EXPERIMENTAL INVESTIGATION OF TRIBOLOGICAL PROPERTIES OF MONO-LAYERED AND MULTI-LAYERED AlCrN-BASED COATINGS

Jasmaninder Singh Grewal[1] and Gurdeep Singh[2]
[1]Professor and Head of department, [2]Student
Production Engineering Department
Guru Nanak Dev Engineering College, Ludhiana – 141006, Punjab, India.

Abstract: This manuscript highlights characterisation and tribological behaviour of AlCrN-based multi-layered and mono-layered coatings deposited on EN-8 high-tensile steel with the help of Physical Vapour Deposition (PVD)-DC Magnetron Sputtering technique. These coatings were deposited to improve the wear-rate of EN-8 substrates. These AlCrN-based Nano-structured coatings were deposited on EN-8 substrates at 600ºC temperature by using OerlikonBalzer's-rapid coating system under the influence of nitrogen atmosphere. After deposition of coatings, the substrates were examined by investigating the thickness, micro-hardness, Porosity percentage &Surface roughness (Ra) values of coatings. Also, SEM/EDAX & XRD analysis were used to identify the various phases present on the substrates surface. The micro-hardness of multi-layered and mono-layered coatings was measured to be 470Hv and 362Hv, whereas thickness was measured to be6.1µm&4.0µm.Also, uncoated and coated EN-8 substrates were analysed for thermal behaviour with the help of Differential Scanning Calorimetry (DSC). This DSC analysis revealed that mono-layered coated substrates were thermally more stable as compare to multi-layered and uncoated substrates. After this, tribological behaviour of uncoated and coated substrates was examined according to ASTM-(G99-03) standard using a Pin-on-disc tribometer by varying sliding-velocity to 0.5 $m.s^{-1}$, 1 $m.s^{-1}$ and 2 $m.s^{-1}$ at constant normal load of 30N.Wear results indicates that multi-layered AlCrN nano-structured coating behave better under different sliding-velocity conditions.

Index Terms –Physical Vapour Deposition, Sliding Wear, Sliding Speed, Frictional Coefficient, Alcrona-pro (AlCrN Mono-layered) & Altensa (AlCrN multi-layered) Coatings

1. INTRODUCTION

Over the past some years, CrN-based PVD coatings with excellent anti-adhesive, anti-corrosive and oxidation properties, have been found a number of industrial applications as protective coatings for various kinds of tools.However, in some applications a permanent failure of CrN-based coatings was observed due to less abrasive wear-resistance and low hardness of these kinds of coatings [1-5]. In order to improve these kinds of properties of CrN-based coatings, another metal has been investigated to develop the multicomponent coatings [1, 6].

The level of performance these multicomponent coatings highly depends upon their coating structure, elemental composition, deposition parameters & the technique used as well as on reliability of technologies used to measurement the characteristics of the coatings like hardness, thickness etc. [6, 7].

Aluminium act as one of the most capable candidates for CrN-based coatings and would form a Cr-Al-N multicomponent system, which have superior oxidation as well as abrasive wear resistance properties[8-10]. On the same time the maximum service temperature for Cr-Al-N multicomponent coatings has around 1100ºC, whereas CrN-based coatings has around 700ºC [11].

With respect to CrN-based coatings, TiN, TiAlN and AlTiN-based coatings are also used widely as protective coatings. The maximum service temperature of these kinds of coatings has around 600ºC for TiN-based coatings and 900ºC for TiAlN and AlTiN-based coatings. Also, due to addition of Al- content more than 65% in TiAlN &AlTiN-based coatings, there is the chance to change the structure of coating from cubic to hexagonal lattice whereas in case of AlCrN-based coatings, Cr-atoms has the capability to hold the Al-content more than 65% without doing any structural change to AlCrN-based multicomponent coatings. Due to this reason, TiN-based coatings has been rapidly replaced with that of CrN-based coatings [1, 12-17].

Accordingly, in this research work an attempt is basically made to investigate the tribological properties of mono-layered and multi-layered AlCrN-based coatings deposited on EN-8 high tensile steel. EN-8 is extensively used in manufacturing of rotavator blades. The rotating-blades of rotavator operates under different environmental condition and provide high impact on the soil-surface and throw the soil particles upward due to its rotational movement and pulverises soil through breaking clods. The environment basically leads to physical and chemical degradation of the blades surface. Therefore, the blades suffer heavy oxidation and abrasive wear, due to which material from blades was continuously detached, which leads to permanent failure of rotavator blades. To reduce this wear phenomenon, PVD-DC Magnetron Sputtering technique has been used to deposit AlCrN-based coating on EN-8 and investigated for their tribological behaviour. The wear behaviour of substrates has been investigated using a Pin-on-disc apparatus and according to ASTM-G99-03 standard. The aim of this research work is to present an experimental database related to frictional coefficients and wear rates in terms of cumulative weight loss of uncoated and AlCrN-based coated substrates under different sliding wear conditions.

