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Temperature-dependent residual stresses in a hetero-epitaxial thin film system

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ABSTRACT

This paper investigates the temperature dependence of residual stresses in a hetero-epitaxial thin film on a sapphire substrate. The X-ray diffraction technique was employed and a theoretical analysis was also carried out. It was found that the magnitude of compressive residual stresses decrease with increasing the temperature, and that the rate of the change can be well predicted theoretically. It was discovered, however, that the residual stresses vary with the film thickness. For a film of 0.3 μm in thickness at all temperatures, the magnitudes of compressive stresses measured are greater than the theoretically predicted; but for that of 5 μm in thickness, the magnitudes measured become smaller than the theoretical. This leads to the conclusion that the mitigation of lattice mismatch, essentially through interface misfit dislocations, could have varied with the change of the film thickness.

1. Introduction

To increase the capacity of an integrated circuit chip, a semiconductor thin film is often grown on an insulation substrate [1] for exploiting the insulation property and low parasitic capacitance. However, homoepitaxy substrates are either expensive or not available, the hetero-epitaxial growth is often used to simplify device processing or reduce processing cost. The disparate microstructures and thermal properties, however, usually bring about substantial residual stresses in the thin film system, which, when large enough, could result in the film delamination or even wafer cracking [2]. The hetero-epitaxial silicon-on-sapphire (SOS) thin film system is just such a product that has significant mismatches of lattice constant (−5.9% and −14.1% in Si [100] and Si [010] directions, respectively) and coefficient of thermal expansion (CTE) (−0.41% and −0.35% in Si [100] and Si [010] directions, respectively). Some experimental studies [3–6] showed that the silicon film is subjected to compressive stresses ranging from −500 MPa to −1000 MPa, depending on the deposition temperature and film thickness. Considering the high lattice mismatch as shown above, the measured residual stresses are much smaller than those from a simple multiplication of the Young's modulus (about 170 GPa for silicon) and the mismatch strain, resulting in a high residual stress of −10.3 and −23.8 GPa in Si [100] and Si [010] directions, respectively. This indicates a significant stress mitigation mechanism in the thin film. Some transmission electron microscopy (TEM) investigations [7–10] revealed that the interface misfit dislocations are the dominant mechanism for accommodating the lattice mismatch. These dislocations superpose a strong converse stress field to the stress field induced by lattice mismatch. Owing to the discreteness of dislocations, the mitigated residual stresses are often thickness-dependent, which has been unveiled by the stress analysis of the successively etched film [10]. However, the abovementioned stress analysis was performed at a room temperature, at which the measured residual stresses are actually influenced by the CTE mismatch as well. In order to quantify the stresses solely induced by microstructures, a high-temperature stress characterization is necessary.

To this end, this study will carry out post-mortem temperature-dependent X-ray diffraction (XRD) stress analyses with SOS samples of film thicknesses of 0.3 and 5 μm. It is expected that the stresses obtained at the deposition temperature will exhibit the net effect of lattice mismatch and misfit dislocations, and that the stress variation with temperature will show how the effect changes during the cooling–heating cycles. For comparison, three-dimensional finite element (FE) simulations, considering the anisotropy of the material system, will also be conducted to calculate the temperature-dependent residual stresses induced by the CTE mismatch.

2. Experiment

The silicon films of 0.3 and 5 μm thick were epitaxially grown on a 600 μm thick R-sapphire substrate by chemical vapor deposition (CVD) at the temperature of 900 °C. The gaseous source in CVD epitaxial growth was silane (SiH_4), and the reaction was SiH_4 (gas) = Si (solid) + $2H_2$ (gas). The orientation relation of silicon and sapphire is Si [001] || Al_2O_3 [$10\bar{1}2$]. This is because the atoms on sapphire ($10\bar{1}2$)

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plane have the closest symmetry to those on Si (001), such that the epitaxial silicon film can be obtained with minimized growth defects. Prior to deposition, the sapphire wafer was carefully polished to minimize the effects of structural defects of sapphire on the silicon growth.

The specimens of in-plane dimension 15 × 15 mm were cleaved from the wafers and measured in a Philips Panalytical (MRD) diffractometer at the high voltage of 45 kV and current of 40 mA. The stress measurement was performed with a line focused high resolution setting, where the highly collimated and intensive incidence beam of CuKα1 (wavelength λ = 1.5604 Å) was obtained by an X-ray mirror and a 4-bounced germanium crystal, i.e., Ge (220) monochromator, providing a high spectrum resolution of 0.006°. In the diffracted beam path, a flat crystal monochromator was mounted before the detector to control the acceptance angle of the diffracted beam. To obtain the precise diffraction angle 2θ, it is necessary to approach the measuring (hkl) planes to the diffraction maxima. This was done by optimizing the rotational (Φ), inclinational (Ψ), and the incident (ω) angles by consecutive scanning around the diffraction position in the Eulerian Cradle. The peak profile was acquired at a step size of 0.015° and a rate of 2 s per step. The raw diffraction data was smoothed and fitted by the Gaussian function, from which the diffraction angle 2θ at the peak was determined by solving the second derivative of the Gaussian curve.

A heating stage (DHS 1100 system) was used to heat the specimen to a temperature ranging from the room temperature to 900 °C. The heating system consisted of an aluminum nitride sample holder with good temperature conductivity and a heater/thermocouple system underneath the sample stage allowing a precise control of heating conditions. The specimen was covered by a graphite dome, which is almost transparent to X-ray and minimizes temperature fluctuation due to radiation and convection.

