Measurement of the mechanical properties of nickel film based on the full-field deformation: An improved blister method

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Abstract To characterize the mechanical properties of thin films, an improved blister method is proposed, which combines a digital speckle correlation method with the blister test. Based on this method, an experimental setup is developed to measure Young’s modulus, residual stress, and interfacial adhesion energy of an electroplated nickel film. The results show that the improved blister method has the advantage of a high accuracy full-field measurement with the simple operation and low requirement on environments, which can be used to characterize the mechanical properties of films with various scales from laboratory to industrial applications.

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1. Introduction

Thin films have been widely used in wear-resistant coating on cutting tools [1], anticorrosion coating of plates [2] and thermal barrier coatings on turbine blades [3]. Unfortunately, the premature fracture usually occurs in films or coatings when they are in service. The mechanical properties of thin films play an important role in their design and applications. Many techniques have been used to characterize the mechanical properties of bulk materials, but they cannot be directly applied to thin films [4,5]. Over the last decades, some new methods have been developed to measure the mechanical properties of thin films such as uniaxial tension [6], indentation [7],...
scratching [8], etc. Among these methods, samples are easily prepared in indentation and scratching tests, however, the measurement results are often affected by substrate [9] and the thickness of a thin film [10]. In contrast, it is difficult to fabricate and handle the samples in uniaxial tensile tests in a micron or submicron scale.

Similar to indentation and scratching tests, the advantage of a blister method is the minimal sample preparation and handling. Furthermore, the blister test can be used to estimate the adhesion energy of film/substrate because the dissipated energy in the test contributes to interfacial debonding. Hence, the blister test has been widely applied to characterize the mechanical properties of silicon nitride [11–17], polymer [18,19], metal [20–24], and diamond films [1]. It is worth noting however, that the accuracy and reliability of the blister test is mainly affected by the assumed load-deflection equation, samples and data measurements [25]. Small and Nix analyzed the influence of initial conditions, such as residual stress and thickness of films, by finite element simulation [26]. Maier-Schneider et al. [27] compared the square film models proposed by Tabata et al. [16] and Vlassak and Nix [11], and with those obtained by electronic speckle pattern interferometry (ESPI). Yan et al. realized deformation measurements, and it is difficult to fabricate and handle the films [10]. In contrast, it is difficult to fabricate and handle the films [10].

Generally, the deformation measurement is essential for the determination of physical and mechanical properties in the blister test. The methods used to conduct the deformation measurement include using a cathetometer [30] and displacement sensor [3]. However, these traditional technologies are only suitable for point measurements, and it is difficult to extract the meso/micro-deformation information such as interface debonding. Hence, it is necessary to measure the three-dimensional deformation morphology of displacements or strains. The whole field measurement techniques include the fringe projection [31], speckle interference [20], and surface profiler [23]. These methods can be used to determine the deformation of a film at different points, but their experimental operation is complex and due to external vibration, the interferometric beam may easily deviate from the original optical path.

The digital speckle correlation method (DSCM) is applied for deformation measurements with the advantage of simple optical path, high accuracy and no requirement of vibration isolation [32,33]. According to the investigation by Zhu et al. [32], an accurate three-dimensional deformation measurement system calibrated with telemetric lens was developed. The measurements on deformation by DSCM are in agreement with those obtained by electronic speckle pattern interferometry (ESPI). Yan et al. realized the orientation function of an optical mouse based on DSCM [33]. The experiments showed that such an orientation function is consistent with simulation results. To the best of our knowledge, there are few studies on the deformation measurement by using DSCM in the blister test. In this paper, DSCM is used to study the deformation characterization of nickel films. Typical mechanical properties are analyzed based on the evolution of the displacement field during the blister test.

