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An analysis technique for extraction of thin film stresses from x-ray data

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We demonstrate a technique for experimentally determining stresses in crystalline thin films without any knowledge of the elastic properties of the thin film material. The results are obtained from interplanar spacing versus \( \sin^2 \Psi \) plots for different stress states. The interplanar spacings are measured by non-symmetric x-ray diffraction. The different stress states are produced by annealing the thin film/substrate samples at elevated temperatures in air and by cooling them in liquid nitrogen. Poisson’s ratio for isotropic films or a similar quantity for anisotropic films can be found through the use of this technique. An extension of this technique also permits the measurement of the coefficient of thermal expansion without removing the film from the substrate. © 1997 American Institute of Physics.

The generalized focusing diffractometer (GFD) is commonly used to evaluate the three-dimensional (3D) ellipsoid of elastic strain in blanket or patterned thin films through measurements of interplanar spacings as a function of \( \sin^2 \Psi \) (where \( \Psi \) is the angle between the surface normal and the normal of the diffracting planes). To calibrate this stress measurement technique, knowledge of the unstrained lattice spacing is required. Unstrained lattice spacings in thin films may differ from values for bulk materials due to thin film anomalies such as variations in stoichiometry, impurities, and interface effects. Thus the literature values for unstrained lattice spacings should not be used for stress calculations. Furthermore, literature values for unstrained lattice spacings for some materials, or for materials which can be grown only as thin films, may not be available. Also, unstrained lattice parameters may not be available at the temperature at which a stress measurement is desired. A common way to resolve this problem involves calculation of the unstrained lattice parameters using known elastic constants for the film. For isotropic materials this is done using Poisson’s ratio, while for anisotropic materials the calculation depends on the elastic compliances. A limitation of this approach is that Poisson’s ratio or the compliances for the chosen material may not be available for the film in question at the chosen test temperature.

In this letter we demonstrate a technique for experimentally determining the unstrained lattice spacing of any crystalline thin film at any temperature using the GFD. Assuming a biaxial stress state for the film, and considering a two-dimensional (2D) section of the 3D ellipsoid of interplanar spacing as shown in Fig. 1, we see how the spacing of crystal planes \( (d) \) varies with direction, as indicated by the angle \( \Psi \). The two ellipses drawn correspond to two different strain states, one corresponding to a biaxial tension stress state and the other corresponding to biaxial compression. The point at which the two ellipses intersect corresponds to a direction in which the lattice spacing is the same for the two different strain states, or for that matter for any strain state. Finding this direction will allow us to determine the spacing of the planes in the unstrained lattice \( (d_0) \). For two different stress states in the same film, the intersection of the plot of the respective \( d \) spacings versus \( \sin^2 \Psi \) indicates the unstrained lattice spacing directly, as seen in Fig. 2. We note that this technique does not require any knowledge of the elastic properties of the film.

In an elastically isotropic material the value of \( \sin^2 \Psi \) corresponding to \( d_0 \) \( (\sin^2 \Psi_0) \) relates to Poisson’s ratio of the material as follows:

\[
\sin^2 \Psi_0 = \frac{2 \nu}{1 + \nu}.
\]

Thus this technique may also be used to measure Poisson’s ratio of any isotropic crystalline thin film material. For an anisotropic material with a (111) fiber texture there exists a relation between the value of \( \sin^2 \Psi \) corresponding to \( d_0 \) and the elastic compliances for that material:

\[
\sin^2 \Psi_0 = \frac{2s_{44} - 4s_{11} - 8s_{12}}{3s_{44}}.
\]
The technique described here allows determination of this particular combination of compliances for such a textured film. Other expressions would be needed for other textures.

Systematic errors which may be present in the described technique will limit the absolute accuracy of the unstrained lattice spacing measurement. This however is not a serious deficiency. In the process of calculating stresses we need to first calculate the strains in the film. The strain is calculated from the difference in lattice spacing and the unstrained lattice spacing:

$$
e = \frac{\Delta d}{d_0}. \quad (3)$$

When calculating $\Delta d$ we lose a possible systematic error in the absolute values of the lattice spacings. We still need to deal with the systematic error in the denominator of eq. (3). If, for example, we could measure $d_0$ with an accuracy of 0.2%, then we could in turn also calculate the stresses with an accuracy of the same magnitude. Whereas an accuracy of 0.2%, then we could in turn also calculate the stresses with an accuracy of the same magnitude. Whereas an accuracy of 0.2%, then we could in turn also calculate the stresses with an accuracy of the same magnitude.

