Evolution of the Laser-Induced Spallation Technique in Film Adhesion Measurement

Laser-induced spallation is a process in which a stress wave generated from a rapid, high-energy laser pulse initiates the ejection of surface material opposite the surface of laser impingement. Through knowledge of the stress-wave amplitude that causes film separation, the adhesion and interfacial properties of a film-on-substrate system are determined. Some advantages of the laser spallation technique are the noncontact loading, development of large stresses (on the order of GPa), and high strain rates, up to $10^8$/s. The applicability to both relatively thick films, tens of microns, and thin films, tens of nm, make it a unique technique for a wide range of materials and applications. This review combines the available knowledge and experience in laser spallation, as a state-of-the-art measurement tool, in a comprehensive pedagogical publication for the first time. An historical review of adhesion measurement by the laser-induced spallation technique, from its inception in the 1970s through the present day, is provided. An overview of the technique together with the physics governing the laser-induced spallation process, including functions of the absorbing and confining materials, are also discussed. Special attention is given to applications of laser spallation as an adhesion quantification technique in metals, polymers, composites, ceramics, and biological films. A compendium of available experimental parameters is provided that summarizes key laser spallation experiments across these thin-film materials. This review concludes with a future outlook for the laser spallation technique, which approaches its semicentennial anniversary. [DOI: 10.1115/1.4050700]

1 Introduction

In thin-film applications, the structural integrity and mechanical reliability of multilayered systems are governed by their interfacial properties. Thus, the ability to quantify film adhesion is essential for design and evaluation of layered media. Numerous experimental techniques have been employed by industries such as micro-electronics, aerospace, and automotive, including stud pull test, blister test, scribe method, tape-peel test, and many others [1]. By 1995, over 300 adhesion measurement techniques were known within the community [2]. For example, the tape-peel test, primarily employed to measure adhesion of coatings or paint layers on a substrate, has been successfully implemented for over 40 years to quantitatively assess adhesion between two films of interest [3,4]. However, it is challenging to extract a quantitative measure of adhesion strength. The tape-peel configuration imparts a mixture of tensile and shear forces, due to the angle of tape application, and must be performed under specific testing conditions while in direct contact with the sample [5]. Several other industrial adhesion measurement methods such as the scratch and stud pull test are also less quantitative and are primarily engaged for qualitative analysis of similar films [6]. Out of the need for noncontact, quantitative adhesion measurement methods that can characterize strongly adhered interfaces, several laser-based techniques, including the laser spallation technique, were developed.

The evolution of laser spallation begins with the generation of shock waves produced by laser vaporization of metal surfaces, which was presented for the first time in 1960 by Askaryon and Morez [7]. Their study on generation of high-amplitude stress waves in materials by impinging a high energy and pulsed laser beam on an unconfined surface was confirmed by White [8], Bell and Landt [9], Panarella [10], and Skee and York [11] between 1963 and 1968. In 1970, Anderholm [12] developed a technique for confinement of plasma and presented that stresses on the order of gigapascals (GPa) could be generated by placing a quartz overlay, transparent to the laser light pulse, on top of the substrate surface. The configuration confines the vaporized materials in the vicinity of the metal surface and significantly increases the peak stresses developed. During the next four years, a number of methods to increase the magnitude of these stress waves emerged, and several different transparent overlays were developed [12–17]. For example, Yang [18] reported the results of detailed experimental studies on peak stress generation as a function of film properties and laser parameters utilizing a transparent glass overlay and a variety of metal absorbers. When the amplitude and duration of this shock wave are sufficient, spallation will occur near the surface of a monolithic material or at the interface of film and substrate. Spallation is defined as “planar separation of material parallel to a wavefront as a result of dynamic tensile stress components perpendicular to this plane” [19]. Spallation due to laser impingement was introduced by Fox and Barr in 1974 who generated spallation within 1.0 mm thick monolithic 6061-T6 aluminum samples [20]. In 1976, the first measurement of interfacial adhesion strength by laser spallation was proposed by Stephens and Vossen [21]. In the laser spallation technique, a nanosecond pulsed laser, classically Nd-YAG, is utilized to create a high-amplitude compressive stress pulse in the substrate. The compressive stress pulse, on the scale of a few GPa, propagates through the substrate and then reflects as a tensile wave at the free boundary with sufficient magnitude to cause deformation of a monolithic material surface. For thin film–substrate systems, if the magnitude of the tensile stress wave at the film–substrate interface is greater than the interface bonding strength, then spallation of the film will occur (i.e., the film separates from the substrate) [19]. In 1988, Gilath et al. utilized the high-pressure and high strain-rate nature of the laser-induced shock waves to understand the stages of failure of aluminum foil including internal cracking, spallation, and target perforation [22]. Laser spallation research exploded from the 1990s to the very early 2000s, with several prolific groups contributing to its body of work. Some of the key
groups involved were led by Ali Argon at Massachusetts Institute of Technology [23], Vijay Gupta at Dartmouth University [23–25] and University of California at Los Angeles [26–28], Eric Auroux and Michel Boustie at Center National de la Recherche Scientifique in France [29,30], Nancy Sottos and Richard Weaver at University of Illinois at Urbana-Champaign [31–33], and Junlan Wang at University of California Riverside [34] and University of Washington [35,36]. During this time, Vijay Gupta also filed the first patent for the laser spallation technique in 1995 [37].

Significant advancements in laser spallation techniques were developed in the early 2000s which include adhesion measurement of submicron to a few micron thick films (0.1–10 μm) with strong interfaces, previously deemed impossible. Wang and coworkers [31,32,38,39] suggested that thinner film testing could be accomplished by loading the interface through nonlinear elastic substrates. Due to the nonlinear elasticity of the substrate such as fused silica, the laser-generated stress wave with an original Gaussian distribution will evolve into a decompression shock after traveling a certain distance, which will significantly increase the interfacial stress produced and the subsequent failure of the thin film/substrate interface [39]. The pulse-shaped shock waves successfully improved the efficiency of the laser spallation technique in interfacial testing of thin (~500 nm) low-dielectric films [38] and super strong tungsten/tungsten interfaces [40]. Grady et al. [41] benefited from the advantages of nonlinear elastic substrates in their work to measure interfacial adhesion of Au films on fused silica substrates functionalized with different molecular species. Further advancements by Wang et al. [31,33,34] included the expansion of interfacial loading from pure tensile stress to mixed-mode and pure-shear; thus, a full range of interfacial loading modes are now possible. In their method, mixed-mode loading was carried out by applying a prismatic substrate so that a high strain-rate shear wave is generated by mode conversion at the angled face of the prism and impinges upon the test film. Another significant advancement of thin-film spallation was the capture of real-time images of spallation evolution and measurement of spallation front velocity. In state-of-the-art laser spallation protocols, displacement measurement is confined to a single point or line using laser interferometry, and no images of the failure process are captured. Kingsstedt and Lambros presented a modified version of the laser spallation technique to capture the real-time and ultrahigh-speed images of the failure process of 100 nm thick aluminum films [42].

Advancements within the laser spallation technique have led to its applications for film adhesion of a variety of substrate/film systems such as metal/metal [29,40], metal/ceramics [24,43], metal/polymer [44], ceramic/ceramic [45,46], fiber/matrix [47], polymer/ceramics [41], ceramic/biomaterial [35], and metal/biomaterial [48], as well as adhesion of granular media/metal-coated-glass-substrates [49]. Recently, more novel applications of the laser spallation technique have arisen including cleaning of ceramics [50], adaption to measure adhesion of biological films [51,52], probing the mechanical response of micro-electromechanical systems under laser impulse loading [43,53], and others [49–51,54].

