ABSTRACT

CrN, TiN thin films and CrN/TiN superlattice thin films were prepared employing Closed Field Unbalanced Reactive Magnetron Sputtering. The deposition was carried out in a TEER UDP-450 system consisting of the cylindrical deposition chamber equipped with two targets located at diametrically positions. The substrate holder is located at the centre of the deposition chamber and it has rotation capability in three discrete values of rotation speed. X-Ray techniques (XRD and XRR) were employed for the structural and morphological characterization respectively. Spectroscopic ellipsometry was employed for the optical characterization using the combined Drude-Lorentz and Tauc-Lorentz model. For the nanomechanical characterization nanoindentation technique was employed.

CrN and TiN thin films were deposited at various values of N_2 flow (reactive gas) and substrate bias voltage, while a set of thin films was deposited on rotated substrate at the three discrete values of substrate holder rotation speed. The microstructural characterization revealed that the N_2 flow is the crucial parameter for the growth of the various phases (Cr_2N, CrN) of the Cr-N system. Moreover, it was determined the ranges of N_2 flow where the sinlge phases of the material dominate. The substrate bias voltage was found to affect the preferred growth orientation, the cell size, the internal stress field and the density of the thin films, since it affects the mobility of the adatoms on the surface of the growing film and the subplantation conditions of the plasma ions during thin film growth. More specific the [100] orientation is favoured when the mobility of the adatoms is increased due to the increase of energy transferred to the growing film during the ion bombardment. Contradictory, when the energy which is transferred to the surface of the growing film is decreased due to either the decrease of energy of the ions or the dissipation of the ions' energy in to the bulk of the film the [111] orientation is favoured. Furthermore, the cell size of the films increases with bias voltage, since the number of the plasma ions subplanted increases and consequently increases the lattice deformation. The above has a result an increase of the internal compressive stresses. On the other hand in the case of thin films growing on rotated substrate has as a result reduce of ion bombardment which affects their microstructure and morphology. The optical characterization of he CrN thin films revealed the interrelation between structure and morphology on the one hand and optical properties on the the other hand. Thus Cr_2N is metal while CrN exhibits semiconductuve behaviour. This was validated both by measurements in the Vis-UV region using the combined Drude-Lorentz model and in the NIR-UV region using the combined Tauc-Lorentz model. The latter enable us to determine the value of the fundamental gap (~0.4 eV). In the case of TiN the results of optical characterization showed that the optical parameters of the material arising by the combined Drude-Lorentz model are in agreement with the results reported in literature. Moreover, the thickness of the TiN thin films was calculated by SE employing a three-phase model. In the case of the thin films growing on rotated substrate the thickness results revealed that the substrate is exposed to the target in the ~20% of the rotation. The nanomechanical characterization revealed the mixed Cr_2N-CrN phase is harder than the CrN phase. Furthermore, the study of the effect of bias voltage on the nanomechanical response of CrN and TiN revealed that the hardness is maximized in the case of pure grain orientation (either [111] or [100]), increase in density and internal compressive stresses. The above analysis was validated by the study of nanomachanical properties of films grown on rotated substrate.

The superlattice thin films were deposited at various values of bias voltage at the three values of substrate rotation speed. The estimation of bilayer period Λ and the thickness of the individual layers was achieve utilizing the data of the deposition rate of the single layers and the deposition geometry. The bilayer period was calculated employing XRD in Small and High diffraction angles. The results of the two techniques and the calculated and estimated results are in good agreement. The thickness of the individual layers was calculated by High-Angle XRD and SE using a formalism base on Bruggemann Effective Medium Theory. The results of both techniques are in good agreement with the estimated values. The nanostructural characterization revealed that the preferred orientation is not affected by the bilayer period in the case of film growth at $V_b=V_f$, while it is affected of films grown on biased substrates. The values of the internal stresses are lower by an order of magnitude compared to the values of corresponding single layers. This is attributed to the presence of semicoherent interfaces which lead to stress relief. As the value of the bilayer period decreases the compressive stresses increase due to the extension of the lattice deformation in to the bulk of the individual layers. The nanomechanical characterization showed that the hardness values are close to those predicted by the rule of mixtures. This can be attributed to the low values of the bilayer period. Finally, the micorstructural characteristics affect the nanomechanical properties of the superlattice thin films in the same way of as in the case of single layers.