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Measurement of interface strength by the modified laser spallation technique. III. Experimental optimization of the stress pulse

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In Part III of this series, experimental optimization of the laser spallation technique is achieved by investigating the effect of the kind and thickness of the constraining materials and the energy-absorbing film on the amplitude and profile of the generated stress pulse. An experimental setup consisting of a 0.5- μm -thick Al film as the energy absorbing film and a 5 μm layer of solid water glass as the constraining material is found to maximize the stress pulse amplitude. In addition, the effect of the substrate material on the shape of the stress pulse is also determined. Finally, it was found that for a given substrate, the pulse profile is independent of the laser fluence, indicating similar laser-material interaction phenomena over the range of fluence levels considered here.

I. INTRODUCTION

In Part I of this series,¹ the old and the modified versions of the laser spallation technique for measuring the tensile strengths of thin film interfaces were discussed. In Part II,² we presented applications of the modified laser spallation technique to carbon/metal interfaces, and interfaces between alumina substrates and Nb coatings with and without interlayers of Cr and Sb. In addition, the interfacial fracture mechanisms controlling its tensile strength were addressed. For potential applications of this technique to other interface systems, and systems involving thin coatings ($< 0.1 \mu\text{m}$ thickness), it is desirable to maximize the amplitude of the generated compressive pulse in the laser spallation experiment. Apart from increasing the pulse amplitude, as demonstrated by the numerical results described in Part I, the stress pulse rise and fall times also control the amplitude of the interface tensile stress. Furthermore, the interface tensile stress is far more sensitive to the decay time of the stress pulse than it is to the pulse's rise time. The aim of this article (Part III) is to optimize the stress pulse generation conditions in the laser spallation technique by changing the type and thickness of the laser-absorbing film, and its constraining medium. In addition, the influence of the substrate and laser fluence on the profile and amplitude of the stress pulse is discussed.

The modified laser spallation technique allows us to perform this optimization process experimentally since the generated stress pulse is detected at the rear surface of the coating or substrate. A competitive numerical model for obtaining the optimal conditions is very difficult to achieve as this requires modeling the multiphoton ionization process within the plasma, created at the film surface upon absorption of the laser energy. The ablating particles in the plasma produce a force normal to the material surface by momentum transfer. Acoustic waves formed by such processes differ markedly from those formed by the thermoelastic expansion of the solid or molten film. Since in the laser spallation experiment a combination of these processes occur at the threshold laser fluences, a direct experimental optimization as pursued here is the only reliable

way of studying such laser-material interactions. In addition, a column of liquid was used as an alternative to the solid confining medium in some experiments, which makes the modeling even more complicated.

The generation of stress pulses in solids by lasers has been pursued actively since 1963, starting with the study by White³ of laser-produced ultrasounds. One of the main interests over the years has been to enhance the stress pulse amplitudes. The first and the simplest approach involved using high-power laser pulses. The second approach involved using constraining layers on the surface of the solids or laser-absorbing films, resulting in an amplitude enhancement through constrained plasma expansion.^{4,5} O'Keefe and Skeen⁶ used coatings of transparent, relatively volatile materials to produce an order of magnitude increase in the pulse amplitudes, and Fox⁷ showed that a thin coat of black paint or drops of distilled water can produce results comparable to the constraining techniques. Yang⁸ investigated the effect of various laser-absorbing metallic films on the amplitudes of the stress pulses produced in a piezoelectric quartz crystal. However, few researchers^{9,10} discussed the effect of these on the stress pulse profile (defined by both the rise and fall times of the stress pulse). In our research results, presented here, we study the changes in both the profile and amplitude of the stress pulses generated by varying the various experimental conditions in the laser spallation setup, including the type and thickness of the constraining material, and the laser-absorbing film. In addition, we also explore the effect of the laser fluence and the substrate material on the stress pulse.

