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► **To cite this version:**

Patrick F. Flowers, Sanichiro Yoshida, Kenji Gomi, Sushovit Adhikari, Katelyn Dreuk, et al.. Evaluation of Thin Film Coating Adhesion with an Opto-Acoustic Technique. Ultrasonics Symposium (IUS), 2011 IEEE International, Oct 2011, Orlando, United States. hal-01092229

HAL Id: hal-01092229

<https://hal.science/hal-01092229>

Submitted on 8 Dec 2014

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Evaluation of Thin Film Coating Adhesion with an Opto-Acoustic Technique

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An opto-acoustic technique to evaluate the adhesion strength at the interface of nano-scale thin film systems has been demonstrated. The specimens have been integrated into a Michelson interferometer as the end mirrors, and driven from the rear with an acoustic transducer at moderate frequencies. The resultant film surface displacement has been detected as a fringe shift of the interference intensity pattern behind the beam splitter with a digital imaging device at a normal frame rate. Comparison has been made between strongly and weakly adhered film specimens. The difference in the adhesion strength has been successfully visualized as the difference in the fringe contrast. Fourier analysis on the fringe pattern has quantified the fringe contrast.

***Index Terms*— Acoustic measurements, Nondestructive testing, Optical interferometry, Thin films.**

I. INTRODUCTION

Evaluation of adhesion strength at the interface of nano-scale thin film systems is important in various applications. To date, several destructive and non-destructive techniques have been proposed. Each of these techniques has advantages and disadvantages. Destructive methods basically scratch the film off the substrate and measure the applied force [1]. They are capable of evaluating the adhesion strength directly, but the measured force always contains the shear component. Therefore, a certain algorithm to estimate the normal force is required. In addition, as destructive testing methods, they are inapplicable to 100% inspection. Most non-destructive techniques available use acoustic excitation. Scanning acoustic spectroscopy [2] focuses acoustic energy at a point inside the substrate, and estimates the stiffness of the adhesion by measuring the phase velocity of the resultant surface acoustic wave. It is a reliable but time-consuming method, since it requires scanning over the surface. Telschow et al [3] demonstrated two-dimensional visualization by generating a wave in the interior of the substrate with an acoustic transducer and measuring the propagation of the acoustic wave to the surface. It does not require scanning over the surface, but high acoustic frequency (typically several hundred MHz) is required to excite acoustic waves in the substrate. This feature makes the resultant surface displacement small. Consequently, lock-in amplification with a high sampling number is necessary; hence, it is also time-consuming.

In this study, we propose an optical interferometric technique to visualize the displacement of the thin film surface. A Michelson interferometer was configured with the coated surface of silicon wafer specimens as the end mirrors of the two interferometric arms. The specimens were driven with an acoustic transducer from the rear surface of the substrate, and the resultant displacement of the film was detected as the relative phase difference behind the beam

splitter. This idea is similar to that of Telschow et al in the use of optical interferometry combined with acoustic excitation of the specimen. However, unlike the Telschow et al technique, the driving frequency was much lower than the resonant frequency of the substrate. Consequently, the substrate oscillated as a rigid body without acoustic wave excitation in the bulk. This feature made the apparatus and procedure of adhesion strength evaluation much simpler; modulation-demodulation scheme and lock-in amplification were unnecessary. Since the specimen was driven at a frequency range much lower than the resonance, the film surface displacement was on the same order as that of the substrate. The nondestructive nature along with the simplicity and low-cost features indicate potentiality for its application to assembly lines in related industries.