The resurgence of interest in laser spallation techniques within the last ten years has resulted in a dramatic increase in film adhesion publications and an expanding array of applications. The primary aim of writing this review article is to consolidate the available knowledge and experience to date in a comprehensive pedagogical publication for the first time. In this review, the physical aspects of generation and propagation of shock waves in the laser spallation technique as a function of laser parameters and mechanical properties of materials are described in Sec. 2. Section 3 addresses the measurement principles of the laser spallation technique including experimental setup and surface displacement diagnostics. Section 4 is focused on the applications of the laser spallation technique in metals, polymers and composites, ceramics, and biological materials. This review is concluded with a future outlook in Sec. 5.

## 2 Physical Aspects of Laser Impingement and Stress-Wave Generation

Stress waves are generated in laser spallation via optical breakdown, ablation, or rapid heating of an absorbing medium. Heating by rapid absorption is a mechanical process where a laser pulse with a short duration (nanoseconds) and high-power density (W/cm²) is focused on the surface of a solid target. A stress wave is generated by the rapid thermal expansion of the absorbing medium, whether the medium is free or confined. In direct laser–matter interaction, without any confinement, the laser-induced plasma will expand in air and become ionized, causing the laser energy to disperse far from the surface. If the sample is covered with a confining layer, depicted in Fig. 1, transparent to the laser pulse, the laser energy is absorbed at the sample surface. With little place to expand, the resulting plasma pressure becomes five to ten times greater than that from direct ablation, and the duration two to three times longer [56,57].

Due to the laser interaction with the absorbing layer, a sudden propagation of uniform pressure generates an assumed elastic compressive wave with a velocity dependent on the material properties of the substrate. This velocity, \( C_{s} \), the dilatational wave speed, is denoted as

\[
C_s = \sqrt{\frac{E'}{\rho}} \tag{1}
\]

where \( E' \) and \( \rho \) are the effective modulus and density of the substrate, respectively [58]. The effective modulus under plane strain assumptions, which is common in laser spallation techniques, can be determined by the linear elastic, isotropic, homogenous material’s Young’s modulus, \( E \), and Poisson ratio, \( \nu \), by the following equation:

\[
E' = E \left(1 - \frac{\nu}{2(1 + \nu)}\right) \tag{2}
\]

The generated acoustic wave is often assumed to be one-dimensional when the loading area is much larger than the substrate thickness. This compressive wave, depicted in Fig. 2, propagates through the substrate and then reflects off of the free surface as a tensile wave, similar to mechanics found in a Newton’s cradle.

Sections 2.1–2.3 describe optimal laser pulse characteristics, energy-absorbing materials, and confining layer materials. The selection of appropriate laser parameters, absorbing, and confining layers yield stronger plasma peak generation, and subsequently stronger stress waves can be generated. For strongly adhered films, high magnitude stress waves are essential to initiate delamination.

### 2.1 Laser Pulse Characteristics

The most common laser source used in laser spallation experiments is a Q-switched Nd:YAG laser [32,59,60], which typically operates on a wavelength range of 1055–1064 nm. Nd-doped glass (Nd:glass) [61] has been less commonly used as a laser source. A Q-switch accumulates energy in the laser-active medium, which is then quickly released, generating pulse durations on the order of a few nanoseconds. Typical pulse energy adopted in the laser spallation technique is in the range of 0.1–10 J with the spot size on the order of 10 mm; however, this spot size is often reduced using optics to 1–3 mm in diameter to increase the laser power density [32,40,48,52,59].

### 2.2 Absorbing Layer Material Selection

The main characteristics of an ideal energy-absorbing film that produces a large magnitude stress wave from an incident laser pulse are [62]:

1. high absorptivity
2. small penetration depth ("much smaller than the thickness of the film")
A variety of absorbing layer materials have been explored for laser spallation applications. These include aluminum [48], lead [63,64], gold [61], zinc [65], and titanium [66]. Aluminum is most frequently chosen, due to its relatively low cost, low toxicity, and a multitude of coating techniques able to generate a uniform layer thickness [67]. Details of the thermodynamic properties of these absorbing layers are tabulated in Table 1 [72].

Optical penetration or absorption depth, determined by the material and wavelength of irradiation, is important to minimize the heat generation by the laser. One of the most effective irradiation sources for laser-induced spallation is a Q-switched Nd:YAG laser which operates at 1064 nm. According to Table 1, titanium, aluminum, and gold, the most common absorbing layers, have penetration depths between 8.3 and 21.3 nm at 1064 nm wavelength. Such small characteristic absorption depths indicate that a majority of the laser pulse is absorbed in the skin depth of the material. In contrast, the higher optical absorption depth for materials such as germanium and silicon oxide, which are 220 nm [73] and 1120 nm [74], respectively, at 1064 nm, results in deeper energy deposition. The high depth of energy deposition in these materials results in weaker plasma formation, thus not suitable for this application. Under optimal absorption, the laser energy should be deposited at the interface of the confining layer and the energy-absorbing layer and not penetrate into the substrate. For this reason, the energy-absorbing layer is chosen to be an order of magnitude or more greater in thickness [32] than the penetration depth of the laser light. As seen in Table 1, the penetration depth of aluminum for a laser with wavelength of 1064 nm is approximately 8.3 nm. Several researchers showed that a 300 nm thick aluminum absorbing layer will provide sufficient amplitude to delaminate strong interfaces [48,75,76].

2.3 Confining Layer Material Selection. The choice of the confining material depends largely on the chosen substrate material as well as its transparency to the selected pulsed laser wavelength. Large acoustic impedance mismatch between the confining and substrate materials will ensure that generated acoustic waves propagate through the substrate and toward the film of interest [77]. For efficient and high-amplitude laser-induced stress-wave generation under the confined regime, the best features of the confinement material are:

1. high acoustic (shock) impedance
2. high dielectric breakdown threshold
3. excellent transparency to the laser wavelength

The laser-induced plasma within the confining material produces a high-amplitude compressive stress wave that propagates through the substrate. Equation (3) calculates how plasma pressure, \( P \), depends on the laser intensity \( I_0 \), the reduced acoustic impedance \( Z_{\text{total}} \), and the fraction of laser energy that contributes to plasma generation, \( z_a \) [57,78]:

\[
P(\text{GPa}) = 0.01 \left( \frac{z_a}{2z_a + 3} \right) Z_{\text{total}} \left( \frac{g}{\text{cm}^2 \cdot \text{s}} \right) I_0 (\text{GW/cm}^2)
\]  

The leading multiplier, 0.01, is applied to the above equation in order to scale the value generated to an appropriate magnitude [78–80]. The value generated, while not resulting in an exact stress-wave magnitude, offers a good approximation of expected magnitudes. Approximate values are often input into numerical modeling and provide initial guidelines before experimental values are obtained. The empirical coefficient \( z_a \), which is
determined experimentally, is normally between 0.25 and 0.4. The plasma length, \( l \), in the confining material is given by
\[
l = \frac{P\tau}{Z_{\text{total}}} \tag{4}
\]
where \( \tau \) is the laser pulse duration, \( Z_{\text{total}} \) (g/cm² s) is the reduced acoustic impedance, and \( P \) is pressure calculated from Eq. (3). The reduced acoustic impedance is calculated from
\[
\frac{1}{Z_{\text{total}}} = \frac{1}{Z_1} + \frac{1}{Z_2} \tag{5}
\]
and
\[
Z_1 = \rho_1 C_{d1} \tag{6}
\]
\[
Z_2 = \rho_2 C_{d2} \tag{7}
\]
where \( Z_1 \) and \( Z_2 \) represent the acoustic impedance of the confining and absorbing layer materials, respectively, which are functions of material density, \( \rho_1 \) and \( \rho_2 \), and dilatational wave speed within the material, \( C_{d1} \) and \( C_{d2} \), respectively. From the relationships in Eqs. (4)–(7), the energy deposited between materials with higher acoustic impedance will result in higher pressure [81]. For example, a 5 ns laser pulse with a power density of 10 GW/cm² for an aluminum absorbing layer \( Z_2 = 17.1 \times 10^5 \) g/cm² s confined with water \( Z_1 = 1.48 \times 10^5 \) g/cm² s results in a peak pressure and plasma length of 4.6 GPa and 17.4 \( \mu \)m, respectively. In this example, the confining layer should be at least 17.4 \( \mu \)m in thickness to generate the peak plasma pressure of 4.6 GPa.

