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IMPROVEMENT OF WEAR RESISTANCE BY VACUUM ARC EVAPORATION PVD THIN FILM TiN COATING

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Abstract

In this paper the wear resistance characteristics of a thin film coating of Titanium Nitride (TiN) applied on a 304 Stainless Steel by Vacuum Arc Evaporation (VAE) Physical Vapour Deposition (PVD) Technique is being investigated for checking the feasibility of thin film ceramic coating application on automotive engine components. So as to improve their wear resistance, life span as well as operating efficiency. Generally, engineering materials must have some specific characteristics and those characteristics which are important in selecting material for specific applications related with material structure and life-time. It is difficult to find all these features especially mechanical and surface properties in a single material. Surface coating enhances performance, reliability and service life and permits lighter, more compact designs. Reduced energy consumption and the use of environmentally benign products in smaller quantities are further advantages that come into play in the building of machines and equipment just as they can in engine and vehicle making. So the solution is found by increasing the strength of bulk material, and increasing wear and corrosion resistance of the surface. Therefore, engineering materials are selected from cheaper materials providing needed structural features, and the other surface characteristics are provided from coatings. Friction and wear of the sliding components in an automobile cause an increase in both fuel consumption and emission. Many engine components involved with sliding contact are all susceptible to scuffing failure at some points during their operating period. Therefore, it is important to evaluate the effects of various surface coatings on the tribological characteristics of the automobile parts.

Index Terms— Ceramic coating, PVD, Thin film, TiN, VAE, wear.

I. INTRODUCTION

Automobile manufacturers all over the world are putting heavy effort to produce more energy reliable, efficient and environment friendly vehicles. The world has to depend on internal combustion engines for some more years till other clean energy concepts like fuel cell technologies gets developed to be used in the automobiles. In the engine and engine components, power losses due to friction add up to 15% of the total energy losses in a vehicle [1]. Most of the friction related power losses occur at the cylinder liner, piston, and piston ring interfaces and in crank train, valve train and injection pumps which contribute to these losses. (Fig.1).

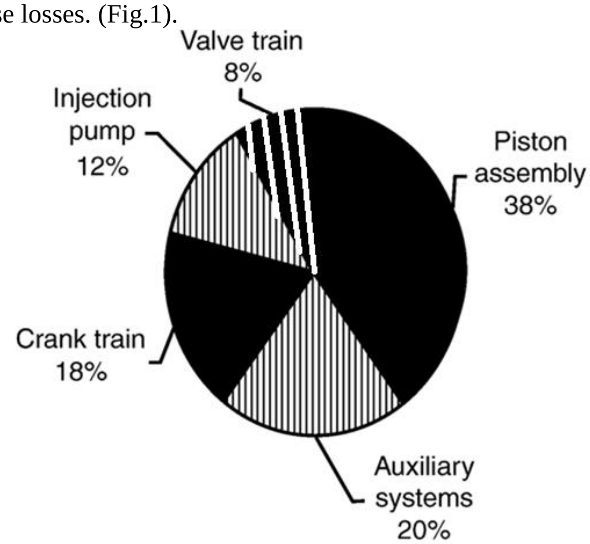


Fig. 1. Typical distribution of frictional losses in internal combustion engine.

Most of the studies on wear mechanisms in tribosystems points to how wear can be combatted:

- Increasing the surface hardness
- Applying inert surface coatings
- Reducing the friction coefficient

By using thin film coatings using PVD technique it is possible to coat metal surfaces with very thin film coatings of a few micro meter or even lower than a micro meter thickness with good adhesion to the substrate, without a costly change of design or material. Surface coatings can provide longer service life, tolerate higher loads, low and ease of maintenance, environmental gains and conservation of resources, improved response in kinetic systems, lower energy consumption, better corrosion resistance, use of lower cost base materials, etc.

