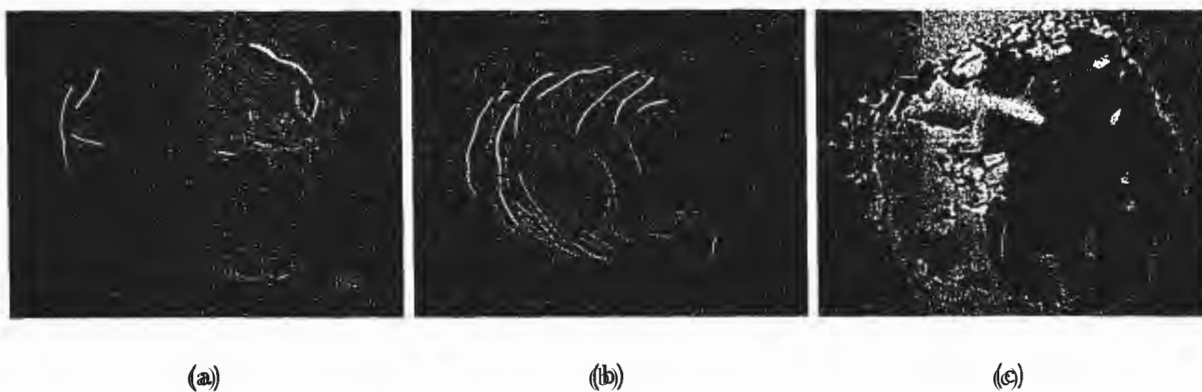


Figure 4.10: Damage patterns observed on a 1.2 μm thick aluminum film with a 4 μm thick waterglass confining layer and laser fluence of: (a) 0.084 J/mm²; (b) 0.112 J/mm²; (c) 0.140 J/mm².

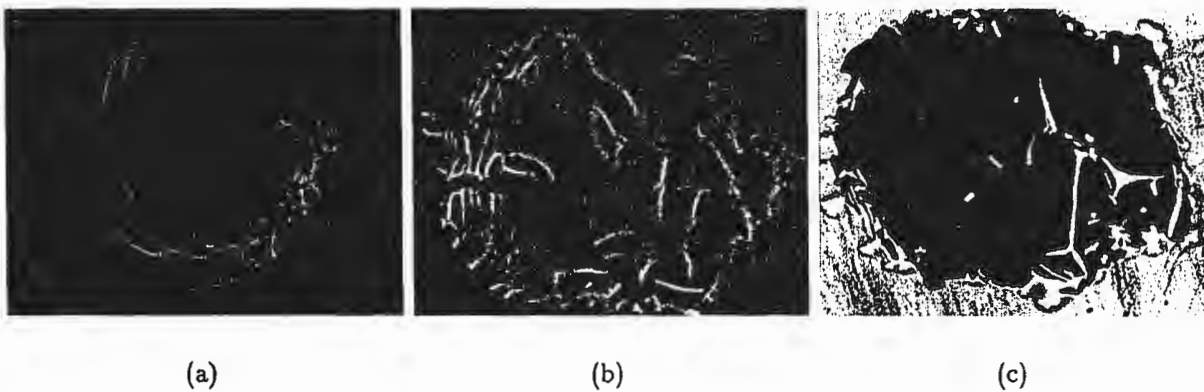


Figure 4.11: Damage patterns observed on a 1.2 μm thick aluminum film with a 6 μm thick waterglass confining layer and laser fluence of: (a) 0.084 J/mm²; (b) 0.112 J/mm²; (c) 0.140 J/mm².

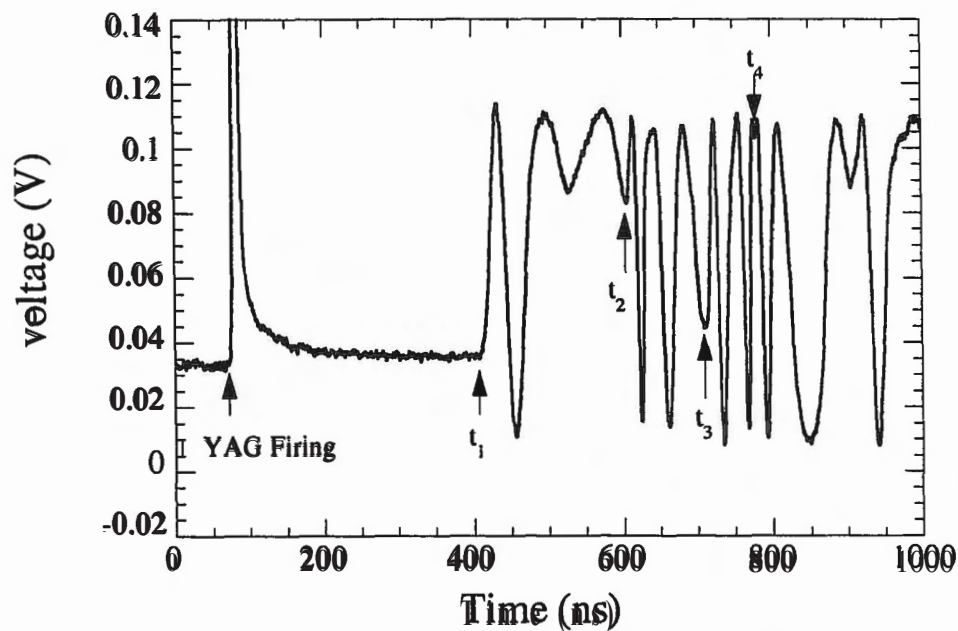


Figure 4.12: Typical interferometric signal obtained in mixed-mode loading experiment for a 1.0 μm aluminum film with 20 μm thick waterglass layer and laser fluence of 0.140 J/mm² at point A.

Fig. 4.12 shows the expected longitudinal and shear arrivals at $t_2 = 600$ ns and $t_3 = 710$ ns, respectively. A shock appears to arrive at $t_4 = 780$ ns (indicated by the sudden turning in the fringe signal), but interpreting the fringe data is difficult since point A is not the center of shear arrival and the shock arrival is much later than the expected 740 ns (30 ns after the initial shear wave arrival). The data also show the arrival of a low-amplitude stress at about $t_1 = 400$ ns, much earlier than the expected longitudinal wave L_2 . This arrival time is approximately equal to the travel time of a longitudinal wave from a point 0.25 mm below C to A in the triangular prism (Fig. 4.1).

In order to confirm the observed wave arrival times and to evaluate the effect of diagonally generated stress waves from C to A (Fig. 4.1), a reference experiment was carried out on a much larger prism with cross-sectional dimensions of 15 mm $\times$ 15 mm as shown in Fig. 4.13. The Nd:YAG laser was incident at point C* on the absorbing layer at a distance of 1.5 mm from the bottom surface and the Argon laser was focused at a point A* 1.5 mm away from the right surface (same distance as in the smaller samples). In this configuration waves

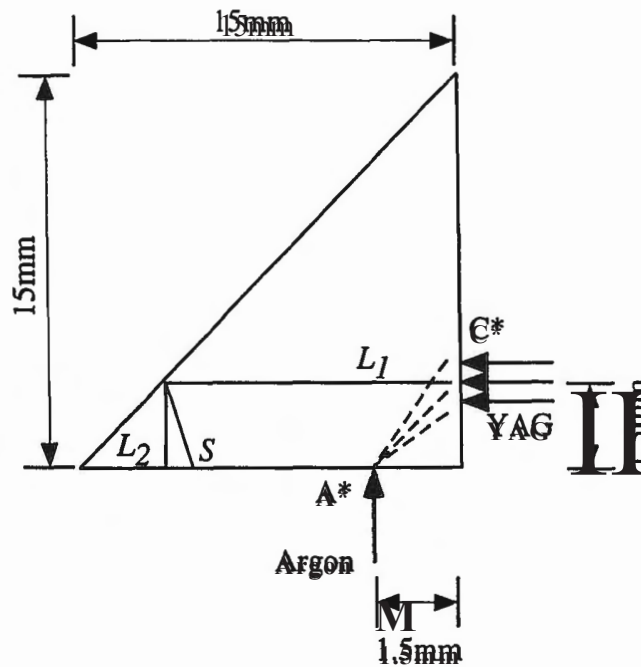


Figure 4.13: Schematic of the reference experiment for diagonal wave evaluation (laser fluence = 0.140 J/mm²).

reflected from the oblique surface only arrived at positions far to the left of point A*, and the signal detected at A* was solely due to diagonal waves propagated directly from point C*.

The interferometric signal detected from point A* is shown in Fig. 4.14. The signal shows two comparatively strong wave arrivals. As expected from a diagonally generated longitudinal wave from C* to A*, the first wave arrival is at $t_1 = 390$ ns. There is also a clear second wave arrival at $t_2 = 572$ ns. The ratio between the first wave arrival time $t_1 = 390$ ns and the second at $t_2 = 572$ ns is approximately equal to the ratio between the longitudinal and shear wave speeds c_L and c_S , i.e.,

$$\frac{t_2 - 80\text{ns}}{t_1 - 80\text{ns}} = \frac{572\text{ns} - 80\text{ns}}{390\text{ns} - 80\text{ns}} = 1.567,$$

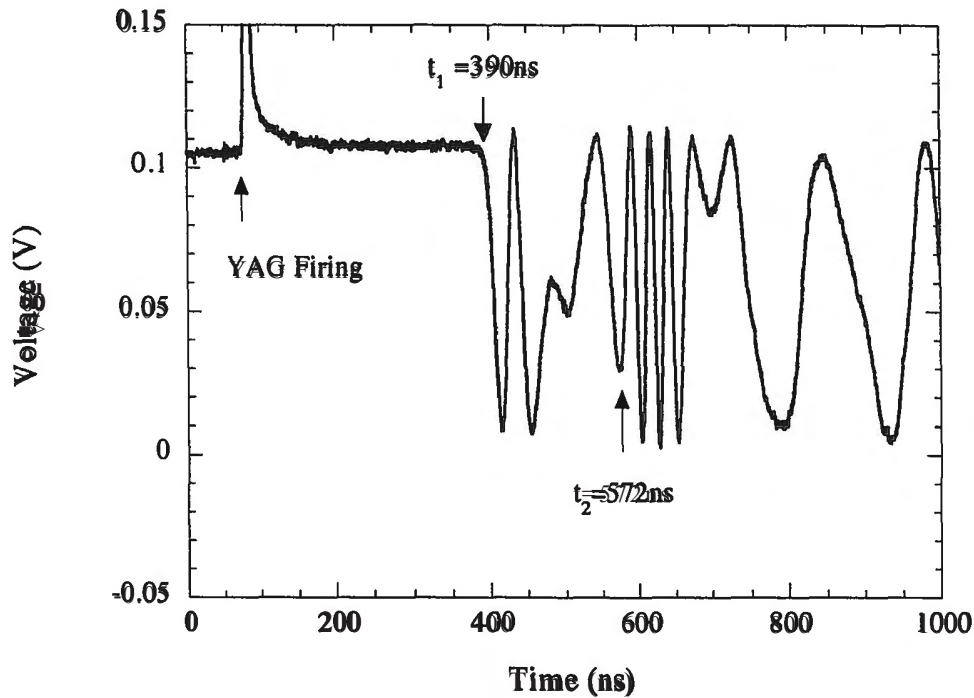


Figure 4.14: Interferometric signal detected at point A* for diagonal wave evaluation.

$$\frac{c_L}{c_S} = \frac{5.94}{3.75} = 1.584.$$

The results of this reference experiment indicate that in the mixed-mode loading experiment of Fig. 4.1, a diagonally generated longitudinal wave will arrive before L_1 at approximately $t_1 = 390$ ns and a diagonally generated shear wave will arrive at $t_2 = 572$ ns (the same time as L_2). The additional waves are possibly due to surface roughness of the confining layer at point C (Fig. 4.1), or sharp edges in the YAG beam. While the stress caused by the diagonal longitudinal wave is clearly quite low, the diagonal shear wave is much stronger than the L_2 wave. Since these two waves are overlapped, extracting information from the longitudinal wave L_2 signal is not reliable. Figure 4.14 also shows that the diagonal waves become weak after 700ns (indicated by the wide spacing of the interferometric fringes). Therefore, the shear part of the interferometric signal in the 3 mm prism can still be used to calculate the displacement of the shear wave.

To quantify better the interfacial stress at film failure, the Argon detection beam was

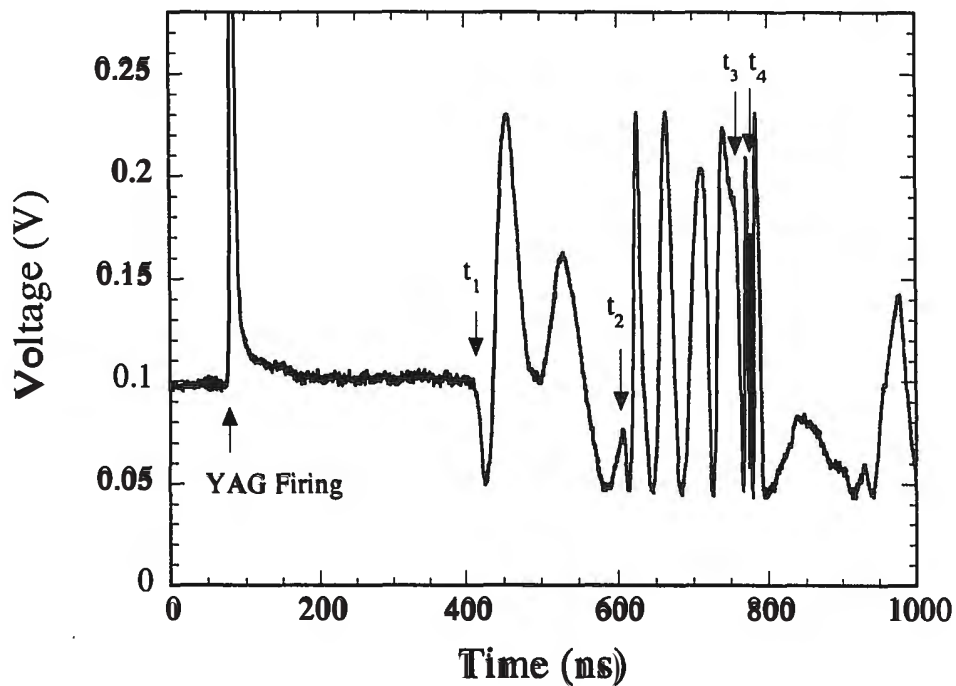
focused at point B (Fig. 4.1), the center of shear wave arrival. Typical interferometric data obtained at point B are shown in Fig. 4.15. Based on the geometry of the sample and the wave speeds in fused silica, the shear wave arrival at point B is expected at 754 ns after firing the Q switch of the YAG laser.