Although water was the original material chosen for plasma confinement, a coating of aqueous sodium silicate, known as waterglass, provides an advantageous confining layer. Applications of laser pulses with higher intensities are possible because of the higher dielectric breakdown threshold for water and waterglass, compared to crystalline materials such as quartz. Parametric experiments show that depending on the substrate thickness, there exists a range of confining layer thicknesses from submicron to a few tens of microns that can produce a sufficient compressive pulse that propagates through the thin-film interface [82]. In the case of waterglass, many researchers have successfully conducted laser spallation experiments with waterglass confining layers whose thickness is less than the predicted plasma length. Waterglass is typically applied on top of the absorbing layer with either a brush or more precisely with a spin coater for thinner and more uniform thickness. A waterglass layer with a thickness less than the predicted plasma length does not adversely affect the ability to generate waves of sufficient amplitude to spall interfaces with a wide range of strengths. For example, Grady et al. [41] selected a 7 \( \mu \)m thick waterglass layer as the confining layer in experiments that successfully delaminated a polyimide interface, which exhibited adhesion strength on the order of 500 MPa.

### 3 Measurement Principles

With the principles governing the physics of laser-generated stress waves sufficiently discussed, Secs. 3.1 and 3.2 examine the practical application of the laser spallation technique. The discussion focuses on how the laser spallation experiments are commonly set up, Sec. 3.1, which includes safety protocols associated with high-energy laser generation and measurements of the substrate stress at debond through calibration experiments relying on laser interferometry, Sec. 3.2. Section 3.2 also includes the analytical equations and finite element analysis (FEA) vital to obtaining interfacial stress at failure, also known as adhesion strength.

#### 3.1 Experimental Setup

A typical laser spallation experimental setup including the stress-wave generation by the pulsed laser and detection by Michelson-type interferometric diagnostic system is shown schematically in Fig. 3. A single pulse with a spatial top-hat profile from the Nd:YAG laser impinges upon the energy-absorbing layer, which is coated on the back surface of the substrate. A spatio-temporal profile of the laser pulse should be provided with the Nd:YAG laser at purchase or can be determined with a beam profiler to determine the pulse shape. The rapid expansion and ionization of the absorbing layer due to confinement generates a high-amplitude acoustic wave that propagates into the substrate in compression. After reflection at the free surface of the test film on the front side of the substrate, the wave loads the test film–substrate interface in tension. Provided that over the central portion of the laser beam the incident energy density is uniform, a one-dimensional plane wave idealization can be adopted as in the following derivations [32].

The quintessential laser spallation test specimen consists of a transparent confining layer, a thin energy-absorbing layer, the substrate, and the test film (Fig. 3). If the test film is nonreflective, a thin (on the order of 100 nm) reflective coating must be implemented to generate significant signal during loading. To determine the displacement history of the free surface of the test film, the velocity is determined using a Michelson-type interferometer. An example of the standard laser spallation setup, which includes a variety of components to control and quantify stress-wave generation, is depicted in Fig. 4. The Nd:YAG laser pulse first passes through a variable attenuator, which can reduce the laser energy and results in a controlled range of energy pulse magnitudes. A focusing lens, specified by the pulsed laser wavelength and results in a controlled range of energy pulse magnitudes. A focusing lens, specified by the pulsed laser wavelength and energy, focuses laser light to an appropriate spot size for testing. According to work by Lev and Argon [83], the ratio of substrate thickness to laser spot size diameter is ideally equal to or less than 1.25 (likewise less than or equal to 2.5 if using spot size radius) to guarantee the least geometrical attenuation of amplitude and the highest stress in the central part of the pressure wave. The focused and energy-controlled laser pulse then contacts the substrate assembly, which generates a stress wave as discussed in Sec. 2. To obtain the transient displacement of the stress-wave loaded surface, a continuous wave diagnostic laser is employed in a Michelson interferometric setup. The continuous wave laser, which must operate in a single longitudinal mode, is collimated to reduce light dispersion, and is then split by a 90 deg cube beam splitter. Half of the beam travels to a fixed mirror, while the other half is directed toward the center of the stress-wave loaded region on the reflective sample, equal distance with the fixed mirror from the beam splitter. The two beams then recombine at the beam.
interested stress-wave signal. The typical displacement diagnostic precisely trigger the data collection for complete capture of the tor to respond to sudden changes in light intensity produced by order of GHz. The rapid rise and fall times enable the photodetector and digitized by an oscilloscope. Additionally, because interferometer patterns are detected on subnanosecond resolution, the interferometric fringes produced during laser spallation experiments have a rise time of a few nanoseconds. Oscilloscopes with sampling rates greater than 10 GS/s are needed in order to accurately resolve the fringes produced during the experiments. Additionally, because interferometer patterns are detected on subnanosecond resolution, the photodetector must have a rise and fall time on the order of picoseconds, which subsequently leads to a high bandwidth often on the order of GHz. The rapid rise and fall times enable the photodetector to respond to sudden changes in light intensity produced by light interference during surface displacement. The oscilloscope must be synchronized with the Nd:YAG laser Q-switch in order to precisely trigger the data collection for complete capture of the interested stress-wave signal. The typical displacement diagnostic laser is a stable linearly polarized solid-state diode but other high-quality linearly polarized laser sources such as He–Ne [84] or argon ion [85] have been successful. When setting up the laser interferometer, alignment of the diagnostic laser and the pulse laser on the sample is crucial for accurate displacement measurements. Any misalignment between the two will lead to displacement and subsequent stress values to be measured lower than those actually experienced during stress-wave loading. Laser absorbing paper can be used to help align the two lasers. Placement of an optical filter specially designed to block the pulsed laser wavelength before the beam splitter is an additional safety measure to prevent transmission of infrared light from contacting the cube. If the Nd:YAG pulse is allowed to reach the beam splitter, the optical adhesive will burn, which will require replacement of the beam cube. The highest risk for this occurrence is during the alignment process when there is often no sample in the laser path, or the laser is fired onto the same spot multiple times for glass substrate systems. Regular inspection of all optics is recommended to ensure good alignment and prevent damage of any equipment. When lasers are operated during the experiments, eye-wear safety is important. Nd:YAG lasers are hazardous for eye exposure (classified as class IV lasers), and over exposure from the solid-state laser (typically class IIIb) can also lead to retinal damage. It is important to wear eyewear with the recommended optical density and incorporate additional safety measures in consultation with laboratory safety personnel.

3.2 Surface Displacement Diagnostic. Movement of the sample surface during stress-wave loading causes a change in the voltage trace of the Michelson interferometer due to the Doppler effect. The voltage curve in Fig. 5(a) is a representative measurement recorded during stress-wave loading and includes the arrival of the mechanical wave resulting in the displacement of the free surface. In order to properly capture the stress-wave arrival at the free surface, a Q-switch laser must be set up in tandem with the oscilloscope to trigger data acquisition after the laser fires. The stress-wave arrival time at the free surface can be calculated by the ratio of the sample thickness to the dilatational wave speed of the material, which can be used to adjust the delay time on the oscilloscope so that only the initial stress-wave arrival is captured. For an example, 1 mm thick common glass with a dilatational wave speed of approximately 5000 m/s, the arrival time of the stress wave to the free surface is 200 ns. Because this value is sometimes greater than or equal to the entire oscilloscope capture window, a delay time is often programed by the user. This delay time is why it appears as if the interferometric fringes begin around 80 ns in Fig. 5(a) but in reality, the fringes for this glass specimen arrived at the photodetector 200–230 ns after voltage trigger was sent by the Q-switch laser. Thus, the time scale on the x-axis is somewhat arbitrary as there is a user-defined delay time between when the Nd:YAG fires and the oscilloscope begins data acquisition. In standard fringe analysis practice, the arrival of the stress wave is set as the zero point as in Fig. 5(b). The portion of the voltage curve where the mechanical wave arrives is isolated, Fig. 5(b), and is unwrapped to calculate the displacement over time from Eqs. (7) and (8), Fig. 5(c). Finally, using an analytical equation described below in Eq. (10), the substrate stress profile is obtained, Fig. 5(d).