II. EXPERIMENTS

All experiments for stress pulse optimization were performed using the modified laser spallation setup discussed in detail in Part I. Except when studying the effect of the substrate material itself, all experiments reported here were performed on substrates of *N*-type Si single-crystal wafers with a 25.4 mm diam and 1 mm thickness. Both (100) surfaces of the Si wafer were polished to within a quarter of a wavelength of light. The deposition of the laser-absorbing

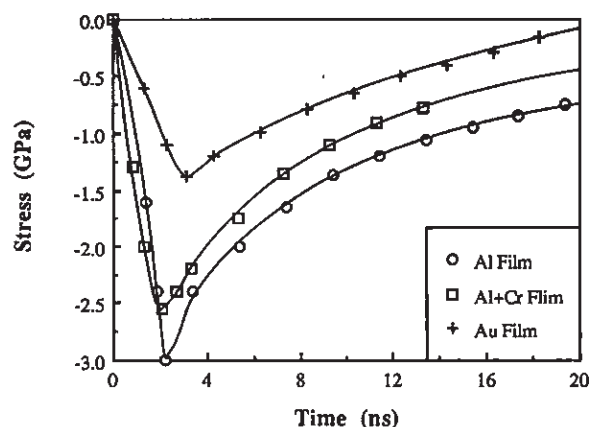


FIG. 1. Influence of the type of laser-absorbing film on the stress pulse profile (laser fluence = $8.67 \times 10^4 \text{ J/m}^2$; constraining material: a fused quartz disk).

films was done by the dc and rf-magnetron sputter system discussed in Part II. The amplitude and the profile of the stress pulse were measured by monitoring the transient free-surface velocity of the Si surface by the Doppler displacement interferometer.

III. RESULTS

A. Influence of the type of the laser absorbing film

Figure 1 shows the profiles of stress pulses generated in the Si wafer with different laser absorbing films. Since each film has a different emissivity at $1.06 \mu\text{m}$, for the purpose of comparison all experiments were performed at a laser fluence of $8.67 \times 10^4 \text{ J/m}^2$, measured before the laser pulse strikes the film. A fused quartz disk was used as the constraining material and lapped onto the film in a manner discussed in Gupta *et al.*^{11,12} The candidates for the laser absorbing films were Au, Al, and Al+Cr. The bilayer of Al+Cr involves depositing a film of Al on the substrate, followed by a deposition of the Cr film. In this arrangement the Al film acts as a stress-generating film because of its high thermal expansion coefficient, while the Cr film maximizes the absorbed laser energy because of its low reflectivity to the $1.06 \mu\text{m}$ laser beam. The thicknesses of Au, Al, and Cr films were chosen to be 0.5, 0.5, and 0.06 μm , respectively. Figure 1 shows that the Al and Al+Cr films produced higher amplitude and lower rise-time stress pulses than the Au film. Specifically, the rise times for the Au, Al, and Al+Cr films are 3, 2.4, and 2.2 ns, respectively, while the corresponding peak stresses, as measured from the interferometer, were 1.34, 2.5, and 3.0 GPa, respectively. The decay time of the pulse is controlled by the extent of the thermal pulse propagation within the film,^{11,12} which is controlled partly by the thermal diffusivities of the film and the constraining medium. Figure 1 shows similar decay times for all three cases, and interestingly, the thermal diffusivities for Al and Au are $0.982 \times 10^{-4} \text{ m}^2/\text{s}$ and $1.30 \times 10^{-4} \text{ m}^2/\text{s}$, respectively. The low rise time for the Al film in comparison to the Au film is due to its lower heat capacity and higher emissivity (7%) resulting in higher

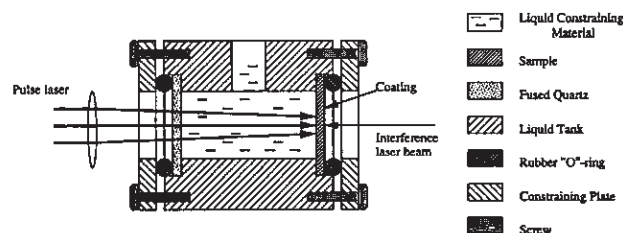


FIG. 2. Schematic of the liquid tank to generate the stress pulse.

temperatures, as the thermal diffusivities of Al and Au are quite comparable. Clearly, the rise time of the stress pulse is limited by the rate at which the heat energy is input, which in turn is controlled by the rise time of the laser pulse, being equal to about 1.5 ns in our experiments. Although the rise time of 2.2 ns for the Al+Cr film is marginally lower than 2.4 ns obtained using the Al film alone, the amplitude of the stress pulse is about 20% lower. Therefore, with respect to both the amplitude and rise-time optimization, a layer of Al was chosen for the modified laser spallation experiment.

