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Comparative Study of the PVD Coating on the Plasma Nitrided Mild Steel

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***Abstract*—In this work, Duplex Coating (Plasma Nitriding + PVD Arc TiN coating) as well as non duplex (PVD TiN) coating has been carried out on substrate i.e. Mild Steel (MS). The substrate material was chosen because Mild steel has wide applications especially in Marine Engineering, Oil & Gas Production & Transmission industry as well it is a cost effective material. Surface Characteristics/Properties were investigated on PVD Titanium Nitride (TiN) Coated and Duplex Coated (Plasma Nitrided + PVD TiN) by Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), Micro Hardness Test and Rockwell Adhesion Test. Duplex coated samples (Plasma Nitrided + PVD TiN coated) showed better Mechanical Properties like Higher Hardness and higher Adhesion strength compared to their non-duplex (PVD TiN coated) counterparts, hence more suitable to be used in Mechanical applications.**

***Keywords*—Duplex Coating, Plasma Nitriding (PN), Physical Vapour Deposition (PVD), Microhardness, Adhesion**

1. Introduction

Physical Vapour Deposition (PVD) TiN coatings are widely used in many engineering applications due to their high hardness, high adhesion strength and low friction coefficient. Though, there has been rapid growth in the application of hard thin film coating in industry, the very few application have been found in general engineering applications because of commonly used low alloy steels with relatively low strength. When the soft substrate is subjected to high loads, deformation of the soft substrate leads to bending and fracture of the hard thin film, since the using substrate must have sufficient hardness and flow strength to support the coating without any plastic deformation when subjected to high loads [1, 2, 3].

Nitrided cases do not show sufficient resistance in the case of very strong attack by wear and corrosion. By an additional protective layer, such as a hard-coating, the load bearing capacity can be further improved. The hard-coating, for instance TiN, after nitriding provides a very hard, wear, heat and chemical resistant outer layer. By this way properties obtained by the combination of nitriding with hard-coating allow function sharing between the core material, the hardened case and the surface [4].

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On Low base hardness; where coating alone would not work, because of not getting enough support for the coating & premature, catastrophic failure occurs, if substrate is subjected high load, Duplex Treatment (Surface modification + Coating) is being used.

Duplex treatments – applying surface modification and coating technology on the same component – produce a very hard and thin surface layer (good wear resistance), and beneath this layer a hard diffusion zone (enhanced fatigue properties).

2. Experimental

2.1 Substrate Preparation

Mild Steel was chosen as the substrate. Chemical composition and Microhardness of material are presented in Table1. Specimens were milled to the dimensions of 20 mm x 20 mm. Prior to plasma nitriding the specimens were grinded and polished to the surface finish of $R_a = 0.2$ µm.

Table 1. Chemical composition of Mild Steel

Material	**Chemical Composition (wt-%) / Structure**	**Hardness, $HV_{0.1}$**
Mild Steel	C-0.18, Si-0.40, Mn-0.70, S-0.04, P-0.04, Fe-98.64	210

2.2 Plasma Nitriding

Plasma nitriding process was carried out at Bhat Metals, Delhi on PlateG setup. The samples were treated at Temperature - 520˚C, Treating Time for Nitriding - 8 Hours, Cooling Time - 4 Hrs. A mixture of Hydrogen (25%) and Nitrogen (75%) was used in the present investigation for plasma nitriding process.

2.3 PVD Coating Process

Coating of Titanium Nitride (TiN) on the Untreated & Plasma Nitrided (PN) samples was performed at Balzers, Pune, India by Commercial PVD Arc coating process at Temp - 450˚C named BALINIT® A, which is used mostly with a broad range of applications. Prior to TiN deposition samples were treated in alkaline solution & ultrasonically cleaned.

2.4 Microstructure and EDS studies

Top surface morphology and Cross section of the specimen was studied under the SIGMA Zeiss Field Emission Scanning Electron Microscopy (SEM) Model. Distribution of chemical elements inside the Duplex Treated (Plasma Nitrided + PVD TiN coated) as well non duplex PVD TiN coated sample was determined by the means of Energy Dispersive Spectroscopy (EDS), which was incorporated in the same SEM system.

