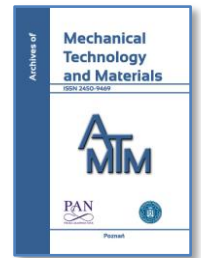


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Tribology of nitrided-coated steel-a review

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ABSTRACT

Surface engineering such as surface treatment, coating, and surface modification are employed to increase surface hardness, minimize adhesion, and hence, to reduce friction and improve resistance to wear. To have optimal tribological performance of Physical Vapor Deposition (PVD) hard coating to the substrate materials, pretreatment of the substrate materials is always advisable to avoid plastic deformation of the substrate, which may result in eventual coating failure. The surface treatment results in hardening of the substrate and increase in load support effect. Many approaches aim to improve the adhesion of the coatings onto the substrate and nitriding is the one of the best suitable options for the same. In addition to tribological properties, nitriding leads to improved corrosion resistance. Often corrosion resistance is better than that obtainable with other surface engineering processes such as hard-chrome and nickel plating. Ability of this layer to withstand thermal stresses gives stability which extends the surface life of tools and other components exposed to heat. Most importantly, the nitrogen picked-up by the diffusion layer increases the rotating-bending fatigue strength in components. The present article reviews mainly the tribological advancement of different nitrided-coated steels based on the types of coatings, structure, and the tribo-testing parameters, in recent years.

1. INTRODUCTION

Machining industry is constantly seeking ways to enhance performance (metal removal rate) and reduce cost of the manufactured parts. One way to enhance the machining performance is to utilize high speed machining. One of the problems associated with high speed machining is the high tool wear which leads to reduction in tool life [1-4]. This is essentially due to the existence of higher cutting temperature generated between the tool tip and the component interface. Higher cutting temperature can also enhance the chemical reactivity between tool and certain work piece materials such as Titanium (Ti). This can lead to higher chemical wear, thereby further reducing the tool life [5]. Lower tool life leads to frequent tool changes resulting in increased machine

down time. This in turn reduces the overall productivity. Hence, it is essential to minimize the tool wear & tear and increase the life. One of the techniques adopted by the industry to minimize wear and to improve is surface treatment and coating [4, 14].

The challenge to improve the properties of protective coatings by thermochemical pre-treatment of the substrate has gained much attention in recent years [7]. One of the approaches adopted by the industry to reduce wear and enhance tool life, and productivity is the use of physical vapour deposition (PVD) and it has been found that pre-nitriding of steel can provide substantial improvements in coating adhesion, which ultimately leads to improvement in tribological performance [1-3, 6].

Hard materials for coatings can be divided into single layer coatings and multilayer or multiphase coatings. Single layer coatings are further classified as:

Metallic Hard Materials - they include borides, carbides & nitrides of transition metals. The examples are TiN, TiC, CrN, WC, TiB₂ etc.

Covalent Hard Materials - they include borides, carbides & nitrides of Al, Si, B & diamond. The examples are SiC, Si₃N₄, AlN, AlB₁₂ etc.

Covalent Hard Materials - they include borides, carbides & nitrides of Al, Si, B & diamond. The examples are SiC, Si₃N₄, AlN, AlB₁₂ etc.

Luo et al. [10] presented a systematic way to select tribological coatings for a specific application. The method consists of a pre-selection tool which is based on database, followed by some simple tests such as nanoindentation test, scratch test etc. to screen the candidate coatings. They have also developed a flexible polar diagram which helps compare coatings from various aspects.

Optimum wear resistance can be achieved by multiphase or multilayer coatings. They seem to be the best compromise in complex requirements such as hardness & toughness, weak adhesion at the surface, and at the same time, good adherence at the substrate-layer boundary. Multilayer materials are used with mutual solubility. The examples include TiC & TiN, Al₂O₃ & AlN etc. Some multilayers are with coherent interfaces such as TiC or TiN & TiB₂ [9].

This article aims to review the research carried in this area, in recent years.

2. THE NITRIDING PROCESS

Nitriding is a heat treatment process which diffuses nitrogen into the surface of a metal to create a case hardened surface. It is predominantly used on steel, but also used with titanium, aluminium and molybdenum alloys. It is a means of imparting a very high surface hardness to steel components. As compared to other processes which offer this, nitriding offers certain advantages such as low process temperature (around 495°C to 535°C), a slow, controlled heating and cooling rate, clean and oxide-free finish to the finished product, ability to harden selected areas of the component, but of course, the entire component can be hardened, if preferred.

