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Opto-acoustic technique to evaluate adhesion strength of thin-film systems

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An opto-acoustic technique is proposed to evaluate the adhesion strength of thin film systems at the film-substrate interface. The thin-film system to be examined is configured as an end-mirror of a Michelson interferometer, and driven from the rear with an acoustic transducer at audible frequencies. The amplitude of the resultant oscillation of the film is quantified as the variation in the contrast of the interferometric fringe pattern observed with a digital camera at 30 frames/s. As a proof of concept, experiment has been conducted with the use of a pair of strongly and weakly adhered Au-coated Si-wafer specimens. The technique successfully differentiates the adhesion strength of the specimens.

Thin-film systems are used in various applications ranging from semiconductor chips to artificial joints. In many cases, the adhesion strength at the film-substrate interface is an important factor for reliability of the end-product, and its inspection at the production line is essential. Ultrasonic imaging techniques are widely used for inspection of film-substrate interfaces. The field has a long history of research and development, and numerous literatures are available. Gilmore¹ reviews industrial applications of the techniques. With these techniques, it is relatively easy to find defects at the interface as they are recognized as contrast in the image. When the interface is defectless, on the other hand, characterization of the adhesion is challenging. Some of the ultrasonic techniques are applicable to defectless interfaces, but they are not necessarily ready for field uses. Scanning acoustic spectroscopy² focuses acoustic energy at various depths in the substrate and estimates the stiffness of the adhesion via analysis of the resultant surface acoustic wave. It is a well-developed technique, but requires scanning along the depth and numerical modeling is necessary to convert the acoustic signal to the stiffness. Hence, it is time consuming, and the final result depends on the model. The full-field visualization technique developed by Telschow *et al.*³ excites acoustic waves in the substrate and measures the film-surface displacement optically. It does not require scanning, but high acoustic frequency, typically 100 MHz or higher, is necessary to excite the acoustic wave. Consequently, the resultant displacement of the film is small, and lock-in amplification with a high sampling number is necessary; hence time-consuming as well.

From the scientific viewpoint, the adhesion of a thin film on a solid substrate is not a simple process. It depends on a variety of the attributes of the interface, and often the chemical bonding at the interface are not fully understood. As Baglin⁴ points out in his review article, further research is necessary in the related areas such as adhesion enhancement technique and interface modeling.

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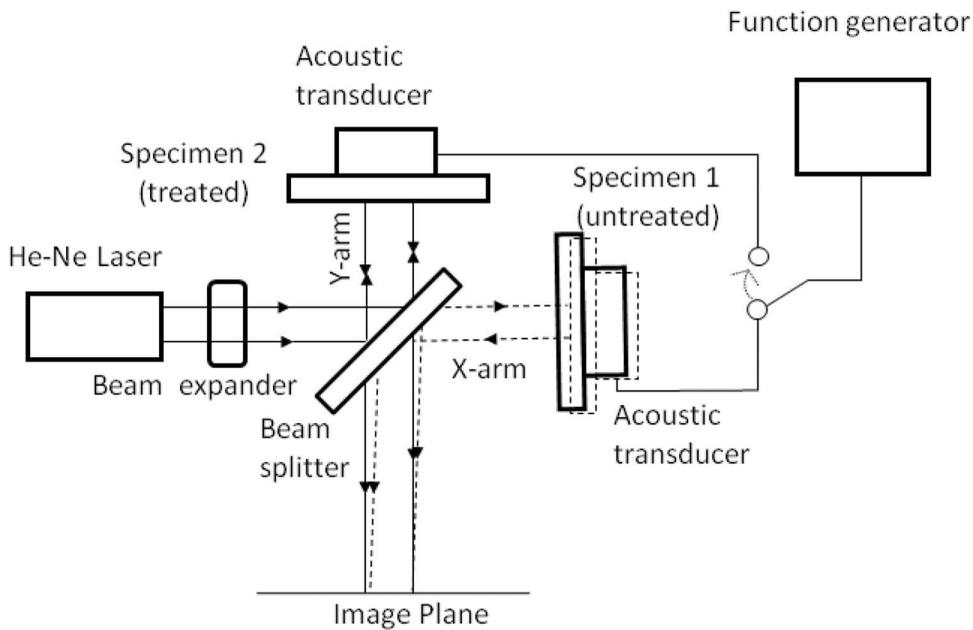


FIG. 1. Experimental setup.

