

Original Article

# Influence of Duplex Plasma Nitriding and PVD Hard Coatings on the Mechanical and Tribological Performance of Tool Steel

Kolse Chandrashekhar Ashok<sup>1</sup>, R. A. Kapgat<sup>2</sup>

<sup>1,2</sup>Department of Mechanical Engineering, Sanjivani College of Engineering, Kopergaon, Savitribai Phule Pune University, Pune.

<sup>1</sup>Corresponding Author : [kolseca@gmail.com](mailto:kolseca@gmail.com)

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**Abstract** - Improving the durability of tooling and formation components remains one of their biggest issues. This is due to the severe wear, friction, and deterioration of the surfaces when exposed to high-concentration stresses. Another example of a commonly used surface engineering method is Physical Vapour Deposition (PVD) coating and thermochemical treatment. The techniques are effective in enhancing the general effectiveness of the tool. The overall impact of plasma nitriding and PVD hard coating on tool steel was systematically studied in the current research. It was dedicated to the mechanical and tribological behaviour of the materials. TiCN and TiAlN were coated in order to enhance the surface hardness and wear resistance, following the production of a hardened diffusion layer through plasma nitriding, which was able to bear external loading. Mechanical characterization was also done using microhardness measurement and tribological performance by the use of a dry sliding wear test. The worn surfaces and the coating integrity were analysed by means of X-Ray Diffraction (XRD), Energy-Dispersive Spectroscopy (EDS), and Scanning Electron Microscopy (SEM). The observation demonstrates that the surface hardness of the duplex-treated samples and the singly treated samples significantly differ in their wear rates and surface hardness. The result of the work develops a mechanistic base for higher hybrid surface treatment strategies. Moreover, they offer a reference framework for further optimization and experimental modeling of the engineering tools of surface forming.

**Keywords** - Nitriding of plasma, PVD hardened coating, TiAlN and TiCN coating, Tribological behaviour.

## 1. Introduction

The complex service conditions that metal forming tools deal with are high contact pressures, sliding interactions repeated multiple times, and repeated thermal exposure [1]. Such conditions tend to lead to severe friction loading. Such conditions eventually decrease the life of the tool and process stability as they promote successive surface degradation processes such as abrasive wear, adhesive transfer, galling, microcracking, and coating delamination. Premature tool wear in industrial forming has not only a higher cost effect, but also results in a reduced dimensional accuracy and quality surface of formed parts [2]. Additionally, this failure has a considerable influence on the effectiveness of production in general. Consequently, it has become one of the leading research topics in the broader surface engineering to enhance the surface endurance of forming tools. [3, 4].

To achieve the optimum of hardness/toughness, tool steel in forming implementation is usually followed through bulk heat treatment methods, like austenitizing, quenching, and tempering [5]. Although this type of treatment is effective in

enhancing core mechanical strength, it is usually inadequate in dealing with the surface-dominated failure modes that control tool performance during service [6]. The mismatch between the bulk strength and the surface durability has led to the incursion of the surface, which has seen the development of advanced surface modification methods to selectively enhance near-surface characteristics without adversely affecting the substrate core. [7].

Plasma nitriding, among other thermochemical surface treatments, has been widely applied since it adds nitrogen to the surface of the steel. The result of this process is the formation of a hardened diffusion zone and, in other instances, a compound layer. The resistance to the plastic deformation, fatigue resistance, and surface hardness under contact loading are also significantly improved with the nitride layer [8]. However, even in dry or boundary-lubricated conditions, as seen in forming processes, some limits to the condition of nitrided surfaces can be observed when exposed to extreme conditions of sliding or adhesion wear. Degradation caused by friction, localized damage, and all these are very challenging.



These are presently limiting the individual performance of nitriding in high-need applications [9].

Physical Vapour Deposition (PVD) coating, such as TiCN and TiAlN, which are generated by deposition of thin, durable, and wear-resistant layers of ceramic, offers and serves as an alternate method of increasing the performance of surfaces [10, 11]. These coatings have great abrasion resistance, oxidation, chemical stability, and hardness that are widely known. In forming tools, PVD coating is used to reduce the coefficients of friction, deferred wear, and material transfer, and create resistance to wear in the part. Nevertheless, there is often premature failure in the PVD coatings in the form of cracking or delamination when applied directly onto untreated (or under-strengthened) base materials. This has been mainly caused by the inadequacy of the load-bearing capacity of the underlying substrate. This condition permits excessive under contact, subsurface deformation [10].

