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Noncontact characterization of adhesion of surface layers by laser-generated acoustic waves

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Abstract: Laser-induced spallation is proposed as a powerful means for the investigation of adhesion properties of solid surface films. Ultrasonic pulses are produced at the back side of the sample by the impact of Nd:YAG-laser pulses. At the front side, the induced ultrasonic surface displacements are monitored with a 300 MHz bandwidth fiber-optic interferometer, which is proposed as a versatile point-detection ultrasonic transducer. The technique is applied to the time-resolved monitoring of laser-induced spallation and debonding of surface films.

1. INTRODUCTION

Thin and thick surface films and coatings play an increasingly important role in many areas of technology. Good adhesion strength of the film to its supporting substrate is vital for the performance of most films. We present the spallation of surface films as a promising tool for bond strength evaluation, being more quantitative than standard techniques [1]. Nd:YAG-laser pulses are used to generate the required acoustic pulses at the back side of the sample. At the front surface of the sample, the compressive pulse is reflected as a tensile pulse. If the sample is coated with a solid surface film, the film will undergo spallation if the effective tensile stress, which is the superposition of the stresses of the incoming and reflected waves, exceeds the adhesion strength of the film. The ultrasonic waveform and the spallation process manifest themselves as transient displacements of the sample surface [2]. The monitoring of these displacements allows the determination of the time-dependent stress history at the interface under test up to the moment of spallation, which in turn gives information on the adhesion properties of the surface film. Detection systems with a bandwidth of at least 100 MHz are required for the monitoring of these acoustic transients. We present a homodyne fiber-optic interferometer, which features the necessary large bandwidth as well as a simple setup. Adhesion tests have been performed on various substrate-film combinations, e.g. stainless steel plates with plasma-sprayed Mo-ceramic layers, the results of which are presented here.

2. EXPERIMENTAL SETUP

Figure 1 shows our setup for the noncontact generation and detection of ultrasonic pulses [3]. The pulses are generated at the back side of the sample by a frequency-doubled pulse of a Nd:YAG-laser with 7 ns halfwidth. The pulse energy is continuously variable from 2 mJ to 500 mJ, producing spallation in most cases. In order to favour the generation of longitudinal waves, we usually either work in the "confined plasma" geometry with a transparent quartz plate pressed against the sample, or by applying a thin oil layer, the former being more effective, and the latter producing very reproducible pulse shapes.

The transient displacements of the sample front surface induced by the ultrasonic waveform as well as the spallation process are monitored by a homodyne fiber-optic interferometer. This type of interferometer has

Figure 1: Setup for the fiber-optic interferometric monitoring of laser-induced spallation.

been previously used for atomic force microscopy in the kHz-range [4]. The beam of an ordinary 1 mW HeNe-laser is coupled into a monomode fiber, whose other end has been perpendicularly cleaved and positioned at a distance of approx. $50 \mu\text{m}$ from the surface to be monitored. Part of the light is reflected at the fiber-end (reference beam), and another part exits the fiber, reflects off the surface of the sample, and re-enters the fiber (signal beam). The interference signal is detected with a fast photodiode and preamplifier and recorded in a 2 GS/s digitizing oscilloscope.

The actual displacement $z(t)$ of the sample surface is then calculated from the interferometer signal $I(t)$ as:

$$z(t, k(t)) = \frac{\lambda}{4} \left\{ k + \frac{1}{2} - (-1)^k \cdot \frac{1}{\pi} \left[\arccos \left(\frac{2 I(t) - I_{\max} - I_{\min}}{I_{\max} - I_{\min}} \right) - \Phi_0 \right] \right\} + \text{const.} \quad (1)$$

where $I_{\max}$ and $I_{\min}$ are the interference maximum and minimum, respectively, λ is the HeNe-laser wavelength of 633 nm, and $k = 0, \pm 1, \pm 2, \dots$ is the slope number of the cosine-shaped fringe pattern of a two-beam interferometer (Fig. 2). Odd values of k correspond to negative slopes of the response function of the

Figure 2: Interferometer signal of an accelerated surface movement, illustrating the definition of the fringe numbers k .

interferometer. $I_{\max}$ and $I_{\min}$ are allowed to follow the changing fringe visibility of an actual interferometer signal. Finding the correct "fringe numbers" $k(t)$, which are a priori not known, can be a tricky task. But nevertheless, with some previous knowledge of the shape of the expected displacement signals, as well as by comparison of multiple signals of comparable events taken for different values of the phase Φ_0 , it is often possible to overcome this ambiguity. We therefore developed an analysis software, that allows an interactive calculation of $z(t)$, with the user specifying the interference maxima and minima where the fringe number changes.