Development of a simple and reliable nondestructive technique for adhesion characterization is important not only for good quality control of commercial products but also for advancement of the coating technology.

In this paper, we propose an opto-acoustic method operatable at lower frequency. The principle is straightforward. The thin-film system to be examined is oscillated at an audible frequency with an acoustic transducer from the rear surface of the substrate, and the resultant displacement of the film surface is measured as a relative phase difference behind the beam splitter of a Michelson interferometer. Unlike the case of acoustic frequency over 100 MHz, the oscillation frequency is orders-of-magnitude lower than that required to excite an acoustic wave in the substrate; hence the substrate oscillates as a rigid body. At the same time, the oscillation frequency is high enough to induce harmonic motion in the interface region where the stiffness is lower than the substrate or the film material. Consequently, the displacement of the film surface is considerably greater than that of the substrate. The use of low oscillation frequency brings about two advantages; lock-in amplification becomes unnecessary, and a digital imaging device such as a CCD (charge-coupled-device) camera can be used at a normal frame rate (30 fps) for signal readout. Below we report on our recent proof-of-concept experiment in which we have demonstrated the proposed method by successfully differentiating the adhesion of two Au-coated Si-wafer specimens that supposedly have different adhesion strengths. The first specimen (the untreated specimen) is prepared with a process known as the sputter deposition without pre-coating treatment on the substrate surface. The second specimen (the treated specimen) is prepared with the same sputter deposition, but the substrate's surface is treated with oxygen plasma prior to the coating. It is empirically known that this surface treatment strengthens the adhesion. With a simple mathematical procedure, the oscillation amplitude has been estimated quantitatively.

Figure 1 illustrates the Michelson interferometer used in this experiment. The end-mirror of one arm is replaced with the treated specimen, and the other with the untreated specimen. Both specimens are originally fabricated as a disk of 76 mm in diameter, and cut into an approximately 5 mm × 5 mm square. The Au film is 100 nm thick and coated on a 750 nm thick substrate cut along the [1,0,0] plane. The coated side of the specimens is faced inward, reflecting the light from the laser source back to the beam splitter. Identical acoustic transducers are attached to the specimens providing oscillation in line with the optical path. The light source is a 1 mW helium/neon laser oscillating at 632.9 nm. The intensity pattern of the interference is projected on a screen placed