The three main methods used are: gas nitriding, salt bath nitriding, and plasma nitriding. The processes are named after the medium used to donate nitrogen.

3. MICROSTRUCTURAL CHARACTERIZATION

Devaraju et al. [3] investigated sliding wear behavior of plasma nitrided (PN) austenitic stainless steel AISI 316LN SS. X-Ray Diffraction (XRD) patterns of PN treated and untreated 316 LN SS sample are shown in Fig. 1a and 1b, respectively. The XRD patterns suggest that PN layer consists of CrN, Fe₄N, and Fe₃N phases along with austenite reflections from the substrate material. A small amount of ferrite (α) phase was also observed which was reported by other researchers, as well.

X-Ray Diffraction (XRD) patterns of the nitrided 316 L steel [6] show that the modified layer, as observed from the SEM micrograph of the cross-section of the nitrided specimen (Fig. 2), on the surface of the nitrided specimen appears to be

a homogenous layer separated from the bulk material by a clear line.

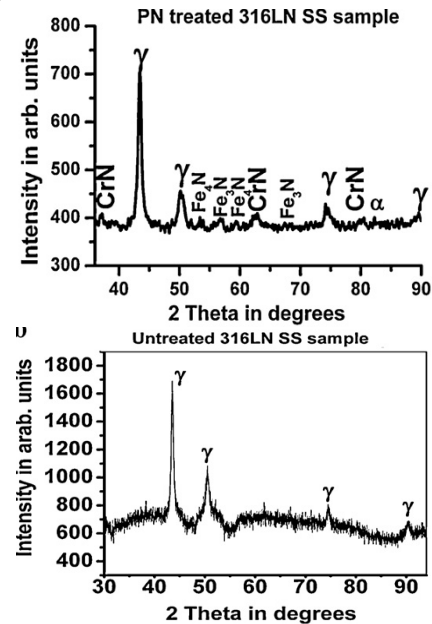


Fig. 1. XRD patterns of (a) PN treated and (b) untreated 316 LN SS [3]

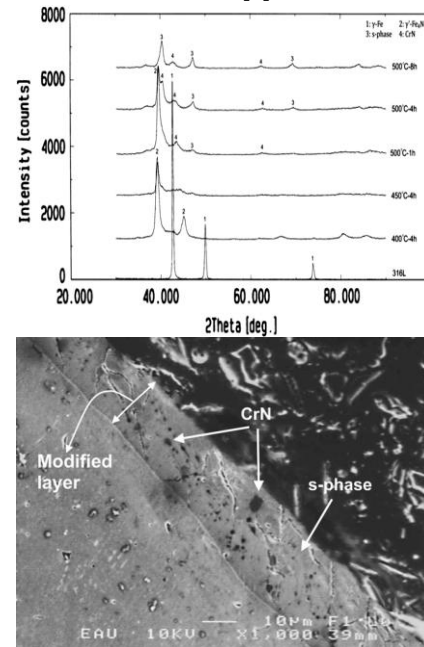


Fig. 2. a) The XRD results b) the SEM micrograph of cross section of the nitrided specimen at 5000C and for 1 hr. [6]

This layer is called as modified layer for austenitic stainless steel which consists of s-phase, CrN and γ -Fe₄N. The formation of s-phase is attributed to the diffusion of nitrogen and chromium atoms. XRD patterns further suggest that diffusion of nitrogen as an interstitial atom to the austenite lattice is greatly encouraged while the diffusion of chromium as a substitutional atom is prohibited at 4000C. The distortion of austenite lattice with the effect of nitrogen atoms causes high compressive residual stresses in the modified layer, which is helpful in increasing the fatigue life of the component.

X-Ray Diffraction (XRD) patterns of the nitro carburized & post-oxidised 35CrMo alloy [9] show many white carbide particles distributed in the matrix. Microstructure observation of the treated specimen shows the three layers formed during nitrocarburizing followed by post-oxidation, the outermost black oxide layer which is composed of Fe₃O₄, then a compound layer, and the innermost diffusion layer. Further it was observed that microstructure of the core remains un-altered after the treatment.

Zhang et al. [11] investigated the effect of nitrocarburizing and post-oxidation on fatigue behaviour of 35CrMo alloy steel. Fig. 3 shows microstructure observation of treated sample. It shows three layers formed during surface treating process: oxide layer, compound layer and diffusion layer. The outermost surface is a loose and black oxide layer, next to it, bright compound layer which is homogeneous and dense, and below the compound layer is the diffusion layer. There is a clear boundary between the compound layer and the diffusion layer. Further, it has been observed that the core remains un-altered after the treatment.