Duplex surface engineering strategies have been used as a feasible method of addressing the flaws of individual surface processes [12]. Plasma nitriding is also used as a pre-treatment in duplex systems to increase the mechanical support of the substrate. The deposition process is then followed by a coating of PVD so as to give a better tribological performance. Through the redistribution of contact stress and providing mechanical strength as an interlayer, the nitride diffusion layer reduces the localization of strain at the interface between the coating and substrate [13]. This synergetic, effective action between the coating and hard material and the nitride substrate enhances coating bonding and inhibits the formation of cracks. In the end, this results in the improvement of the wear resistance. [14].

The solution to the challenges linked to the separate surface treatment processes has been identified in duplex surface engineering methods. These combination therapies are more successful than individual therapies. Plasma nitriding as an initial treatment on the duplex system is taking place to enhance the mechanical bearing of the substrate. In order to provide enhanced tribological performance, a PVD coating is then deposited on top of this step. The layer of diffusion of nitride serves as a mechanically robust layer when diffusion interfaces that spread the contact stresses in a more uniform way. This act consequently lessens the strain that is concentrated at the coating-substrate interference [15].

Despite broad studies on plasma nitriding and PVD coatings, limited effort has focused on their joint influence on AISI 4140 tool steel under dry sliding situations. In particular, comparative assessment of TiAlN and TiCN coatings and their correlation with microstructural and tribological behavior remains inadequate. Therefore, this study examines the effect of duplex plasma nitriding and PVD coatings to launch structure–property–performance relationships. Recent studies have highlighted important progress in PVD coatings and

duplex surface treatments for enlightening tool performance. TiAlN coatings are reported to reveal greater oxidation resistance and thermal stability, while TiCN coatings deliver good toughness and wear resistance under sliding circumstances. Duplex treatments linking nitriding and coatings have revealed better adhesion and load-bearing capacity. However, most studies emphasise individual coatings or different substrates, with restricted comparative analysis under like circumstances.

Although duplex plasma nitriding and PVD coatings have been extensively examined, most present studies emphasise single coating systems, different substrate materials, or do not provide a direct contrast under the same working conditions. In addition, limited labours have been conducted to correlate coating composition, microstructural features, and tribological performance in a unified framework.

The novelty of this effort lies in incorporating mechanical, tribological, and microstructural analyses to clarify coating performance through a structure–property–performance method. Furthermore, this study offers a relative understanding of coating consistency and wear mechanisms under matching test conditions, which is important for choosing appropriate coatings in forming tool applications.

The objective of the research is to be used as a theoretical framework for an experimental study on liberal surface metalworking techniques used on the machining of instruments. The given work offers the mechanistic benefits of duplex surface treatment, and thus presents future studies of surface conditioning methodology, parameter optimization modeling on a sound scientific base, and its contributions in the formative of experimental databases that predict performances of tools.

## **2. Materials and Methods**

### ***2.1. Substrate Material and Substrate Preparation***

The substrate material selected for the present investigation was a medium alloy tool steel. The material is widely used in forming and tooling applications due to its favourable combination of machinability, hardness, and strength. The steel was delivered as bars, which were then machined into specimens with uniform dimensions suitable for tribological testing and mechanical characterization. All specimens underwent standard metallographic preparation before surface treatment. To achieve a consistent and repeatable surface finish, this required consecutive grinding using a silicon carbide abrasive sheet and polishing. After polishing, the specimens were ultrasonically cleaned in acetone. This process was performed to remove residual contaminants, debris, and oils from the surface. Before being processed further, the cleaned samples were air-dried and kept in a controlled environment to avoid surface conditions. The substrate and later surface treatment procedures interacted reliably.

A minimum of three samples were arranged for each condition (untreated, nitrided, TiAlN-coated, and TiCN-coated) to confirm repeatability of outcomes.

