Journal of Materials Science & Surface Engineering, 5(5): 627-632
ISSN (Online): 2348-8956; 10.jmsse/2348-8956/5-5.3

Investigation of Tribological Behaviour of Nitrided and Coated AISI 4140 Steel

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ABSTRACT

In the present study, samples made of AISI 4140 steel, pre-treated with plasma nitriding (PN) and coated with coatings like Titanium Nitride (TiN), Titanium Carbo Nitride (TiCN), Chromium Nitride (CrN), Aluminium Titanium Nitride (AlTiN) using Physical Vapour Deposition (PVD) technique, were investigated in terms of their dry and wet sliding wear behaviour. Wear tests, were performed with a pin-on-disc machine. The results of the duplex treated samples were compared with the conventional hard chrome coated AISI 4140 steel.

The results showed improved wear properties of the duplex-treated specimens compared to the hard chrome coated AISI 4140 steel. TiCN coated and nitrided 4140 steel has shown the best performance among the investigated materials. Furthermore, the compound layer formed during nitriding was found to act as an intermediate hard layer leading to superior sliding wear properties. The improved performance of the duplex treated samples can be attributed to the presence of a nitrided subsurface.

ARTICLE HISTORY
Received 24-05-2017
Revised 27-06-2017
Accepted 04-07-2017
Published 27-07-2017

KEYWORDS
Adhesion
Duplex Treatment
Plasma Nitriding
PVD
Tribology
Wear

Introduction

AISI 4140 steel is extensively used in the manufacture of many different parts and components, which encompass conveyor rolls, hydraulic machinery shafts, connecting rods, hollow shafts, axles, forming dies, ejectors, crankshafts, trim dies, and guides. Components of forming tool dies such as draw ring, ejector pin use AISI 4140 as material for their manufacturing. The integrity of the die cutting tools is essential to accomplish adequate product quality. An improperly formed tool will bring about inferior product shape. The components of forming tool dies are generally subjected to tribological loads which are greatly influenced by the material being formed [1], and, ultimately subjected to friction and wear. Moreover, such tools are also subjected to the normal and tangential forces created by the system [2]. However, as-received AISI 4140 steel shows poor tribological properties such as poor wear resistance and unstable friction behaviour. It also has strong adhesion when it slides against itself and other metals [3]. Such parts require pretreatment before being put to service. One of the ways of modifying the surface of forming tool to improve its wear resistance is thermo-chemical surface treatment such as nitriding, carbonitriding etc. [4]. Surface designing, which may incorporate surface treatment, are by and large, utilized to enhance wear resistance of steel substrates by improving surface hardness and limiting surface adhesion leading to reduced friction [5]. One of the methodologies adopted by the industry to reduce wear and improve life of the part is the utilization of physical vapour deposition (PVD) technique [3]. PVD hard coatings are outstanding for furnishing surfaces with high surface hardness and enhanced tribological properties [8]. In any case, it is verifiable truth that the use of PVD hard coatings to the substrate materials does not really prompt to ideal tribological properties, if the substrate material does not experience reasonable pretreatment. It is due to plastic deformation of the substrate, which may result in coating failure [8]. The prime necessity to enhance the wear properties of the substance is the correct and solid attachment of the coat to the substrate. Moreover, the substrate must have an ability to support hard and brittle coating [2]. Presence of hard nitride layer on the lower surface accomplishes legitimate attachment of the coat to the substrate [5, 8]. Nitriding prior to coating deposition emphatically influences the development and properties of the hard coating [9]. Iron nitride formed in the hardened surface layer after plasma nitriding increases surface hardness substantially [10]. Moreover, nitriding could promote the development of compressive stresses in the surface of the substrate; therefore, lessening the difference in stress environments in the coating and steel substrate [9, 11]. This was supported by the fact that the best adhesion results were obtained in work pieces with a larger diffusion zone and a thicker compound layer [3]. However, in order to increase the shear properties of the resulting coat, development of the interface layer between the coat and the substrate may be useful. Zeghni and Hashmi [5] and Staia et al. [12] studied the beneficial effects of nitriding of tool steel prior to PVD coating and found improved hardness and wear resistance. The authors, Devaraju et al. [3] and Yildiz et al. [11] experimented with plasma nitrided AISI 316 LN stainless steel and found improved wear behaviour at elevated temperature and improved fatigue properties, respectively. The nature and the prevailing wear mechanisms of nitrided and coated tools had been the subject matter of interest in many investigations. To name a few; Yilbas and Nizam [9] and Nickel et al. [13] experimented with PN treated and TiN coated drills, and observed longer tool lives under all machining conditions. The improvement in machining performance of the pre-nitrided drills was attributed to the role of the plasma nitriding in improving coating adhesion, and, thus, increasing the wear resistance of the coating. Abrasive