After the room temperature characterization, the residual stresses were investigated at elevated temperatures of 325 °C, 500 °C, 700 °C, and 900 °C respectively. When a temperature step was reached, a holding time of 5 min was applied at the temperature to make sure that the temperature fluctuation had been minimized before the new measurement.

The microstructures of the SOS samples were inspected by the TEM, using the cross-sectional specimens along silicon [110] prepared by a Nova 200 Nanolab focused ion beam system, using the Ga ion beam at 30 kV and 0.63 nA. The TEM specimen was tilted around silicon <110> zone axis by a double tilt holder in a Philips CM-200 TEM (200 keV) to highlight the planar defects and dislocations. The topologies of the films were examined by the in-situ Scanning Probe Microscopy (SPM) imaging in the Hyistron TI-950 TriboIndenter. A Berkovitch indentor with tip radius of 150 nm was utilized in the scanning. The probe force applied was 2 μN.

3. XRD analysis of temperature-dependent stresses

Although the least square algorithm has been well established to solve for the complete stress tensor of a single-crystalline thin film [11,12], the accuracy is influenced by the stress-free lattice constant a_0. In theory, a_0 changes with temperature owing to the thermal expansion of the lattice. However, for a film-on-substrate system, both the film thickness [13,14] and microstructural defects [14] can lead to the uncertainty in quantifying a_0 when using the XRD. In the present study, therefore, a_0 was treated as an unknown and the multiple regression method was employed for resolving both a_0 and the residual stress tensor [15,16] simultaneously. This method led to more accurate results than those directly using a_0 from the bulk single crystal.

Fig. 1 schematically illustrates the coordinate relation in the XRD stress analysis. The directions of the normal stresses σ_{11}, σ_{22}, σ_{33} coincide with silicon crystallographic directions [100], [010] and [001], respectively. d_{hkl} (or $d_{\Phi\Psi}$) is the lattice spacing measured along the silicon [hkl] direction, where Φ and Ψ are the azimuth and inclinational angles relative to the silicon [100] and [001] directions. The previous study of the authors has shown that in the SOS system, the three in-plane film stresses (i.e., σ_{11}, σ_{12} and σ_{22}) are much larger than the other stress components (i.e., σ_{31}, σ_{32} and σ_{33}) [12], which indicates that the stress state can be simplified to a plane-stress state. With sufficient numbers of d_{hkl}, the intercept a_0 and variables σ_{11}, σ_{22}, and σ_{33} could be solved using the least-square method from the linear equation set of

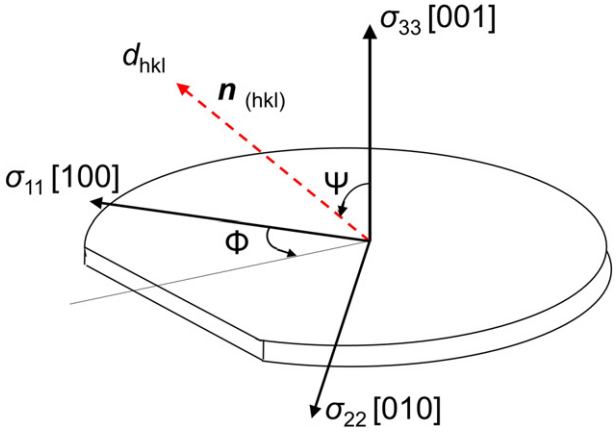

Fig. 1. Coordinate in the XRD stress analysis.

$$D_{hkl}^{\xi} = a_0 + \sum_{i,j=1,i\le j}^{2} B_{ij}^{\xi}(\Phi,\Psi,S_{mn})a_0\sigma_{ij}, \tag{1}$$

where ξ refers to the individual measuring direction pertaining different crystallographic orientations; $D_{hkl}^{\xi} = d_{hkl}^{\xi}\sqrt{h^2+k^2+l^2}$ are the experiment results; and $B_{ij}^{\xi}(\Phi, \Psi, S_{mn})$ are the regression coefficients determined by the diffraction directions (Φ and Ψ) and the elastic compliance tensor of silicon S_{mn}. The detailed derivation of Eq. (1) is given in Appendix A. In the measurement, nine crystallographic orientations, i.e., [004], [224], [115], [404], [315], [206], [335], [353], and [444] were identified and listed in Table 1. They give rise to high diffraction angles $2\theta_{hkl}$ and thus less errors in the strain measurement [17]. The maximized multiplicity of Φ and Ψ could make the solution of σ_{ij} more reliable by avoiding the multi-collinearity [18] in a multiple-regression analysis. In the high temperature stress analysis, the temperature-dependence of the compliance tensor of silicon were considered, which were calculated from the equations given in [19] as listed in Appendix B.

4. Results and discussion

Fig. 2 is the plot of the diffraction peaks measured at various temperatures from the silicon (335) diffraction planes. As can be seen from the plot, the diffraction angles $2\theta_{[335]}$ was left shifted from the room

Table 1
The stress-free diffraction angles $2\theta_0$, the azimuth (Φ), and inclinational (ψ) angles for individual (hkl) diffraction planes.

(hkl)	$2\theta_0$	Φ (°)	ψ (°)
004	69.126	0	0
206	127.534	0	18.44
404	106.702	0	45
315	114.084	18.44	32.31
115	94.947	45	15.79
224	88.025	45	35.26
335	136.880	45	40.32
444	158.604	45	54.736
353	136.880	59.04	62.77