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# Substrate effect on the morphology, structural and tribology properties of Titanium carbide thin film grown by RF Magnetron Sputtering

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## Abstract-

Substrate – coating behaviour plays a crucial role in determining the efficacy of the resulting properties of thin film coating. This research work studies the impact of two different substrates (Ti6Al4V and CpTi) on the mechanical properties of Titanium carbide thin film coating deposited using RF Magnetron Sputtering. The tribology properties were characterized using Nanoscratch and Tribo-tester. GIXRD and FESEM were used to study the structural and surface morphology of the coatings. The micrograph images of the FESEM show a similar trend for the film distribution while the GIXRD highest peak intensities vary for the coating. Spallation, bending crack and delamination of the film were noticed under high load condition and the micro-scratch result shows good adhesion between the coating and the substrates.

**Key words:** Substrate, Coating, RF Magnetron, Characterization

## 1. Introduction

With the global advancement in technology and research, the demand for materials with specific and modified properties are increasing sporadically. Different manufacturing techniques are currently available for developing new materials or modification of the existing one to meet demand. One of such manufacturing techniques that is becoming a new heart in the pursuit of globalization is the surface modification process. Surface modification involves modifying surface properties such as wear resistance, fatigue, creep strength, corrosion and erosion resistance, thermal resistance etc. of materials using techniques like thermal treatments, thermo-chemical treatment, implantation, plating and coating to achieve the desired properties [1-8]. Titanium carbide TiC is a material of choice with excellent properties for surface modification process. It is a synthetic, hard, high melting, good thermal and wear resistance, excellent biocompatibility, superior surface hardness and high electrical conductivity material [9, 10]. With these outstanding properties, it possesses the potential for superficial modification of materials. Out of the several surface modification techniques, radio frequency (RF) magnetron sputtering which is a variant of physical vapour deposition is a favourable technique for modifying surface of materials due to advantages like simple apparatus, low deposition temperature, high deposition rate, good adhesion of coating, uniformity and suitability of large scale and easily controlled process parameters [11]. RF sputtering deposition involves the ejection of neutral atoms from the target and condensation of the atoms on the substrate by exchange of the momentum process performed by the impingement of energetic ions us RF voltage source[7, 12]. There are several factors that determine the quality of thin film grown by



RF magnetron sputtering process. These factors include working pressure, temperature, substrate type, post-annealing treatment, reactive gas, atmosphere, drying condition, deposition time and target-substrate distance and to achieve the optimal desired properties, it is imperative to select the proper deposition technique and deposition condition [13-17]. However, the effect of most of these factors have been extensively studied but the substrate effect is still generally understudied. Substrate behaviour is one of the key determinants of the properties of thin film. Excellent bonding between the substrate and the coating is very pertinent for the development and application of coating. Hence, in this study, TiC thin films were deposited on commercially pure Titanium (CpTi) and Titanium grade IV (Ti6Al4V) alloys. We investigated the effect of the substrates on the morphology, structural and tribology properties of the films.

## 2. Methodology

### 2.1 Preparation of Substrates and thin film deposition process

HHV TF500 versatile box coater located at Botswana Institute of Science and Technology, Palapye, Botswana was used for the RF Magnetron sputtering process. The coater is versatile and possesses the abilities to be used in three different coating configuration modes namely Ion Beam deposition, Evaporation and Magnetron Sputtering. A solid Titanium carbide (TiC) target of 99.5% purity was used as the target source. The substrates used are CpTi and Ti6Al4V alloys. Before deposition, the surfaces of the substrates were ground with Silica carbide papers (#320, #400, #600) and polished to remove surface impurities. The substrates were additionally cleaned with acetone, isopropanol and finally with deionized water for 15mins respectively. The substrate-target distance was maintained at a constant position throughout the deposition process and the substrate was located directly above the target on a rotating disc to ensure uniform distribution and condensation of the target on the surface of the substrate. The sputtering chamber was first evacuated to a pressure of  $1.13 \times 10^{-5}$  mbar to provide a long mean free path for collision between the target and substrate and minimizes contaminants. The process parameters varied for the coating process is presented in table 1 while other working parameters were kept constant. After thorough evacuation of the chamber, the system was filled with argon to a partial pressure of 0.1-10 Pa to initiate the plasma formation. After the plasma formation, the sputtering of the target is triggered, and the shutter is removed, and pre-sputtering was done on all the samples for 5 mins to remove contaminants from the target surface and maintain system stability before the main sputtering deposition.

