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Deposition of TiC Thin Films Using Dense Plasma Focus Device

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Abstract
Thin films of titanium carbide (TiC) are deposited on stainless steel-304 substrates using dense plasma focus device. Thin films of TiC are deposited using different numbers of focus shots, like 10, 20 and 30. XRD patterns in all three cases are found to have diffraction peaks for (111), (200), (220) and (311) TiC crystalline planes. The XRD pattern for the 20 focus shots deposition matches well, in terms of not only positions but also relative intensities of diffraction peaks, with the Standard TiC pattern indicating the formation of good quality TiC film. Diffraction peaks related to stainless steel-304 substrate are also seen on XRD patterns. Scanning electron micrographs show increasing clustering of TiC grains with the increase in number of focus deposition shots. The EDX spectra show the presence of expected constituent elements. These results infer the first ever deposition of TiC thin films using dense plasma focus device.

1. Introduction

The dense plasma focus [1,2] is a simple device in which a short but finite two dimensional non-cylindrical Z-pinch compresses the plasma to high densities ($\approx 10^{25\text{-}26}$ m^{-3}) and high temperatures (≈1-2 keV). Historically, the dense plasma focus has been developed as a fusion device with most of its studies being done in hydrogen and its isotopes. More recently, the use of energetic ions of plasma focus for inducing phase change in materials [3-5] and for deposition of thin films [6,7] have been reported. The electrons from this device have been used for electron microlithography [8]. A high repetition rate, compact plasma focus has been successfully used for soft x-ray lithography, with line width down to below 0.2 μm [9].

Titanium carbide is one of the most commonly used industrial coating materials in many tribological applications [10] because of its outstanding wear resistance, high hardness, high strength and rigidity, good stability at high temperatures, and low coefficient of friction. Other attractive properties that make TiC a potential candidate material for use as electron injecting electrode in organic light emitting diodes are its good electrical conductivity and low work function [11]. In this paper we report, for the first time, the deposition of TiC films at room temperature on 304-stainless steel substrates using a pulsed plasma device called dense plasma focus.

2. Experimental Setup and Methodology

The deposition of TiC films was accomplished by using a simple single-capacitor dense plasma focus device designated as United Nation University/International Center for Theoretical Physics Plasma Focus Facility (UNU/ICTP PFF). It is a 3.3 kJ Mather-type focus device. The experimental setup along with the focus subsystems is sketched in figure 1. Details of the plasma focus facility are provided elsewhere [2]. Conventionally, the

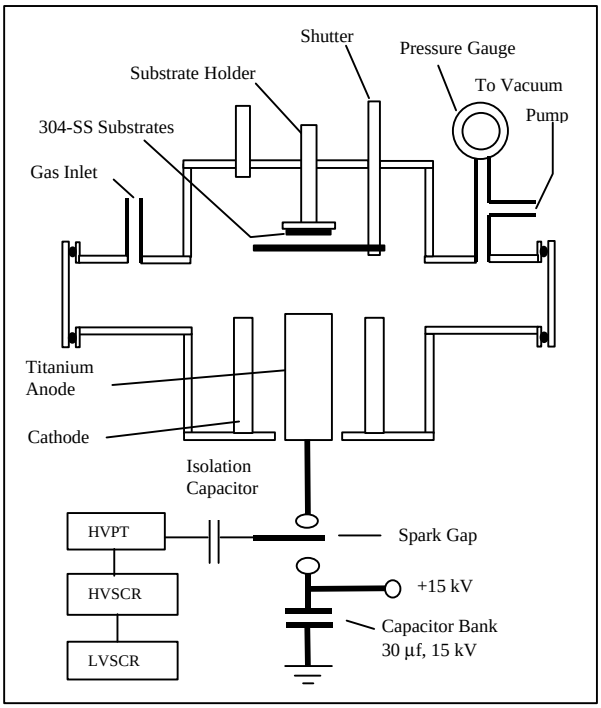

Fig.1. Schematic of experimental set up.

electrode assembly of UNU/ICTP PFF possesses a hollow copper anode being surrounded by six cylindrical copper cathode rods in a squirrel cage fashion. For deposition of TiC films, the central hollow copper anode was changed to solid titanium one whereas outer cathode rods were kept same. The argon-acetylene admixture was used as the working gas. Throughout the experiment, the filling gas pressure of argon-acetylene admixture (in the ratio 7:3) was kept at 1.5 mbar.