2. EXPERIMENTAL DETAILS

2.1 Substrate Material Selection:

Selection of EN-8 high tensile steel has been done on the basis of manufacturing of rotavator blades. The cylindrical pins of 8mm diameter and 30mm length have been prepared from EN-8 bar and chemical composition of the prepared samples has been compared with that of actual composition using Atomic Emission Spectrometer according to IS:8811-1998 standard. Table 1 represents the comparison between nominal composition and actual composition of EN-8 material.

Table 1: Chemical Composition (Wt. %) of the EN-8 Steel Substrate

EN-8	C	S	P	Si	Mn	Cr	Cu	Ni	Fe
Nominal	0.37-0.45	0.035	0.035	0.17-0.37	0.50-0.80	0.25	0.25	0.25	Balance
Actual	0.42	0.015	0.026	0.20	0.65	0.09	0.04	0.01	Balance

2.2 Deposition of Coatings:

A front loading Balzer's-RCS (i.e. rapid coating system) machine has been used for deposition of multicomponent coatings. Two kinds of coatings generally known as *Alcrona-pro* (i.e. AlCrN-based Mono-layered) and *Altensa* (i.e. AlCrN-based Multi-layered) coatings have been deposited at 500^{o}Con cylindrical pins of EN-18 by using DC Magnetron Sputtering-PVD technique. The surface roughness of the mono-layered and multi-layered coated substrates was found to be 0.252 and 0.037 µm.The coatings have been deposited by OerlinkonBalzers Coating India Pvt. Ltd, Gurugram, India and parameters used during deposition of multicomponent coatings is shown in table 2

Table 2: Process parameters used during deposition of coating

	Alcrona-Pro	**Altensa**
Machine used	Inlenia BAL 1200 XL (OerlikonBalzers)	RCS-rapid coating system (OerlikonBalzers)
Deposition temperature (°C)	500	500
Targets	Al and Cr	Al and Cr
Pressure in vacuum chamber(bar)	10^{-3}	10^{-3}
No. of targets per machine	8	8
Time interval (hrs)	8-10	8-10
Micro-hardness (HV)	362	470
Maximum Service temperature (°C)	1100	>1100
Coating Thickness (µm)	4.0	6.1
Colour of coatings	Bright grey	Light grey

2.3 Characterization of Coating

An inverted metallurgical microscope attached with Metallurgical Image Analysis Software (MIAS) has been used to measure the porosity percentage of the multicomponent coatings deposited on EN-8 substrates. The coated substrates were characterized using XRD analysis to identify the phases present on the substrates surface. A Bruker D-8 Advanced Powdered X-ray-Diffractometer with 2.2 kW Cu Kα anode was used to perform the XRD analysis. The speed and angle of goniometer has been kept 20 min^{-1} and 5-100^o respectively. After this, Field Emission-Scanning Electron Microscope (FE-SEM Quanta 200 FEG) attached with EDAX through Genesis Software has been used to characterize the morphology of the substrates surface. The samples have been analysed for thermal behaviour with the help of Differential Scanning Calorimetry (DSC).The micro-hardness and surface roughness (Ra)of the coated substrates has been measured by using Vickers hardness tester and Mitutoyo-Surftest SJ-410 equipment.

2.4 Sliding Wear Studies

This study has been made to investigate the tribological properties of uncoated and coated substrates. The wear in terms of cumulative weight loss and frictional coefficient has been measured using Pin-on-disc test rig. Figure 1 represents the front and top view of Pin-on-disc test rig whereas table 3 represents the specifications of Pin-on-disc test rig. The uncoated and coated pins were tested by varying sliding velocity to 0.5, 1 and 2 m.s^{-1} at constant normal load of 30 N for a time interval of 90 min. The cumulative weight loss of uncoated and coated substrates has been plotted against time interval under different parameters as mentioned above.

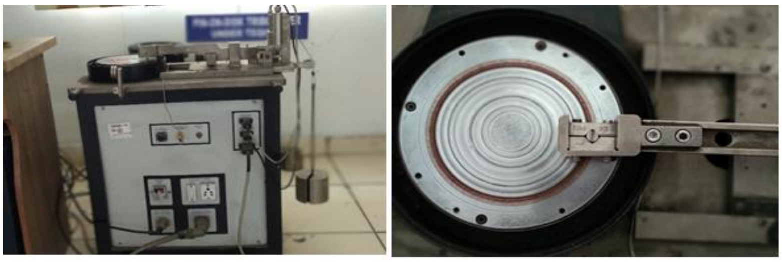


Figure 1:Front and top view of Pin-on-disc test rig

Table 3:Specifications of Pin-on-disc test rig

Specification	Machine Capabilities	Selected Range
Disc Size	165mm x 8mm	165mm x 8mm
Wear track diameter	60mm-160mm	120mm
Normal Load	0-200N	30N
Disc rotation	50-2000 RPM	0.5m/s (79.58 rpm), 1m/s(159.15rpm) & 2m/s (318.3rpm)
Pin Size	3-10mm	8mm

The specifications of Pin-on-disc test rig was provided by DUCOM Bengaluru, India and the range of parameters was selected accordingly. The rotating disc made of EN-31 provided by DUCOM Bengaluru, India in the Pin-on-disc apparatus has hardness value around 63HRC whereas hardness value of EN-8 material has 35 HRC, which is much less than that of EN-31 material. So, experimentation has been done with EN-31 as counter material on Pin-on-disc test rig.

