Experimental Study of Wear Performance of Tool Steel Undergone DUPLEX Surface Treatments for Hot Forging Applications

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Abstract

In recent years, the DUPLEX surface treatment, consisting of vacuum gas nitride followed by physical vapor deposition (PVD) coating, has earn a lot of interest for enhance the wear performance of high temperature forming application, especially hot forging. In this work the wear performance of hot forging tool steel grade AISI H13 undergone DUPLEX treatments with different top coating have been investigated. Three types of PVD coatings, e.g. TiAlN, AlCrN and AlCrTiN, were studied. The microstructure, physical and mechanical properties and surface roughness were also investigated. The wear tests were performed using a high temperature pin-on-disc arrangement at the working temperature of the hot forging tool, i.e. 300°C. The experimental results indicate that the wear behavior is strongly influenced by the level of contact stress developed at the surface. At low contact stress, harder coating shows the better wear performance while, at high contact stress, the tougher coating serves better. Examination of worn surface suggests that this might be caused by change in failure mechanism from ductile fracture to a more brittle manner at high contact stress for hard material. The understanding of the influence of working conditions and coating properties on the wear resistance of tool steels obtained from this work can be very useful in the improvement of wear performance in hot-forging tools and also other high temperature applications.

Keywords*: DUPLEX surface Treatment; Hot-forging; Wear; Nitride Coatings; PVD Hard Coating*

1. Introductions

Hot forging is one of the most common manufacturing processes. Hot forging tool is subjected to repeat mechanical and thermal loading which finally lead to severe damage of tool surface due to plastic deformation, thermal and mechanical fatigue [1]. The most popular hot-working tool is the AISI H13 steel owing to its high toughness and resistance to softening. Surface treatment has been used to improve the forging tool material performance. One of the most popular treatments employed in industries is gas nitriding producing a surface hardened material with the hardened depth as deep as a few hundred micrometers [2-3]. In recent years, a development of process which comprises the sequential application of two or more surface treatments producing a surface composite has been great interest. This is so called as DUPLEX treatments [4-6]. Many studies have been done on these DUPLEX treatments including one involves nitriding process followed by physical vapor deposition (PVD) coatings. Such DUPLEX treatment combines the advantages of both processes leading to improved tribological properties, i.e. high wear resistance, low friction, at the top surface with higher surface hardness to the depth of a few micrometers deep.[8-12] However, at present, DUPLEX surface treatments have not yet been extensively employed in the manufacturing industries due to insufficient information on the material behaviors under different service conditions. In this work, the propertied of forging tool steel (AISI H13) undergone the DUPLEX surface treatment, i.e. mechanical, adhesive and high temperature wear properties, have been investigated. The treatments being studied include gas nitriding process followed by three different high temperature resistance PVD coatings, e.g. TiAlN, AlCrN, and AlCrTiN.

2. Experimental Procedures

2.1 *Preparation of specimens*

AISI H13 discs (56 HRC, diameter 25 mm, thickness 5 mm) were cut from a cylindrical bar. The nitriding process was carried out by means of Vacuum Gas Nitriding which increases the surface hardness to 1100 HV. After nitriding process, all specimens were grinded and polished to remove the white layer. The polished specimens have the mean surface roughness, R_a, of 0.402 µm (measured by Taylor-Hobson Profilometer). Three types of PVD coatings e.g. TiAlN, AlCrN and AlCrTiN were deposited on nitride specimens.