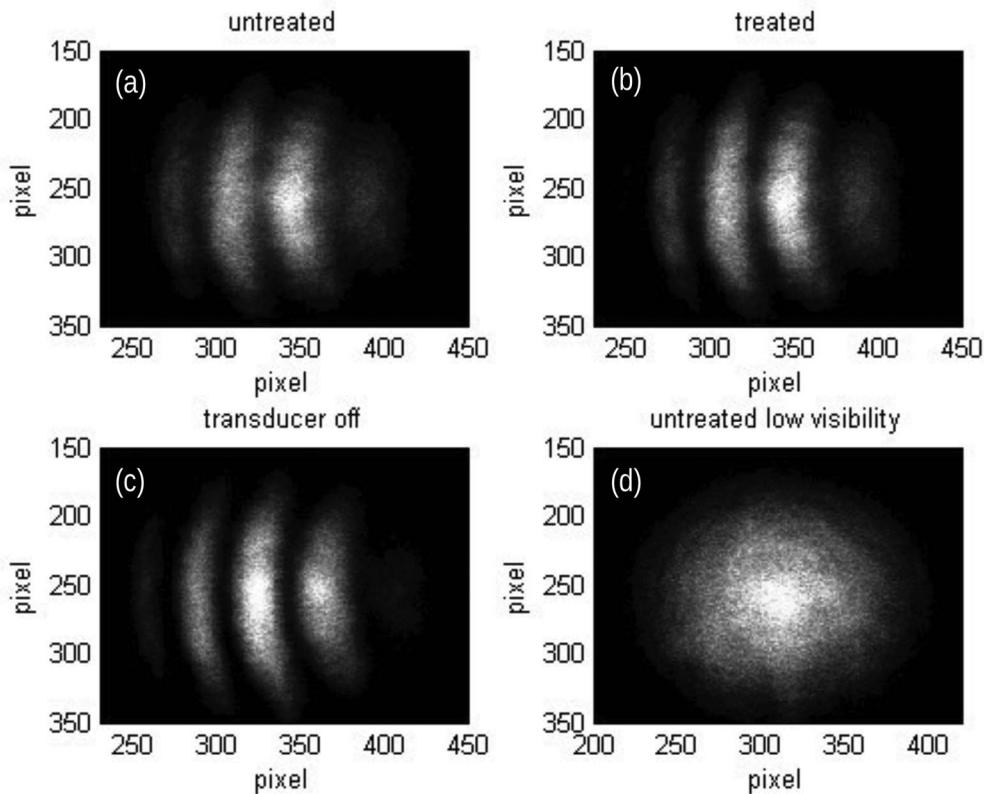


FIG. 2. Typical fringe images. (a) : drive untreated specimen; (b): drive treated specimen; (c): both specimens undriven; (d): drive untreated specimen harder.

behind the beam splitter, which a standard CCD camera captures at a frame rate of 30 frames/s . The end-mirror of the X-arm is tilted horizontally so that its returning beam's angle of incidence to the screen is slightly off the normal direction. In this way, the optical path difference between the two beams varies in proportion to the transverse distance from the optical axis on the screen, making the interference pattern consist of vertical fringes (dark stripes). One specimen at a time is oscillated at a driving frequency ranging from 10 to 50 kHz. When either specimen is oscillated by the acoustic transducer, the vertical fringes dither horizontally at the same frequency. Since the CCD camera's frame rate is much lower than the acoustic frequency, this causes the contrast of the fringe pattern to be decreased. Fig. 2 shows a set of fringe patterns observed at a driving frequency of 11.5 kHz. The slightly curved feature of the fringes is due to the curvature of the laser's wave fronts caused by the beam expander. The condition for each pattern is as follows. (a) : the untreated specimen driven and the treated specimen undriven; (b): the treated specimen driven and the untreated specimen undriven; (c): both specimens undriven; (d): the untreated specimen driven at 11.5 kHz more strongly than (a) with the treated specimen undriven. The amplitude of the input voltage to the transducer is the same for (a) and (b). The following observations can be made: (1) comparison of (a) and (b) against (c) shows that the fringe contrast is decreased when either specimen is oscillated; (2) comparison of (a) against (b) shows that when the untreated specimen is oscillated, the degree of decrease in the fringe contrast is greater than when the treated specimen is oscillated at the same frequency and amplitude. (3) When the untreated specimen is oscillated more strongly, the film's oscillation amplitude comes to the point where the fringe becomes invisible. These observations qualitatively indicate that when oscillated at 11.5 kHz with an appropriate strength, the film surface causes an oscillatory optical-path change detectable as a decrease in the fringe contrast and that the oscillation amplitude of the thin film is greater on the untreated specimen than the treated specimen. The latter is consistent with the condition that the film adhesion is stronger on the treated specimen.