3. RESULTS AND DISCUSSION

3.1 Porosity Percentage

The porosity analysis plays a key role in wear studies. The dense and uniformed coatings are supposed to provide better wear resistance as compared to less dense and porous coatings. The porosity percentage of multicomponent AlCrN based mono-layered and multi-layered coatings measured by an inverted metallurgical microscope attached with MIAS software was found to be 0.48% and 0.46%. This represents that the deposited coatings are less porous in nature.

3.2 X-Ray Diffraction Analysis (XRD)

The X-ray analysisofmulticomponent AlCrN-based mono-layered& multi-layered coatings deposited on EN-8 has been shown in figure 2 respectively. This technique helps to identify the various phases present on surface of substrates. The diffractograms shows the presence of AlN, CrN and combinations of both phases.

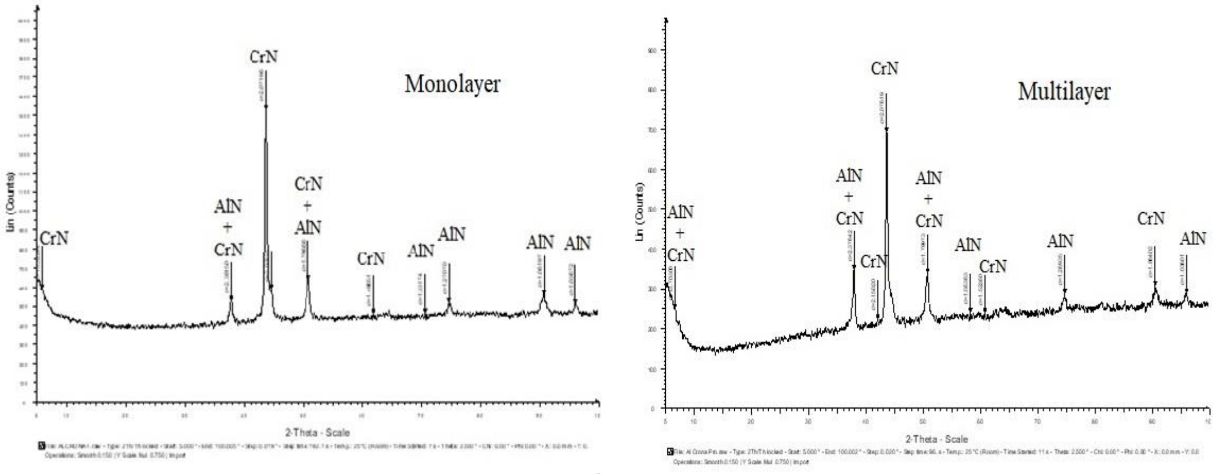


Figure 2: XRD analysis of coated substrates

3.3 Surface Morphology of Coatings

SEM (Scanning Electron Microscopy) micrographs w.r.t Energy Dispersive X-ray analysis (EDAX) represents the surface morphology of multicomponent AlCrN-based coatings deposited on EN-8 substrates as shown in figure 3. A grey matrix w.r.t black & white contrast regions has been represented by the SEM micrographs. SEM/EDAX analysis represents the less amount of Al and higher amount of Cr at black coloured areas as compared to white contrast region.