The light intensity recorded by the photodiode detector that is related to the fringe count, \( n(t) \), is

\[
I(t) = \frac{I_{\text{max}} + I_{\text{min}}}{2} + \frac{I_{\text{max}} - I_{\text{min}}}{2} \left(\sin\left(2\pi n(t) + \varphi\right)\right) \tag{8}
\]

where \( I_{\text{max}} \) and \( I_{\text{min}} \) are the maximum and minimum intensities of the interference fringes, and \( \varphi \) is the phase angle. Once \( n(t) \) is obtained by Eq. (8), the free surface displacement \( u(t) \) can be determined by
One complete fringe shift corresponds to a displacement of $\lambda_0/2$ of the free surface, where $\lambda_0$ is the wavelength of light of the continuous laser. Since one whole fringe must be present to calculate the displacement, using a 532 nm laser, a minimum displacement of 266 nm can be measured using the interferometer. The compressive stress propagating through the substrate, $\sigma_{\text{sub}}(t)$, is calculated by using free surface displacement and the following equation:

$$
\sigma_{\text{sub}}(t) = \frac{1}{2} \rho C_d \frac{\partial u(t)}{\partial t}
$$

(10)

where $\rho$ is the density of the substrate, $C_d$ is the dilatational wave speed of the substrate, and $\partial u(t)/\partial t$ is the temporal velocity of the free surface, corresponding to the derivative of Eq. (9). For sufficiently thin films, with a thickness, $h$, that satisfies

$$
h \ll C_d \tau
$$

(11)

where $\tau$ is laser pulse rise time, a simple relation for interface stress is applicable. This relation is equivalent to Newton’s second law (the interface stress, $\sigma_{\text{int}}$, is the mass density of the film multiplied by the outward acceleration of the center of mass of the film) [32] which has the form

$$
\sigma_{\text{int}}(t) = -(\rho h) \frac{\partial^2 u(t)}{\partial t^2}
$$

(12)

Because Eqs. (10) and (12) are simplified versions of Eqs. (13) and (14), respectively, the latter can be used regardless of thickness and should be applied when unsure of satisfaction of Eq. (11). When displacement measurements cannot be made directly on test films due to their nonreflective nature (e.g., polymers, bio-materials, and rough films), displacement can be calibrated from measurements made directly on the substrate if reflective (e.g., silicon) or coated with a thin (e.g., 100 nm) layer of reflective material (e.g., aluminum). During calibration experiments, the same laser fluences used during spallation testing are impinged upon the calibration specimens so that multiple interferometer recordings are obtained, and calculated substrate stresses are consistent.

Other numeric models that can be adopted to derive interface stresses include wave transmission equations and finite element models. The stress at the thin film/substrate interface can also be calculated using wave propagation principles. For this purpose, one can consider two plates with a parallel arrangement and multiple back and forth reflections. At each interface, a portion of the wave is transmitted, and the remaining portion is reflected. The reflection, $R$, and transmission, $T$, coefficients at a substrate/film interface are given by

$$
T = \frac{2 \rho_1 C_1}{\rho_1 C_1 + \rho_2 C_2}
$$

(15)

$$
R = \frac{\rho_2 C_2 - \rho_1 C_1}{\rho_1 C_1 + \rho_2 C_2}
$$

(16)

where $\rho_1$ and $\rho_2$, and $C_1$ and $C_2$ are the densities and dilatational wave speeds of the substrate and film materials, respectively. If the materials for both the substrate and the film are known, and the substrate stress profile is known, these coefficients can be applied to determine the magnitude of the laser-induced compressive wave as it passes through the interface of the two materials [59,62].

Apart from the implementation of analytic equations to solve for interface stress, numerical methods have also been applied, including FEA [86]. FEA is a numerical method to simulate the response of the thin film by considering time-step and elements. Figure 6 shows the combination of spectral, cohesive, and volumetric elements to capture the film and substrate response due to stress-wave loading. Similar to the calibration protocol designed with the analytical equations (Eqs. (7)–(13)), the substrate stress profile serves as an input to a numeric simulation of each film within the system, resulting in interface stresses. Figure 6 depicts a complex FEA model. Simpler one-dimensional FEA models, as
well as the complex FEA models, have shown good agreement with analytical equations. As such, more complex FEA models have been abandoned in favor of one-dimensional models for a simple bimaterial interface under stress-wave loading [86].

4 Applications

High deformation rates, on the order of $10^3$/s, noncontact loading, and applicability to thick and thin films, make the laser spallation technique ideal for a broad range of film adhesion systems. In this section, a number of material specific applications of the laser spallation technique, and experiments previously performed using the technique, are discussed. Several interface systems are examined including metallic, polymeric, composite, ceramic, and biological films. Publications in this field have predominantly been produced by academic institutions, and its adoption within industries is relatively unknown possibly due to restrictions on publishing over proprietary concerns. The numerical details of published experimental testing parameters and results from adhesion measurements across major types of substrate–film combinations are summarized in Table 2. Significant advancements within the four major film types, metals, polymers and polymer composites, ceramics, and biological materials are each discussed below.

4.1 Metals. Many applications require thin metallic coatings for desired function such as reflection coatings, radiation protective coatings, and corrosion/erosion resistant coatings [100–102]. In 1976, the first laser spallation test of a metallic coating was reported by Stephens and Vossen [21] to investigate the adherence of metal films to glass substrates. Later advancements expand upon Vossen’s work and include adhesion measurements of multilayer systems, determination of the influence of textured surfaces on interfacial adhesion, mixed-mode loading, a dynamic fracture energy measurement, and paired molecular dynamics (MD) modeling.

In multilayer systems, there is a critical need to determine the reliability of thin-film stacks under mechanical loading. However, conventional tests often failed to delaminate the interface of interest within the stack. Gupta et al. extended the laser spallation test to multilayer systems [87] by coating tungsten and copper on Si substrates with a thin layer (2500 Å) of SiO$_2$, which is commonly found in integrated circuit devices. A schematic of the setup employed by Gupta et al. is shown in Fig. 7(a). It was demonstrated that sufficient tensile stress for interface separation could be generated in multilayer systems without an absorbing layer, while still avoiding cohesive failure. The measured interface strength, shown in Fig. 7(b), between a polyimide film and silicon nitride substrate demonstrates that as humidity is increased the interface adhesion strength decreases. Courapied et al. implemented laser spallation testing for thermal sprayed coatings of yttria-stabilized zirconia (YSZ) and intermetallic NiAl alloy on textured surfaces [88]. To determine improvements in coating adhesion due to laser texturing of a substrate surface, the adhesion of different coatings of NiAl on different pretreatment surfaces was compared using the laser spallation technique. They report that laser treatment of substrates improves the adhesion of NiAl films on 2017 aluminum and stainless steel SS304L substrates from 256 MPa for grit-blasting surface to 550 MPa for laser treated surface.

Similar to NiAl films, many film systems are so adherent; they require much higher tensile stresses to delaminate during an adhesion test. The ability of the laser spallation technique to generate different loading modes led to significant progress in creating higher tensile stress. In 2002–2004, Wang et al. reported interfacial strength of a thin aluminum film with thickness in the range of 0.5–1.8 µm on fused silica substrates measured using laser spallation with both tensile and mixed-mode loading [31,33]. As shown in Fig. 8(a), interference mixed-mode loading, the planar substrate is replaced with a triangular prism. Mode-conversion of the longitudinal wave occurs at the angled face of the prismatic substrate, generating a high-amplitude shear wave which propagates toward the test film. Due to the oblique incidence of the shear wave, a mixed-mode loading is obtained at the film/substrate interface, and the failure of the film is shown in Fig. 8(b). During these studies, a consistent interfacial tensile strength (~490 MPa) for different thickness of film (0.68–1 µm) on fused silica substrates was obtained [33]. Experiments performed under mixed-mode loading, and results from one such study are shown in Fig. 8(c). A higher interfacial strength was observed under mixed-mode loading (with an average value of shear stress 523 ± 52 MPa, along with a 227 ± 27 MPa tensile component), with a different wrinkling and tearing pattern [31].