As expected, the diagonally generated longitudinal and shear waves arrive at $t_1 = 408$ ns and $t_2 = 607$ ns, respectively. The shear wave S arrives at $t_3 = 757$ ns, at the expected time. A shock is developed 21 ns later, indicated by the tightening of the fringe spacing and a sudden turning of fringe data at $t_4 = 778$ ns. This fringe pattern is similar to that observed for shocks in the tensile experiment (Fig. 3.15a). After mode-converting at the oblique surface, the stress wave has retained the characteristics of the shock developed in the initial longitudinal propagation. The development of the shock is due to the nonlinear elasticity of the fused silica substrate and its generation mechanism was extensively discussed in Section 2.4.

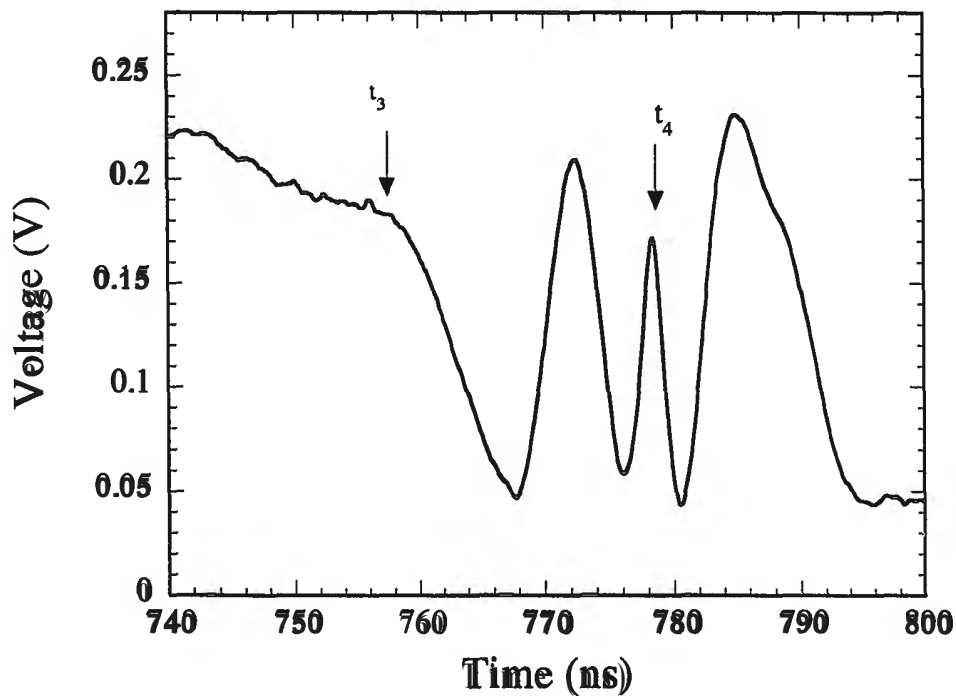
Waterglass Thickness Optimization

As was the case in the tensile-spallation experiment, there is an optimal thickness for the waterglass confining layer in the mixed-mode loading experiments. When the waterglass layer is too thick, a very high amplitude stress wave is generated, resulting in significant damage to the test film. However, excessive damage of the test film leads to inaccuracy in the interferometric measurement. When the waterglass layer is too thin, the generated stress wave is weak and no film damage can be induced. In order to capture the interface stress just prior to film failure, the waterglass layer thickness must be optimized.

In the mixed-mode experiment, the travel distance of the longitudinal waves L_1 , L_2 , and the shear wave S are all about 1.5 mm. Wave L_1 has the highest stress amplitude. It is expected that the shock will develop in the L_1 wave and any significant shock attenuation only occurs in the L_1 wave, i.e., over the first 1.5 mm. Once it is mode-converted at the oblique surface, shear wave S should retain the features of the shock. Only geometric attenuation



(a)



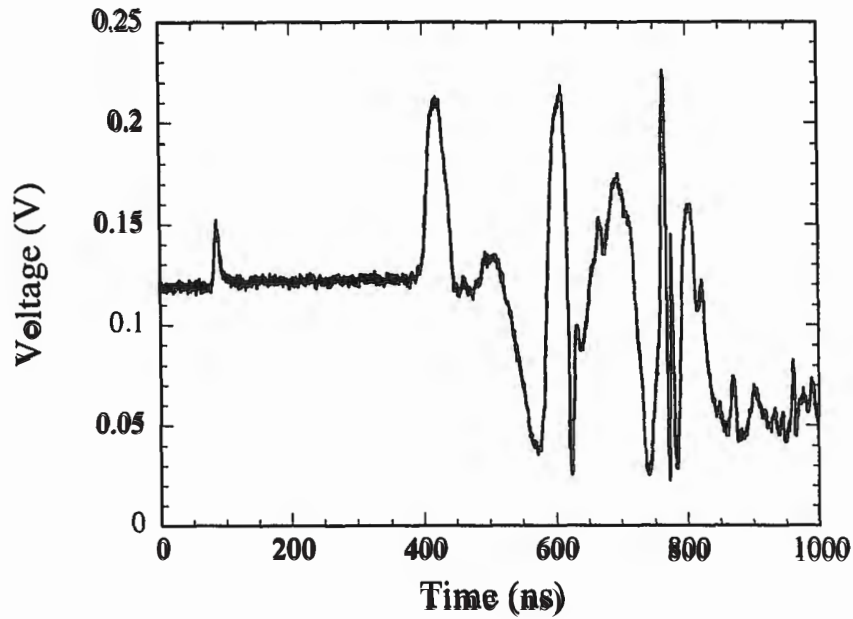
(b)

Figure 4.15: (a) Typical interferometric signal obtained for a 0.57 μm thick aluminum film with 4 μm thick waterglass (laser fluence of 0.140 J/mm²) at point B; (b) expanded view detailing the shear wave portion of the signal in (a).

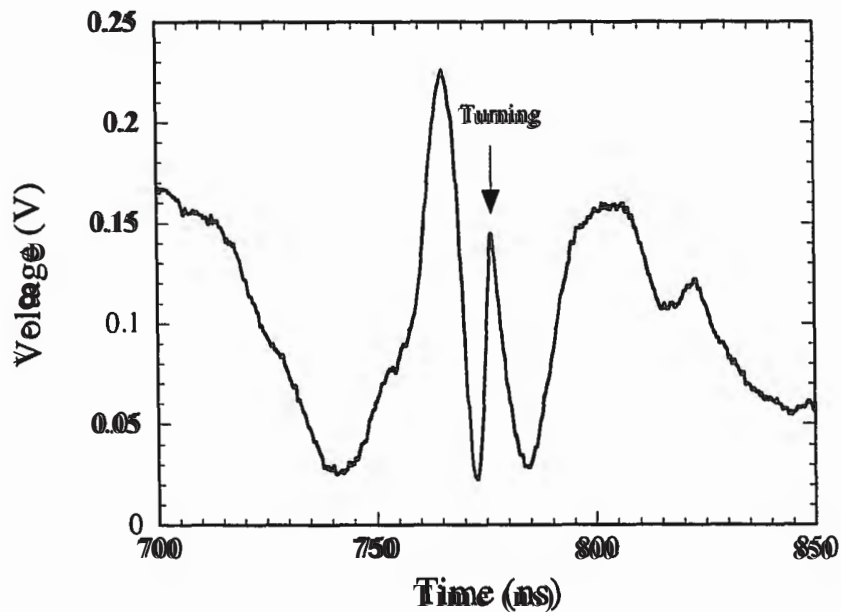
is expected in the second 1.5 mm travel distance. Based on the parametric study in the tensile case (Section 3.4.4), the required waterglass thickness should be somewhere between 0.4 μm (optimal thickness for the 1.5 mm thick fused silica substrates) and 8 μm (near optimal thickness for the 3.0 mm thick fused silica substrates). Three different waterglass thicknesses—2 μm , 4 μm , and 6 μm —were studied. The interferometric signal obtained at point B for a 0.5 μm thick aluminum film for all three waterglass layer thicknesses are shown in Figs. 4.16, 4.17 and 4.18, respectively. The measurements were all obtained at full YAG laser power.

A clear shock was detected in the samples with a 2 μm and 4 μm thick waterglass confining layer, but little or no film damage was observed. Although no shock was detected in the fringe data for the sample with a 6 μm thick waterglass layer (no turning point), the test film was significantly damaged. Much higher substrate stress was generated with increasing waterglass thickness. The maximum shear stress generated in the 2 μm waterglass sample was about 0.27 GPa and the maximum shear stress generated in the 4 μm thick waterglass samples was about 0.42 GPa. Interferometric data are difficult to interpret in the 6 μm thick waterglass sample due to extensive film damage. For accurate characterization of the interface strength at film failure, interferometric measurements should be carried out right at the initiation of film failure and the shock must be accurately detected. Under the present experimental conditions, the 4 μm thick waterglass layer was determined to be optimal for the mixed-mode case.

Additional experiments were carried out for two different film thicknesses, 0.57 μm and 1.0 μm , both with waterglass thickness of 4 μm . All tests were performed at full YAG power. The interferometric signal for the 0.57 μm thick aluminum film is shown in Fig. 4.15. From several measurements obtained at point B (Fig. 4.1), the average maximum shear stress in the substrate was 0.49 ± 0.07 GPa at full YAG power. For this film thickness, little or no failure was observed. The average maximum interfacial shear stress at point B was 413 ± 27 MPa and the maximum interfacial tensile stress at point B was 94 ± 8 MPa. For the

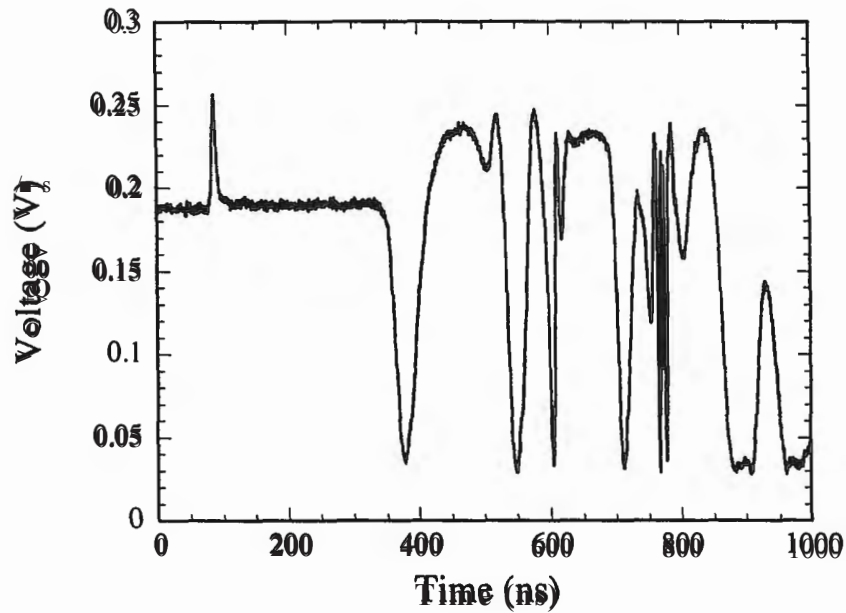


(a)

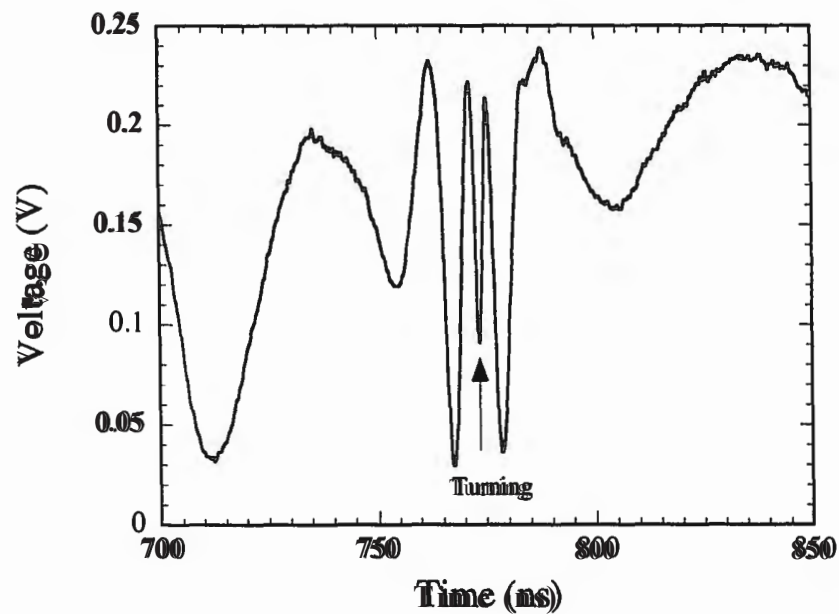


(b)

Figure 4.16: (a) Interferometric signal obtained at point B for a 0.5 μm thick aluminum film with 2 μm thick waterglass confining layer (laser fluence of 0.140 J/mm²); (b) expanded view detailing the shear wave portion of the signal in (a). Arrow points to the turning of the fringe, which indicates a shock.