B. Influence of the type of the constraining material

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

Three kinds of assembly were utilized to keep the constraining material in contact with the energy-absorbing film. When fused quartz was used as a constraining material, the assembly process described in Refs. 11 and 12 was used. The fused quartz was 3 mm in thickness and 32 mm in diameter.

As a second alternative, several liquids were also used as constraining materials. In these cases, a special liquid tank was designed and built. A schematic of the liquid tank is shown in Fig. 2. The liquid in the tank was held by a fused quartz wall and the surface of the sample was coated by the laser-absorbing film. The fused quartz also acts as a transparent window for the $1.0 \mu\text{m}$ Nd:YAG laser pulse and allows it to reach the laser absorbing film through the liquid column. The "O" rings prevent the liquid from leaking from the tank. The thicknesses of the liquid column and fused quartz were 25 and 3 mm, respectively. As shown in Fig. 2, a 12-mm-diam hole was provided on the upper part of the tank to release the air, otherwise the air pressure buildup upon evaporation of the liquid, would break the substrate. In the present experiments, the liquids

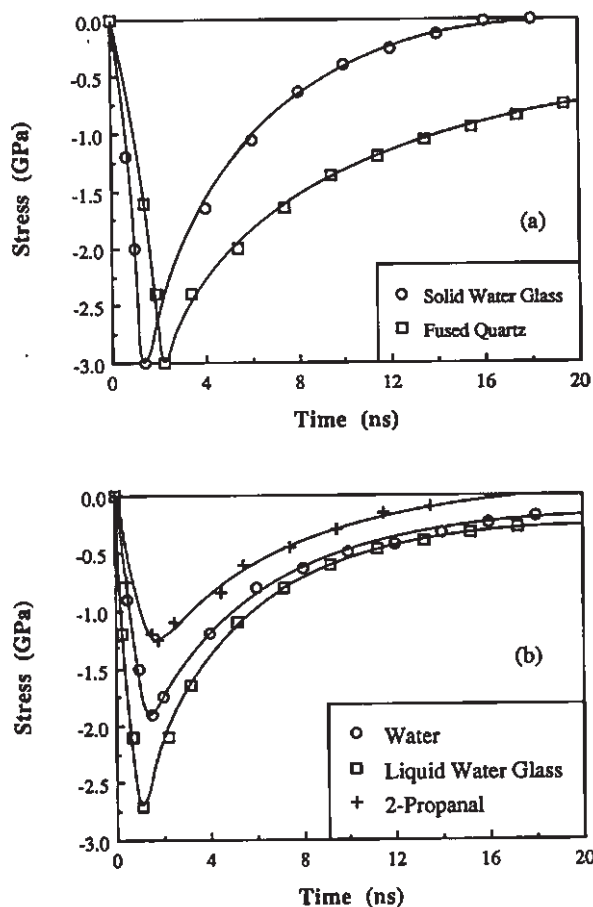


FIG. 3. Influence of the type of the constraining material on the stress pulse profile. All stress pulses were generated by a 0.5- μm -thick Al film and at a laser fluence of $8.67 \times 10^4 \text{ J/m}^2$. (a) Using a solid constraining media. (b) Using a liquid constraining media.

chosen were water, 2-propanol, and water glass $[\text{H}_2\text{SiO}_3:(\text{H}_2\text{O})_x]$.

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

For all the constraining materials, Al film was used as the laser-absorbing film. The results at laser fluence of $8.67 \times 10^4 \text{ J/m}^2$ (measured at the Nd:YAG source) for 0.5- μm -thick Al film are presented in Fig. 3. Remarkably, a 5- μm -thick layer of solid water glass generates the strongest and sharpest stress pulse. Although the fused quartz plate produces the same amplitude of 3.0 GPa, the rise time of 3.05 ns is almost 2.7 times that obtained by the layer of solid water glass. Furthermore, the stress pulse shows a much sharper decay in the case of solid water glass, a feature which is critical to increasing the interface

tensile stress. The shallow decay in the case of the fused quartz is due to a decrease in the effective thermal diffusivity and to the presence of interface air pockets. As shown by Gupta *et al.*¹² such a decrease in the thermal diffusivity will result in a longer and more gradual tail of the stress pulse profile. In fact, earlier experiments by Vossen,¹³ where no constraining medium was provided, showed even slower decay times of almost 100 ns.