In 2006, Hu and Wang further modified the sample geometry to introduce pure-shear loading at the film/substrate interface and obtained a much higher shear strength (720 ± 126 MPa) for the same Al/fused silica interface [34].

In 2008, Kandula et al. [103] developed a dynamic fracture test to delaminate thin strips of aluminum. By an energy balance approach, they measured interface fracture toughness as 5.6 J/m$^2$ for a 2 µm thick Al film on silicon substrates. Because interface fracture toughness can be difficult to measure by conventional methods, the adaptation of the laser spallation technique provided a new approach. Images of the unique failure that occurs from loading the thin Al strips is shown in Fig. 9(a). In 2009, Hu et al. used the laser spallation technique to measure the adhesion of tungsten films on tungsten substrates as well as the spallation strength of bulk tungsten [40]. They measured tungsten-tungsten interfacial strength as 875±61 MPa, and spall strength of bulk tungsten was estimated to be between 2.7 and 3.1 GPa. In 2014, Grady et al. studied the effect of self-assembled monolayer (SAM) interfaces on interfacial failure between gold films and fused silica substrates by laser spallation. Optical images of film failure of two SAM chemistries as stress increases are shown in Fig. 9(b). The black region demonstrates spallation of the film from the substrate, which increased with increasing tensile stress. They measured interface strengths of 19–80 MPa for SAMs with different end groups amine 11-amino-undecyltrimethoxysilane, dodecyltriethoxysilane, 11-bromo-undecyltrimethoxysilane, and 11-mercaptop-undecyltrimethoxysilane (Fig. 9(c)) and found that interfacial failure can be effectively controlled by SAM chemistry and laser fluence. By comparing Figs. 8(b) and 9(b), the different failure mechanisms for tensile and shear stress waves are quite obvious. While tensile stress results in a first crack nucleation followed by film rupture, mixed-mode loading results in slight film wrinkling in shear direction followed by film ripping.

Experiments needed predictive capabilities and major advancements in numerical modeling moved the laser spallation technique forward. In 2016, Awasthi et al. [104] implemented MD to determine the spallation response and nanoscale cohesive interaction of SAMs with different attachments to a gold layer. They
| Material type | Test film (thickness) | Substrate (thickness) | Absorbing layer (thickness) | Confining layer (thickness) | Laser type and parameters | Compressive stress-wave amplitude (GPa) | Peak interfacial tensile stress (MPa) |
|---------------|-----------------------|-----------------------|-----------------------------|----------------------------|---------------------------|-----------------------------------------|---------------------------------------|
| Metal         | Al (0.5–1 μm) [31]    | Fused silica (prism 3 mm × 3 mm × 15 mm) | Al (0.4 μm) | Waterglass (N.R.) | Nd:YAG 0.11 max 2.9 1 | 0.47 substrate shear stress | 227±27° tensile together with 523±52° shear (mixed-mode loading situation) |
|               | Al (0.6–3 μm) [32]    | Si (0.4–3 mm) fused silica (1–6 mm) | Al (0.4–1 μm) | Waterglass (50–100 μm) | Nd:YAG 0.11 max 2.9 1–2 | 0.55–2.3 for Si and 0.3–1.2 for fused silica | 60–100 for Si and 50–1000 for fused silica |
|               | Au (150 nm)           | Fused silica (1500 μm) | Al (0.4 μm) | Waterglass (1 μm) | Nd:YAG 0.044–0.075 10 2 | N.R. | 20–80° dependent on interfacial chemistry |
|               | Au (0.3–1.2 μm) [85]  | 45 deg fused silica prism (10 mm × 10 mm × 10 mm) with Si (730 μm)/Si3N4 (400 μm); Fused silica (500 μm) with Si (730 μm)/Si3N4 (400 μm) | Al (0.4 μm) | Waterglass (10 μm) for mixed-mode Waterglass (1 μm) for pure tensile mode | Nd:YAG 0.01–0.068 8 1.2 | 0.25–0.49 substrate shear stress | 142±5° tensile together with 426±15° shear (mixed-mode loading situation)245±15° (pure tensile mode) |
|               | Cu (2 μm) [87]        | Si (<1 mm) with SiO2 (250 nm) | Al (0.3 μm) | Waterglass (10–100 μm) | Nd:YAG 0.35–1.4 2.5 3 | ~0.8–3.5 | 320–730° |
|               | Cu (35 μm) [76]       | Ni alloy (235 μm) | N.R. | Waterglass (10–100 μm) | Nd:YAG 36–54 10 ~4 | N.R. | 3100 and 3200 as spall strength |
| NiAl (80 μm)  | 2017 Al alloy (N.R.)  | N.R. | Water | Nd:YAG (532 nm) | N.R. 7.1 N.R. | N.R. | 268–550° dependent on surface treatment |
| Polymer       | W (1–7 μm) [40]       | W (0.2–2 mm) with fused silica (0.5 mm) | Al (0.4 μm) | Waterglass (10 μm) | Nd:YAG 0.3 max 5 1–2 1–5.4 | 875±161° |
|               | W (0.5 μm) [87]       | Si (<1 mm) with SiO2 (250 nm) | Al (0.3 μm) | Waterglass (10–100 μm) | Nd:YAG 0.35–1.4 2.5 3 | ~0.8–3.5 | 110° |
|               | PBO (5 μm) [59]       | Si (725 μm) with Si3N4 (30–400 nm) | Al (0.4 μm) | Waterglass (10 μm) | Nd:YAG 131 5 1.6 | Up to 3.5 | 240–340° dependent on Si3N4 thickness and cure cycle |
|               | Polymide (2.5 μm) [86] | Si (<1 mm) with Si3N4 (750 nm) | Al (0.3 μm) | Waterglass (10–100 μm) | Nd:YAG 0.35–1.4 2.5 3 | ~0.8–2.3 | 140–304° dependent on humidity and time |
| Material type | Test film (thickness) | Substrate (thickness) | Absorbing layer (thickness) | Confining layer (thickness) | Laser source | Laser pulse energy (J) | Pulse duration (ns) | Spot size (mm) | Compressive stress-wave amplitude (GPa) | Peak interfacial tensile stress (MPa) |
|---------------|----------------------|----------------------|-----------------------------|-----------------------------|--------------|------------------------|-------------------|----------------|----------------------------------------|-------------------------------------|
| Polyimide (6 μm) [41] | Si (425 μm) with SiO2 (2.3 mm); Si (730 μm) with SiN4 (29 nm); Si (730 μm) with SiO2N4 (1 μm) | Al (0.4 μm) | Waterglass (7 μm) | Nd:YAG | 0.043–0.11<sup>a</sup> | N.R. | 2 | 0.2–2.6 | 275–429<sup>a</sup> for Si with SiO2, 150–375<sup>a</sup> for Si with SiN4, 175–390±30<sup>a</sup> for Si with SiO2N4, dependent on UV-exposure and cure cycle |
| Polystyrene (1.1 μm) [89] | Fused silica (1.5 mm) | Al (0.4 μm) | Waterglass (1 μm) | Nd:YAG | 0.057–0.15<sup>b</sup> | N.R. | 1.9 | Up to 1 | 112–223 |
| Polyurea (2 μm) [67] | Fused silica (3 mm × 3 mm × 20 mm) | Al (0.1 μm) | Waterglass (10 μm) | Nd:YAG | 0.2 max | 5 | 1 | Surface velocity reported | Energy dissipative properties reported |
| Polyurea (1000 μm) [90] | Stainless steel (0.72 mm) | Al (0.5 μm) | Waterglass (50–100 μm) | Nd:YAG | 6.5<sup>b</sup> | N.R. | 3 | 1.45 | 93±5 spall strength |
| Epoxy (1 μm) [91] | Fused silica (1.5 mm) | Al (0.4 μm) | Waterglass (1 μm) | Nd:YAG | 0.093–0.159<sup>b</sup> | N.R. | 1.5 | 0.9 min | 120–190 |
| Composite | Six ply T700/M21 CFRP (1.5 mm) [92] | N.R. | Water (N.R.) | Nd:Glass | 20 max | 30 | 4 | N.R. | N.R. debonding determined by laser intensity |
| | Four ply CFRP with adhesive paste (0.72 mm) [93] | Four ply CFRP (0.72 mm) | Black tape | Water (N.R.) | Nd:YAG | 0.4–1.2 | 8 | 4 | N.R. | 150<sup>a</sup> for weak bond area 340<sup>a</sup> for strong bond area |
| | Four ply CFRP with adhesive paste (0.72 mm) [93] | Four ply CFRP (0.72 mm) | Black tape | Water (N.R.) | Nd:YAG | 0.4–1.2 | 8 | 4 | N.R. | 150<sup>a</sup> for weak bond area 340<sup>a</sup> for strong bond area |
| Steel (700 μm) with polyurea (100 μm) [94] | E-glass composite (800 μm) | Al (0.5 μm) | Waterglass (50–100 μm) | Nd:YAG | 1.2<sup>b</sup> | 8 | 5 | 0.67 | 370±20<sup>a</sup> for polyurea/composite 486±20<sup>a</sup> for steel/polyurea |
| Ceramic | Diamond (84 μm) [95] | SiC (5 mm) | Silicon grease containing MoS2 particles (15–20 μm) | Silica (6 mm) | Nd:YAG | 0.041–0.2 | 3–5 | 0.16–1.88 | 0.1–0.5 | 1100<sup>a</sup> |
| | PZT–sol–gel film (160 nm) [96] | Si (325 μm) with SiO2 (500 nm) and ODS monolayer (~3 nm); Si (325 μm) with SiO2 (500 nm) | Al (0.4 μm) | Waterglass (6 μm) | Nd:YAG | 0.15 max | 8 | N.R. | ~0.35–1.05 | 55–480<sup>a</sup> dependent on surface treatment |
| Material type | Test film (thickness) | Substrate (thickness) | Absorbing layer (thickness) | Confining layer (thickness) | Laser source | Laser pulse energy (J) | Pulse duration (ns) | Spot size (mm) | Compressive stress-wave amplitude (GPa) | Peak interfacial tensile stress (MPa) |
|---------------|-----------------------|-----------------------|----------------------------|-----------------------------|--------------|-----------------------|-------------------|--------------|-----------------------------------------|---------------------------------|
| Biological material | YSZ (80 μm) [88] | Stainless steel 304 L (N.R.) | N.R. | Water (N.R.) | Nd:YAG | N.R. | 7.1 | N.R. | N.R. | 80–250° dependent on surface treatment |
| | ZrO₂ (20–140 μm) [97] | Stainless steel (1–2 mm) | Al (0.3 μm) or none | Waterglass (40–50 μm) | Nd:YAG | N.R. | 2.5 | 3 | N.R. | 271 b |
| | MC3T3-E1 Pre-osteoblast (1 μm) [51] | Fibronectin-coated polystyrene (300 μm) | Al (0.5 μm) | Water (10 cm) | Nd:YAG | 0.05–0.3 | 2.5 | 3 | N.R. | 34.9 b for fibronectin coated and 22.6 b for untreated |
| | MG 63 osteosarcoma [52] | Glass (1 mm) with smooth and roughened Ti (0.1 μm) | Al (0.3 μm) | Waterglass (5.5 μm) | Nd:YAG | 0.06–0.3 b | ~10 | 2.2 | 0.2–2.2 | 143 b for smooth Ti, 292 a for roughened Ti |
| | H19-7 neuron [35] | Si (N.R.) | Al (0.4 μm) | Waterglass (5 μm) | Nd:YAG | 0.3 max | 5 | 1–2 | N.R. | Cell detachment is reported as a function of laser fluence |
| | Pseudomonas aeruginosa [98] | NiTi stents and stainless steel screw | Ti (N.R.) | N.R. | Nd:YAG | 0.008–0.012 | 4 | ~7 | N.R. | Biofilm area is reported as a function of laser energy |
| | Streptococcus mutans (21–25 μm) [52] | Glass (1 mm) with smooth and roughened Ti (0.1 μm) | Al (0.3 μm) | Waterglass (5.5 μm) | Nd:YAG | 0.3 | ~10 | 2.2 | 0.2–2.2 | 320 b for smooth Ti, 332 a for roughened Ti |
| | Staphylococcus epidermidis biofilms [66,99] | Pig skin (N.R.) | Ti (0.5 μm) on polycarbonate (0.127 mm) | Waterglass (200 μm) | Nd:YAG | 0.66 b | N.R. | 3 | N.R. | 263.4 a |
| | Streptococcus mutans [48] | Glass (1000 μm) with Ti (0.1 μm) | Al (0.3 μm) | Waterglass (5.5 μm) | Nd:YAG | 0.06–0.3 b | ~10 | 2.2 | 1.7–3.5 | Biofilm detachment is reported as a function of laser fluence |