Currently most of the commercially available piston rings are coated with chromium electrochemically [2]. For the last few decades, TiN coatings have been used for tools, dies, and many mechanical parts to enhance their life and performance owing to their attractive properties such as high hardness, good wear resistance, marginally superior adhesion and chemical stability. TiN forms an excellent protective coating because of its hardness, refractory nature and corrosion resistance. However, the tribological behaviour of TiN film is found to vary with substrate, deposition method, coating film thickness, stoichiometry, heat treatment and type of wear. Like most ceramic materials, TiN films are very brittle; they have tendency to fracture and separate from the substrate during wear, especially on softer substrates. These tendencies are seen to enhance with increasing applied load and coating thickness [3].

Adhesion of the coating to the substrate is also an important factor with respect to wear protective properties. If the coating does not adhere well to the substrate the coating can easily delaminate, hence exposed substrate surface can increase. The adhesion properties of the coating to the substrate are a function of the coating type and surface cleanliness. Also high-energy ion bombardment and heating of the coated surfaces may cause diffusion of the metallic layer between the metal and the coating to the substrate, hence wear resistance of the substrate could be improved. Another possibility for the improvement of wear resistance is the deposition of inter layers and/or multilayered coatings. Such inter layers may be deposited by physical or electrochemical processes (PVD, ECD). [4].

In this study, the wear resistances properties of PVD-TiN coatings were investigated and the relations between wear resistance properties were studied.

II. EXPERIMENT DETAILS

The TiN coated samples were prepared by vacuum arc evaporation (VAE) process on a 304 SS substrate of dimension 10x10x60mm. The samples were polished as per requirement of VAE process standards. The coating thickness of the sample was analyzed using Fischerscope and adhesion was ensured by indentation method.

The hardness of the coating layer was compared with that of substrate by Nano indentation method. In nano indentation test, the properties of the film is measured without removing the film form the substrate. Indentation hardness is the mean contact pressure of the contact, and is found by dividing the indenter load by the projected area of the contact. The mean contact pressure, when determined under conditions of a fully developed plastic zone, usually defined as the "indentation hardness: HIT of the material. The displacement of the intender is measured and the size of the contact area (at full load) is estimated from the depth of penetration with the known geometry of the indenter. In the depth sensing indentation technique used in nano indentation, the elastic modulus of the specimen determined from the slope of the unloading of the load-displacement response. The modulus measured by this method called "indentation modulus" Err. The Berkovich indenter is used for testing. The mean contact pressure is usually determined from a measure of the contact depth of penetration hc, such that the projected area of the contact is given by:

$$A = 3\sqrt{3}h_c^2 \tan^2\theta \tag{1}$$

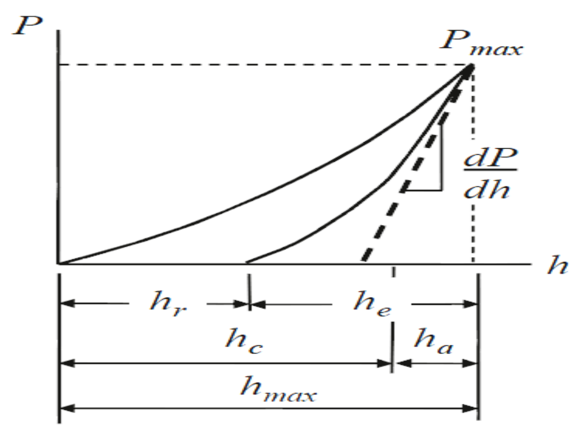
which for θ = 65.27°, evaluates to:

$$A = 24.494\, h_c^2 \tag{2}$$

and hence the mean contact pressure, or hardness, is:

$$H = \frac{P}{24.5\, h_c^2} \tag{3}$$

The principal goal of nano indentation testing is to extract elastic modulus and hardness of the specimen material from experimental readings of indenter load and depth of penetration. Force and depth of penetration are recorded as load is applied from zero to some maximum and then from maximum force back to zero. Plastic deformation occurs, then there is a residual impression left in the surface of the specimen. The size (and hence the projected contact area) of the residual impression for nano indentation testing is too small to measure accurately with optical techniques. The depth of penetration together with the known geometry of the indenter provides an indirect measure of the area of contact at full load, from which the mean contact pressure, and thus hardness, may be estimated. When load is removed from the indenter, the material attempts to regain its original shape, but it prevented from doing so because of plastic deformation. An analysis of the initial portion of this elastic unloading response gives an estimate of the elastic modulus of the indented material.



