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INVESTIGATION OF NANO-INDENTATION ON FREE-STANDING FILMS FOR THE DETERMINATION OF TENSILE TEST PARAMETERS

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Abstract – With the aim to extract the mechanical properties of small volumes of materials, nowadays several methods have been proposed and are applied in practice. How to compare the results of these methods, therefore, gains more and more interests. Instrumented indentation with a spherical indenter on circularly supported free-standing thin films is believed to be an effective way to connect and compare the material testing methods including nanoindentation and micro-tensile testing. In this paper an experimental setup for making indents on free-standing films is presented, in which a modified bright-field microscope with optical sectioning capability is applied to determine the zero-point of nanoindentation, and to in-situ and real-timely measure the deflection of the film. Preliminary experimental results are reported, which coincide with the theoretical model.

Keywords: Nanoindentation instrument, free-standing film, spherical indentation, optical microscopy, optical sectioning, topography measurement.

1. INTRODUCTION

As thin films/coatings are now widely applied in microsystems, microeletromechanical systems (MEMS), etc., for protective and/or functional aims, the determination of the mechanical properties of such thin layers gains more and more importance. As a rule, most of the knowledge in characterising bulk material behaviour frequently fails to describe the material response of thin layers. Therefore a variety of methods has been proposed to investigate the mechanical properties of small volumes of material, including nanoindentation testing [1-2], laser acoustic technique [3], vibration method for microstructure [4], micro-bending method [5], micro-tensile testing [6-8], etc.

Compared with other methods, the micro-tensile test, in which the specimens to be investigated are in general free-standing films, can provide more relevant information since it measures stress and strain directly. Unfortunately, however, the clear and straightforward principle of micro-tensile testing for free-standing films cannot be realised as a simple experimental exercise, and the measurement results usually suffer a lot from the error sources within the experimental system. Therefore, how to verify or evaluate the results of testing free-standing thin films remains one of the key problems of this test.

Instrumented indentation testing in the nanometer range, also referred to as nanoindentation testing, has already been used extensively to characterize the mechanical properties of micro-scale materials including thin films, coatings, etc. In some cases nanoindentation testing has been proven to be the only means for determining both elastic and plastic mechanical properties of small volumes of material. As a result, it would be a natural choice to verify the micro-tensile test by means of nanoindentation testing on free-standing films.

2. PRINCIPLE OF MAKING INDENTS ON FREE-STANDING FILMS

As shown in Fig. 1, during the indentation test with a spherical indenter onto a circularly supported thin film, the relationship between the deformation of the thin film/membrane (h) and the indentation force (F) should be as follows [9, 10]:

$$F = \frac{9\pi}{16} \cdot \frac{E \cdot T \cdot R_{indenter}^{\frac{1}{4}}}{R_{film}^{\frac{9}{4}}} h^3 . \qquad (1)$$

in which E is the Young's modulus of the specimen's material, T the thickness of the film, $R_{indenter}$ the radius of the spherical indenter, R_{film} the in-plane radius of the film.

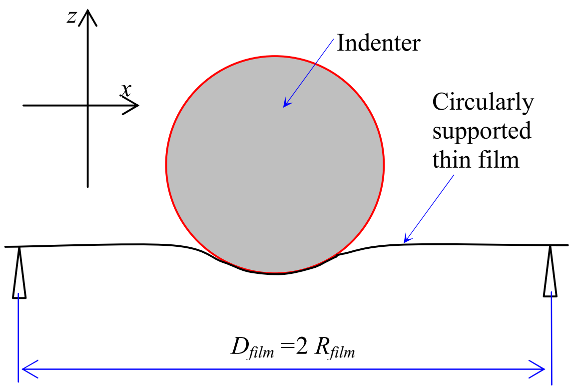

Fig. 1. Schematic diagram of the indentation testing on a free-standing thin film.

However, the clear and straightforward principle of indentation testing on free-standing films cannot be realised as a simple experimental exercise. A few technical key problems prevent experimental testing from being realized practically, including

(1) the alignment between the spherical indenter and the thin film specimen, since Eq. (1) has been deduced on the basis of axial symmetric assumption,

(2) determination of the zero-point for indentation testing, since the stiffness of the specimen is usually quite small.

