Mechanical and Tribological Analysis of Nitrided AISI 4140 Steel With Coatings

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***Abstract*— The objectives of this paper is to systemically discuss the coating selection process, to develop a coating pre-selection tool and to propose some approaches for evaluating and comparing coatings, sequentially to help the selection of Tribological coatings.AISI 4140 is the most commonly used of the high tensile steels with a wide range of applications in automotive, gear and engine construction, crankshafts, steering knuckles, connecting rods, spindles, intermediate gears, pump and gear shafts. axles, nuts and bolts.This paper presents review of literature on Mechanical and Tribological Analysis of Nitrided AISI 4140 Steel. It is found that Coating is one of the most important and basic features of any components now a days. Because by applying the coating on the material the performance of the components increases. In present review work, various mechanical and tribological tests are conducted on AISI 4140 steel and same steel nitrided and coated with few low friction surface coatings such as CrN, TiAlN, DLC, TiC, AlCrN, AlTiN, TiCN,WCC etc. Based on the investigation, the base material is selected i.e AISI 4140. So from study and there outcomes it desire to coat the substrate with different coatings for improving its characteristics. And for getting more improvement in the subtrates they are nitrided. Various tests such as Microhardness test, Surface roughness, SEM, XRD, Scratch test, Pin on disk are being discussed. Coating is done by using PVD coatings, the common coating are CrN, TiAlN, DLC, TiC, AlCrN, AlTiN, TiCN,WCC etc.**

***Index Terms*— AISI 4140,PVD, DLC, XRD, Scratch test, Pin on disk**

I.INTRODUCTION

In Tribological applications, using coatings is an effective and relatively economical measure to reduce friction and protect the substrate surface from wear. However,selecting the appropriate coating for a given Tribological application is still difficult and complicated because the tribological response of a coating system depends on many factors (coating properties, counterpart, substrate, interface and running conditions). There is no general rule to help the coating selection.The use of coatings to improve the tribological properties of components such as tools for metal cutting and forming, and machine elements e.g. sliding bearings, seals and valves is constantly increasing. [1,2]

Additionally, many new deposition techniques and new coatings are being continuously developed, which provide a wide range for the selection of tribological coatings. At the same time, it also brings the coating users a problem: how to find the most suitable coating from numerous possibilities for a specific tribological application? The current method is trial and error, which is time-consuming and expensive, and some better coatings are probably missed.[1]

Tribology is the study of the thermal, mechanical and chemical interactions that occur when solid surfaces in or near contact move in relation with each other. A typical tribological system consists of three participating materials, namely, two solids in partial mechanical contact and a third material that can be a fluid or a solid, located in the intervening space between the contacting solids. Because of the multiplicity of processes encountered in tribological systems, and the many size scales involved, Tribology is necessarily an interdisciplinary area of study. Insights from the following disciplines are often essential for an understanding of tribological problems:

1. Physics and Chemistry
2. Materials Science and Engineering
3. Surface Science and Engineering
4. Continuum (Solid and Fluid) Thermo-Mechanics

1.1 NITRIDING

Nitriding is a thermo chemical surface treatment in which nitrogen is transferred from a media into the steel at temperatures completely within the ferrite and carbide phase field. After nitriding, a compound layer and an underlying diffusion zone (i.e. case) are formed at the surface of the steel.

Nitriding minimizes distortion and deformation of the heat treated parts. Therefore, nitriding is an important surface heat treatment for ferritic steels and can be widely used.[1,3]

1.2 COATING

Surface engineering, including surface treatments and coatings, is one of the most effective and flexible solutions for Tribological problems. Coatings change Tribological systems by inducing residual compressive stresses, decreasing the friction coefficient, increasing the surface hardness, altering the surface chemistry, changing the surface roughness. So, they improve the wear resistance of surfaces and extend the lifetime of relevant components. During last several decades, numerous coatings and deposition methods have been

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successfully developed and used to reduce friction or/and to protect surfaces from damage in mechanical systems.The increasing use of coatings in Tribological applications is mainly based on the following reasons:

1. More and more scientists recognize that the surface is the most important part in many engineering components, and most failures have a relationship with the properties of the surface area.
2. Many other functionally important properties depend on the surface area, such as electronic, magnetic, optical, bio-compatible characteristics.
3. Higher and higher performance is required for mechanical components and tools, which cannot be realized just by selecting materials or improving structures. The use of coatings can improve the performance of surface regions for friction reduction, wear resistance, corrosion resistance and other functionally attributes; at the same time, the substrate remains its original properties, responsible for the strength and toughness [1,3].