The most interesting feature of Fig. 3 is that a use of liquid water glass produces a stress pulse amplitude that is only 10% lower than that obtained by its solid counterpart, and by the fused quartz. Interestingly, the rise time of 1.33 ns is lower compared to that of 3.05 ns obtained through the use of fused quartz. The same is true for the post-peak fall profile. In fact, compared to the fused quartz, both the rise and fall times are much sharper even for water (1.52 ns rise time) and for a liquid solution of 2-propanol (1.90 ns), albeit at lower amplitudes of 1.88 GPa for water and 1.25 GPa for the propanol solution. This presumably occurs due to a better interface, free of air bubbles.

The process of stress pulse generation with liquids as constraining media involves reaching the nucleation condition through a nonequilibrium, super-heating of the liquid. Although a quantitative modeling of this phenomenon is under consideration, here we provide a physical understanding of the phenomenon. For liquid water glass, both the amplitude and rise time of the pulse is almost the same as its solid counterpart, because at very high strain rates, the liquid provides all the inertia and acts exactly like a solid. Therefore, for time intervals shorter than the laser pulse rise time, the liquid water glass acts like a solid with similar thermo-mechanical properties, leading to a similar rise time. The sharper post-peak decay compared to the fused quartz results, due to a sudden cooling at the liquid/film interface from the liquid nucleation process. The cooling of this interface should significantly reduce the thermal pulse diffusion distance towards the substrate. As discussed earlier, this will result in a sharper decay of the stress pulse. Although a more quantitative understanding is required to select the most efficient liquid, nevertheless, the selection of various liquids based on a physical understanding given here indicates that a 5- μm -thick layer of solid water glass provides the desirable profile for the modified laser spallation experiment.

C. Influence of the thickness of the laser absorbing film

The thickness of the laser-absorbing film also influences the characteristics of the stress pulse. Since the Al film, when constrained by a 5- μm layer of solid water glass, provides the optimal pulse profile, experiments were carried out with Al film thicknesses of 0.3, 0.5, and 1.0 μm , and constrained by a 5- μm -thick layer of solid water glass. Figure 4 shows the profiles of the stress pulses for different Al thicknesses, all obtained at a laser fluence of $8.67 \times 10^4 \text{ J/m}^2$. No trend, either for the rise time or for the amplitude of the stress pulse, was observed with the coating thickness. The highest amplitude (3.0 GPa) and lowest

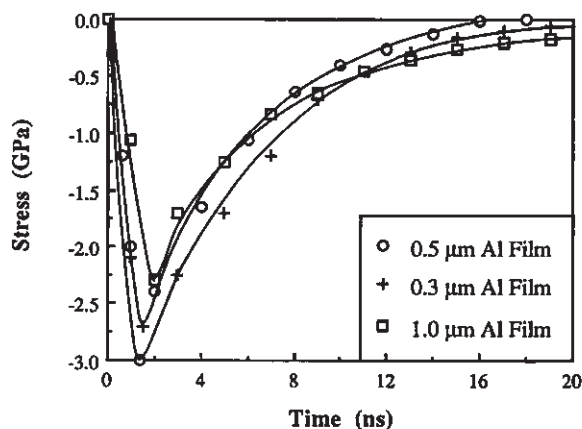


FIG. 4. Influence of the thickness of the laser-absorbing film on the stress pulse profile. (Constraining medium: 5- μ m-thick layer of solid water glass; laser fluence= 8.67×10^4 J/m²).

rise time (1.14 ns) was obtained with a 0.5- μ m-thick Al film. The rise time/maximum amplitude pair of 2.19 ns, 2.3 GPa, was obtained with a 1.0- μ m-thick film, while the pair 1.62 ns, 2.68 GPa, resulted with a 0.3- μ m-thick film. The lower amplitudes at thicknesses less than 0.5 μ m are due to a reduction in the total laser energy absorbed by the Al film. Clearly the penetration depth at a laser wavelength of 1.0 μ m should define the smallest thickness of the film for maximum pulse amplitudes. Furthermore, higher rise times are obtained at thicknesses lower than a critical film thickness (here, 0.5 μ m) because the poor thermal diffusivity of the confining medium starts to dominate, leading to lower spatial temperature gradients within the film, and resulting in a higher rise time. In conclusion, a 0.5- μ m-thick Al film confined by a 5- μ m thick layer of solid water glass is used for the modified laser spallation experiment.