2.2 *Experimental Details*

The thickness and surface roughness of all specimens after DUPLEX treatments were measured using Calotester and Taylor-Hobson Profilometer, respectively. The cross-section of specimens were also examined using optical microscope to investigated the coating layer and the depth of diffusion zone resulted from the nitriding process. Before optical microscope investigation, all specimens were polished and then etched with 4% Nital acid. Vickers micro-hardness testes were performed at the load of 50 gf. on cross-sectioned specimens at different depths under the surface up to a depth of 120 µm. Nano-indentation tests were also carried out on the top surface at the load of 20 mN with the loading rate of 40 mNmin^{-1} and the dwell time of 15 s to determine the surface hardness and Young's modulus of the coatings. The adhesions of all coatings were studied using CSM scratch tester. During the scratch tests a diamond indenter was drawn across the surface under progressive load from 0.9N to 150 N at the loading rate of 10 N min^{-1} with the table speed 10 of mm min^{-1} and the total scratch length of 15 mm. The acoustic emission signal and the reported coefficient of friction with the optical microscopic image were used to determine the adhesive properties of the coated system. The ball-on-disc arrangement was used to determine the wear resistance properties at high temperature of the coated specimen. The tests were performed at 300 ˚C (forging tools temperature [10]) with WC ball with the diameter of 6 mm. The wear tests were investigated at different load i.e. 1N, 5N, 10N, and 15N with the linear speed of 10 cms^{-1} with 150 m total sliding distance. The volume of the material worn off from the disc surface under different load was calculated. The wear tracks were studied to investigate the character of the each individual worn surface using optical microscope.

3. Results and discussion

3.1 *Characteristic of DUPLEX coatings*

The DUPLEX coating thickness and roughness are shown here in Table1 together with the surface hardness and Young's modulus. The average value of indentation depth was estimated to be approximately 215 nm, 190 nm and 195 nm for TiAlN, AlCrN and AlCrTiN coating, respectively. The effect of substrate on the surface hardness of the coatings is minimized at such indentation depth to coating thickness ratio. All coatings were found to be very hard with the surface hardness greater than 30GPa with the AlCrTiN showing the highest hardness of approx. 34 GPa.

Table 1: Average surface roughness (R_a), the average thickness (t), surface hardness (H) and Young's Modulus (E) of the top coating.

Type of coatings	t (µm)	R_a (µm)	$H_{0.02}$ (GPa)	E (GPa)
DUPLEX AlCrN	1.80	0.532	32.4±3.6	513±48
DUPLEX AlCrTiN	2.62	0.508	34.1±3.2	431±51
DUPLEX TiAlN	3.52	0.441	31.0±2.1	352±27

The micro hardness of the coated specimen at the different depth under the surface is shown here in Figure. 1. It can be seen that, for all DUPLEX coatings, the maximum hardness of 1200HV was observed for all nitride layer. The value of hardness was then found to dropped to approx. 650 HV at the depth of 110-120 µm. causing by the greater influence of softer substrate. This is consistent with the observation shown by the optical microscopic images of specimens' cross-sections shown in Figure. 2 which indicate the diffusion layer of the thickness of 110-130 µm.

