Study of texture effect on elastic properties of Au thin films by x-ray diffraction and Brillouin light scattering

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Abstract. We have shown a strong texture effect on elastic properties of gold thin films deposited by physical vapour deposition. Elastic properties of non-textured and {111} fiber textured gold thin films were investigated by x-ray diffraction combined with in-situ tensile testing and Brillouin light scattering. These static and dynamic methods allowed characterizing the local and macroscopic elastic behavior of gold films.

1. Introduction
Mechanical properties of thin films have received considerable attention because of their length-scale dependence and its implications on the reliable operation of micro-electronic systems. The small dimensions, interfaces and singular structures are intrinsic to thin films. Thus, in light of ongoing efforts to reduce characteristic feature sizes in such systems, it is imperative to study mechanical properties of materials at small scales [1,2]. Polycrystalline thin films elaborated by sputtering methods show generally textures that may be very strong. Particularly, {111} texture is often encountered in the case of face centered cubic (fcc) materials such as Au. For elastically anisotropic materials, texture induces macroscopic elastic anisotropy which amplitude depends on the degree of texture and on the elastic anisotropy of the crystallites that compose the polycrystalline thin films (local anisotropy). In many cases, macroscopic elastic anisotropy is very elevated and cannot be neglected. As for common fcc materials, <hhh>- and <h00>-type directions are the stiffest and the more compliant, respectively. In the case of gold, Young’s modulus ratio between these two direction types is about 2.8. Indeed, the elastic behavior of polycrystalline gold films strongly depends on grains orientations distribution [3]. In this paper, we show a study of elastic properties of non-textured and {111} fiber textured gold thin films by x-ray diffraction (XRD) combined with tensile testing and Brillouin light scattering (BLS). The interest here is to compare static (XRD) and dynamic (BLS) measurements of elastic properties. We will discuss the results, especially the texture effect on the macroscopic elastic constants.

2. Experimental procedure
2.1 Gold films preparation and microstructure
Gold thin films were produced by physical vapor deposition on Kapton and GaAs substrates. Texture of the as-deposited Au thin films were characterized by XRD analysis. XRD texture characterizations were carried out using a four-circles diffractometer (Seifert XRD 3003) using a Cu X-ray source. The incident beam was focused on the samples mounted on an Eulerian cradle for Ψ tilting, where Ψ is the angle between the measurement direction and the normal of the specimen surface. φ is the rotation...
angle around the normal of the specimen surface. In figure 1, the \{111\} textured gold film (full line) is characterized by sharp peaks observed at $\Psi = 0^\circ$ and at $\Psi = 70.5^\circ$. These are characteristics of a strong \{111\} texture. In contrast, the non-textured gold film (dotted line) is characterized by an intensity showing small variation when $\Psi$ is varied. It should be noted that as expected no significant variations in intensity were found when $\phi$ was changed (rotational symmetry around the normal of the specimen surface, i.e. fiber texture).

Figure 1. $\Psi$-scan obtained for the \{111\} planes family of gold films. The \{111\} textured film (full line) is characterized by sharp peaks observed at $\Psi = 0^\circ$ and at $\Psi = 70.5^\circ$. In contrast, the non-textured films (dotted lines) is characterized by an intensity showing small variation when $\Psi$ is varied.

2.2 X-ray diffraction and in-situ tensile testing
Elastic behavior of supported thin films can be determined by combining uniaxial tensile test to the film/substrate composite and X-ray diffraction. Intragranular strain is determined by peak shifts in XRD measurement, while macroscopic applied stresses in thin films are known thanks to a load cell, which is disposed on a Deben\textsuperscript{TM} micro-tensile machine. The origin of the measured strain, by using this method, is only the known applied stress state [4]. Since thin film diffraction involves only very small volumes, x-ray strain measurements with accuracy of below 0.01% have been performed using a four-circle goniometer at the H10 and DW22 beam lines at the synchrotron radiation facility LURE (Orsay, France).

2.3 Brillouin light scattering
The Brillouin spectra were taken in air, at room temperature, with typical acquisition times of 1-2 hours. The light source was an Ar$^+$ laser on a single mode of the 514.5 nm line. 300 mW of p-polarized light was focused on the surface of the sample and the scattered light was analyzed by means of a Sandercok-type 3+3 pass tandem Fabry-Perot interferometer characterized by a finesse of about 100 and a contrast ratio higher than $10^{10}$. In the present work we used the backscattering interaction geometry, so that the value of the wave vector of the surface acoustic waves probed is fixed experimentally to the value: $Q_z = 2k\sin(\theta)$, where $k$ denotes the optical wave vector in air and $\theta$ the angle of incidence.