## 2.2. Plasma Nitriding Treatment

A thermochemical surface treatment called plasma nitriding was used to improve the steel substrate's near-surface mechanical characteristics [16, 17]. Under strictly regulated process conditions, the treatment was performed in a controlled plasma nitriding surface. Prior to nitriding, the chamber was evacuated to remove atmospheric gases. Then, a nitrogen-containing gas mixture was introduced to initiate plasma formation. In order to facilitate efficient nitrogen diffusion while preventing excessive compound layer formation that would cause surface brittleness, the nitriding temperature and treatment period were chosen. The nitriding factors were carefully chosen based on the literature to confirm the development of a uniform diffusion layer without extreme brittleness.

## 2.3. PVD Hard Coating Deposition

After plasma nitriding, hard ceramic coatings were applied to chosen specimens utilizing a PVD method. Because of their proven wear resistance, high hardness, and appropriateness for tooling applications, TiCN and TiAlN coatings were selected. For a comparative assessment, coatings were applied to non-nitrided specimens to evaluate the effect of the nitride interlayer on the performance of the coating. High vacuum was used during the deposition procedure to guarantee the consistency and purity of the coating [18]. In-situ and pre-coating consisted of plasma cleaning the specimen to increase surface activation and adhesion of the coating. The coating parameters, such as the deposition temperature, the bias voltage, as well as gas composition, were maintained the same in all samples to ensure reproducibility.

Deposition constraints were organised to get uniform coating thickness and good adhesion, as described in previous studies.

## 2.4. Mechanical Characterization

The untreated, nitride-coated, and duplex-treated specimens were tested by microhardness testing to determine their mechanical response [19]. The hardness in Vickers microhardness tests was measured using a hardness tester with constant measurement of weight and time. To account for surface variability and guarantee statistical reliability of the data, indentations were created at several points on each specimen. For nitride samples, hardness evaluations were conducted close to the surface to determine the extent of nitrogen diffusion in improving surface hardness. Hardness levels in coated and duplex-treated samples showed the combined effect of the underlying substrate and coating. The average hardness values were utilized for comparative analysis among various treatment conditions.

Hardness values were gained from at least three indentations at dissimilar locations, and the average value was stated to reduce experimental error.

## 2.5. Tribological Testing

Dry sliding wear experiments on a pin-on-disc tribometer were used to evaluate tribological performance. The experiment was conducted in a laboratory environment with a regulated normal load, sliding speed, and sliding distance. The coefficient of friction was continually measured as a function of sliding distance through testing.

Following the end test, the dimensions of the wear track were assessed, and the wear rate was determined based on material loss. To assess the impact of surface treatments on wear resistance and friction response, the tribological behaviour of untreated, nitrided, coated, and duplex-treated specimens was methodically examined.

Each wear test was repeated three times under matching conditions, and the average values of wear depth and wear volume were considered for analysis.

## 2.6. Surface And Microstructural Analysis

Surface characterization after wear was carried out using SEM to analyse wear track morphology and determine the primary wear mechanism. Features including grooves, material transfer, microcracks, and coating delamination could be observed in great detail [20]. EDS was used alongside SEM to examine elemental distribution in the wear tracks and evaluate coating integrity following sliding. XRD was used for phase identification and structural study of the treated surface.

# 3. Results and Discussion

## 3.1. Surface Hardness Evaluation After Surface Treatment

The effect of surface modification on hardness was qualitatively assessed using Vickers and Rockwell C scales, as outlined in the hardness data shown in Table 1. The untreated AISI 4140 substrate showed a relatively low hardness of about 207 HV and 13 HRC, which is typical of tool steel that has been heat-treated normally without surface reinforcing. A significant rise in the hardness was noted after the deposition of the PVD coating.

The AISI 4140 specimen with a TiAlN coating demonstrated a Vickers hardness of about 734 HV, reflecting a nearly 3.5 times enhancement compared to the uncoated substrate. In the same vein, the Rockwell C hardness significantly improved from 13 HRC to 62 HRC. The TiCN-coated samples also exhibited significant hardness improvement, reaching a Vickers hardness of around 719 HV and a Rockwell C hardness of 61 HRC. TiAlN and TiCN coatings differ slightly in hardness, which can be explained by differences in coating chemistry, bonding properties, and microstructural elements created during PVD deposition [21, 22].

