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Effect of interface profile and incident wave characteristics on aluminum/epoxy dynamic adhesion strength

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ABSTRACT

The effect of interface profile on aluminum/epoxy adhesion strength under extreme dynamic loading condition is investigated. Test samples are prepared by depositing thick epoxy film onto polished aluminum substrates of roughness less than 200 nm. Failure is instigated at the interface by using laser induced stress waves. The adhesion strength of the film, evaluated by employing hybrid experimental/numerical approach, is observed to increase with decreasing interface roughness. To get more insight into the effect of interface profile and the incident wave characteristics on transient stress fields, two dimensional bi-material geometries are modeled with sinusoidal interface and the wave propagation analyses are performed. The simulations indicate increasing stress amplification at crest locations with an increase in the wavy interface amplitude (A_{int}). The interface wave length (λ_{int}) on the other hand shows a reverse trend. While the interface stress peaks appear to be uniquely dependent upon A_{int}/λ_{int}^2, the effect of asperity remains constrained to the vicinity of the interface. It is demonstrated that the stress amplification at wavy interface increases with increasing incident energy flux whereas the shape of the loading pulse has no effect on the interface stress magnitude. On the contrary the peak stress at a planar thick film interface is only influenced by the incident pulse amplitude and not by its shape or the energy content.

1. Introduction

Metal polymer bonded layers are used in several engineering components, ranging from sensors and miniaturized devices to automobile and aerospace structures. Often adhesion is preferred (over bolting and riveting) for joining dissimilar layers due to the method's advantage of achieving high strength to weight ratio, easier applicability, uniform load transfer, capability to join very small and/or intricate geometries and significantly reduced stress concentration. Inevitably, the layers' adhesion characteristics govern the reliability and the functionality of the bonded components. Prior to fabricating layered structures, the surfaces are chemically, electrochemically and/or mechanically pretreated to enhance interfacial adhesion. In the first two processes a thin layer of functionalized groups is placed at the interface to promote adhesion whereas in case of mechanical processing the surface interlocking is increased by developing controlled asperity. Adams et al. [1] have discussed a range of pretreatment methods and adhesion measurement techniques along with the design methodology and the mechanics of adhesive bonded joints.

The literature review on surface pretreatment processes indicates that the effect of interface asperity on the adhesion characteristics has been investigated by only a few researchers. Albers and White [2] reported that the knurling process on aluminum significantly enhanced aluminum/epoxy interfacial shear strength whereas phosphoric acid anodization in combination with silane treatment increased the interfacial tensile strength by several manifold. Uehara and Sakurai [3] used milling process to develop the roughness of few microns on metallic surfaces prior to bonding them. They conducted peel, pull and lap joint tests for analyzing their interface strength. They suggested that the maximum tensile and shear strengths were achieved at an optimal roughness value whereas the peel strength did not show specific correlation with the interface roughness. Teixeira et al. [4] also reported similar results in regards to the lap shear tests in which they used grinding process to vary the interface roughness. Budhe et al. [5] observed the maximum adhesion strength under shear loading at optimal roughness values of the epoxy bonded aluminum/wood layers. By employing double cantilever beam (DCB) for measuring aluminum/epoxy interfacial fracture energy, Zhang et al. [6] demonstrated enhanced fracture resistance at higher surface roughness values. They also indicated that the local mode-mixity, bridging and friction besides actual contact area played a major role in the fracture mechanisms. While the quasi-static test methods are commonly used in adhesion

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characterization, they pose several challenges, especially when the bonded layer dimensions are in microns and submicron range. Various adhesion measurement techniques pertaining to submicron films are reviewed by Mittal [7]. He discussed the potentials and limitations of each method along with the involved test complexities. Among a few studies conducted under dynamic loading conditions, Syn and Chen [8] reported that the energy dissipation at aluminum/epoxy interface increased with increasing interface roughness. They had performed four point bend tests by loading test samples using split Hopkinson pressure bar (SHPB). Ju et al. [9] employed SHPB setup to analyze energy dissipation in bonded rocks. They suggested that only beyond a critical roughness value the energy dissipation increased with increasing roughness at the joints.

In contrast to the relatively complex test procedures of the above discussed methods, the laser spallation technique dynamically loads the interface in non-contact fashion where the substrate is subjected to a strain rate of the order $\sim 10^7$/s. In addition the sample preparation process as well as the data reduction involved with this method is very simple. The technique was introduced by Yang [10] and Vossen [11]. Gupta et al. [12,13] further developed the method and incorporated Michelson interferometer in combination to perform in situ out-of-plane displacement measurements. The method has been successfully employed in past by Gupta et al. [12,13] and Sottos et al. [14–16] to evaluate the interface strength of metallic, polymer, ceramic and multilayer low dielectric films. Bossi et al. [17] extended the laser spallation method to thick bonded layers and relatively thick interfaces. They demonstrated its applicability to characterize weak adhesion (kissing bonds) of composite joints by inducing interfacial failure in graphite/epoxy laminates of variable adhesion strength. They also showed a correlation between the failure laser fluence and the tensile and fracture parameters. Arrigoni et al. [18] employed the method in combination with Fabry-Parot interferometer to analyze the effectiveness of various adhesive layers in epoxy bonded aluminum joints. They reported that the fast fourier transform (FFT) of the free surface velocity signals obtained from the damaged samples were distinctly different from the ones observed in the undamaged interface cases.