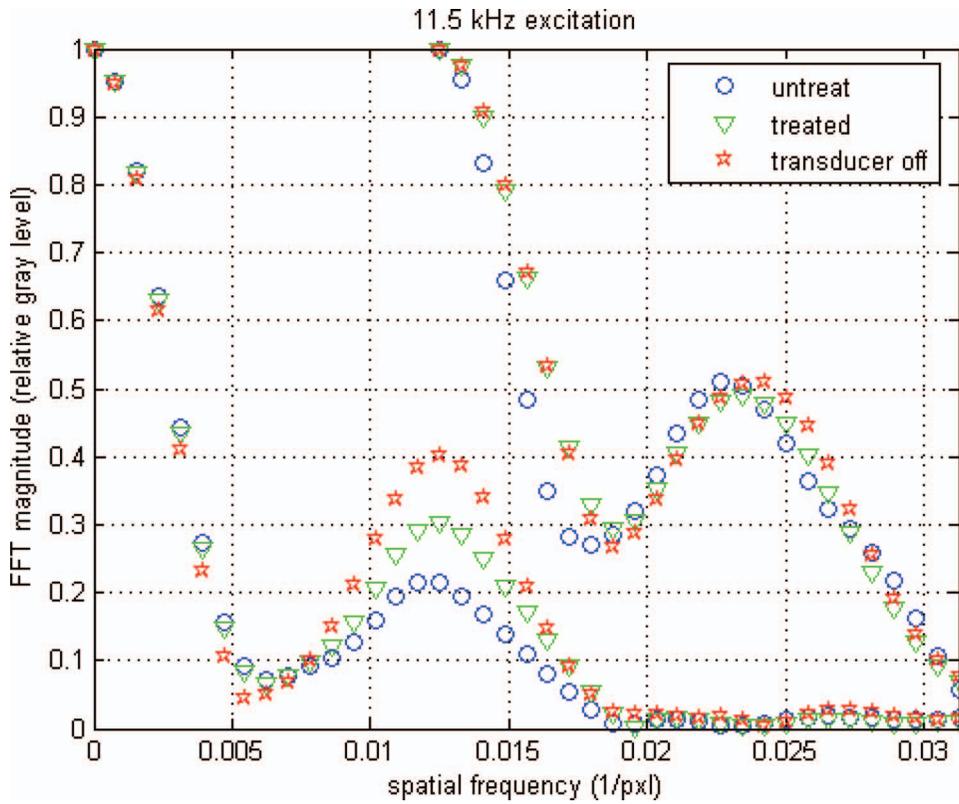


FIG. 3. Fourier spectra of intensity profile along central rows of Fig. 2(a)–2(c) (left) and corresponding spectra for uncoated Si wafers (right). The right spectra are shifted horizontally by 0.02 (1/pixel) for better visibility.

The fringe contrast can be quantitatively evaluated via Fourier analysis of the fringe patterns. Fig. 3 are the magnitude of the Fourier spectra obtained by performing Fast Fourier Transform (FFT) on the horizontal intensity variation along the central row of the three fringe patterns Fig. 2(a)–2(c). It is clearly seen that the peak value decreases in the order of the cases “both undriven”, “the treated driven” and “the untreated driven”, whereas the magnitude at the low and high frequency sides of the peak envelope is very similar among the three cases. Here the higher peak value corresponds to the higher contrast, as discussed mathematically below. The three spectra inserted on the right in Fig. 2 are the FFT spectra obtained with both specimens replaced by uncoated Si wafers. The transducer and the driving conditions for the uncoated-wafer cases are exactly the same as the two coated-specimen cases. The three spectra of the uncoated wafer cases are overlapping one another indicating that the observed difference in the coated specimen cases is not caused by a difference in the transducer performance or other difference in the experimental condition but the coating condition.

The relationship between the film’s oscillation amplitude and the peak value of the FFT spectrum can be explained mathematically by considering the output signal of the CCD camera. The optical intensity I on the screen placed behind the beam splitter can be written as follows.

$$I = I_1 + I_2 + 2\sqrt{I_1 I_2} \cos(\phi_1 - \phi_2), \quad (1)$$

where I_1 and I_2 , and ϕ_1 and ϕ_2 are the intensity and the phase of the light returning from the moving and stationary end-mirrors to the screen. As a function of time, ϕ_1 and ϕ_2 can be expressed as follows.

$$\begin{aligned} \phi_1(t) &= kl_1(t) - \omega t = k(l_{10} + d \sin \Omega t) - \omega t, \\ \phi_2(t) &= kl_2 - \omega t = kl_{20} - \omega t, \end{aligned} \quad (2)$$

where l_1 and l_2 are the distances from the moving and stationary end-mirrors to the screen, $k = 2\pi/\lambda$ is the wave number of the laser light with λ being the wavelength, Ω is the oscillation frequency of the thin film, ω is the optical angular frequency of the laser light, and suffix 0 used in l denotes the distance when the transducer is turned off. Putting $I_1 = I_2 = I_0$ as the intensity of the two beams set to be the same and using eqs. (2) for the phases, we can rewrite eq. (1) as follows.