**Table 1. Result of surface hardness test**

Sr. No.	Type of hardness	AISI 4140	TiAlN coating	TiCN coating
1	Hardness, Vickers	207	734	719
2	Hardness, Rockwell	13	62	61

### 3.2. Tribological Behaviour Under Dry Sliding Conditions

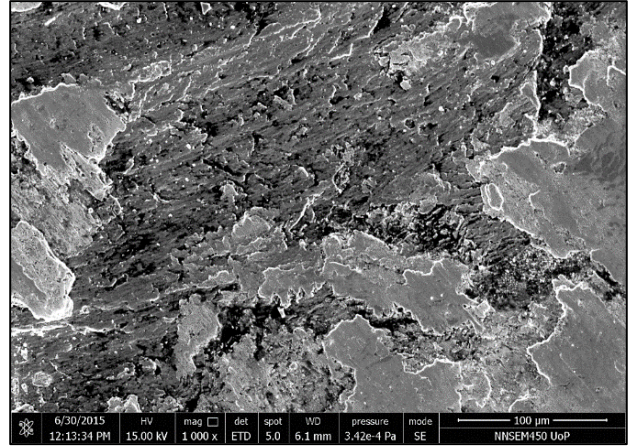
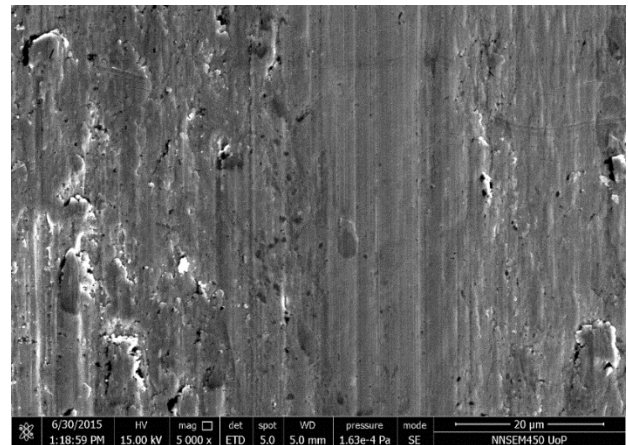
The tribological behaviour of the AISI 4140 specimens with surface treatment was assessed under dry sliding conditions, and the recorded wear depth and volumetric wear loss are presented in Table 2. The nitride AISI 4140 specimen showed a volumetric wear loss of 7.381 mm<sup>3</sup> and a wear depth of around 148.84 µm. In a similar way, the volume wear loss significantly dropped to 4.172 mm<sup>3</sup>, showing improved resistance to material loss. This enhancement is linked to the elevated hardness and chemical stability of the TiAlN coating, which suddenly minimizes asperity penetration and restricts the development of abrasive grooves during sliding. With a wear depth of roughly 90 µm and a volumetric wear loss of 4.5 mm<sup>3</sup>, the TiCN-coated specimen likewise showed notable wear resistance. Although the TiCN coating's overall tribological performance is still significantly superior to that of the nitride substrate, the wear values are only marginally higher than those of the TiAlN-coated sample. The marginally increased wear noted in the TiCN-coated sample could be attributed to variations in coating microstructure, bonding properties, and frictional behaviour during dry sliding conditions [23, 24].

### 3.3. SEM Analysis Of Worn Surfaces

SEM was utilized to examine the wear morphology of AISI 4140 specimens coated with TiAlN and TiCN following dry sliding tests. The goal was to determine prevailing wear mechanisms and link surface characteristics with the noted tribological performance. An investigation of the TiAlN-coated specimen using SEM shows the presence of a well-defined shallow wear track characterized mainly by scratches oriented parallel to the direction of motion, as shown in Figure 1. The lack of deep ploughing marks or extensive surface suggests that abrasive wear was greatly minimized. The morphology of the wear track indicates that the TiAlN layer is relatively unaffected by the process of sliding. No significant delamination of the layer is observed, so there is strong bonding of the layer to the surface material. The sparse presence of microcracks additionally suggests that the coating had adequate toughness to endure contact stresses without experiencing catastrophic failure [25].

