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ESPI TECHNQIUE FOR MEASURING MICRO-TENSILE PROPERTIES OF THIN FILM MATERIALS

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Abstract: In-plane ESPI(electronic Speckle Pattern Interferometric) technique was developed to measure the micro-mechanical properties for thin film materials. The properties were determined from the micro-tensile stress and strain curve by measuring micro-tensile strain in micro-sized specimen 0.5 μm thick and 200 μm wide. The specimen was monotonically loaded by the micro-tensile loading system developed in this study. The micro-tensile strain during tensile loading was measured using the in-plane ESPI technique. In order to continuously measure the strain during loading, the subsequent strain measurement algorithm was developed. Furthermore, sensitivity to measurement of the strain was enhanced by phase estimation algorithms: sinusoidal fitting technique and object-induced dynamic phase shifting technique. Using these algorithms, the micro-tensile stress-strain curves were generated. It is shown that the sensitivity was increased by 6 times to a maximum

Keywords: ESPI technique, micro-tensile properties, thin film materials, subsequent strain measurement, sensitivity

1. INTRODUCTION

As nano technology is expected to be a core technology in future, micro/nano materials required for fabricating the M(N)EMS have been actively developed. Properties of these materials may be significantly essential for evaluating the reliability of the system, as well as in design, manufacturing process and usage.

Several testing methods have been proposed to measure the micro-mechanical properties for nano/micro-materials including thin films.[1] In order to obtain the exact stress-strain curve and determine the reliable properties by these methods, strain measurement technique, which can be used in measurement of the strain corresponding to the stress generated during mechanical loading, may be essential. The novel strain measurement techniques, like ISDG technique[2], DIC[3] and laser interferometry [4,5] etc., were proposed instead of using the conventional strain gages or extensometer.

In this study, in-plane ESPI(Electronic Speckle Pattern Interferometry) technique, as a laser interferometry technique, was introduced to measure micro-tensile properties for thin film materials, Au and TiN. To apply the ESPI technique successively for measurement of the properties, subsequent strain measurement algorithm was developed to continuously obtain the strain data during loading. Furthermore, the resolution of the strain measurement was improved by using the newly developed algorithms for enhancement of the sensitivity.

2. STRAIN MEASUREMENT ALGORITHMS

2.1. Subsequent strain measurement algorithm

ESPI is one of the most appropriate methods for highly sensitive measurement of out-of-plane and in-plane displacement/strain in non-contact. For in-plane ESPI technique, two collimated laser beams, generated from a laser source, illuminate the specimen surface at equal incident angle θ on either side of the surface normal. Interference speckle patterns may be produced by superposition of the scattered lights. As the specimen is deformed, the interference speckle patterns are moved and fringe patterns are also varied. By comparing a fringe pattern representing the reference object state and the other representing a deformed state, the value of deformation will be calculated. Speckle correlation fringes obtained by subtracting the intensity of the deformed surface from that of the initial surface state can be presented as the following:

$$I_{sub} = \left| I_i - I_d \right| = 4a_1a_2 \sin(\phi_R + \frac{\Delta\phi}{2}) \sin\frac{\Delta\phi}{2} \quad (1)$$

, where i indicates the frame at the initial state, d indicates that at the deformed state, and $\Delta\phi$ is the phase difference introduced in the original object beam due to object deformation. And, ϕ_R (= ϕ_1-ϕ_2) represents the random phase, and a_1, ϕ_1, a_2, ϕ_2 are the amplitude and phase of the beam scattered from the surface in initial state and of the reference beam that is used, respectively.

During tensile loading, the deformed states of the specimen can be presented by a sequence of speckle patterns, compared with a reference speckle pattern. Thus, the number of the fringe patterns varies with amount of the deformation of the specimen: the number of fringes is increased as the deformation of the specimen gets relatively large. Therefore, the relatively large deformation makes the fringe patterns, compared to a reference pattern, become considerably complex and indiscernible. It means that the deformation beyond its considerable level cannot be measured reliably and the amount of the deformation corresponding to subsequent load levels can not be determined. Therefore, in this algorithm, several references, rather than a reference image, are taken during full tensile loading, as shown in Fig. 1. Several patterns, compared to a reference frame, are subsequently taken and then the last pattern is selected as a new reference. Compared to the new reference, several frames are subsequently taken. Succeedingly, the same procedure is iterated to full load. To get reliable deformation data during test, the subtraction interval against the renewed reference should be kept constant. By following this procedure, the full tensile deformation can be continuously measured from the start to end of the tensile loading