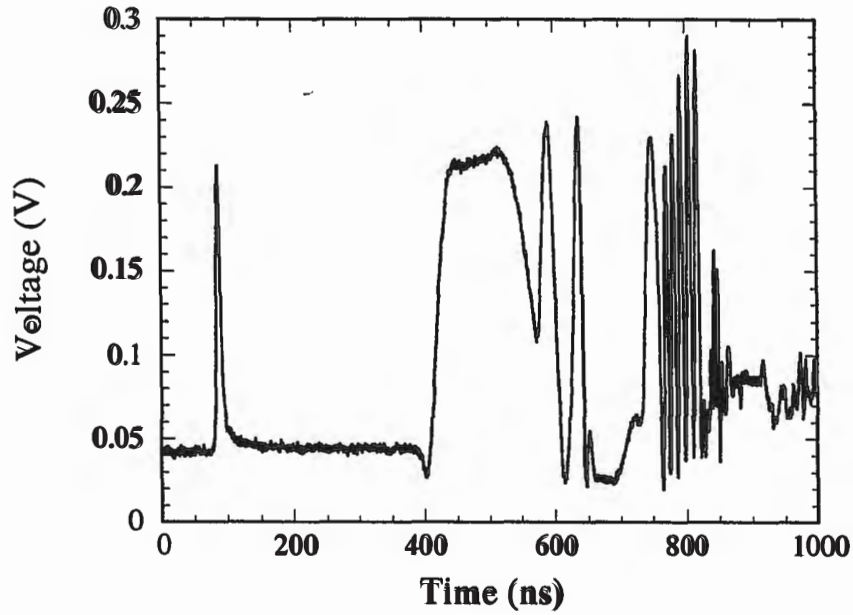


(a)

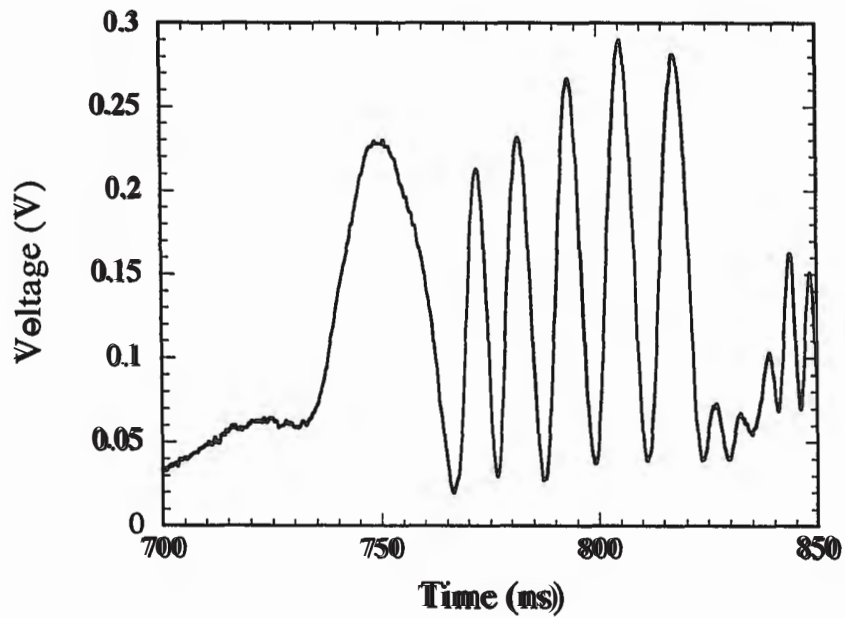


(b)

Figure 4.17: (a) Interferometric signal obtained at point B for a $0.5 \mu\text{m}$ thick aluminum film with $4 \mu\text{m}$ thick waterglass confining layer (laser fluence of 0.140 J/mm^2); (b) expanded view detailing the shear wave portion of the signal in (a). Arrow points to the turning of the fringe, which indicates a shock.



(a)



(b)

Figure 4.18: (a) Interferometric signal obtained at point B for a $0.5 \mu\text{m}$ thick aluminum film with $6 \mu\text{m}$ thick waterglass confining layer (laser fluence of $0.140 \text{ J}/\text{mm}^2$); (b) expanded view detailing the shear wave portion of the signal in (a).

1.0 μm test film at full YAG power, significant damage occurred to the film, but no shock was detected in the interferometric data. The extensive film damage once again made calculation of the interfacial stress difficult.

4.5 Shear Strength of Aluminum/Fused Silica Interface

Given the power limitation of the YAG laser and the difficulty interpreting interferometric data for thicker films, one needs to carefully choose the test film thickness to accurately measure the stress at or just prior to failure. Based on the test results of the previous section, it is estimated that the film thickness for capturing the failure initiation is between 0.57 and 1.0 μm and the laser power required to initiate failure will vary with film thickness. A series of experiments were performed on prisms with test film thickness ranging from 0.57–1.0 μm , absorbing layer thickness of 0.4 μm , and waterglass confining layer thickness of 4 μm .

In order to determine the aluminum/fused silica interface shear strength, experiments were carried out in two steps. In the first step, 9 prisms were fabricated with 3 different test film thicknesses, 1.0 μm (4 samples), 0.8 μm (3 samples), and 0.68 (2 samples). A summary of the laser power required to initiate film failure is provided in Table 4.1. As the film thickness decreases, greater YAG laser power is required to initiate the failure.

The four samples with 1.0 μm thick aluminum test films were tested at four different YAG laser powers, 60%, 80%, 90%, and 100% (corresponding to laser fluences at 0.084, 0.112, 0.126, and 0.140 J/mm²). Three samples failed at laser powers 80% and greater, and one failed at all four laser powers. The one that failed at lower power had poor film quality from evaporation and made this test less reliable. For two of the sample failures at 80% and above, no shock was observed in the fringe data due to extensive film damage, prohibiting accurate calculation of the interface stress. However, a clear shock was detected at 60%

laser power. The measured maximum interface stress at this laser power was 438 MPa. On one sample that failed at 80%, although a small amount of film delamination was initiated, a shock was still detected. The maximum interface stress from this measurement was 537 MPa.

Three samples with a film thickness of $0.8\ \mu\text{m}$ were tested at YAG laser powers of 70%, 80%, 90%, and 100%. Two samples failed at laser powers 80% and greater, and one failed at laser power of 90% and greater. No shock was detected at failure, but clear shocks were detected at 70% laser power just prior to film failure. The measured maximum interface stress prior to film failure was 475 MPa.

The two prisms with a $0.68\ \mu\text{m}$ film thickness were tested at 70%, 80%, 90%, and 100% YAG powers. Both films failed at laser powers 90% and greater. Again, no good data were obtained at failure, but the measured maximum interface stress before film failure (80% laser power) was 460 MPa.

A final prism was fabricated with a $1.2\ \mu\text{m}$ film and tested at four laser powers 60%, 80%, 90% and 100%. Significant film damage occurred at all four powers. None of the interferometric measurements were suitable for interpreting the interface strength due to extensive film damage.

For the 1.0 , 0.8 and $0.68\ \mu\text{m}$ thick aluminum film samples, the maximum interface stresses measured at laser powers just below the level required to cause failure are listed in the first two columns of Table 4.2 together with the corresponding power level. Due to film damage at failure, only one data point was obtained for the interface stress at film failure, which is listed in the first two columns of Table 4.3.

Given the limitation on laser power with the 110 mJ Nd:YAG laser used in the present experiments, it was difficult to obtain data just at failure. Rather than testing more prisms as described above, a different approach was adopted to estimate the stress at failure. In this second stage of experiments, eight prism samples were fabricated with a very thin test film thickness of $0.2\ \mu\text{m}$. This film thickness guarantees no film damage will occur and the shock

Table 4.1: Laser power required to initiate failure for different film thicknesses.

Film thickness	Sample No.			
	1	2	3	4
1.0 μm	80%	80%	80%	60%*
0.8 μm	90%	80%	80%	
0.68 μm	90%	90%		

* problematic data

Table 4.2: Interface shear stress and laser power prior to film failure initiation.

Film thickness	Measured		Estimated	
	τ_{int}^S (MPa)	Laser power	τ_{int}^S (GPa)	Laser power
1.0 μm	438	60%	485	60%
	373	60%		
	410	60%		
0.8 μm	475	80%	417	70%
	427	70%		
	370	70%		
0.68 μm	377	80%	382	80%
	460	80%		

Table 4.3: Interface shear strength and laser power at film failure initiation.

Film thickness	Measured		Estimated	
	τ_{int}^S (MPa)	Laser power	τ_{int}^S (MPa)	Laser power
1.0 μm	537	80%	562	80%
	N/A	80%		
	N/A	80%		
0.8 μm	N/A	90%	450	80%
	N/A	80%		
	N/A	80%		
0.68 μm	N/A	90%	490	90%
	N/A	90%		

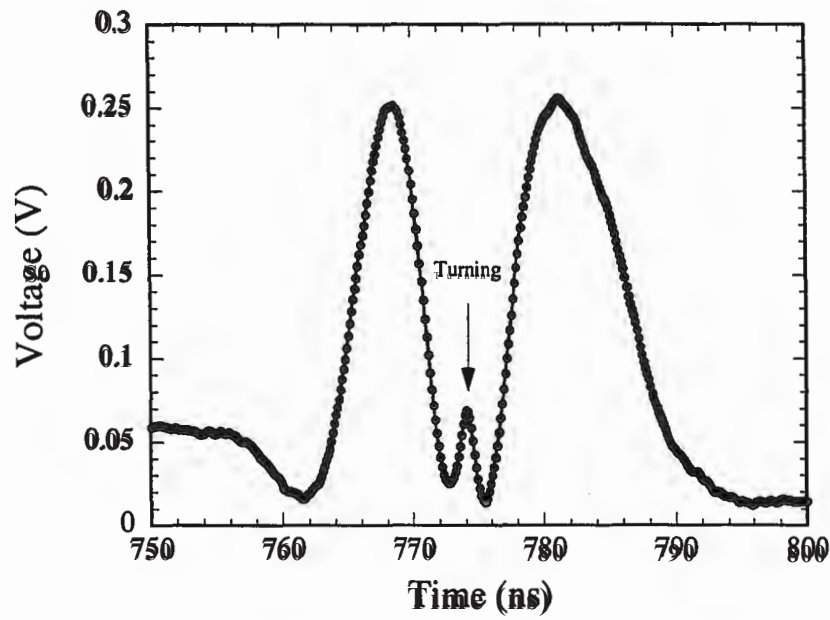
Table 4.4: Correlation of maximum substrate/interface stress and applied laser fluence.

Sample No.	Fluence: 0.084 J/mm ²		Fluence: 0.112 J/mm ²		Fluence: 0.140 J/mm ²	
	τ_{sub}^S (GPa)	τ_{int}^S (MPa)	τ_{sub}^S (GPa)	τ_{int}^S (MPa)	τ_{sub}^S (GPa)	τ_{int}^S (MPa)
1	-0.36	128	-0.35	144	-0.44	212
2	-0.31	83	-0.36	109	-0.46	129
3	-0.37	*	-0.44	108	-0.51	175
4	-0.45	85	-0.37	98	-0.45	122
5	-0.33	81	-0.40	145	-0.52	185
6	-0.41	94	-0.48	116	-0.54	221
7	-0.28	84	-0.33	85	-0.38	128
8	-0.37	116	-0.39	100	-0.48	219
Average	-0.35±0.04	95±18	-0.39±0.05	112±21	-0.47±0.05	176±40
Top-3-Ave	-0.39±0.02	113±17	-0.44±0.04	135±16	-0.52±0.02	217±5

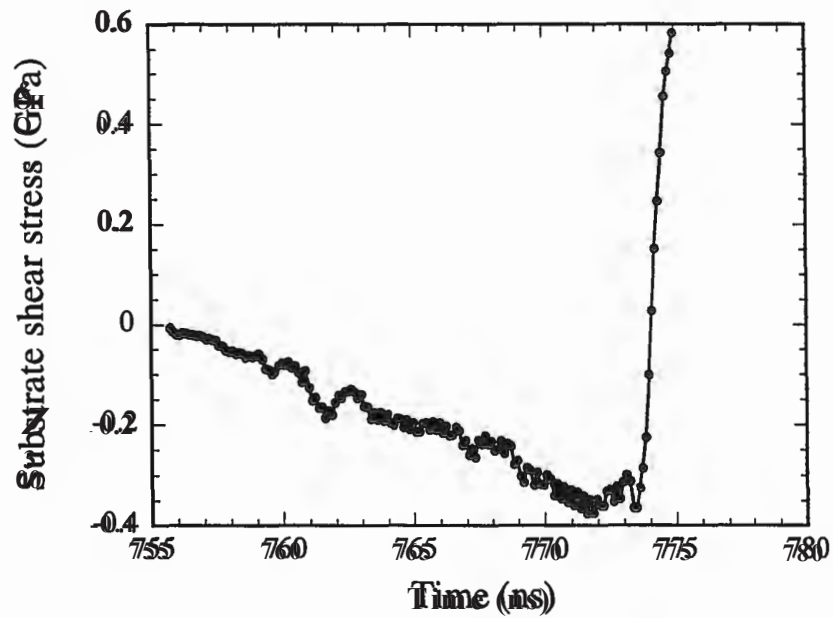
* problematic data is deleted due to uncertainty in fringe data.

will be accurately detected. Thus, these data can be used to calibrate the stress at different laser powers. On each sample, 3-4 laser powers were applied at different test spots. A typical interferometric signal obtained for one of the 0.2 μ m films at point B (laser fluence of 0.140 J/mm²) and the corresponding substrate shear stress are shown in Fig. 4.19. Through these experiments, a correlation between the maximum substrate/interface shear stress and the YAG laser power is established. The measured maximum substrate and interface shear stress at three laser fluences, 0.084 J/mm², 0.112 J/mm², and 0.140 J/mm² are summarized in Table 4.4. The substrate and interface stress are also plotted in Fig. 4.20 for each laser fluence with error bars for each individual data point.