2. The principle of DSCM

In the application of DSCM, speckle patterns on a specimen surface before and after deformation are digitized into source and target images. As illustrated in Fig. 1(a), points \( P(x_P, y_P) \) and \( Q(x_Q, y_Q) \) in the source image move to \( P^* \) and \( Q^* \) in the target image. The relationship of these two points can be written as

\[
\begin{align*}
    x_Q &= x_P + \Delta x \\
    y_Q &= y_P + \Delta y
\end{align*}
\]

where \( \Delta x \) and \( \Delta y \) are the distances between points \( P \) and \( Q \) along \( x \) and \( y \) directions, respectively. After deformation, displacements of point \( P \) are \( u_P \) and \( v_P \) in the \( x \) and \( y \) directions, respectively. Then

\[
\begin{align*}
    x_P^* &= x_P + u_P \\
    y_P^* &= y_P + v_P
\end{align*}
\]

Similarly, displacements of point \( Q \) are

\[
\begin{align*}
    x_Q^* &= x_Q + u_Q \\
    y_Q^* &= y_Q + v_Q
\end{align*}
\]

In consideration of tensile and shear effects with very small \( \Delta x \) and \( \Delta y \), \( u_P \) and \( v_Q \) can be represented as

\[
\begin{align*}
    u_Q &= u_P + \frac{\partial u_P}{\partial x} \Delta x + \frac{\partial u_P}{\partial y} \Delta y \\
    v_Q &= v_P + \frac{\partial v_P}{\partial x} \Delta x + \frac{\partial v_P}{\partial y} \Delta y
\end{align*}
\]

Substituting Eqs. (1) and (4) into (3), we have

\[
\begin{align*}
    x_Q^* &= x_P + u_P + \frac{\partial u_P}{\partial x} \Delta x + \frac{\partial u_P}{\partial y} \Delta y \\
    y_Q^* &= y_P + v_P + \frac{\partial v_P}{\partial x} \Delta x + \frac{\partial v_P}{\partial y} \Delta y
\end{align*}
\]

That is, for an arbitrary point \( Q \), we have

\[
\begin{align*}
    x^* &= x + u + \frac{\partial u}{\partial x} \Delta x + \frac{\partial u}{\partial y} \Delta y \\
    y^* &= y + v + \frac{\partial v}{\partial x} \Delta x + \frac{\partial v}{\partial y} \Delta y
\end{align*}
\]

In the case of a very small \( PQ \), the following relationships can be obtained:

\[
\begin{align*}
    dx &= x_Q - x_P \\
    dy &= y_Q - y_P \\
    dx^* &= x_Q^* - x_P^* \\
    dy^* &= y_Q^* - y_P^*
\end{align*}
\]

It is obvious that the distances of \( PQ \) before and after deformation are

\[
\begin{align*}
    |PQ|^2 &= (dx)^2 + (dy)^2 \\
    |P^*Q^*|^2 &= (dx^*)^2 + (dy^*)^2
\end{align*}
\]

Thus, the strain in the \( x \) direction can be defined as

\[
\epsilon_{xx} = \frac{|P^*Q^*| - |PQ|}{|PQ|} \approx \frac{\partial u}{\partial x} + \frac{1}{2} \left[ \left( \frac{\partial u}{\partial x} \right)^2 + \left( \frac{\partial v}{\partial x} \right)^2 \right]
\]

Following the same procedure, the strain components in other directions can be written as

\[
\epsilon_{xx} = \frac{\partial u}{\partial x} + \frac{1}{2} \left[ \left( \frac{\partial u}{\partial x} \right)^2 + \left( \frac{\partial v}{\partial x} \right)^2 \right]
\]
The in-plane displacement field \((u, v)\) can be determined by matching subsets \(S\) of \((2M+1) \times (2M+1)\) pixels and the corresponding subset \(S^*\) before and after the deformation, as shown in Fig. 1(a). A typical correlation function is defined as:

\[
K = \sum_{i=-M}^{M} \sum_{j=-M}^{M} [f(x_i, y_j) - \bar{f}] [g(x_i', y_j') - \bar{g}] \left(\frac{\partial f}{\partial x} \frac{\partial g}{\partial x'} + \frac{\partial f}{\partial y} \frac{\partial g}{\partial y'}\right)
\]

where \(K\) is the correlation coefficient, \(f(x_i, y_j)\) and \(g(x_i', y_j')\) are the gray values of points in subsets before and after deformation, respectively, and \(\bar{f}\) and \(\bar{g}\) are the average gray values of \(f(x_i, y_j)\) and \(g(x_i', y_j')\), respectively.