Table 1 RF Magnetron Sputtering Process Parameters

Experimental number	run	Sputtering Parameters					
		RF (W)	Power	Sputtering (Hrs)	Time	Temperature (°C)	Substrate
A1		150		2		80	CpTi
A2		200		2		90	CpTi
B1		150		2		80	Ti6Al4V
B2		200		2		90	Ti6Al4V

### 2.2 Film Characterizations

Field Emission Scanning Electron Microscope FESEM (ZEISS Gemini\*2, Germany) capable of capturing nanoscale images effortlessly at very high magnification was used to observe the surface morphology evolution. The FESEM images were taken at 50,000x magnification.

Grazing Incidence X-ray Diffractometer (PANalyticals's Xpert Pro with Cu K-alpha and wavelength 1.540598 Å GIXRD) at a very low angle of incidence ( $0.02^\circ$ ) that allows penetration into the thin film was used to study the structural properties of the thin film. The peeling behaviour of the coatings was evaluated using a linear displacement micro scratch tester (Anton Paar Micro Combo Tester, Austria). A Rockwell diamond ball with a radius of 100  $\mu\text{m}$  was used for the microscratch test to determine the film adhesion toughness and coefficient of friction. For each measurement, the applied load was progressive from 50 mN to 2 N at a sliding speed of 2 mm/min and sliding length of 1mm. All these experiments were carried out in an ambient atmospheric condition. The failure mode of the film under high load was performed on a Rtec Universal ball-on-disk tribometer at ambient condition. (temperature, pressure, and humidity). An E521000 Alloy Steel Grade 25 ball (6.350 mm in diameter) was used as the counter body, and the obtained thin film coating specimens served as the disk. The tests were carried out at a normal load of 10N, sliding velocity of 1mm/s, for 2 mins and sliding displacement of 1mm. The friction coefficient was monitored continuously during the experiments by a linear variable displacement transducer and recorded on a data acquisition computer attached to the tribometer. After completion of the wear, the wear scars were observed on the microscope for further analysis of the surface deformation. All the data are collected from the average test value for three samples with the same deposited parameters to confirm valid values in this work.

### 3. Result and discussions

The surface micrographs of the films measured from scanning electron microscope are illustrated in figure 1. The morphology revealed a similar trend for the thin films on different substrates. Sparse distribution of the thin film coating was noticed at RF power of 150 W and temperature of 80  $^\circ\text{C}$  for both substrates. As the process parameters increase, columnar growth pattern was noticed and the crystalline quality improved. TiC thin film becomes continuous, homogeneous, densely packed with no void and porosity on the surface. Higher process parameters favour interfacial reaction between the substrate and the target. The improvement in the microstructural evolution might be presumed to be higher rate of bombardment of the ions with the target and rapid migration of the ejection atoms to the surface of the substrate. It can be seen that the morphology depends more on the RF power and temperature than the substrate type.