Overall, higher substrate and interface stress were obtained at higher laser fluence. However, there is some variation from sample to sample for each laser fluence. These variations may be due to delamination of the absorbing and waterglass layer before testing, inaccuracy in aligning the Argon detection beam or variation in the wave generation mechanism at high laser power. In some samples, the waterglass layer starts to delaminate prior to testing, which results in lower values of the measured substrate and interface stress. Although extreme care is given to the alignment of the Argon detection and YAG laser beam, some inaccuracy such

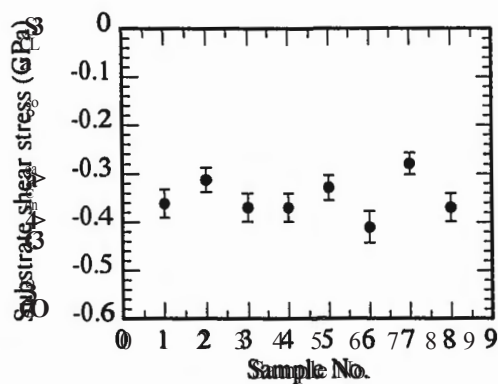


(a)

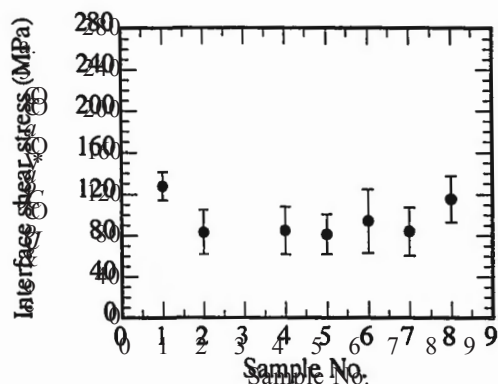


(b)

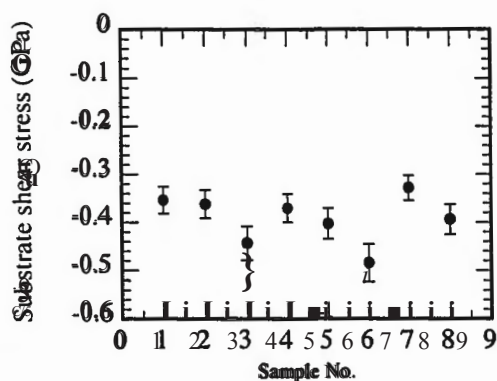
Figure 4.19: Typical interferometric data obtained on a $0.2 \mu\text{m}$ film at laser fluence of 0.140 J/mm^2 : (a) fringe data; (b) substrate shear stress.



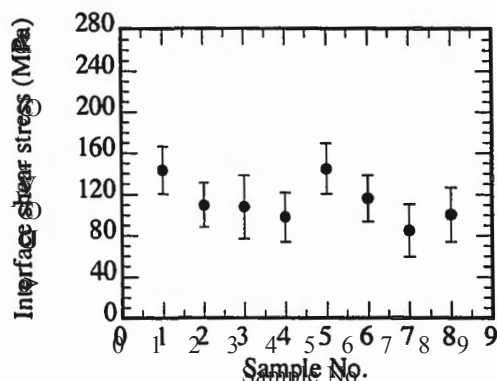
(a)



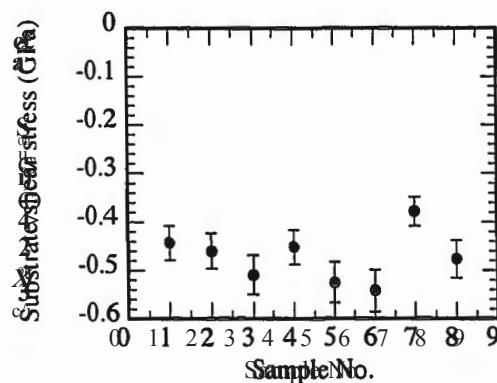
(b)



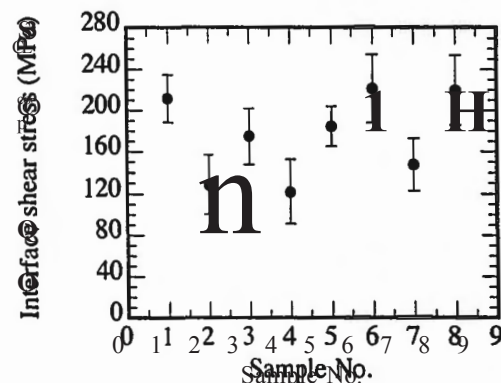
(c)



(d)



(e)



(f)

Figure 4-20: Maximum substrate and interface shear stress on prism samples with 0.2 μm thick aluminum film, 0.04 μm thick absorbing layer and 44 μm thick water glass layer: (a,b) laser fluence at 0.084 J/mm²; (c,d) laser fluence at 0.112 J/mm²; (e,f) laser fluence at 0.140 J/mm²

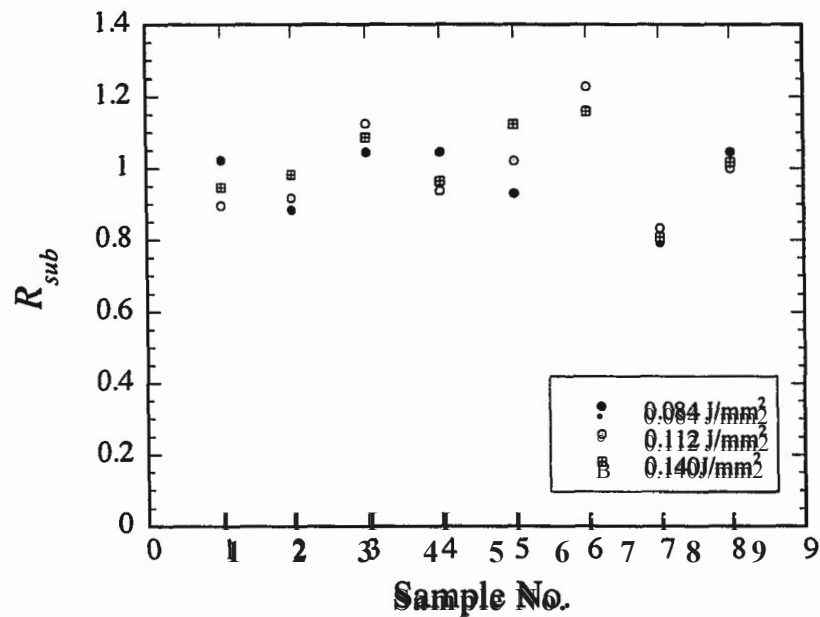
as sample tilting in the vertical plane is sometimes unavoidable. The laser beams are only aligned in the window located in the center section of the prism sample (Fig. 4.6). After the alignment is complete, the sample is translated vertically to each different test spot. Thus, any tilting of the sample in the vertical plane moves the Argon detection beam off the center of the shear wave arrival position. If all the other conditions are optimal, the signal detected at the center of the shear arrival should yield the highest amplitude of stress. Assuming the absorbing layer did not delaminate before testing, the higher values listed in Table 4.4 are more likely to reflect the highest stress acting on the interface.

In order to evaluate the overall variation between the measurements performed on different samples, two new variables R_{sub} and R_{int} are introduced as the ratios of each individual substrate and interface stress over the average substrate and interface stress,

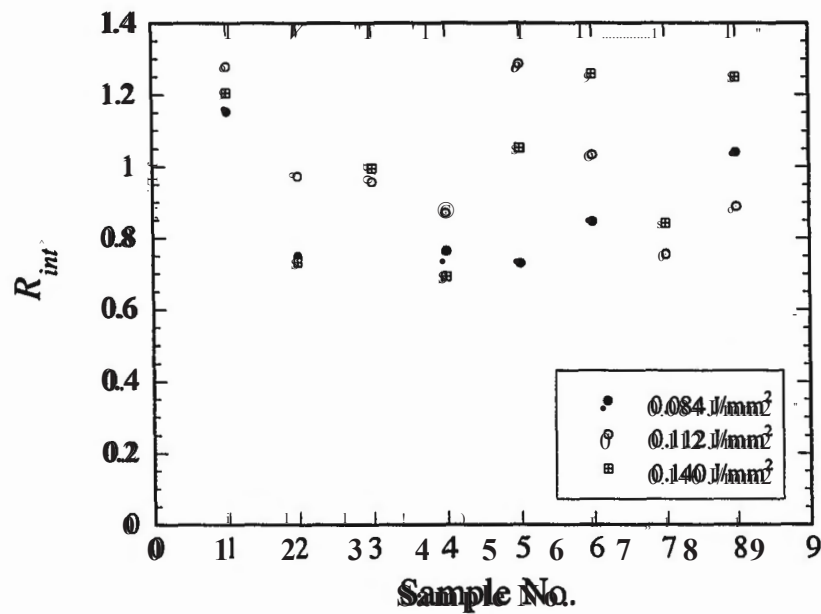
$$R_{sub} = \frac{\tau_{sub}^S}{\bar{\tau}_{sub}^S}, \quad R_{int} = \frac{\tau_{int}^S}{\bar{\tau}_{int}^S}; \quad (4.16)$$

where τ_{sub}^S is the substrate stress for each individual measurement, $\bar{\tau}_{sub}^S$ is the overall average substrate stress at each laser fluence, τ_{int}^S is the interface stress for each individual measurement, and $\bar{\tau}_{int}^S$ is the overall average interface stress at each laser fluence. In Fig. 4.21, R_{sub} and R_{int} are plotted for each measurement at the three different laser fluences. The deviation of the substrate stress from the average value is within the range of $\pm 20\%$. The deviation of the interface stress from the averaged value is $\pm 30\%$.

Following the calibration experiments, both the interface stress prior to failure and at failure for the previous three film thicknesses 1.0 μm , 0.8 μm and 0.68 μm were estimated using the data in Table 4.4 and the power levels at or just below those listed in Table 4.1. The estimated interface stress was obtained by multiplying the interface stress in Table 4.4 at the corresponding laser fluence just prior to or at failure by the ratio of the actual thickness to 0.2 μm (the film thickness used in the calibration experiments). The estimated values for interface stress are summarized in the right hand two columns of Tables 4.2 and 4.3. From



(a)



(b)

Figure 4.21: Variations in data between samples: (a) R_{sub} ; (b) R_{int} .

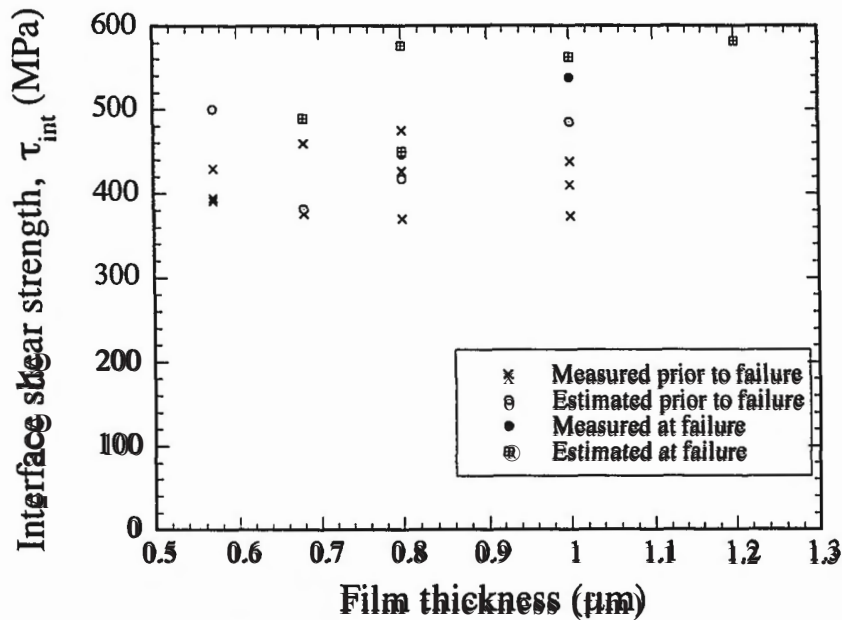


Figure 4.22: Interface stress prior to and at failure initiation.

Table 4.4, the highest interface stress measured prior to film failure was 475 MPa while the highest interface stress estimated was 485 MPa, very close to the measured value. The only measured data point available for interface stress at failure was 537 MPa. As listed in Table 4.3, the estimated lowest interface stress is 450 MPa. Averaging all the measured and estimated values in Table 4.3, it appears that the interface shear strength of aluminum/fused silica substrate is approximately 523 ± 52 MPa. The interface stress just prior to and at film failure are also plotted in Fig. 4.22 for all available measurements.

4.6 Discussion and Conclusions

In this thesis, a new test method was developed for studying mixed-mode interfacial failure of thin films. According to the wave propagation and reflection theory, a high amplitude shear wave was generated by allowing the initial longitudinal wave to mode convert at an oblique surface. After optimizing the thickness of the waterglass confining layer, a shear wave with large enough stress to fail an aluminum film/fused silica interface was obtained. The resulting

shear stress was calculated from high-speed interferometric displacement measurements. The interferometric fringe data revealed the development of a shock due to nonlinear softening of the fused silica substrate. After mode converting at the oblique surface, the stress wave retained features of the shock, similar to those observed in previous tensile experiments. The interfacial strength in shear was estimated at 523 ± 90 MPa, slightly higher than that required to cause failure under tensile loading, which was about 490 ± 70 MPa, i.e., the same interface has higher strength under mixed-mode loading. This observation is consistent with the quasi-static mixed-mode experiments on bi-layer samples [59, 68], in which interfacial fracture energy has been found to increase with increasing mode II component.