For a Berkovich indenter:

$$h = \sqrt{P}\left[(3\sqrt{3}H\tan^2\theta)^{-\frac{1}{2}} + \left[\frac{2(\pi-2)}{\pi}\right]\frac{\sqrt{H\pi}}{2\beta E^*}\right] \tag{4}$$

Upon elastic unloading:

$$h = \sqrt{P}\left(\frac{\pi}{2E^*}\right)^{\frac{1}{2}}\left(\frac{\pi}{3\sqrt{3}}\right)^{\frac{1}{4}}\frac{1}{\tan\theta'} \tag{5}$$

where in Eqs. (4) and (5) the quantities R′ and θ ′ are the combined radii and angle of the indenter and the shape of the residual impression in the specimen surface. The indentation modulus is usually determined from the slope of the unloading curve at maximum load. Equation (6) shows that the indentation modulus (here expressed as E*) as a function of dP/dh and the area of contact [10]

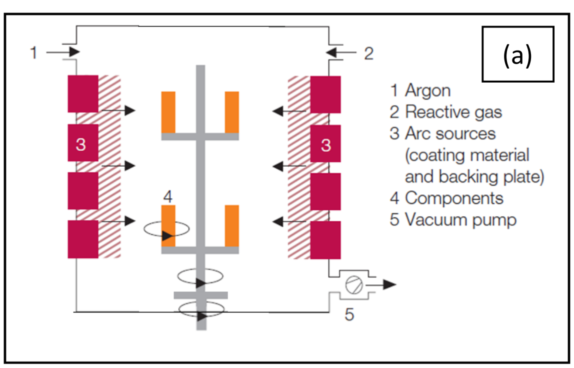
$$E^* = \frac{1}{2}\frac{\sqrt{\pi}}{\sqrt{A}}\frac{dP}{dh} \tag{6}$$

The coated samples were inspected using SEM and TEM analysis for coating morphological study and compositional analysis. The wear properties of coated and uncoated samples were investigated using pin-on-disc testing method. Wear experiment parameters are described in Table 2.1

TABLE 2.1 WEAR EXPERIMENT PARAMETERS

Parameters	Normal 304 SS	TiN Coated 304 SS
Sliding Velocity (m/s)	0.4	0.4
Sliding Distance (m)	200	200
Applied load (N)	20	20
Track Diameter (mm)	80	80
Experiment Duration (s)	500	500
Disc Speed (rpm)	96	96

. The wear test is conducted in Pin-on-disc wear test rig. The disc diameter is 80mm. The testing is conducted under constant load of 20N and constant speed of 0.4m/s i.e. 96rpm and for a distance of 200m. The sliding time was 8.3s. Since the hardness of TiN is much higher than the steel disc used on the Pin-on-disc testing equipment an alternate method was designed. A P600 grade electro coated silicon carbide emery sheet was cut to the disc's diameter and placed on the top surface of the disc, so that the wear takes place between the emery sheet surface and tested sample.