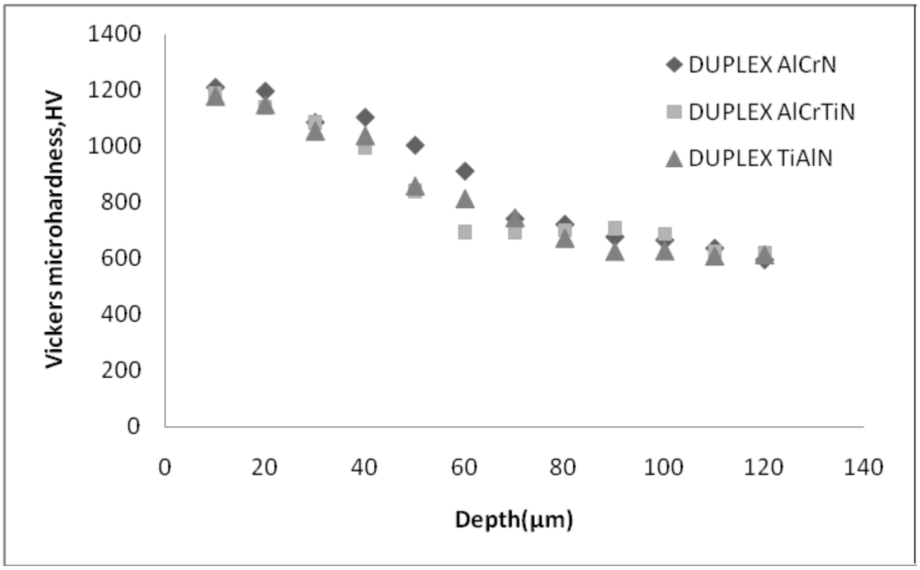


Figure 1: Microhardness depth distribution for DUPLEX specimen s

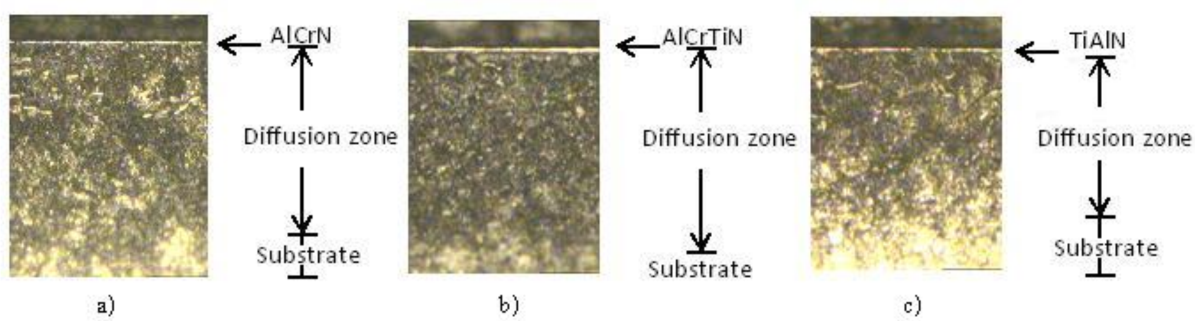


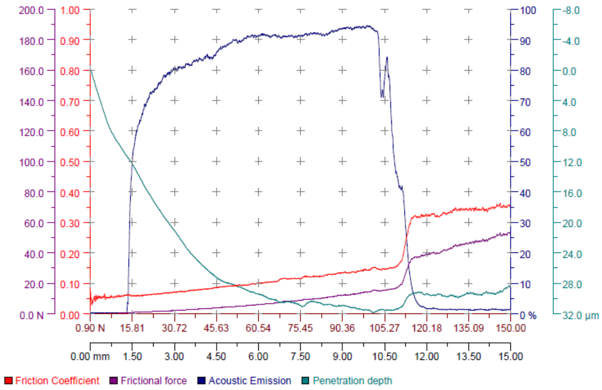
Figure 2: Optical microscopic images of polished cross-sectioned specimens of: a) DUPLEX AlCrN, b) DUPLEX AlCrTiN, and c) DUPLEX TiAlN coating

Table 2: Adhesive properties of DUPLEX coatings

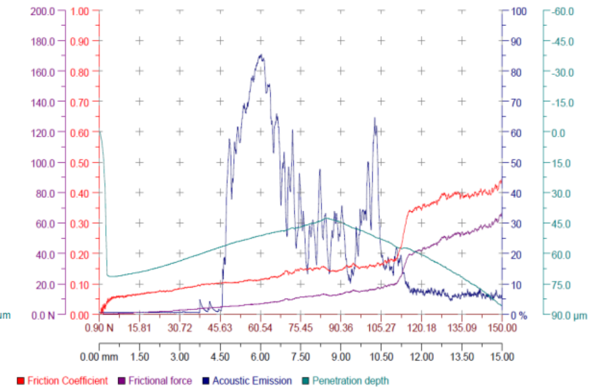
Type of coatings	Lc_1(N)	Lc_2 (N)	Adhesion behavior observed
DUPLEX AlCrN	16	114	Particle chipping =>First Delamination => Full Delamination
DUPLEX AlCrTiN	45	117	First Crack => Progressive Crack => Full Delamination
DUPLEX TiAlN	23	65	First Chipping => Progressive Chipping => Full Delamination