Mechanical and Tribological Analysis of Nitrided AISI 4140 Steel With Coatings and the methodologies employed to measure toughness for thin films fall into one of these methods: bending, buckling, scratching, indentation, and tensile tests [10,13].

Tribological coatings is to obtain an increased lifetime. There are, however, several other positive effects.

1. The improved wear resistance of coated metal cutting tools is usually utilized to increase the cutting speed and thereby the productivity, rather than to give a prolonged tool life.

2. Reduced friction often means reduced energy consumption. In some cases, a lowered friction may permit the exclusion of lubrication or of cooling stages.

3. Increased or controlled friction may be a beneficial effect in other applications such as brakes, bolted joints and safety connectors.

4. Reduced tendency to sticking and material pick up from the counter surface is crucial to the performance of forming tools and many sliding applications. Anti-sticking agents may be omitted in forming tool applications.

5. Components of reduced weight can be designed by application of coatings. Reduced weight means e.g. an increased ratio of power to weight of car engines, which in turn may give lower fuel consumption.

An important benefit of PVD and CVD processes is the high flexibility as to composition and structure of the coatings [5,10].

II. LITERATURE REVIEW

Podgornik, J [1] et.al in their research presented, samples made of AISI 4140 steel pre-treated with plasma nitriding and coated with different PVD coatings (TiN, TiAlN and ta-C) were investigated in terms of their microhardness, surface roughness, scratch adhesion and dry sliding wear resistance. Wear tests, in which duplex-treated pins were mated to hardened ball bearing steel discs, were performed with a pin-on-disc machine. To examine the influence of the nitrided zone on the performance of the coating–substrate composite, coatings were deposited on hardened as well as on plasma nitrided samples, prepared under different nitriding conditions. The results of the investigation showed improvedmechanical and wear properties of the plasma nitrided hard-coated specimens compared to the uncoated and pre-hardened ones. Furthermore, the compound layer was found to act as an intermediate hard layer leading to superior sliding wear properties of the composite.

S.Hogmark [2] et.al in their research presented tribological design considerations of coating composites and recommended methods and techniques for their evaluation. Current design concepts of coatings for tools and machine elements are exemplified together with successfully coated components. Techniques for evaluation of some of the more important fundamental and tribological properties will be presented and several examples are given mainly with thin physical vapour deposition (PVD) coatings. The evaluation techniques can be utilized both in coatings selection and in development of new coatings.

W.C. Oliver, G.M. Pharr [3] carefully documented the load displacement behavior of six materials by using Berkovich indentor to develop an improved technique for determining the hardness and elastic modulus from indentation load displacement data. It is shown that the load displacement curves during unloading in these materials are not linear, even in initial stages thereby suggesting that flat punch approximation used so often in analysis of unloading data is not entirely adequate. An analysis technique is presented that accounts for the curvature in unloading data and provides a physically justifiable procedure for determining the depth which should be used in conjunction with the indenter shape function to establish the contact area at the peak load. The hardness and elastic modulus of six materials was computed using the analysis procedure and compared with values determined by independent means to access the accuracy of method.

B. Podgornik and S. Hogmark [4] reported in the paper entitled“Tribological properties of plasma nitrided and hard coated AISI 4140 steel” that samples made of AISI 4140 steel pre-treated with plasma nitriding and coated with different PVD coatings (TiN, TiAlN and ta-C) were investigated in terms of their microhardness, surface roughness, scratch adhesion and dry sliding wear resistance. Wear tests, in which duplex-treated pins were mated to hardened ball bearing steel discs, were performed with a pin-on-disc machine. To examine the influence of the nitrided zone on the performance of the coating–substrate composite, coatings were deposited on hardened as well as on plasma nitrided samples, prepared under different nitriding conditions. The results of the investigation showed improvedmechanical and wear properties of the plasma nitrided hard-coated specimens compared to the uncoated and pre-hardened ones. The basic requirements for satisfactory performance of acoated component are sufficient coating–substrate adhesion and the ability of the substrate to support the coating.

