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OF TIN COATINGS UNDER BOUNDARY LUBRICATION  
CONDITION**

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## Abstract

In engineering sciences, there is an increasing demand to reduce or control friction and wear of engineering materials. For a tool to be able to resist wear it must have a high hardness and strength, be chemically stable and possess a high toughness and low friction. In order to reduce friction, it must be capable of forming a stable compound at the contact interface to control the ensuing friction and wear behavior. However, it is impossible to combine all these properties together in a single conventional tool material. Nowadays composite coatings are being developed so as to attain desired properties. The actual top development in this regard is the development of Titanium Nitride (TiN) coatings, using Physical Vapor Deposition (PVD) technique.

In the present study, TiN coating was produced on HSS using the physical vapor deposition system. The resultant coating was then characterized for their adhesion measurement, coefficient of friction and wear resistance. The results show that TiN coatings have quite high wear resistance and hence can be used in cutting tools to increase the tool life.

**Key Words:** Co-efficient of friction, Wear rate, Physical Vapor Deposition, Titanium Nitride, Tool Life, Tribology, Adhesion

## 1. INTRODUCTION:

A number of surface modification processes are being used to improve the life of a tool. There are certain advantages, disadvantages and applications of each process. In some cases surface modification processes can be used to modify the substrate surface prior to the deposition of a film or coating. For example a steel surface can be hardened by plasma nitriding (ionitriding) prior to the deposition of a hard coating by PVD process.

Physical vapor deposition (PVD) is fundamentally a vaporization coating technique, involving transfer of material up to atomic level. It is an alternative process to electroplating technique. The process is similar to chemical vapor deposition (CVD) except that the raw materials or precursors, i.e. the material that is going to be deposited starts out in solid form, whereas in CVD, the precursors are introduced to the reaction chamber in the gaseous state <sup>[1]</sup>.

Wear is the deformation and material loss of a material in moving contact with another material. Wear and erosion of a film can be measured by: weight loss, material transfer and wear scars. Wear is extremely sensitive to the application, temperature, material etc. So most wear tests are functional type test <sup>[2][3]</sup>. The mechanical properties of films are important in their response during subsequent processing and to mechanical stresses <sup>[4][5]</sup>.

In the present study, TiN coating was produced on high strength steel (HSS) substrates using PVD technique. The resultant coatings were then characterized for their tribological

properties at high temperature in the presence of lubricant oil. The wear tracks thus produced were analyzed using Scanning Electron Microscope.

## 2. EXPERIMENTAL WORK

### 2.1 Sample Preparation

High Strength Steel (HSS) coupons were used in present study. Surface of the substrate was degreased using Carbon Tetra Chloride ( $\text{CCl}_4$ ) in ultrasonic bath for 5 minutes. After washing, samples were thoroughly treated with distilled water to remove the traces of  $\text{CCl}_4$ . The samples were then placed in Cleano-Gel cleaning solution at temperature of  $60^\circ\text{C}$  for about 10 minutes. Finally the samples were cleaned in ultrasonic bath with acetone followed by careful transfer to coating chamber avoiding any contamination.

### 2.2 Deposition of Coating

PLATIT  $\pi^{80}$  coating system was used to deposit TiN upon HSS substrates. The system is fully automated and is controlled by the process computer. The coating deposition was conducted in a flowing pure nitrogen atmosphere. The cleaned HSS coupons were mounted on carousel substrate holders which rotate continuously around the vertical central axis at a speed of 12 rpm. During deposition, a negative DC bias of -70 V was applied to the substrates. The parameters are shown in Table 1.