The failure patterns developed under mixed-mode loading (mostly shear mode) are different from those observed in the tensile-spallation experiment. Significant wrinkling and tearing occurs in the films due to the shear loading. The current film failures also differ from other mechanisms reported in literature, such as film cracking due to residual tension and blister-like buckle-driven delamination due to residual compression. These results indicate that mode dependence of interfacial toughness plays a critical role in determining the sequence of events leading to film failure. The use of laser generated shear waves provides a new tool to explore a range of thin-film interfacial failure modes as well as the role of such extrinsic effects as residual stress, surface roughness, and imperfections.

Chapter 5

Concluding Remarks and Future Work

5.1 Concluding Remarks

The goal of this dissertation was to develop a new technique to characterize the intrinsic interfacial strength of thin films under mixed-mode conditions using laser-generated stress waves.

In the theoretical analysis, the wave-generation mechanism following both a delta pulse and Gaussian laser pulse was analyzed. The plane wave regime was determined for a finite YAG beam size with a temporal Gaussian distribution. For a fused silica substrate, which has negative nonlinear elasticity, the mechanism for shock development and subsequent attenuation were thoroughly studied. The theoretical analysis provided meaningful insights into the nature of the wave generation and propagation in the laser spallation experiments.

The experimental parametric study on the tensile laser spallation was essential for the successful extension of this technique to mixed-mode loading experiments. The spallation behavior of aluminum thin films and the corresponding interferometric measurements were investigated as a function of film thickness, substrate thickness, confining layer thickness, laser fluence, and various parameters governing the source. Two different substrate materials, single crystal silicon (100) and fused silica, were considered. For both fused silica and silicon

substrate samples, the maximum stress in the substrate and the maximum stress at the interface increased with increasing YAG laser fluence and increasing confining layer thickness. The maximum interface stress also increased with increasing film thickness. Hence, thicker films led to higher interface stress and failed more easily. For both types of sample, the maximum substrate stress and maximum interface stress decreased with increasing substrate thickness due to geometric attenuation caused by the finite size of the YAG beam. In the case of the fused silica substrate, there was also attenuation due to the shock evolution. A thicker confining layer helped to generate a higher compressive stress in the substrate and greater damage on the test film. Several factors, including YAG spot size, film thickness, substrate thickness, and confining layer thickness, needed to be coordinated to obtain film failure.

Observations of damage revealed both substrate and film failure in silicon substrate samples but only film failure in fused silica substrate samples. Interferometric measurements confirmed the difference in stress development for the two kinds of sample. In fused-silica-substrates, the compressive pulse evolved into a decompression shock. The existence of a shock created a large stress at the film interface, promoting failure. The development of the shock was due to the nonlinear elastic properties of fused silica. This shock mechanism was advantageous for failing thin-film interfaces on thick substrates. The experimental parametric study revealed important design variables for improving the tensile spallation test and developing the mixed-mode loading experiments.

Based on the results of the tensile parametric study, a new technique for mixed-mode loading of thin-film interfaces was successfully developed. The planar geometry of the tensile specimen was modified to a triangular prism so that a high-strain-rate shear wave was generated (by mode conversion at an oblique surface) and then allowed to impinge on the test film. By allowing the initial longitudinal wave to mode-convert to a shear wave and a secondary longitudinal wave at an oblique surface, mixed-mode loading of thin film interfaces was realized. After optimizing the thickness of the watertight confining layer, a shear wave

with large enough stress to fail an aluminum film/fused silica interface was obtained. The resulting shear stress was calculated from high-speed interferometric displacement measurements. The interferometric fringe data revealed the development of a shock due to nonlinear softening of the fused silica substrate. After mode converting at the oblique surface, the stress wave retained features of the shock, similar to those observed in previous tensile experiments. Quantitative data revealed that the mixed-mode interfacial strength was larger than tensile values for the same interface.

The failure patterns observed under mixed-mode loading (mostly shear mode) are different from those observed in the tensile spallation experiment. Significant wrinkling and tearing occurs in the films due to the shear loading. These results indicate that mode dependence of interfacial toughness plays a critical role in determining the sequence of events leading to film failure. The use of laser-generated shear waves provides a new tool to explore a range of thin-film interfacial failure modes as well as the role of such extrinsic effects as residual stress, surface roughness, and imperfections.

5.2 Future Work

Analysis of the experimental data in this dissertation depends on accurate interpretation of the interferometric data. For a standard Michelson interferometer, each fringe corresponds to a change in normal displacement given by Eq. (3.3). However, special care must be taken to account for the sudden turning in the fringe data. The turning point occurs when the acceleration changes sign, indicated by the abrupt termination of one fringe and the beginning of a new one. The turning point becomes difficult to detect, however, at a the fringe limit, i.e., the peak or valley of a fringe. Some modifications could be made to improve the accuracy of the current interferometric set-up. Quadrature coding is often employed in shock-wave experiments to eliminate any ambiguity in the sign of the acceleration [69, 70]. By adding a quarter-wave retardation plate and two polarizing beam splitters, two

quadrature optical signals can be obtained from the horizontally and vertically polarized components of light that differ in phase because of the retardation plate. Simultaneous in-plane surface displacement measurement could also improve the accuracy. However, most optical techniques for measuring in-plane displacement require the application of a diffraction grating to the sample surface. Such gratings would alter the failure behavior of the films. Techniques that do not require a grating, such as confocal Fabry-Perot interferometry [71] or heterodyne speckle interferometry [72] may prove useful.

The Michelson interferometer used in this dissertation is essentially a displacement interferometer, where the displacement is proportional to the fringe count of the interferometric signal. In order to obtain the stress in the substrate and at the thin film interface, the displacement-time profile has to be differentiated (first derivative for substrate stress and second derivative for interface stress). As a result, higher noise is induced as more derivatives are taken. A normal-velocity interferometer (NVI) as developed by Barker and Hollenbach [73] could be adopted, in which the surface velocity instead of displacement is proportional to the fringe count of the signal. In the velocity interferometer, normally reflected laser light from the sample surface is collected and split into two separate beams, which are subsequently recombined after traveling through different path lengths. The interferometer sensitivity is a function of the delay time between the two interfering beams, and the resulting fringe signal is related directly to changes in normal velocity. In this technique, no differentiation is needed for the substrate stress and only one differentiation is needed for the interface stress. The resolution of the stress data could be increased. The NVI technique, however, is limited in the low-velocity range ($0.1\text{--}0.25\text{ mm}/\mu\text{m}$), which is a typical range for the current experiment. Velocity in this range leads to delay times in the neighborhood of 5 ns or more in order to obtain three or four fringes. This in turn leads to significant averaging of the velocity measurements [70]. Thus in order to use an NVI, higher YAG laser power and thicker confining layers are necessary to generate velocities higher than $0.1\text{--}0.25\text{ mm}/\mu\text{m}$.

In the mixed-mode loading experiment, alignment of the argon detection beam and YAG

laser beam is critical to guarantee that the signal is detected right at the center of the mixed-mode loading point (point B in Fig. 4.1), where the strongest signal is expected. Any misalignment due to tilting of the sample makes the argon beam deviate from point B and results in a reduced signal. In the current experiments, the beams are aligned only in the center window of each prism sample. Any tilting or shifting of the sample in the vertical plane could induce alignment error, especially at positions far from the center window. A possible solution is to leave an alignment window between every two test spots. Alternatively, alignment could be performed at each test spot, depositing aluminum film only around point B, leaving point A available for alignment. In both cases, a larger prism (also higher power YAG) is desirable to guarantee enough working space on the test-film side of the prism.

The mixed-mode loading technique developed in this dissertation can be used to investigate the interfacial strength for a range of mode mixity. Mode dependence of the interface toughness plays a critical role in determining the sequence of events in the propagation of a delamination [74]. By a systematical changing of the incident angle θ_i in Fig. 4.1, which will also subsequently change the mode conversion of the incident longitudinal wave to a shear wave (Fig. 4.2), the interfacial strength could be measured for a full range of mixed-mode loading conditions from purely tensile to purely shear. The results may provide significant insights into thin-film failure mechanisms.

In the laser-spallation experiments, the interfacial adhesion strength is characterized in terms of stress. In many other adhesion measurement techniques, such as the scratch [8], blister [19] and indentation tests [21, 22], the adhesion is measured in terms of energy. In order to relate the measurements from laser-spallation technique to that obtained from other techniques, future work can be carried out to calculate the energy associated with laser spallation. One such parameter is the kinetic energy stored in the thin film, which can be directly calculated from the measured surface velocity of the film, the film density, and thickness. Since this energy contains all the energy available in the film, it can serve as an upper bound for the fracture energy in the laser-spallation technique.

Such extrinsic effects as friction, substrate roughness, and imperfections also may play a significant role in thin-film failure modes and contribute to the apparent interface toughness. Recently, Balint and Hutchinson [74] analyzed the effect of surface roughness on steady-state mode II edge delamination of compressed thin films. Their results showed that nano-scale surface roughness together with the associated friction at the interface tends to increase the effective mode II toughness of thin films with thickness in the micron range. Although the film failure mechanism in this dissertation is different from that considered by Balint and Hutchinson model, it is possible to explore the influence of surface roughness on film failure mode and the corresponding interfacial strength using the currently developed mixed-mode loading experiments. The current technique may also be used to investigate fracture in the presence of controlled flaws at the thin-film interface or on the test film. For example, a circular flaw may be introduced in the process of film deposition. By precisely controlling the incident position of the stress waves, the failure may initiate and develop along the flaw edge.

In the current research, the intrinsic interfacial strength for aluminum/fused-silica substrate is determined for both tensile and mixed-mode loading cases. Fused silica is found to be an ideal substrate material for launching high-amplitude shock waves (its negative nonlinear elasticity being critical for the development of a decompression shock). By inserting an extra layer of the desired substrate material between the fused silica and the thin film, the current technique can be used to test a variety of substrates and films, especially those of interest to the microelectronics industry, such as polyimide, copper (Cu), aluminum (aluminum) and polysilicon on single-crystal silicon substrates.

Appendix A

Analytical Solution of the Thermo-elastic Stress Pulse

The solution of the heat transfer problem described by equation (2.1) with boundary conditions (2.5) and (2.6) is obtained by Laplace transformation. The transformation of equation (2.1) yields a partial differential equation in the Laplace domain

$$\tilde{T}(x,s) = \frac{s}{D_i} \tilde{T}_i, \quad (\text{A.1})$$

with boundary conditions

$$\begin{cases} \tilde{T}_1(0,s) = \tilde{T}_2(0,s) \\ k_1 \tilde{T}_1(x,s)|_{x=0^-} - k_2 \tilde{T}_2(x,s)|_{x=0^+} = \phi \end{cases}, \quad (\text{A.2})$$

and

$$\begin{cases} \tilde{T}_1(x,s) \rightarrow 0 \text{ as } x \rightarrow -\infty \\ \tilde{T}_2(x,s) \rightarrow 0 \text{ as } x \rightarrow +\infty \end{cases}. \quad (\text{A.3})$$

The solution to the above system of equations is

$$\begin{cases} \tilde{T}_1(x, s) = \frac{\phi}{\sqrt{k_1 \rho_1 C_1 + \sqrt{k_2 \rho_2 C_2} s}} \frac{1}{s} \exp\left(\sqrt{\frac{s}{D_1}} x\right) \\ \tilde{T}_2(x, s) = \frac{\phi}{\sqrt{k_1 \rho_1 C_1 + \sqrt{k_2 \rho_2 C_2} s}} \frac{1}{s} \exp\left(-\sqrt{\frac{s}{D_2}} x\right) \end{cases}.$$

The solution to the initial heat transfer problem is then found by inverse Laplace transformation.

The solution of the elastic wave propagation problem given by Eqs. (2.11) and (2.12) entails use of a Green's function when x is large and positive. The thermal source can be located in material 1 or 2.

