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Measurement of Poisson's Ratio of a Thin Film on a Substrate by Combining X-Ray Diffraction with *in situ* Substrate Bending

Ho-Young Lee,[1] Su-Jeong Suh,[2,*] Sung-Ryong Kim,[3] Se-Young Park,[2] and Young-Chang Joo[1]

[1]Department of Materials Science and Engineering, Seoul National University, Seoul 151-742, Korea
[2]School of Advanced Materials Science and Engineering, Sungkyunkwan University, Suwon 440-746, Korea
[3]Department of Polymer Science and Engineering, Chungju National University, Chungju 380-702, South Korea

A method to determine Poisson's ratio of a crystalline thin film on a substrate without sophisticated equipment and/or specimens is presented. The method is based on the combining x-ray diffraction and *in situ* substrate bending and the three components of the induced strains were measured by x-ray diffraction experiments using the well-known $\sin^2\psi$ method. The method allows one to extract the Poisson's ratio of the unpatterned thin films on substrates in a simple way and with a good precision. As an example, Poisson's ratio of a sputter-deposited copper film on a low-carbon steel substrate was measured.

Keywords: poisson's ratio, thin films, x-ray diffraction, copper film

1. INTRODUCTION

Mechanical properties of thin films and multilayers are essential parameters for advanced devices.[1] Thus, there has been an increasing interest about the mechanical properties and the elastic properties of thin films and multilayers which can differ significantly from the bulk metal ones.[2] Poisson's ratio is one of the fundamental parameters of thin films characterizing mechanical behaviors and the performance of the devices made of thin films. Many previous researchers have tried to measure the Poisson's ratio of thin films using various methods. Moram *et al.* measured Poisson's ratio of the epitaxially grown (111)-oriented scandium nitride thin films on silicon by evaluating in-plane and out-of-plane strains using high-resolution x-ray diffraction technique.[3] Renault *et al.* determined Poisson's ratio using a synchrotron x-ray diffractometer equipped with an extra tensile tester.[4] Kim *et al.* evaluated Poisson's ratio using nano-indentator with double-ring shaped specimen.[5] J. Ye *et al.* determined the Poisson's ratio of thin films by detecting thermal expansion in two directions perpendicular to each other.[1] Zhao *et al.* measured Poisson's ratio of thin films on silicon substrates by using wafer curvature technique.[6] However, the methods mentioned above often require sophisticated equipment and/or specially designed specimens. Therefore, a simple method for measuring Poisson's ratio is strongly necessary. We describe a simple method for the measurement of the Poisson's ratio of the thin films on substrates without any additional surface micromachining process.

*Corresponding author: suhsj@skku.ac.kr

2. THEORY

The method used in this work is based on the well-known $\sin^2\psi$ method.[7] It consists of *in situ* bending of film/substrate system in an x-ray diffractometer (Fig. 1). The Poisson's ratio of the films is determined by measuring three strain components induced by substrate bending (Fig. 1). The main assumption is the linear elastic behavior of both the substrate and the thin film, and perfect adhesion between the substrate and the film.

Consider a body under external loadings in a Cartesian coordinate system. We define measurable or apparent strain component, ε_i^m, and true or effective strain component, ε_i^t. Then each component of the measurable strain may be expressed as

$$\varepsilon_1^t = \varepsilon_1^m + \nu_{12}\varepsilon_2^t + \nu_{13}\varepsilon_3^t \tag{1}$$

$$\varepsilon_2^t = \varepsilon_2^m + \nu_{23}\varepsilon_3^t + \nu_{21}\varepsilon_1^t \tag{2}$$

$$\varepsilon_3^t = \varepsilon_3^m + \nu_{31}\varepsilon_1^t + \nu_{32}\varepsilon_2^t \tag{3}$$

where is the Poisson's ratio defined as

$$\nu_{ij} = -\frac{\varepsilon_i}{\varepsilon_j}(i \neq j) \tag{4}$$

In case of a thin film on a substrate, there is no external loading along film normal direction (ε_3^t=0). Thus