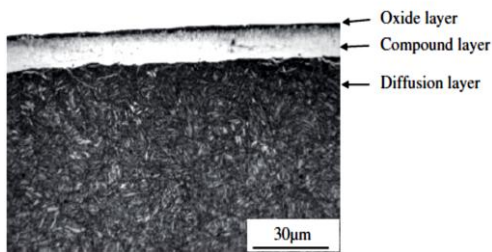


Fig. 3. Microstructure of nitrocarburized and post-oxidised 35CrMo alloy steel [11]

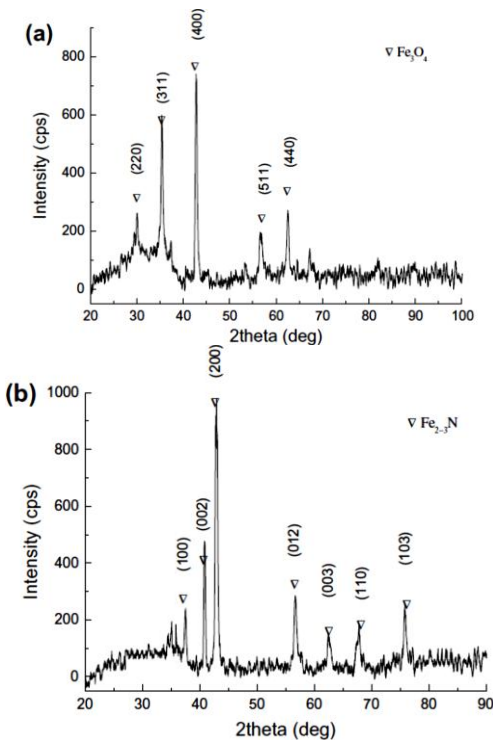


Fig. 4. X-ray diffraction patterns of (a) the oxide layer, (b) the compound layer, of nitrocarburised and oxidised 35CrMo alloy steel [11]

XRD results of treated specimens are shown in Fig. 4, which shows the oxide layer formed on the outermost surface, which is composed of Fe₃O₄, and the layer under it is the compound layer, which consists of ε-Fe₂₋₃N. The authors, however, found very little carbonization in the compound layer.

Zhang et al. [12] investigated the effect of oxy-nitrocarburizing on medium carbon railway axle steel. Fig. 5 shows microstructure observation of the treated specimen.

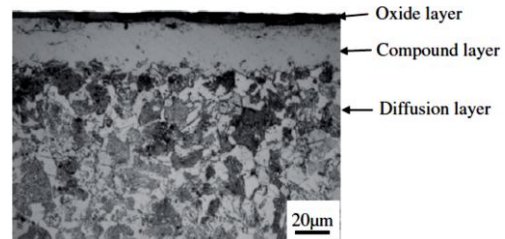


Fig. 5. Microstructure observation of the treated specimen [12]

They also observed three layers in the treated specimen: oxide layer, compound layer, and diffusion layer. The outermost oxide layer having a thickness of 6 μm and a compound layer beneath it having a thickness of 26 μm, followed by the diffusion layer. It was further observed that the microstructure of core remains unchanged even after the treatment.

Abdalla et al. [13] investigated the effect of plasma thermochemical treatments including nitriding, nitrocarburizing and nitrocarburizing after oxidation, on AISI 1020 steel at three temperatures, 673, 773, and 873 K. The X-ray patterns of plasma nitrided AISI 1020 steel at 773 and 873 K are shown in Fig. 6a and 6b, respectively.

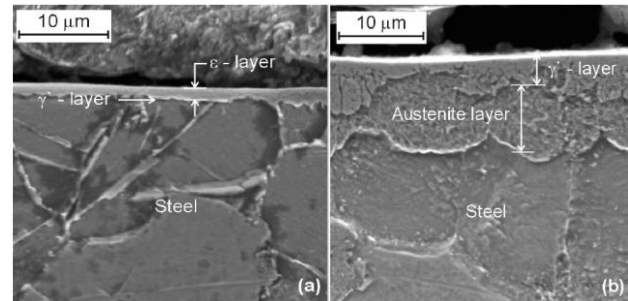


Fig. 6. Cross-sectional SEM micrographs of AISI 1020 steel samples nitrided for 30 min in H₂-75%N₂ at (a) 773 K and (b) 873 K [13]