Thin films of Al and Au were deposited onto oxidized (100 nm) Si wafers (orientation [111]) of thickness 470 μm. The Al film was DC sputtered at about 120 °C while the Au film was formed by evaporation. A low pressure chemical vapor deposition (LPCVD) nitride was grown on the oxidized Si wafer to prevent the Au film from forming a silicide with the substrate and a 50 Å thick Ti layer was deposited under the Au film to promote adhesion.

The samples were annealed in flowing air at 400 °C and 500 °C for 1 hour in a wafer curvature system. The temperature was ramped at a rate of 5 °C/min and the curvature was recorded every 10 °C during heating and cooling. Interplanar spacing measurements were made at room temperature by non-symmetric x-ray diffraction before and after ev-ery annealing cycle. Films were subsequently cooled to 77 K by a liquid nitrogen dip for 1 min and warmed in air. The interplanar spacing was again measured at room temperature after the cooling cycle. This treatment cycle was repeated five times for Al to achieve different tensile and compressive stress states and six times for Au to achieve a range of tensile stress states.

Scans performed on the GFD showed that the films were predominantly (111) textured. This texture did not change with thermal cycling for the Al film. For the Au film two additional x-ray peaks stemming from (100) grains appeared during heating. The development of (100) oriented grains is believed due to the smaller strain energy of (100) grains compared to (111) grains. The (111) orientation predominantly survived annealing. This texture did not change from the compliance values given in Ref. 7.

<table>
<thead>
<tr>
<th>TABLE I. Comparison of experimental values to literature values of $\sin^2 \Psi_0$ and $d_0$ for Al and Au.</th>
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<td><strong>Al (422)</strong></td>
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a The cited value of $\sin^2 \Psi_0$ was calculated from the compliance values given in Ref. 7.

b Reference 6.
sample remained (111) textured during the anneal whereas the Au sample became partially (100) textured. The literature value for $\sin^2 \Psi_0$ was calculated for purely (111) textured Au.

The observed deviations in $d_0$ values of 0.121% (Al) and 0.189% (Au) are partially due to a real difference in the unstrained lattice parameter values in thin films and bulk materials, and partially due to systematic errors in the measurement. As explained before, these deviations in $d_0$ will lead to deviations in the stress measurement of less than 0.2%, which is excellent.

The present GFD technique would also permit a measurement of the coefficient of thermal expansion of the film without removing it from the substrate and without any knowledge of the elastic constants of the film material. The unstrained lattice spacing would have to be measured at different temperatures for different stress states created by different thermal histories. Thus we would again look at differences in interplanar spacings. The error analysis described above is equally valid here and thus the expected relative errors in thermal expansion coefficient (due to systematic errors in $d_0$ values) can also be expected to be of the same magnitude as the relative error in $d_0$.

This shows that the technique described, although it is not generally useful for accurately determining lattice spacings of well characterized materials, does provide a straightforward, rapid, and accurate method for measuring stresses and thermal expansion coefficients of “new” and poorly characterized thin film materials.

In summary, we have shown that it is possible to achieve significantly different stress states in crystalline thin films on silicon substrates using appropriate heating and cooling cycles. Measurements of interplanar spacings versus angular direction in these differently stressed films lead to direct measurements of unstrained lattice parameters. The unstrained lattice parameters can then in turn be used to calibrate x-ray stress measurements in films of new or poorly characterized materials. These measurements do not require any knowledge of the elastic properties of the film. Indeed, they permit a measurement of Poisson’s ratio for the case of isotropic films and a similar elastic property for textured films. Finally, an extension of this technique would permit the measurement of the coefficient of thermal expansion without removing the film from the substrate and without any knowledge of the elastic properties of the film material.

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