**Table-1:** Technological Parameters of PVD Process

Parameter	Coat	Substrate Temperature ( $^\circ\text{C}$ )	Nitrogen Pressure (Pa)	Accelerating Voltage (Volts)	Coat Period (Min)
Set Value	TiN	450	1.5	70	120

## 3. CHARACTERIZATION OF COATINGS

### 3.1 Wear Testing

Friction co-efficient of the coating was measured by CSM Pin-on-Disc Tribometer. Two types of experiments were performed:

- Without Lubricant (dry condition)
- With Lubricant (wet condition)

Steel 100Cr6 balls with diameter 6mm were used during experiments. All the experiments were run for 25000 laps. Machine oil was used as the lubricant whose volume was  $50\mu\text{l}$ . The volume was measured by Micro-Pipette. All experiments were run at 2 to 5N load.

**Table-2:** Pin-on Disc Test Parameters

Pin	Speed (cm/s)	Number of Cycles	Pin Load	Temperature ( $^\circ\text{C}$ )
100Cr6 Steel ball, $\Phi$ 6mm	2	25000	2N	25
	4		3N	
	6		4N	
	8		5N	

### 3.2 Scanning Electron Microscopy (SEM) Analysis

A Hitachi S-3700N Scanning Electron Microscope was used to investigate the tracks produced by Tribometer. The scratches and wear damages after tests were thoroughly observed. Images were taken using secondary electron detector and typical imaging condition of 10 kV.

### 3.3 EDX Analysis

The Energy Dispersive X-Ray (EDX) analysis of the tracks was carried out to analyze the chemical composition of the tracks and confirm whether the coating has delaminated or not.

## 4. RESULTS AND DISCUSSIONS:

### 4.1 Wear Testing

#### 4.1.1 Without Lubricant (dry condition)

Table-3 summarizes the co-efficient of friction of each track at a varying load from 2 to 5 N. It can be clearly seen that the co-efficient increases as the load increases. The weight on the ball is also decreasing while increasing the load which is the indication of good coating adhesion with the substrate.

**Table 3:** Summary of Test results in Dry environment

Sr. #	Load (N)	Initial weight of the sample, $W_{1s}$ (gm)	Final weight of the sample, $W_{2s}$ (gm)	Initial weight of the Static partner, $W_{1sp}$ (gm)	Final weight of the Static partner, $W_{2sp}$ (gm)	Co-efficient of Friction, $\mu$
1	2	5.77306	5.77254	1.04901	1.04884	0.568
2	3	5.77254	5.77302	1.04884	1.04869	0.622
3	4	5.77302	5.77297	1.04869	1.04813	0.553
4	5	5.77297	5.77288	1.04813	1.04783	0.991

#### 4.1.2 With Lubricant (wet condition):

The table-4 shows the co-efficient of friction of tracks at varying loads of 2 to 5N in the lubricating environment. Table 5 shows that there is no significant loss in the weight of the coating. The EDX analyses of these tracks confirm that there is no sign of the deposition of ball material on the coating.

**Table 4:** Summary of Test results in lubricating environment.

Sr. #	Load (N)	Initial weight of the sample, $W_{1s}$ (gm)	Final weight of the sample, $W_{2s}$ (gm)	Initial weight of the Static partner, $W_{1sp}$ (gm)	Final weight of the Static partner, $W_{2sp}$ (gm)	Co-efficient of Friction, $\mu$
1	2	4.90536	4.90537	1.04348	1.04343	0.093
2	3	4.90537	4.90558	1.04343	1.04339	0.104
3	4	4.90558	4.90549	1.04339	1.04332	0.077
4	5	4.90549	4.90545	1.04332	1.04324	0.103

## 5. SCANNING ELECTRON MICROSCOPY (SEM) WITH EDX ANALYSES:

Fig. 1 (a), (b), (c) and (d) show scanning electron micrographs along with their EDX analysis. It can be clearly observed from the micrographs that the coating is quite adherent to the substrate and at very high number of cycles (25000) in dry conditions, the coating does not delaminate and instead the ball material is deposited on the coating which is clearly shown by the loss in weight of the ball in table-3. The traces of elements of steel present at the wear tracks, as shown by the EDX analysis is not the indication of the appearance of substrate. It is rather the indication that the material of steel ball has been transferred to the wear tracks during wear testing, which can be seen in table 3.