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Contents lists available at http://www.jmsse.org/ & http://www.jmsse.in/

wear properties of steel reported to depend upon their microstructure, and chemical composition, as well [14].

In this context, in recent investigation, Prabakaran et al. [15] carried out study in order to analyze the effect of TiCN coated AISI 410 steel against high carbon steel and found reduction in friction coefficient and wear loss. The results reported in the literature [16] indicate that the friction and wear of TiCN coated tribo-system is mainly affected by hardness and thickness of the coating. TiCN-coated cutting tool inserts performed 9 times longer than uncoated inserts due to higher hardness and lower friction coefficient [17]. The main factors affecting tool performance being coating adhesion and substrate preparation, followed by coating wear resistance and toughness. Investigation conducted by Podgornik et al. [18] indicates that hard PVD coatings can successfully be used in blanking and piercing applications. Puchi-Cabrera et al. [19] found that SAE 4340 steel substrate coated with a TiCN film deposited by plasma assisted physical vapour deposition (PAPVD) resulted in significant increase in fatigue life. This increase in fatigue life has been attributed to high mechanical strength of the film, its compressive residual stress state, and excellent adhesion of the coat to the steel substrate. Zukerman et al. [20] concluded that the presence of the nitride layer and compound layer between the steel substrate and the coating acts as a diffusion barrier which ultimately helps maintain the wear resistance of the nitrided and TiCN or TiN/TiCN coated specimens. The sliding wear tests of AlCrN single-layer coatings, and TiAlN/AlCrN nano-multilayer coatings on cemented carbide disc, in dry sliding condition by W. Liew et al. [21] found to have improved behaviour. Chandrashekhar et al. [22] investigated the influence of carbon content of the substrate, and coating thickness on scratch and wear resistance of the AlCrN films, and reported an increase in adhesion force between the substrate and the coating with increasing carbon content of the substrate (EN 353 and EN 31). They further reported positive effect of coating thickness and carbon content of the substrate on the microhardness of the AlCrN-coated specimens. In another research conducted by Chandrashekhar et al. [23], wherein they compared performance of TiAlN and AlCrN-coated EN 353 steel, and showed that load carrying capacity of AlCrN coating was better than TiAlN coating. The compound hardness of both coatings increased with increase in coating thickness. Literature [24] discussed the high temperature wear behaviour of AlCrN coated Titanium alloy, and, results confirmed superior mechanical and tribological properties of the coated Titanium alloy as compared to the uncoated one due to the hard nature of AlCrN particles. Malarvannan et al. [25] have reported that TiN and AlCrN bilayer PVD coated high speed steel showed less amount of corrosion, in addition to the enhanced wear resistance, which was attributed to the dense microstructure and less porosity of the coating. Cadena et al. [26] conducted research in which a heat-treated monolayer coating of AlCrN was deposited on Tungsten Carbide substrate and results confirmed superior performance of the heat-treated monolayer coating, in comparison with a conventional monolayer AlCrN coating.

Components of forming tool dies are subjected to high thermal and mechanical loads due to the high temperature difference between the hot blank and the cold tool surfaces. Due to relative motion between the tool surface and the work piece, it has been quite often observed that tool surface leads to excessive wear in terms of adhesion on the tool surface [27]. One of the ways to reduce wear of components of forming tools is the application of coating on tool surface. Performance of coated tool is strongly affected by the coating-substrate adhesion. One of the methods of improving coating-substrate adhesion may be the inclusion of nitride diffusion layer in the substrate before the coating is initiated. Consequently, investigation into the mechanical and tribological properties of the resulting coated surface formed on the nitride layer becomes essential.

