A PRELIMINARY INVESTIGATIONS ON SECOND GENERATION NANO COMPOSITE SUPER NITRIDE COATINGS ON ASTM A681 TOOL STEELS

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Abstract

It is essential to sustain ever increasing challenges looked by cutting tool producers in enhancing the performance of the tools amid machining of hard to-cut materials which have been created as of late. Though coated tools have discovered wide application in industries, there still remains an extensive scope of change of the properties of coatings with a view to accomplish friendly cutting environment. Surface treatment has been recognized as one of the conceivable avenues which can possibly expand properties and performance of coated tools in machining. The current research work has utilized a second generation AITiN super nitride nano composite coating commercially called Hyperlox® was deposited on ASTM A681 tool steels by PVD magnetron sputtering technique. The friction and wear properties were investigated using pin on disc test, has been correlated to microstructural and micromechanical characteristics. The outcomes demonstrated that the Hyperlox® coatings introduced lower friction coefficient and lower wear rate among all loading conditions.

1. Introduction

Medium carbon steels are widely used in fabrication of components in engineering, construction and mining industries due to its strength and high hardness. Many sliding materials were subjected to severe wear, abrasion, high stresses, corrosive or aggressive environments due to its robust construction in nature (1). Hence, wearable materials are much attentive topic to the specific researchers. Usually medium carbon steels are considerably modified by thermo mechanical processing and heat treatment for withstanding such destructive forces not only to the tool/work piece interface, but also to a substantial depth below that interface. On the other hand, tremendous research activities around the world dealt with new types of protective nano-coatings and their corresponding developments, which showed excellent wear resistant, hardness, thermal stability, fracture toughness enable them to improve tool life greatly (2, 3). These tendencies gained a massive support to the respective researchers that allowed to study micro and nano structure of coating materials. Many commercial surface modification techniques available in the market for steels namely surface implantation, plating or coating, thermo chemical or thermal treatments. Surface implantation change surface chemistry similar to thermo chemical treatments; however, surface layer implantation is considered as a separate surface treatment. Whereas in plating or coating, a thin layer of hard particles of nitrides, carbides are deposited on the surface that exhibit a little change in surface chemistry as a result tool life can be increased. A historical review of the advancement of CVD and PVD processes shows a steady difference in viable coating materials through changes in the manufacturing quality and coating strategy. Especially nano structured PVD coatings made sense of how to develop themselves accessible surprisingly quickly. Hence, manufacturing industries often searching new coating materials and their corresponding techniques involved for their own standard / customized tools, especially where high stresses, temperature or wear induced directly and indirectly, in particular, cutting tools for rapid manufacturing processes (4-7). Two different processing approaches are used in coatings to deposit a thin film layer of nitrides or carbide's
namely physical vapor deposition (PVD) and chemical vapor deposition (CVD). PVD is a plasma coating process involves a physical process of depositing thin solid layers of another material onto a surface. PVD does not require any chemical reaction to be happen to deposit a durable particle. In CVD process, required many inorganic chemicals and the surface to be well cleaned, sometime it takes more time and is not environmental friendly. Nevertheless, the main limitation of PVD process is the rate at which the deposition is relatively very slow when coating undercuts and obtaining similar surface features. Especially, if the product has cylindrical or curved features therefore complex machines, and skilled operators are required. Quality of PVD coating on a tool steels are depending on the following: chemical inertness, a good adhesion, a high hardness and high wear resistance. Attaining these facts by a monolayer of nitride or carbide coating increases the hardness, brittleness and lowers the yield strength. Therefore, a good solution to the above discrepancies is binary / ternary carbide or nitride coatings (8, 9). In recent year's combination of aluminum titanium nitride (Al-Ti-N) coatings have been deposited as binary and ternary layer systems such as TiAlV (10), TiAIN (11-14), TiCN (2), CrAlN (15, 16). Several studies confirmed the effects of coatings have much superior in enhancing the mechanical, corrosion, tribological properties and to extend tool life (17-20). However, due to their implementing difficulties they were not drawn much attention by the researchers when comparing to commercial tribological coatings (21). The major limitations of binary / ternary nitride and carbide coatings are extremely difficult, consumes more time and numerous trials to be required to obtain good surface integrity. Hence, the present investigation mainly focuses on bringing surface integrity of ASTM A681 Super nitride coating on tool steel.