3.2 *Adhesive properties of DUPLEX coatings*

Adhesive properties of DEPLEX coatings were studied in the laboratory at room temperature. The load under which the first sudden variation in the acoustic emission signal observed was used to define the first critical load for first damage, called as Lc_1. Lc_2 was taken as the load at which the full delamination or the expose of substrate could be identified which corresponds to the sudden change in the friction coefficient. This is confirmed by the optical investigation of the scratch track. The critical load Lc_1 and Lc_2 of DUPLEX surface treatment were presented in Table3. The optical images of the scratch tracks are given in Figure. 5-7.



a)



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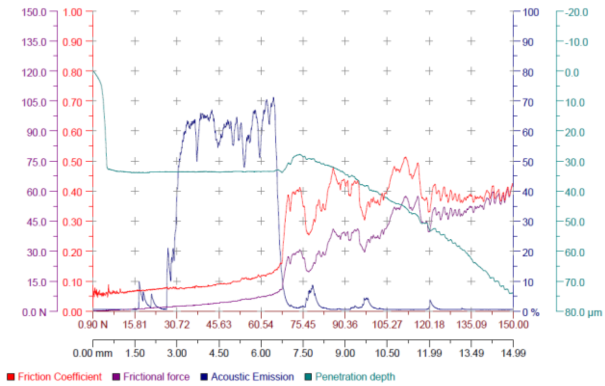


c)

Figure 3: Friction coefficient and Acoustic emission signal of scratch tests for a) DUPLEX AlCrN, b) DUPLEX AlCrTiN ,and c) DUPLEX TiAlN

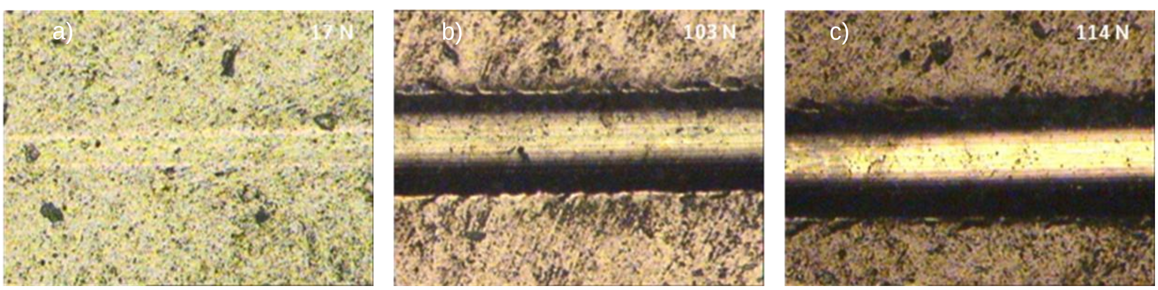


Figure 4: Scratch tracks of DUPLEX AlCrN a) particle chipping, b) first delamination and c) full delamination

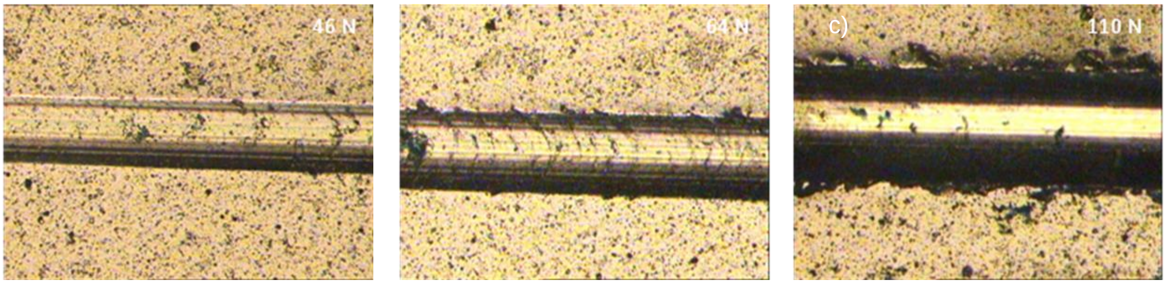


Figure 5: Scratch tracks of DUPLEX AlCrTiN a) first crack, b) progressive crack , and c) full delamination