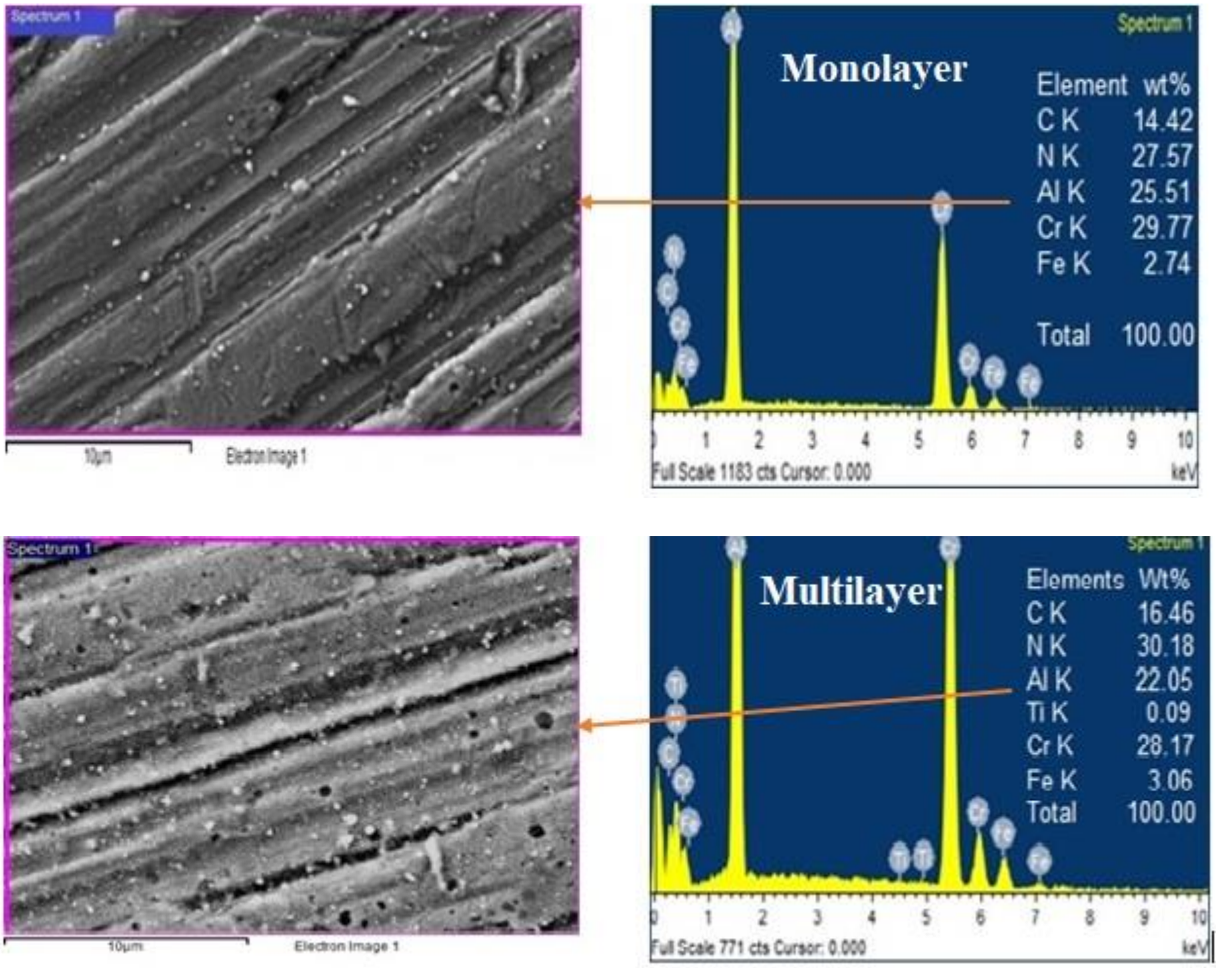


Figure 3: SEM/EDAX analysis of coated substrates

3.4 Thermal Analysis

The thermal analysis of uncoated, mono-layered (*Alcrona-pro*) and multi-layered (*Altensa*) samples has been done by using Differential Scanning Calorimetry (DSC). The curve of all samples has been generated against heat energy in mW w.r.t temperature (ºC). The first half cycle represents the heating cycle ranging from 30ºC to 350ºC whereas the next half cycle represents the cooling cycle ranging from 350ºC to 30ºC. The time interval for carrying out the thermal analysis was 1 hr and 5 min i.e. 32.5 minutes for each cycle. Figure 4 represents that mono-layered coated sample absorb more amount of heat energy as compared to multi-layered coated and uncoated sample. It has been observed more heat energy was required by mono-layered coated sample to reach the temperature of 350ºC whereas less heat energy was absorbed by multi-layered coated and uncoated sample to attain the same temperature i.e. mono-layered coated sample is thermally more stable as compared to multi-layered coated and uncoated sample.

3.5 Wear & Frictional Behaviour

The wear and frictional analysis of coated as well as of uncoated samples has been investigated using Pin-on-disc tribometer by varying sliding velocity to 0.5 m.s^{-1}, 1 m.s^{-1} and 2 m.s^{-1} at constant load of 30 N. Figure 5 represents the wear behaviour of uncoated and coated samples at various sliding velocities. It has been observed that more cumulative weight loss was occurred when sliding velocity was high i.e. 2 m.s^{-1} at constant load of 30N in all the material cases. Figure 5 also represents that multi-layered coated samples has lower wear rate (i.e. cumulative weight loss) as compared to mono-layered coated and uncoated samples at all sliding velocities i.e. 0.5, 1 & 2 m.s^{-1}.