The above reported studies were limited to the bonded surfaces with the roughness values more than a few microns. Also, the investigations did not demonstrate precise adhesion measurements when the layer dimensions were in tens of microns. In current investigation a systematic study is performed to quantify the effect of interface asperity characteristics on aluminum/epoxy bonding strength under extreme dynamic loading conditions. Test samples with less than 200 nm interface roughness are prepared and the experiments are performed to instigate interfacial failure by using laser induced high amplitude short duration stress pulses. In-situ displacement history, obtained by performing interferometric measurements, in combination with computational (transient) stress analysis is used to assess the adhesion strength. Additional computational analyses are performed to get more insight into the effect of interface asperity and the incident pulse characteristics on transient stress fields.

2. Material characterization and sample preparation

Bars of Al 6063-T6 aluminum alloy are machined (milled) into 750 μm thick plates, followed by cutting them into 25 mm × 25 mm square samples. The aluminum substrates are polished by using abrasive slurry to develop a range of surface finish. The polishing material consisted of the sand papers and the alumina powder of grit sizes ranging from 1 μm to 35 μm. The representative specimen surface profiles, scanned by using stylus type profiler (Dektak-XT), are shown in Fig. 1(a). The root mean square (RMS) roughness value, R_q, of the prepared surfaces is calculated by applying,

$$R_q = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(y_i - \bar{y})^2} \tag{1}$$

where $\bar{y}$ is the distance of the average surface profile from the reference axis and y_i represents the abscissa of the ith digitized data point on the asperity. The substrates with five different roughness values, ranging from 35 nm to 530 nm, are considered in the current investigation. The R_q corresponds to each of the surface profiles are illustrated in Fig. 1(a). The roughness values are also tabulated in Table 1. The surface profile with R_q = 530 nm corresponds to the unpolished (only milled) sample whereas the others are developed by polishing the substrate.

The test specimens are prepared next by depositing an epoxy layer on top of the polished aluminum surface. First, Diglycidyl Ether of Bisphenol A (DGEBA) epoxy resin is mixed with Tri-ethyl tetra amine (TETA) curing agent in 10:1 ratio by weight and stirred slowly for about 20 min. Care is taken to avoid the entrapment of air bubbles into the mixture. Few drops of epoxy are placed onto the substrate. This is followed by adjusting a cellophane tape wrapped glass plate on top of it and putting a pre calibrated dead weight onto the glass plate to achieve the epoxy film of desired thickness. The entire assembly is left at room temperature in humidity controlled environment for about 15 h to ensure the uniform curing of the epoxy layer. The aluminum/epoxy specimen is carefully detached from the covered glass plate and placed on a dry flat surface for at least 15 days. Prior to conducting the experiments the back surface of the aluminum is spin coated using a sodium silicate solution (water glass) such that a 10 μm confinement layer is achieved. The layers of a test specimen are illustrated in Fig. 1(b). Epoxy films of thickness (h) varying between 12 μm to 30 μm are considered in this investigation. In addition the calibration specimens are prepared by depositing 100 nm aluminum film (using thermal evaporator) onto the mirror finished (R_q = 35 nm) aluminum plate which is followed by spin coating a water-glass layer at its back, similar to the actual test sample case.

For conducting experimental and computational analysis, the physical and the acoustic properties of constituent materials are measured separately. The density (ρ) of epoxy and aluminum alloy, calculated by weighing 10 mm × 10 mm × 10 mm cubes, is tabulated in Table 2. Ultrasonic thickness gage Parametrics 35DL (OLYMPUS), in combination with 10 MHz longitudinal and 5 MHz shear wave transducers (M112-RM and V154-RM, respectively) are employed to measure the wave speeds using pulse echo method. The longitudinal (C_d) and shear (C_s) wave speeds in the epoxy and aluminum are presented in Table 2. The dynamic elastic modulus (E_d) and the Poisson's ratio (ν_d), determined by applying the following plane strain equation, are also included in the table.

$$C_d = \sqrt{\frac{E_d(1-\nu_d)}{\rho(1+\nu_d)(1-2\nu_d)}}, \quad C_s = \sqrt{\frac{E_d}{2\rho(1+\nu_d)}}. \tag{2}$$

3. Experimental details

The optical set up used in current investigation is schematically illustrated in Fig. 2. In the figure, S and F represent the substrate and the film, respectively. A 5 ns Gaussian pulse of variable energy content (0–300 mJ) from Q-switched Nd:YAG laser (λ = 1064 nm) is focused onto the water-glass constrained aluminum substrate. The localized ablation at the back surface of the aluminum develops high amplitude compressive stress pulse which propagates towards the film. The stress wave, transmitted into the epoxy, gets mode converted into a tensile pulse at the front (free) surface of the film, subjecting the development of tensile stresses at the substrate/film interface. The laser energy is gradually increased until the interfacial failure is detected.

In-situ transient out-of-plane displacement is measured at the free surface of the film by employing Michelson interferometer as shown in Fig. 2. Components of experimental setup are adjusted such that the