2. Experimental procedures

2.1. Specimen preparation

A second-generation AlTiN supernitride coating layers was deposited onto machined premium grade ASTM A681 tool steel substrate via DC reactive PVD magnetron sputtering is used in an Ar/N₂ gas mixture. The ASTM A681 tool steel samples were cut into small size and subsequently austenitized at 1030°C for 30 min, quenched in circulating air and finally tempered at 620°C for 2 h yielding a hardness of 434±20 HV. With a specific end goal to minimize the impact of the substrate surface severity, samples were mechanically cleaned utilizing precious diamond slurry in addition to lubricant to a normal roughness (Ra) smaller than ~0.01 m before they were nitrided to a case depth of 5±2microns. A vacuum chamber was evacuated to a base pressure of 3 × 10⁻⁴ Pa and the operating pressure consisting of pure 99.99% of Ar and N₂ gases with a constant flow was set at 0.3 Pa during all supernitride coatings. A Cemecon DC sputtering with booster technology coating system (Model: Cemecon-CC800®) is used for depositing second generation super nitride coatings on all ASTM A681 tool steel samples. The nominal composition of ASTM A681 tool steel is 0.32% - 0.40% C, 5.13% - 5.25% Cr, 1.33% - 1.4% Mo, 1.0% Si, 1.0% V. All the A681 samples were rotated at 10 rpm throughout the deposition process in order to attain a homogeneous coating therefore each sample were located at a distance of approximately 160mm from the target. A 10mm holder is used to hold the samples of ASTM A681 tool steel samples for each coating at a pulsed DC bias voltage of 25V was applied during the deposition process at a temperature of 250°C. A Zeiss Sigma VP -Schottky thermal field emission electron gun scanning electron microscope (FEG - SEM) with an Oxford INCA model energy dispersive X-ray analysis (EDS) was employed to analyze the topography on both coated and uncoated surfaces of A681 tool steel. The phase structure of coated surface layers was analyzed using PANalytical X’Pert Pro MPD X-ray Diffractometer (XRD) equipped with Cu Kα radiation for qualitative and quantitative identification of crystalline phases. In order to conduct a thorough investigation, diffractometer was operated with a scan rate of 0.02° s⁻¹ was used with a grazing incidence of 0.5°.

3. Results and Discussion

3.1. Surface morphology

Figure 1(a) and Figure 1(b) show the high magnification SEM micrograph of heat treated sample of ASTM A681 samples. The samples are relatively cleaned and typically coarser, grooved with small pores as shown in Figure 1(a). On few samples, it can be observed a macro crack having adhered debris particles in plane of machined direction as presented in Figure 1(b). Figure 1(d) show the XRD result
confirm no contaminations on the surface to be coated and the presence of great amount of chromium oxide, iron oxide on the heat-treated samples. The surface morphology of coated and heat-treated ASTM A681 tool steel was characterized by using Nanosurf© - compact Atomic Force Microscopy (AFM) (Model: NaioAFM). AFM is best suitable for measuring the thin film coatings for structural analysis on the nano scale also used to study the mechanical properties of the coatings with a variation of loads.

Figure 1. Heat treated ASTM A681 tool steel samples (a). Grooved, (b). macro cracked

Figure 2. AFM image of heat treated A681 sample

Figure 3. Coated ASTM A681 tool steel samples (a). Macro droplets of TiN Compounds, (b). Macro droplets of AlTiN compounds

Figure 4. AFM morphology of AlTiN Coated sample
Figure 2 show AFM topography of heat treated ASTM A681 steel that seems machined wafer surface include narrow machining streaks consequences a coarse surface finish. The surface roughness of heat treated samples was in the range between 270 μm to 290 μm. In addition to that, it can be observed that the coating was inconsistent due to low agglomeration as a result a dotted wafer coated surface exhibited on the coating surfaces. The magnitude of change in color of different regions is due to variation in signal with depth which corresponds to the height difference and surface roughness of each sample. therefore, an average surface roughness was considered to understand the effective coating roughness in each coating. The coating surface has less uniformity, micro holes and macro droplets were enclosed by TiN intermetallic compounds as shown in Figure 3(a) and Figure 3(b). Consequently, the surface roughness of 208 μm was observed irrespective of all samples when fabricated by AlTiN coatings. At last, usually wear resistant coatings onto tool steels consequences substantial increase of titanium, oxides of iron, silicon and chromium substrates. Therefore, XRD analysis was performed on the all layers coating samples, for instance, AlTiN coated and heat-treated sample results were graphically presented in Figure 5. The obtained results confirm the presence of addition of titanium, oxides of iron, silicon and chromium substrates. The coating surfaces display heterogeneities covered by debris are in the form of elongated micro-particles which are originated probably due to splattering on the substrate surface during coating process. With this it was concluded that, the adhered nano composite particles (Ti,Al,N) have significantly increasing micro hardiness over the surface layer and contributing to decrease in wear intensity of ASTM A681 tool steel.

![Graphical representation of XRD pattern of coated AlTiN sample](image)