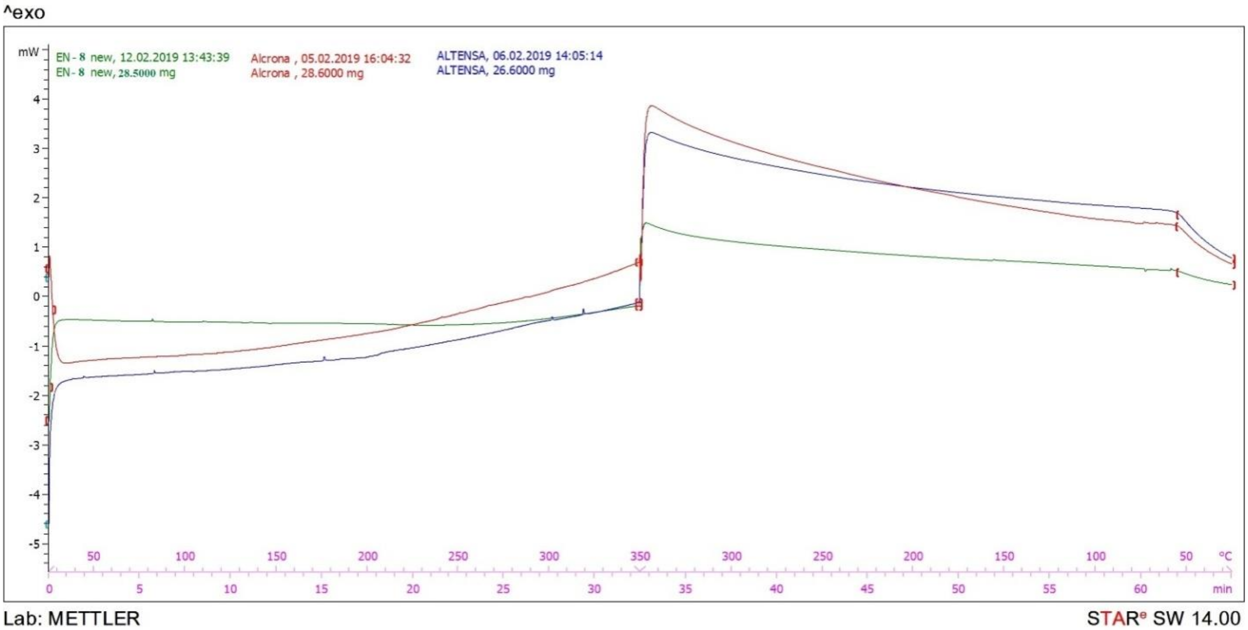


Figure 4: DSC of uncoated and coated samples

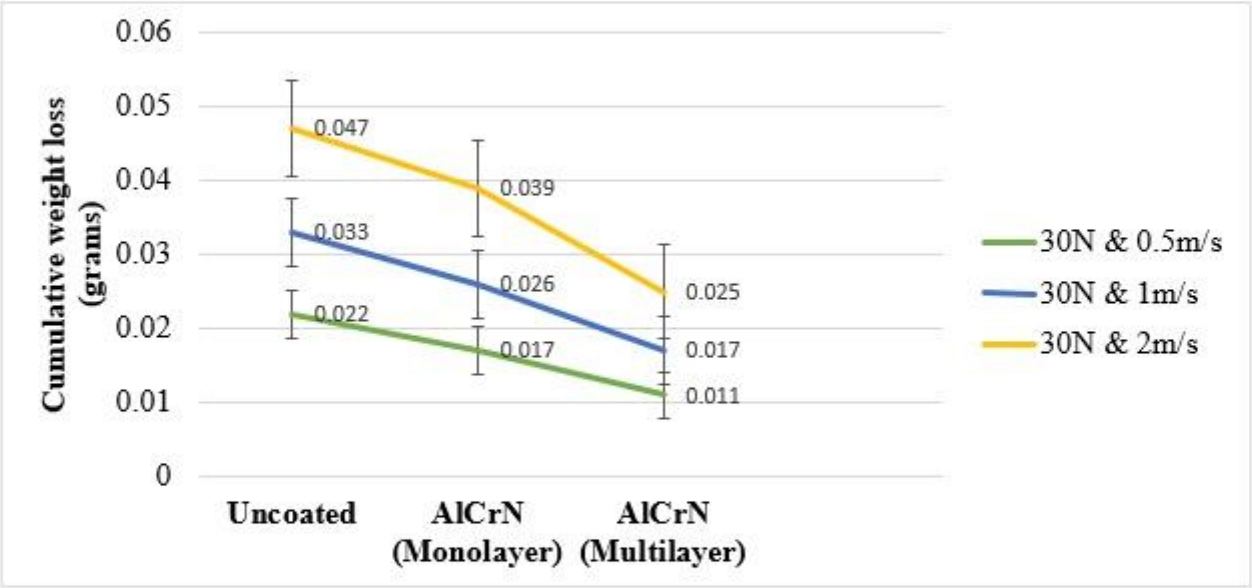


Figure 5: Comparative weight loss (in grams) of uncoated & coated sample at constant load & different sliding velocities

The frictional coefficient (µ) values collected from winducom software attached with Pin-on-disc tribometer through a control panel and computer system has been shown in figure 6. The frictional coefficient of all the uncoated and coated samples was found to be in the range of 0.167-0.448. It has been observed that frictional coefficient was decreases as the sliding velocity was increases from 0.5 $m.s^{-1}$ to 1 $m.s^{-1}$ and 2 $m.s^{-1}$ at constant normal load in all the material cases. Figure 6 also represents that multi-layered coated samples has lower frictional coefficient as compared to uncoated and mono-layered coated samples at all the varying sliding velocities w.r.t constant load.

