

# Adhesion Strength of Al/Epoxy Resin Interface over a Wide Range of Loading Rates

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**Abstract.** This study evaluated the interfacial adhesive strength between aluminium alloy and epoxy resin (Al/epoxy resin) over a wide range of strain rates (loading rates). We conducted three types of tests with different loading rate, i.e., a quasi-static tensile test for the range of lower loading rate, a Split Hopkinson Bar (SHB) for the range of middle loading rate, and Laser Shock Adhesion Test (LaSAT) for the range of higher loading rate. LaSAT is a unique test of adhesion evaluation, since laser induced shock wave is employed to lead interfacial fracture. In parallel, finite element method (FEM) is conducted in order to calculate stress distribution at the interface during LaSAT. As a result, it was found that the interface between the aluminium alloy and the epoxy resin interface shows significant loading rate dependency of the adhesion strength and this tendency is very similar to that of bulk epoxy materials.

## 1 Introduction

Adhesive bonding with a polymer material has various advantages such as significantly reduced stress concentration and higher strength to weight ratio of component. Therefore, such a bonding method has been widely used in various engineering components. In actual use, these components are not only subjected to static loading but also dynamic and impact loading. Deformation and fracture of such a bulk polymer including resin adhesive are well known to significantly depend on loading rate (i.e., strain rate sensitivity: SRS) [1]. Therefore, it is expected that the interfacial strength (adhesion strength) shows loading rate dependency due to SRS of resin adhesive. To elucidate this mechanism, it is essential to quantitatively evaluate the strength of the metal/resin interface under various loading rates. Experiments of dynamic and impact loading including Split Hopkinson Bar (SHB) method were well conducted previously, and the strain rate ranges from  $10^1$  to  $10^3$  s<sup>-1</sup>. However, the study on the interfacial strength at high-speed loading such as the strain rate of the  $10^{6\sim7}$  s<sup>-1</sup> has not been conducted so far. Recently, non-contact testing methods using pulsed laser for shock

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wave fracture have been developed [2]. This method is called Laser Shock Adhesion Test (LaSAT) [2][3], which employs laser ablation to generate strong elastic waves. Tensile stress is applied to the interface and delamination (interfacial fracture) occurs. The stress at the interface is calculated using numerical simulations such as the finite element method (FEM). As a result, the interfacial strength under impact loading can be quantitatively evaluated. Thus, it is expected that the quasi-static test, SHB test and LaSAT can be used to evaluate the interfacial strength over a wide range of loading rates. This information will be useful for the design of adhesives under various load factors. Finally, a comparison of the results of the quasi-static tensile test, SHB test and LaSAT was made to discuss SRS of adhesion strength.

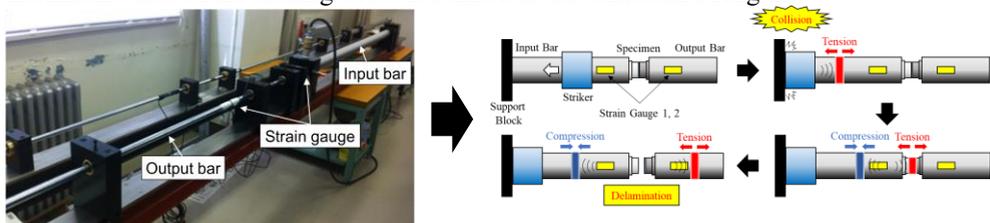
## 2 Sample and Experimental Details

### 2.1 Split Hopkinson Bar Test

#### 2.1.1 Methods of SHB Test

Fig. 1 shows a photograph of the SHB test apparatus. The split-Hopkinson bar tensile test apparatus consists of a coaxially lined impact tube, input bar, output bar, and support block. The specimen is mounted in between the input and output bars. The striking tube is a straight circular tube. Strain gauges are separately attached on the input and output bars, and the strain signals are amplified by a bridge circuit and amplifier, and then recorded using the oscilloscope. A compressed air system with a compressor was used to launch the striker, and the air pressure was set to 0.020 MPa. In addition, four polyethylene films (30 mm in outer diameter and 20 mm in inner diameter) were inserted between the impact tube and the support block as buffer material.

