ABSTRACT

The work presented in this thesis addresses the parametric study of ion beams emitted from Mather type plasma focus devices and their flourishing utilization in materials processing. Experiments have been performed by using two different plasma focus devices; a conventional 2.3 kJ plasma focus device developed under the joint venture of the United Nations University (UNU) and the Abdus Salam International Centre for Theoretical Physics (ICTP) designated as the UNU/ICTP device operational at the GC University Lahore and a modified version called the Nanyang X-ray source-2 designated as the NX2 device (a repetitive plasma focus) operational at the National Institute of Education (NIE), Nanyang Technological University (NTU), Singapore. The measurements of ion parameters such as energy, energy distribution, number density and current density are carried out in the ambient gas pressure by employing a BPX65 photodiode and a Faraday cup (FC) using time of flight technique.

A major motivation is to establish the optimum processing conditions for ion nitriding, surface modification, phase changes and carburizing of materials of industrial interest like Ti, $AlFe_{1.8}Zn_{0.8}$ alloy and SS-321 in plasma environment. The processed samples are characterized for structural and morphological changes, compositional profile and surface hardness by employing X-ray diffraction (XRD) at GC University Lahore, scanning electron microscopy (SEM) at University of Peshawar, field emission SEM (FESEM) and energy dispersive X-ray spectroscopy (EDX) at the NIE NTU Singapore, X-ray photoelectron spectroscopy (XPS) at the National University of Singapore (NUS) Singapore, Raman spectroscopy and Vickers microhardness test at Quaid-i-Azam University Islamabad, Pakistan. The SRIM code and microindentation measurements are used to estimate the depth profile of the modified layers.

Nanocrystalline spatially uniform TiN thin films with petal like features are developed on Ti substrates exposed to 30 focus shots at various axial positions. The surface roughness and the relative proportion of the TiN films are strongly influenced by the ion beam energy flux. The film acquires eminent appearance with maximum relative proportion of nitrogen at 7 cm axial position. The probable energy of the ions reaching this position is 64 keV with the maximum ion number density of 5.9×10^{13} cm^{-3}. The corresponding energy flux and current density are 2.69×10^{13} keV cm^{-3} nsec^{-1} and 1142 A cm^{-2}

respectively. The grain size of the film is estimated to be about 90 nm while the compound layer thickness is about 0.66 μm. The surface microhardness is also maximum at this axial position with typical value of 7650±10 MPa.

The SEM images of a typical microcracked TiN thin film and the SRIM code estimations of ion penetration help in understanding the growth mechanism of the film in terms of ion dose. The granular nanostructures appearing on the substrate surface are grown from nucleates of a few nm size developed by the energetic ions induced collision cascades. The predeposited nitride layer or nitrogen ions interstitially implanted into the substrate surface are also redistributed by the successive pulses of the ion beams leading to layer densification along with possible resputtering. Moreover, the temperature evolution during the DPF ions irradiation also enhances the reactivity of the nitrogen already introduced during the preceding pulses. The residual tensile stresses on the sample surface are transformed to the compressive stresses after DPF ion irradiation.

Nitrogen ions induced surface changes in $AlFe_{1.8}Zn_{0.8}$ alloy are investigated as functions of axial and angular positions for 30 shots. The expanded fcc phase of Al is evolved owing to the incorporation of nitrogen along with Fe and Zn into the Al lattice. A comparatively smooth and crack free nitride layer is formed on the sample treated at 7 cm axial and 10^0 angular position with 4- to 5-fold increase in Vickers hardness.

$TiN_{0.9}$ and $(Fe,Cr)_2N$ are deposited on SS-321 along with formation of non-stoichiometric $(Fe,Cr)_xN$ phase by exposing the samples to multiple focus shots in nitrogen plasma at different axial and radial positions. The transformation from $(Fe,Cr)_xN$ to $(Fe,Cr)_2N$ is attributed to an increased nitrogen ion dose. The point-like structures of flakes reveal the nucleation of crystal growth with the increased ion doses. The nitride layer is golden in colour and is spatially uniform with improved surface hardness.

Multiphase nanocrystalline titanium oxycarbide TiC_xO_y thin films composed of TiC_2, $TiO_{0.325}$, Ti_2O_3 and carbon phases are deposited on titanium substrate in CH_4 discharges by the UNU/ICTP and the NX2 devices. The nanocomposite films are non-porous and microcrack-free with grain-like surface morphology having spatially uniform carbon distribution. XRD, Raman and XPS results reveal the favorable evolution of multiphase coatings having a stoichiometric TiC_2 phase and graphitic carbon adsorbates along with the residual oxide ($TiO_{0.325}$, Ti_2O_3) phases with the lower energy flux and lower repetition rate in the UNU/ICTP treatment. Whereas, the deposition of carbon and a non-stoichiometric $TiO_{0.325}$ phase is favored due to the improved oxide removal and enhanced disorder in the substrate surface during the NX2 treatment. In addition, TiC_2 phase is also suppressed, possibly due to the enhanced substrate temperature caused by the higher energy flux of the ion beams and the higher repetition rate. The granular profile of the films attains a definite coagulation pattern. The energy flux of the ion beam and the repetition rate are found to be critical parameters which influence the preferred evolution of a particular phase during the restructuring of various phases.