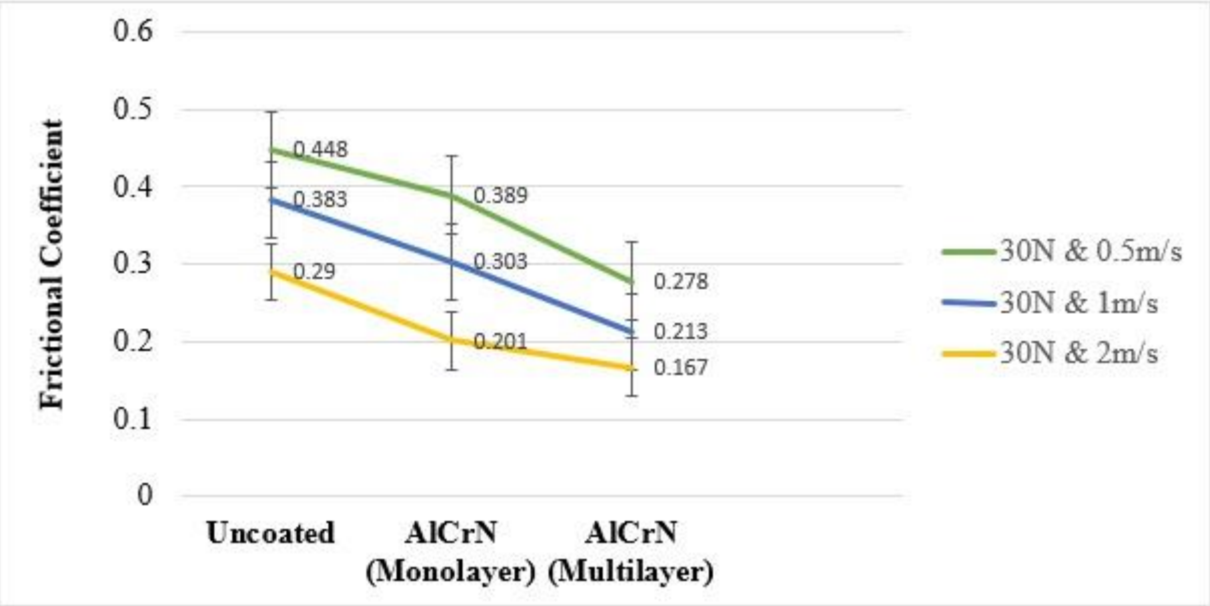
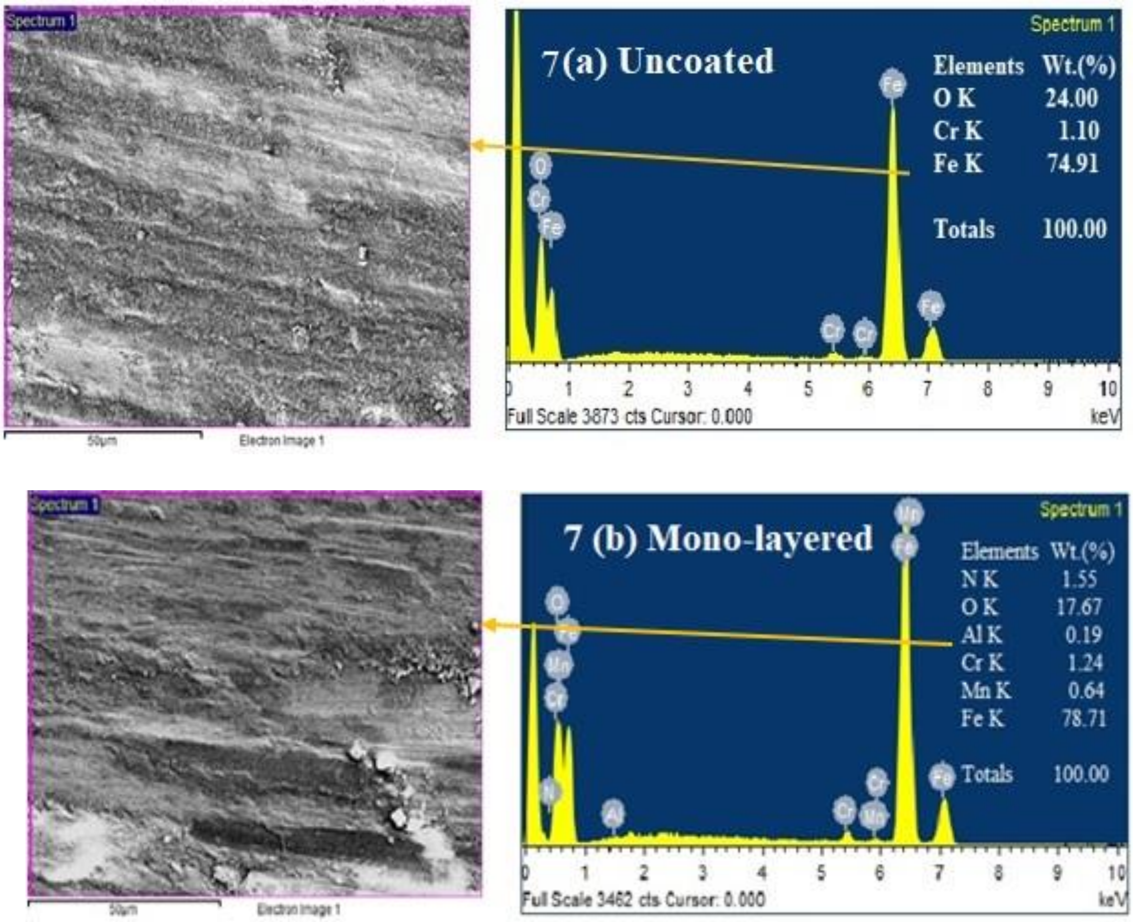