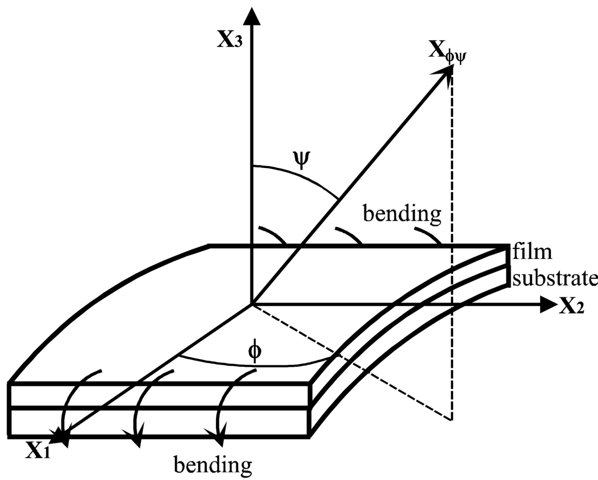

Fig. 1. Representation of the specimen coordinate system, *X*, and the angle ϕ and ψ. $X_{f\psi}$ is the normal to the diffracting plane.

$$\varepsilon_1^f = \varepsilon_1^m + \nu_{12}\varepsilon_2^f \quad (5)$$

$$\varepsilon_2^f = \varepsilon_2^m + \nu_{21}\varepsilon_1^f \quad (6)$$

$$\varepsilon_3^f = -\nu_{31}\varepsilon_1^f + \nu_{32}\varepsilon_2^f \quad (7)$$

Considering Eqs. 5 and 6, ε_1^f and ε_2^f are given by

$$\varepsilon_1^f = \frac{\varepsilon_1^m + \nu_{12}\varepsilon_2^m}{1-\nu_{12}\nu_{21}} \quad (8)$$

$$\varepsilon_2^f = \frac{\varepsilon_2^m + \nu_{21}\varepsilon_1^m}{1-\nu_{12}\nu_{21}} \quad (9)$$

Therefore, Eq. 7 can be rewritten as

$$\varepsilon_3^m = -\nu_{31}\frac{\varepsilon_1^m + \nu_{12}\varepsilon_2^m}{1-\nu_{12}\nu_{21}} - \nu_{32}\frac{\varepsilon_2^m + \nu_{21}\varepsilon_1^m}{1-\nu_{12}\nu_{21}} \quad (10)$$

Assuming in-plane isotropic (ν_{12}=ν_{21}) and out-of-plane isotropic (ν_{31}=ν_{32}), then Eq. 10 becomes

$$\varepsilon_3^m = -\nu_{31}\frac{\varepsilon_1^m + \varepsilon_2^m}{1-\nu_{12}} \quad (11)$$

In case of fully isotropic (), one may write

$$\varepsilon_3^m = -\nu_f\frac{\varepsilon_1^m + \varepsilon_2^m}{1-\nu_f} \quad (12)$$

If the film is perfectly adhered to the substrate ($\varepsilon_2^m = -\nu_s\varepsilon_1^m$), then Eq. 12 becomes[8]

$$\varepsilon_3^m = -\nu_f\frac{1-\nu_s}{1-\nu_f}\varepsilon_1^m \quad (13)$$

where ν_f and ν_s are the Poisson's ratio of the film and the substrate, respectively.

In case of biaxial isostrain ($\varepsilon_1^m = \varepsilon_2^m$) and isotropic films, Eq. 12 becomes[9]

$$\varepsilon_3^m = \frac{-2\nu_f}{1-\nu_f}\varepsilon_1^m \quad (14)$$

It is common to use x-ray diffraction to measure the spacing of the lattice planes in crystalline materials. When a crystal is subjected to a stress in an elastic range, it gives rise to change of the lattice plane spacing. The strain state can be determined from a set of measured interplanar spacing as a function of $\sin^2\psi$, where ψ is the angle between the normals of the films surface and of the diffracting plane. This technique has been described by Flinn.[10]