The aim of this paper is to report on a series of experiments that we recently conducted to prove the principle of the proposed method. In these experiments, we used two groups of thin film coated silicon wafer specimens: those prepared with a pre-surface oxygen plasma treatment (hereafter referred to as treated specimens) and those prepared without any pre-surface treatment (untreated specimens). Treated specimens are empirically known to show stronger adhesion than untreated specimens. All of the experiments we conducted showed clear differences between the treated and untreated specimens in the displacement of the film surface resulting from acoustic excitation of the specimens; the untreated specimens consistently showed greater displacement amplitude, whereas the treated specimens showed greater phase lag. The phase difference between the film surface and the acoustic transducer indicates that the treated specimens have higher damping behavior at the interface. These observations agree with the consensus that treated specimens have stronger adhesion because the pre-surface treatment makes the interface rougher; hence, increasing the effective surface area of adhesion. In the following sections we describe these experiments along with the results.

II. SPECIMENS AND PRINCIPLE OF THE METHOD

A. Specimens

Three film materials (polystyrene, platinum, and gold) were tested. All specimens used in this study were coated on a silicon substrate. The polystyrene specimens were prepared with a coating technique known as spin-coating [4], while the platinum and gold specimens were prepared by sputter deposition [5]. Prior to the coating, the silicon substrates for specimens chosen to fall into the group of treated specimens were treated with oxygen plasma. The silicon substrate was cut along the [1,0,0] plane. The thickness of the silicon substrate and the film were 750 nm and 100 nm, respectively. The specimens were fabricated as a disk with a 75 mm diameter.

B. Principle of the method

Consider one of the above-described specimens driven with an acoustic transducer from the rear of the substrate at a frequency much lower than the resonant frequency of the silicon substrate. At such a frequency the substrate is expected to oscillate as a rigid body. On the other hand, depending on the stiffness of adhesion at the interface, the film is expected to show different oscillatory motion relative to the substrate; the lower the stiffness, the higher the amplitude. To the first-order approximation this dynamic can be considered as a harmonic oscillation. The transfer function from the substrate's displacement to the film surface displacement can be fully characterized by the resonant frequency and damping coefficient of the adhesion.

If the film displacement is measured in the time domain, the magnitude and phase of the transfer function can be directly measured. Our first experiment [6] was designed to observe this effect. However, when the film displacement is detected with a digital camera (CCD, charge coupled device) of a normal frame rate as a fringe shift behind the beam splitter of a Michelson interferometer, the dynamic is observed in a different fashion. Since the CCD's sampling rate is much lower than the acoustic frequency, the film displacement lowers the visibility of the interferometric fringe; i.e., the dark stripes in the fringe pattern become blurred. Our second experiment with a Michelson interferometer was designed to observe this effect.

III. RESULTS AND DISCUSSION

To prove the above principle, we conducted two types of experiments. In the first experiment, Laser Doppler Vibrometry [7] was used to directly measure the displacement of the film surface resulting from excitation by an acoustic transducer from the rear surface of the substrate. In the second experiment, a Michelson interferometer [8] was arranged using the film surface as the end mirrors' reflective surfaces. The film surface displacement was then observed behind the beam splitter as the relative phase change of the fringes by driving one specimen at a time.

A. Experiment with Laser Doppler Vibrometry

Fig. 1 illustrates the configuration of the first experiment. The specimen was bound with epoxy to an acoustic transducer driven by a function generator with a sinusoidal signal. The Laser Doppler Vibrometer measured the displacement of the coated surface of the specimen as a function of time. This experiment was conducted with the polystyrene-coated specimens as they were expected to show the greatest surface displacement among the three film materials.

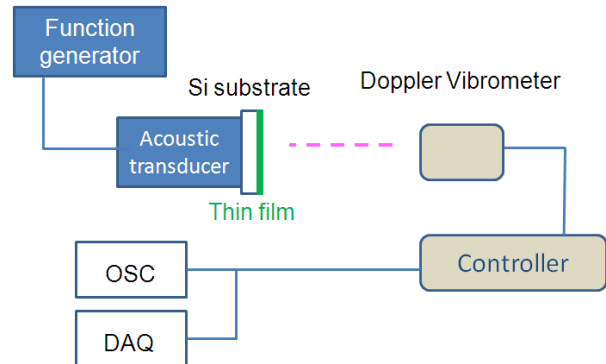


Fig. 1 Arrangement of first experiment for direct measurement of film surface displacement with Doppler Vibrometer. OSC: oscilloscope, DAQ: Data Acquisition