In this paper, a modified bright-field optical microscope with optical sectioning capability is applied to solve the aforementioned problems.

3. A MODIFIED BRIGHT-FIELD MICROSCOPE WITH STRUCTURED LIGHT ILLUMINATION

Ordinary optical bright-field microscopes have been commonly used in material testing laboratories, e.g. for measuring the in-plane dimension of a post-indent after instrumented indentation test. However, they usually fail to detect the axial displacement of the specimen, and therefore have no capability to image the three-dimensional surface topography of the specimen.

Recently, a simple method [11] was applied to an ordinary microscope, resulting in a modified bright-field optical microscope with sectioning capability. Its principle is shown in Fig. 2:

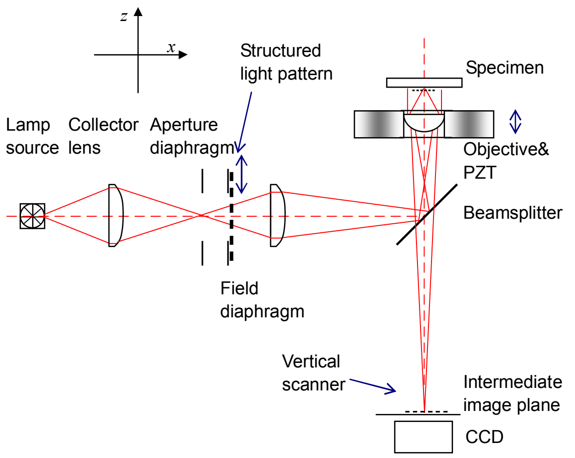

Fig. 2. Principle of a modified optical microscopy with optical sectioning capability.

The light coming from a lamp source is collected by a collector lens and projected onto the aperture diaphragm (condenser aperture), and then evenly illuminates the field diaphragm (field aperture). The position of the field aperture is conjugate to that of the specimen and of the intermediate image plane, where a CCD camera is placed. Therefore, once a structured optical element (e.g. a one-dimensional transparent diffraction grating) is inserted directly behind the field aperture, on the CCD we can see that the specimen's image is now modulated with the image of the structured element, creating the image I_i.

Let us assume that the structured pattern is a one-dimensional grid with the spatial frequency f_s. For this reason, the phase-shifting technique can be applied to remove the structured pattern and to obtain the image I_p. In the case that the objective is driven to move along the optical axis (z-axis) and a perfect plane mirror is used as the specimen, the relationship between I_p and z is found as follows [11]:

$$I_p(z) = \left| 2\frac{J_1[2uf(1-f/2)]}{[2uf(1-f/2)]} \right| \quad (2)$$

where $u = 8(\pi/\lambda)z\sin^2(\alpha/2)$, $f = M\cdot\lambda f_s/NA$, and λ is the light wavelength, $\sin(\alpha)$ is the numerical aperture (NA) of the microscope objective.

For comparison, Fig. 2 shows the typical axial responses obtained by the modified microscope and by that of a confocal microscope, which is normally defined as $I(u) = [\sin^2(u/2)/(u/2)]^2$, respectively.

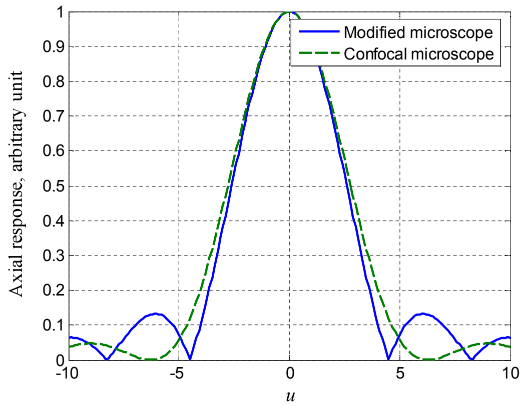

Fig. 3. Comparison between the axial responses of the modified microscope and the typical confocal microscope.