Adhesion strength.

bCalculated from reported laser fluence and laser spot size.

N.R. = not reported.
predicted the interfacial strength ratio of dodecyltriethoxysilane to 11-mercapto-undecyltrimethoxysilane to be about one-fourth which was quite comparable with Grady’s results [86] but MD magnitudes were about 50 times higher than the experimentally obtained results. Their simulation and experimental results showed that for those functional groups with weaker interaction with gold, the lower cohesive strength was observed and separation from the gold layer occurred without bond rupture.

4.2 Polymers and Composites. Polymers are ubiquitous in electronic, construction, automotive, and biomedical industries. Adhesion at the polymer/substrate interface is a complex subject, which includes polymer chemistry, polymer physics, and stress-fracture analysis. The presence of chain and cross-linking structures produce different deformation characteristics and adhesion mechanisms in polymer/substrate systems compared to metallic systems. Laser spallation, regardless of adhesion mechanism, offers an applicable method for macroscale adhesion measurement in film/substrate systems with multi-adhesive mechanisms such as those exhibited by polymeric films. In the following key publications, the laser spallation method was implemented to study adhesive strength, dynamic tensile stresses, and induced chemical reactions in polymer films of interest to micro-electronic packaging and other coating applications.

Kandula et al. [59] applied the laser spallation technique to measure the adhesive strengths of different formulae of 5 μm thick polybenzoxazole (PBO) with 30–400 nm Si₃N₄ interface in a multilayer thin-film stack. They examined the influence of dehydration procedures in film processing, substrate surface roughnesses, and PBO formulations on interfacial adhesion strength. As evident in Fig. 10(a), film failure progressed as stress increased. For these PBO films, delamination precedes spallation under stress-wave loading. Their work revealed that PBO formulation (type A and B) and surface roughness of the substrate correlate strongly with interface strength. They increased the film roughness by increasing Si₃N₄ thickness from 30 to 400 nm for both PBO formulas. They found that by increasing the roughness, the interfacial strength increased by 40 MPa for type A and 60 MPa for type B. Optical images in Fig. 10(a) show that while complete spallation occurs in films of type A (bottom row), films of type B (top row) only initiated delamination for the same Si₃N₄ thickness and laser fluence. Results of this study show that interfacial stress between Si₃N₄ and PBO changes as thickness of the sample increases.