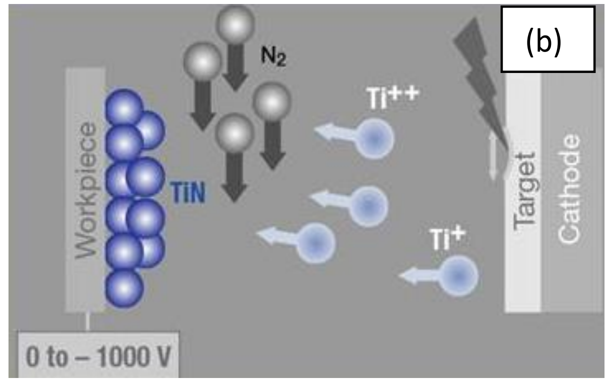


Fig. 2 (a) Schematic diagram of PVD vacuum arc evaporation technique, (b) Schematic representation of VAE PVD mechanism.

III. Results and discussion

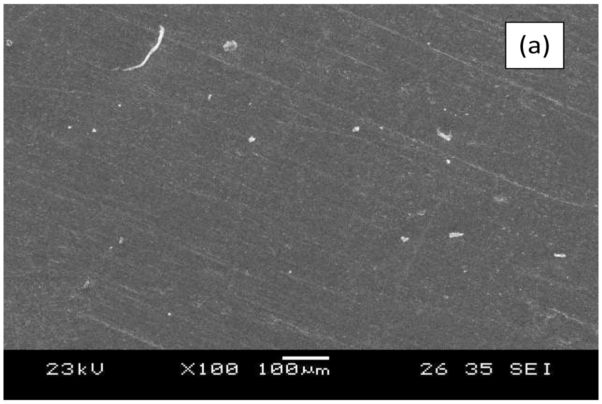
A. Coating characterization

The coating thickness was checked using a Fisherscope and found to be in the range 1.9µm. A coating thickness of 2µm has been found to give the best tool life in steel tools [5]. The coating adhesion test was done by indentation method and was found good. The hardness of the VAE PVD applied coating layer was checked using Nano-indentation method. And the results are as furnished in table 3.1

TABLE.3.1 NANO INDENTATION TEST RESULTS

Max Depth (nm)	Load (Nm)	Hardness (Gpa)	Young's modulus (Gpa)
69.68	2.86	14.9	243.71
111.85	5.85	11.64	224.79
146.7	8.89	13.24	181.89
194.5	11.74	11.25	174.28
222.84	18	15.28	183.04
334.87	26.25	12.44	149.85
337.76	28.65	11.51	193.22
449.51	37.7	11.79	128.77
504.63	47.75	12.91	122.58
521.36	51.9	13.83	122.1

The average coating hardness found to be 12.879Gpa i.e. 1312 HV where as the substrate hardness is about 128.5HV.



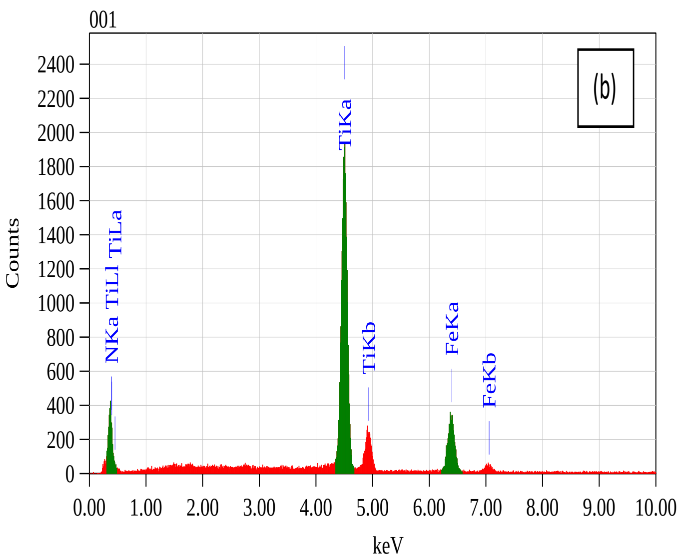
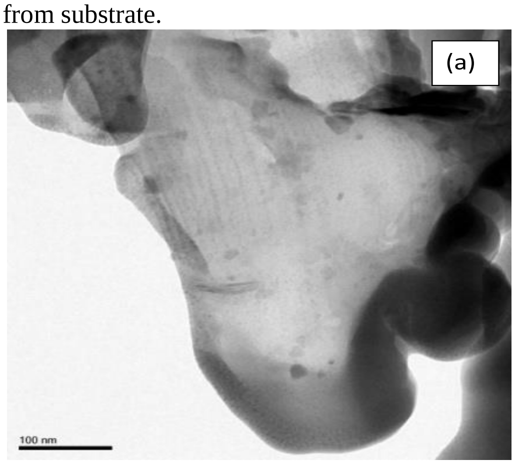