Under consideration that part of the left or right side of the axial response of the modified microscope demonstrates a quite good linearity, Lee *et al.* [12] proposed a linear fringe subdivision method, with which the axial resolution of the modified microscope can be increased to several nm. In the next section, the surface deformation of the free-standing film will be monitored in-situ and real-timely by this microscope.

4. EXPERIMENTAL INVESTIGATION

The experimental setup for making an indent on a circularly supported free-standing thin film is illustrated in Fig. 4.

A precise micro-force transducer, produced by Hysitron Inc. [13], is employed in the experiment, which can generate an indentation force with a resolution of sub-micro-Newton. However, the inner spring constant of the transducer amounts to about 150 μN/μm, which is far larger than the stiffness of the specimen, resulting in the difficult determination of the zero-point in the experiment. A spherical indenter with a diameter of 800 μm is mounted onto the transducer, which is moved by a piezoelectric stage (PZT 1, P840, Physik Instrumente, 1 nm resolution with closed-loop control) to engage the specimen.

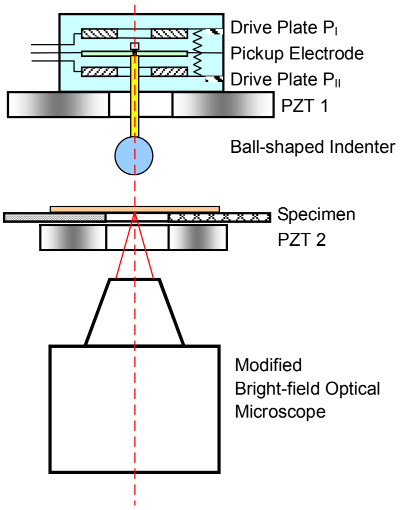

Fig.4. Schematic diagram of the experimental setup.

A silver foil with a nominal thickness of 3 μm is fixed onto a steel plate with a hole of 2 mm diameter, forming the specimen in Fig. 4. Further, the specimen is mounted on a piezoelectric stage (PZT 2, P713, Physik Instrumente, Germany, scan stroke: 15 μm x 15 μm, 1 nm resolution in closed loop), with which surface scanning of the specimen along the z-axis can be realized.

The modified microscope has been developed on the basis of a reverse bright-field microscope [14]. A 10-bit CCD camera is used in Fig. (4), the typical exposure time for one frame is 2 ms. The resonance frequency of the piezoelectric translator for driving the optical grid is 14 kHz, however, the actual working frequency of the translator is designated to 15 Hz. In order to obtain the 3D topography of the specimen, three frames of images have to be captured, therefore the maximum imaging speed of the modified microscope is 15 Frame/s. Finally the photography of the experimental setup is shown in Fig. 5.

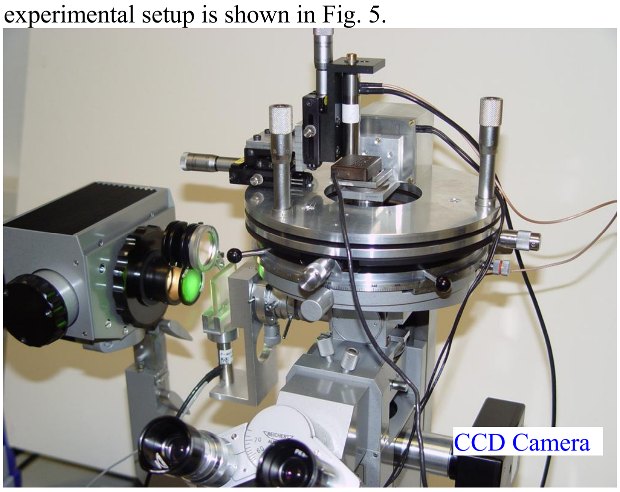

Fig. 5. Photography of the experimental setup for making indents on a free-standing film.

In the experiment, an objective with 0.90 *NA* (80×) was firstly employed to determine the zero-point of the indentation, which has a linear region of 0.36 μm and a measurement uncertainty of 2.3 nm [14]. In order to in-situ image the deformation of the silver film during the indentation test, an objective with 0.25 *NA* (16×) is applied. The corresponding linear region and z-axis uncertainty are about 5 μm and 56.7 nm, respectively, as shown in Fig. 6.