The mechanism of the SHB test is also shown in Fig. 1. First, the striker is launched by compressed air and collide with the left end of the input bar. This generates an elastic tensile stress wave in the input bar, propagating towards the specimen. When the wave reaches the left side of the specimen, a part of wave is transmitted through the specimen, and the rest of the wave propagates as a reflected wave in the input bar. The transmitted wave then propagates in the output bar. When the transmitted wave reaches the adhesive bonding interface between the aluminium alloy and epoxy resin, the tensile stress is loaded to the interface. At the critical moment, delamination occurs at the interface. Each strain value of the input bar and the output bar is recorded by strain gauge (#1 and #2). From these strain values, the loading stress-strain diagram and the strain rate-strain diagram are obtained. The maximum value of the loading stress is defined as the adhesion strength.



**Fig. 1** Experimental setup and overview of SHB Test.

#### 2.1.2 Specimens of SHB Test

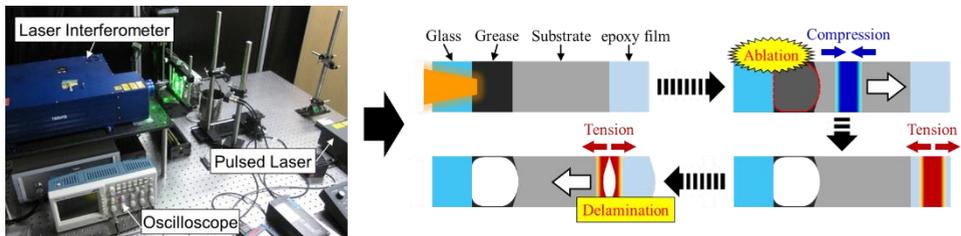
This study established a method to carefully adjust the thickness of the epoxy resin layer and its axiality of tensile loading. First, the specimen after bonding was clamped in a developed

jig and a vise to ensure the axiality. Furthermore, the resin layer thickness was measured using a digital microscope. After adjusting the thickness of the adhesive layer to  $100 \pm 10 \mu\text{m}$ , the bonded specimens were left for at least two days to complete the bonding process. To prevent resin leakage from the bonded surface, polyimide adhesive tape was wrapped around the edge side near the interface during resin curing.

## 2.2 Laser Shock Adhesion Test (LaSAT)

### 2.2.1 Methods of LaSAT

Fig. 2 shows an experimental setup of the laser shock adhesion test (LaSAT). The Nd:YAG laser (Tempest 300, New Wave Research) irradiates pulsed laser (wavelength 1064 nm, pulse duration 3-5 ns) towards the grease as shown in Fig. 2. The grease, which is composed of silicone oil and graphite powder, absorbs energy from the pulsed laser and expands rapidly due to the phase transition from the liquid phase to the gas or plasma phase (laser ablation). This phenomenon causes shock wave, propagating in the substrate. The shock wave propagates through the substrate toward the epoxy resin surface. At the free surface of epoxy film, the wave mode is converted from compressive stress into tensile stress due to free edge reflection. The converted shock wave applies tensile stress to substrate/film interface. The magnitude of the tensile stress can be controlled by the pulsed laser energy. Therefore, the laser energy (where a delamination or failure is first detected) can be defined as the critical laser energy.



