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Stress-free deposition of [001] preferentially oriented titanium thin film by Kaufman ion-beam source

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ABSTRACT

We proposed a method to control and minimize residual stress in [001] preferentially oriented Ti thin films deposited by a Kaufman ion-beam source using a substrate temperature during deposition (T) as the parameter. We determined the residual stress, corresponding lattice parameters, and thickness of deposited films using X-ray diffraction and X-ray reflectivity measurements. We showed that the Ti film deposited at $T \approx 273$ °C was stress-free with corresponding lattice parameters a_0 and c_0 of (2.954 ± 0.003) Å and (4.695 ± 0.001) Å, respectively. The stress-free sample has the superior crystallographic quality and pure [001] orientation. The Ti thin films were oriented with the c-axis parallel to the surface normal. We also investigated root mean square of surface roughness of deposited films by atomic force microscopy and it was in the range from ≈0.58 nm to ≈0.71 nm. Such smooth and stress-free layers are suitable for microelectromechanical systems.

1. Introduction

Ti is a commonly used material in planar technologies either for the fabrication of integrated circuits, microelectromechanical systems (MEMS) or microfluidic systems. Ti has a reasonably good electrical conductivity, an excellent thermal stability, high hardness, high melting point, high elasticity and a very low concentration of crystallographic imperfections [1]. Ti belongs to a group of biocompatible materials and is also compatible with the complementary-metal-oxide-semiconductor (CMOS) process; thus, it is capable of being used in fabrication lines dedicated to CMOS production and research [2]. Ti thin films are used in MEMS applications such as cochlear implants [3], infrared bolometers [4–6], flexible and wearable heartbeat sensors [7], piezoelectric energy harvesters [8], microfluidic devices [9], and piezoelectric resonators [10]. Layers used in MEMS technologies are typically required to have minimal residual stress as this significantly affects the mechanical [11] and electrical [12] properties of a final device. Once the stress level exceeds a certain limit it can even cause damage to the structural integrity of the device [13].

Ti thin films are usually deposited by processes such as physical vapor deposition, including thermal and electron beam evaporation, pulsed laser deposition, or sputtering, either magnetron or ion-beam based. Each technique results in the formation of layers with different properties, such as crystallographic parameters, roughness, residual stress, electrical sheet resistance, and thermal coefficient of resistance [14]. Control of the Ti crystallographic orientation is of crucial significance as it directly affects its properties and, together with surface roughness, it is important for properties of layers subsequently deposited on top of the Ti layer, such as AlN piezoelectric material [10,15,16]. The magnetron sputtering technique allows residual stress control by modulating Ar gas pressure, magnetron power, or substrate deposition temperature (T) [17]. Unfortunately, these parameters cannot be set independently from each other. The other extended technique employing the assisted ion-beam source for concurrent substrate ion-beam bombardment during the layer growth allows only limited control of residual stress [18].

Here, we report on the preparation of stress-free highly [001] oriented Ti thin films deposited using a 3-grid radio frequency-inductively coupled plasma (RFICP) Kaufman ion-beam source. The major advantage of this ion-beam source type is a well-defined process control that allows us, contrary to conventional deposition techniques, to set parameters like ion-beam energy and atomic sputtering direction independently, and also in one order magnitude lower operational pressure compared with magnetron sputtering [1]. On the other hand, its disadvantage is a

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rather more complex control of residual stress. In our work, we concentrated on the investigation of the control of residual stress by *T* and its influence on crystallography and surface roughness of a Ti layer deposited on a plasma enhanced chemical vapor deposited (PECVD) SiO_2 layer.

2. Experimental details

2.1. Titanium deposition process

For all experiments, we used p-type Si wafers with diameter of ≈100 mm, crystallographic orientation of [100], thickness of ≈375 μm, and specific resistivity in the range from ≈6 Ω cm to ≈12 Ω cm covered with PECVD SiO_2 with a thickness of ≈200 nm. These wafers were diced into individual substrates with dimensions of ≈20 mm × ≈20 mm.

The Ti layers were deposited by an ion-beam sputtering system inside a vacuum chamber with the RFICP Kaufman ion-beam source (Kaufman & Robinson—KRI®) with a ≈ 4 cm diameter Mo 3-grid dished focused ion optics with an ≈45° ellipse pattern. We used an Ar ion-beam with purity of 99.99999% to provide the bombardment of an ≈100 mm × ≈100 mm Ti target with the purity of 99.995% under an incidence angle of ≈45°. Reduction of the ion-beam space charge was provided by a KRI LFN 2000 charge neutralizer (KRI®). The vacuum chamber was evacuated to the pressure of $\approx 5 \cdot 10^{-7}$ Pa before each deposition process. The final Ti thickness (t_f) of (80 ± 1) nm was monitored in-situ by quartz crystal microbalance method and subsequently verified by X-ray reflectivity (XRR) measurement.