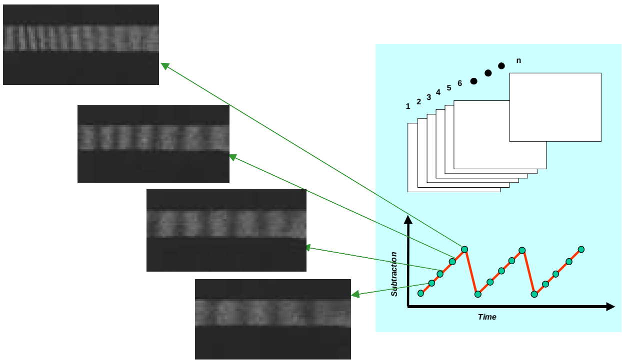

Fig. 1. Subsequent strain measurement algorithm for ESPI technique

2.2. Algorithm for enhancement of sensitivity

The sensitivity to measurement of the strain by ESPI technique can be physically defined and the maximum sensitivity may be approximately λ/2. However, in order to measure the micro-strain in the smaller scaled object, like MEMS and thin film materials, the more increased measurement sensitivity than the maximum theoretical sensitivity, which can be obtained by counting the number of fringe over the testing section in common, may be required. Therefore, to get the enhanced sensitivity to deformation measurement, the deformation within an interfringe should be quantitatively analyzed.

As found in Eq. (1), high frequency noise is included in the light intensity of the speckle pattern. It can be removed by Gaussian low pass filtering. The modulated intensity of the fringe patterns over the specimen test section may be varied with a frequency between interfringe. Thus, it can be simulated in sine wave, as shown in Fig. 2. During subsequent tensile deformation, the amplitudes of the waves for each speckle pattern, representing the different deformed states, may be nearly identical. Therefore, all the fringe patterns corresponding to the subsequent deformed states can be described as a sine function with the same amplitude and different phase, $I = A\sin(\omega X)$. Figure 2 shows examples for describing the modulated speckle fields with one or two fringes as this relation. Based on this relation, as shown in Fig. 2, the fringe pattern within the interfringe, corresponding to relatively small deformation, can be presented as a part of the sine wave. Thus, the relatively small deformation can be determined by fitting the intensities, presenting as the pattern within interfringe, with a function of sine. By following this sinusoidal fitting procedure for the modulated intensity, the sensitivity to in-plane deformation measurement may be enhanced.

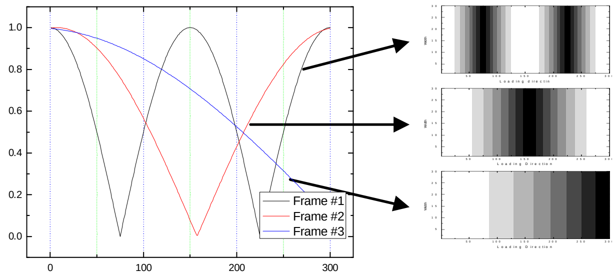

Fig. 2. Profiles and fringe patterns for the image acquired sequentially

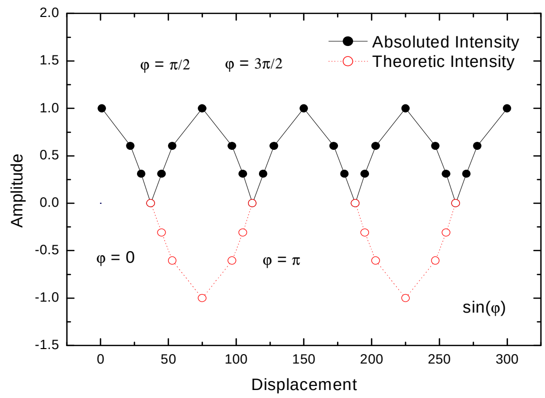

Fig. 3. Theoretical variation of fringe patterns with increasing tensile deformation

As another algorithm for obtaining the phase of the fringe pattern within the interfringe, object-induced dynamic phase shifting technique[6,7] can be used. The intensities of the pattern at an arbitrary point within the subsequently deformed field are varied with load levels, as shown in Fig. 3. As can be seen from Fig. 3, the amount of the subsequent deformation can be simulated with the phase shift of the intensity. Thus, in this technique, the phase shift can be determined by N-bucket method. In this study, 4-bucket method is used. From the speckle patterns obtained by phase shift, π/2, according to 4-bucket, phase map is determined. The inclination angle of the phase map may be used in calculating the strain. To enhance the sensitivity, the patterns within the interfringe should be used to calculate the phase shift. Then, the deformation corresponding to the interfringe can be detgermined.