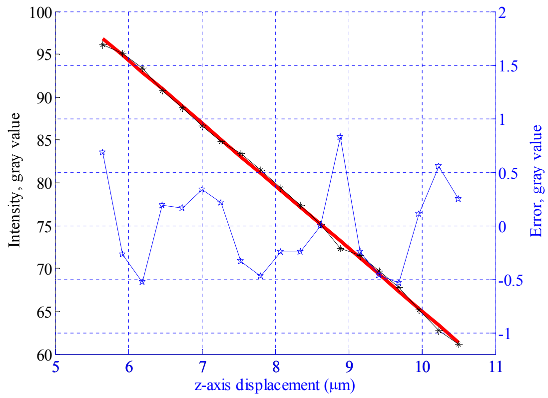

Fig. 6. Linear region and measurement uncertainty of the modified microscope with an objective of 0.25 *NA* (16×).

During the nanoindentation test, the loading and unloading procedure takes 20 s, the hold time is defined to 5 s. Preliminary experimental results are shown in Fig. 7. The theoretical curves are calculated with the parameters: $E = 83$ GPa, $T = 3$ μm, $R_{indenter} = 400$ μm, $R_{film} = 1000$ μm.

Figure 7(a) shows the relationship between the indentation force and the deflection of the film under nanoindentation test without hold time, while Fig. 7(b) is obtained with an additional hold time of 5 s. It can be seen that due to creep of the specimen, the indentation force generated by the micro-force transducer cannot be maintained anymore, which indicates that a better force transducer is expected for the experiment in the next step.

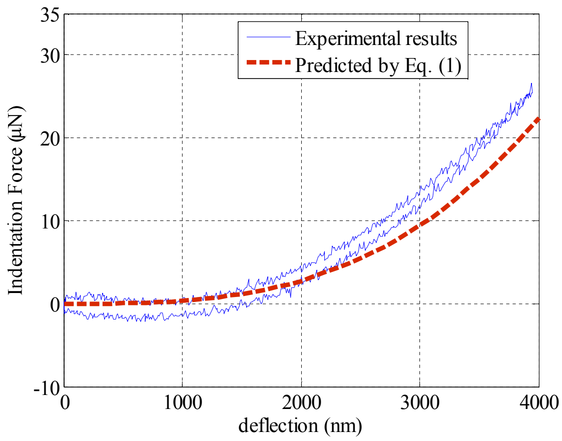

Fig.7(a). Experimental results of nanoindentation testing without hold time.

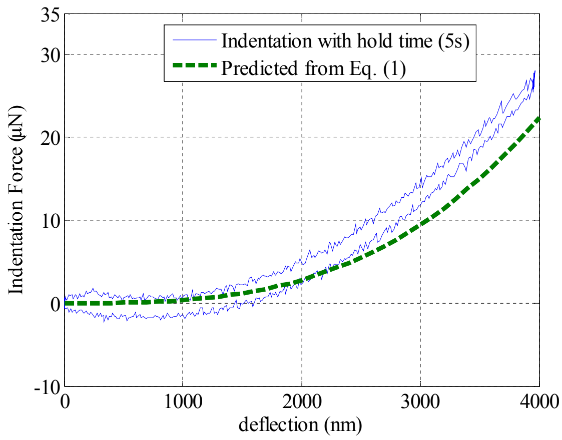

Fig. 7(b). Experimental results of nanoindentation testing with hold time (5 s).

The deviation between the experimental data and the theoretical model can be caused by a few error sources within the experiment, including the thickness tolerance of the silver film, friction between the spherical indenter and the film which is not taken into consideration in Eq. (1), residual stress within the film, etc.

5. CONCLUSION

In this paper an experimental set-up for the determination of the mechanical properties of thin films by means of nanoindentation has been developed, which employs a micro-force transducer with sub-micro-Newton resolution and a ball-shaped indenter to make indents on a circularly supported silver film. A modified optical microscope with axial sectioning capability was applied for in-situ and real-timely inspection of the surface deflection of the silver film. Preliminary experimental results coincide with the theoretical models for spherical indentation on free-standing films. However, further improvements on the experimental setup would be necessary.

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