It clearly indicates the formation of Fe₄N (γ') and Fe₂₋₃N (ε) nitrides. However, in this carbon-free atmosphere, a predominantly ε-phase compound layer with a thin γ' sub layer was observed to be formed for the samples treated at 773 K and in an atmosphere containing 75%N₂ and 25%H₂. At 873 K, which is the temperature just above the Fe-N eutectoid temperature, the microstructure of the compound layer, was found to be an austenite sub layer, which is located between the nitride surface layer and the diffusion zone. In both, nitrocarburizing and nitrocarburizing followed oxidation, the remaining compound layer was predominantly found to be composed of γ'-phase.

4. EFFECT OF NITRIDING ATMOSPHERE AND TIME

Many researchers, while investigating the effect of nitriding on wear characterization of steel, observed that nitriding has potential to offer good wear resistance to the tool steels by increasing their hardness [1, 11]. This rise in hardness has mainly been attributed to the presence of nitrided layer [1]. Moreover, application of nitriding prior to coating provides extra support for coating, by way of increase in substrate hardness [11].

It was found that as the modified layer thickness, the treatment temperature, and time increased, the surface microhardness values increased [6]. This was attributed to the fact that as the treatment time, and temperature increase, the amount of nitrogen atoms in the modified layer also increase, which ultimately results in higher hardness. Table 1 presents results obtained by Yildiz et al. [6] which clearly indicate that the treatment temperature of 500 C and 8 hours duration results in the modified layer thickness of 77-80 μm and the surface hardness of 1650-1700 HV0.01.

Table 1. Changes in modified layer thickness and surface hardness of nitrided AISI 316L for different parameters [6]

Nitriding parameters		Modified layer (μm)	Surface hardness HV _{0.01}
Temperature °C	Time h		
400	4	9-12	890-920
450	4	17-20	1150-1200
500	1	12-15	970-1000
	4	40-45	1300-1350
	8	77-80	1650-1700
Untreated 316 L		-	270-300

Further it was reported that high temperature nitriding treatment has a detrimental effect on the corrosion resistance of stainless steel as CrN formed at high temperatures causes depletion of chromium atoms from bulk material.

Zhang et al. [11] investigated the effect of nitrocarburizing and post-oxidation on 35CrMo alloy steel which was nitrocarburized and post-oxidized in salt bath. Fig. 7 shows the variation of microhardness with respect to the distance from the surface.

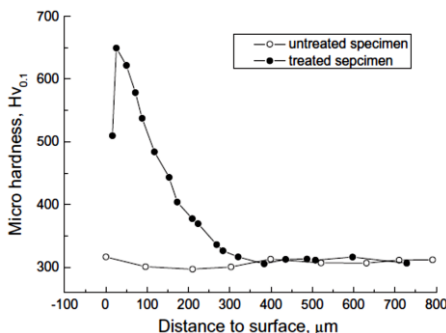


Fig. 7. Variation of microhardness with respect to the distance from the surface [11]

They found that microhardness, which was measured by Vickers microhardness tester with a 0.98 N load and holding for 15 s, of the oxide layer on the outmost surface is comparatively low (360HV0.1), while the compound layer was found to have the highest hardness value of 650 HV0.1. It was, however, noted that the core hardness remained the same as that of the hardness of the untreated sample. Abdalla et al. [13] conducted plasma nitriding of mild steel at varying temperatures & time to investigate the influence of three temperatures, 673, 773, and 873 K with nitriding time varying from 10 to 60 min. Fig. 8 shows variation of surface hardness (HV0.05) with respect to the nitriding time. It has been observed that at 673K, the microhardness values were much higher as compared to those at 773K.

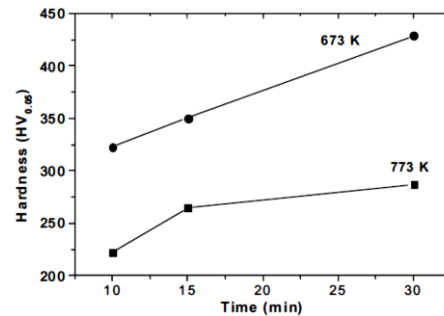


Fig. 8. Surface hardness as a function of nitriding time for samples treated at 673 K and 773 K in H₂-50%N₂ [13]

The compound layer produced was found to be below 0.5μm, at temperatures up to 673 K. However, at 773 K, with increasing nitrogen concentration, the thickness of 2 μm was observed. The nitriding time in both the cases was 30 minutes. This higher thickness of compound layer ultimately results in improved wear resistance. However, for short treatment times, between 10 to 30 min., the surface hardness remained below the bulk hardness.