**Figure 5. XRD pattern of coated AlTiN sample**

### 3.2. Coating analysis

The hardness (H), elastic modulus (E) and indentation load (P) is a displacement function of coated and heat-treated samples of A681 tool steel. For measuring these parameters Berkovich tip indenter showing a face of 78.9 deg is used. A graphical representation of the elasto-plastic indentation of heat-treated and coated samples are presented in Figure 6(a) to Figure 6(b). \( h_c \) is the contact depth, \( h_t \) is the final depth of the contact impression after unloading, \( P \) is the indentation load, and \( b_{max} \) is the indenter displacement at peak load. The area \( A_1 \) (OBC) represents the plastic work done in the viscoelastic-plastic segment and the area \( A_2 \) (CBC) represents the elastic work recovered during the unloading. If the material has completely plastic, the unloading curve is a straight line (BD) and \( h_{max} = \) therefore \( A_2 = 0 \). \( S \) is the contact compliance and \( P_{max} \) is the peak load. It can be seen that, the area of \( A_1 \) is less when compared to area \( D_1 \) which reveal the coated sample relatively low displacement compared to heat treated sample. The hardness of the heat treated and coated was determined using nano indenter at several points. Six different loads were applied while testing: 5.91N, 15.8N, 19.7N, 30N, 35N and 39.7N. The average hardness values were calculated and plotted against load for all coatings and heat-treated samples.
Figure 6 (a) and Figure 6(b) elasto-plastic indentation of heat-treated and coated samples
The young’s moduli of heat treated and coated samples were presented graphically in Figure 7(a). The average young’s modulus of 200 GPa was observed in AlTiN coatings whereas 170 GPa was observed in heat treated samples. It is observed that, the young’s moduli of all coatings were high when low indentation load whereas it has low at high indentation load. It is due to the variation in the coating substrate thickness around the periphery of each sample. Figure 7(b) show a plot of average hardness of all coatings and heat-treated sample. It is observed that the samples coated with AlTiN exhibited high average hardness value of 13.47 GPa with an average load of 24 mN whereas 10.21 GPa with an average load of 16 mN was observed in heat treated samples. Similarly, the maximum penetration depth of all coatings and heat-treated samples is presented in Figure 7(c). It is observed that an average penetration depth of 422.70 nm and 480.833 nm on AlTiN coatings and heat-treated samples respectively. The lowest penetration depth of 201nm was observed in AlTiN sample. In general, the penetration depth is a function of applied load of the coated sample. Therefore, as the load increases to 5.91N to 19.7N, penetration depth is increases with decrease in hardness of the all coatings irrespective of type of coated substrate. In addition to that, based on the hardness readings observed, the hardness of the all coated samples more or less same and no significant hardness improvement can be seen in any samples when increasing load. The increase in hardness was related to utilizing of multilayered materials with nanometrics thickness kept over mechanical steel substrate. The nanometric multilayer’s of AlTiN have a microstructure subject to the measure of the bilayer period, which decides the improvement of the mechanical properties. Similar observation was observed by Noorakma et al (22) in hydroxyapatite coated magnesium based bio-degradable alloys. The hardness increased from substrate to outer surface of the coated samples.

**Figure 7** (a). Young’s modulus, (b). Hardness, (c). Penetration depth of AlTiN coated and heat-treated samples
Figure 8(a) Result of coefficient of friction of AlTiN coated sample

Figure 8(b) Wear result of AlTiN coated sample
3.3. Tribological properties

Tribological performance of the coatings were carried out using pin on disc method on the Ducom tribometer at room temperature. All coated samples were mechanically polished after deposition of coating using a comparable level. The substrates of coated sample of all multi-layer systems were tested at room temperature against steel disc and the obtained coefficient of friction and wear rate results of
coated and heat-treated samples were presented in Figure 8(a) – Figure 8(d). Test load and sliding speed 2N and 0.1m/s were given in order to establish the initial contact between pin and disc. The initial Coefficient of Friction (CoF) is begun at low level 0.3 was observed in each coated and heat-treated surface. The dependency of friction coefficient was evaluated on the number of cycles with the standard number of cycles 12000 for AlTiN coatings and heat-treated samples. The heat-treated samples have characterized run with rapid increase of the CoF value, while the AlTiN coating samples comparatively steady wear regime after about 2200 cycles with noticeable fluctuation. The rapid increase of wear rate of the pin, wear debris around the pin stimulated until the friction reached a steady-state. Using wear track profiles, the wear rate of the coatings was calculated. Each coating wear rate was estimated at room temperature using the formula, \( w = V / (F \times S) \), where \( V \) is wear volume in mm\(^3\), which was evaluated according to the cross-sectional area of the wear track, \( F \) is load in N and \( S \) is the sliding distance in m. Volume of the material removed on the wear track during the tests were measured to estimate wear rate of each coating. In addition to that, built up edge is also to be considered for estimating the wear of the coatings, since, built-up edge is the growth of material around or in the wear track. However, no significant built-up edge can be seen around the wear track. The estimated wear rate is presented graphically in Figure 9. The highest wear rate can be observed in heat treated samples. Wear rates of coating and heat-treated samples such as AlTiN and heat-treated samples are 180 and 453 m3/N/m × 10\(^{-17}\).

![Figure 9. Wear rate of AlTiN and heat-treated sample](image)

4. Conclusion

A Second generation AlTiN based coatings characterize the state-of-the-art for PVD hard coatings used in industrial machining applications. However, new difficulties primarily in the field of metal cutting in stamping and framing applications require constant enhancements. Other than mechanical and physical properties, oxidation protection and the affinity between the workpiece material and the cutting tools are essential for further advancements. These properties can be affected from one viewpoint by different coating parameters and coating innovations yet additionally by a specific chemical composition of the elements utilized for the coatings. As this coating is quite new in the cutting tool market further machining tests have to be done to define the potential for this type of coating.

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