On the other hand, the SEM micrograph of the TiCN-coated sample in Figure 2 indicates a relatively rough wear track with deeper grooves. Although severe wear removal is not apparent, the fact that the groove is deeper than in the TiAlN-coated sample indicates a greater degree of abrasive wear. Localized wear of coating, thinning, and small microcracks are evident, suggesting concentrated stress during sliding. However, there was no major delamination of the coating, indicating that the TiCN coating retains sufficient

adhesion and structural integrity under the applied test conditions [26].

**Fig. 1 SEM micrograph of TiAlN****Fig. 2 SEM micrograph of TiCN**

### 3.4. EDS Compositional Analysis

Following tribological testing, the elemental composition and chemical integrity of the AISI 4140 specimen coated with TiAlN and TiCN were examined using EDS.

The effective deposition of the Ti-Al-based nitride coating is confirmed by the prominent Ti and Al peaks in the EDS spectrum of the coated specimen. With a normalized atomic percentage of roughly 46.5% for Ti and 30.3% for Al, these two elements dominate the discovered composition. Due to minimal coating thinning inside the wear track, a minor iron (Fe) content of about 3.3% indicates localized exposure of the underlying steel substrate. In accordance with the chemical composition of AISI 4140 substrate, trace amounts of alloying elements like chromium (Cr), manganese (Mn), silicon (Si),

and molybdenum (Mo) were also found. Following dry sliding, the coating maintained its integrity, as evidenced by the comparatively low Fe concentration relative to Ti and Al.

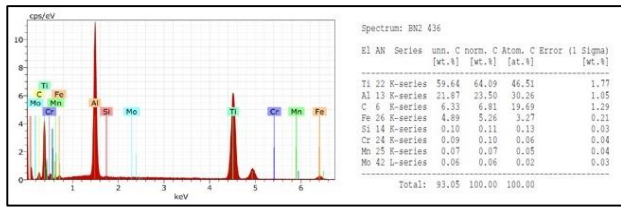


Fig. 3 EDS spectrum and elemental composition of TiAlN-coated specimen

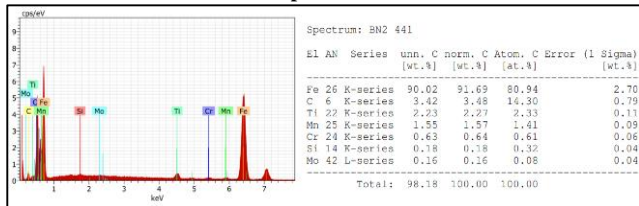


Fig. 4 EDS spectrum and elemental composition of TiCN-coated specimen

Conversely, the TiCN-coated specimen's EDS spectrum indicates a significantly higher content, with Fe representing approximately 80.9% of the detected elements. Increased steel substrate exposure in the wear area is indicated by the notable Fe presence, which may indicate considerable coating thinning or partial loss during sliding. The presence of the TiCN coating system was confirmed by the detection of carbon at about 14.3 at.% and titanium at around 2.3 at.%. The TiCN coating exhibited a comparatively lower Ti concentration than the TiAlN-coated specimen. This suggests that the TiCN coating experienced greater wear or localized degradation under identical test conditions. As with the later alloying materials, some trace levels of silicon, chromium, manganese, and molybdenum were also detected. Although the Fe signal is greater, there is the presence of Ti and C, meaning that there is residual TiCN coating protecting against severe wear to some extent. The wear depth and volumetric wear loss experienced during tribological tests were slightly greater in the TiCN-coated specimen that had a greater substrate exposure, as was indicated.

The EDS outcomes were compared in terms of evaluations. The review shows the high compositional stability of the TiAlN coating compared to TiCN under dry sliding. In the TiAlN-coated specimen, there was less Fe and more Ti-Al concentration. This implies that it has superior coating stability and less engagement between the substrate and wear mechanism.

