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Influence of Substrate of the Carbon Contents and Coating Thickness on Scratch and Wear Resistance of AlCrN Films

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Abstract Influence of carbon content substrates and different coating thickness Aluminium Chromium Nitride (AlCrN) coatings was investigated and reported in the studies. Low carbon steel (EN353) and high carbon steel (EN31) rectangular blocks were used as substrates. AlCrN coatings with two different thickness was deposited on these substrates using Balzers rapid coating system machine. The morphology, crystal structure, mechanical and tribological properties (surface hardness, wear resistance and coefficient of friction (COF)) of the coatings were examined using SEM, Optical Microscope, AFM analysis, Micro-Hardness tests, Scratch Tester TR-101 and Pin on Disc testing tribometer at atmospheric conditions. It was shown that surface morphology of D C Arc deposited AlCrN coatings is affected by the substrates properties (carbon content). The increase in the carbon contents of the substrates resulted in the increase of adhesion force between the substrates and coatings. It was also found that, AlCrN/EN31 steel with smooth roughness has the higher wear resistance than AlCrN/EN353 steel substrate.

Keywords: *AlCrN coatings, SEM, Optical microscopy, AFM analysis, Micro-Hardness test, Scratch test, Pin on disc test*

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1. Introduction

In many industrial applications, the most common requirement is of enhanced tribological properties of the machine components viz. high surface hardness, good wear resistance and low coefficient of friction [1]. One of the techniques which can be used to obtain enhanced surface properties is hard coating on the machine components. Hard coatings have success story in various mechanical cutting applications, due to their noteworthy properties such as high hardness, high melting point, wear resistance etc., The hard coating layer plays a very important role in surface engineering as it reduces the friction between two mating surfaces by eliminating direct contact of the surfaces [2,3]. From early 1970s onwards, the Titanium Nitride (TiN) coating played an important role in the field of surface engineering due to its hardness of over 20GPa [4,5]. However, it can be noted that, nowadays a wide range of Physical Vapor Deposition (PVD) hard coatings are available for variety of applications. Recently, researchers studied and reported that, Aluminium Chromium Nitride (AlCrN) is one of the most representational commercial coatings available. As it exhibits excellent adhesion, hardness, high oxidation resistance at high temperature, good thermal stability and better resistance to chemical breakdown than Titanium Aluminium Nitride (TiAlN), Titanium Nitride (TiN) and Titanium Carbo-Nitride (TiCN) coatings. Furthermore, AlCrN coatings have been used to enhance the properties of cutting tools, bearing spindle and many other high-performance mechanical components [6,7]. Moreover, many recent publications have proven that AlCrN coatings possess superior wear resistance and low coefficient of friction than those of the TiN and TiAlN coatings [8,9,10,11].

Researchers have studied and analyzed a series of Aluminium Chromium Nitride coatings with different substrates. Chawla et al. [12] concluded that AlCrN coating exhibits better corrosion resistance in comparison to TiAlN coating. Liew [13] reported that under vacuum, using ball on disc machine, TiN produced lower COF than AlCrN whereas in atmospheric air AlCrN exhibited lower COF than TiN with high debris retention on the sliding interface due to the effect of oxidation. Grzesik et al. [14] observed that coefficient of friction decreases with the increase in normal load as a typical behaviour of steels. Also, it is reported that, TiAlN/cast iron produced lower COF compared to TiAlN/stainless steel and TiAlN/carbon steel. Mo et al. [15] concluded that the AlCrN coating exhibited lower COF and wear rate compared to TiAlN coating under same testing conditions. Muthuvel et al. [16]

concluded that the coating on the texture surface exhibits higher sliding distance to reach the steady state in coefficient of friction at all normal loads compared to the coating on the lapped surfaces due to prolonged existence of AlCrN film inside the dimples.

In most of the mechanical industries, EN-353 low carbon steel is widely used because of its good ductility and weldability. However, this material has poor tribological properties such as high coefficient of friction, low wear resistance, and low hardness [17]. Two methods are most commonly used to improve the steel of tribological properties. First step, in steel adding alloy elements during smelting of the integral alloy and second step, surface modification techniques. The former is impractical because number of interstitial elements doped into the steel is harmful to ductility and increase the cost of products. Thus, in recent years the most popular research fields, surface modification became an most effective and economical method. The improvement of tribological properties, most commonly using surface modifications of steels such as PVD and ion plating. An advantage of the technology is that the coatings are metallurgically bonded to the substrate with good interface. This technology for complex shaped substrates also exhibits controllability of coating thickness, high deposition rate and good coating uniformity.