$$I(t) = 2I_0 + 2I_0 \cos \{k(l_{10} - l_{20}) + kd \sin \Omega t\}. \quad (3)$$

Since one of the end-mirrors is tilted, the relative phase difference on the screen can be put as a linear function of x as $k(l_{10} - l_{20}) = \alpha x$. Here x is the horizontal coordinate on the screen surface whose origin is at the central fringe and α is a constant. With these, eq. (3) becomes

$$I(x, t) = 2I_0 \{1 + \cos(\alpha x) \cos(\delta \sin \Omega t) - \sin(\alpha x) \sin(\delta \sin \Omega t)\}, \quad (4)$$

where $\delta = kd$. Using Bessel functions, we can further rewrite eq. (4) as follows.

$$I(x, t) = 2I_0 [1 + \cos(\alpha x) \{J_0(\delta) + 2J_2(\delta) \cos(2\Omega t) + \dots\} - \sin(\alpha x) \{2J_1(\delta) \sin(\Omega t) + 2J_3(\delta) \sin(3\Omega t) + \dots\}]. \quad (5)$$

Now consider the intensity captured by the CCD camera whose frame rate is much lower than the oscillation frequency of the film, $\Omega/2\pi$. From eq. (5) the CCD camera's output signal $S(\tau) \propto \int_0^\tau I(x, t) dt$ can be put as follows.

$$\begin{aligned} S(\tau) \propto & \int_0^\tau \{1 + \cos(\alpha x) J_0(\delta)\} dt \\ & + 2 \cos(\alpha x) \sum_{m=1}^N J_{2m}(\delta) \int_0^\tau \cos(2m\Omega t) dt \\ & - 2 \sin(\alpha x) \sum_{m=1}^N J_{2m-1}(\delta) \int_0^\tau \sin\{(2m-1)\Omega t\} dt. \end{aligned} \quad (6)$$

where τ is the exposure time of the CCD camera and m is an integer. Since $\tau \gg 2\pi/\Omega$, the integrands of the second and third terms on the right-hand side of eq. (6) [$\cos(2m\Omega t)$ and $\sin\{(2m-1)\Omega t\}$] sweep through a number of periods; consequently, these terms oscillate with an increase of τ . On the other hand, having an integrand independent of time, the value of the first term increases in proportion to τ . Therefore, when the CCD's frame rate is much lower than the film's oscillation frequency, $S(\tau)$ is dominated by the first term. As this term contains the fundamental spatial frequency, this in turn means that on the FFT spectrum like Fig. 3 the value of the first peak (call the first peak value) increases in proportion to τ while other peaks don't.

As apparent from the first term of eq. (6), the first peak value is proportional to $J_0(\delta)$. Since $J_0(\delta)$ is a monotonically decreasing function of δ from $\delta = 0$ to the first zero at $\delta = 2.405$, and δ is proportional to d , this means that for a given τ the first peak value decreases as the film's oscillation amplitude increases. From this viewpoint, the FFT spectra in Fig. 3 can be interpreted as follows. When the two specimens are oscillated at 11.5 kHz, the thin-film on the specimens experiences an oscillation with a certain amplitude d , and this decreases the first peak value in proportion to $J_0(\delta)$. When the amplitude corresponds to $\delta = 2.405$ the peak becomes zero and the fringes become totally invisible. Since the untreated specimen causes a greater δ , the peak value decreases more than the treated specimen. (Imagine a small mass is attached to a larger mass with a spring, and the larger mass is oscillated. The weaker the spring, the greater the oscillation the small mass would experience.) Since the value of $J_0(\delta)$ is uniquely determined by δ and $J_0(\delta) = 1$ for $\delta = 0$, it is possible to estimate the value of δ , hence the oscillation amplitude d , from the first peak value relative to that of the case when the transducer is off (call the peak ratio). Table I shows the amplitude of the film's oscillation estimated in this fashion for the driving frequency of 11.5 kHz.