### 3.5. Impact Strength

Table 2 gives a summary of the impact strength of the specimens of AISI 4140 treated on the surface. The findings demonstrate the effect of nitriding and PVD coating on dynamic loading resistance. The nitride specimen exhibited

the highest impact energy of 4J and therefore the best ability to endure stresses caused by impact. It is explained by the fact that the formation of a diffusion layer in the course of nitriding occurs, leaving the ductile core. Dissipation of the energy can occur with the help of this structure by plastic deformation.

Table 2. Impact strength values for the investigated sample

Sr. No.	Specimen	Impact strength
1	Nitrided AISI 4140	4
2	TiAlN-coated AISI 4140	3.5
3	TiCN-coated AISI 4140	3.25

PVD-coated specimens, on the other hand, displayed a slight decrease in impact strength. The TiAlN-coated specimen absorbed 3.5 J. The TiCN-coated specimen exhibited a slightly lower value of 3.25 J. The ceramic nature and high-hardness coatings are the main causes of the decrease in impact energy for coated samples. These features encourage localized stress concentration under abrupt loading and limit surface flexibility.

With marginally higher impact resistance, TiAlN suggests it better accommodates stresses at the coating-substrate boundary compared to TiCN. The coated specimens retain acceptable toughness levels for forming tool applications, even though their impact strength has decreased. High impact energy absorption is not as crucial in these applications as improved wear resistance and surface durability [27].

### 3.6. Ultimate Tensile Strength

Table 3 displays the surface-treated AISI4140 specimen's ultimate tensile strength. These findings show how PVD coatings and nitriding affect the load-bearing capacity under uniaxial tensile loading. The nitrided specimen exhibited a tensile strength of 655 N/mm<sup>2</sup>. This represents a baseline strength achieved through thermomechanical surface modification alone, without the contribution of a hard surface coating. The PVD-coated specimens showed a notable increase in tensile strength. The TiAlN-coated AISI 4140 sample exhibited the highest tensile strength at 788.4 N/mm<sup>2</sup>, representing approximately a 20% improvement over the nitrided specimen.

Table 3. Ultimate tensile strength values for the investigated samples

Sr. No.	Specimen	Ultimate tensile strength (N/mm <sup>2</sup> )
1	Nitrided AISI 4140	655
2	TiAlN-coated specimen	788.4
3	TiCN-coated specimen	760.5

The TiAlN coating's strong interfacial adhesion and high stiffness are responsible for this improvement. These characteristics become relevant during tensile deformation and help in maximising the transmission of the stress and

delaying the occurrence of necking. Also, the tensile strength of the TiCN-coated specimen was enhanced substantially and thus achieved 760.5 N/mm<sup>2</sup>. Even though the TiCN coating was a bit lower in comparison to the TiAlN-coated sample, it was effective in improving the tensile response of the substrate by inhibiting crack formation as well as encouraging a more homogenous stress distribution.

### 3.7. X-Ray Diffraction Analysis

Figure 5 presents the X-ray diffraction patterns of the TiAlN and TiCN-coated AISI 4140 samples, measured in the 2θ range of 30 ° - 80 °. The diffracted profile can be used to determine the phase constitution, crystallographic orientation, and structural stability of the deposited coatings of the material.

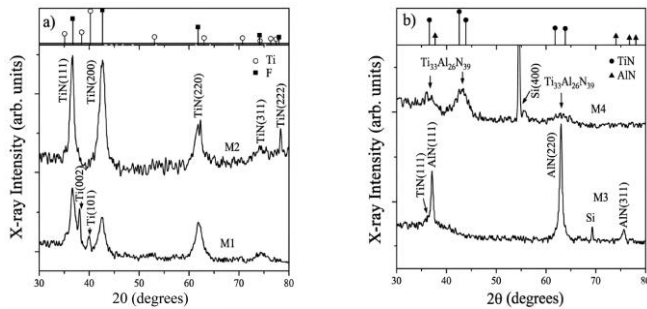


Fig. 5 X-ray diffraction patterns in the 2θ range from 30 to 80 for the TiAlN and TiCN-coated sample

Evaluation of the TiAlN-coated specimen indicates that the XRD pattern of the coated specimen is dominated by TiN-based solid solution phases. The pattern feature has distinct diffraction reflections, i.e., indexed to the (111), (200), and (220) planes. These peaks indicate that the TiN lattice has been replaced with Aluminium atoms to form an FCC TiAlN structure. The XRD peak broadening that has been observed indicates that the TiAlN coating has a non-crystalline structure, which helps to increase hardness and improve tribological performance. Small peaks shifts with respect to stoichiometric TiN are caused by lattice distortion due to the inclusion of Aluminium as well as residual stresses developed during the PVD process.