The deflection \(\omega_0\) can be expressed as:

\[
l = MN = L\omega_0 / (H - \omega_0)
\]

where \(L\) is the distance between the projector and CCD camera and \(H\) is the distance from the CCD camera to specimen. Due to \(\omega_0 \ll H\), Eq. (12) can be simplified as:

\[
\omega_0 = (H/L)l = Al
\]

where the coefficient \(A\) is a constant, which is dependent only on the experimental setup.

3. An improved blister method

The schematic diagram of the blister test is shown in Fig. 2. A uniform pressure is loaded on a thin film with the thickness of \(t\) and the radius of \(a\). The film blisters under the uniform pressure \(P\), and as pressure increases by \(\Delta P\), the film debonds from the substrate with the debonding radius of \(\Delta a\) and the deflection of \(\Delta \omega_0\). With the help of a theoretical model, the mechanical properties can be evaluated based on the height of blister and the corresponding pressure.
To verify the accuracy of this setup, the deflection curves for the film center \( a_0 \) versus pressure \( P \) are measured by ESPI and DSCM, respectively. As shown in Fig. 3(b), the images of the specimen surface are concentric circles for ESPI and speckles for DSCM, respectively. It is seen that the results of DSCM are in agreement with those of ESPI and the deviation is less than 0.4 \( \mu m \) based on \( P-a_0 \) curves.

Fig. 3  (a) Sketch map of the experimental setup and (b) the deflection curve of the membrane center \( a_0 \) versus pressure \( P \) measured by DSCM and ESPI, respectively.

4. Typical experiments

4.1. Specimens

The substrate is a cylinder stainless steel with diameter and thickness 34 mm and 3 mm, respectively. A hole with the radius of 1.6 mm is machined at the center of substrate. To measure the mechanical properties of electroplated nickel films, the specimens are divided into two groups. The specimens of group A are fabricated by gluing the freestanding electroplated nickel film to the stainless steel substrate with epoxy. Here, the Ni film can be peeled off from stainless steel by weakening the adhesive strength between the Ni film and the substrate during an electroplating process, such as shortening the activated time of the substrate surface, reducing the surface cleanliness of substrate, and increasing the thickness of coating. When it is peeled from the substrate, however, the freestanding film becomes flat because residual stress is released.

In order to determine the residual stress and interfacial adhesion energy, the specimens of group B are prepared by electroplating the nickel film on substrate. The film is obtained with nickel sulfate electrolyte, which is composed of 250 g of \( NiSO_4 \cdot 6H_2O \), 50 g of \( NiCl_2 \cdot 6H_2O \) and 35 g of \( H_2BO_3 \) per liter. Pure nickel is used as the anode. The \( \text{pH} \) value is adjusted with sulfuric acid to 4.0 at 42 °C. A conventional rotating disc electrode is used in electrodeposition. Before electroplating, pretreatments are necessary to get rid of impurities. The thickness of the glued and electroplated specimens is 30 \( \mu m \).