The deposition parameters were selected under conditions determined in our previously reported work [1]. We set the ion-beam energy to ≈200 eV, acceleration voltage to ≈−220 V to control the extraction and optical parameters of the ion-beam, ion-beam current to ≈5 mA at the target, radio frequency power to ≈70 W supplied to the plasma discharge, Ar flow to ≈2.2 sccm, and *T* was used as the parameter. We performed a contact temperature measurement using a type K thermocouple during the sputter deposition process to investigate the *T* caused by the combination of deposited material energy and the built-in substrate heater. The lowest value of *T* was ≈105 °C which is generated only by the deposited material flux with no additional heating. We controlled the *T* in the range from ≈105 °C to ≈295 °C with an accuracy of ±3 °C. The pressure during the all depositions was constant with the value of $\approx 9 \cdot 10^{-3}$ Pa.

2.2. X-ray scattering methods

We characterized the Ti layers by several X-ray diffraction (XRD) methods (Fig. 1). We used a Rigaku SmartLab system with a Cu X-ray tube.

We used the standard Bragg-Brentano setup (Fig. 1A) to determine the lattice parameter (*c*) of the planes parallel to the surface. The lattice parameter (*a*) in the perpendicular direction was measured in a grazing-incidence setup (Fig. 1B) using a parabolic multilayer mirror as a monochromator and parallel plate collimator with a divergence of ≈0.15° and ≈0.11° in the incident and the scattered beam, respectively. We performed this measurement at an angle of incidence (α_i) of 0.3°, which is close to the critical angle of total external reflection of 0.298°. The diffracted signal was integrated over the exit angle (α_f) from 0° up to 2°. We extracted values of lattice parameters by curve fitting using Rigaku PDXL2 software.

We measured the t_f in the XRR configuration using a two bounce Ge (220) channel cut monochromator.

We determined the preferential orientation of the Ti lattice via the pole figures measurement. We performed the measurement with the parallel beam and the parallel plate collimator as in the grazing incidence setup (Fig. 1B). The diffractometer with an in-plane arm allowed to map the full half space above the sample's surface. The background of pole figure was measured 2° off the peak position.

We measured the curvature (Fig. 1C) of the Si substrate before (R_0) and after (*R*) Ti deposition to determine the elastic stress of Ti layers. The curvature radius was determined from the dependence of Bragg angle (θ) on the substrate position. Its slope is inversely proportional to the curvature radius of crystallographic planes. The stress of a Ti layer (σ_f) can be calculated by the Stoney formula (Eq. (1)) for [001] oriented Si [19]:

$$\sigma_f t_f = \frac{h^2}{6} \cdot \frac{1}{s_{11} + s_{12}} \cdot \left(\frac{1}{R} - \frac{1}{R_0} \right) \quad (1)$$

where *h* is the substrate thickness and s_{11} and s_{12} are the components of the Si elastic compliance tensor. We used a four bounce Ge (220) Bartels-type monochromator and measured the position of Si 004 diffraction peak dependence on the sample position.

2.3. Surface roughness measurement method

We determined the root mean square of surface roughness (R_{RMS}) from surface scanning by atomic force microscope (AFM) Dimension Icon by Bruker in the ScanAsyst®-Air mode. We used the ScanAsyst-Air probe with a cantilever spring constant of ≈0.4 $N \cdot m^{-1}$ and tip radius of ≈2 nm.

3. Results and discussion

3.1. Crystallography and stress characterization

We performed phase analysis measurement using XRD in the Bragg-Brentano setup with the 2θ angle in the range from 30° to 85° for all layers with *T* as the parameter. The interval from 60° to 78° was excluded

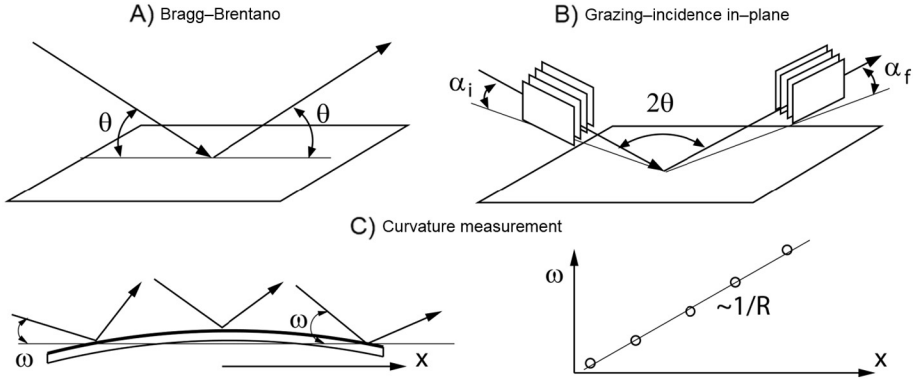

Fig. 1. Setup of the XRD experimental methods: A) Bragg-Brentano setup to measure the out-of-plane *c* lattice parameter; B) In-plane diffraction for *a* lattice parameter determination was measured with the parallel plate collimator and analyzer shown in the figure; C) Curvature measurement determines average σ_f in the layer. The angle of incidence with respect to the mean surface is noted as ω.