Evidently, to the best of the knowledge of the authors, influence of varying load and sliding speed on tribological characteristics of duplex treated AISI 4140 steel has not been observed systematically. Hence, the influence of varying load and sliding speed on tribological properties of duplex treated AISI 4140 steel coated with TiN, TiCN, CrN, and AlTiN coatings has been investigated in present work. In this work, an effort has been made in that direction. Further, the study has been motivated with an intention to investigate the applicability of AISI 4140 as forming tool material. Moreover, being conventional coating material, tribological performance of Hard Chrome (HC) coated AISI 4140 steed has also been investigated and compared with the above-mentioned materials. The present investigation has been conducted on AISI 4140 steel in five different conditions: plasma-nitrided and coated with TiN (N-TiN 4140), plasma-nitrided and coated with TiCN (N-TiCN 4140), plasma-nitrided and coated with CrN films (N-CrN 4140), plasma-nitrided and coated with AlTiN films (N-AlTiN 4140), and Hard Chrome coated AISI 4140 steel (HC-4140).

Experimental

Materials

The present investigation has been conducted with samples of an AISI 4140; its chemical composition is presented in Table 1.

Table 1: Chemical composition of AISI 4140 steel

Composition (wt. %)	C %	Si %	Mn %	P %	S %	Cr %	Mo %	Ni %
Specifications	0.35–0.45	0.10–0.35	0.45–0.70	0.05 Max	0.05 Max	0.90–1.40	0.20–0.35	1.30–1.80
Observed Values	0.381	0.225	0.656	0.021	0.006	0.947	0.217	1.345

This material was chosen in view of its ability to be nitrided without losing its toughness. The material was given as a bar, from which samples of 8 mm diameter and 30 mm height were cut. The ends of specimens were polished so as to achieve the surface roughness of approximately 0.4 µm, as indicated in the standard, ASTM G99-04 [28].

Preceding nitriding, the samples were polished with alumina, followed by cleaning with acetone. The nitriding of the specimens was performed by plasma nitriding (PN) process at ~540^{0}C in a 75%H_2-25%N_2 atmosphere, at a pressure of 450-500Pa, for ~16 hrs. The process parameters were selected based on the literature [2-3, 10, 13] which suggested that nitride results have direct relation with concentration of nitrogen, treatment temperature, and treatment time. Moreover, higher treatment temperature helps in achieving greater diffusivity of nitrogen into the surface [10].

Subsequent to nitriding, the specimens were machined and polished again in order to dispose off the white layer. Coatings were deposited on nitrided surfaces, with the help of PVD process, the technique developed by 'Oerilikon Balzers Coating Pvt. Ltd', at a substrate temperature of ~ 400⁰C to 450⁰C. A group of 5 samples each was coated with coatings such as TiN, TiCN, CrN, and AlTiN. In order to compare the sliding wear behaviour of these materials with the conventional coating material, hard chrome, a group of 5 samples of AISI 4140 was coated with hard chrome.

Response Measurements

Wear tests were carried out on duplex treated samples. The schematic of the experimental setup is shown in Fig. 1.