3. MICRO-TENSILE TESTING SYSTEM

3.1 In-plane ESPI system for micro-tensile strain measurement

In-plane ESPI system for micro-tensile strain measurement during micro-tensile loading was arranged. A beam generated from a He-Ne laser source with capacity of 22 mW was split into two identical beams in intensity. These laser beams, which are symmetric with respect to the observation direction, were exposed on the surface of the tensile specimen. Image of the speckle pattern produced by simultaneous illumination of the surface was focused on the screen of the CCD camera through a magnifying lens. The speckle pattern images were sequentially captured in real time through a frame grabber and digitally stored.

3.2 Micro-tensile testing

Micro-tensile properties for Au film 0.5 μm thick and TiN film 1 μm thick, were measured using the micro-tensile loading system, as shown in Fig. 4, developed in this study. These two thin films were prepared by deposition on respective silicon wafers using sputtering technique. The specimen for thin films had a parallel length of 2 mm and a width of 200 μm. The actuator consisted of the system was driven by linear motor with stroke resolution of 4.5 nm and the maximum displacement of 10 mm. The load cell has a maximum capacity of 500 mN.

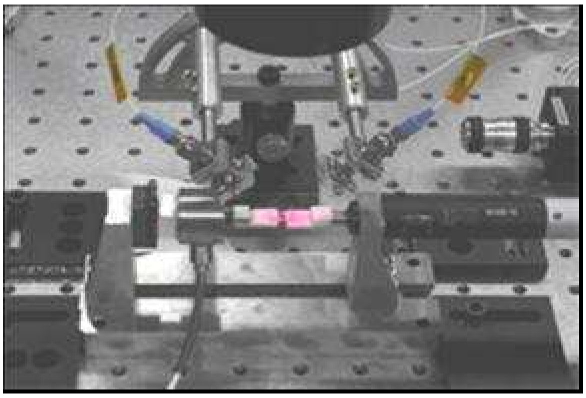

Fig. 4 Micro-tensile testing system

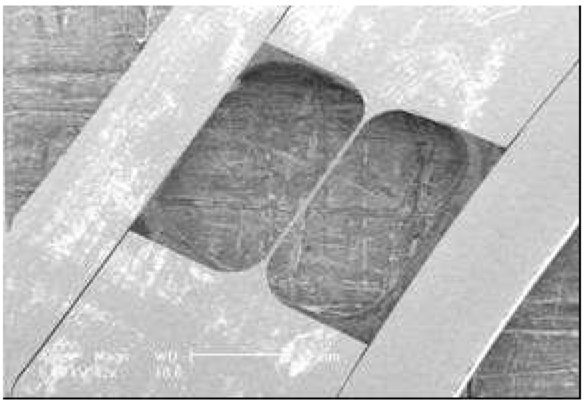

Fig. 5 Micro-sized tensile specimen

The micro-sized tensile specimens were fabricated by electromachining process, as shown in Fig. 5. The specimen was installed on the grips with UV adhesives. Micro-tensile test was carried out in displacement control at a rate of 10μm/min. During tensile testing, dual illumination generated from a He-Ne laser were exposed on the specimen with an incident angle, θ, of 45°. Speckle patterns were captured by CCD camera, equipped with zoom lens, with a two-dimensional array of 640x480 pixels. Five images of speckle patterns per second were taken during tensile loading with the frame grabber with a capture capacity of 30 frames per second. After tests, the tensile strain was measured using the algorithm, as stated above, for the continuous measurement of micro-tensile strain.

4. RESULTS AND DISCUSSION

4.1 Measurement of micro-tensile strain

Using subsequent strain measurement algorithm, the micro-tensile strain data for thin film materials, Au and TiN, were acquired as shown in Fig. 6. From the contour map of the correlation fringes over the specimen surface, the in-plane deformation over the surface can be determined by counting the fringe spacing. The in-plane deformation was calculated as the following:

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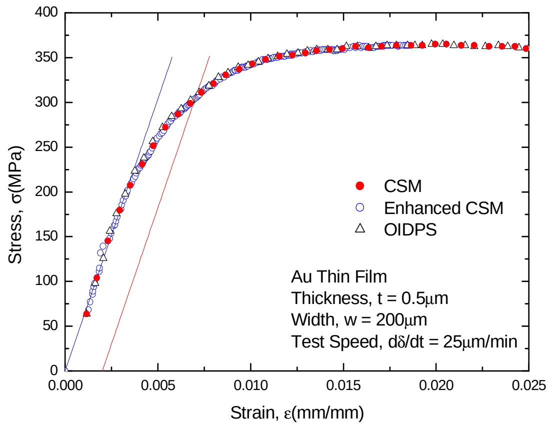
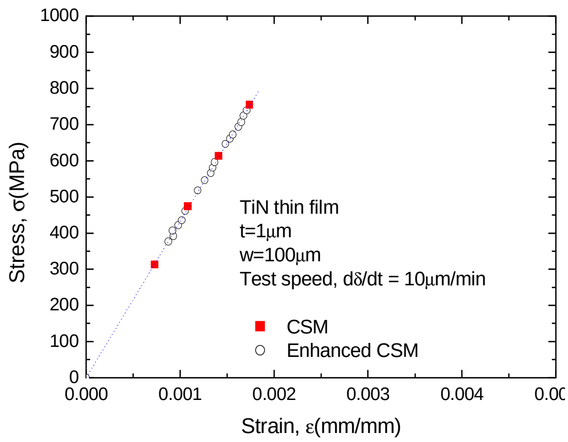
$$u = \frac{n\lambda}{2\cos\theta} \qquad (2)$$



(a) for Au thin film



(b) TiN thin film

Fig. 6. Typical micro-tensile stress-strain curve

Here, λ is the wave length of laser light source (632.8nm for He-Ne laser), n represents the number of correlation fringes and θ means incident angle of dual illumination lights. From this deformation, the strain on the test section was calculated as the following;

$$\varepsilon = \frac{\lambda}{2D_p \cos\theta} \quad (3)$$

Here, D_p represents spacing between two fringes. Using Eq. (3), values of micro-tensile strain for these thin films were determined as shown in Fig. 6.

The sensitivity to the tensile deformation measurement was enhanced by phase estimation between interfringe with sinusoidal fitting algorithm and object-induced dynamic phase shifting technique described above. The intensities of all speckle fringe patterns could be described in the form of sine wave function like $I = A\sin(\omega X)$. As shown in Fig. 7, the maximum and minimum intensities of each fringe pattern, corresponding to the successive deformation, were nearly identical, respectively. So, the intensities of the fringe patterns along the specimen axis for all frames could be represented in the following wave with the different phase, ω, and the same amplitude, A.

$$I = A\sin(\omega X - \delta) + y_o \quad (4)$$

where A and y_o were 12.3 and 29.7, respectively, for Au film. Using this algorithm, the fringe patterns, as shown in Fig. 8

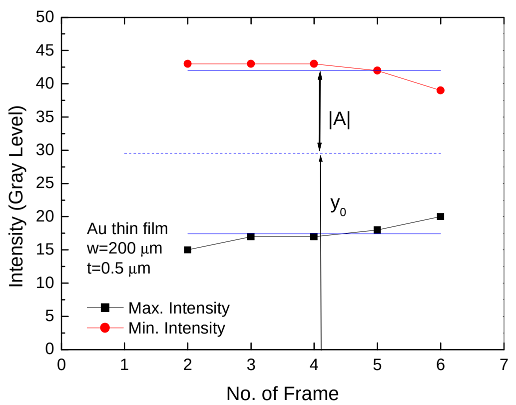
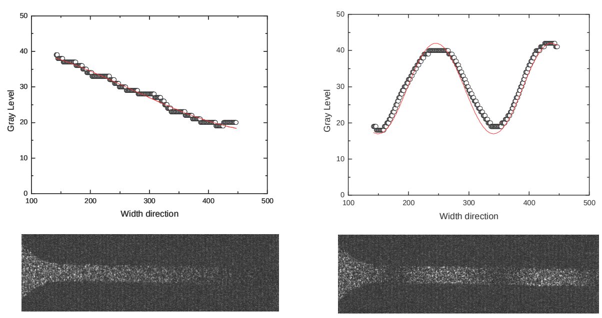


Fig. 7. Peak intensities of the fringe patterns acquired at five successive deformed states



(a) for interfringe pattern **(b) for fringe pattern**

Fig. 8. Typical fringe patterns fitted into the sine function

(b), could be fitted into a sinusoidal type like Eq. (4). Furthermore, the interfringe pattern like Fig. 8(a) could be fitted into the sine function, as well. From the fitted curve, the phase of the pattern was determined and the strain corresponding to the fringe pattern could be calculated using the following relationship.

$$\varepsilon = \frac{\omega\lambda}{4\pi f \cos\theta} \quad (5)$$

, where ω and λ are the phase determined from fitted curve and wavelength of laser source, respectively, and f is the length of inter-pixel. The micro-tensile strain data(open symbol) obtained by this procedure are plotted in Fig. 6 along with the data(solid symbol) determined by continuous strain measurement algorithm. As shown in Fig. 6, it is found that the sensitivity was enhanced by 5 ~ 6 times, compared to the sensitivity determined by the continuous strain measurement algorithm.