5. ADHESION CHARACTERIZATION

A good adhesion of the coating to the substrate is always a crucial factor in most applications of coated components. Adhesion quality of the coatings can be evaluated using Rockwell C indentation test which should be performed as per the VDI 3198:1992-08 [14] norms (Verein Deutscher Ingenieure Normen [VDI], 1991). This test may be performed to enquire two distinctive properties of coating, i.e. interfacial adhesion and film brittleness, and cohesion, as well [15]. The samples are indented using Rockwell C indenter with indentation force of 1471 N (150 Kgf), and then investigated using an optical microscope. This test distinguishes between 6 levels of adhesion quality. Fig. 9 represents the distinction between acceptable and unacceptable failure criteria in adhesion test [16]. The adhesion quality of the coat can be evaluated by comparing the indentation pattern. Six levels range from excellent adhesion (HF-1) with only minor radial cracks observed around the indentation mark to the poorest adhesion (HF-6) where coating completely peels off the substrate. Well adherent coatings, manage to withstand the shear stresses and prevent extended delamination circumferentially (Adhesion quality HF-1 to HF-4). On the other hand, extended delamination at the vicinity of the

indentation indicates poor interfacial adhesion, and can be indexed as HF-5 and HF-6.

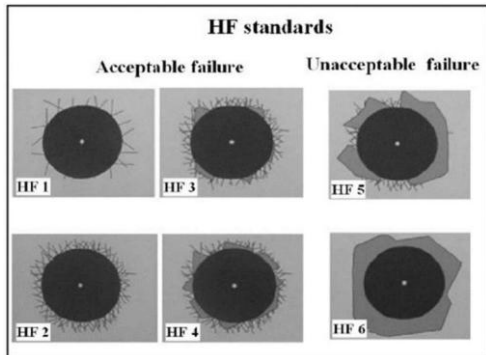


Fig. 9. Distinction between acceptable and unacceptable failure in adhesion test [16]

Adhesion evaluation of untreated & prenitrided samples was carried out [1] and results suggest that surface roughness, coating material, and type of nitriding have an influence on the coating-nitrided substrate adhesion. Table 2 shows the results obtained by Zeghni and Hashmi [1]. As evident from the results, Vanadis 4 steel coated with TiC, TiN, and Al₂O₃ has shown superior results in adhesion test as compared to the D3 tool steel.

Table 2. Adhesion quality of the samples [1]

Sample	Adhesion quality (untreated samples)	Adhesion quality (prenitrided samples)
D ₃ / TiC	HF 2	HF 4
D ₃ / TiN	HF 5	HF 3
D ₃ / Al ₂ O ₃	HF 6	HF 4
V ₄ / TiC	HF 2	HF 3
V ₄ / TiN	HF 5	HF 2
V ₄ / Al ₂ O ₃	HF 6	HF 2

Generally, it is claimed that the compound layer formed during nitriding reduces coating-substrate adhesion, which is attributed to the porosity of compound layer, and its poor adhesion to the steel surface.

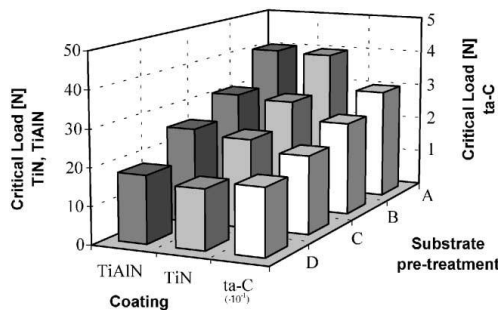


Fig. 10. Critical loads for first failure of the coatings during scratch adhesion test [2]

However scratch test employed to examine adhesion of the coatings [2] on to the substrate did not necessarily lead to a reduced coating-substrate adhesion (refer Fig. 10). Plasma nitriding, however, was found to improve coating-substrate adhesion and load carrying capacity of the substrate, as well. Adhesion of the CrN layers on to plasma nitrided and as-received surfaces of hardened AISI 4140 steel were quantified as HF-5 and HF-1, respectively [8]. Fig. 11 shows SEM micrographs of the damage formed around the indents during Rockwell C tests in single treated, and the duplex treated steel, respectively.