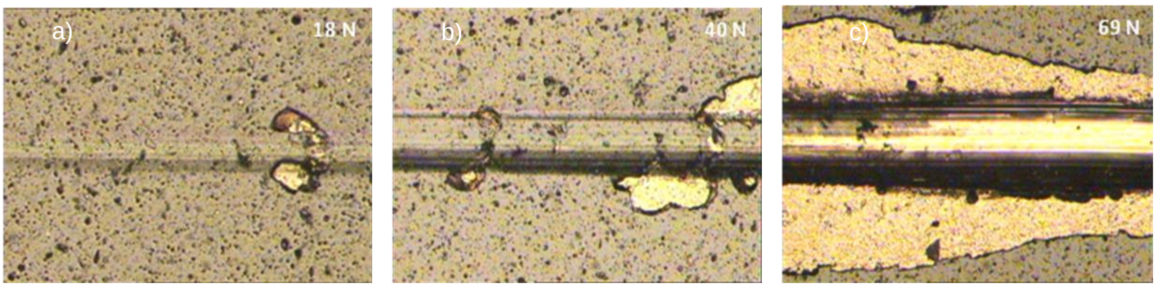


Figure 6: Scratch tracks of DUPLEX TiAlN a) first chipping, b) progressive chipping , and c) full delamination

From Figure. 3a, The DUPLEX AlCrN showed first damage at approx. 16N. A small degree of particle chipping could be observed under such load (see Fig. 4). From the microscopic image, it was found that no severe chipping or crack is observed for the whole test suggests that the AlCrN is the toughest coating in such work . The first partial delamination is observed at the load of approx. 103 N followed by the full delamination at approx. 114N DUPLEX AlCrTiN showed best adhesion with the first critical load (i.e. first damage) at approx. 45 N (see Fig. 3b). Fig 5b indicated that small cracks were founded on side of scratch track under the first critical load. Progressive cracks were found at the load of approx. 64N suggests higher failure resistance with lower resistance to crack propagation compared to DUPLEX AlCrTiN coating (see Fig.5). The friction coefficient signal suggested the full delamination load of approx. 117N. DUPLEX TiAlN coating show a moderate value of the first critical load with a lowest value of final critical load relative to the other two coatings with Lc_1 and Lc_2 of approx. 23N and 65 N, respectively. Optical Microscopic images indicated chipping at first critical load. The severity of chipping is increased at higher load causing full delamination at lower load than the other two coatings (see Fig.6b). Fig. 6c showed that, at full delamination load, there are intensive chipping outside the scratch track.

3.3 *Sliding wear behaviors of DUPLEX coatings*

The pin on disc tests were performed on all coatings under different load. The wear volume of the disc was calculated based on the topography change of the worn surface and the total sliding distance and is plotted against the applied normal load in Figure. 7. The maximum Hertzian [13] contact pressures corresponding to different normal load together with the coefficient of friction obtained are given here in Table 3. The increase in friction coefficient with load was found for DUPLEX AlCrN and TiAlN. The minimum variation in the coefficient of friction with load was observed for the DUPLEX AlCrTiN. This may be resulted from a combined effect of wear phenomena (i.e. wear debris character and behavior) and reduction in shear strength at high temperature and should be fully investigated (the work is currently ongoing).