Sam Zhang, Deen Sun [5] stated that there is neither standard test procedure nor standard methodology for assessment of toughness of thin films. However, researchers have long been trying to make such measurements, thus a spectrum of test methods have been developed, mostly each in its ownway. As qualitative or semiquantitative assessment, a simple plasticity measurement or scratch adhesion test can mostly suffice. For quantitative description, however, a choice of bending, buckling, indentation, scratching, or tensile test has to be made. These testing methods are either stress-based or energy-based. This paper gives a critical review on these methods and concludes that, for thin films, the energy based approach, especially the one independent of substrate, is more advantageous.

Erdman N. et al. [06]explained in the paper entitled "Multispectral imaging in an FEG-SEM" that in the field emission gun scanning electron microscope (FEG-SEM) the function of gun is to produce extremely narrow beam of electrons of precisely controlled energy. It is called "field emission" gun because an electric field causes high energy electrons to be emitted from a tungsten filament emitter having a point as small as 10 nm. State of the art field emission guns feature a Schottky emitter, which is made up of tungsten filament with a tip as small as 10 nm coated with thin layer of zirconium oxide to raise electrical conductivity.

Escobar C. et al. [07] explained in paper entitled "Improve on Corrosion Resistant Surface for AISI 4140 Steel Coated with VN and HfN Single Layer Films" that to improve the corrosion resistance of different industrials application, we studied vanadium nitride (VN) and hafnium nitride (HfN) single layer thin film, which were deposited onto Si (100) and AISI 4140 steel substrates via r.f. magnetron sputtering technique in Ar/N2 atmosphere with purity at 99.99% for both V and Hf metallic targets. Both films were approximately 1.2 ± 0.1 μm thick. The crystallography structures that were evaluated via X-ray diffraction analysis (XRD) showed preferential orientations in the Bragg planes VN (200) and HfN (111). The chemical compositions for both films were characterized by EDX. Atomic Force Microscopy (AFM) was used to study the morphology; the results.

Tlili Brahim et al.[08] explained in paper entitled "Hardness and scratch response of PVD multilayer coatings" that Cr/CrN, CrN/CrAlN and Cr/CrN/CrAlN multilayered coatings thin films have been developed by dual RF magnetron sputtering. They consist of superposing Cr, CrN and CrAlN layers of 50–500nm thick, up to a total thickness of 0.45–1μm. These coatings were grown on AISI4140 steel samples. The mechanical properties of these coatings were studied by scratch-tests and nano-indentation measurements. The hardness of the films reaches 15.8 GPa for a Cr/CrN multilayered coating sputtered at a bias voltage of -900V. High peak load tests were used to estimate the film adhesion on steel substrates; critical loads (Lc2) of 11N showed weak adhesion properties of the film. Moreover, an inventory of the major scratch-testsfailure modes was established, which were classified into plastic deformation and different forms of cracking,spallation and coating perforation events. No evidence of interfacial failure(s)of the sub-layers was observed after the adhesion and nanoindentation tests. Weak adhesion of the coatings deposited to thesubstrate should be connected with the existence ofthe interface and the difference in surface roughnessfrom the pure metal and the PVD coatings.

Singh Shiva Kumar et al. [09] have explained in the paper entitled "Exploring the Superconductors with Scanning Electron Microscopy (SEM)" that Scanning electron microscopy (SEM) is one of these characterization techniques, whose data is used to estimate the properties, determine the shortcomings and hence improve the material. SEMhas been widely used to explore the superconductingbehavior. Grain size matters a lot in deciding the superconducting parameters of cuprate HTSc. Some of the important aspects with which SEM deals with, is the grain size, morphology and alignment, structural defects, chemical composition. With the time ways to use SEM and to extract information from superconductors got improved. Earlier for HTSc's it was used simply in finding grain size, grain connectivity and to figure out impurity regions.