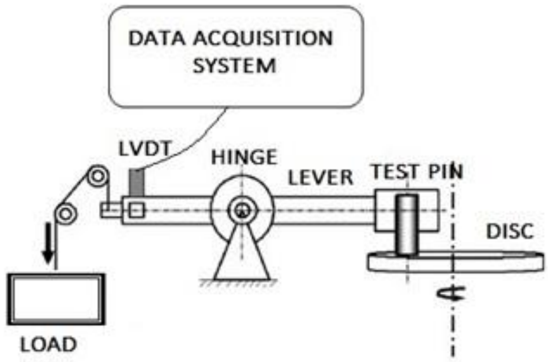


Figure 1: Schematic of Experimental Setup

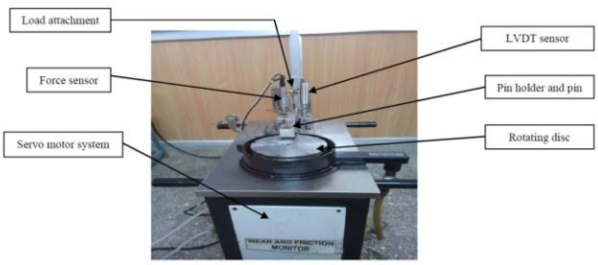


Figure 2: Actual Experimental Setup

Pin-on-disc friction and wear monitor TR-20LE-PHM400 of DUCOM make was employed in the study. The specimens were loaded normally and slid against an EN 8 steel disc having diameter of 160 mm and thickness of 8 mm, with hardness of ~60 HRC. In order to have accuracy in the results obtained, the sliding end of pin and disc surface were cleaned with acetone, and then dried before testing. Unlubricated wear tests with a sliding distance of ~2000 m were carried out at room temperature of ~27 ⁰C, a relative humidity of ~ 50 %, and at one atmosphere pressure. In order to study the effect of load on wear performance, the wear tests were conducted at normal load of 10 N and 20 N. Moreover, to study the effect of sliding speed on wear performance, the wear tests were conducted at 4.19 m/s and 5.23 m/s. The tests were conducted in dry (without lubrication) and in wet condition, in presence of lubricant, SAE 1040 oil. The response, wear track depth was measured in µm with the help of Linear Variable Differential Displacement Transducer (LVDT). The friction force, and hence, friction coefficient was determined with the help of a load cell available with the setup. The tests were conducted according to the ASTM G99-04 standard [28].

Scanning Electron Microscopy (SEM) techniques were used. Field Emission Gun-Scanning Electron Microscope (FEG-SEM) JSM-7600F was used to acquire the SEM micrographs of the cross sections of untreated and PN treated specimens. The PN treated specimen was chemically characterized using Energy Dispersive X-Ray analysis (EDX), where, the electron beam from the SEM serves to excite characteristic X-rays from the area of the specimen being probed. The actual experimental test setup is as shown in Fig. 2.

Results and Discussion

Wear Characterization of Duplex Treated 4140

A cross sectional SEM micrograph of untreated AISI 4140 and that of PN treated 4140 specimen is shown in Fig. 3a, and 3b, respectively. Fig 3a illustrates typical microstructure of AISI 4140 steel employed in the present work. Plasma nitriding of the steel substrate gave rise to the development of nitride layer. The cross sectional SEM micrograph (Fig. 3b) reveals that the average thickness of the PN layer which was measured with the help of image analysis technique, and, it was found to be ~45 µm. EDX Spectrum of cross-section of nitrided 4140 is presented in Fig. 3c which clearly indicates the presence of nitrogen. It shows the element composition of 83.25 at. % Fe and traces of Cr, Si, etc., in addition to the presence of nitrogen. Nitrogen observed on the surface supports the formation of Cr- and Fe- nitrides on the surface [10].

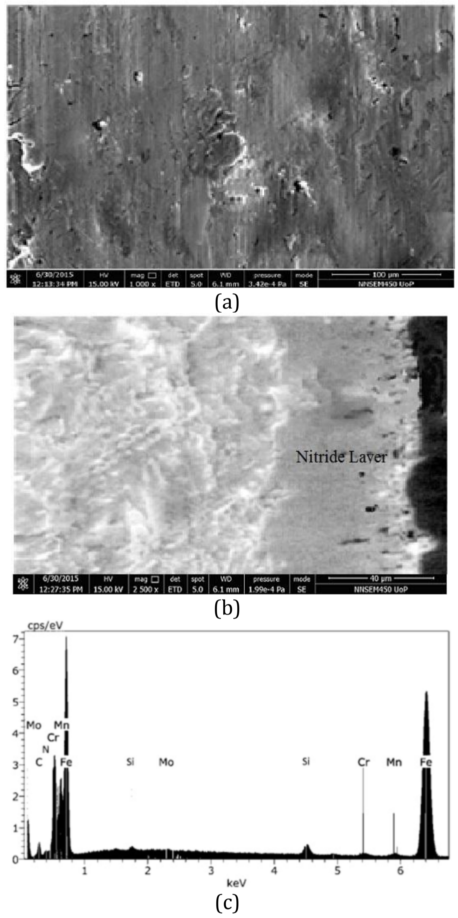
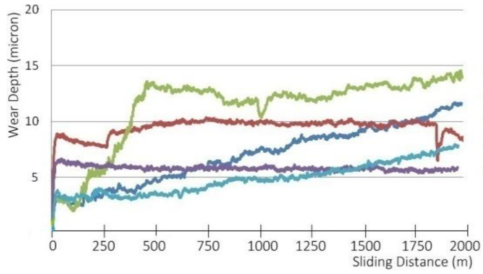