Fig.3.1 (a) SEM Image of TiN Coated Sample, (b) EDS MAPPING of Coated Sample

The image of SEM analysis (Fig.3.1a) shows a continuous film of coating on the substrate and the EDS graph ensures (Fig.3.1b) that the coating material is TiN. Since the film coating is in 2 micron range the EDS analysis shows the peaks of Iron from substrate.



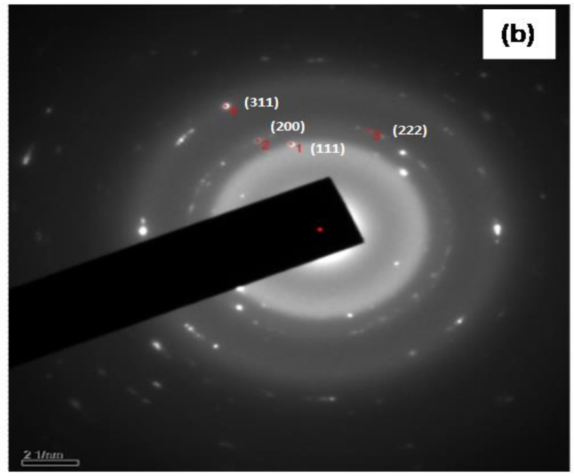


Fig: 3.2 (a) TEM Image of Coated Sample (b) SAD Pattern of TiN Coated Sample

TEM is an indispensable analytical tool in the study of the microstructure of coatings. Tem analysis results as shown in Fig 3.2 a & b provides a strong backing of coating continuity and it also gives an indication of mixed nature that is amorphous and crystalline nature of TiN. The d-Spacing indexing of SAD Pattern ensures the particle is TiN. Since Coating is done through VAE PVD Method, the TiN may be partially converted to amorphous in nature, which will improve the corrosion resistance.

B. **Wear studies**

The parameters as mentioned in Table 2.1 shows the setting parameters are same for both the TiN coated and uncoated 304 SS sample and run for the same duration of 8.3s. And consequently the graphs are generated between frictional force generated at wear interface and time.

Table 3.2 WEIGHT LOSS OF THE SAMPLES

Parameters	Uncoated 304 SS	TiN coated 304 SS
Initial weight (gm)	64.433	41.540
Final weight (gm)	64.407	41.529
Mass loss due to wear (gm)	0.026	0.011

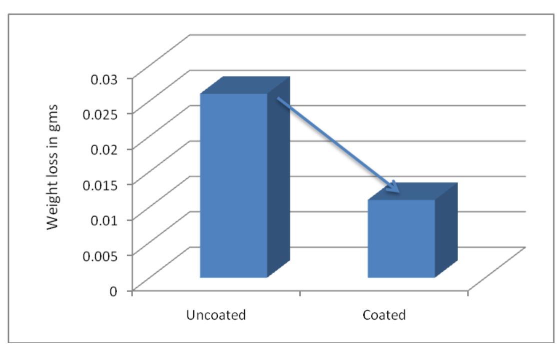


Fig 3.3 Weight loss of Coated and uncoated sample

From the Table 3.2 on the weight loss of the samples noted before and after the wear test and Fig 3.3, the better wear resistance of the coating layer is clear . The uncoated sample lost 0.026gm, where as the coating restricted the weight loss due to wear to 0.011gm which is only 42.3% of the uncoated sample which confirms the superior wear resistance with the coating.