Fayomi O. S. I et al. [10] explained in the paper entitled "An Investigation of the Properties of Zn Coated Mild Steel" that zinc coatings on steel are rather unending because of the unique properties and the very low cost that it offers. The mechanical (wear and hardness) and corrosion behaviours of Zn Coated Mild Steel in 3.65% NaCl are described. A thin film of Zn on steel substrates was prepared by electro deposition technique using Zn particles to form a bath plating solution. Scanning electron microscope and Atomic force microscope were used to study the surface morphology, the topography and the surface adherent properties of the coatings. The crystal particles present were observed by X-ray diffraction pattern (XRD) and energy dispersive X-ray diffraction spectrometer (EDS). The uniform deposits of Zn showed fine grains and good protection against corrosion as appreciated 75% hardness value was achieve The average increase in microhardness of the mild steel substrate was 55 HVN to deposited sample Zn 3 was 108 HVN, about twice the microhardness value of the mild steel was achieved. The corrosion resistance of mild steel was improved after zinc deposition.

III.PROBLEM DEFINITION

A.Problem Statement

. There is growing demand for low friction coatings such as TiN, TiAlN, CrN, WC, DLC, etc. that allows coating surfaces to rub against each other with reduced friction and wear. The use of low friction coating is to improve the Tribological properties of tools for metal cutting, forming and machine elements. It is therefore important to do research on materials that have low friction so that they can be used as surface coatings. Therefore the present study aims to evaluate the Mechanical, Tribological and Microstructural properties of TiN, AlCrN coatings on Nitrided and Non-nitrided AISI4140.

B.Objectives

1. To find the mechanical properties of coatings such as hardness, impact strength and surface roughness.
2. To conduct tribological tests to find the scratch resistance, wear resistance.
3. To perform the microstructural analysis.
4. To conduct the comparative analysis of properties for various specimens coated with different types of coatings.

C.Methodology

1. Selection of substrate material for experiment.
2. Selection of coatings to be coated on substrate.
3. Preparation of substrate samples for each test as per the given standards.
4. Nitriding and Coatings application on prepared substrate samples.
4. Carrying out various Mechanical Tests such as Hardness and surface profiliometry.
8. Carrying out Scratch test and wear test to find the tribological properties.
9. Carrying out microstructural analysis of weared samples.
10. Carrying out comparative analysis.
11. Report completion and other activities.

D.Coatings Selection

The TiN and AlCrN PVD coatings with approximately 4µm thickness were applied on AISI 4140 and nitrided AISI 4140. The coatings were applied at Oerlikon Balzer's Coatings Pvt. Ltd. Pune. Some of the coating characteristics given by Oerlikon Balzer's are shown in Table

TABLE 1.
COATING CHARACTERISTICS GIVEN BY OERLIKON BALZER'S

Coating	TiCN	TiAlN
Colour	Blue-grey	Violet-grey
Coating temperature (^{o}C)	450	450
Maximum service temperature	400	900
Structure	Multilayr,graded	Nano-structured

IV.EXPERIMENTAL

A.Microhardness

Hardness is the property of a material that enables it to resist plastic deformation, usually by penetration. Microhardness test was carried out using Vickers microhardness tester as shown in fig.1 using ASTM E10-01e1.[18]

Fig.1. Vickers Microhardness Tester. (Make-Reichert Austria. Load -100 grams)

B.Sample preparation

Samples prepared for microhardness testing were of size, diameter= 30mm and thickness= 18mm with both surfaces flat and polished to 0.4µm. Three samples were salt bath nitrided at temperature around 450oC and two of these were PVD coated and one AISI 4140 without nitrided and one with nitriding was prepared.

C.Coating Thickness

Thickness of coatings was calculated using X-Ray Fischeroscope at Oerilikon Balzer's Coating Pvt. Ltd.

Sample Details: Samples used for measurement of coating thickness were each of length 38mm and diameter 13mm.

D.Surface Topography

A quantitative complimentary method to examine the surface morphology is obtained by analysing the surface roughness using surface profilometry. The surface roughness test was carried out using Mitutoyo SJ-210 Surface roughness tester shown in fig.2

Fig. 2. Mitutoyo SJ-210 Surface roughness tester

Sample preparation:

For surface roughness testing two square rods of dimension 204mm*24mm*11mm of AISI 4140 were prepared and the surfaces were extremely smooth finished with Ra approximately 0.4µm. The specimens were salt bath nitrided and after nitriding they were coated with TiN and AlCrN respectively.

a.TiCN coated nitrided AISI4140

b. TiAlN coated nitrided AISI4140
Fig.3 a.TiCN b. TiAlN. coated nitrided AISI4140

D.Friction and wear test:

A pin on disc tribometer is the equipment used to determine the sliding friction coefficient and wear resistance of surfaces. Pin-on disc friction and wear monitor TR-20LE-PHM-400 shown in fig.3 was used to carry out tests.