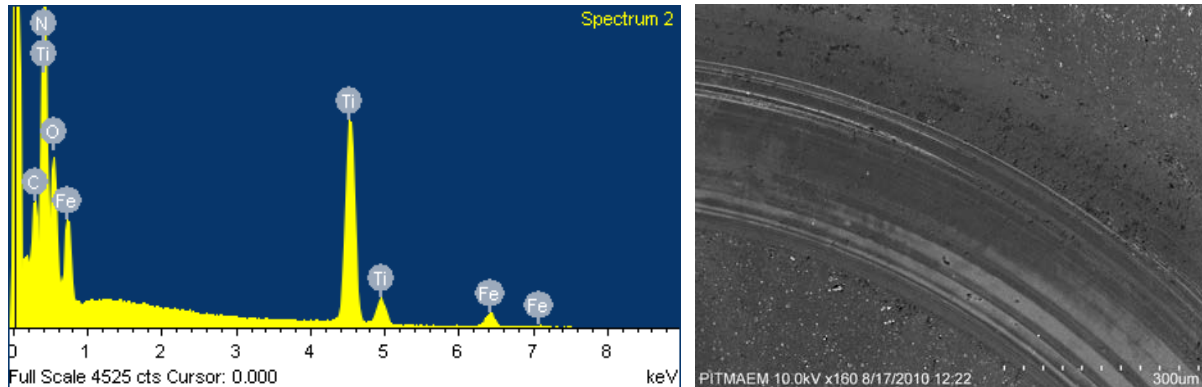


Fig.1(a) EDX analysis of wear track at 2N in dry conditions, showing highest percentage of coating and traces of ball material deposited on the coating.

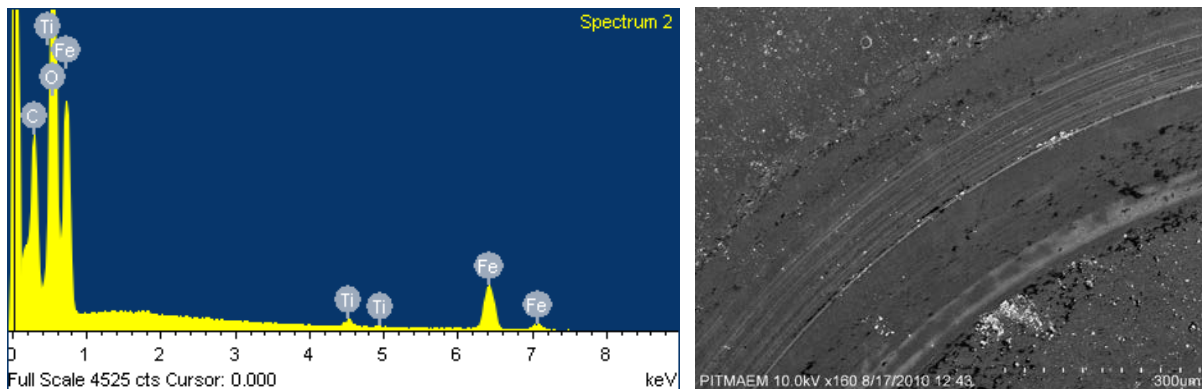


Fig.1(b) EDX analysis of wear track at 3N in dry conditions, showing highest percentage of coating and increased traces of ball material deposited on the coating.