The linear relationship between crystallographic interplanar spacing d and $\sin^2\psi$ has been established for the measurement of the macroscopic stress.[7] To obtain the oisson's ratio, it is necessary to get the $\varepsilon-\sin^2\psi$ straight lines as a function of applied bending strain using the x-ray diffraction. The conversion from $d-\sin^2\psi$ relationship to $\varepsilon-\sin^2\psi$ relationship could be possible by measuring the d at different applied bending strains. For example, if the d is measured at zero applied bending strain (no substrate bending) and consequently, the d is measured under applied bending strain in a linear elastic range, the conversion can be attainable. On the other hand, since there must be non-zero residual strain of the films in spite of zero applied bending strain, Eq. 4 should be replaced by the following equation in a linear elastic range.

$$\nu_{ij} = -\frac{\Delta\varepsilon_i}{\Delta\varepsilon_j}(i \neq j) \quad (15)$$

The strains in the crystalline lattice of films induced by substrate bending lead to a shift of the diffraction peak with respect to its previous position. It is apparent that the strain state of the film on the bended substrate is an anisotropic biaxial one. And when substrate thickness is much greater than film thickness, the stress gradient along the thickness direction of the film is negligible.[11]

3. EXPERIMENTS

5 cm-by-5 cm-sized low-carbon steel-plate with 0.5 mm thickness (commercial name: bright steel sheet, provided by Dongbu Steel Co. Ltd. in Korea) was prepared as a substrate and cleaned. And then, 0.5 μm-thick Cu thin film was deposited on the substrate by a sputtering system (200 W at 4 mtorr), and the Cu-deposited steel substrate was loaded on the specially designed substrate holder. The substrate holder was designed to bend the substrate properly during the x-ray diffraction. The orientation of the sample is characterized by the ϕ angle, denoting the rotation angle of the specimen around its surface normal, and by the angle ψ, being the angle between the normal to the surface and the normal to the diffracting lattice planes (Fig. 1). The specially designed substrate holder (Fig. 2) mounted on a specially designed

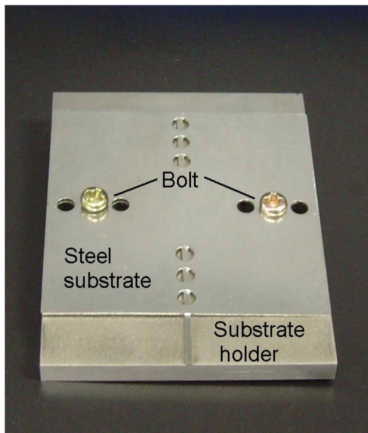

Fig. 2. A low-carbon steel substrate mounted onto a substrate holder.

sample holder on goniometer. The $\sin^2\Psi$ method was carried out using a Bruker D8 Discover system with a Cu Kα radiation source. The operating voltage and current were 40 kV and 40 mA, respectively. The location of Bragg peak was determined by fitting the peak with the Gaussian function (Fig. 3).

4. RESULTS AND DISCUSSION

According to the x-ray diffraction patterns of as-deposited Cu thin film and steel substrate obtained by θ-2θ scan (Fig. 4), it is investigated that many diffraction peaks of the Cu thin film, except (311) as well as (111) peaks, coincide with those of the steel substrate. Therefore, we have chosen (111) plane as a strain measuring diffracting plane. Since different {111} planes can be found at different angles to the film normal direction, the angle ψ was fixed at 0° and 70.53°, respectively. Then, the angle ϕ was fixed at 0° and 90° at every ψ (Fig. 5).

The additional strain induced by substrate bending was calculated by the following equation.

$$\Delta\varepsilon_{\phi\psi} = \frac{\Delta d}{d} = -\frac{\pi}{360}\frac{\Delta(2\theta_{Bragg})}{tan(\theta_{Bragg})}\ \text{(deg.)} \tag{16}$$

where $\Delta\varepsilon_{\phi\psi}$ is the elastic lattice strain in the direction defined by the Euler angles ϕ and ψ with respect to the reference frame of the sample. Neglecting the shear strain components, $\Delta\varepsilon_{\phi\psi}$ may be expressed in terms of the strain components, $\Delta\varepsilon_i$ induced by substrate bending in the sample coordinate system.