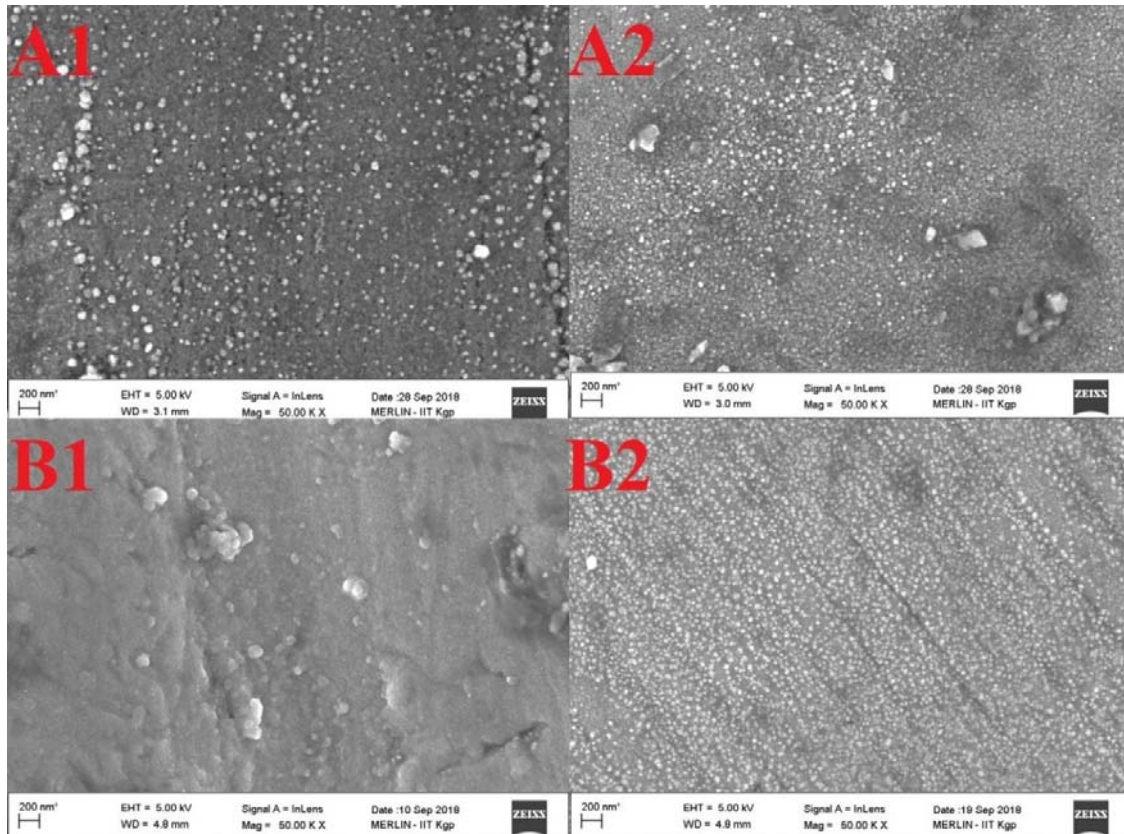


Figure 1 Micrography of Surface Topography of TiC thin film under different Process Parameters

The XRD spectra for the film are presented in figure 2. The characteristic XRD peak intensity of the TiC films are different for the two substrates. The preferential orientation (111) plane was observed to be the maximum plane for the CpTi substrate while preferential orientation at (200) plane was noticed for the Ti6Al4V substrate. Other noticeable diffraction peaks associated with TiC film were found at (220), (222) and (311) planes. The preferential orientation plane of (111) found in samples A1 and A2 are in good agreement with the work of Djafer et al [18]. They suggested that this orientation plane is attributed to the smallest surface energy storage in the stress state and this is associated with coating deposited at low energy level. Pelleg et al [19] explained that the preferential orientation of (200) plane would grow in the initial stage of deposition because of its lowest energy surface while only considering the thermodynamics without factoring the kinetic features deposition parameters, ion bombardment, incidence angle and energy. Chun [20] reported that the preferential orientation peak is critically affected by the deposition parameters and the stress state of the coating which is made up of strain and surface energies. The preferential orientation at (200) is favoured if the surface energy is dominant which has the lowest surface energy while preferential orientation of (111) plane is foremost if the strain energy is the dominant factor. The characteristic peaks range from  $32^{\circ} < 2\theta < 85^{\circ}$  for both substrates. The main peak of Ti6Al4V substrate at  $2\theta = 40.3243^{\circ}$ , and the corresponding main peak for CpTi at  $2\theta = 38.3091^{\circ}$ . As the temperature and RF power increases, the diffraction peak intensity becomes more defined. Zhang et al attributed this phenomenon to the kinetic energy and lattice position relationship. At low temperature and power, the adatoms

have low kinetic energy which weakens the surface mobility of the adatom to be positioned in the preferred lattice orientation and reduces the quality of the films [17, 21]