Grady et al. [41] performed experiments to determine the interfacial adhesion of photodefinable polyimide films on passivated silicon substrates. In this research, consistency of substrate stress profiles was demonstrated by loading multiple times at single laser fluences and creating an overlap plot, shown in Fig. 10(b). They examined effects of different cure cycles and UV-exposure on...
film–substrate adhesion and showed that UV-exposure and longer cure cycle are necessary to have a well adhered film.

Spall strength is defined as the strength needed to initiate planar fracture (parallel to the backside of film) [105]. Youssef and Gupta [90] studied the dynamic tensile strength and constitutive behavior of polyurea films on steel substrates at ultrahigh strain rates by using ns laser-induced stress waves. At sufficient loading amplitude, the polyurea film spalls, and the damage to the film is evident in the side profile microscopy image in Fig. 10(c). At failure, a strain rate of $1.67 \times 10^7/s$ was calculated with the spall strength of $93.1 \pm 5$ MPa. As shown in Fig. 10(d), the tensile stress peak, which was obtained by finite element method using experimentally measured substrate stress profiles as input, was considered as the spall strength.

The early 2000s saw the introduction of mechanophores, molecules that undergo a chemical reaction due to mechanical loading [106,107]. In 2014, Grady et al. [89] demonstrated mechanical activation of a mechanophore-linked glassy polymer via laser-induced stress waves. In their research, activation initiated at stresses greater than 180 MPa, corresponding to a strain of 7% and fluorescence intensity increased with increasing applied stress, up to 200 MPa where saturation occurred. More recently, Sung et al. [91] developed a novel approach to activate mechanophores,
which are covalently attached to a solid interface (epoxy-fused silica), by loading with laser-induced stress waves. A schematic of the mechanophore-linked interface is illustrated in Fig. 11(a). They observed a sudden increase in florescence of mechanophores at an activation threshold of 149 to 163 MPa, where spallation occurred. Optical and fluorescence images with photopatterned epoxy (upper left) and fluorescence pattern epoxy (lower right) which show activation of interfacial mechanophore by loading are shown in Fig. 11(b). Measurement of energy-absorption response of thin films is another application of laser spallation. Jajam and Sottos [67] studied the energy-absorption behavior of polyurea through the surface velocity history in a mixed-mode laser spallation experiment. A value of 50–65% energy absorption was recorded at various laser fluences ranging from 157 to 217 mJ/mm².

In polymer matrix composites, the adhesion of polymer matrix and reinforcement material affects bond quality. Bond quantification is more accurate with the availability of a reliable noncontact method that can ensure the integrity of the joint [108]. Weak adhesion between composite layers can lead to poor mechanical properties and premature failure. Using the appropriate laser parameters such as spot diameter, pulse duration, and pulse energy, the laser spallation technique can be employed as a non-contact method to determine the different level of adhesion and bonding quality in polymer matrix composites. The induced debonding by the loading stress wave has been detected and quantified through different diagnostics systems such as interferometric confocal microscopy (ICM), ultrasound emission, optical microscopy, and X-ray radiography. Ecault et al. [92] and Perton et al. [93] utilized the laser spallation technique to test the mechanical quality of the fiber/matrix bonds in carbon fiber-reinforced polymer composites (CFRP) and laminate assemblies (four-ply laminate on top of another four-ply laminate), respectively. Crack propagation and layer separation occur during laser spallation of weakly bonded composites shown in Fig. 12(a). A schematic of the laser absorption and ply separation of a layered composite is shown in Fig. 12(b). Ecault and coworkers determined several different weak bonds in the sample containing two CFRP parts bonded with an adhesive film and proved that it is possible to discriminate different degrees of contamination in CFRP by laser spallation testing. They evaluated back face and bond damage threshold laser power density of a certain level of contamination as 0.49 GW/cm² and 0.66 GW/cm², respectively. ICM images of damage and deformation, in the z direction, that occurred at the back face of a CFRP sample are shown in Fig. 12(c). From the ICM images, the correlation between laser intensity and damage size can be observed. Perton and coworkers measured different adhesion strengths from two areas within the laminate, and the adhesion strength was evaluated by increasing the laser pulse energy. They found that good bonds were unaffected by certain levels of stress generation what would lead to failure in weaker bonds. In their study, a range of adhesion strengths from 150 to 340 MPa were measured. The depth and size of the debonds were determined by a post-test inspection through the laser ultrasonic technique. Adaptations to the laser spallation technique provide opportunities to determine composite bond quality within composite structures with less sophistication than is typically required for accurate adhesion strength measurement.

4.3 Ceramics. Since their introduction in the 1970s, ceramic films have been the material of choice in a number of coating applications such as thermal protection, corrosion protection, friction and wear reduction, and electrical insulation [109]. Metallic surfaces are covered in ceramic coatings through several methods such as thermal spraying (plasma spraying) and chemical vapor deposition. Unfortunately, crack initiation and propagation at the interface of coating and substrate may occur due to residual stresses or other driving forces [110]. In subsequence, quantifying the adhesive strength of thin ceramic films has been one of the major challenges since the first ceramic film application. Over time, significant research has been conducted to measure the tensile adhesion strength of the ceramic/substrate interface by laser spallation. In Sec. 4.3, major advances in adhesion strength measurement of ceramics such as zirconia, yttria-stabilized zirconia, lead zirconate titanate (PZT), and diamond, materials often widely employed in thermal barrier coatings, cutting tools, and ultrasonic transducers, are discussed.

![Fig. 11](a) Interfacial mechanophore specimen preparation. (b) Top row depicts optical images of 1-photopatterned epoxy and 2-maleimide-anthracene functionalized silica substrate, while the bottom row depicts the same images under fluorescence where the higher amplitude stress waves have activated the specimens, denoted by the fluorescence present. Each figure is adapted with permission from Sung et al. [91]. Copyright 2018 by American Chemical Society.
In 2004, Kobayashi et al. measured the interfacial strength of plasma sprayed zirconia (ZrO$_2$) coatings with a thickness that varied between 20 and 140 $\mu$m on stainless steel by the laser spallation technique [97]. A cross section of a ZrO$_2$ coating, which includes some large pores near the substrate, is shown in Fig. 13(a). ZrO$_2$ is widely used as a thermal barrier coating for high temperature protection of metallic structures. Higher interface strength, with an average value of 271 MPa, at shorter spray distance was observed, shown in Fig. 13(b). Ikeda et al. [95] was also able to employ the laser spallation technique, illustrated in Fig. 13(c), to achieve failure of vapor deposited diamond films. Failure of these diamond films are shown in Fig. 14(a). The calculated substrate stress from compression and free surface stress due to tension waves which reaches 1.1 GPa at a certain laser energy are shown in Fig. 14(b). This large magnitude of adhesion strength demonstrates one of the key advantages of laser spallation, which is the ability to fail very strong interfaces impossible by conventional tests [95].

Another ceramic material that is widely used as a thermal barrier coating is plasma sprayed YSZ. Like all plasma spray coatings, the preparation of the substrate prior to thermal spraying is required to promote strong adhesion between the coating and substrate. Courapied et al. [88] implemented laser-assisted surface texturing to control the surface topography of stainless steel before coating. Using the laser spallation technique, they showed that laser-assisted surface texturing improved the adhesion of a YSZ coating on stainless steel, which resulted in an adhesion strength increase from 80 MPa to 250 MPa. Relatively recently, Berfield et al. examined the adhesion strength of PZT sol–gel thin films on both octadecyltrichlorosilane (ODS) functionalized substrates and oxidized silicon (SiO$_2$/Si) [96]. Failure of the films resulted in rupture and tearing similar to failure observed in metallic films, Fig. 14(c). The two surfaces achieved remarkably different interface adhesion strengths, illustrated in Fig. 14(d), concluding that the adhesion strength of PZT sol–gel is largely determined by the functionalized surface it is adhered to.