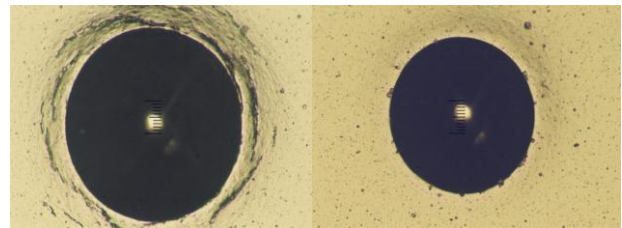


Fig. 11. SEM micrographs showing the indents during Rockwell C tests for (a) single treated and, (b) duplex treated AISI 4140 steel [8]

Moreover, application of nitriding process before PVD coating was found to enhance load carrying capacity and adherence of CrN coating to the substrate.

6. RESIDUAL STRESS CHARACTERIZATION

Zhang et al. [11] investigated the effect of nitrocarburization and post-oxidation on fatigue behavior of 35CrMo alloy steel, and found that there was no residual stress in the untreated specimen, while a compressive residual stress field was found along with the tensile residual stress in the core. Distribution of residual stress at the surface of 35CrMo alloy steel is shown in Fig. 12.

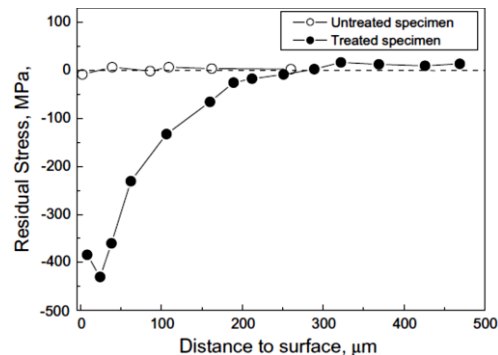


Fig. 12. Longitudinal residual stress profile at the surface layer [11]

The value of the tensile residual stress was about 7 MPa. The maximum compressive residual stress found at the subsurface zone was about 430 MPa.

Zhang et al. [12] investigated the effect of oxynitrocarburizing on fatigue properties of medium carbon railway axle steel. Fig. 13 shows longitudinal residual stress profile of the treated and untreated specimen. It can be seen that there is a compressive residual stress field at the surface balanced with a slight tensile residual stress field in the core.

The surface residual stress for the treated specimen was found to be 260 MPa, and the maximum compressive residual stress found at the compound layer was ~506 MPa. Unlike the results obtained by Zhang et al. [11], the untreated specimen has shown the compressive residual stress of 40 MPa.

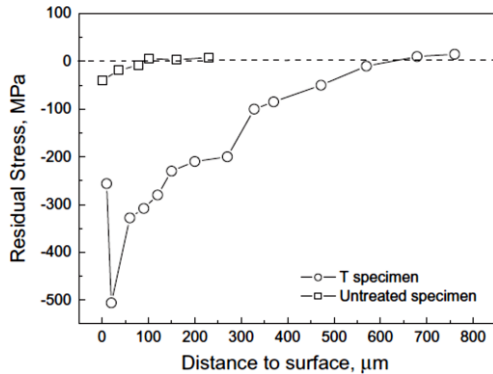


Fig. 13. Residual stress distribution at the surface of treated and untreated specimen [12]

7. TRIBOLOGICAL STUDIES OF COATED-NITRIDED STEEL

To evaluate the friction and wear behavior of coated-nitrided steels, researchers have used a variety of tribo-testers with different contact configuration. The various contact configurations employed are pin on disc, reciprocating type etc.

Roughness characteristics

Roughness is generally an undesirable property, as it may cause friction, wear, drag, and fatigue, but it is sometimes beneficial, as it allows surfaces to trap lubricants and prevents them from welding together. Many authors have investigated change in the surface roughness caused by nitriding and by the coating deposition. Many of them found that after nitriding, both, the average roughness value, R_a and the maximum peak to valley height, R_{max} of the original ground surface, increased. The degree of increase in surface roughness varied with the nitriding conditions. Podgornik et al. [2] have reported an increase from $0.35\mu\text{m}$ to approximately $0.65\mu\text{m}$ in average roughness with respect to the original ground surface, using plasma nitriding in a nitrogen-poor atmosphere (99.4% H_2 -0.6% N_2), whereas, plasma nitriding in a nitrogen-rich atmosphere (75% H_2 -25% N_2 gas mixture) resulted in a rougher surface of about $1.27\mu\text{m}$ average roughness. However, deposition of PVD coatings on thermo-chemically treated substrates or on polished substrates did not result in any measurable increase in the surface roughness. Similar results, obtained by Yildiz et al. [6] are presented in Table 3.

