THERMALLY-INDUCED STRESSES IN THIN ALUMINUM LAYERS GROWN ON SILICON

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ABSTRACT

Elevated-temperature X-ray diffraction (XRD) was used to evaluate residual stresses in aluminum thin films on Si (100). The films with a thickness of 2 \(\mu\)m were deposited by magnetron sputtering at different temperatures, and XRD measurements were carried out with the heating stage DHS 900 mounted on a Seifer 3000 PTS diffractometer. The strains were characterised always in temperature cycles from room-temperature up to 450°C with steps of 50 °C. Stress values in weakly textured thin films were calculated using the Hill model applying temperature-dependent X-ray elastic constants of aluminum. The thin films exhibit specific temperature hysteresis of stresses depending on the deposition temperature (being from the range of 50–300°C). The results allow to quantify contributions of intrinsic and extrinsic stresses to the total stress in the layers as well as to evaluate phenomena related to plastic yield. The comparison of the data from thin films deposited at different temperatures indicate a dependence of intrinsic stresses on the substrate temperature during deposition as well as the presence of the plastic yield in films during the cool down after deposition.

INTRODUCTION

Aluminum has been widely applied for the production of interconnects in micro- and optoelectronic devices [1]. During operation of such devices, the aluminum layers are exposed to thermally loads that cause thermally-induced (extrinsic) stresses [2,3]. Those stresses result from the mismatch of thermal expansion coefficients (TECs) of the layer and the substrate and are responsible for most mechanically-induced damages. In the case of Al layers on Si substrates, TEC of aluminum (\(\alpha=23,1 \times 10^6 /K\)) is about ten times higher than that of silicon (\(\alpha=2,6 \times 10^6 /K\))[4,5]. Manufacturing stresses (intrinsic), those develop during the layer deposition, are superimposed on extrinsic stresses and depend on the microstructure development of the layer during deposition. The intrinsic stresses are influenced by the substrate temperature, deposition rate, mobility and energy of incoming atoms, etc [6]. The influence of the deposition conditions and the thermal treatment on the total stresses in aluminum on silicon is still not fully elucidated. Though the thermal stresses can be calculated approximately from TECs [7, 16], the connection between specific deposition conditions on the magnitude of intrinsic stress is still unclear.
Here we present elevated-temperature X-ray diffraction studies of residual stresses in aluminium layers on silicon deposited at different temperatures. The measurements were performed using a newly developed heating-stage for four-circle diffractometers DHS 900 (Domed Hot Stage up to 900°C) [8].

EXPERIMENTAL DETAILS

As a substrate for the specimen preparation, 0.6 mm thick Si(100) wafers with a size of about 5 x 5 mm were used and cleaned using isopropanol and acetone. Thin layers of polycrystalline aluminum were deposited on those native oxide silicon-wafers using magnetron sputter deposition at substrate temperatures of $T_D$: 50, 150, 250 and 300°C (Table 1). Each deposition was done at $4 \times 10^{-3}$ mbar and took about 15 min. The thickness of the layers was about 2 µm.

The elevated temperature characterization of strain/stress in the specimens was performed using a heating attachment DHS 900 mounted on a Seifert 3000 PTS four circle X-ray diffractometer [7]. The used setup featured a 1mm collimator and a scintillation-detector. It was set up in a Bragg-Brentano-geometry with a copper target X-ray tube (Cu Kα), which was operated at 40 kV and 40 mA. A dome was used to homogenise the specimen temperature and to protect the specimen against environmental influences. The melting of the synthetic material of the dome was prevented by an air cooling system.

STRESS EVALUATION

Before performing strain characterization of the samples, Al 111 and 100 pole figures were measured documenting that the layers were isotropic [9]. As a next step, $hkl$ reflections with sufficient intensity were selected (Table 1) [10]. Specimens were measured in a temperature cycle from room temperature up to 450°C in steps of 50°C. The peak positions of measured diffraction profiles at selected tilt angles $\psi$ were determined with a fitting program applying a Voigt function.

For stress calculations, the temperature-dependent X-ray elastic constants, $S_1^{Hill(T)}$ and $S_2^{Hill(T)}$ calculated using Hill model, were deployed. From lattice spacing $d_{hkl}^T$, measured for a specific $hkl$ reflection at the temperature $T$, the isotropic in-plane stress $\sigma^T$ and the unstressed lattice parameter $a^T$ were calculated according to

$$d_{hkl}^T = \frac{a^T}{\sqrt{h^2 + k^2 + l^2}} \left(1 + 2 S_1^{Hill(T)} + 0.5 S_2^{Hill(T)} \sin^2 \psi \right) \sigma^T$$

where $\psi$ is the angle between the normal vectors of the specimen surface and $(hkl)$ crystallographic plane [9,13,15].
EXPERIMENTAL RESULTS

In Figures 1 and 2, diffraction data, obtained from Al 331 reflections (sample 2) during heating and cooling, respectively, are presented. During heating, the \(2\theta\) peak position shifts about \(-0.1^\circ\) per \(50^\circ\)C and on the cooling part it is on the other way around. It can be seen, that the intensity of the diffraction profiles decreases by about 5% per \(\Delta T=50^\circ\)C.