The most popular technique is to investigate the adhesion strength of the thin coating on substrate via scratch test method. In scratch tester where in a normal load applied on the coating surface is stepwise increased and load, at which coating detached adhesively, is called as the critical load failure. To investigate wear behaviour of a coating material under different stresses, this test can be used. The strength of adhesion is influenced by following many factors effect on the critical normal load value: the substrate hardness, the coating thickness, the surface quality, the coating hardness, the loading rate, interface bonding, indenter dimensions, and the friction between the indenter and a coating.

Evidently, to the best of the knowledge of the authors, influence of carbon percentage of substrates on the structure and mechanical properties of AlCrN coatings have not been observed systematically studied. Hence, the influence of a different carbon percentage of substrates on the structure and mechanical properties of AlCrN coatings has been investigated. In this work, an effort has been made in that direction. AlCrN coating was deposited on the low carbon steel (EN353) and high carbon steel (EN31) blocks with coating thicknesses of ~2µm and ~4µm using commercially available PVD method at Oerlikon Balzers Coating India Limited, Pune, India. Influences of different carbon percentage of substrates were investigated at atmospheric conditions using a scratch tester (adhesion and wear mechanism) and pin on disc testing tribometer (COF and wear rate) with various operational conditions.

2. Materials and Methods

2.1. Materials

The flat specimens for scratch test experiment were machined with dimensions of 60x25x10 mm and for pin on disc experiment the cylindrical specimens of diameter 10.0±0.1 mm and length 28±0.1 mm with working surface roughness of Ra: 0.32 µm for low carbon steel (EN353) and high carbon steel (EN31) substrates have been used. The flat specimens were polished on one surface by progressively using emery papers of 150 to 1000 grit sizes, and the other surface is machined for scratch testing. The cylindrical specimens were polished on both sides using emery papers for pin on disc test. The chemical composition of EN353 and EN31 steel substrates are shown in Table 1.

Table 1. Chemical composition of EN353 and EN31 steel

Material	C	Mn	Si	Ni	Cr	Mo	Fe
EN-353	0.2170	0.57762	0.1895	0.0317	0.05306	0.004590	Bal.
EN-31	1.0223	0.4536	0.3136	0.0779	1.20730	0.02430	Bal.

2.2. Deposition Method

Prior to deposition, substrates were initially degreased in ultrasonic bath to remove excessive oils and greases. This was followed by cleaning with alkaline solutions with ultrasonic and rinsing with Demineralized Water (DM). Final stage of cleaning included drying with hot air blower. Cleaning of these parts was carried out using Oerlikon Balzers proprietary (standard) cleaning procedure.

AlCrN coating was carried out with Arc evaporation method using Oerlikon Blazers commercial coating process. Arc evaporation of AlCr targets was carried out in presence of Nitrogen to form stoichiometric AlCrN films. The thicknesses of the AlCrN coatings were approximately 2±0.3 µm and 4±0.2 µm. Coating was carried out at temperature of about 500±10ºC with nitrogen as reactive gas. A DC-substrate bias voltage was maintained in the range of -50 to -150V during coating. Figure 1 shows the microstructure for the AlCrN coatings of different thicknesses in as-coated condition.

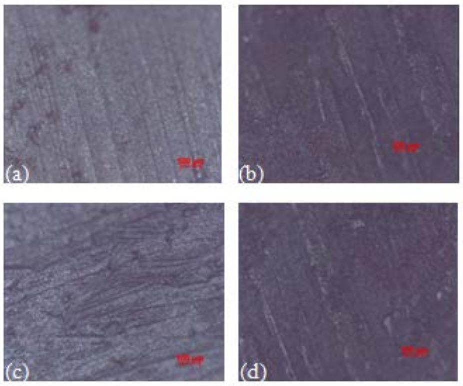

Figure 1. Optical microstructure images (100µm) of AlCrN coated surfaces of (a) 2 µm on EN353, (b) 4 µm on EN353, (c) 2 µm on EN31 and (d) 4 µm on EN31