Fig. 2 shows the result of measurements at a driving frequency of 50 kHz. The plot labeled "transducer" indicates the displacement of the transducer's surface. The amplitude of displacement for the treated specimen is significantly lower than that of the untreated specimen, and even that of the transducer itself. In addition, the treated specimen shows much greater phase lag to the transducer than the untreated specimen. From these observations, it is considered that the thin film of the treated specimen is over-damped. This is consistent with the speculation that the oxygen plasma treatment makes the substrate surface rougher and thereby strengthens the adhesion.

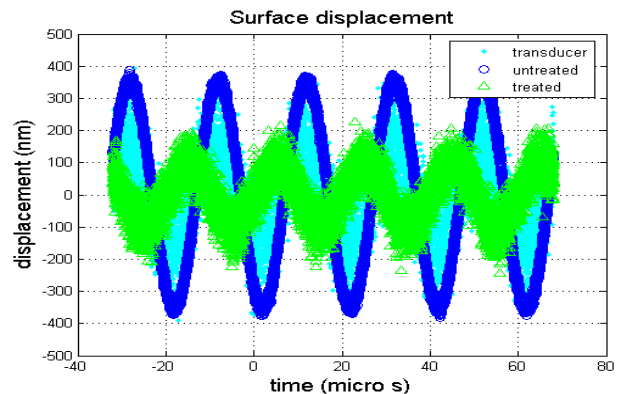


Fig. 2 Surface displacement observed in first experiment

B. Experiment with Michelson Interferometer

Fig. 3 illustrates the arrangement of the second experiment. The two end mirrors of a Michelson interferometer were replaced with a pair of gold-coated specimens; one treated specimen, and the other untreated. The coated side of the

specimens was faced inward, reflecting the light from the laser source back to the beam splitter. Identical acoustic transducers were adhered to the specimens providing oscillation from the rear, in line with the optical path. The light source was a 1 mW helium/neon laser oscillating at 632.9 nm. The intensity pattern of the interference was projected on a screen (labeled “image plane” in Fig. 3) placed behind the beam splitter which a standard CCD camera operating at a frame rate of 30 frames/s captured. The end mirror for the horizontal optical path was tilted so that its angle of incidence to the image plane was slightly off the normal direction. In this fashion, the optical path difference between the two beams depended on the transverse distance from the optical axis on the image plane; hence, the interference pattern consisted of vertical fringes (dark stripes). When either specimen was oscillated by the acoustic transducer, the vertical fringes were expected to shift horizontally. Since the CCD camera’s frame rate was much lower than the acoustic frequency, this caused the fringe pattern to be blurred, i.e., lower fringe contrast. One specimen at a time was oscillated at a driving frequency ranging from 10 kHz to 20 kHz.

