The Structural and Mechanical Properties of Ti Films Fabricated by Using RF Magnetron Sputtering


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Titanium films were deposited on various substrates, such as pure Si(100), oxide/Si(100), nitride/Si(100), TiO$_x$N$_y$/Si(100), PI(polyimide) and glass, by using r.f. magnetron sputtering. X-ray diffraction (XRD) data showed a distinct change in major film growth direction with the substrate. In the case of Ti film growth on pure Si(100), oxide/Si(100) and nitride/Si(100) surfaces, the film was strongly grown in the (100) direction while Ti films with growth preferentially in the (002) direction were observed on TiO$_x$N$_y$/Si(100), PI and glass surfaces. The surface morphologies were observed using atomic force microscopy (AFM). The mechanical properties were observed by using a nano-indenter.

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I. INTRODUCTION

Titanium, as well as its alloys, has good mechanical properties, like a very high specific strength and remarkable corrosion resistance. Therefore, they have been widely used in several industries, such as aerospace engineering, motor science, chemical engineering sectors and have been applied in medical science as human joint replacements and dental implants [1–4]. They have also been used as functional thin film layers, like diffusion barriers, buffer layers and interlayers, etc., in semiconductor devices [5,6]. Even though pure titanium is a highly reactive metal, it is normally protected by an oxide film consisting mainly of TiO$_2$ and it is very stable. It shows several characteristics like excellent corrosion resistance even in many aqueous media and excellent biocompatibility [2].

The basic film properties are well known to depend mainly on its microstructure. Even though the growth and nucleation of Ti thin films has been extensively studied, the roles of the substrate’s properties, like the surface energy, electric conductivity and substrate structure, are not yet fully understood. Therefore, we investigated the particulars of the growth of Ti thin films deposited on varied substrates (pure Si(100), oxide/Si(100), nitride/Si(100), TiO$_x$N$_y$/Si(100) [7], PI (polyimide), glass) by r.f. magnetron sputtering. The main goal of our experiment was to clarify the growth behavior of Ti thin films on various substrates, which is influenced by the conditions of the substrates and by the distance between the target and the substrate.

II. EXPERIMENT

The Ti thin films were deposited on varied substrates by using a r.f. magnetron sputtering system. Pure Si(100), oxide/Si(100), nitride/Si(100), TiO$_2$N$_y$/Si(100), PI (polyimide) and glass were used as substrates. They were cleaned by using an acetone, alcohol, HF (48% for Si wafer) solution, DI (distilled) water and nitrogen gas for drying. A round-type, 2-inch sputtering target was designed for high-purity Ti (99.99 %). The sputtering system was evacuated to 5 × 10$^{-6}$ Torr by using a turbo molecular pump. The working pressure was fixed at 1 × 10$^{-2}$ Torr and it was controlled mainly by using high-purity Ti (99.999 %) gas. Ti thin films were deposited at room temperature (RT) with an r.f. power of 200 W after cleaning the target with an Ar plasma for 5 min. The argon flow rate was uniformly 100 sccm.

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We mainly studied the variation of film growth behavior with the distance between the target and the substrate ($D_{ts}$). The crystallinity of the Ti thin films was investigated by using X-ray diffraction (XRD). The surface morphologies were investigated by using atomic force microscopy (AFM) and we investigated the mechanical properties by using a nano-indenter for films with identical thicknesses.

### III. RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction pattern for each type of Ti thin film. From Figure 1(a), we see three typical peaks attributed to (100), (002) and (101) diffractions. Since Ti has a cubic or a hexagonal structure [8], our XRD data revealed that all of the films are polycrystalline with a hexagonal structure. In addition, Figure 1(a) clearly shows different growth behaviors as various substrates. It is noticed that in the case of pure Si(100), oxide/Si(100) and nitride/Si(100), the Ti films are mainly grown in the (100) direction while Ti on glass and PI substrates exhibited a (002) orientation, indicating a substrate dependence. As Figure 1(a) shows, Ti thin films seem to be influenced by the surface energy and the crystallinity of substrates Similar results for the substrate effect have already reported for the growth of CrN thin films [9]. However, to now, there have been no reports on how the distance between the target and the substrate ($D_{ts}$) affect the growth direction.