The diffraction pattern of the TiCN-coated material has mixed phases of nitride-carbide characterized by reflections attributed to Ti(C, N) solid solution. The carbon in the lattice of TiN is the cause of variation in the lattice parameters. All these alterations are reflected in the changed values of the peak positions and intensities as compared to TiAlN. The sharper peaks of the TiCN coating represent a very coarse crystalline structure. This structural variation is consistent with the marginal low hardness and wear in the TiCN-coated specimens compared to the TiAlN-coated sample. Weak diffraction peaks associated with the steel substrate can be observed with both coatings, showing that the coatings are continuous and sticking in places, however, slim enough to

allow some X-rays to penetrate. Notably, there are no unpleasant secondary phases or oxide peaks determined. This proves the stability of the coating with regard to the deposition conditions used.

## 4. Conclusion

The influence of nitriding of plasma-treated and then PVD-hardened tool steel in AISI 4140 was thoroughly examined in this work. The evaluation was on the product's mechanical, tribological, and microstructural attributes. In the research, it is demonstrated that in adverse service environments that require interaction between touch and mechanical loads, surface engineering plays the key role in ensuring that the tool performance is maximized.

Plasma nitriding was able to create a hard diffusion zone, which greatly enhanced the surface hardness and gave strong support to the subsurface. In combination with PVD coating, the hardened coating improved the transfer of loads and decreased the surface deformation. TiAlN as well as TiCN coating made a significant contribution towards the rise of the surface hardness. TiAlN was a little harder, which was due to its formed crystalline structure and solid solution strengthening effect. Dry sliding tribological tests had revealed that coated samples underwent much lower wear depth and had lesser volumetric loss than samples that were pure nitride. Observation of SEM showed better wear tracks and wear coating reduction, which justifies TiAlN having higher wear resistance amongst the observed coatings. On the contrary, TiCN-coated surfaces demonstrated somewhat more pronounced abrasive properties, which agrees with their relatively larger wear values.

This was also corroborated by the compositional and structural analysis, which verified the performance trends. Superior coating integrity and reduced substrate exposure for the TiAlN-coated specimen were confirmed by EDS results. Additionally, XRD analysis verified that stable TiN-based solid solution phases formed in both coatings. Enhanced hardness and wear resistance in TiAlN coatings resulted from refined microstructure and lattice distortion. The specimen's impact strength was somewhat reduced by the coatings because they were ceramic in nature. On the other hand, the tensile strength was greatly increased by improved stress distribution and coating-substrate interaction. TiAlN-coated specimens exhibited the highest ultimate tensile strength, indicating superior resistance to tensile loading and premature failure.

From an industrial viewpoint, the enhanced hardness and wear resistance witnessed in TiAlN- and TiCN-coated samples specify robust potential for application in forming and tooling operations, where reduced wear and extended tool life are serious. However, certain boundaries must be considered. The extra processing stages involved in duplex treatment may increase production cost and processing time. Furthermore,

coating performance is sensitive to process restrictions and substrate preparation, which may affect consistency in large-scale applications. The study is also incomplete for laboratory-scale dry sliding situations and does not account for complex industrial surroundings, including lubrication, thermal cycling, or impact loading. Overall, the results show that TiAlN and TiCN coatings successfully enhance AISI 4140

tool steel's substrate surface performance. TiAlN has the best combination of mechanical stability, wear resistance, and hardness among them. The findings offer a solid experimental foundation for the creation of sophisticated duplex surface treatment techniques. Future initiatives centred on the optimization and predictive modelling of forming tool performance are also supported by this work.

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