Surface roughness of untreated AISI 316 L was observed in the range 0.06 to $0.08\mu\text{m}$, whereas, the values reported for plasma-nitrided specimens were in the range of 0.08 to $0.43\mu\text{m}$. Roughness values increased with increase in nitriding time and nitriding temperature, as well.

Table 3. Changes in modified layer thickness and surface roughness of nitrided AISI 316L for different parameters. [6]

Nitriding parameters		Modified layer (μm)	Surface roughness R_a (μm)
Temperature $^\circ\text{C}$	Time h		
400	4	9-12	0.08-0.11
450	4	17-20	0.30-0.43
500	1	12-15	0.21-0.39
	4	40-45	0.24-0.27
	8	77-80	0.37-0.43
Untreated 316 L		-	0.06-0.08

Friction behavior and mechanism

The general objective of nitriding & then applying coating to a surface for any tribology based application is to impart both smoothness and hardness to the surface so that friction and wear are reduced. Among the two degrading phenomena, friction is a critical factor dictating the efficiency of mechanical assemblies that involve sliding surface contact, and hence, minimization of the friction of the coating is a vital need.

Podgornik et al. [2] found that coating of surface-treated AISI 4140 steel with TiN or TiAlN coatings increased the steady-state coefficient of friction from 0.3 to 0.4, which was found to be more or less independent of the substrate pretreatment used, while, with ta-C, steady-state coefficient of friction was found to be between 0.1 and 0.15. With plasma nitrided substrate, the coefficient of friction showed a very constant value throughout the test. These low values of coefficient of friction can be attributed to the formation of a carbon transfer film. Yilbas et al. [7] investigated wear behavior of plasma-nitrided and TiN coated AISI H11 and AISI M7 twist drills, and compared the results with previous experiments. The variation of wear and friction coefficient with respect to sliding time is presented in Fig. 14a and 14b, respectively.

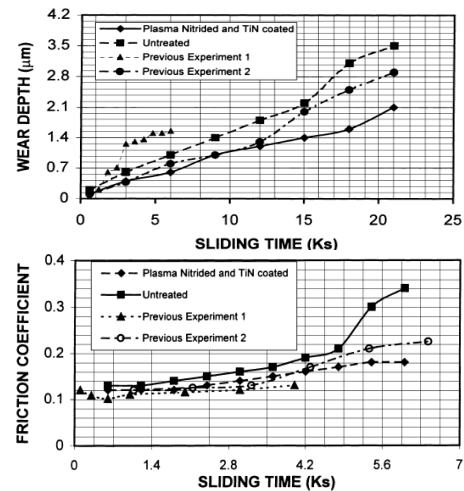


Fig. 14. Variation of a) wear depth b) friction coefficient, with sliding time [7]

They found that friction coefficient appears to be the same for treated as well untreated surfaces in the initial stage of the friction test. Though plasma nitrided surface results in slightly higher friction coefficient, but it is less than that for untreated surface. Higher value of friction coefficient for plasma-nitrided surface may be because of the brittle nature of the white layer formed on the plasma-nitrided surface, which, in turn, results in less wear in the early stage of the sliding time. Moreover, wear occurs more slowly in duplex treated samples than for untreated work pieces.

Friction behavior and mechanism at elevated temperature

The coefficient of friction for plasma nitrided surface [3] was observed to be less than the untreated surface, but, as temperature increases, friction coefficient steadily increases. The reason for the low friction is due to the fact that PN ring has high surface hardness and fine grain structure, which eliminates adhesion.

Staia et al. [5] studied high-temperature wear behaviour of nitrided AISI D2 tool steel, prior and after plasma assisted PVD (PAPVD) coating. Variation of friction coefficient with distance, at 25oC, 300oC, and at 600oC, in case of plasma nitrided, and TiN, and TiAlN coated D2 tool steel is presented in Fig. 15.

