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Stress and strain in titanium nitride thin films

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ABSTRACT

Titanium nitride (TiN) films, with thickness ranging from 0.02 μm to 1.9 μm, were grown by reactive unbalanced magnetron sputter deposition on silicon substrates. The average film stress is highly compressive in thin films and less compressive in thicker films.

Two films, with thicknesses of 0.53 μm and 1.63 μm, were subjected to detailed X-ray diffraction (XRD) analysis. $\sin^2\Psi$ analysis was performed, both on films attached to the substrate, as well as on free-standing flakes of the film. The flakes were obtained by dissolving the substrate. $\sin^2\Psi$ analysis, both on the films attached to the substrate as well as on the flakes, did not yield straight lines. By combining the $\sin^2\Psi$ measurements on films attached to the substrate with the $\sin^2\Psi$ measurements on the flakes we were able to distinguish between a residual deformation of the lattice and the deformation due to the biaxial stress. Following this procedure the stress obtained from wafer curvature and from XRD strain measurements coincides.

A residual strain parallel to the growth direction of the crystallites with the <111> direction parallel to the growth direction combined with a changeover in film texture from <001> parallel to growth direction to <111> parallel to growth direction leads us to propose a model explaining the dependence of stress on film thickness in TiN thin films.

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1. Introduction

Polycrystalline titanium nitride (TiN) thin films are widely-used as wear protective coatings or as diffusion barrier layers in IC technology. Grown by Physical Vapor Deposition (PVD) on a rigid substrate, these films are usually in a stressed state, considerably influencing the film's performance. A too high level of stress can lead to cracking of the film in the case of tensile stress and to buckling in the case of compressive stress. Two different techniques are commonly used to measure the stress in the film: a) measuring the stress-induced strain of the film's lattice parameter by X-ray diffraction (XRD) and b) deducing the film stress from measuring the elastic deformation of the substrate.

With both techniques it has been observed that for thinner films the average biaxial film stress is more compressive [1,2]. With increasing thickness the film stress decreases, yet, remains compressive. In earlier work we have shown that this decrease of average film stress is due to a stress gradient in the film [3]. We performed detailed XRD analysis on two films of different thickness and different stress levels. Plots of the lattice strains obtained from the 111, 200 and 311 reflections vs. $\sin^2\Psi$ yielded no straight lines. Various authors have dealt with the non-linearity of $\sin^2\Psi$ plots [4–6]. The non-linearity is caused by the direction dependent elastic properties of the grains, a grain orientation distribution and the requirement of continuity of either in-plane stress or in-plane strain over grain boundaries. However, in our case we think that there is still another factor responsible for the non-linearity of the measured $\sin^2\Psi$ plots. Based on the changeover in texture from 001 to 111, accompanied by a decrease in biaxial compressive film stress with increasing thickness, we argue that the ion bombardment during film growth generates more compressive stress in the crystallites with their 001 crystal direction parallel to growth direction than in the crystallites with their 111 crystal direction parallel to growth direction. TiN has a titanium face centered cubic crystal structure with nitrogen on the octahedral positions. Therefore crystallites with their 001 crystal direction parallel to the growth direction are more open in growth direction than crystallites with their 111 direction parallel to the growth direction and hence more susceptible to ion-peening. A higher stress generation in the crystals with their 001 direction parallel to growth direction will lead to a residual deformation of the other grains, as we do observe.

In order to obtain the stress in the film from the XRD data, we need to distinguish between elastic deformation and residual deformation. Therefore we also analyzed film flakes removed from the substrate. With no constraint imposed on the film flakes by the substrate, we can measure the residual, Ψ-dependent strain of the diffracting planes, without the macroscopic biaxial stress. Subtracting the two sets of $\sin^2\Psi$ measurements will yield the effect of the biaxial stress on the lattice deformation.

2. Experimental procedures

2.1. Film growth

The examined TiN films were grown by reactive unbalanced magnetron sputter deposition in an industrial PVD system (Hauzer HC

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Table 1
Film thickness and average residual stress from wafer curvature measurements of the two analyzed TiN films.

Film	Film thickness (μm)	Average residual stress measured (wafer curvature) (GPa)
A	0.53	−1.4
B	1.63	−1.0

750) at a temperature of 480±20 °C. The homologous temperature ($T_{deposition}/T_{melting}$) is 0.24. The monocrystalline silicon substrates (100 mm wafers with a 001 crystal orientation, 525 μm thick) performed a planetary motion in front of a 600×120 mm titanium target; the nitrogen and argon flows during deposition were 37 sccm and 115 sccm, respectively, yielding a deposition pressure of 0.4 Pa. The growing film was ion bombarded by applying a substrate bias voltage of −125 V; the target power was 5 kW; the target voltage was 600 V, resulting in a deposition rate of 4.5 nm/s. The film thickness was controlled by adjusting the deposition time. We obtained a series of films in the thickness range of 0.02 μm to 1.9 μm. Two of those films were extensively analyzed. Details of films A and B are given in Table 1.

2.2. Stress measurement

From wafer curvature measurements before and after deposition, which were performed with a two-laser-beam setup, the average stress in the films was calculated according to the Stoney equation [7]. For all films the thickness was calculated from the film weight and the TiN density (5.21 g/mm^3). The accuracy of the weight measurements was 0.1 mg, which corresponds to 12% error for a 0.02 μm film and 0.1% for a 1.9 μm film. With scanning electron microscopy we measured films with the thickness of 0.02 μm, 0.07 μm, 0.3 μm; a 1.9 μm film was measured by transmission electron microscopy. The thickness from microscopy agreed with the thickness from initial weight measurement within 5% [3]. The error of the calculated stress lies within 7% of the measured values for the films A and B.

