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Hysteresis effect and film characterization in DC reactive sputtering of titania and alumina

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Abstract – In preparation to the fabrication of TiO_2/Al_2O_3 nanolaminates, titania and alumina thin films were deposited on Si single crystals using DC reactive sputtering. Optimal reactive gas flow was determined from a hysteresis loop obtained with simultaneous operation of Ti and Al targets. Film stoichiometry, deposition rate, phase composition and nanohardness were determined. Results indicate that thermal treatment might be necessary to achieve ultrahardness in nanolaminates fabricated under the conditions studied .

Based on the model of restricted dislocation movement [1], TiO_2/Al_2O_3 nanolaminates are potential candidates for ultrahard coatings of superior thermal and chemical stability in the presence of O_2. In preparation to the fabrication of TiO_2/Al_2O_3 nanolaminates, we deposited and characterized titania and alumina thin films on Si single crystals.

Deposition took place using DC reactive sputtering, which compared to RF processing offers higher deposition rates and thus is more suitable for industrial applications. DC reactive deposition of metal oxides, however, is unstable as a result of target poisoning [2]. To prevent poisoning without compromising oxide stoichiometry we performed real-time monitoring of the oxygen partial pressure as a function of flow rate. Substrate temperature varied from room temperature to 450°C.

Figure 1 shows the hysteresis loop corresponding to the simultaneous operation of the Ti and Al targets. The observed loop is intermediate between those observed for Ti and Al single target operation. Based on the data, an optimal reactive gas flow rate of 2.7 sccm O_2 was determined. Total pressure was 4 mTorr.

Titania stoichiometry and deposition rate were investigated using Rutherford backscattering spectrometry. Results show that TiO_2 samples are free of contaminants; data analysis reveals an O/Ti ratio of 2:1. Alumina film quality was investigated using spectroscopic ellipsometry, which indicated minor absorption and a refractive index of 1.5 to 1.7 at 0.6 µm. Deposition rates were 2.4 and 0.9 nm min^{-1} for titania and alumina, respectively. Phase composition was determined using X-ray diffraction (Figure 2). Titania films are crystalline with balance between rutile and anatase depending on deposition temperature, while alumina films are amorphous, with metallic aluminum in the structure. Mechanical indentation was used to determine film hardness, which varied from 9.5 to 11 GPa for titania and from 8 to 18 GPa for alumina, also depending on deposition temperature. These results indicate that additional thermal treatment might be necessary to achieve ultrahardness in nanolaminates fabricated under our deposition conditions.

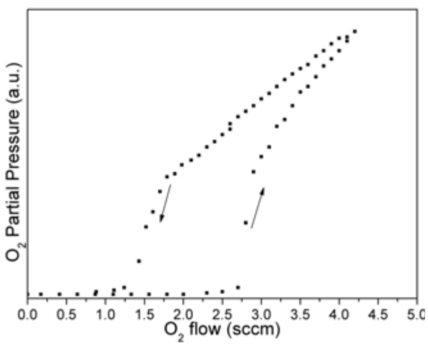

Figure 1: Hysteresis loop for simultaneous operation of Ti and Al targets.

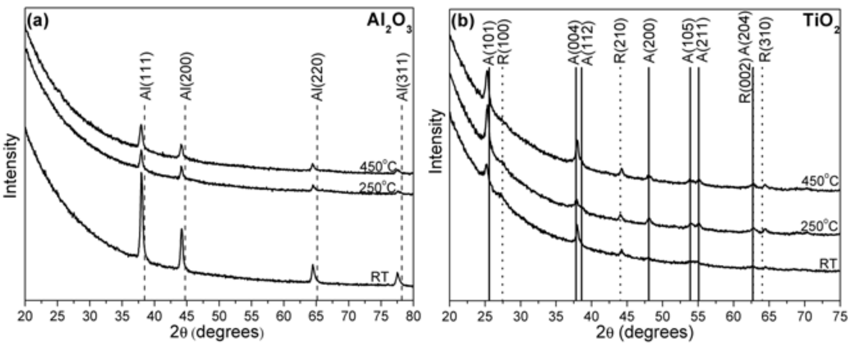

Figure 2: Diffractogram of (a) alumina's film at different temperatures and (b) titania's film at different temperatures.

References

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