TiC films were deposited on 20mm×20mm×1mm stainless steel-304 substrates. Substrates were cleaned in ultrasonic bath using organic solvent and then mounted along the anode axis at the distance of 11.5 cm from the top of the anode. Three different thin films were deposited using 10, 20 and 30 focus shots.

The deposited TiC films were characterized by variety of techniques. The crystallinity was studied using Siemen s D 5005 X-ray diffractometer. The surface morphology of the films was studied using Jeol JSM 5310-V Scanning Electron Microscope. The recognition of elemental constituents of the film and their distribution mapping on the film surface was conducted using Energy Dispersive X-ray spectroscopy.

3. Results and Discussion

XRD patterns of all three deposited films are shown in figure 2. All three deposited films are polycrystalline and contain diffraction peaks for (111), (200), (220), and (311) TiC crystalline planes, as labeled in figure 2. These patterns show the successful growth of as-deposited crystalline TiC films on 304-stainless steel substrates for all three Samples. The respective positions of all TiC peaks are in agreement with the JCPDS standard data. It can be seen that the XRD pattern of film deposited with 20 focus shots matches quite well, in terms of not only positions but also relative intensities of diffraction peaks, with the JCPDS standard data. An additional diffraction peak, corresponding to (222) crystalline plane, can be seen in the XRD spectrum of this 20 focus shots deposition film. Other peaks that are observed in the XRD patterns of 10 and 20 focus shots deposition films correspond to that of the Chromium Iron Nickel 304-stainless steel substrate, as marked in figure 2.

It is interesting to note that the XRD pattern of 30 focus shots deposition shows the emergence of new phase corresponding to Chromium Iron Nickel diffraction peak, having different stoichiometery in comparison to 304-stainless steel ($Cr_{0.19}Fe_{0.70}Ni_{0.11}$). This induction of Chromium Iron Nickel phase has probably taken place at the film substrate interface because of the exposure to more plasma focus shots which causes greater ion energy deposition and hence the greater radiation damage. This hypothesis of greater radiation damage with more number of focus shots is also validated by a careful examination of XRD spectra of figure 2. It can be noted that the intensities of most of the TiC diffraction peaks have decreased with the increase in the number of focus shots. Only

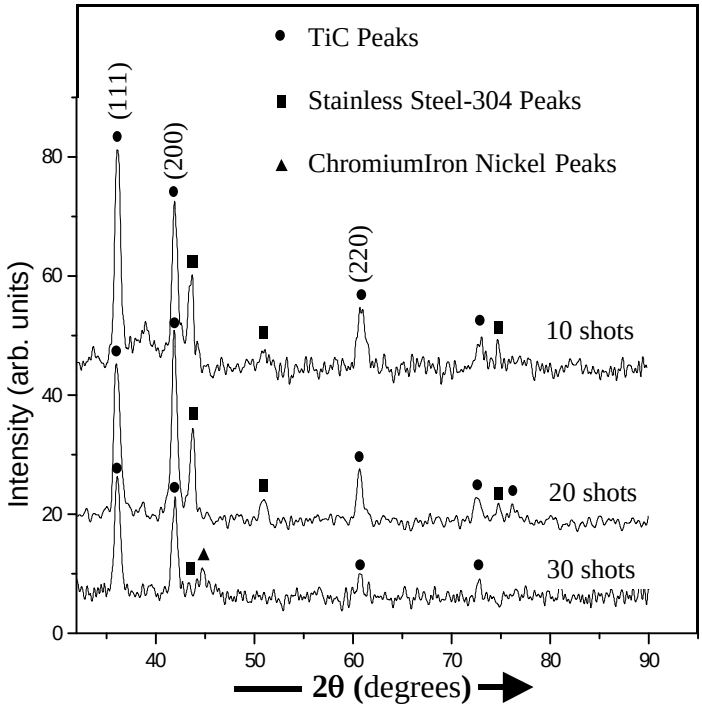

Fig.2. X-ray Diffraction patterns of films.

the intensity of (200) diffraction peak has increased from 10 to 20 focus shots deposition and another peak (222) has emerged in 20 shot film because of the proper phase induction in it. The XRD pattern of 30 focus shots deposition shows that the intensities of TiC diffraction peaks are least and, moreover, an altogether new phase of Chromium Iron Nickel is induced. This decrease in the degree of crystallinity of the deposited TiC film and induction of new phase on substrate indicate increasing radiation damage with the increase in number of focus shots.