For well reflecting sample surfaces, we achieve a noise level of 1 nm at a bandwidth of 300 MHz, measured at the quadrature point. In contrast to most other optical detectors, our interferometer also works fine with very rough sample surfaces like plasma-sprayed Al_2O_3 ceramic coatings, resulting in an increased noise level of 5 nm. The low drift of the interferometer due to the short interferometer-cavity length renders any active stabilisation unnecessary for single-shot experiments. The detection area on the sample surface measures only approx. 15 μm in diameter. Based on its robustness and simplicity of use, we consider this type of fiber-optic interferometer an ideal non-contact point-detection transducer, also for other applications requiring displacement monitoring.

Once $z(t)$ is determined, we get the surface velocity $v(t)$ by calculating the time derivative of z . The time dependence of the pressure pulse normal to the surface is then given by

$$\tau(t) = -\frac{1}{2}v(t)c_\ell\rho = -\frac{1}{2}v(t)Z \quad (2)$$

where c_ℓ is the longitudinal wave speed, ρ the density, and Z the acoustic impedance of the surface material. For many types of coatings, it may be difficult to determine accurate values of the impedance, because the material properties of surface films are only poorly known and can differ considerably from the data of the corresponding bulk material.

3. SPALLATION STUDIES

Thin film spallation studies have been performed on silicon wafers, 0.36 mm thick, with evaporated gold dots, 500 nm thick and 0.8 mm in diameter. Thin film spallation is caused by the tensile tail of the first longitudinal arrival L^1 . These studies have shown [3] that the fast debonding process takes place in approx. 1 ns. If the spalled-off piece of gold film moves directly towards the fiber-tip of the interferometer, a fast regularly moving fringe pattern is produced. The calculated displacement confirms a steady movement of the film away from the substrate at a constant speed reaching 95 m/s in our experiments. The velocity signal, calculated as the time derivative of the displacement signal, implies that the acceleration takes place within 5 ns, yielding $1.9 \cdot 10^{10} \text{ m/s}^2$!

Recently, the behaviour of 120 μm thick plasma-sprayed molybdenum ceramic layers on 1.9 mm thick steel substrates has been studied. Laser pulses of 100 mJ energy at 532 nm were slightly focused to a spot of 1.5 mm diameter on the back side of the substrate. A thin layer of oil was applied to the surface prior to each laser shot, permitting easy and reproducible acoustic pulse generation. Owing to the toughness of the layer, only a small area of layer with a diameter of less than 1 mm is debonded during spallation, and subsequently held in place by the surrounding layer still adhering to the substrate. In the example shown in Fig. 3, seven consecutive laser shots were fired onto the same spot, the first three of which are shown. The analysis of the displacement signals of the molybdenum layer opposite to the laser spot reveals that the first shot (numbered 1) produced a delamination. The thickness of the delamination gap between substrate and layer can be estimated to be around 1 μm (Fig. 3b). Because of this delamination gap, the longitudinal substrate arrival cannot reach the on-axis detection point directly, but circumvents the gap by its still adhering rim. The resulting extension of the path explains the observed delay of the arrivals of the signals 2 and 3. This delay of approximately 20 ns (Fig. 3c) corresponds to a lateral diameter of the delamination of 320 μm , assuming 4000 m/s for the longitudinal wave speed in the layer. Figure 3c also reveals that shot # 2 slightly enlarges the gap, seen in the additional delay of shot # 3. Although this delamination can clearly be detected, the surface of the ceramic layer doesn't show any visible damage.

A conventional, e.g. microscopic, inspection of the surface is thus not an adequate procedure for the detection of spallation of thick coatings, whereas our technique is capable of producing and detecting minute delaminations.

Figure 3: 120 μm plasma-sprayed Mo / Mo₂O₃ ceramic layer on 1.9 mm of stainless steel.

- Interferometer signals at 100 mJ/pulse: 1: Spallation signal, 2,3: Consecutive signals at same spot.
- Calculated displacements of layer surface.
- Detailed view of the first arrivals, as indicated in b), revealing a delay of approx. 20 ns owing to the delamination caused by the spallation at shot 1.

4. CONCLUSIONS

The time-resolved monitoring of laser-induced spallation of surface layers is introduced as a promising alternative to standard techniques, one main advantage being its purely noncontact character. Results obtained on plasma-spray coated steel samples provide new insight into the delamination process, when the interface is subjected to high tensile stresses normal to the interface, which are produced in a non-contact manner with pulsed lasers.

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