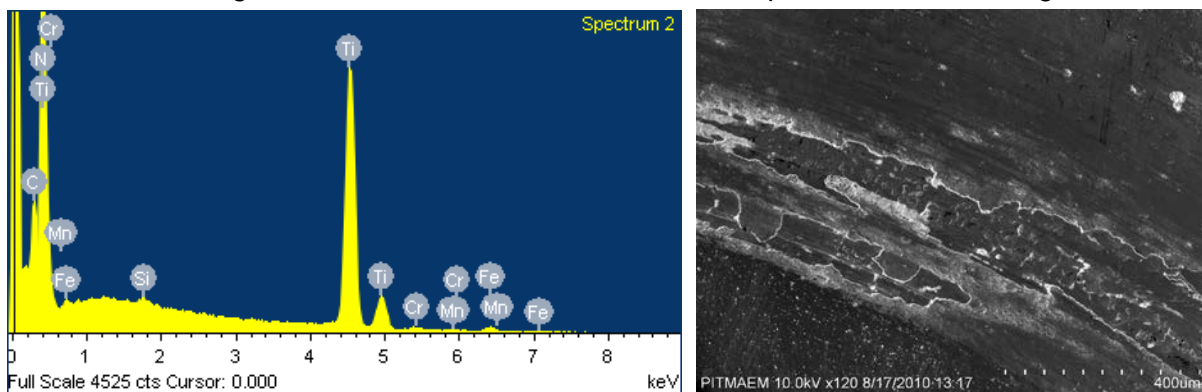


Fig.1(c) EDX analysis of wear track at 4N in dry conditions, showing highest peaks of coating and more intense traces of ball material deposited on the coating.

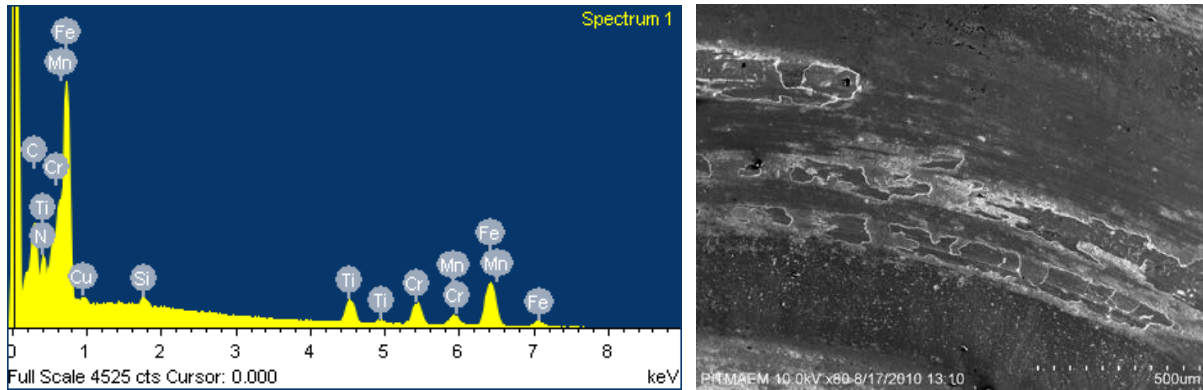


Fig.1(d) EDX analysis of wear track at 4N in dry conditions, showing highest peaks of coating and more concentrated traces of ball material deposited on the coating.

Fig. 2 (a), (b), (c) and (d) show scanning electron micrographs of wear tracks produced in the presence of lubricant along with their EDX analysis. It can be clearly observed that there is no appreciable wear on the coating after 25000 cycles. There is even no transfer of ball material which is clearly shown by in table-4.

