ABSTRACT

The work presented in this thesis shows the study of different parameters of ion beam emanated from a conventional 2.3 kJ plasma focus device which are used for the deposition of thin films on zirconium substrate. These energetic ions are characterized by employing BPX65 photodiode detector placed at 9 cm along the anode axis and is used to estimate ion energy (range from 40 *keV* to 1.2 *MeV*) and ion number density (9.7×10^{19} m^{-3} to 1.796×10^{19} m^{-3}) of the ions emitted during one focus shot. The relativistic electrons emitted from DPF device play a vital role to incorporate the third element (ablated from the insert material placed at the anode tip by the bombardment of relativistic electrons) into the substrate matrix and to deposit composite films on different substrates.

The composite films are characterized by employing different characterization techniques such as XRD, SEM, EDS, Raman Spectroscopy and Vickers microhardness tester to explore the diverse surface properties (crystal structural, surface morphology, crystallite size, microstructure features, elemental compositions and surface microhardness) of the composite films. TRIM code and micro-indentation measurements are used to estimate the depth profile of the modified layers.

Nitriding of zirconium by employing the energetic nitrogen ions emanated from plasma focus device for multiple focus shots when the samples are placed at different angular positions confirms the formation of zirconium nitride films on zirconium substrate. The crystallinity and crystallite size of nitride phases increase with the increase of focus deposition shots while residual stresses are maximum at lower nitrogen content (lower number of focus shots) which decrease with the increase of nitrogen contents. Smooth and uniform surface morphology showing granular features of nitrides is observed at 10^{o}. The nitrogen content in the deposited films increases with the increase of focus deposition shots. The microhardness of the deposited film enhances up to 400% and increased by increasing the nitrogen ion dose and decreases rapidly by increasing the angular position. Hence different features of the deposited films are strongly influenced by ion energy flux, ion number density and sample angular position.

The XRD patterns of zirconium oxynitride nanocomposite film (deposited for 40 focus shots at different axial positions) show that the crystallinity of ZrN, Zr_3N_4 and ZrO_2

depends on the sample axial position as well as ion energy flux. The SEM images show the formation of compact layer at lower axial position which is due to high ion energy flux. The nitrogen content decreases with the increase of sample angular position. The maximum microhardness of the layers is found to be 7200 ± 12 MPa for 10 gram imposed load. The EDS exhibits the distribution of different elements present in the composite films.

ZrAlON composite films are deposited on zirconium substrate showing the formation of ZrN, Al_2O_3 and AlN compounds. Five focus shots are sufficient to initiate the nucleation of ZrN and Al_2O_3 while 10 focus shots are enough to initiate the nucleation of AlN. The crystal growth of nitrides/oxides increases with increasing focus shots (up to 30 focus shots) and after that re-sputtering of the previously deposited film takes place. Smooth surface is observed for 20 focus shots at 0^o angular position while net type microstructure is obtained for 30 focus shots. Rough surface morphology is observed for 40 focus shots. A typical microhardness value of the deposited composite films is 5255 ± 10 MPa for 10 grams imposed load which is 3.3 times the microhardness values of unexposed sample. The microhardness values of the exposed samples increase with increasing focus shots (up to 30 Focus shots) and decrease for 40 focus shots due to re-sputtering of the previously deposited composite film.

Nanocrystalline zirconium carbonitride (Zr_xCN_y) composite films composed of ZrN, Zr_2N and Zr_3N_4, ZrC and Zr_2CN compounds are deposited on zirconium substrate at room temperature for multiple focus shots. The average crystallite sizes estimated for ZrN (200) and Zr_2CN (111) planes are found to vary from 10 to 20 nm. Maximum compressive stresses (~ 3.9 GPa) in Zr_2N (002) plane for 30 focus shots while maximum tensile stresses (~ 6.5 GPa) in ZrN (200) plane for 20 focus shots are observed. Tensile stresses in Zr_2CN (111) plane are transformed to compressive one after 30 focus ion irradiation. Raman analysis exhibits the emergence of D and G bands relating to carbides phases during the deposition process. The SEM microstructure shows agglomerates of size 30 to 300 nm. The Vickers microhardness values of the composite films increase with the increase of focus shots and is found to be 5.6 ± 0.45 GPa for 10 g imposed load which is 4.5 times that of the untreated substrate and 1.5 ± 0.11 GPa for 200 g imposed load which is 1.3 times that of the virgin one.

Zirconium silicon nitride composite films consisting of Si_3N_4 and Zr_3N_4 compounds are deposited on silicon substrate at room temperature through the reaction of sputtered silicon by ion beams and ablated zirconium from the insert by using plasma focus device. The crystallinity of Si_3N_4 and Zr_3N_4 compounds increases with increasing ion irradiation. The crystallite size of nitride increases (from 9 ± 1 nm to 33 ± 3.5 nm) while % microstrain values developed in the nanocomposite films decrease (from 3.65 to 0.61) with increasing focus shots. The SEM images reveal different microstructure patterns like granular, oval and cauliflower types of the deposited films. It is found that crystal growth and stress relaxation inhibit the pit formations during the deposition process. The EDS results confirm the presence of silicon, zirconium, nitrogen and oxygen in the composite films.