$$\Delta\varepsilon_{\phi\psi}=\Delta\varepsilon_1\sin^2\psi\cos^2\phi+\Delta\varepsilon_2\sin^2\psi\sin^2\phi+\Delta\varepsilon_3\cos^2\psi \tag{17}$$

Using Eq. 17 we can get $\Delta\varepsilon_3$ directly by setting $\psi = 0$, then $\Delta\varepsilon_{\phi\psi}$ becomes $\Delta\varepsilon_3$. And $\Delta\varepsilon_1$ and $\Delta\varepsilon_2$ can be obtained by changing ϕ with the knowledge of $\Delta\varepsilon_3$. The Poisson's ratio of the film, v_f, can be obtained by the following equation, which is the same form of the Eq. 12.

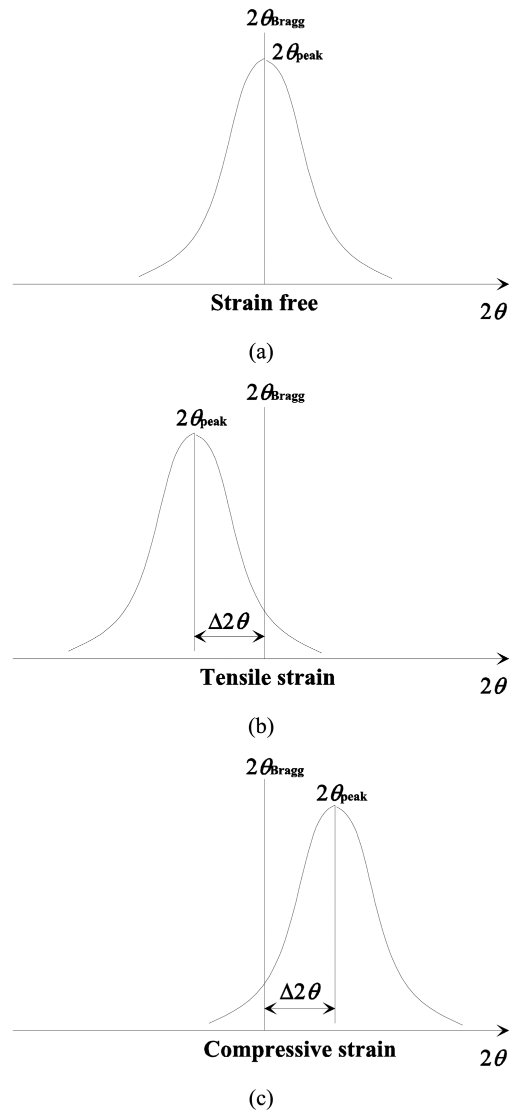

Fig. 3. Relationship between peak shift and strain state: (a) strain-free state ($2\theta_{peak}=2\theta_B$), (b) tensile strain state ($2\theta_{peak}<2\theta_B$), (c) compressive strain state ($2\theta_{peak}>2\theta_B$).

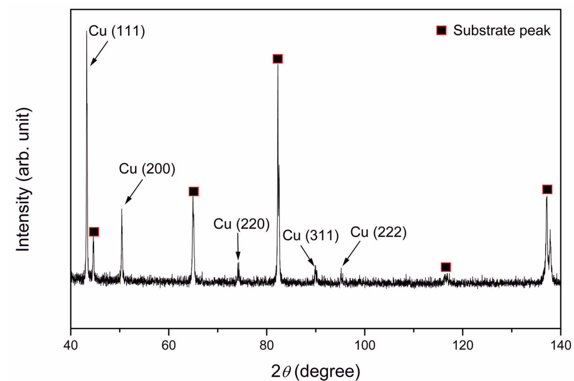

Fig. 4. X-ray $\theta/2\theta$ diffraction patterns of 0.5 μm-thick Cu film on a low-carbon steel substrate.