3. Results

3.1 XRD and in-situ tensile testing
In this part, we show briefly experimental results obtained for the two film types. In the case of non-textured gold film, the measured strains for \{400\} plane family as a function of $\sin^2 \Psi$ are shown in figure 2. From this graphs, it is possible to characterize macroscopic isotropy (linear relationship between strain and $\sin^2 \Psi$). The measured strains for textured gold film are plotted as a function of $\sin^2 \Psi$ in figure 3. The macroscopic anisotropic elastic behavior of the material is observed on a $\varepsilon - \sin^2 \Psi$...
Ψ plot and characterized by the non-linearity of the curves regardless of the loaded state. This anisotropy is due to strong \{111\} fiber texture. By fitting the experimental data (straight line in figure 2, dotted line and open symbols on figure 3), by using Neerfeld-Hill (NH) model, we determined in-plane Young’s modulus of the two films. It should be noted that NH model is a micro-mechanical model that allow describing realistically the elastic fields in polycrystalline materials [4]. We founded 91.8 GPa for the textured film and 83.0 GPa for the non-textured film, i.e. a discrepancy of 9.6%. This experimental result is in good agreement with calculations taking account of grains orientations distribution of each film (discrepancy of 9.1%) [4].

**Figure 2.** Experimental x-ray strains for \{400\} plane family as a function of \(\sin^2 \Psi\), for the non-textured gold film. Strains were measured for three loaded states (F = 0.7 N, 1.5 N and 2.8 N).

**Figure 3.** Experimental (full lines and closed symbols) strains as a function of \(\sin^2 \Psi\), for the \{111\} fiber-textured gold thin film. Strains were measured for five loaded states (F = 1.1 N, 2.2 N, 3.6 N, 5.6 N and 6.6N).

### 3.2 Brillouin light scattering

**Figure 4.** Brillouin spectrum a) of the gold layer 740 nm thick with \{111\} texture, b) of the isotropic gold layer 120 nm thick on a AsGa(001) substrate. The angle of incidence is \(\theta = 65^\circ\). RW denotes the Rayleigh surface wave, \(S_i\) the \(i^{th}\) Sesawa guided waves. Below we have indicated the calculated power spectrum at the free surface of the displacement components \(<u_z(0)>\) (vertical, associated to the ripple scattering mechanism) and \(<u_x(0)>\) (longitudinal).

For nearly opaque films thicknesses lower or around the acoustic wavelength (0.3 - 0.4 μm), supported by substrates with acoustic phase velocities higher than that of the films (slow film / fast
substrate) such as a gold film on a GaAs substrate, we can only observe using the Brillouin light scattering technique the surface acoustic waves with a sagittal polarization (see figure 4): the Rayleigh wave and the so-called Sesawa guided waves [5], and the corresponding phase velocity can be measured. These modes are dispersive, so that measurements of the acoustic waves frequencies are usually performed for different angles of incidence (i.e. different values of the in-plane wave vector) that enable a determination of the elastic constants.

By taking account only the ripple mechanism for the scattered intensity by the surface acoustic waves, the experimental spectra for the non-textured film (120 nm) is well fitted. In the case of textured film (740 nm, i.e. thicker), the agreement is qualitative. The contribution of the Au/GaAs interface and of the photo-elastic coupling should be considered for a better description of the intensity at higher frequency (more than 8 GHz). The adjustment of surface mode positions leads to elastic constants values (see table 1). The effective elastic constants calculated by NH model, by considering experimental textures, are also reported in table 1. We experimentally and theoretically show discrepancies of effective elastic properties between the non-textured and the \{111\}-textured gold films. Especially, the $C_{44}$ increases drastically (20%) from the textured to the non-textured film. The agreement between experiments and calculations is also good for $C_{13}$ and $C_{33}$. The experimental values for $C_{11}$ are more surprising, but it should be noted that theoretical discrepancy for this constant is very weak (2.5%).

|   | $C_{11}$ (GPa) | $C_{12}$ (GPa) | $C_{13}$ (GPa) | $C_{33}$ (GPa) | $C_{44}$ (GPa) |
|---|----------------|----------------|----------------|----------------|----------------|
| Au\{111\} | | | | | |
| Exp. | 190 | - | 142 | 227 | 18.5 |
| Neerfeld-Hill | 212 | 154.3 | 145.8 | 220.4 | 22.7 |
| Au isotropic | | | | | |
| Exp. | 214 | 168 | 168 | 214 | 23 |
| Neerfeld-Hill | 207.4 | 152.3 | 152.3 | 207.4 | 27.5 |

Table 1. Elastic constants derived from BLS measurements on a \{111\} textured and an isotropic Au film. The NH average is calculated from the experimental orientations distribution function.

4. Summary

We have shown that combining tensile testing with XRD and BLS are very powerful and complementary techniques to study the structural effects on elastic behavior of supported thin films. The diffractive method allows a selective assessment of crystalline phases and crystallographic planes of materials. Thus, this technique is very powerful to study anisotropy of materials (selective study of different diffracting planes) and mechanical properties of multi-phased materials, like multilayers (selective study of each phase). BLS is a very complementary technique to XRD, since it allows calculating elastic constants of the whole material (diffracting and diffracting parts such as interfaces, amorphous phases…). Especially, it can be very powerful to reveal mixing at interfaces in multilayers [6]. Moreover, combining these two techniques will be of great interest to study magneto-elastic properties of thin films.

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