Figure 6: Variation in Frictional coefficient of uncoated & coated samples at constant load & different sliding velocities

3.6 SEM/EDAX of Worn-Out Samples

The SEMmicrographs clearly depicts various kinds of wear mechanisms of metal removal as well as of wear track and indicate the wear of the samples. Adhesive and Abrasive were the two main wear phenomenon's observed during testing of pins. SEM micrographs along with EDAX analysis of multicomponent coated and uncoated samples was shown at maximum sliding velocity i.e. 2 m.s^{-1} w.r.t constant load of 30 N in figure 7. In uncoated samples Fe almost 75% was the main constituent and EDAX indicates the formation of oxides of ferrous due to oxygen as represented by figure 7 (a). AlCrN multi-layered coating micrographs revealed the retention of coating and hence protect the substrate. EDAX analysis shows the presence of Al, Cr, N and O as main constituents and are shown by figure 7 (c) whereas SEM/EDAX analysis of mono-layered coating as shown in figure 7 (b) revealed the oxide fracture and removal of coating from the surface. The EDAX confirms that the coating has removed partially from the surface and fails to protect the substrate. Although with increase in sliding velocity, wear rate (Cumulative weight loss) also increases.



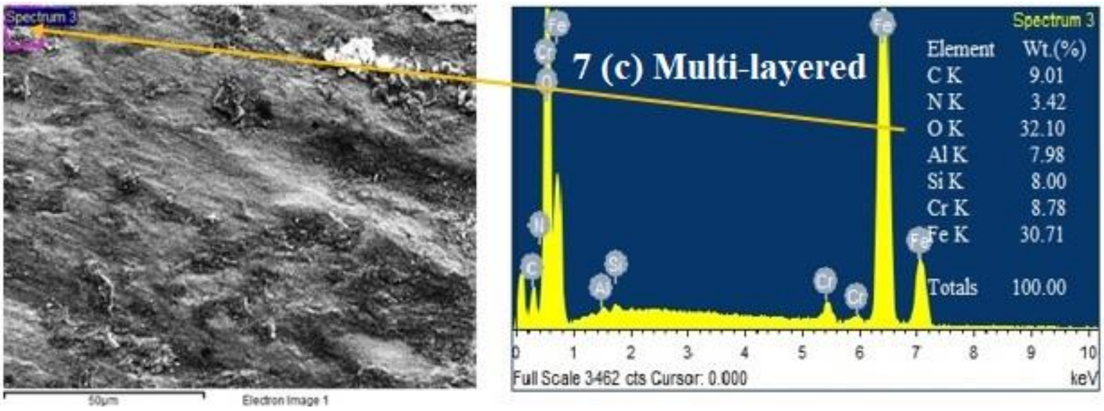


Figure 7: SEM/EDAX analysis of worn-out samples at maximum sliding velocity of 2 m.s^{-1} w.r.t constant load of 30 N

4. CONCLUSIONS

The tribological properties of multicomponent AlCrN-based coatings in the form of monolayer and multilayer deposited on EN-8 high tensile steel by Magnetron Sputtering-PVD technique have been analysed. Wear and frictional properties were studied using Pin-on-disc tribometer by varying sliding velocities at constant load. The following conclusions are made:

- The multicomponent AlCrN coatings deposited at 500°C exhibited negligible percentage of porosity value. The porosity percentage of mono-layered and multi-layered coating was observed to be less than 0.5%.
- The XRD analysis represent presence of various phases like CrN &AlN on the substrate surface.
- The DSC results reveals that mono-layered coated sample is thermally more stable as compared to multi-layered coated and uncoated sample.
- Wear (i.e. cumulative weight loss) of the coated as well as of uncoated samples was increased as the sliding velocity was increased and multi-layered coated samples has lower wear rate as compare to mono-layered coated and uncoated samples. Based upon the present data of cumulative weight loss at varying sliding velocities i.e. 0.5, 1 & 2 m.s^{-1} w.r.t constant load of 30N, can be arranged in the order of;
  Uncoated samples > AlCrN-based mono-layered coated samples > AlCrN-based multi-layered coated samples
- Frictional coefficient of the coated as well as of uncoated samples was decreased as the sliding velocities was increased i.e. at 0.5 m.s^{-1} sliding velocity w.r.t constant load of 30 N, frictional coefficient was maximum.
- Oxidation and Abrasive wear were the two main wear phenomenon’s observed during testing of coated and uncoated samples of EN-8 high tensile steel.