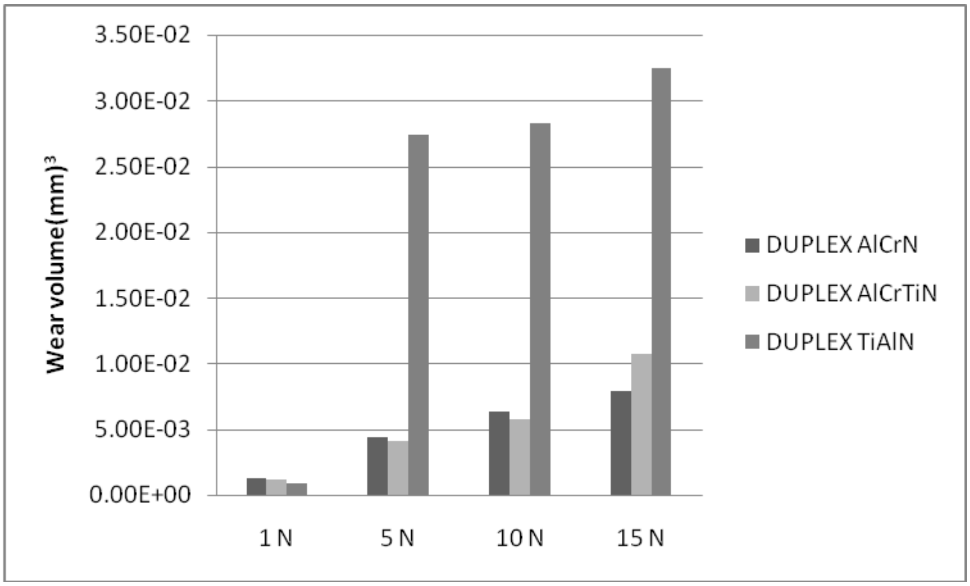


Figure 7: Wear volume of DUPLEX coatings

Table 3: Friction coefficient and Coefficient of wear of DUPLEX surface treatment

Types of coatings	Load (N)	Maximum Hertzian Contact Pressure (MPa)	Friction coefficient	Specific Wear coefficient , K
DUPLEX AlCrN	1 N	1136.78	0.50	1.35×10^{-3}
	5 N	1943.87	0.53	
	10 N	2449.12	0.70	
	15 N	2803.54	0.80	1.36×10^{-3}
DUPLEX AlCrTiN	1 N	1074.728	0.60	1.29×10^{-3}
	5 N	1837.759	0.62	
	10 N	2315.43	0.56	
	15 N	2650.5	0.58	2.11×10^{-3}
DUPLEX TiAlN	1 N	1000.27	0.40	5.62×10^{-3}
	5 N	1710.44	0.75	
	10 N	2155.03	0.80	5.26×10^{-3}
	15 N	2466.89	0.90	