For the case where the thermal source is in material 2, the P.D.E to solve for the Green's function is given by

$$\begin{cases} \rho \frac{\partial g_v}{\partial t} = \frac{\partial g_t}{\partial x} \\ \frac{\partial g_\sigma}{\partial t} = (\lambda + 2\mu) \frac{\partial g_v}{\partial x} - \delta(x - x_s, t - t_s) \quad \text{where } x_s \geq 0 \end{cases}, \quad (\text{A.4})$$

where

$$\begin{cases} v(x, t) = g_v * (\lambda + 2\mu) \rho \frac{\partial T}{\partial x} \\ \sigma(x, t) = g_\sigma * (\lambda + 2\mu) \rho \frac{\partial T}{\partial x} \end{cases}. \quad (\text{A.5})$$

Equation (A.4) is equivalent to

$$\begin{cases} \rho \frac{\partial g_t}{\partial t} = \frac{\partial g_\sigma}{\partial x} \\ \frac{\partial g_\sigma}{\partial t} = (\lambda + 2\mu) \frac{\partial g_v}{\partial x} \end{cases}, \quad (\text{A.6})$$

with boundary conditions

$$\left\{ \begin{array}{l} g_1|_{x=0^-} \equiv g_1|_{x=0^+} \\ g_1|_{x=x_2^+} = \frac{1}{(\lambda+2\mu)_2} \delta(t - \frac{x_2}{c_2}) \\ g_2|_{x=0^-} \equiv g_2|_{x=0^+} \\ g_2|_{x=x_2} = g_2|_{x=x_2^+} \end{array} \right. \quad (\text{A.7})$$

Using Laplace transformation, equations (A.6) and (A.7) become

$$\left\{ \begin{array}{l} H s \tilde{g}_1 = \frac{\partial \tilde{g}_1}{\partial x} \\ s \tilde{g}_2 = (\lambda + 2\mu)_2 \frac{\partial \tilde{g}_2}{\partial x} \end{array} \right. , \quad (\text{A.8})$$

with boundary conditions

$$\left\{ \begin{array}{l} \tilde{g}_1|_{x=0^-} \equiv \tilde{g}_1|_{x=0^+} \\ \tilde{g}_1|_{x=x_2^+} \equiv \frac{1}{(\lambda+2\mu)_2} e^{-s \frac{x_2}{c_2}} \\ \tilde{g}_2|_{x=0^-} \equiv \tilde{g}_2|_{x=0^+} \\ \tilde{g}_2|_{x=x_2} = \tilde{g}_2|_{x=x_2^+} \end{array} \right. \quad (\text{A.9})$$

The solution in the Laplace domain is

$$\left\{ \begin{array}{l} \tilde{g}_1(x, s) = \frac{1}{2\mu_2} \left[e^{-s(t + \frac{x-x_2}{c_2})} + \frac{P_1 C_1 - P_2 C_2}{P_1 C_1 + P_2 C_2} e^{-s(t + \frac{x+x_2}{c_2})} \right] \\ \tilde{g}_2(x, s) = -\frac{1}{2C_2} \left[e^{-s(t + \frac{x+x_2}{c_2})} + \frac{P_1 C_1 - P_2 C_2}{P_1 C_1 + P_2 C_2} e^{-s(t + \frac{x-x_2}{c_2})} \right] \end{array} \right. \quad (\text{A.10})$$

By inverse Laplace transformation, the Green's function when $x \gg x_2 \gg 0$ is obtained as

which is a sum of a direct pulse and a reflection off the interface.

For the case when the thermal source is located in material 1, solution follows as above.

The Green's function when $x \gg 0$ and $x_s < 0$ is given by

$$\begin{cases} g_1(x, x_s, t - t_s) = \frac{1}{c_1(\rho_1 c_1 + \rho_2 c_2)} \delta(t - t_s - \frac{x}{c_2} + \frac{x_s}{c_1}) \\ g_2(x, x_s, t - t_s) = -\frac{\rho_2 c_2}{c_1(\rho_1 c_1 + \rho_2 c_2)} \delta(t - t_s - \frac{x}{c_2} + \frac{x_s}{c_1}) \end{cases}, \quad (\text{A.11})$$

which consists of a single pulse that has been transmitted through the interface. Solution to the one-dimensional wave equation (2.11) and (2.12) is then obtained using

$$\sigma(x, t) = \int_{-x}^{+x} \int_0^t g_2(x, x_s, t - t_s) * (3\lambda + 2\mu) \alpha \frac{\partial T(x_s, t_s)}{\partial t_s} dx_s dt_s. \quad (\text{A.12})$$

which yields equation (2.15).

To find the displacement field, equation (A.12) is integrated in parts to obtain

$$\sigma(x, t) = \frac{\partial}{\partial t} \int_{-x}^{+x} \int_0^t (3\lambda + 2\mu) \alpha g_1(x, x_s, t - t_s) T(x_s, t_s) dx_s dt_s. \quad (\text{A.13})$$

Given that for $x > 0$,

$$\sigma(x, t) = \rho_2 c_2 \frac{\partial u(x, t)}{\partial t}, \quad (\text{A.14})$$

one obtains

$$u(x, t) = -\frac{1}{\rho_2 c_2} \int_{-x}^{+x} \int_0^t (3\lambda + 2\mu) \alpha g_1(x, x_s, t - t_s) T(x_s, t_s) dx_s dt_s, \quad (\text{A.15})$$

which yields equation (2.16).

Appendix B

Stress Calculation Using Wave Transmission and Reflection Coefficients

Standard wave transmission and reflection theory is adopted [44] to obtain an exact expression of the stress in the substrate and at the interface in terms of the measured surface displacement u_0 . The substrate is considered as a homogeneous, isotropic semi-infinite media and the film of thickness h and different material properties is to be attached to the surface of the semi-infinite media as show in Fig. B.1. In the analysis, $\lambda_1, \mu_1, \rho_1, c_1$ and $\lambda_2, \mu_2, \rho_2, c_2$ are the Lamé moduli, density and dilatational wave speed of the substrate and film, respectively.

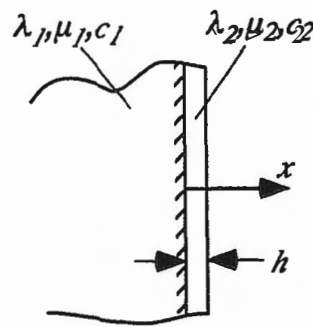


Figure B.1: A film of thickness h on a semi-infinite substrate.

Assuming a plane harmonic wave strikes the interface from the negative infinity, the

one-dimensional elastic wave equation in both media is

$$(\lambda + 2\mu)_i \frac{\partial^2 u_i}{\partial x^2} = \rho_i \frac{\partial^2 u_i}{\partial t^2}. \quad (\text{B.1})$$

The stress-displacement relation is given by

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

The non-trivial boundary conditions for the problem are

$$u_1 = u_2, \quad \sigma_1 = \sigma_2 \quad \text{at} \quad x = 0, \quad (\text{B.3})$$

$$u_2 = u_{\text{surface}} = u_0; \quad \sigma_2 = 0 \quad \text{at} \quad x = h. \quad (\text{B.4})$$

Solutions to equation (B.1) can be obtained in the form of

$$\begin{cases} u_1 = A_1 e^{-i\omega(t + \frac{x}{c_1})} + A_2 e^{-i\omega(t - \frac{x}{c_1})} \\ u_2 = A_3 e^{-i\omega(t + \frac{x}{c_2})} + A_4 e^{-i\omega(t - \frac{x}{c_2})} \end{cases} \quad (\text{B.5})$$

The corresponding stress can be written in the form,

$$\begin{cases} \sigma_1 = -i\omega\rho_1 c_1 [A_1 e^{-i\omega(t + \frac{x}{c_1})} - A_2 e^{-i\omega(t - \frac{x}{c_1})}] & x \leq 0 \\ \sigma_2 = -i\omega\rho_2 c_2 [A_3 e^{-i\omega(t + \frac{x}{c_2})} - A_4 e^{-i\omega(t - \frac{x}{c_2})}] & x \geq 0 \end{cases} \quad (\text{B.6})$$

By applying the boundary conditions at the interface and free boundary, one gets

$$\begin{cases} A_1 + A_2 - A_3 - A_4 = 0 \\ \rho_1 c_1 (A_1 - A_2) - \rho_2 c_2 (A_3 - A_4) = 0 \\ A_3 e^{-i\frac{\omega h}{c_2}} + A_4 e^{i\frac{\omega h}{c_2}} = \tilde{u}_0 \\ A_3 e^{-i\frac{\omega h}{c_2}} - A_4 e^{i\frac{\omega h}{c_2}} = 0 \end{cases} \quad (\text{B.7})$$

where $\tilde{u}_0$ denotes the fourier transformation of u_0 .

Solving for the coefficients gives

$$\begin{pmatrix} A_1 \\ A_2 \\ A_3 \\ A_4 \end{pmatrix} = \frac{\tilde{u}_0}{2} \begin{pmatrix} \cos \frac{\omega h}{c_2} + i \frac{\rho_2 c_2}{\rho_1 c_1} \sin \frac{\omega h}{c_2} \\ \cos \frac{\omega h}{c_2} - i \frac{\rho_2 c_2}{\rho_1 c_1} \sin \frac{\omega h}{c_2} \\ \cos \frac{\omega h}{c_2} + i \sin \frac{\omega h}{c_2} \\ \cos \frac{\omega h}{c_2} - i \sin \frac{\omega h}{c_2} \end{pmatrix}. \quad (\text{B.8})$$

The amplitude of the incident stress in the substrate is therefore given by

$$\begin{aligned} \tilde{\sigma}_{\text{sub}} &= i\omega \rho_1 c_1 A_2 \\ &= \frac{\tilde{u}_0}{2} (i\omega) \rho_1 c_1 \left[\cos \frac{\omega h}{c_2} - i \frac{\rho_2 c_2}{\rho_1 c_1} \sin \frac{\omega h}{c_2} \right], \end{aligned} \quad (\text{B.9})$$

and the amplitude of the stress acting on the interface is given by

$$\begin{aligned} \tilde{\sigma}_{\text{int}} &= -i\omega \rho_2 c_2 (A_3 - A_4) \\ &= \omega \rho_2 c_2 \tilde{u}_0 \sin \frac{\omega h}{c_2} \\ &= -(i\omega)^2 \rho_2 c_2 \tilde{u}_0 \frac{\sin \frac{\omega h}{c_2}}{\omega}. \end{aligned} \quad (\text{B.10})$$

For small film thickness h , $\cos \frac{\omega h}{c_2} \approx 1$, $\sin \frac{\omega h}{c_2} \approx 0$, then

$$\tilde{\sigma}_{\text{sub}} \approx (i\omega) \rho_1 c_1 \frac{\tilde{u}_0}{2}, \quad (\text{B.11})$$

$$\tilde{\sigma}_{\text{int}} \approx \omega^2 \rho_2 h \tilde{u}_0 = -(i\omega)^2 \rho_2 h \tilde{u}_0. \quad (\text{B.12})$$

By inverse Fourier transformation, one obtains

$$\sigma_{\text{sub}} = -\frac{1}{2} (\rho_1 c_1) \frac{\partial u_0}{\partial t}, \quad (\text{B.13})$$

$$\sigma_{\text{int}} = -\rho_2 h \frac{\partial^2 u_0}{\partial t^2}. \quad (\text{B.14})$$

which are the same as Eqs. (2.46) and (2.47) used in Section 2.2.4 on page 18. Note that the displacement at the free surface, u_0 , is twice the displacement of the wave in the substrate, u , given by Eqs. (2.46) and (2.47) .

Without the assumption of small film thickness h , Eq. (B.9) leads to the following expressions

$$\sigma_{\text{sub}} \equiv -\frac{1}{2} \frac{\partial}{\partial t} \left\{ u_0 * \left[\rho_1 c_1 \text{IFFT} \left(\cos \frac{\omega h}{c_1} \right) + \rho_2 c_2 \text{IFFT} \left(\sin \frac{\omega h}{c_2} \right) \right] \right\}, \quad (\text{B.15})$$

$$\sigma_{\text{int}} \equiv -\rho_2 c_2 \frac{\partial^2}{\partial t^2} \left[u_0 * \text{IFFT} \left(\frac{\sin \frac{\omega h}{c_2}}{\omega} \right) \right], \quad (\text{B.16})$$

where

$$\begin{aligned} \text{IFFT} \left(\cos \frac{\omega h}{c_2} \right) &= \frac{1}{2} \left[\delta \left(t - \frac{h}{c_2} \right) + \delta \left(t + \frac{h}{c_2} \right) \right], \\ \text{IFFT} \left(\sin \frac{\omega h}{c_2} \right) &= \frac{i}{2} \left[\delta \left(t - \frac{h}{c_2} \right) - \delta \left(t + \frac{h}{c_2} \right) \right], \\ \text{IFFT} \left(\frac{\sin \frac{\omega h}{c_2}}{\omega} \right) &= \begin{cases} \sqrt{\frac{\pi}{2}} & |t| \leq \frac{h}{c_2} \\ 0 & \text{otherwise} \end{cases}. \end{aligned}$$

Equations (B.15) and (B.16) yield the final expression for substrate and interface stress

$$\begin{aligned} \sigma_{\text{sub}} &\equiv -\frac{1}{2} \frac{\partial}{\partial t} \left\{ u_0 * \left(\frac{\rho_1 c_1}{2} \left[\delta \left(t - \frac{h}{c_2} \right) + \delta \left(t + \frac{h}{c_2} \right) \right] + \frac{\rho_2 c_2}{2} \left[\delta \left(t - \frac{h}{c_2} \right) - \delta \left(t + \frac{h}{c_2} \right) \right] \right) \right\} \\ &\equiv -\frac{1}{4} \rho_1 c_1 \frac{\partial}{\partial t} \left\{ \left[u_0 \left(t - \frac{h}{c_2} \right) + u_0 \left(t + \frac{h}{c_2} \right) \right] + \frac{\rho_2 c_2}{\rho_1 c_1} \left[u_0 \left(t - \frac{h}{c_2} \right) - u_0 \left(t + \frac{h}{c_2} \right) \right] \right\} \quad (\text{B.17}) \end{aligned}$$

$$\begin{aligned} \sigma_{\text{int}} &= -\rho_2 c_2 \frac{\partial^2}{\partial t^2} \left\{ u_0 * \sqrt{\frac{\pi}{2}} \left[H \left(t - \frac{h}{c_2} \right) - H \left(t + \frac{h}{c_2} \right) \right] \right\} \\ &\equiv -\rho_2 c_2 \sqrt{\frac{\pi}{2}} \frac{\partial^2}{\partial t^2} \int_{-\frac{h}{c_2}}^{\frac{h}{c_2}} u_0 \left(t + \xi \right) d\xi \quad (\text{B.18}) \end{aligned}$$

which are valid for large film thickness.

Appendix C

Residual Stress Measurements

The thin films used in this project were either vapor deposited using a thermal evaporator or sputtered using a sputtering system. During the deposition process, large residual stress can be induced. Because the residual stress state could affect the failure pattern of the test film and the measured interface strength value, the residual stress in the aluminum films tested in this project were determined using a wafer curvature measurement technique.