### 4.4 Biological Materials

Many industries including marine, food production, and health are adversely affected by the adhesion of biological substances [111–114]. Bacterial biofilms, which consist of a viscous community of micro-organisms attached to any surface [112], are particularly harmful to these industries. For...
example, biofilms affect drag on ship hulls, contribute to contamination in food production, and can cause infection. Specifically, the formation of bacterial biofilms onto medical device surfaces poses a significant threat due to their increased antibiotic resistance [111] and typically need to be physically removed in order to properly treat. The ability to quantify the adhesive strengths of these films is critical in order to determine what surface characteristics will prevent strong adherence. Counting methods are the most prominent nonquantitative techniques used to measure the adhesion of biological films, but they fail to relate adhesion to a critical force [115]. Attempts to implement conventional critical force techniques require good grip on the biological material, which is slippery and has low cohesive strength [116]. The adaptation of the laser spallation technique to measure adhesion of biological films fulfilled the need for a quantitative, noncontact, critical force adhesion test. The first reports on laser spallation testing of biological materials appear in 2006–2008, followed by a gap in biological laser spallation research. In 2018, laser spallation testing of biological materials reemerged and expanded to include bacterial biofilms.

In 2006, Hu et al. [35] adopted the laser spallation technique to quantify the adhesion of neuron cells cultured onto a Si wafer; the setup used is illustrated in Fig. 15(a). Quantifying the adhesion of cells onto various surfaces is vital in the study of host response to...
differing medical devices. The study performed by Hu et al. was the first to report laser spallation experiments that evaluate the adhesion of biological materials. Experiments were able to initiate ejection of adhered cells from a silicon surface, as shown in Fig. 15(b). Even though the authors used critical laser fluence to signify adhesion strength (as opposed to determination of imposed stresses), they demonstrated that laser spallation is an effective method for investigating the adhesion between biological cells and inorganic surfaces. Since biological films preclude in situ interferometric measurements, fluence to failure was reported (Fig. 15(c)), which shows increasing critical fluence required as cell growth time increases. Hagerman et al. [51] employed an Nd:YAG laser system to measure adhesion strength of mouse pre-osteoblasts (MC3T3-E1) on polystyrene culture dishes. Stress-wave loading causes ejection of cells from the dish leading to a partially bare surface, while the surrounding cells remain adhered, depicted in Fig. 16(a), a red circle has been included to draw attention to area of spalled cells. While the adhesion strength values they measured using the laser spallation technique (22.6–34.9 MPa) were orders of magnitude larger than values they measured using jet impingement (50–150 Pa), the authors note laser spallation was more advantageous compared to jet impingement due to the noncontact nature of testing and rapid onset of loading and failure. Figure 16(b) demonstrates the ability of laser spallation to indicate strong and weak cell adhesions strength due to fibronectin and untreated surface, respectively.

More recently, Boyd et al. conducted laser spallation experiments to measure the adhesion strength of biofilms of Streptococcus mutans and MG 63 osteosarcoma cells on pure titanium [48,52]. The main purpose of this research was to determine factors that significantly influence macroscale adhesion strength of biofilms on dental implant surfaces. Specially designed substrate assemblies (Fig. 17(a)) were constructed that allowed for facile interchangeable substrate configurations. Laser spallation experiments found that sucrose concentration in growth media initially increased adhesion strength of S. mutans, and then caused a decrease in adhesion strength at higher concentrations [48]. Film failure before and after loading with increasing fluence is depicted in Fig. 17(a). Further experiments found that increasing surface roughness caused a significant increase in adhesion strength of MG 63 cell monolayers with minor increases found for adhesion of S. mutans biofilms [52]. The effect of surface roughness is

Fig. 16  (a) SEM image of region with detached cells after loading occurs, a circle is used to indicate spalled region; see online version for better image contrast. (b) Adhesion strength of cells on fibronectin-coated and uncoated surfaces; the fibronectin coating results in a larger stress needed for detachment. Each figure is adapted with permission from Hagerman et al. [51]. Copyright 2007 by Wiley.

Fig. 17  (a) Cultured biofilms with loading before and after indicated, multiple regions are loaded on a single biofilm to account for the variability associated with biological films, with Example substrate assembly inlaid (Adapted with permission from Boyd et al. [48]. Copyright 2019 by Springer Nature). (b) Evolution of failure for bacterial biofilms as well as cell monolayers with increased stress values on smooth and rough titanium (Adapted with permission from Boyd et al. [52]. Copyright 2021 by Elsevier).
Weibull analysis performed on failure of cells and bacterial biofilms at increasing interface strength; roughened titanium surfaces have a significant impact on cell adhesion but minimal effect on bacterial adhesion (Adapted with permission from Boyd et al. [52]. Copyright 2021 by Elsevier).

(a) Image detecting software is used to measure distance of testing spot from center of biofilm and a graph illustrating this effect on percentage of region spalled (Adapted with permission from Kearns et al. [117]. Copyright 2020 by American Chemical Society).

(b) Schematic of reflective panel attached to both smooth and roughened titanium surface in order to measure effect of stress-wave generation (Adapted with permission from Boyd and Grady [118]. Copyright 2020 by AIP Publishing).

(c) Average peak substrate stresses generated on both smooth and rough titanium using the reflective panel at two fixed fluences (Adapted with permission from Boyd and Grady [118]. Copyright 2020 by AIP Publishing).

Fig. 18 (a) Weibull analysis performed on failure of cells and bacterial biofilms at increasing interface strength; roughened titanium surfaces have a significant impact on cell adhesion but minimal effect on bacterial adhesion (Adapted with permission from Boyd et al. [52]. Copyright 2021 by Elsevier). (b) Image detecting software is used to measure distance of testing spot from center of biofilm and a graph illustrating this effect on percentage of region spalled (Adapted with permission from Kearns et al. [117]. Copyright 2020 by American Chemical Society). (c) Schematic of reflective panel attached to both smooth and roughened titanium surface in order to measure effect of stress-wave generation (Adapted with permission from Boyd and Grady [118]. Copyright 2020 by AIP Publishing). (d) Average peak substrate stresses generated on both smooth and rough titanium using the reflective panel at two fixed fluences (Adapted with permission from Boyd and Grady [118]. Copyright 2020 by AIP Publishing).
shown qualitatively by the initiation of spallation at higher fluence values in Fig. 17(b) and quantitatively by the failure statistics of each loaded fluence value in Fig. 18(a). During the initial stages of these studies, two enabling works were conducted validating laser spallation as an appropriate technique [117, 118]. The first study determined that there was no impact on adhesion strength based on loading regions within the biofilm from any internal film tension, Fig. 18(b). Because location, relative to the biofilm, played no role in adhesion measurements, multiple loaded regions could be tested on a single biofilm, increasing the throughput of biofilm adhesion measurements. The second is a study used to determine the effect of surface roughness on stress-wave propagation by adding special reflective panels, Fig. 18(c). This study concluded that the micron-sized roughness common in medical implants does not significantly impact stress-wave generation, indicating that smooth substrates of the same thickness and material are a valid substitute when interferometric calibrations cannot be performed on a rough substrate, Fig. 18(d).

Laser spallation of biomaterials is still a young field. There are some open questions and challenges that should be addressed. One of these is how cavitation in hydrated films affects stress-wave propagation. For example, traumatic brain injury researchers demonstrate a very low threshold for the generation of cavitation at the arrival of a compressive stress wave in brain surrogate materials [119]. The mechanism behind biofilm or cell removal under stress-wave loading may be due to cavitation, and more experiments in this area are needed. Another open question is how laser spallation adhesion measurements of biological materials compare to experiments like shear flow. Would adhesion moduli determined by laser spallation show the same trends when compared to experiments like shear flow. Would adhesion moduli be performed on a rough substrate, Fig. 18(d). A direct comparison of methods is needed. Finally, another challenge issued for researchers is how cavitation in hydrated films affects stress-wave propagation. For example, traumatic brain injury researchers concluded that the micron-sized roughness common in medical implants does not significantly impact stress-wave generation, indicating that smooth substrates of the same thickness and material are a valid substitute when interferometric calibrations cannot be performed on a rough substrate, Fig. 18(d).

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