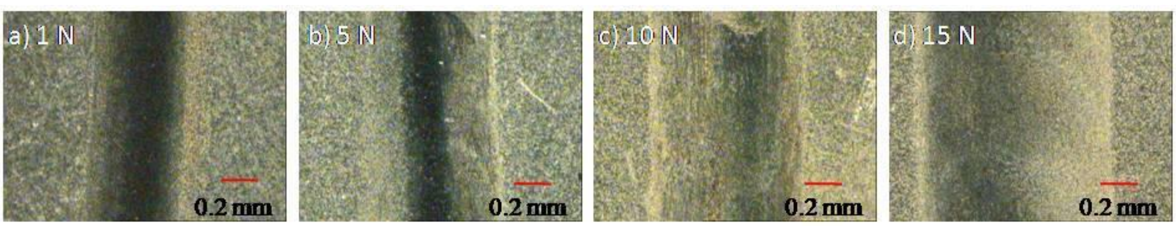


Figure 8: Optical Microscopic images of wear tracks of the DUPLEX AlCrN coating

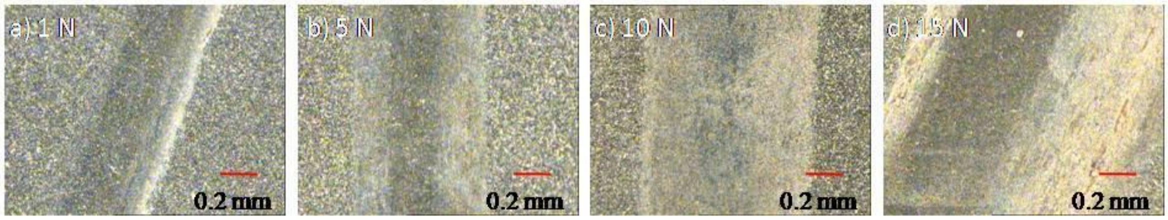


Figure 9: Optical Microscopic images of wear tracks of the DUPLEX AlCrTiN coating

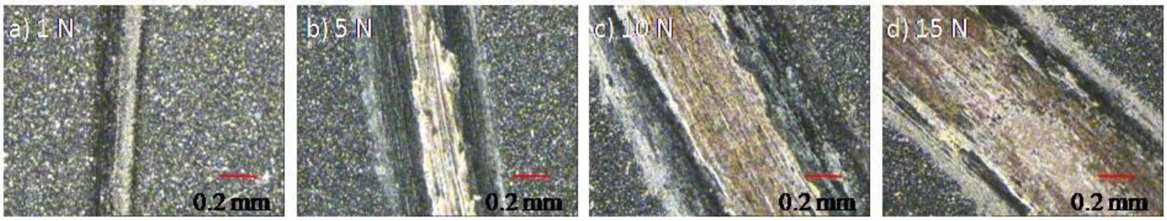
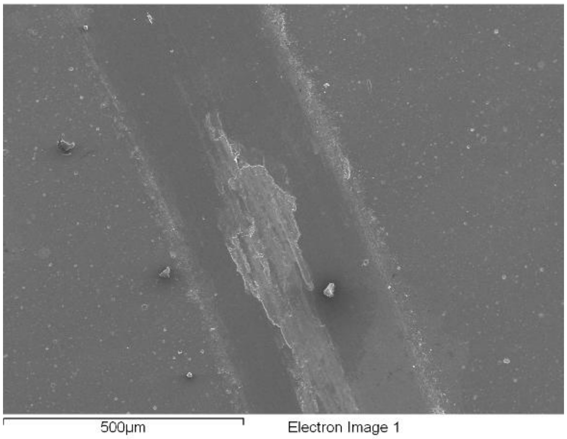


Figure 10: Optical Microscopic images of wear tracks of the DUPLEX TiAlN coating

Fig.7 show higher wear volume at higher load. This is consistent with the results suggested by the optical investigation of the wear tracks shown here in Figure. 8-10. Wear tracks widths of all DUPLEX coatings were found to be increased with increasing load due to However, a bi-linear relationship between the wear volume and normal load is observed. Different dimensionless wear coefficient so called as specific wear rate, K, were different for low and high load suggesting different wear mechanism. The wear coefficient for different load level were calculated from Archard equation [14] and listed in Table 3. At low contact pressure, DUPLEX AlCrTiN has a lower wear rate than DUPLEX AlCrN resulting from a higher hardness of DUPLEX AlCrTiN. It can be seen from Fig 8a, b and Fig.9a, b that no crack is presented in all wear tracks. Whereas plastic deformation is presented in wear track of DUPLEX AlCrN suggesting ductile failure behavior of materials, i.e. plouging abrasive wear is expected to dominate wear.