2.3. Coating Characterization

Micro-hardness measurements for the compound system of substrate + coating were carried out (as per the IS 1501-2002 procedures) by using Vickers hardness tester (MH6). The microhardness of the coatings on steel was measured using a MVH-1 (METATECH) Microhardness tester, applying a load of 10, 25 and 50gms and a dwell time of 15 seconds. A conventional scratch tester set-up (DUCOM TR-101 M4 scratch tester) was used to determine the scratch resistance and behaviour of the deposited thin coating on the substrate. The radius of the diamond indenter was 0.2mm. The test was carried out under the progressive load range of 10-200 N, 10 N/mm of loading rate, sliding speed of 1.0 mm/sec and the scratch length was around 14 mm. The scratch tester plots/records the coefficient of friction, applied normal load and traction force, simultaneously. Sliding wear tests were carried out in a DUCOM TR-20 LT pin on disc machine tribometer to evaluate the friction and wear behaviour of the coated specimens. All tests were carried out at room temperature, ambient humidity and under dry sliding conditions.

AISI 316 Austenitic stainless steel disc was used as a counter body. The pin diameter was 8mm and the disc was made of high chromium high carbon tool steel. The loads applied on the specimens are 5N and 10N at the sliding speeds of 0.5 and 1 m/sec. The wear rate was calculated by measuring the weights of a specimen before and after the test.

Zeiss Axiovert 200 MAT inverted optical microscope, loaded with image software Zeiss Axiovision Release 4.1, was used to investigate the morphologies of wear scars left behind after each scratch and wear tests. The porosity measurements were made with image analyzer, loaded with software Dewinter Materials Plus 1.01 based on ASTM B276. PMP3 inverted metallurgical microscope was used to obtain the images.

Scanning electron microscope (SEM-JEOL, Model-JSM 6380) was used to characterize the surface morphology of the coatings. SEM micrographs were taken with electron beam energy of 15keV. The surface morphology (2D and 3D) of the thin coatings was characterized by AFM (Innova SPM Atomic Force Microscope) to calculate the surface roughness and particle size.

Table 2. Vicker's hardness of AlCrN coating on EN353 and EN31 steel at various loads

Samples (Thickness)	Load (gms)		
	50gms	25gms	10gms
EN353 (2μm)	634	1034	1094
EN353 (4μm)	718	1102	1216
EN31 (2μm)	1560	2779	2886
EN31 (4μm)	1812	2984	3252

3. Results and Discussions

3.1. Micro-Hardness

Figure 1 (a-d); shows the optical microstructure images (100μm) of AlCrN coated surfaces of 2 μm & 4 μm on EN353 and 2 μm & 4 μm on EN31 respectively. The corresponding Vicker microhardness values for two different thickness coatings under 50, 25 and 10 gms. are listed in Table 2. AlCrN deposited on high carbon steel specimens' display a higher compound hardness value in comparison to AlCrN on the low carbon steel specimen.

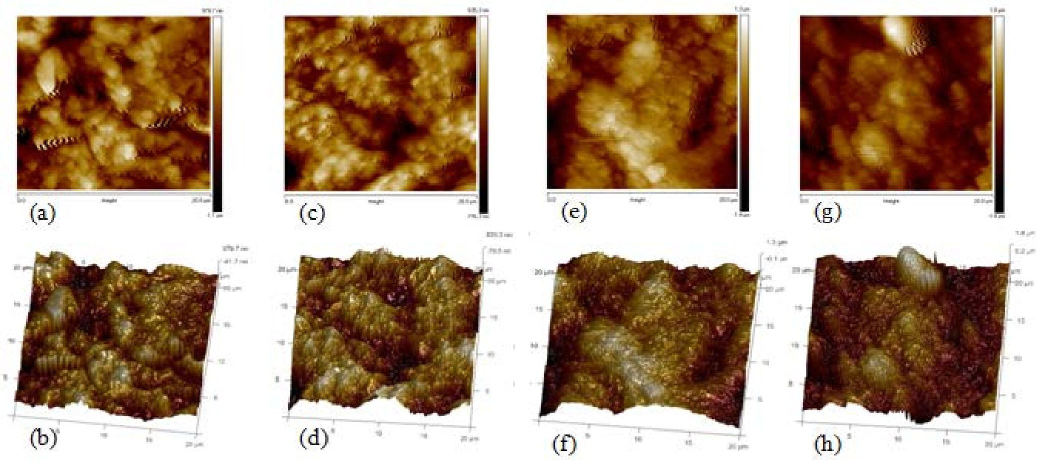

Figure 2. 2D and 3D AFM images of AlCrN coating deposited on EN353 (2μm (a, b) & 4μm (e, f)) and EN31 steel (2μm(c, d) & 4μm(g, h)).