2.5 Microhardness Measurements

Vickers microhardness at the load of 100 gm was measured at the top surface of untreated, Nitrided, Duplex (PN + PVD TiN coated) and Non Duplex (PVD TiN coated) for an average of 3 indentations per sample using a LECO's Micro indentation testing system (LM 300 series). Low load of 100 gm ensures the limited indenter penetration depth to eliminate the substrate effect.

2.6 Adhesion Strength

Rockwell Hardness Test system FIE Make, Model BAB 250 was used to measure the adhesion properties of thin films on substrates i.e. Mild Steel. This test method is very easy to perform. The Rockwell-C adhesion test was developed in Germany and is standardized in the VDI guidelines. A 120˚ spheroid-conical diamond indenter (Brale) is used for Rockwell-C adhesion test. The hardness tester (applied load is 150 kg) causes layer damage adjacent to the boundary of the indentation. When the indenter penetrates the coating, cracks propagate from the indentation point. The damage to the coating was compared with a defined adhesion strength quality shown in Fig. 1. HF1 shows excellent adhesion properties with a few crack networks while HF6 shows the poorest adhesion properties showing complete de-lamination of the film [5, 6].

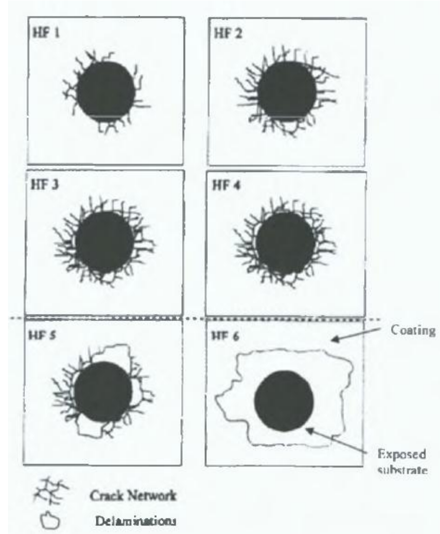

Fig.1 Adhesion strength quality HF 1 to HF 6 [6]

3. Results and Discussion

3.1 Microstructure Studies

A very smooth and featureless surface was noticed in the PVD TiN coating as per Fig. 2. Grain size of the Duplex (PN+PVD TiN) coating are seen smaller comparison to non duplex PVD TiN coating, hence from Hall-Petch equation (1).

$$\sigma_y = \sigma_o + k_y/\sqrt{d} \quad \ldots\ldots\ldots(1)$$

Where σ_y is the yield stress, σ_o is a materials constant for the starting stress for dislocation movement (or the resistance of the lattice to dislocation motion), k_y is the strengthening coefficient (a constant unique to each material), and d is the average grain diameter.

From Hall-Petch equation (1), it may be concluded that because of smaller grain size of duplex (PN+PVD TiN) coated surface; it is harder than non duplex PVD TiN coated surface. It has also been confirmed through Microhardness testing.

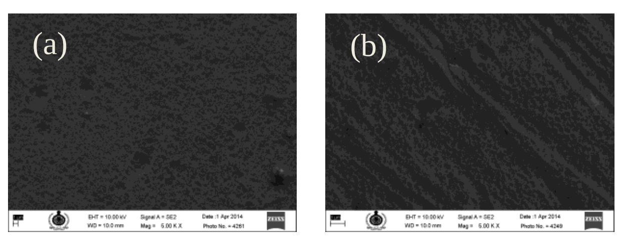

Fig. 2 SEM images of top surface of (a) MS PVD TiN coated (b) MS Duplex (PN+ PVD TiN coated)

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3.2 EDS Analysis

An Energy Dispersive Spectroscopy (EDS) line profile analysis at a certain spectrum was performed on the top surface of the substrate after PVD TiN coating for elemental analysis and chemical characterization of the sample. It confirmed the presence of the atoms like N & Ti and also small quantity of C & Fe. Weight percentage of Ti is found in range of 63% - 67%, while N is found in range of 28% - 30%, which confirms the proper deposition of TiN layer on the surface.