D. Influence of the thickness of the constraining material

We also explored the optimal thickness for solid water glass layer in order to maximize the pulse characteristics. A 0.5- μ m-thick Al film was used as the laser-absorbing material. Our experiment showed that the stress pulse does not change when the water glass thickness changed from 1 to 10 μ m, which were the thicknesses typically used in the experiment. Within this range in thickness, the solid water glass layer and the molten Al film are completely removed after the generation of the stress pulse. As shown by Gupta *et al.*¹² in the laser spallation setup, one compressive pulse is produced in each the substrate and the constraining medium. It is the reflection of the compressive pulse into a tension pulse from the free surface of the solid water glass that leads to its removal. As explained in detail elsewhere,¹⁴ the removal of the Al and solid water glass occurs within 1.27 ns and all of the heat trapped within these layers is completely detached from the substrate. Consequently, the stress pulse within the substrate is free of any thermal tail and leads to a hitherto unreported stress pulse profile with 1.14 ns rise time, and with no post-peak

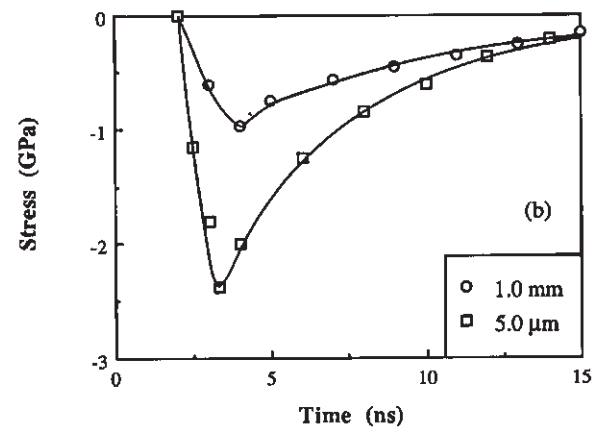
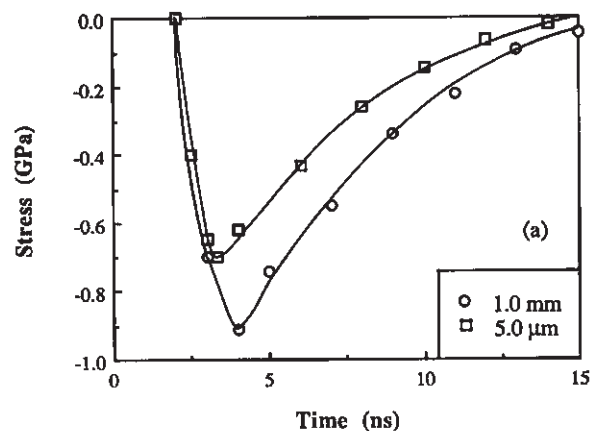


FIG. 5. Influence of the thickness of the constraining material on the stress pulse profile. All experiments were performed with a 0.5- μ m-thick laser absorbing Al film. The laser fluence used to generate the stress profile in (a) was 1.07×10^4 J/m² and for those in (b) was 4.18×10^4 J/m².

decay tail. As shown in Fig. 3, the stress becomes zero at 17 ns, whereas for other constraining media, the tail of the pulse assumes an asymptotic behavior. This effect is analogous to the mechanism of stress pulse generation through an impact of a thin flyer plate in the plate impact experiment.

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

E. Influence of the laser fluence

Within the range of laser fluences used in the experiment, we have found that a higher fluence results in an

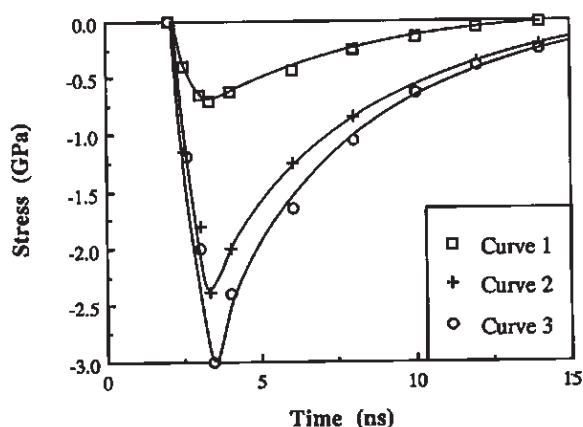


FIG. 6. Influence of the laser fluence on the stress pulse profile. Curve 1: $1.07 \times 10^4 \text{ J/m}^2$; Curve 2: $4.18 \times 10^4 \text{ J/m}^2$; Curve 3: $8.67 \times 10^4 \text{ J/m}^2$. All pulses were generated by using an Al film and 5- μm -thick layer of solid water glass.