3.2. AFM

Figure 2(a-h) shows the variations in AFM surface morphology (2D and 3D) of AlCrN coatings for two different thicknesses deposited on low carbon steel and high carbon steel specimens. The difference in morphology of these two different thicknesses substrate coatings can be observed by comparing the 2D images in Figure 2(a), (c), (e) and (g). Moreover, a clearer comparison of these coatings can be recorded by viewing 3D images in Figure 2(b), (d), (f) and (h). With increasing substrate carbon content and coating thickness, the surface of the coatings became smoother and smoother, which indicates that coatings on substrates have higher compact structure. As the axis scale indicates the overall roughness value, referring to Figure 2(h) one can note that, the high carbon steel substrate with 4 μm AlCrN coating has smoother surface in comparison to other substrates (see Figure 2 (b), (d) and (f)). Lower roughness is exhibited for the case of low carbon steel substrate with thicker coatings and structures have finer crystallites and coatings have less compact structure. The particle sizes of the coatings measured by AFM Analysis are presented in Table 3. It can be observed from Table 3 that, the 2μm of AlCrN/EN31 specimen have the larger particles (320 nm) as compared to 2μm of AlCrN/EN353 (235nm), 4μm of AlCrN/EN353 (165nm) and 4μm of AlCrN/EN31 (308nm) specimen [18,19,20]. This may be due to the presence of Cr.

Table 3. Comparison of the particle sizes of the AlCrN coatings obtained using AFM analysis

Type of Coated samples (Thickness)	Surface Roughness			Appearance of surface features
	Arithmetic mean roughness (nm)	Maximum height (nm)	Point mean roughness (nm)	
EN353 (2μm)	235	1034	1094	Irregularly spaced peaked features
EN353 (4μm)	165	1102	1216	Irregularly spaced peaked features
EN31 (2μm)	320	2779	2886	Irregularly spaced peaked features
EN31 (4μm)	308	2984	3252	Irregularly spaced peaked features

3.3. Scratch Testing

The normal load at which the detachment of the coating from substrate occurs is referred as the critical load value and is used as the measure of bonding strength. The ramp incremental load was applied over the thin coating substrates by a spherical diamond indenter. The coating adhesion increases with increase in substrate carbon content and coating thickness. Figure 3 and Figure 4 correspond to the coefficient of friction and traction force recorded during the scratching with respect to the stroke length. The variation of the coefficient of friction vs stroke length for different coating thicknesses and carbon contents of the substrates is shown in Figure 3. It was noticed that in the case of low carbon substrate with a higher coating thickness, the coefficient of friction increases at the beginning and then decreases at 0.4mm scratch length. In case of coating on low carbon steel and smaller coating thickness, the coefficient of friction along the stroke length were all higher than those of the other substrates and almost the same is observed by Valli et al. [21]. As it was pointed out that the coefficient of friction gradually increased along stroke length as the ramp load increased and the mode of failure changed from cohesion related to adhesion related. It shows that, by increasing the carbon content of the substrates, the scratch coefficient of friction decreases. The higher carbon content substrate and coating thickness specimen bear lesser traction force during the scratch test as shown in Figure 4. Larger indentation stress is needed for the higher carbon substrate and coating thickness, which makes the sufficient compression to delaminate the coating under the effect of indentation stress and the shear stress.

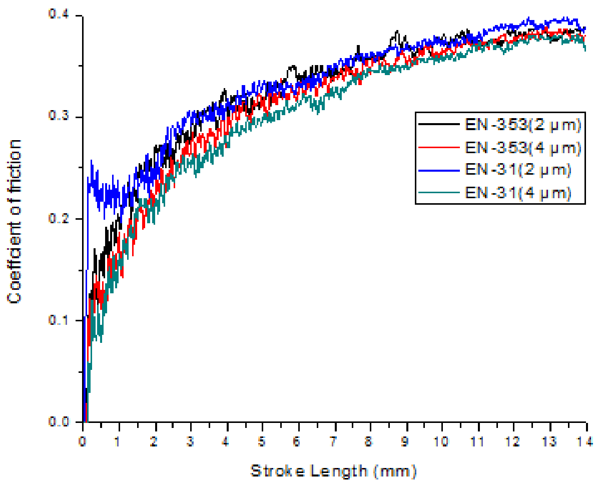

Figure 3. Variation of coefficient of friction vs stroke length for different coating thicknesses and carbon contents of the substrate

