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Quantitative evaluation of adhesion quality of surface coating by using pulse laser-induced ultrasonic waves

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ABSTRACT

This study aims to evaluate delamination characteristics of surface coating by using strong ultrasonic wave induced by pulsed laser irradiation. Strong tensile stress wave is induced by pulse laser irradiation, and delamination of the coating layer/substrate interface (or spallation) is produced by confined silicone grease breakdown (i.e. ablation). Upon various levels of energy of irradiation laser, the coating delamination and displacement are measured in-situ. Parallel computation of elastic wave propagation using FDTD (finite difference time domain) yields the wave propagation in the specimen, and estimates the interfacial strength with stress wave distribution. The delamination area is visualized by laser ultrasonic wave scanning technique after the laser spallation test. The interaction between displacement waveform and delamination is explored. The present technique may shed some light on various coating depositions for assessing the coating adhesion quality.

1. Introduction

Thin films and surface coatings are widely used for wear/contact resistance, which plays a protective role against a soft/ductile substrate. In use, delamination or exfoliation is often experienced due to weak bonding, driven by thermal stress and stress concentration at the edge. Bonding quality is always important and such material integrity imposes a challenge during the process of deposition of films and coatings. Quantitative evaluation of bonding quality through effective and rapid mechanical testing is strongly demanded in industry. An overview of several adhesion testing methods including peel, stud-pull, scratch, bulge, and four-point bending was given by Lacombe [1]. In general, peel tests imposes a high tensile stress to the coating, which may rupture the coating prior to interfacial delamination. In a stud-pull test, a rigid metallic stud is bonded to the target coating with epoxy and then pulled at a controlled rate until interfacial failure occurs; however, if the adhesion of coating is strong, stud separation may occur before the desired interfacial delamination. Interfacial fracture toughness may also be extracted from four-point bend [2,3], blister [4] or superlayer [5–7] tests. The effectiveness and repeatability of the test are strongly dependent on generation and propagation of a pre-crack along the interface of interest. Specimen preparation is often time consuming due to the difficulties in introducing the pre-crack. Blister and superlayer techniques also require significant sample preparation and are not practical for rapid interface characterization.

The laser ultrasonic wave and laser spallation technique [8–27] appears to be a promising way of fast and efficient measurement of interfacial strength. High amplitude laser-induced acoustic stress pulses are employed to dynamically load a film/coating interface. Vossen [22] initially developed the spallation technique to estimate the bond strength of millimeter thick aluminum (Al) films on substrates using a high energy laser-induced acoustic pulse loading. An absorbing layer was first coated on the back side of the substrate, and a laser pulse impinging the absorbing layer generates a strong acoustic pulse. The advantage of this technique is use of the pulse wave, inducing the coating delamination in a non-contact manner. Gupta et al. [12–14] and Yuan et al. [15–18] extended Vossen's technique to investigate a wide variety of thin film interfaces. They determined the critical laser power at which the spallation initiated, and extracted the interface strength. One issue is that the measured strength at the level of coating spallation is possibly overestimated, since small delamination first occur prior spallation (i.e. complete delamination and is followed by coating fracture). In fact, most previous studies used high laser power, inducing a large coating

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spallation clearly (thus, this technique is called laser "spallation" technique).[1] In addition, the interfacial strength was estimated through a combination of experimental calibration and corresponding one-dimensional (1D) wave propagation simulations. More recently, Ito et al. [28] conducted simulation of elastic wave propagation using FDTD (finite difference time-domain method) with two-dimension, to improve the estimation of the interfacial stress. In the light of technical development, the evaluation process for interfacial strength by laser spallation technique is still challenging. Specifically, both detection of delamination and calculation of interfacial stress are critical.

In this work, we improve the laser spallation technique to measure the adhesive strength of hard coating on steel substrate. As a representative material, electroplated Ni coating deposited on stainless steel substrate is used in this study. In order to evaluate the interfacial strength accurately, new detection techniques for delamination and stress computation procedure are explored. The former one is the use of a correlation factor, which captures the variation of elastic waveform (output wave) since the existence of delamination may affect the output wave. In addition, scanning technique with laser ultrasonic wave (called laser scanning) was also employed to visualize the delamination area. The latter (stress computation) is an improved analysis based on the stress wave propagation by FDTD (similar with Ito et al. [28].). The main objective of the present paper is to further improve the accuracy of laser spallation technique for measuring interfacial strength of various coating systems.

2. Experimental setup

2.1. Laser spallation technique

The present setup for laser spallation technique is shown in Fig. 1. A pulse laser generated by a Q-switched Nd: YAG laser (Tempest 300, New Wave Research) irradiates onto the break down layer, which is formed on the back surface of the substrate. For the YAG laser, the wavelength is 1064 nm. In addition, the pulse duration of half bandwidth is about 5 ns (3–5 ns) and the rise time is 5 ns. The laser is focused to a circular spot of 3.0 mm on the break down layer to induce ablation. This layer is molybdenum disulfide (MoS_2) with viscosity grease. The grease layer with thickness of 50–80 μm is confined by fused quartz plate as shown in Fig. 1. The laser-induced ablation of the confined absorbing layer results in the generation of a high amplitude acoustic pulse wave. This elastic wave propagates through the substrate thickness to the coating, and reflects at the free surface of the coating. This leads to reflection and interference of elastic wave, impinging on interfacial stress. When the stress reaches the interfacial strength of coating, delamination and spallation of the coating may occur.