increased stress pulse amplitude. However, the effect of the laser fluence on the shape of the stress pulse is also important. For a given substrate, if the profile of the stress pulse remains similar (they can have different amplitudes) for different laser fluences, then one stress pulse profile can be used as an input into the computer program to calculate the transfer coefficients (discussed in Part I), and this can be used throughout the entire experiment. Otherwise, the computer program has to be run for every stress pulse shape, which can be time consuming. Figure 6 presents the results for such experiments performed with 0.5 μm Al film and 5 μm solid water glass at increasing laser fluences. It shows that as the fluence level is increased, the profile of the stress pulse is retained, albeit with different amplitudes. Therefore, it is feasible to resort to the computer code only once for each substrate/coating system. The similarity in

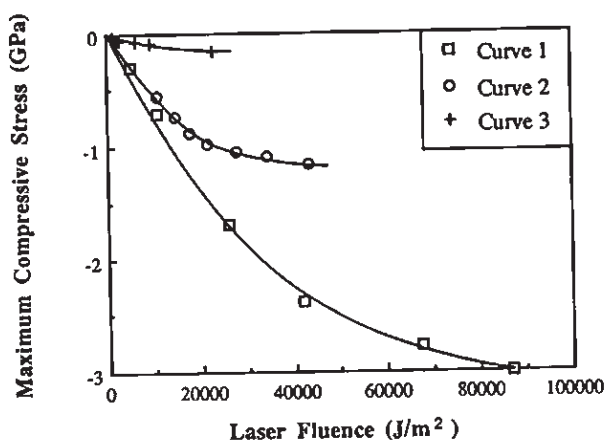


FIG. 7. Plots of the maximum compressive stress vs the laser fluence for several different experimental conditions. Curve 1. Substrate: Si; energy absorbing medium: 0.5- μm -thick Al film; constraining material: 5- μm -thick solid water glass. Curve 2. Substrate: polycrystalline Al_2O_3 ; energy absorbing medium: 0.5- μm -thick Al film; constraining material: a fused quartz disk. Curve 3. Substrate: PG; energy absorbing medium: 0.5- μm -thick Al film; constraining material: 5- μm -thick solid water glass.

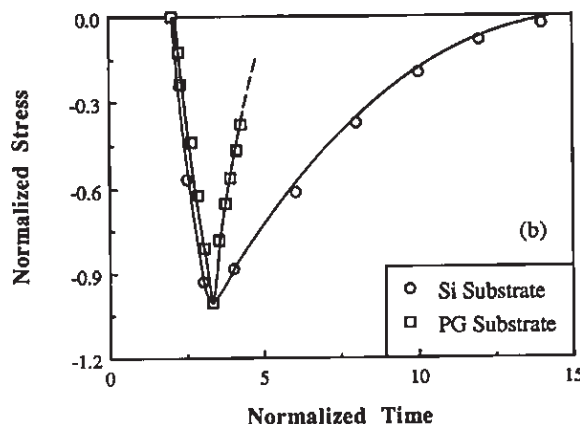
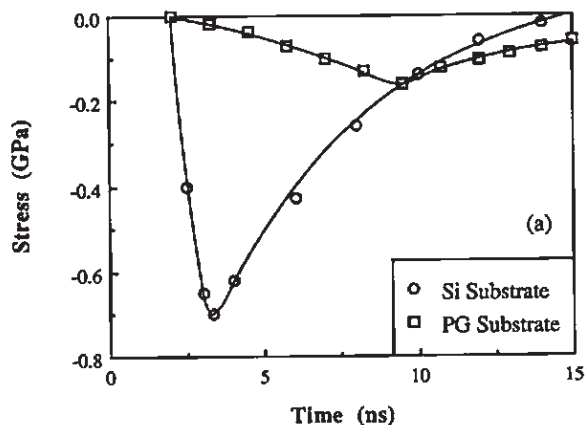


FIG. 8. Influence of the substrate material on the stress pulse profile. (a) Actual stress pulses. (b) Normalized stress pulses.

shape of the stress pulse implies the same mechanism for generation of the stress pulse within the fluence levels considered here.