Therefore, the critical load value is higher, when the coating thickness and carbon content in substrate is higher. Many authors have reported that the surface roughness and coating thicknesses improve the bonding properties of the coatings [22,23,24]. Therefore, the results indicate that the best adhesion will be achieved at higher carbon content of the substrate and coating thickness, which deduces the adhesion force between the coating and the substrate is affected by the morphology of the substrate carbon content and thickness of the coating.

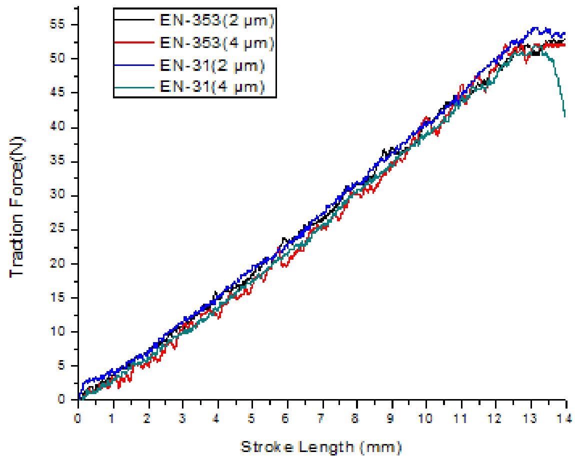

Figure 4. Variation of traction force vs stroke length for different coating thicknesses and carbon contents of the substrate.

The scanning electron micrographs of tracks in the AlCrN coatings on the low and high carbon steel substrates, each with two coating thicknesses are shown in the Figure 5(a-d). These show cohesive and adhesive chipping events, at the edges of track Figure 5(a). The coated substrates showed cohesive and adhesive failure rather than stress caused by the cracking was enough to remove the coatings. This is observed by the tearing action at the edges of the scratch. The scratch revealed the presence of progressively increasing debris appearing within the plastically deformed edge of the track and adhesive chipping at the cannel edge corresponding to a load of around 13N, which indicates the tensile cracks, i.e. burnished with cracks perpendicular to the scratch direction, and appearance with a typical fracture at the sides (see Figure 5(b)). This kind of behaviour is observed only when hardness of coating is much higher than the substrate [25, 26]. At both edges of the track chipping observed (Figure 5(c)). It showed that the spalling damage on the track and the ramp load generate the chips of coating outside of the track, which proves the partial loss of the cohesive property of the coating. The conformal tensile cracks showed good adhesion coating. For the ramp load it is observed that no clear delamination between the coating and substrate as shown in Figure 5(d). Around 16N load, no severe wear was found for the AlCrN coating with high carbon steel substrate and coating thickness. This worn out track shows that, only edge plouging, i.e. adhesive failure occurred. There was small amount of debris on the scratched surface or side of the track. Under these conditions coefficient of friction and traction force were found to be low. It indicates that some of the scattered debris of the coating material still adhered to the substrate at around 3.5mm scratch length. Examination of track confirmed the good wear resistance [27,28,29].

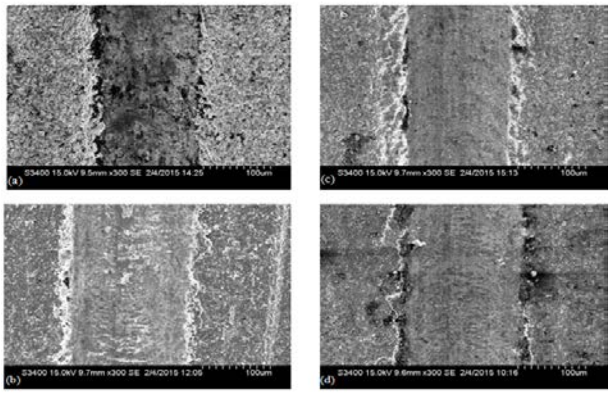

Figure 5. SEM scratch test image of AlCrN deposited on (a) EN353/2μm; (b) EN353/4μm; (c) EN31/2μm and (d) EN31/4μm

During the scratch test of coated high carbon steel substrates, there was no delamination in coatings, which may be due to its yield strength or higher microhardness, surface coefficient of friction and coating thickness. Comparing coated with low carbon substrates (Low carbon content coated substrates) exhibited higher coefficient of friction under the ramp loads.