Acknowledgement

The authors are highly grateful to manufacturing & research lab, Guru Nanak Dev Engineering College, Ludhiana.

REFERENCES

1. Mo, J. L., & Zhu, M. H. (2008). Sliding tribological behavior of AlCrN coating. Tribology International, 41(12): 1161-1168.
2. Vetter, J., Knaup, R., Dweletzki, H., Schneider, E., &Vogler, S. (1996). Hard coatings for lubrication reduction in metal forming. Surface and Coatings Technology, 86: 739-747.
3. Cunha, L., Andritschky, M., Pischow, K., & Wang, Z. (1999). Microstructure of CrN coatings produced by PVD techniques. Thin Solid Films, 355: 465-471.
4. Mo, J. L., Zhu, M. H., An, J., Lei, B., & Zhou, Z. R. (2006). Development in PVD CrN coating. China Surf Eng, 19(4-11): 71-75.
5. Reiter, A. E., Derflinger, V. H., Hanselmann, B., Bachmann, T., &Sartory, B. (2005). Investigation of the properties of Al1− xCrxN coatings prepared by cathodic arc evaporation. Surface and Coatings Technology, 200(7): 2114-2122.
6. Hafedh, D., Kaouthar, K., & Ahmed, B. C. L. (2015). Nanoindentation of Thin Films Prepared by Physical Vapor Deposition. World Academy of Science, Engineering and Technology, International Journal of Chemical, Molecular, Nuclear, Materials and Metallurgical Engineering, 9(2): 368-371.
7. Kawate, M., Hashimoto, A. K., & Suzuki, T. (2003). Oxidation resistance of Cr1− xAlxN and Ti1− xAlxN films. Surface and Coatings Technology, 165(2): 163-167.
8. Vetter, J., Lugscheider, E., &Guerreiro, S. S. (1998). (Cr: Al) N coatings deposited by the cathodic vacuum are evaporation. Surface and Coatings Technology, 98(1-3): 1233-1239.

9. Wuhrer, R., & Yeung, W. Y. (2004). A comparative study of magnetron co-sputtered nanocrystalline titanium aluminium and chromium aluminium nitride coatings. ScriptaMaterialia, 50(12): 1461-1466.
10. Uchida, M., Nihira, N., Mitsuo, A., Toyoda, K., Kubota, K., &Aizawa, T. (2004). Friction and wear properties of CrAlN and CrVN films deposited by cathodic arc ion plating method. Surface and Coatings Technology, 177: 627-630.
11. Singh, G., Singh, S., & Grewal, J. S. (2019). Erosion wear characterisation of DLC and AlCrN-based coated AISI-304/316 steels. Surface Engineering, 35(4): 304-316.
12. Richter, A. (2005). Recipe for enhancement. Cutting Tool Eng, 57(10): 10-12.
13. Fox-Rabinovich, G. S., Beake, B. D., Endrino, J. L., Veldhuis, S. C., Parkinson, R., Shuster, L. S., &Migranov, M. S. (2006). Effect of mechanical properties measured at room and elevated temperatures on the wear resistance of cutting tools with TiAlN and AlCrN coatings. Surface and coatings technology, 200(20-21): 5738-5742.
14. Endrino, J. L., Fox-Rabinovich, G. S., &Gey, C. (2006). Hard AlTiN, AlCrN PVD coatings for machining of austenitic stainless steel. Surface and Coatings Technology, 200(24): 6840-6845.
15. Bobzin, K., Lugscheider, E., Nickel, R., Bagcivan, N., &Krämer, A. (2007). Wear behavior of Cr1− xAlxN PVD-coatings in dry running conditions. Wear, 263(7-12): 1274-1280.
16. Willmann, H., Mayrhofer, P. H., Persson, P. Å., Reiter, A. E., Hultman, L., &Mitterer, C. (2006). Thermal stability of Al–Cr–N hard coatings. ScriptaMaterialia, 54(11): 1847-1851.
17. Scheerer, H., Hoche, H., Broszeit, E., Schramm, B., Abele, E., & Berger, C. (2005). Effects of the chromium to aluminum content on the tribology in dry machining using (Cr, Al) N coated tools. Surface and Coatings Technology, 200(1-4): 203-207.
18. Grewal, J. S., Sidhu, B. S., & Prakash, S. (2016). Effect of Sliding Velocity on Wear Behaviour of TiAlN Coatings. In Advanced Materials Research, 1137: 24-38. Trans Tech Publications.
19. Grewal, J. S., Sidhu, B. S., & Prakash, S. (2016). Characterization of Nanostructured and Conventional TiAlN Coatings Deposited on AISI-304 Boiler Steel. In Advanced Materials Research, (1137): 1-14. Trans Tech Publications.
20. Aihua, L., Jianxin, D., Haibing, C., Yangyang, C., & Jun, Z. (2012). Friction and wear properties of TiN, TiAlN, AlTiN and CrAlN PVD nitride coatings. International Journal of Refractory Metals and Hard Materials, 31: 82-88.