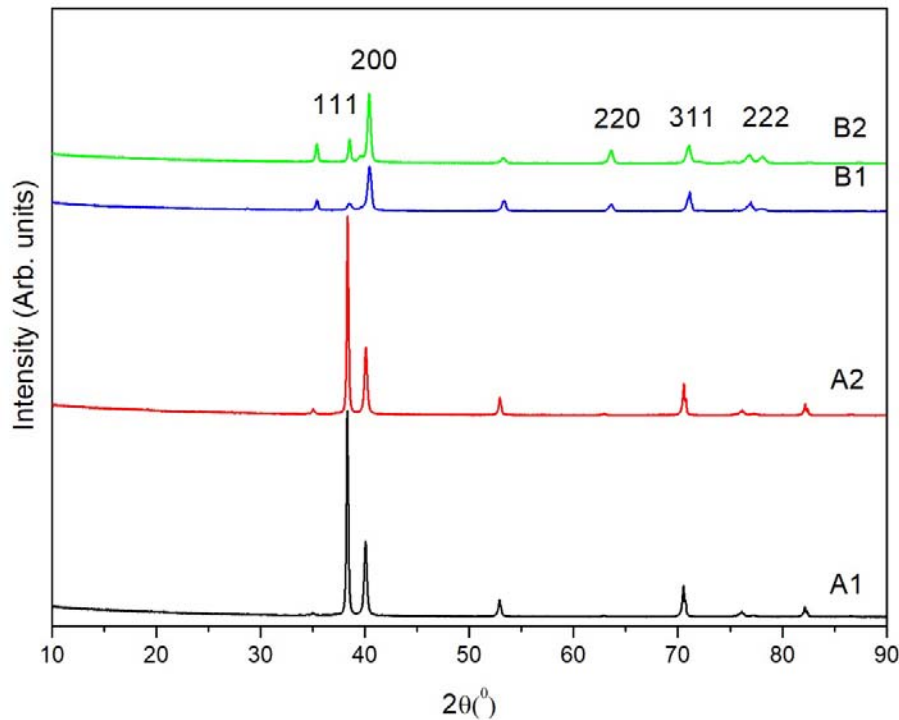


Figure 2 XRD Spectra for Sample A1, A2, B1 and B2

To determine the coefficient of friction COF, adhesion strength and failure modes, tribology tests were performed on the samples on two different scales of micro and macro levels under influence of different loadings configurations. The microscratch tester used for determining the adhesion strength and scratch resistance shows that the friction coefficient is influenced by the substrate type and process parameters. High coefficient of frictions was noticed at samples A1 and A2 compared to B1 and B2 respectively. No plastic slippage was noticed for all the scratch test which implies that the load used for the scratch test were below critical load for the failure of TiC thin film. The same coefficient of friction behavioural pattern was noticed for the macro scratch test at higher load. The coefficient of friction for samples with CpTi substrate was higher than the samples with Ti6Al4V substrates which agrees with the work of Kim et al [22] on the influence of substrate on the tribology properties. Kim et al [22] reported that the COF is also affected by the mechanical properties of the substrate at high loading. The increase friction coefficient for samples A1 and A2 is influenced by the (111) preferential orientation peak noticed for the samples. This agrees with the study of Feng et al [23] and Zhang et al [24] which reported that increase in hardness causes increase in the scratch friction coefficient and (111) plane is established to have higher hardness than (200) plane.



Table 2 Coefficient of Friction for Micro and Macro Scratch tests

Experimental run number	Friction Coefficient	
	Microscratch	Wear
A1	0.031	0.46
A2	0.071	0.49
B1	0.02	0.38
B2	0.0543	0.40

The micrograph of the wear tracks is present in figure 3 to study the thin film failure mode. Noticeable failure mode includes thin film spallation, delamination of the film and bending cracks. Chipping out of the TiC thin film were seen at the edges of the wear track. It was generated from the interaction behaviour between the superficial thin film and the sliding ball indenter. The size of TiC thin film coating removed to end of the wear track reduces from A1 to A2 and B1 to B2 respectively. As the coating density increases, the adhesion bond between the coating and the substrate become stronger and making it difficult for the thin film to undergo rapid plastic deformation even at high load. This phenomenon is responsible for low chip deposition at end edges and wear of the wear tracks for samples B1 and B2.