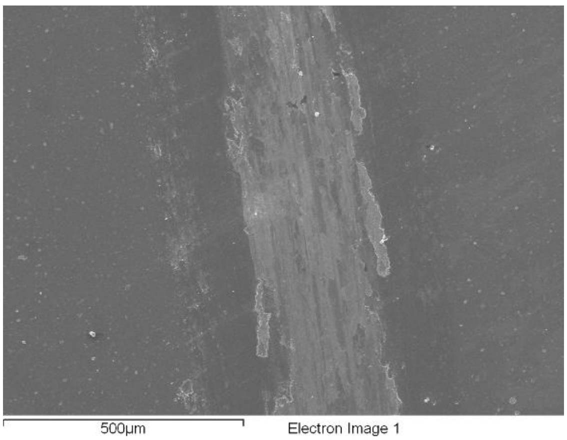
At high contact pressure, DUPLEX AlCrN found to give a better wear performance than DUPLEX AlCrTiN. This may be caused by different failure mechanism predominant wear behavior at such load. Optical investigation of DUPLEX AlCrTiN wear track at high load show that a sign of cutting abrasive wear is found at the edge of wear track. This may result in higher wear rate of DUPLEX AlCrTiN as observed due to the low crack propagation resistance of such coating.

The DUPLEX TiAlN coating was showed lower wear resistance at high temperature at all load under investigation. Figure. 10 illustrates optical microscopic images of the wear tracks of DUPLEX TiAlN coating at load 5-15 N. The analysis of the wear track of DUPLEX TiAlN coating showed the delamination of the coating and pronounced abrasive wear mechanism. Oxidation was expected to take place over the exposed surface inside the track which is unprotected by the coating. The EDS analysis of DUPLEX TiAlN wear tracks under different loads has been carried out. The results are presented here in Figure. 11-12.



Element	Weight%	Atomic%
N K	25.75	43.64
O K	9.52	14.13
Al K	27.38	24.09
Ti K	32.13	15.92
Cr K	0.34	0.16
Fe K	4.87	2.07
Totals	100.00	

Figure 11: EDS of DUPLEX TiAlN wear track at load 5N



Element	Weight%	Atomic%
N K	18.50	32.15
O K	17.99	27.38
Al K	23.02	20.78
Si K	0.23	0.20
Ti K	26.45	13.45
Cr K	0.84	0.39
Fe K	12.97	5.65
Totals	100.00	

Figure 12: EDS of DUPLEX TiAlN wear track at load 15 N

Figure.11 and 12 showed the increase in % Fe and O with increasing load indicates oxidation of substrate caused by delamination as expected. As a result of unprotected, the lower hardness and high temperature resistance of the substrate lead to severe wear of the substrate after the coating is removed giving significant rise in wear rate relative to the other two coatings.

4. Conclusions

In this paper, DUPLEX surface treatment comprises of gas nitriding followed by 3 types of PVD coatings e.g. TiAlN, AlCrN, and AlCrTiN were deposited on AISI H13 tool steel. The mechanical and tribological properties were investigated. Based on the experimental results, the following main conclusions can be drawn:

1) All DUPLEX treatment had approximate 120-130 μm thick diffusion zone resulted from nitriding process giving riser in the steel hardness to a maximum of 1200HV.

2) The top PVD coatings increase the surface hardness to the value of greater than 30 GPa. The increased in the surface hardness is in the following order: DUPLEX TiAlN coating < DUPLEX AlCrN coating < DUPLEX AlCrTiN coating.

3) The DUPLEX AlCrTiN coating showed the best adhesive property with the highest first failure critical load. Whereas the DUPLEX AlCrN coating was found to be tougher with higher crack propagation resistance. DUPLEX TiAlN coating showed high degree of chipping along the edge of scratch tracks with lower value of full delamination load.

4) The results showed that different wear mechanism dominant wear behavior of coatings at different load.

At low contact stress, harder coating (DUPLEX AlCrTiN) shows the better wear resistance whereas at high contact stress, the tougher coating (DUPLEX AlCrN) serves better. Examination of worn surface suggested that this might be caused by change in failure mechanism from ductile fracture to a more brittle manner at high contact stress for hard material. The DUPLEX TiAlN coating showed the lowest wear resistance at high temperature due to low chipping and full delamination load leading to exposure of steel substrate at the contact.

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