4.2. Young’s modulus and residual stress

Based on the DSCM technology, the three-dimensional deformation of the nickel film and its corresponding contour map is given in Fig. 4. The deformation profile of the film can be offered and its center deflection \( a_0 \) can be gained from the maximum height (see Fig. 4(a) and (b)). The deflection of the film center \( a_0 \) versus pressure \( P \) for nickel films in group A is shown in Fig. 4(c). Because the edge of the circle film is clamped on substrate, the boundary conditions are

\[
\omega = 0 \quad \text{and} \quad \frac{da}{dr} = 0, \quad \text{for} \quad r = a
\]

\[
\omega = a_0 \quad \text{and} \quad \frac{da}{dr} = 0, \quad \text{for} \quad r = 0
\]

when the deflection of a thin film is much smaller than its thickness, the relationship between \( P \) and \( a_0 \) can be written as [31]

\[
P a^4 \frac{d^4}{E} = \epsilon(v) r^2 a_0 + f\left(\frac{\sigma}{E}\right) r^2 a_0
\]

where \( a \) and \( t \) are the radius and the thickness of a film, respectively, \( P \) is the pressure, \( E \) is the Young modulus, \( a_0 \) is the out-of-plane deflection of the film center, \( \nu \) is the Poisson ratio, \( \sigma \) is the residual stress in the film and functions \( \epsilon \) and \( f \) are given by

\[
\epsilon(v) = \frac{5.333}{(1-v^2)}
\]

\[
f\left(\frac{\sigma}{E}\right) = 0.552 \sigma \left[2.755 + v-2.755v-v^3\right].
\]

If residual stress in a film can be ignored, Eq. (15) can be simplified as
where the stiffness $D$ is determined by the following expression:

$$D = \frac{E t^3}{12(1-\nu^2)}$$

The residual stress of nickel film in group A can be ignored because the samples are fabricated by gluing the free-standing nickel film to substrate. Young’s modulus of the nickel film can be obtained from the slope of the fitted curve by Eq. (18). Given that Poisson’s ratio of nickel film is 0.3, its Young’s modulus can be calculated as 229.36 GPa. To confirm this result, the uniaxial tensile experiment is performed on free-standing films with the same thickness. Fig. 5(a) shows the shape and size of a tensile specimen. The test is performed on a RG2000 micro-machine-controlled universal tensile machine with the tensile rate of 0.01 mm/s. As shown in Fig. 5(b), Young’s modulus is 221.71 GPa, which agrees well with the result obtained by the blister test.

The residual stress in the electroplated nickel film can also be obtained from the center deflection $\omega_0$ versus pressure $P$ of specimens in group B. The residual stress calculated by Eq. (15) is $143.75 \pm 5.44$ MPa.

### 4.3. Interfacial adhesion energy

As shown in Fig. 6(a), the deflection of the nickel film increases monotonically with the increase of pressure at the initial stage. After point $T(P_c, \omega_c)$, there is a debonding between film and substrate with the decrease of pressure and the quick increase of its corresponding deflection. Fig. 6(b) is the deflection of the nickel film in the $xz$ plane at different pressures. When the pressure reaches to the critical value of 0.393 MPa, the film debonds from the substrate. The volume of the blister quickly increases and the pressure applied on nickel film decreases to 0.391 MPa.

In consideration of the influence of residual stress on the film, the interfacial adhesion energy $G_a$ of film/substrate can be represented as

$$G_a = 0.516 P_c \omega_c$$

where $P_c$ and $\omega_c$ are the critical pressure and the corresponding deflection of the film center, respectively. The interfacial adhesion energy of nickel film/stainless is obtained as $9.33 \pm 1.90$ J/m$^2$.

### 5. Conclusions

In this paper, an improved blister method with the high accuracy and full-field measurement has been suggested by combining DSCM and the blister test. A setup with the measuring precision up to 0.4 μm is designed based on this method. Young’s modulus, residual
stress and interfacial adhesion energy of electroplated nickel film are measured. Young's modulus of nickel film is 229.36 GPa, which is in agreement with that obtained by the uniaxial tensile test. The residual stress and interfacial adhesion energy of electroplated nickel film before and after debonding and (b) is its deflection in the x-z plane at different pressures.

Fig. 6  The deflection of film in group B, where (a) is the $\varepsilon_0$-$P$ curve of nickel film before and after debonding and (b) is its deflection in the x-z plane at different pressures.

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