To clarify this, we deposited Ti films on pure Si(100) substrates at different $D_{ts}$ conditions. Figure 1(b) shows the diffraction patterns obtained from Ti films that deposited on pure Si(100) with various value of $D_{ts}$. These data show a change in the major film growth direction with various $D_{ts}$. The Ti thin films exhibit a highly preferred (002) direction with increasing $D_{ts}$. The XRD data also show that even though the film growth rate decreases with increasing $D_{ts}$, the FWHM values increases, which suggest relatively good crystallinity. Figures 2(a) and 2(b) show the diffraction patterns obtained from Ti films deposited on nitride/Si(100) or PI substrates, respectively. With long $D_{ts}$, the main Ti film growth direction is (002) for both substrates. However, with de-
creasing $D_{ts}$, a strong preference for a (002) preferred growth direction is observed on the PI substrate. Figure 2(b) shows a strong (002) diffraction peak with two small peaks, (100) and (101), at 30 mm distance, indicating relatively good crystallinity. However, in the case of nitride/Si(100), the main growth direction has gradually changed from (002) to (100) with decreasing $D_{ts}$. This means that the crystallinity of the substrate itself can influence the film’s crystallinity, as well as the film’s growth direction. Also, the materials with low surface energy, such as polymers, prefer the (002) orientation while materials with high surface energy, such as silicon or nitride substrates, prefer the (100) direction. In our case, moreover, the effect of $D_{ts}$ is less for a PI substrate than for a nitride substrate. $D_{ts}$, together with surface energy, will mainly affect the film’s growth direction and crystallinity.

Figure 3 shows AFM images obtained from Ti thin films deposited on various substrates at room temperature with a $D_{ts}$ of 70 nm. From Figure 3, we realize one typical thing; the RMS roughnesses of the Ti thin films grown on pure Si(100), oxide/Si(100) and nitride/Si(100) substrates are relatively small (at least three times lower) compared with the values for Ti thin films grown on glass, PI and TiO$_2$N$_x$/Si(100) substrates, reflecting the substrate effect. This is why we obtained two different growth directions. When the film has a smooth surface, it prefer the (100) orientation while a film with a rough surface prefers the (002) orientation. Figure 4 shows AFM images obtained from the Ti thin films grown with various $D_{ts}$. From these figures, we realize that the $D_{ts}$ affects the surface roughness. Consequently, the RMS roughness value increases as a function of the $D_{ts}$. By comparing the Figures 1 and 2 with Figure 3, we conclude that the Ti thin films prefer the (100) direction for value of $D_{ts}$ below 50 nm and the (002) direction for value of $D_{ts}$ above 50 nm.
The hardness was observed using a nano-indenter. The data indicate only for the Ti thin film not including the substrate. Figure 5(a) shows that the hardness value of Ti on a Si substrate was 6.8 GPa and that of Ti on a glass substrate was 5 GPa. Figure 5(b) also shows the hardness of Ti films with various $D_{ts}$. The hardness value at a $D_{ts}$ of 30 nm is higher than that at a $D_{ts}$ of 70 nm, which means that mechanical property of Ti thin films does not correlate with crystallinity. Rather, a typical orientation, such as the (100) plane, will influence the hardness, unlike (002) or other planes.

IV. CONCLUSIONS

Ti thin films are grown with a (100) preferred direction on Si(100) and oxide/Si(100) substrates. Ti thin films with a (002) oriented direction were synthesized on glass, PI and TiO$_x$N$_y$/Si(100) substrates. Therefore the grown characteristics of Ti thin films are influenced by the surface energy and the crystallinity of the substrates. Also, Ti thin films exhibit a highly preferred (002) direction with increasing $D_{ts}$. In addition, the RMS roughness value changes with $D_{ts}$ and with the substrate type. If a Ti film has smooth surface, it prefers a (100) orientation. Also, the hardness of Ti thin films with a (100) direction is higher than that of Ti thin films with a (002) direction.

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