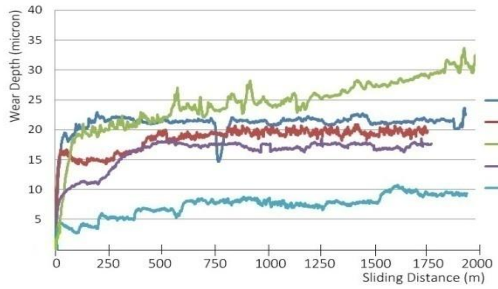
Figure 3: SEM micrograph of (a) cross-section of AISI 4140, (b) cross-section of nitrided 4140 and (c) EDX Spectrum of cross-section of nitrided 4140

Wear Characterization at Varying Load

In order to study the effect of normal load on wear characteristics, the tests were conducted with normal load of 10N and 20 N. Fig. 4a and 4b show variation of wear track depth in dry sliding condition at 10N and 20N, respectively, whereas, variation of friction coefficient at 10N and 20N load, in dry sliding condition, is depicted in Fig. 5a and 5b respectively.

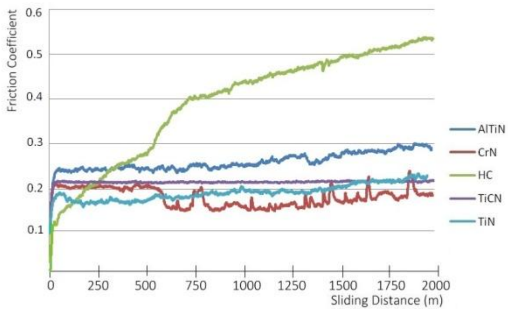


(a)

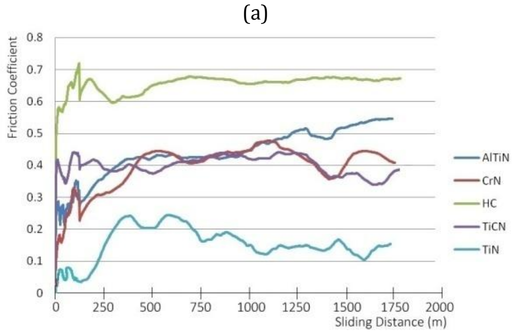


(b)

Figure 4: Variation of wear track depth in dry sliding condition at (a) 10N and (b) 20N load



(a)

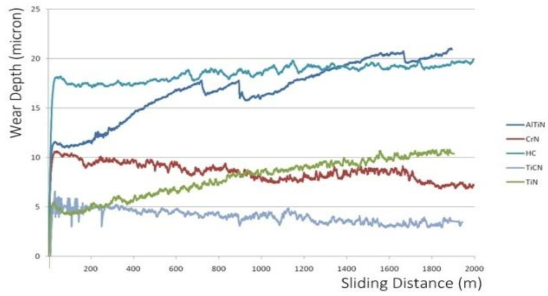


(b)

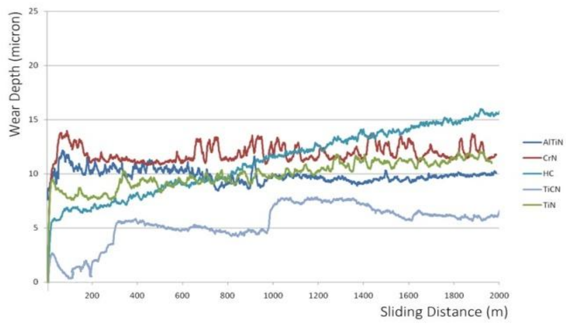
Figure 5: Variation of friction coefficient in dry sliding condition at (a) 10N and (b) 20N load