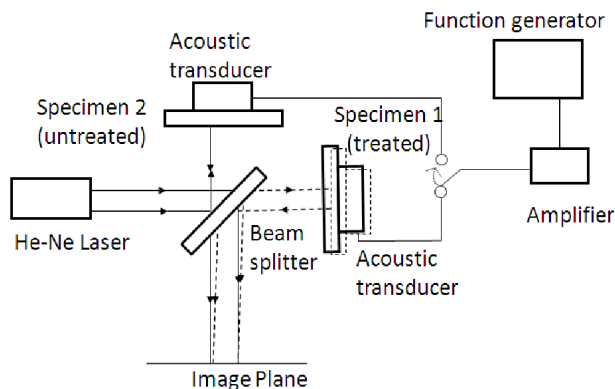


Fig. 3 Arrangement of second experiment utilizing Michelson interferometry

Fig. 4 shows a set of fringe patterns observed at a driving frequency of 11.5 kHz. The slightly curved feature of the fringe patterns is due to the finite curvature of the wave fronts of the laser beams. When driven by their transducers one at a time, the fringe pattern of the untreated specimen (left) showed significantly lower fringe contrast compared with that of the treated specimen (middle). As expected, the fringe pattern observed with both transducers off (right) showed the highest contrast. The same experiment was conducted with the platinum-coated specimens using a video camera. The video also clearly showed significantly lower fringe contrast when the untreated specimen was oscillated compared with that when the treated specimen was oscillated.

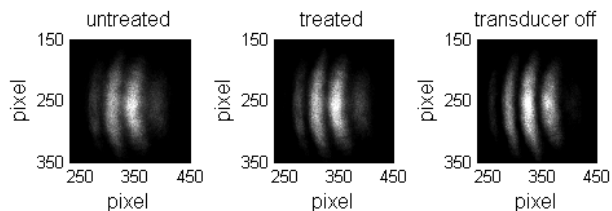


Fig. 4 Fringe patterns for untreated and treated specimens independently driven at 11.5 kHz compared with that when both transducers were off.

For a more quantitative analysis, the intensity of the above fringe patterns at the middle row (row number 250) was extracted as the gray level contained by the corresponding pixels. Fig. 5 shows the extracted gray levels as a function of horizontal pixel number. This plot represents the intensity profile of the fringe pattern along the middle horizontal line. The peaks correspond to the bright fringes observed in Fig. 4. Each fringe pattern shows the peaks at different horizontal locations because the CCD camera’s position relative to the optical axis of the interferometer was not always the same. However, the distance between the neighboring peaks (i.e., the spatial frequency of the fringes) is the same, indicating that the spatial frequency is solely determined by the relative orientation of the interferometer’s end mirrors.

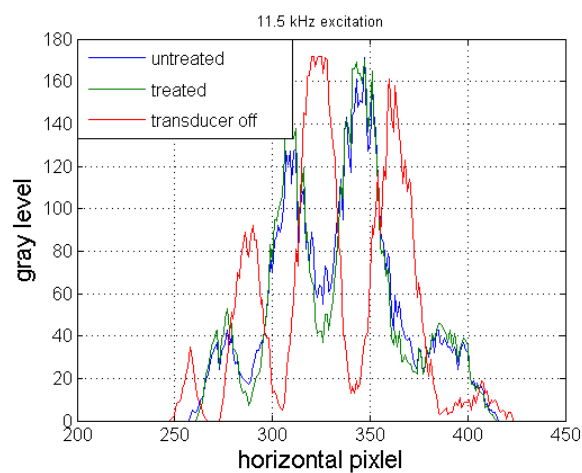


Fig. 5 Intensity profile of fringe patterns at middle row

The differences in the fringe contrast (the level of blurriness) among the cases represented in Fig. 5 are not clear. Thus, in Fig. 6 the Fourier spectra of the intensity profiles shown in Fig. 5 were plotted. Fig. 5 indicates that the spatial period of the intensity profile is approximately 80 (pixels), i.e., $1/80 = 0.0125$ (1/pixel) in frequency. Correspondingly, the highest peak in Fig. 6 (the main peak) appears around the spatial frequency of 0.0125 (1/pixel); the correlation proves that the peak in Fig. 6 represents the main fringes seen in Fig. 5. It is clear from Fig. 6 that the heights of the peaks were lower for specimens undergoing oscillation than when both transducers were off; moreover, the peak corresponding to the untreated specimen undergoing oscillation is lower than that of the treated specimen when oscillated at the same frequency. This indicates that the film of the untreated specimen experienced greater amplitude of oscillation than the treated specimen.