3.4. Friction and Wear Behaviour

In case of pin on disc tests, the coefficient of friction values were obtained at two load and speed conditions with AlCrN coatings on high carbon steel and low carbon steel substrates under dry conditions. The peak values of COF Figure 6 are found to be around 0.35 for lower carbon steel and smaller coating thickness, and for higher carbon content steel and 0.24μm for the higher coating thickness. It can be noted that the coefficient of friction values are different for the AlCrN coatings with deposition thicknesses and different carbon contents of the substrates. It can be clearly seen that the lower thickness and carbon content specimen exhibits slightly higher coefficient of friction than other coated substrates. This is due to, the frictional behaviour of the deposited layer that was strongly affected by its mechanical properties(i.e. as the hardness increases the coefficient of friction decreases) [30,31].

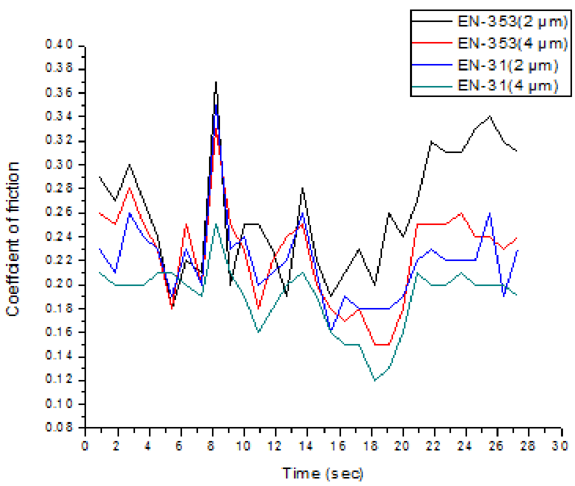

Figure 6. Variation of coefficient of friction vs stroke length for different coating thicknesses and carbon Contents of the substrate.

Figure 7 shows the wear rate of substrates with two different thicknesses of AlCrN coatings obtained using the DUCOM wear test machine. The 2μm/EN353 substrate showed the highest wear rate, followed by other substrates. The AlCrN with 4μm thickness coated on high carbon steel substrate shows the drastic reduction in the wear rate. From Figure 7, it can be observed that, AlCrN coated on lower carbon substrate with higher coating thickness was found to be an effective material to reduce the wear rate than smaller coating thickness substrates. However, the wear resistance of higher carbon content and coating thickness (smoother surface) was even better than that of lower carbon content with higher coating thickness.

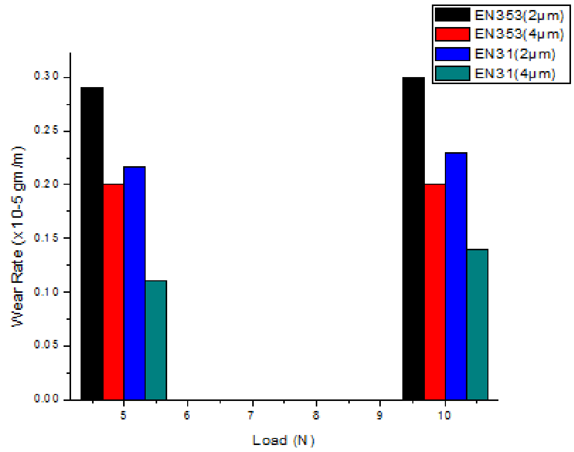

Figure 7. Measurement of wear rate for different coating thickness and carbon contents of the substrate

In case of lower carbon steel with small coating thickness, the coefficient of friction increased because of the wear debris generated during the wear process, which indicates severe wear in the coatings. It shows that the coated higher carbon substrate is about 2 times more wear resistant than the lower carbon substrates. Lesser coating thickness with carbon substrates shows higher wear rate than higher carbon and coating thickness. It demonstrates that AlCrN coated higher carbon content and smoother surface substrate presents better wear resistance when compared to lower carbon content steel substrates, due to its superior coated surface hardness [32,33,34]. In general, the wear rate depends on the coating thickness and carbon content. Here, we can derive that higher content of carbon and smoother coatings possess excellent tribological properties, i.e. lower wear rate and low coefficient of friction.