Scanning electron micrographs of all three films are shown in figure 3. Surface morphologies are almost identical. Figure 3 (a), (b) and (c) show that, on all films, depositions have taken place along the grooves on the 304-stainless steel substrates as the substrates were not polished prior to deposition. The surface of 10 focus shots deposition film seems to have more individual grains in many different sizes. When viewed with greater magnification, the larger grains are seen to be clusters of many smaller grains. It can also be noticed that at many places grains begin aggregating along the grooves on the substrate surface. Aggregation increases with the increase in number of focus deposition shots (from 10 to 30 focus shots deposition). The increasing aggregation of TiC grains with the increase in the number of focus deposition shots may also be attributed to the increasing radiation damage caused by higher ion energy flux.

The EDX spectrum of one of the films, 10 focus shots deposition, is shown in figure 4. Peaks corresponding to elements present in the film and the substrate can be identified. The EDX spectrum is similar for all three samples, except that the relative intensity of TiK_α peak, arising from the deposited film, changes with respect to the FeK_α peak, which is generated in the substrate. The relative intensity ratio (TiK_α/FeK_α) of EDX peaks increases with the increase in the number of focus deposition shots because of the increase

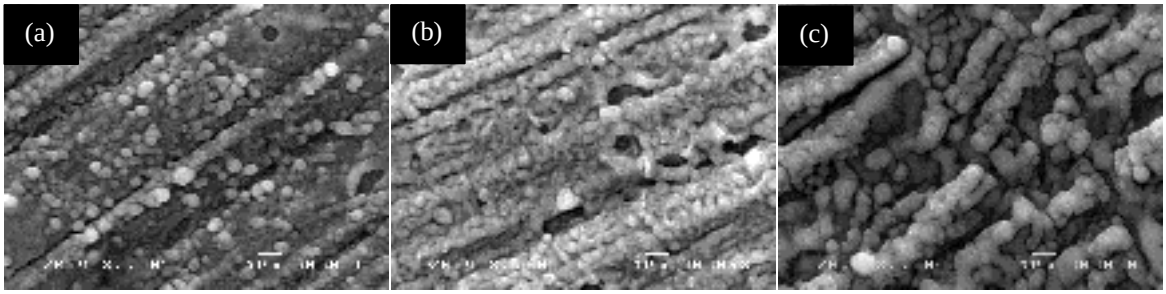

Fig.3. Scanning electron micrographs of (a) 10, (b) 20 and (c) 30 shots deposition.

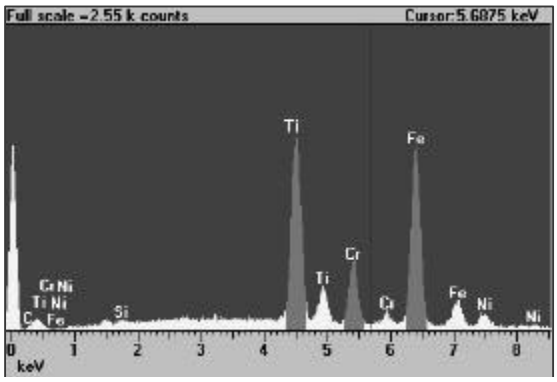

Fig.4. EDX spectrum of 10 shots deposition film.

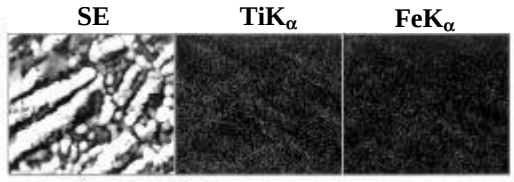

Fig.5. EDX map of 30 shots deposition film.

in the thickness of the deposited TiC film. The experimental value of TiK_α/FeK_α ratio, from EDX spectra, can be used to estimate the approximate average thickness of the deposited film by using a commercial software package Electron Flight Simulator[fi]. The estimated average thicknesses of three films are 6100, 6800 and 7700 Angstroms for 10, 20 and 30 focus shots depositions respectively.

Typical EDX map for 30 focus shots deposition film is shown in figure 5. The intensity of TiK_α and FeK_α maps, are qualitatively related to elemental concentration, with the strongest Ti signal arising from the larger TiC grains. Conversely, the iron signal is most intense in regions where the film is thinner. Similar results were obtained for the films deposited using 10 and 20 focus shots.

4. Conclusions

XRD results confirm the successful growth of as-deposited polycrystalline films of TiC. EDX analyses have confirmed the constituent elements of the deposited films, while x-ray mapping established the nearly even distribution of TiC. It is demonstrated, for the first time, that as-deposited polycrystalline thin films of titanium carbide can be deposited using pulsed plasma focus device at room temperature substrates.

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