Thin film residual stress in both aluminum/silicon and aluminum/fused silica samples were measured with a KLA-Tencor Flexus Model 2908 equipped with a laser reflectance measurement system. A schematic of the apparatus is shown in Fig. C.1. This particular model measures the angle of reflections of two laser beams which are vertically incident on the wafer. To improve accuracy, a line scan traces 100 data points on the surface, from which the radius of curvature is calculated by a parabolic fit of the deflection curve.

The measured radius of curvature is then used to calculate the residual stress in the thin film using Stoney equation [75]

$$\sigma = \left(\frac{E}{1-\nu} \right)_s \frac{t_s^2}{6t_f} \left(\frac{1}{R} - \frac{1}{R_0} \right), \quad (\text{C.1})$$

where the biaxial elastic modulus of the substrate is $E/((1-\nu))_s$, and t_s and t_f are the substrate and film thickness, respectively. Here, R_0 is the radius of curvature before deposition (reference) and R is the radius of curvature after film deposition. The above equation is

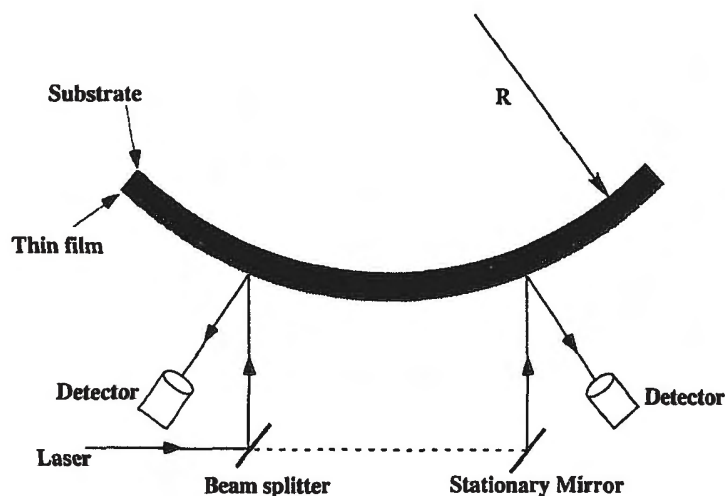


Figure C.1: Schematic of the stress measurement apparatus.

based on beam bending mechanics and assumes that the film thickness is much less than that of the substrate. This assumption allows calculation of stress in the film from the thickness of the substrate and film and the elastic properties of the substrate only. In the current work, all films were greater than two orders of magnitude thinner than the substrate thickness of $300 \sim 500 \mu\text{m}$.

In Figure C.1, the sign of the wafer curvature ($1/R$) is assumed positive (concave up). If an additional stress is generated within the film, the curvature will either become more concave (R will decrease) for tensile stress, or the curvature will increase or eventually become negative if the additional stress is compressive. The converse situation arises when the initial radius of curvature is negative.

Residual stress in aluminum films deposited on $350 \mu\text{m}$ thick silicon substrate was measured for two film thicknesses, $0.5 \mu\text{m}$ and $1.0 \mu\text{m}$. Fig. C.2 shows the deflection curves before and after the film deposition for both film thicknesses. The measured radius of curvature before and after film deposition as well as the calculated residual stress are summarized in Table C.1. The residual stresses were measured 30 minutes after the deposition. The residual stress in both films is tensile, which is typical for evaporated metal films [76]. The

Table C.1: Residual stress measurement in aluminum thin films deposited on 350 μm thick silicon substrates.

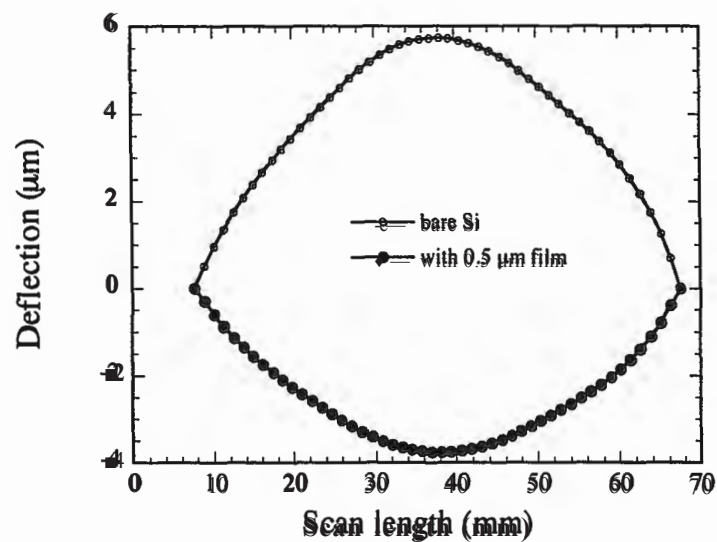
Film thickness (μm)	R_0	R	Residual stress σ (MPa)
0.5	-77	117	66
1.0	-34	-115	151

thinner film (0.5 μm) has a larger tensile stress, 151 MPa. And the thicker film (1.08 μm) has a lower tensile stress, 66 MPa.

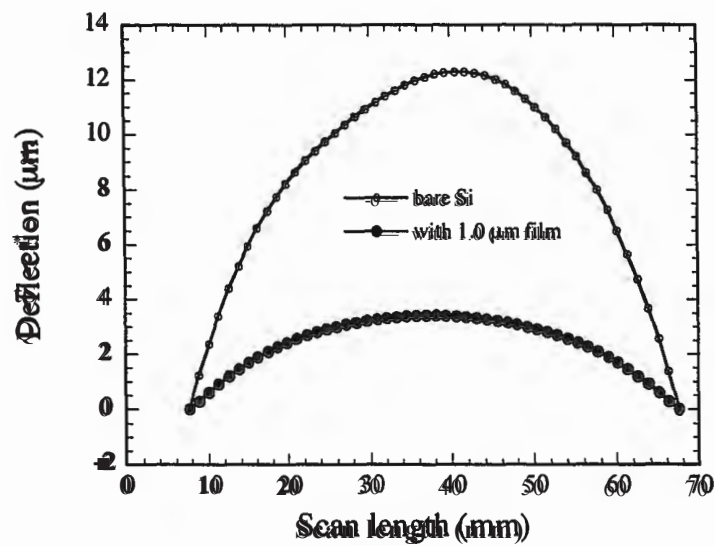
The residual stress in the 1.0 μm thick aluminum thin film was also monitored for 48 hours (in 30 minutes intervals) following the deposition. The residual stress was calculated for every measured R value and is plotted in Fig. C.3 as a function of time. The residual tensile stress in the 1.0 μm thick aluminum film relaxes nearly 17% of the initial value within 48 hours. After remaining in the lab for two months, the residual stress in the same film was measured again and found to be only 40 MPa. This relaxation behavior is common for evaporated metal films with low melting point [77]. At room temperature, stress-driven diffusion releases the residual stress in the film.

Residual stress in aluminum thin films deposited on 500 μm thick fused silica substrates were measured for several film thicknesses. Because fused silica is transparent, intensity of the reflected laser light was quite low for the base line measurement of R_0 and significant variation in the measurement occurs. In order to increase the reflected light intensity and the accuracy of the base line curvature measurements, a "prestress" strategy was adopted. This strategy is shown schematically in Figure C.4. Prior to any measurement, a prestress film was deposited on the top surface of the bare wafer. The radius of curvature measured with this top film was used as the initial radius of curvature R_0 . After establishing the base line, the film of interest was deposited on the bottom side of the wafer and the radius of curvature, R , was measured. The values of R_0 and R were then substituted into equation (C.1) to calculate the residual stress in the second film.

According to this strategy, a 0.4 μm thick aluminum film was used as the prestress film



(a)



(b)

Figure C.2: Deflection of a 3 inch diameter 350 μm thick silicon wafer before and after the deposition of a (a) 0.5 μm ; (b) 1.0 μm aluminum film.

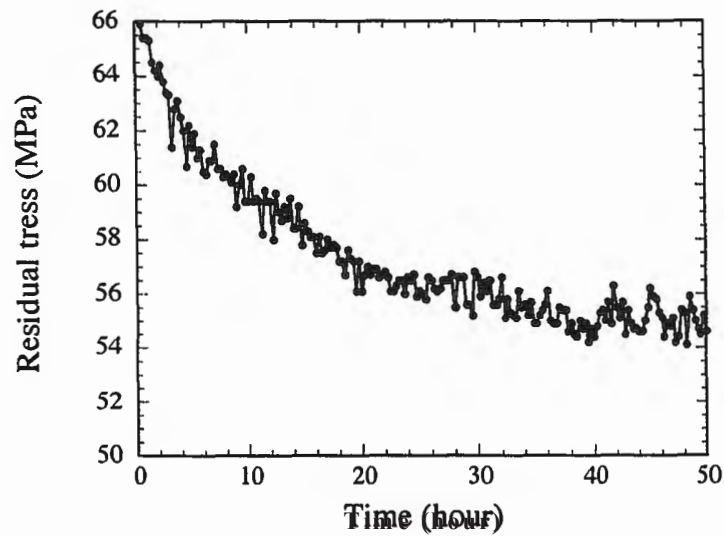


Figure C.3: Relaxation of the residual stress with time in a $1.0\ \mu\text{m}$ thick aluminum film deposited on a $350\ \mu\text{m}$ thick silicon wafer.

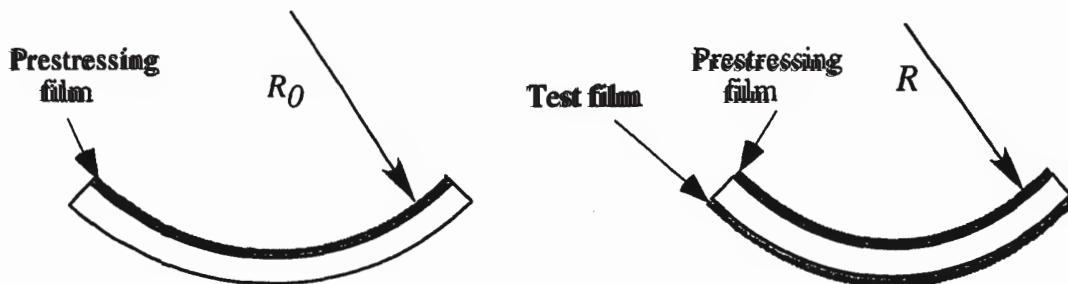


Figure C.4: Schematic of the curvature measurement by prestress strategy.

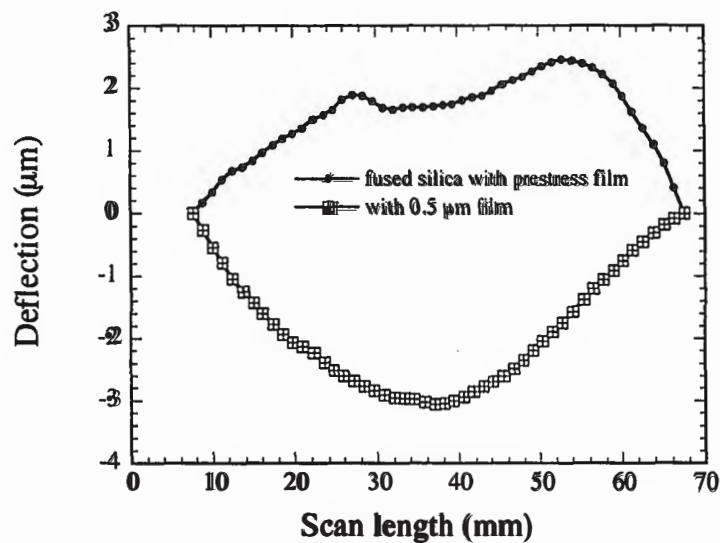
Table C.2: Residual stress measurement in aluminum thin films deposited on 500 μm thick fused-silica substrates.

Film thickness (μm)	R_0	R	Residual stress σ (MPa)
0.5	-185	163	70
1.35	69	22	68

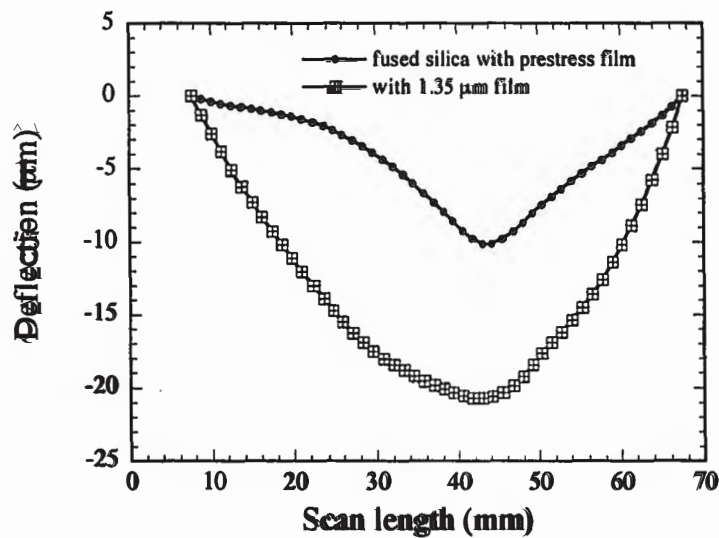
for three measurements. The addition of the prestress film is actually closer to the real situation in the experiment, in which a 0.4 μm thick aluminum absorbing layer is always deposited on one side of the substrate first. Two test film thicknesses, 0.5 μm and 1.35 μm , were studied. Figure C.5 shows the deflection curves before and after the second film (test film) deposition for both film thicknesses. The measured radius of curvature as well as the calculated residual stress are summarized in Table C.2.