Fig. 4 shows the peak ratio as a function of the driving frequency. In the range of 10 - 50 kHz, the untreated specimen consistently shows substantially lower peak ratios as compared with the treated

TABLE I. Oscillation amplitude of thin-film estimated from the peak ratios.

	peak value	peak ratio	δ from $J_0(\delta)$	displacement (nm)
transducer off	3709	1.0	-	
treated	2665	0.72	1.1	106
untreated	1894	0.51	1.51	141

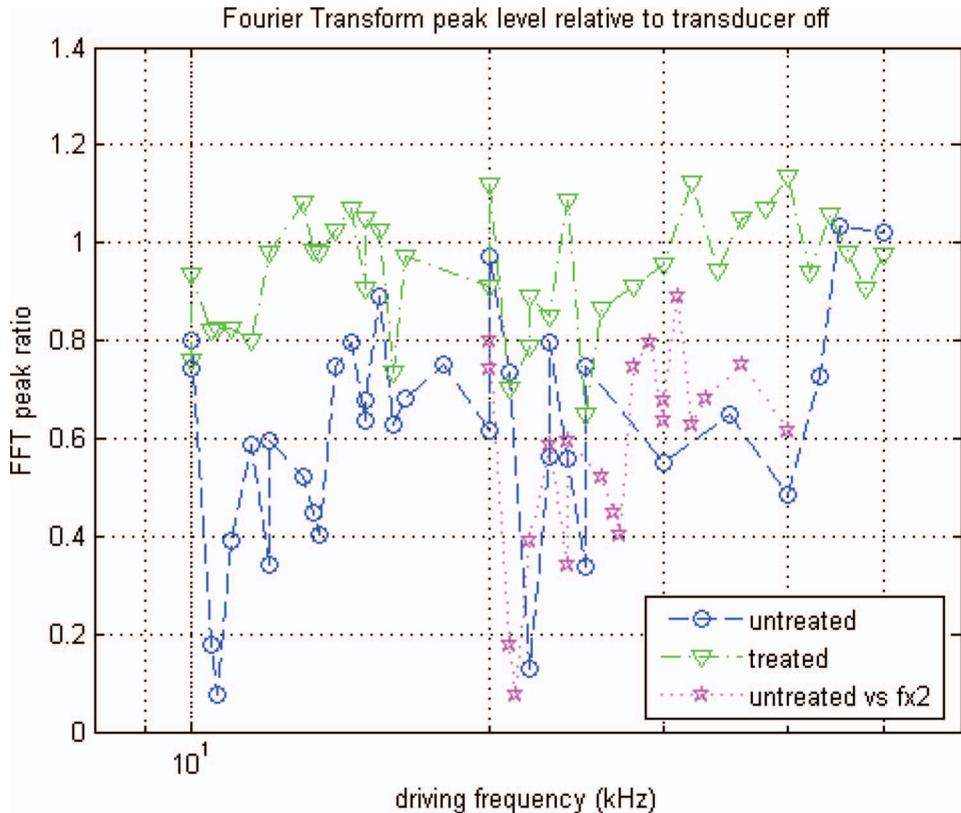


FIG. 4. Fourier transform peak value ratio for untreated and treated specimens.

specimen. The plot of the untreated specimen appears to be consisting of two envelopes; the first ranging from 10 - 20 kHz and the second 20 - 50 kHz. In the first envelope, there is a fairly sharp peak centering at 10.6 kHz. From the shape, this peak can be interpreted as representing a certain resonant phenomenon. With this interpretation, it is possible to view the similar peak observed around 22 kHz as representing the second harmonic of the 10.6 kHz peak. To explore this possibility, we plot the first envelope against the double frequency (20 - 40 kHz) in Fig. 4. This plot appears to follow the 20 - 40 kHz part of the second envelope, supporting the interpretation that the 10.6 kHz represents a resonant phenomenon. It is interesting to note that this frequency range is similar to that in which Penn *et al.*⁵ observed the first- and second-mode mechanical resonances of the optical coating on a fused silica substrate with a similar coating thickness. If this peak represents dynamics at the film-substrate interface, it will be useful for study of adhesion mechanism. Identification of this resonance-like behavior is a subject of our future study.

In summary, this study has demonstrated that the proposed technique is able to differentiate the adhesion strength between two differently prepared Au-coated Si-wafer specimens. With the simple mathematical procedure, the film's oscillation amplitude can be estimated. The setup is quite simple and field applications are expected.

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