Fig.4 Pin on Disc apparatus

Specimen Preparation:

Wear testing requires two materials one for the pin and other for the disc. The typical pin specimen is cylindrical or spherical in shape. Typical cylindrical or spherical pin specimen diameters range from 2 to 10 mm. The typical disk specimen diameters range from 30 to 100 mm and have a thickness in the range of 2 to 10 mm.

The cylindrical pins are made of AISI 4140 material. The pins are made of diameter 6mm and length 30mm. Both the ends of the pins are made perfectly flat with surface roughness Ra less than 0.4µm. The pins are salt bath nitrided and the PVD coated with the coating thickness of approximately 4µm. Two pins of AISI4140 are directly coated. The Disk is made up of EN 31 with 60 HRC having diameter 165 mm and thickness of 8 mm which was electroplated. Test is carried as per ASTM G-99 04. [19]

E.Scratch Resistance Test.

Scratch test was carried on a scratch resistance test rig as shown in fig. below. A simple 500g load was applied on a diamond tip indenter which rests on the specimen and specimen is pulled gently so that the specimen is been scratched.

Sample preparation:

Samples used were same as those used for surface roughness measurement test.

V.RESULTS AND DISCUSSIONS

A. Hardness:

TABLE 2
Microhardness (HV)

Sample	Hv
AISI 4140	278
AISI 4140+ Nitriding	692
AISI 4140+ Nitriding+ TiCN	1013
AISI 4140+ Nitriding+TiAlN	1523

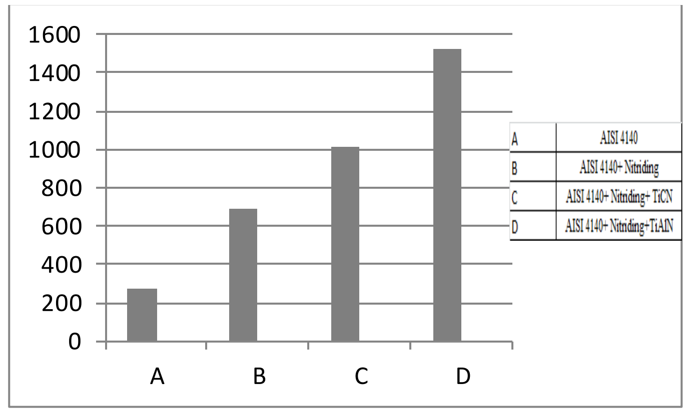

Fig.5 Microhardness Test Result

Vickers microhardness test performed on coated specimens, showed improved composite hardness obtained by nitriding of the substrate. This was observed for both coatings.(fig.) By increasing the hardness of the substrate by nitriding, it improves the load carrying capacity of the substrate and provides good support for the coating as well as reduces the large hardness gradients at the coating-substrate interface. AISI 4140 nitrided and coated with TiAlN showed the highest hardness which is nearly about six times the hardness of only substrate i.e AISI 4140. So TiAlN coating is preferable.

B. Coating thickness:

Coating thickness for both the coatings was found to be nearly same and was approximated to 4 microns.

TABLE 3
COATING THICKNESS

Coating	TiCN (µm)	TiAlN (µm)
n1	4.444	6.402
n2	4.539	6.611
n3	4.490	6.647
n4	4.481	6.176
Mean	4.489	6.474
Std. deviation	0.039	0.214

C. Surface roughness:

TABLE 4
SURFACE ROUGHNESS

SAMPLE	READING 1	READING 2	AVERAGE
TiCN Coated	0.787	0.727	0.757
TiAlN Coated	0.652	0.601	0.626

Stylus profilometry was used to measure the change in the surface roughness caused by nitriding and by the coating deposition. After nitriding, the average roughness value Ra of the original ground surface increased.