2.3. Film detachment

By dissolving the silicon substrate, the substrate imposed constraint on the film was removed. The selective silicon etching was done in an 80 °C solution of purified water (500 ml) and isopropanol (125 ml), containing 100 g diluted potassium hydroxide pellets. Precautions were taken to avoid the detached thin film to interfere with the stirring device. The silicon was completely removed after 24 h. The remaining film flakes were carefully rinsed with water and left to dry at 40 °C. In Fig. 1 we show the remaining flakes of film B, fixed with laboratory grease to a <510> oriented silicon wafer. From here on we will refer to flakes when describing measurements on these film remains. Notably, the flakes remain bent to accommodate the intrinsic stress gradient [3]. Further, due to the handling, some flakes lie in such a way on the substrate that the former film-substrate is facing upwards.

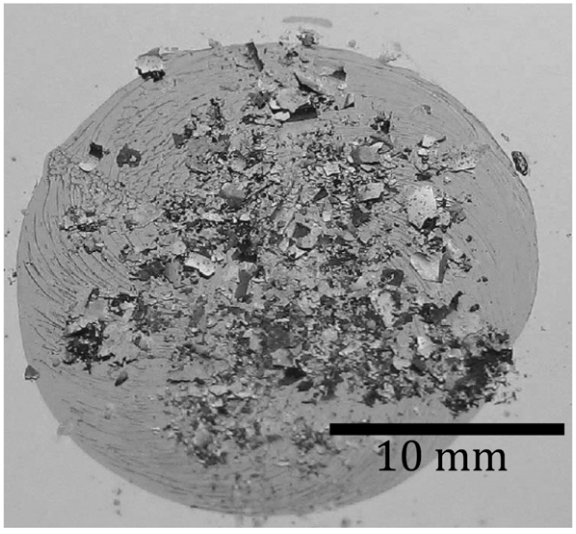

Fig. 1. In order to be able to measure the lattice parameter of the TiN films without the constraint imposed on the film by the rigid substrate, the film was detached by selectively dissolving the silicon substrate. The figure shows the remaining film B, broken into flakes. For XRD analysis the flakes were fixed with laboratory grease onto a <510> oriented silicon wafer.

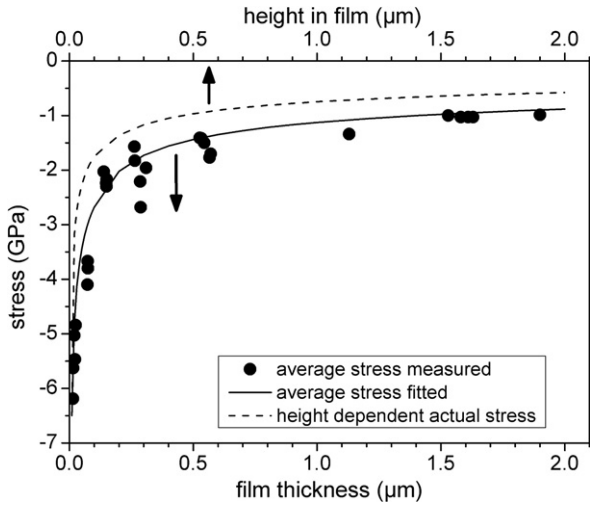

Fig. 2. From wafer curvature measurements we obtain a high compressive average residual stress for thinner films, which decreases with increasing film thickness (dots). The average residual stress is fitted with a power law (bottom axis). The dotted line indicates the derived actual stress in the film as a function of height in the film (top axis).

2.4. XRD measurements

The X-ray stress analysis (XSA) on the films and flakes was performed with the $\sin^2\Psi$ method [4,6] on a Bruker D8 diffractometer with a Vantec position sensitive detector, using the CoKα radiation. Ψ, the specimen tilt angle, describes the angle between the specimen surface normal and the diffraction plane. The data were corrected for background radiation and the CoKα$_2$ component. We examined the 111, 200 and 311 reflections; the peak position was found by a parabolic peak fit above 50% peak intensity. The instrumental errors were determined by measurements on a standard sample of LaB_6. From this we find the absolute accuracy to be +/−0.0001 nm. Due to the circumstance that the flakes do not all face with the same surface upwards, as mentioned in Section 2.3, we can neglect the effect of X-ray absorption and as the flakes are free-standing, the measured lattice parameter represents the crystals' lattice parameter free from external constraint. The Θ–2Θ scans were done in the same system with CoKα radiation. In order to decrease the intensity of the substrate reflections, an ω offset of 2° was applied. On film A a scan was also performed using CuKα radiation.

3. Results

In Fig. 2 we plot with dots the average stress of a number of films vs. the films' thickness (t). For thin films of 0.02 μm we measured up to −6 GPa compressive stress, which decreases rapidly for thicker films. For a 1.9 μm thick film we measured a compressive stress of −1 GPa. Based on the model of Dammers and Radelaar describing the grain growth in polycrystalline films with a power law of the film thickness [8], Janssen et al. were able to describe the stress–film thickness relationship in chromium thin films with a power law of the film

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