Although the two film thicknesses are quite different, the measured residual stresses are very close, 70 MPa for 0.5 μm , and 68 MPa for 1.35 μm . There could be some error associated with these stress values due to the complex deflection of the wafer after prestressing. In wafer curvature measurement, the radius of curvature is obtained from a parabolic fit of the deflection curve. For both wafers in Fig. C.5, the initial deflection deviates from standard parabolic trend, which may induce errors in the curvature fitting. Despite this inaccuracy, the sign of the residual stresses are obviously tensile for both measurements.

The relaxation behavior of the residual stress in the 1.35 μm thick aluminum film was also monitored for 48 hours following the deposition in 30 minute increments. Similar to the case for silicon substrate samples, the residual stress in the aluminum film also relaxes with time (Fig. C.6). Most of the relaxation occurred during the first 10 hours following film deposition. The residual stress reached a stable value in about 30 hours. More than 57% of the initial residual stress relaxed in 48 hours. Thus, much more relaxation occurred in the fused silica samples than silicon samples, which is probably due to the difference of the substrate. The silicon substrate is single crystal and could induce stronger texture in the deposited aluminum film, resulting in higher residual stress. Fused silica is amorphous and



(a)



(b)

Figure C.5: Deflection of fused-silica wafer ($500\text{ }\mu\text{m}$) before and after the second film deposition of a (a) $0.5\text{ }\mu\text{m}$, (b) $1.35\text{ }\mu\text{m}$ thick aluminum film.

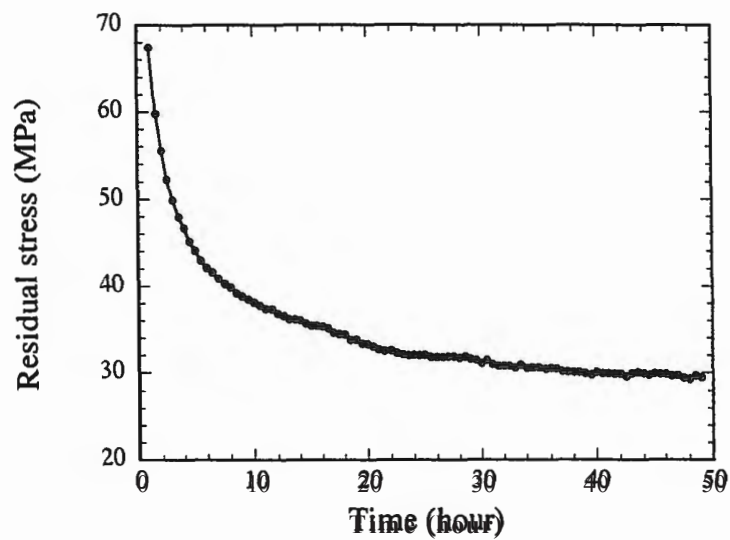


Figure C.6: Relaxation of the residual stress with time in a 1.35 μm thick aluminum film on a 500 μm thick fused-silica wafer.

could favor more diffusion at the interface and hence more relaxation.

Appendix D

Time Constant of Photodetector and Deconvolution Fringe Correction

During the spallation experiments, the shock usually has a fall time of about 1 ns. Thus the data must be resolved with sub-nanosecond resolution. The photodetector used in this project is a Silicon Pin Detector ET-2030, which was manufactured by Electro-Optics Technology, Inc. The main features of the detector are listed in Table D.1, among which the rise time and detection area are most important to this application. A small enough detection area guarantees that the signal is obtained from the center of the interference fringe and the light intensity is not reduced by averaging. A fast rise and fall time is essential for accurately resolving the fastest features of the shock. If the rise time of the detector is not fast enough, the real shock could be distorted. In order to evaluate the time constant of the photodetector, a single 35 ps impulse obtained from a Nd:YLF laser with a wavelength of 527 nm was measured by our ET-2030. This experiment provides the impulse response function of the detector and its electronics. The raw fringe data detected in the experiment can then be corrected by deconvolving with the impulse response function. The deconvolution may be affected in the Fourier transform domain. The correction is realized by taking the inverse Fourier transformation of the ratio of Fourier transformed raw fringe data, to the Fourier transformed impulse response function,

Table D.1: Main features of ET-2030 photo-detector.

Rise time	< 300 ps
Fall time	< 300 ps
Impedence	50 Ω
Detection area	0.01 mm ²
Sensitivity	0.4A/W @ 830 nm
Saturation	10 mA

$$I_{\text{corrected}} = \text{IFFT} \left[\frac{\text{FFT}(I_{\text{raw}})}{\text{FFT}(I_{\text{impulse}})} \right] \quad (\text{D.1})$$

where I_{raw} is the light intensity of the raw fringe data and I_{impulse} is impulse response function.

The impulse response function is plotted in Fig. D.1. The algorithm in Eq. (D.1) is applied on the raw fringe data plotted in Fig. D.2. The amplitude of the Fourier transformation of the raw fringe and impulse are plotted in Fig. D.3. The phase information is not plotted. Since the cut-off frequency of the impulse (~ 1.5 GHz) is much higher than that of the raw fringe data (~ 0.3 GHz), very little distortion is expected. The corrected fringe data and the corresponding displacement, substrate and interface stress are plotted in Fig. D.4 together with those from the raw data. The turning point is a little bit sharper in the corrected fringe data. The amplitude of the calculated displacement and substrate stress are very close for the corrected and raw data. The shock profile in Fig. D.4c is also a little bit sharper in the corrected signal, which then results in an interface stress about 34 MPa higher than that from the raw data. Although the profiles are sharpened, the deconvolution also introduces higher noise in the stress data.

Thus, the deconvolution shows little effect on the amplitude of substrate stress and only a small amount (8%) of increase in the interface stress. Comparing this effect and the effect of exact interface calculation (Eq. B.18), which tends to decrease the calculated interface stress as shown in Table ??, the effects of the large film thickness and photodetector detector

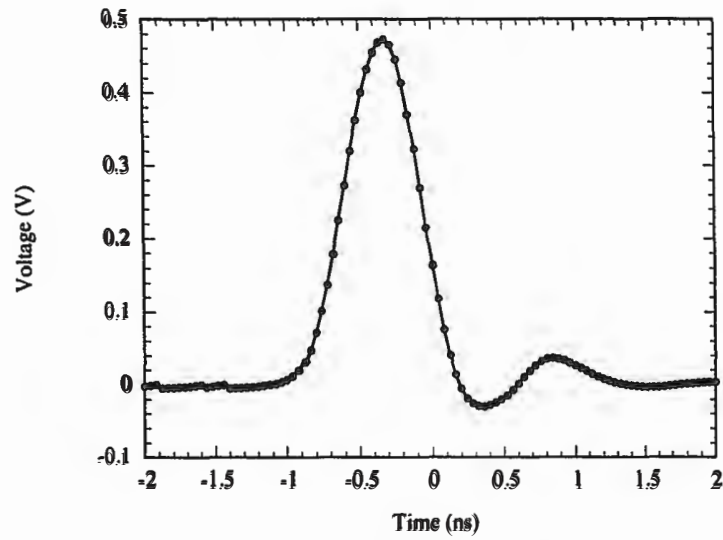


Figure D.1: A 35 ps impulse detected by ET-2030 photodetector and the detection electronics.

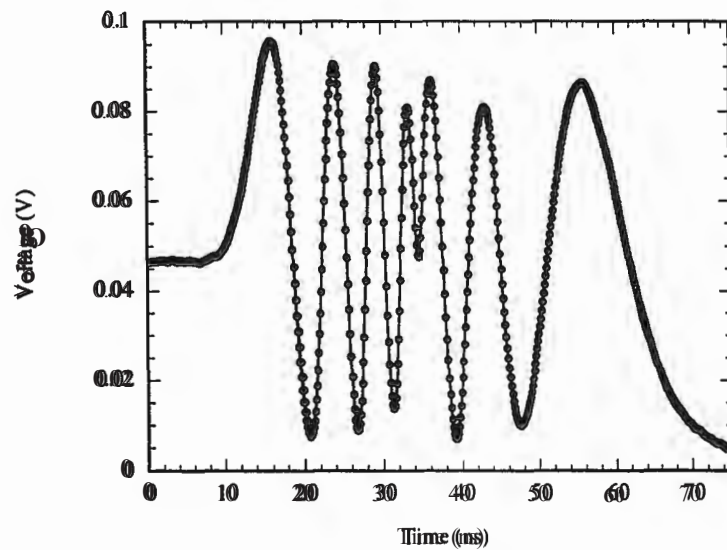


Figure D.2: Raw fringe data measured in tensile spallation experiment.

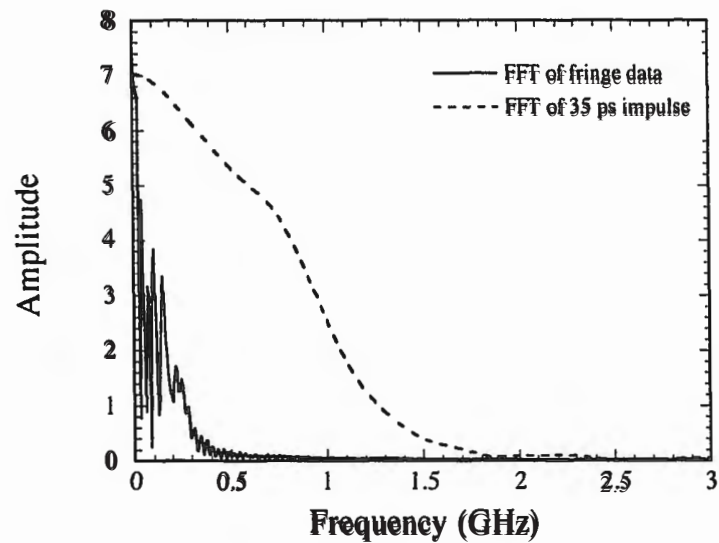


Figure D.3: Amplitude of the Fourier transformation of the impulse and fringe data.

distortion can both be safely neglected.

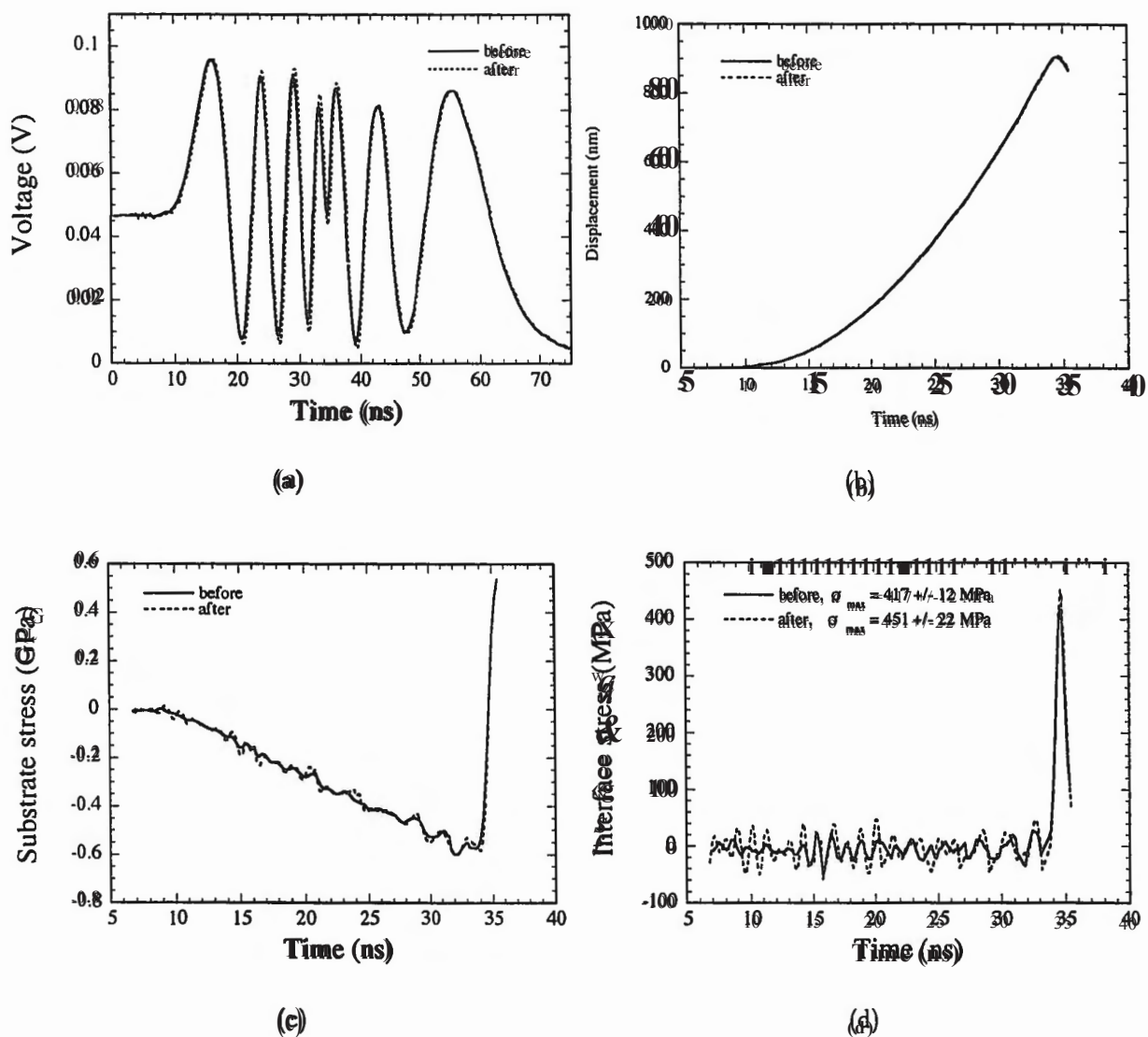


Figure D.4: Data before and after deconvolution: (a) fringe; (b) displacement; (c) substrate stress; (d) interface stress.

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Vita

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