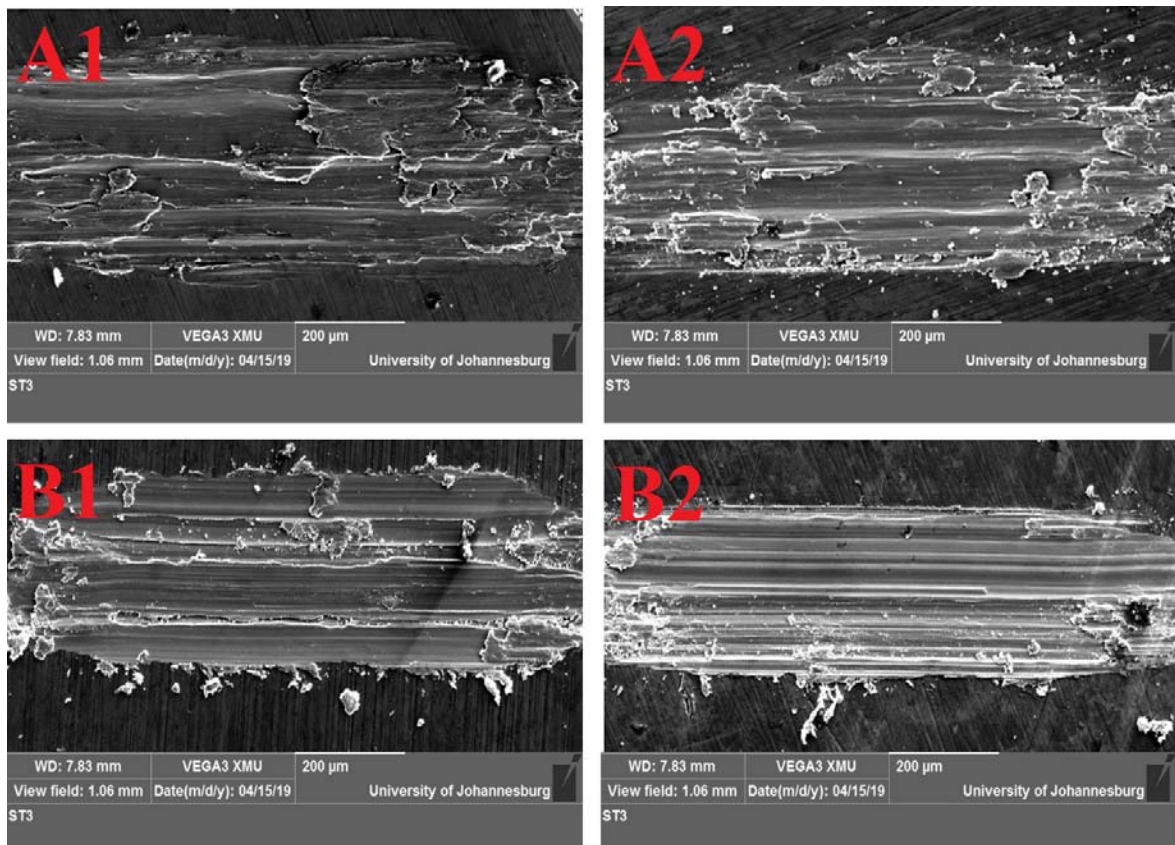


Figure 3 Micrographs of the Wear Track for Samples A1, A2, B1 and B2 respectively

#### 4. Conclusion

We investigated the effect of substrates on the morphology, structural and wear properties of TiC thin film. It can be concluded that the properties of TiC thin film are dependent on the substrate type as well as sputtering parameters. Higher RF power and temperature favour the film properties and the substrate type have a prominent influence on the properties of the films. The morphology becomes homogenous and densely packed as the process parameters changed from 150 W and 80 °C to 200 W and 90 °C. The preferred orientation of the XRD varied with the substrate type as two different peaks were noticed for the substrates and the peak intensity increases with rise in process parameter. The mechanical properties of the substrates influence the COF at high loading and the micro-scratch also revealed dependent on the COF on the substrate type.

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