Figure 1. Shift of Al 331 reflections (specimen 2) during the heating up to 450 °C. Note the decreasing peak intensity with the increasing temperature.

Figure 2. Shift of Al 331 reflections (specimen 2) during cooling down to room-temperature. The peak-intensity increases with the decreasing temperature.

Figure 3 represents the relationship between lattice parameter \(a^T\) and the tilt angle \(\psi\) for sample 2. The linear dependencies \(a^T = f(\sin^2 \psi)\) indicate an isotropic (texture-free) feature of the layer, while positive and negative slopes (Figure 3) correspond to the tensile or compressive stress states, respectively, in the layer [9,10,12,14].

The stress-temperature dependencies from specimen 1 to 4 are plotted in Figure 4 including a theoretical elastic behavior in terms of the slope from a stress-triangle. The triangle represents the slope of the Hook’s straight line for unstressed aluminum while ideal elastic deformation, using values of Young’s modulus \(E=72\) GPa and Poisson’s ratio \(v=0.34\) [4, 5, 12]. Stress dependencies for specimen 1 to 4 (Figure 4), exhibit a different behavior. There are always two hysteresis-forms with open and closed behavior correlating with the deposition temperature (Table 1). The layers deposited at temperatures higher than 150°C show always a closed hysteresis. To understand this behavior, repeated measurements were done on specimen 2 that was thermally strained in cycles up to 150°C, 300°C and 450°C (Figure 2).

Figure 3. Temperature dependence of lattice parameter \(a^T\) on the sample tilt angle for specimen 2. (--- cooling part, — heating part, the vertical dotted line represents stress free lattice)
Figure 4. The temperature hysteresis of in-plane-stresses for specimens 1 to 4 with parallel slopes in the elastic area and with open comportment for specimen prepared at lower temperatures.

Figure 5. Repeated thermal cycles up to 150°C, 300°C and 450°C from sample 2 are plotted to visualize the reversibility of the stress behavior.

As it is demonstrated in Figure 5, the hysteresis form closes after a thermally induced straining for temperatures higher than 300°C. It is supposed that the plastic yield in combination with a strain hardening of the layer is most probably responsible for this behaviour [11, 18, 19]. In that way it is justifiable to note that there is a plastic yield during the manufacturing process for samples deposited at temperatures higher than 150°C.

DISCUSSION

The “open” hysteresis of temperature dependence of stresses in samples 1 and 2 indicate the elastic behavior of the film after deposition. This feature allows us to extrapolate magnitudes of intrinsic stresses \( \sigma_i \) in sample 1 and 2 from the temperature dependencies of the total stresses in Al thin films (Table 1). On the other hand, in samples 3 and 4, the yielding occurred already during deposition or cooling down with typical “closed” hysteresis of temperature-stress dependence, and therefore the extrapolation of intrinsic stresses is questionable.

Additionally, the results in Figure 4 and 5 indicate that there exists relatively weak strain hardening in the films even after the yielding occurred – the slopes \( \partial \sigma^T / \partial T \) during the heating procedure are for all four films comparable [17]. The threshold between elastic and plastic behavior in the films can be clearly recognized. It depends decisively on the stress state at room temperature. For specimens with higher tensile stresses at room temperature, a heating to higher temperatures must be applied in order to remove tensile stress component, reach the compressive region and observe yielding.
Table 1. Deposition and measured properties of the samples [10]. The abbreviations stand for: 

$T_D$ – deposition temperature, $\sigma_I$ - intrinsic stress, $\sigma_{\text{max}}$ – max. tension stress, $\sigma_Y$ - yielding stress, 

$T_Y$ - yielding temperature, $d$ – layer thickness

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<th>sample</th>
<th>$T_D$ (°C)</th>
<th>hkl</th>
<th>$\psi$ (degrees)</th>
<th>$\sigma_I$ (MPa)</th>
<th>$\sigma_{\text{max}}$ (MPa)</th>
<th>$\sigma_Y$ (MPa)</th>
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CONCLUSION

X-ray diffraction has been successfully used to evaluate temperature dependencies of residual stresses in aluminum thin films on Si (100) in the temperature range of 25-450 °C. It has been observed that the temperature behavior of stresses depend decisively on the deposition conditions influencing magnitude of intrinsic stresses as well as the occurrence of plastic yield.

ACKNOWLEDGMENTS

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LITERATURE