To estimate the interfacial stress accurately, numerical simulation of elastic wave propagation is carried out (Section 4.1). For this simulation, dynamics of input pulse wave induced by laser ablation is required. However, the ablation is a very complicate physical phenomenon [29, 30],[2] and is difficult to clarify theoretically in this study. Therefore, the out-of-displacement velocity at the back surface of laser irradiation epicenter is employed to determine the dynamics of ablation (for input wave). In order to measure the out-of-plane displacement velocity, laser ultrasonic interferometer (Tempo-1D, Bossa Nova Technologies) is used. The spot diameter of this laser is set 0.15 mm. Note the alignment of two laser beams is carefully adjusted. Therefore, the present technique involves two laser systems as shown in Fig. 1, in which pulse laser ablation induces high amplitude elastic wave, and travels through the specimen thickness. The out-of-plane displacement at the opposite surface (coating free surface) is measured by laser ultrasonic interferometer. Note that a roller pump is used to circulate MoS_2 grease of ablation layer, such that reproducible laser irradiation can be achieved readily.

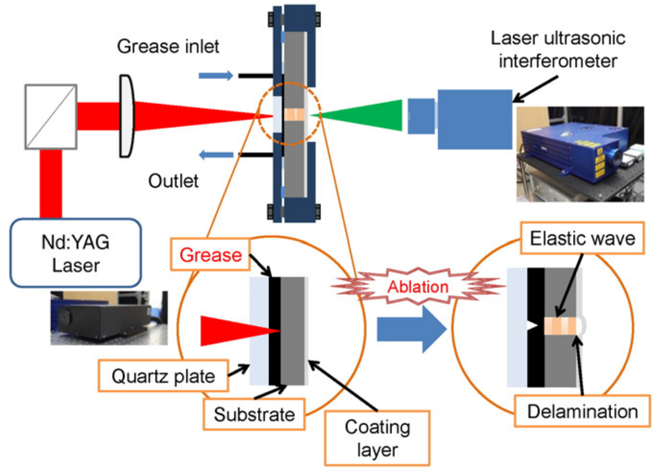

Fig. 1. Experimental setup of laser spallation technique.

2.2. Specimen

The electroplated Ni coating is deposited on a stainless steel substrate. This is a widely used coating and quite representative to demonstrate the versatility of the proposed measurement technique. The austenitic stainless steel SUS304 is employed as substrate. The substrate is plate shape with 50 mm × 75 mm × 3 mm. Mechanical polishing is carried out in order to make the substrate surface of mirror finish. The thickness of Ni coating is 20 μm and 50 μm. Table 1 shows the deposition condition of Ni electroplating. In addition, material properties (elastic property, density, longitudinal wave velocity) are shown in Table 2. The property of Ni coating is referred to the literature [31]. These material properties will be used for the computation of elastic wave propagation and interfacial stress. Note that the residual stress induced by deposition seems to be minor, since the deposition temperature is not so high (below 50 °C) and the post-heat treatment is absent.

3. Experimental result

3.1. Effect of break down layer

Before conducting the laser spallation test, the effect of break down layer (grease layer) is investigated. We prepare a simple plate of SUS304 as specimen and set to the experimental fixture as shown in Fig. 1. The energy of pulse laser is set to 30 mJ, and the grease layer thickness is set to 50 μm. Fig. 2 show the measurement waveform obtained by the laser ultrasonic interferometer, so that we examine the effect of break down layer. Fig. 2(a) is out-of-plane displacement and Fig. 2(b) is the velocity of out-of-plane displacement. In Fig. 2(a), it is found that the existence

Table 1
Ni electroplating condition.

Composition	g/l	Plating	
$Ni_2SO_4 \cdot 6H_2O$	250	Temperature	50 °C
$NiCl_2 \cdot 6H_2O$	40	pH	4.0–4.6
H_3BO_3	40	Current density	30 mA/cm^2

[1] It may be difficult to stop the coating spallation, with increase in the power of irradiated pulse- laser, since it requires detecting the coating delamination before the spallation with a non-destructive way.

[2] In the previous study [29,30], physical model of laser ablation has been investigated, and very illustrative to determine the input wave (resulting in elastic wave propagation and coating delamination). However, since the present ablation media is MoS_2 grease with adjustment by machine oil, and may be difficult to be described in the physical model. Thus, we use numerical simulation and output waveform to determine input pulse wave (dynamics) of laser ablation. This procedure will be explained later.