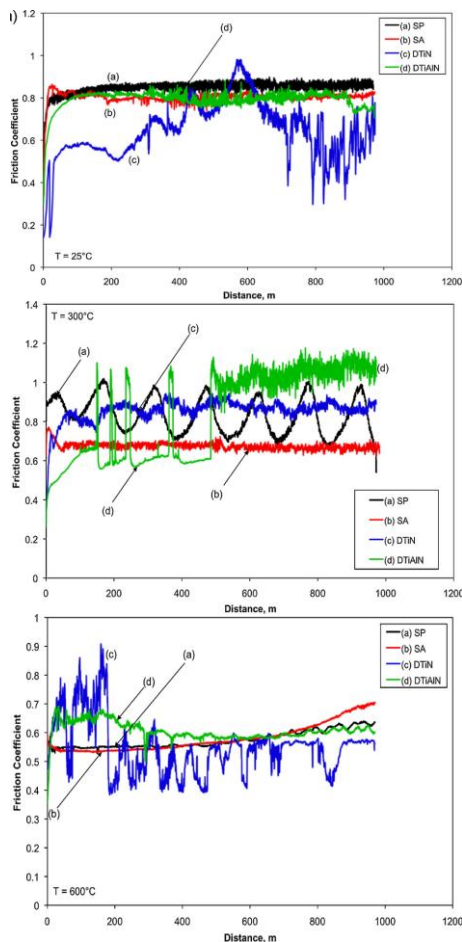


Fig. 15. Variation of friction coefficient with distance at a) 25oC, b) 300oC, and c) 600oC [5]

Results indicate that as temperature increases, friction coefficient of the coated specimens goes on reducing and is less than uncoated specimens up to 300oC. However, at 600oC, the uncoated substrate exhibited the highest resistance to sliding wear. At this temperature, the formation of well bonded surface glazed layer may give rise to a significant reduction in the friction coefficient.

Wear resistance and mechanism

The results obtained by various researchers strongly suggest that nitriding treatment of the steel improves the wear resistance [1, 3, and 7]. Pre-nitriding of tool steels [1] before coating with Al₂O₃ and TiN significantly improved the wear resistance, which was attributed to the presence of the hard nitriding layer on the lower surface. Podgornik et al. [2] found that wear of the duplex-treated pins was strongly influenced by the substrate pre-treatment. They found that plasma nitriding carried out at 5400C and in nitrogen-rich environment (75%H₂-25%N₂ gas mixture) resulted in the least wear. Reduced wear is attributed to the increased substrate hardness. Moreover, compared to coated hardened substrates, the nitrided and coated specimens showed improved sliding wear resistance, which can mainly be attributed to a higher substrate hardness and improved coating–substrate adhesion due to introduction of nitriding process [4].

Wear resistance and mechanism at elevated temperature

Staia et al. [5] conducted experiments with nitrided and coated D2 tool steel at elevated temperatures and observed improvement in the wear behaviour up to certain temperature, but found significant wear, at elevated temperature, than that observed in case of uncoated steel. Fig. 16 shows variation in wear volume with temperature for the materials under investigation.

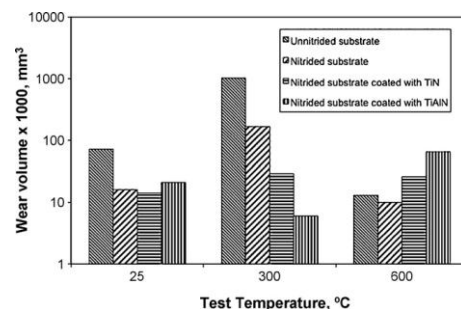


Fig. 16. Variation of wear volume for materials under investigation [5]

At elevated temperature, besides oxidation of the coatings, mechanical strength of the substrate decreases leading to fracture and delamination of the films, and the formation of third-body particles, which ultimately contributes to increased wear.

Similar work carried out by Devaraju et al. [3] at high temperature and under high vacuum reveals similar results. Poor tribological behaviour for untreated specimens was attributed to strong adhesion, high friction; heavy surface damage which ultimately resulted in very high metal loss at all tested temperatures. However they observed improved results at higher sliding speed.

8. CONCLUDING REMARKS

For satisfactory performance of coated components, the basic requirements are sufficient coating-substrate adhesion and the ability of the substrate to support the coating. By enhancing the hardness of the steel substrate by nitriding, the load carrying capacity of the substrate can be improved, which can then effectively support a hard and brittle coating. From the present review, it can be seen that nitriding can definitely improve the hardness and wear resistance of tool steels at room temperature. Even in high temperature applications and under high vacuum, nitriding followed by coating results in improvement in the machining performance of the pre-nitrided components. It has also been shown that inclusion of nitrogen atoms at the surface results in improvement in plain and fretting fatigue life of steel.

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