**Fig.2** Experimental setup and overview of LaSAT.

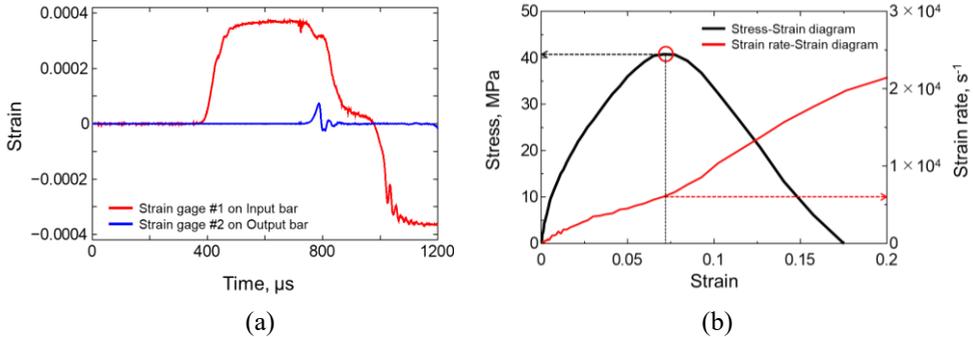
### 2.2.2 Specimens of LaSAT

This study used a bilayer specimen composed of an aluminium substrate and an epoxy resin layer to mimic the Al/epoxy resin interface. The aluminium alloy substrate (thickness 3 mm) was cut into a plate shape with the dimensions of  $75 \text{ mm} \times 50 \text{ mm}$ . The surface was polished with buff and cleaned with ethyl alcohol. Next, epoxy resin (bisphenol A-type) was mixed with curing agent in a 1:1 ratio by volume. During the mixing, we ensure that air bubbles were not entrapped in the mixture. The epoxy resin was applied to the substrate surface, and  $100 \mu\text{m}$  spacers were placed at the four corners, and a PVC plate was pressed against the substrate from above, so that we control the film thickness to be  $100 \mu\text{m}$ . The specimens were left for two days to fully cure in an environment where the room temperature was controlled at  $20^\circ\text{C}$ .

### 3 Results

#### 3.1 Results of SHB test

Fig. 3 (a) shows a transition of measured strain during SHB test, and calculated stress-strain diagram in Fig.3 (b). The interfacial strength was found to be 40.8 MPa and the strain rate just before failure was  $5.69 \times 10^3 \text{ s}^{-1}$  as shown in Fig. 3 (b). In the same way, a total of five tests were conducted, and the data is shown in Table 1. The averaged adhesion strength was 43.2 MPa and the averaged strain rate just before failure was  $5.52 \times 10^3 \text{ s}^{-1}$ .



**Fig. 3.** The results of transition strain at each strain gage (a) and stress-strain and strain rate-strain curves (b).

**Table 1.** Conditions and results for each test specimen.

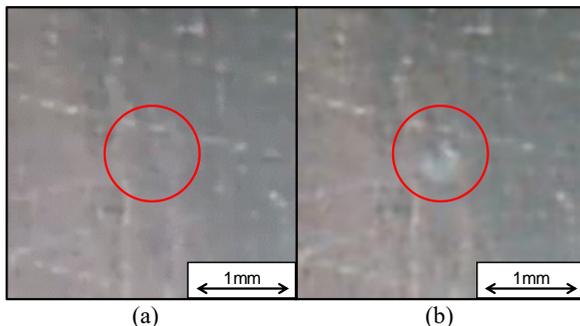
Test number	Test 1	Test 2	Test 3	Test 4	Test 5	Ave.	±
Epoxy's thickness [μm]	108.9	104.6	104.0	106.4	106.1	106.1	1.73
Maximum stress [MPa]	42.8	47.7	40.8	39.2	45.5	43.2	3.08
Strain rate [s <sup>-1</sup> ]	4130	6180	5690	5310	6290	5520	779

#### 3.2 Results of LaSAT

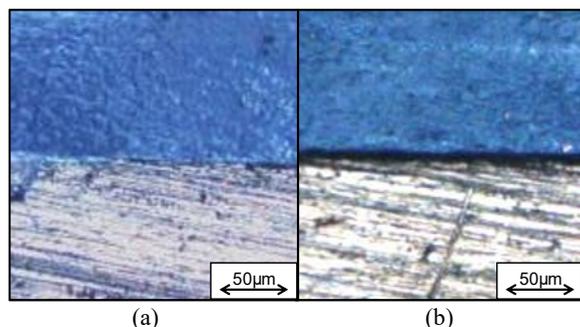
During LaSAT, the delamination was detected by observing the interface from the epoxy resin surface using a microscope. Fig. 4 (a) and (b) are pictures of the interface taken with a microscope before and after the LaSAT experiment. Fig. 4 (b) shows that a small cloudy white circle is observed after LaSAT. Its diameter was about 300 μm, which was taken as a sign of delamination occurrence. To confirm this, the cross sections before and after the observation of the cloudy circles were observed under an optical microscope. These photographs are shown in Fig. 5 (a) and (b). A clear difference can be observed near the interface, Fig. 5 (b). Therefore, it can be said that this method is effective for identifying the timing of epoxy resin delamination and determining the critical laser energy for delamination to evaluate adhesion strength.