As evident from Fig. 4, wear track depth goes on increasing with increase in load. HC coated 4140 has shown sudden rise in wear track depth. It shows sudden increase in friction coefficient (refer Fig. 5a and 5b) at 250-300m of sliding distance, which is an indication of adhesion of work material to the tool surface [1-2, 4]. However, no such sudden rise is observed in case of duplex treated specimens. Among the duplex treated specimens, N-TiN 4140 has shown the lowest wear track depth among the materials under investigation. As load increases from 10N to 20N, the friction coefficient increases for all the materials. It is observed that for duplex treated specimens, at 10N load, the friction coefficient shows very constant value throughout the test. Its average value for duplex treated samples ranges between 0.15 and 0.25. N-TiCN 4140 has shown extremely constant friction coefficient of 0.22 throughout the test. However, as load increases from 10N to 20N, the friction coefficient increases rapidly. At 20N normal load, the average values of friction coefficient, for the entire duplex treated specimens, lie between 0.15 and 0.45, as against 0.65 for HC coated 4140. The oscillations observed in the friction curve [12] indicate that debris of both coating and counterpart is produced, which moves away from the contact area. For all the materials, at 20N load, the friction coefficient increases rapidly during first 200m displacement and thereafter, it remains fairly steady.

In order to study the effect of sliding speed on wear characteristics, the tests were conducted at 4.19 m/s and 5.23 m/s with normal load of 10N. Fig. 6a and 6b show variation of wear track depth in dry sliding condition at 4.19 m/s and 5.23 m/s, respectively. It has been observed that as sliding speed increases, wear track depth goes on reducing, for all the materials under investigation. Reduced wear is attributed to the reduced friction (refer Fig. 7 and Fig. 8) due to hydrodynamic squeeze film action, discussed later. It has been observed that N-TiCN 4140 has shown a very constant wear depth throughout the test.



(a)



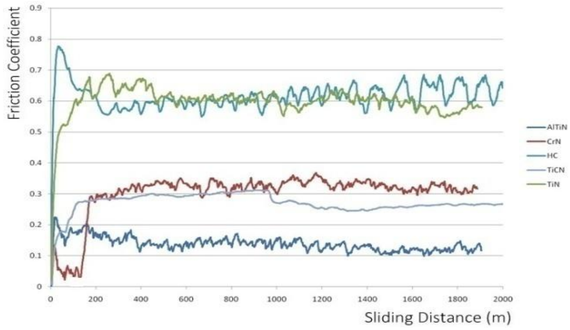
(b)

Figure 6: Variation of wear track depth in dry sliding condition at (a) 4.19 m/s and (b) 5.23 m/s

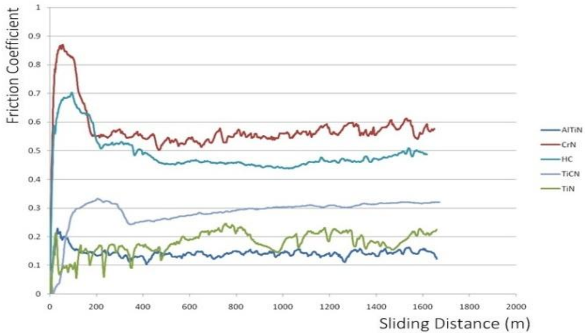
Friction Characterization in Dry and Wet Sliding Condition

Variation of friction coefficient with respect to sliding distance in dry and wet condition at sliding speed of 4.19

m/s and at 5.23 m/s are presented in Fig. 7 and Fig. 8, respectively.

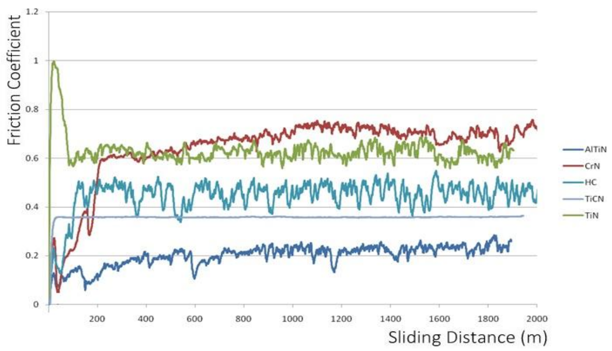


(a)

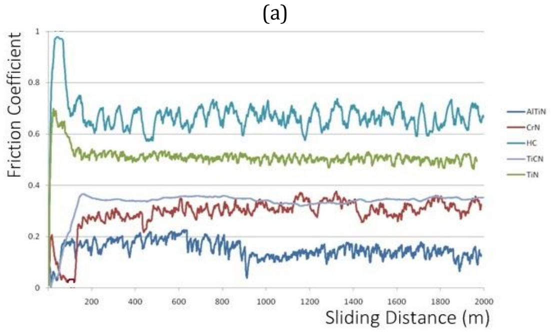


(b)