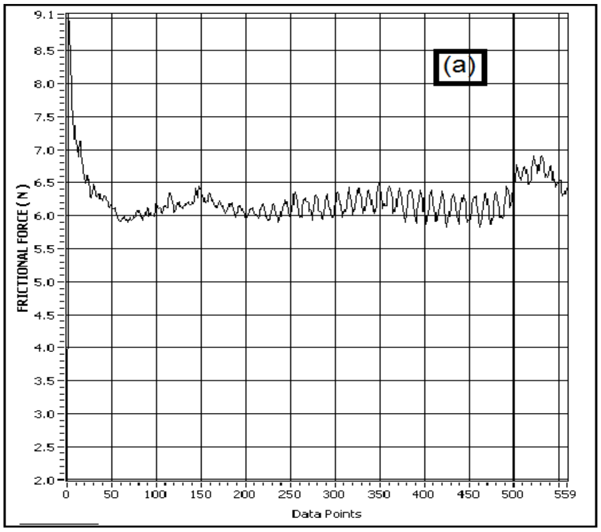


Fig 3.4 (a) Frictinal force generated versus Time for the Uncoated 304 SS sample

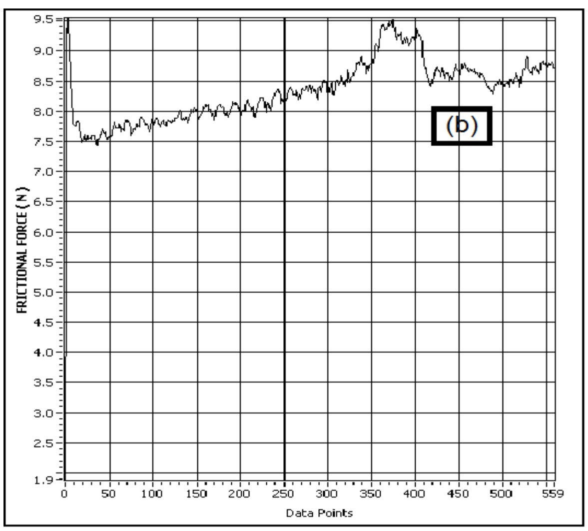


Fig 3.4 (b) Frictinal force generated versus Time for the TiN coated 304 SS sample

Fig 3.4 (a) & (b) shows the frictional force generated versus time during the test in dry condition. It was found that coefficient of friction values of uncoated and TiN coated specimens were similar. SS 304 substrate shows frictional coefficient about 0.785 and coated sample shows a frictional coefficient of 0.805, this is due to the higher hardness of the coating material than the wearing surface. The coated sample shows a higher frictional coefficient and is about tenfold harder than the substrate, even then the material loss is only 43.2% lesser than the uncoated substrate. And this will be much better in wet or lubricated condition, especially in engine components like piston ring and liner interface where there is hydrodynamic lubrication in affect. These results are similar to the results reported in references [5-8]. TiN coatings often yield decreased wear rates and, usually, reduced friction coefficients [9].

IV. Conclusion

According to the testings done on the TiN coating deposited on 304 stainless steel substrate using the vacuum arc evaporation PVD technique the conclusions are as follows:

- The coating thickness measured with Fischerscope showed the thickness to be 1.9µm which is the optimum coating where there is sliding friction.
- The Rockwell adhesion test gave good adhesion of coating layer to the substrate.
- The nano indentation test showed the TiN coating hardness to be 1312Hv which is tenfold harder than steel.
- The SEM and TEM analysis confirmed the coating layer material as TiN and the presence of 2 phases crystalline and amorphous which enhances corrosion resistances.
- The wear test shows a significant reduction in the wear rate with TiN ceramic thin film coating of thickness 2µm. The wear is reduced to 43.2%. it is be clear from the results that the coating will improve the wear resistance and thus it is recommended to be used on engine components susceptible to severe wear conditions.

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