It is interesting to note that the three spectra are practically identical both on the high frequency and low frequency sides of the main peaks. From the viewpoint of the fringe patterns, the spectra in these frequency regions are considered to be noise. It is likely that the low frequency noise is due to slow random fluctuation in the index of refraction in the interferometer’s optical path caused by air temperature

variation. The high frequency noise is due to the speckle nature of the intensity pattern; since the light experiences diffusive reflection on the film surface, the intensity field on the image plane consists of a number of speckles. Careful observation of the fringe patterns in Fig. 4 reveals the speckle nature of the intensity field. Since these factors are independent of the specimen's adhesion strength or motion, they are common to the three plots in Fig. 6; it is understandable that the spectra are identical in the corresponding frequency regions.

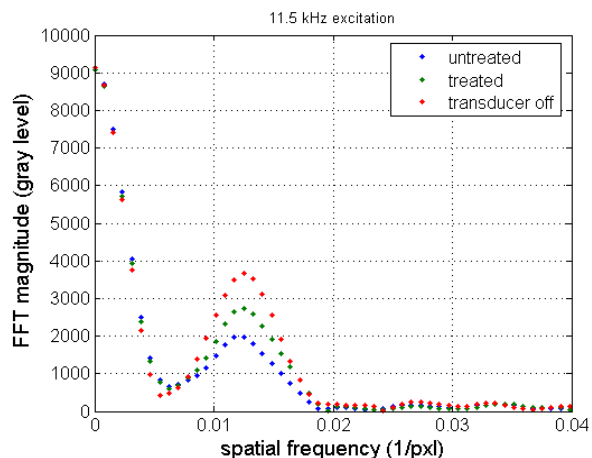


Fig. 6 Fourier transform of fringe patterns

The Fourier spectrum was also obtained by the same procedure as that of Fig. 6 at various driving frequencies. Fig. 7 summarizes the peak values at the main peak of the spectra. It is seen that in the entire frequency range of 10 kHz to 16.5 kHz, the untreated specimen shows significantly lower peak values than the treated specimen, supporting the above argument that the untreated specimens displayed inferior adhesion.

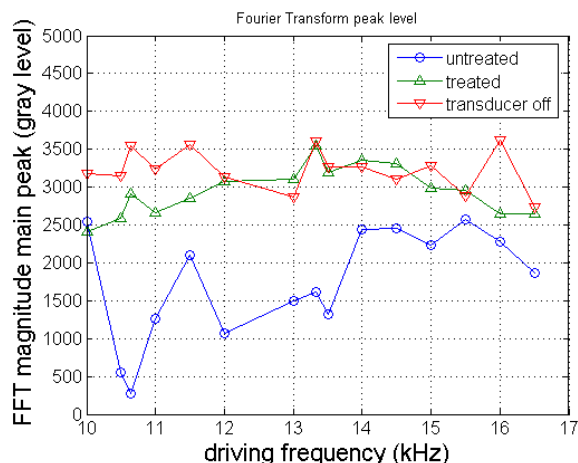


Fig. 7 Peak gray level at the maximum of the main peak of Fourier spectra

IV. CONCLUSIONS

This study has proved the principle of the proposed method for evaluating adhesion strength in thin film systems. The

Fourier analysis on the intensity profile of the fringe patterns has been shown to be a good indication of the strength of adhesion at the interface. The nondestructive nature, simplicity, and low cost in the assembly and operation of the apparatus indicate the potential of the proposed method for fast analysis of specimens in the assembly line.

ACKNOWLEDGMENT

The present research was supported in part by Southeastern Louisiana University under the 2012 Science and Technology Awards for Research (STAR) program, and the South Korean National Science Foundation. Part of the experiment was conducted on the campus of Tokyo Denki University, Tokyo, Japan, as part of the National Science Foundation's International Research Experience for Student Grant (IRES-0927033) awarded to Southeastern Louisiana University. The polystyrene specimens were prepared by Ali K. Kalkan of Oklahoma State University.

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