In Fig. 6, the strain data determined by object-induced dynamic phase shifting technique are plotted along with the data obtained by the continuous strain measurement algorithm and phase estimation with sinusoidal fitting technique. As stated before, the phase of the fringe pattern was estimated from phase map. The map was calculated from four phase-shifted interferograms, shifted by phase of π/4, by discrete Fourier transform. Figure 8 shows an example of the phase map and the unwrapped phase map for Au thin film. From the phase map, phase angle, α, could be determined and the corresponding strain, ε, was calculated by the following relation:

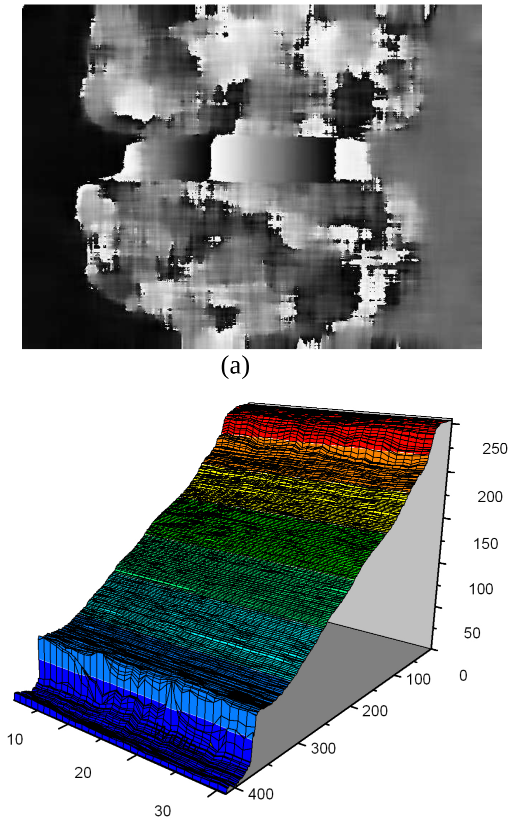
$$\varepsilon = \frac{\lambda \tan\alpha}{4\pi \sin\theta} \quad (6)$$

As shown in Fig. 6, the sensitivity of the strain data determined by the object-induced dynamic phase shifting technique is enhanced, compared to the sensitivity by continuous strain measurement algorithm.

The sensitivity to strain measurement by these two phase estimation methods might be increased with number of speckle fringe pattern images acquired during tensile loading. Therefore, it is expected that the sensitivity can be increased within a limited range by updating testing speed and image sampling speed.

4.2 Micro-tensile stress-strain curves

As shown in Fig. 6, the strain data, calculated with the subsequent strain measurement algorithm, could be correlated to the stress generated on the film by continuous micro-tensile loading. From these data, the micro-tensile stress-strain curves for Au and TiN thin films were determined. Furthermore, using strain values measured according to the sensitivity enhancement algorithms, the stress-strain curves were presented in Fig. 6 along with the curve using the data obtained by subsequent strain measurement algorithm. As can be seen in Fig. 6, the curves determined using the sensitivity enhancement algorithms like sinusoidal fitting and object-induced dynamic phase shift algorithm were identical to the curve by subsequent strain measurement algorithm. Therefore, from these stress-strain curves, elastic modulus, yielding strength, tensile strength, and elastic-plastic properties can be determined.



(a) (b)

Fig. 9. Phase map calculated from discrete Fourier transform (a) and unwrapped phase map by the addition of integral multiples of 2π(b)

4. CONCLUSION AND SUMMARY

In-plane ESPI technique was developed to measure in-plane tensile strain in micro-sized specimens of TiN and Au thin film materials with thickness of 1 μm and 0.5 μm, respectively. The micro-sized tensile specimens 200 μm wide and 2 mm long, were prepared using the electromachining process. The micro-tensile strain for these materials was measured with subsequent strain measurement algorithm developed in this study. Furthermore, algorithms for enhancing the sensitivity to measurement of in-plane tensile strain were suggested by sinusoidal fitting method and object-induced dynamic phase shift method. According to algorithms for enhancement of sensitivity, micro-tensile strain data between interfringe were calculated. It was shown that the algorithms for enhancement of the sensitivity suggested in this study make the resolution of the measured strain increased.

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