A FEM simulation of wave propagation was performed. Note that this simulation method is based on our previous studies [4]. From the results, it can be seen that the front waves of the compressive stress components were reflected at the free surface due to free edge reflection, and reached the interface again as the tensile stress component. When it reached a critical

value, delamination occurred. The maximum value of tensile stress was 90.2 MPa from the FEM computation. Therefore, the adhesive strength was estimated to be 90.2 MPa.



**Fig. 4.** Micrograph of epoxy resin surface before (a) and after (b) the delamination was detected.

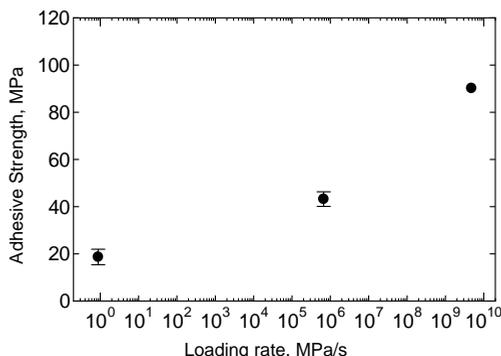


**Fig. 5.** Optical micrograph of cross section of room temperature cured specimen before (a) and after (b) the delamination.

### 3.3 Effect of strain rate dependence

Fig. 6 shows the summary of obtained results from quasi-static tensile test, SHB test and LaSAT. It was found that the adhesive strength increased with increasing loading rate. This is due to the fact that the yield strength of epoxy resins, such as polymeric materials, is generally known to increase due to strain rate dependence [1]. This comes from a change in deformation behaviour of molecular structure. They evaluated larger yield strength of epoxy resin at the strain rate of  $10^3 \text{ s}^{-1}$ . In our LaSAT, the applied interfacial stress is lower than the yield strength of epoxy resin, suggesting that the failure occurred in the elastic deformation range.

In addition, Yildiz, S. et al. found similar phenomenon of adhesive strength, which is strongly dependent on strain rate. They conducted unique experiment of shock wave loading, so that they evaluate adhesive strength of epoxy resin/aluminium and steel joint components at the level of  $10^3$  to  $10^4 \text{ s}^{-1}$  [5]. This trend is similar with our interfacial strength as shown in Fig. 6. Indeed, our LaSAT enables us to evaluate adhesion strength at very high loading rate, i.e., around  $10^9 \text{ MPa/s}$  (same order with  $10^6 \text{ s}^{-1}$ ). Such a higher strain rate is not reported so far for adhesive evaluation and discussion of the mechanism. This paper may be a first report of adhesion strength of such a higher strain rate and reveal SRS of adhesion strength over a wide range of loading rate.



**Fig. 6.** Comparison of between adhesive strength of quasi-static tensile test, SHB test, and LaSAT for the interface between epoxy resin adhesive and aluminum alloy.

## 4 Conclusion

This study evaluated the interfacial adhesive strength between aluminium alloy and epoxy resin over a wide range of strain rates. The details of our results are summarized below.

- (1) The LaSAT experiment quantitatively evaluated the adhesion strength. In parallel with the experiment, we observed the delamination with a microscope and identified the delamination of the white circle using an optical microscope.
- (2) Dynamic analysis of LaSAT for elastic body was conducted using FEM. The data from the experiment was input to the FEM computation and the interfacial stress was estimated to obtain adhesion strength.
- (3) Interfacial strength (adhesion strength) of aluminium and epoxy resin was measured by the quasi-static test, SHB test and LaSAT. It was found that the adhesion strength showed significant strain rate dependency which is similar with a general homogeneous bulk polymer.

## References

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