To examine the wear properties, the microstructure of the coated worn out surface of the substrates were analyzed by optical and SEM microscopy (Figure 8). Repetition of experiment was observed that the substrate coating got peeled off for low carbon steel with higher coating thickness and the wear debris formed due to plastic deformation (Figure 8(a, c and e)). As noticed from Figure 8 the worn out surfaces showed mainly abrasive and partially adhesion mechanism. However, the AlCrN coating on high carbon and coating thickness substrate showed mild plough wear region (Figure 8(b, d and f)), which indicates an adhesion mechanism with strengthened wear resistance [35]. The worn out surface images by SEM and optical (Figure 8) revealed that the surface has a little damage during the pin on disc test. Morphology of scratched wear surface of high carbon steel is shown in Figure 8(b). The pattern of excessive wear can be seen in the coated substrate specimen, as shown Figure 8(c). It shows that the worn out surface of asperities coating ejected out of contact surface was indicating that wear of the AlCrN shows high in coefficient of friction results. Whereas less wear becomes evident in the coated substrate after the travel distance of 30m, as shown in Figure 8(d), which may be due to low debris removal efficiency. The wear morphologies of the AlCrN with high thickness coating on high carbon steel was found to be very different from that of low carbon steel coating. The tribological behaviour of the AlCrN coating with Low carbon steel worn out surface exhibited large amount of debris removal and at the edge of the pin surface the debris was removed directly from the contact surface. In case of AlCrN coating with high carbon steel, there was thick oxidized debris layer formed and due to this a small amount of debris removed from the worn out surface. The behaviour of debris removal was the main difference between these coated substrates.

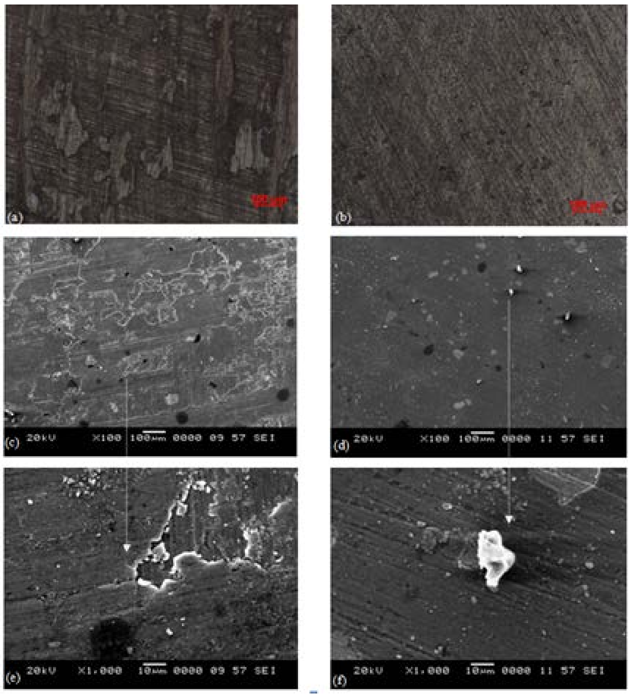

Figure 8. Wear test image of AlCrN deposited on (a) Optical of EN353/4μm; (b) Optical of EN31/4μm; (c) SEM of EN353/4μm; (d) SEM of EN31/4μm; (e) SEM of close-up view of EN353/4μm and (f) SEM of close-up view of EN31/4μm

4. Conclusion

In this work, AlCrN thin coatings were successfully deposited on EN31 and EN353 steel substrates with ~2 and ~4μm coating thicknesses by PVD arc technique. The mechanical properties and structure of AlCrN coatings on different carbon content substrates with two types of coating thicknesses have been systematically studied. The microstructural morphologies were examined in the present study. AlCrN coating is mainly affected by the carbon content of the substrate and coating thickness. The AFM studies showed that the coatings with high carbon content substrates have smoother surface than low carbon content coatings. The carbon content substrates effect the adhesion force of the coating-substrate system. It can be concluded that the good adhesion will be achieved at carbon content substrates. The wear test results showed that the high carbon content coated substrates exhibit adhesive wear which shows the abrasive particles. Whereas, the low carbon content coated substrates showed completely abrasive mechanism. Wear test shows that the coatings with high carbon content surface have better wear resistance.

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