The effect of the laser fluence on the maximum compressive stress is one of the most important characteristics of the stress pulse, since the maximum interface tensile stress is calculated by multiplying this value by the transfer coefficient (see Part I). Figure 7 shows plots of the maximum compressive stress versus the laser fluence for several different experimental conditions. Based on the results presented so far, it is apparent that such curves depend on the kind of substrate material, the kind and thickness of the energy absorbing material, and its constraining material.

F. Influence of the substrate material

Figure 7 shows that the amplitude of the stress pulse varies in different substrates even at the same laser fluence. As expected the profile of the stress pulse is also influenced significantly by a change of the substrate material. One of the examples is given in Fig. 8(a), which shows that the stress pulse generated in silicon substrate is much stronger (0.71 GPa) and sharper (1.2 ns rise time) than that in the pyrolytic graphite substrate (with 0.05 GPa amplitude and 4.5 ns rise time). Even the post-peak decay is sharper for Si due to its higher thermal diffusivity.

It will be of interest to see if a universal stress-pulse shape can be obtained in the normalized coordinates so that only one transfer function chart can suffice for all substrates in the laser spallation experiment. Normalizing the amplitude by using its maximum amplitude, and the time by using the rise time, normalized profiles were obtained for different substrates. Unfortunately as shown in Fig. 8(b) the normalized shapes are not similar. Therefore, the transfer coefficient charts as provided in Part I are associated with a given stress pulse profile.

IV. DISCUSSION

The results presented here indicate that an experimental setup which consists of a Al film of 0.5 μm as the energy-absorbing film and a layer of solid water glass several micrometers thick when used as the constraining material in the modified laser spallation experiment, gives the highest and the sharpest stress pulse. This has the best potential for spalling thin-film interfaces. This setup is an improvement over an earlier version described in Gupta *et al.*,¹² where a 1.0- μm -thick gold film, confined by a 2-mm-thick fused quartz was used.

In addition to the higher and sharper stress pulse profiles, there are several advantages of this setup over the old version. First, the contact between the energy-absorbing film and constraining medium is improved dramatically. In cases where a fused quartz is used as the constraining medium, even a very flat surface can lead to trapping of air gaps between the film/quartz interface due to small local roughness. The air gap reduces the amplitude of the stress pulse and results in a longer post-peak decay time. In addition, after the first shot, the thermal expansion and ablation of the energy-absorbing film and local microcracking of the fused quartz, causes additional air gaps near the ablated area. The replicability of the results in the modified setup is improved considerably due to the coherent, air bubble free interface. In addition to the elimination of air gaps, the experiment shows that the stress pulse is independent of the thickness of the constraining solid water glass provided the thickness is between 1 and 10 μm . This modification makes the experiment much easier to handle.

For a given substrate, the stress-pulse rise time is controlled predominantly by the rise time of the Nd:YAG laser pulse.¹² In all of the experiments reported here, as expected, the stress-pulse rise time is relatively less sensitive to the experimental variables, remaining within 1–3 ns. In contrast, the decay time or the fall time of the stress pulse is a strong function of the experimental variables. Fortunately, as shown by the numerical results presented in Part I, the interface tensile stress is far more sensitive to the decay time of the stress pulse than it is to the pulse's rise time.

V. CONCLUSION

In this article (Part III), we presented an experimental optimization program for obtaining higher amplitudes and sharper profile stress pulses produced by a pulsed laser. The results can be summarized as follows.

(1) Among the Al, Au, and Al+Cr films, the Al film is most efficient for producing the strongest and the shortest rise-time stress pulses. Furthermore, a film thickness of 0.5 μm maximizes the stress. Of the constraining materials, a layer of solid water glass of 5 μm is demonstrated to be the best choice. Therefore, an experimental setup consisting of a 0.5- μm -thick Al energy-absorbing film, and a layer of solid water glass 5 μm in thickness is chosen for the modified laser spallation technique.

(2) The optimized experimental setup has advantages over the old version with respect to having a coherent, air bubble free contact, resulting in a high repeatability of spallation results.

(3) The shape of the stress pulse for a given substrate is independent of the laser fluence. This feature further simplifies the experimental process of the modified laser spallation technique.

(4) The shape of the stress pulse depends strongly on the kind of the substrate material.

(5) In the modified laser spallation experiment, the dominant mechanism for generating the stress pulse is through a plasma-induced momentum transfer process.

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