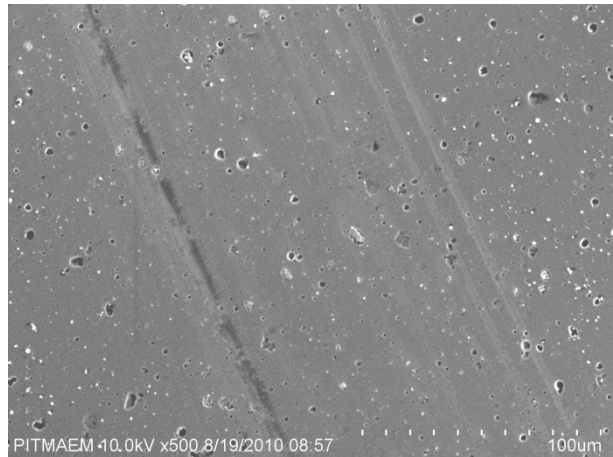
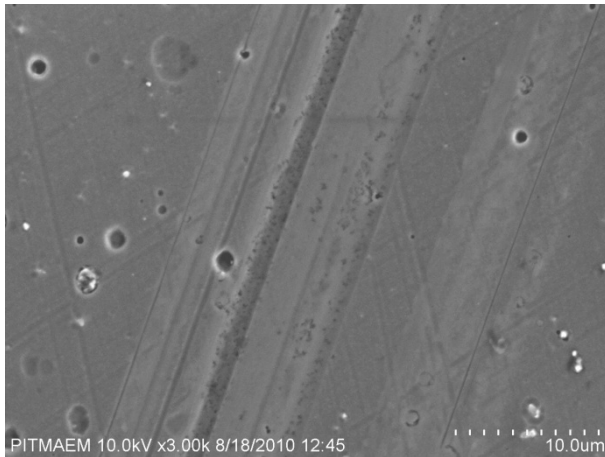


Fig.2 (a) Track at 2N in lubricating environment

Fig.2 (b) Track at 3N in lubricating environment

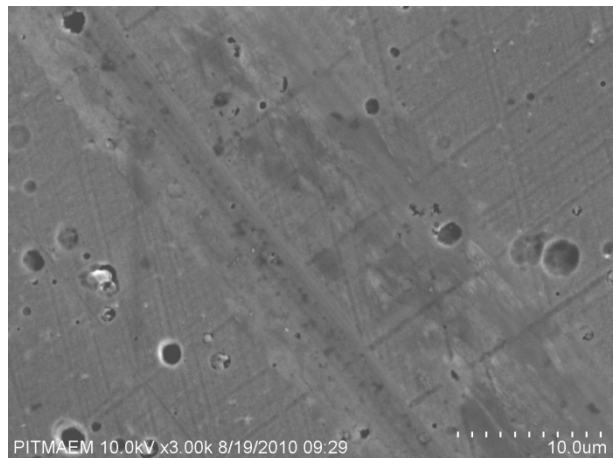
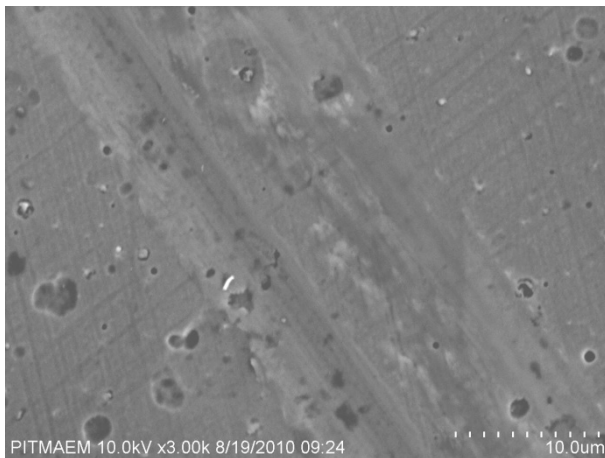


Fig.2 (c) Track at 4N in lubricating environment

Fig.2 (d) Track at 5N in lubricating environment

## 6. CONCLUSIONS:

- a. The presence of lubricant greatly reduces the co-efficient of friction.
- b. Even at very high number of cycles, the substrate surface is not revealed which is proved by the EDX analysis of tracks.
- c. The co-efficient of friction increases while increasing the load.
- d. The excellent adhesion of the coating is also proved by testing in the dry conditions where coating was not delaminated.
- e. Negligible loss in weight indicates very low wear rate of the coating.
- f. Tin coatings can be quite efficient for automotive moving parts and cutting tool. The efficiency of the part demands low co-efficient of friction and low wear rate of the coating, hence increasing the part's life to a great extent.

## 7. REFERENCES:

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