Figure 7: Variation of friction coefficient at 4.19 m/s in (a) dry and (b) wet sliding condition



(a)



(b)

Figure 8: Variation of friction coefficient at 5.23 m/s in (a) dry and (b) wet sliding condition

It has been observed that as sliding speed increases, friction coefficient goes on reducing. It is due to the fact that as speed of the disc increases, the radial and tangential components of the centrifugal force tend to draw more lubricant into the space between stationary pin and the rotating disc. This hydrodynamic squeeze film action is responsible for reduction in friction coefficient which ultimately leads to reduced wear. Among all the materials under investigation, N-TiCN 4140 has shown very constant and consistent value of friction coefficient under all the conditions of load and sliding speed. Low values of friction coefficient in case of duplex treated specimens are in good agreement with previous results [9]. At increased sliding speed, duplex treated specimens produce much better results than at lower sliding speed [3]. Basically, nitriding of steel prior to PVD coating improves wear resistance of steel [8]. In general, deposition of the coatings on PN treated specimens decreased the pin wear significantly. It may be noted that the wear of the investigated coatings decreases with increased substrate hardness due to nitriding. Compared to untreated substrates, the duplex treated specimens showed improved sliding wear resistance, which can mainly be attributed to the higher substrate hardness, and improved coating-substrate adhesion [19]. Adhesion of the coat to the substrate may be attributed to the presence of interface layer between the coat and the substrate [12, 14]. Increased wear resistance may be attributed to the formation of this layer which further leads to improved coating-substrate adhesion.

As discussed in 'Introduction', in the application of forming tools, the ability of the tool surface to avoid adhesion with work material and to provide a desired surface quality of the product is often equally important as the wear resistance of the tool. Therefore, hard abrasive resistant surfaces, with high friction and tendency to work material pick-up do not represent an optimum solution. In general, plasma nitriding of the substrate improves coating-substrate adhesion as well as load-carrying capacity and wear resistance of coated tool steel. Moreover, nitriding of tool steel prior to PVD coating results in improved hardness and wear resistance. PVD hard coatings have proved to be outstanding for furnishing surfaces with enhanced tribological properties.

Conclusions

TiN, TiCN, CrN and AlTiN coatings were deposited on plasma nitrided AISI 4140 steel, using PVD process. Also, conventional hard chrome was coated on untreated AISI 4140 steel and the tribological characteristics were studied, in detail. Following conclusions are drawn from the test results:

1. The wear experiments that have been conducted indicate that nitriding of 4140 steel prior to coating the steel substrate, deposited by PVD process, leads to improved friction and wear behaviour.
2. The nitriding process applied to the steel substrate was found to provide satisfactory load carrying support which leads to the improved tribological behaviour of the coated system.
3. Reduction in friction may be attributed to the long treatment duration that helps nitrogen penetrate deeper into the surface, which ultimately leads to the formation of thick nitride layer.
4. In case of TiCN coated tribo-system, the nitrided zone prior to coating improves coating-substrate adhesion, most probably through the inter-diffusion between iron nitride at the nitrided surface and thin Ti-interlayer deposited at the beginning of the coating process.

5. TiCN coated on PN treated 4140 has proved to be the best suitable coating in terms of steady friction behaviour, among the investigated coating materials.
6. All the materials under investigation have proved to be superior to the conventional hard chrome coating.
7. All the duplex treated systems have shown far better performance as compared to the conventional coating. Hence, they can be implemented for forming tool applications.

Limitations
Adhesion test was not conducted in the study.

Future Scope
Adhesion test may be employed to comment on the quality of adhesion of the coat to the substrate.

Acknowledgements
Authors would like to express sincere thanks to Dr. A.G. Thakur, Head, Mechanical Engineering Department and Dr. A.V. Damale, Lab In-charge of 'Materials Testing Lab' of Sanjivani College of Engineering, Kopargaon, MS, India for their cooperation in conduction of the tests. The authors declare that there is no conflict of interest regarding the publication of this paper.

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