3.3 Cross Section SEM Image Analysis

Cross Section SEM images of the samples are shown in Fig. 3.

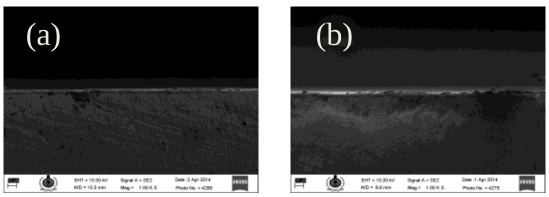

Fig. 3 Cross section SEM images of (a) MS PVD TiN coated (b) MS Duplex (PN+ PVD TiN coated)

A uniform layer of Titanium Nitride (TiN) coating thickness of 5.1 µm & 5.2 µm is observed on PVD TiN coated and Duplex (PN+PVD TiN coated) samples respectively.

3.4 Microhardness Measurements

Results of the MicroIndentation test for Microhardness values are brought in Table 2.The Plasma Nitrided substrate showed higher hardness than the non-nitrided substrate by a factor of two and a half. Duplex treatment (PN+TiN coating) has increased the hardness by more than six folds on Mild Steel substrates. The significant increase of the substrate hardness is believed to be increased by the composite effect of the thick hard nitrided surface underneath the coating, load bearing capacity of the coating.

Sample	**Microhardness ($HV_{0.1}$)**
Untreated	210
Plasma Nitrided (PN)	564
PVD TiN Coated	730
PN+PVD TiN coated	1319

Table 2. Microindentation values of the samples

3.5 Adhesion Test

Results of the Rockwell C Indentation test as 100X magnified SEM images are brought in Fig.4

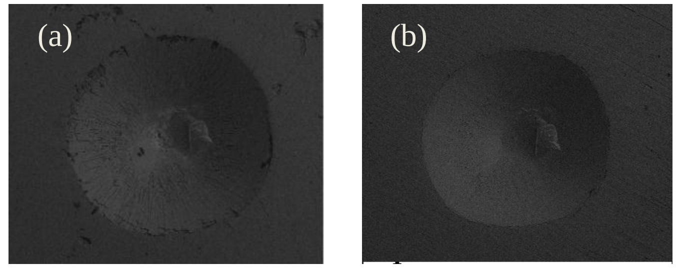

Fig.4 Rockwell Indentation Test for Adhesion of (a) MS PVD TiN coated (b) MS Duplex PN+ PVD TiN coated

From the Rockwell C indentation spot of the duplex and the non-duplex coating system, it was found that plasma nitriding significantly reduced crack network formation and plastic deformation beside the edge of the indenter. It can be clearly observed from Fig. 4 (a), that the coating on the non duplex (PVD TiN coated) sample showed a large number of the crack networks around the indentation spot. This feature represents adhesion in HF3 scale (bad adhesion). However, no crack network and no swelling type detachment or deformation were observed around the edge of the duplex treated sample's indentation spot indicating relatively good adhesion, as shown in Fig. 4(b). This feature represents adhesion in HF1 scale (good adhesion).

4. Conclusions

1. Duplex Coating (Plasma Nitriding + PVD TiN coating), which is being successfully used in materials like HSS, Tool steel for application in Tools, Molds and cutting operations. From this present work, it was successfully performed the Duplex Coating on economical & structural application oriented material like Mild Steel.

2. Micro-indentation test confirmed the hardness of the duplex (PN+TiN) samples to be increased up to six-fold, while the non duplex (PVD TiN) coated sample's hardness increased up to three and a half times.

3. Qualitative Rockwell Adhesion Test of the duplex treated (Plasma Nitrided + PVD TiN coated) samples confirmed the best quality Adhesion compared to non duplex PVD TiN coated samples. Adhesion of duplex coated samples was of the good quality (HF 1).

4. SEM images showed the smooth, homogeneous surface and smaller grain size of duplex coating compared to non duplex coating, which confirmed the higher hardness & better adhesion of duplex coating.

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