D. Friction and wear test:

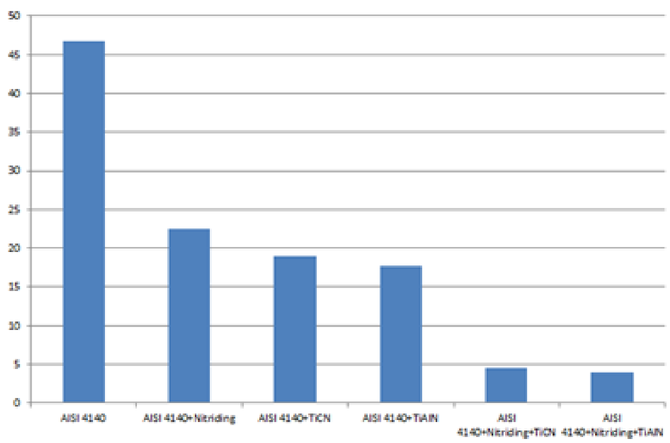

Fig.6 Wear(µm) of Samples

From fig.6 it is observed that for nitride samples wear rate is decreased drastically then Substate sample. Wear of AISI 4140 was 46.77 and it reduced to 3.93 for Nitrided AISI 4140 with TiAlN coating.

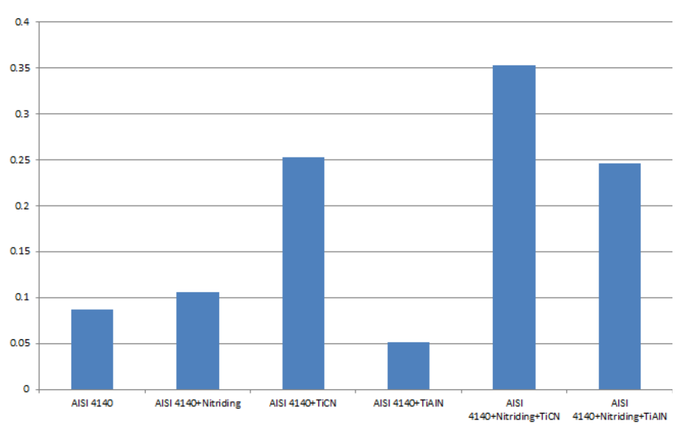

Fig.7CoF of Samples

From fig.7 it is observed that Cof of nitride subsrate with TiAlN or TiCN coatings increased. But the least Cof is preferable. So the increase of CoF is not much so the increase can be neglected over the increase of other properties.

E.Scratch resistance:

Scratch marks were observed for both the coatings but scratch haven't penetrated upto the bear material.

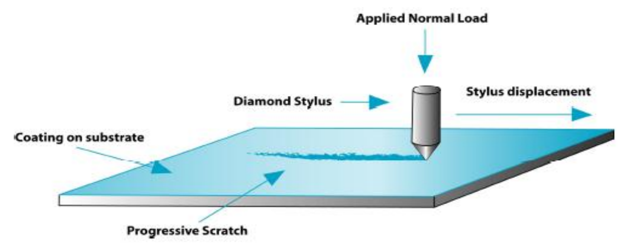

Fig.7 Principle of Sratch test

CONCLUSION

Thus, from the literature TiAlN, TiN, TiCN,AlCrN,DLCcoated on nitrided AISI 4140 steel proved as best coating. Hence, TiAlN and TiCN can be best suited for coating applications than conventional coatings.TiAlN and TiCN can resist high temperatures and is extremely hard.It is seen that AISI 4140 steel gives highest wear rate while AISI 4140 steel with nitriding and TiAlN coating gives lowest wear rate. TiAlN TiCN coated on AISI 4140 steel proved to be the best coating among selected coatings for further study.Thus by watching the above parameters the Substrate material with nitriding,and coating has good mechanical and Tribological properties also it has good machinability so they can be used for forging tools.By providing above coatings on nitrided AISI 4140 40%-50% of its life is increased. From the Microhardness results it is observed that, by applying coating and nitriding the surface hardness increase drastically.AISI 4140 Nitrided and coated with TiAlN showed the highest hardness which is nearly about six times the hardness of only substrate.From the Wear test TiAlN shows the less Wear then other substrate.For all the coatings "Scratch mark observed but not penetrated upto the bear material".

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