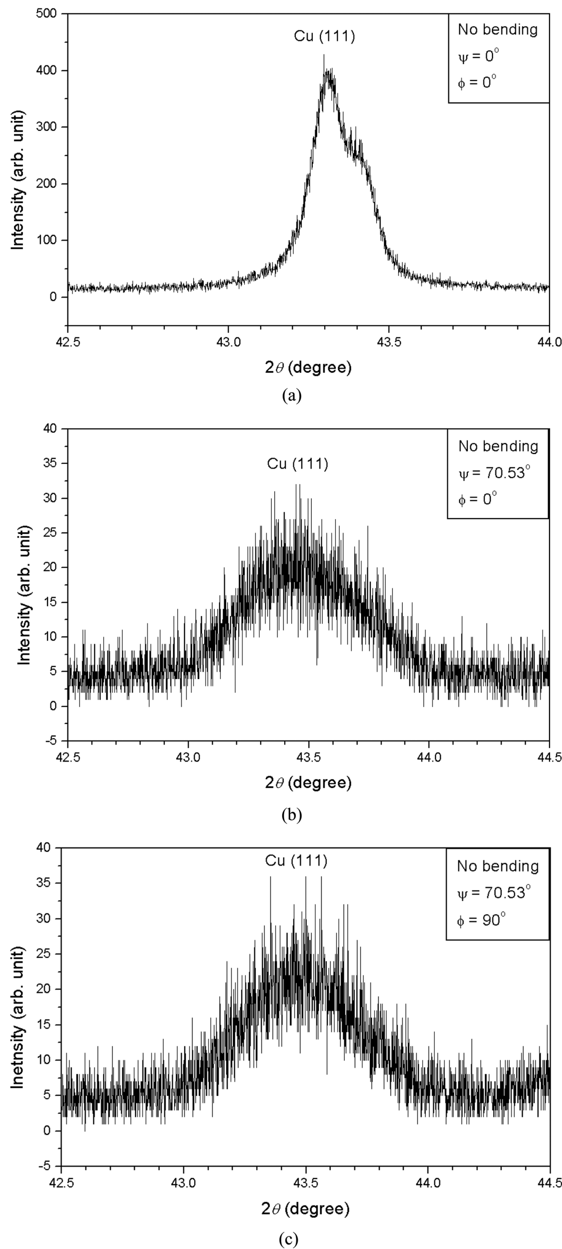

Fig. 5. Cu (111) diffraction peaks of 0.5 mm-thick Cu film on a low-carbon steel substrate with respect to ψ and ϕ under no-bending state: (a) $\psi = 0°$, $\phi = 0°$, (b) $\psi = 70.53°$, $\phi = 0°$, (a) $\psi = 70.53°$, $\phi = 90°$.

$$\Delta\varepsilon_3 = -\nu_f \frac{\Delta\varepsilon_1 + \Delta\varepsilon_2}{1-\nu_f} \quad (18)$$

Under the assumption that the Cu thin film is elastically isotropic (this condition is almost met for FCC metals with an anisotropy factor of 1.2),[12] the Poisson's ratio of the Cu thin film was measured to be 0.347 ± 0.029. Comparing the bulk one appeared in the literature (0.343),[13] the measured value are found to be in good agreement with the bulk one. The deviation of 1.17% from the literature value for polycrystalline Cu can be explained by the anisotropy of Cu which has not been taken into account in the calculation and by the texture in the film.[12]

5. CONCLUSION

An original and useful method is introduced for determining Poisson's ratio of thin films on a substrate without sophisticated equipment and additional surface micromachining process. The proposed method is based on the x-ray diffraction strain measurement of three strain components induced by an *in situ* elastic substrate bending, which induces an anisotropic biaxial strain on the thin film. The measurement of the three strain components was carried out using the well-known $\sin^2\psi$ method. The method has the following main advantages: (i) no elastic constant of the substrate or the film is necessary, and (ii) the unstrained lattice parameter of the film needs not to be known, furthermore, (iii) no sophisticated equipment and process for the surface micromachining of the specimen are needed. As an example, the Poisson's ratio of a Cu thin film on a low-carbon steel substrate was determined by using the proposed method. It should be noted that the Poisson's ratio determined by the presented method are intragranular properties.

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