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Research Article

Adhesion Failure Behavior of Ni-TiO_2-Al_2O_3 & Ni-Al_2O_3 Composite Layers Coatings Evaluated using Microscratch Testing

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Abstract

It is generally accepted that Nanostructure composite layers (NCLs) find their potential applications in industry due to increased hardness, wear resistance and corrosion resistance. Titanium oxide and Alumina Oxide is one of the most important nano materials in implant coating applications mainly because of its excellent properties. This entails that a better understanding of the mechanical properties of a both coating is a must especially its behavior and the mechanisms involved when subjected to stresses which eventually lead to failure. The mechanical properties of the coating may be evaluated in terms of its adhesion strength. In this study, the coatings were subjected to series of micro scratch tests, taking careful note of its behavior as the load is applied. The adhesion behavior of the coatings showed varying responses. It was revealed that several coating process related factors such as thickness, post heat treatment and deposition parameters, to name a few, affect its scratching behavior. Scratch testing related factors (i.e. loading rate, scratch speed, scratch load, etc.) were also shown to influence the mechanisms involved in the coating adhesion failure, evaluation of the load. Displacement graph combined with optical inspection of the scratch confirmed that several modes of failure occurred during the scratching process. These include trackside cracking, tensile cracking, radial cracking, buckling, delamination and combinations of one or more modes. The various parameter are studies like controlling current, pH of the bath solution, agitation etc.; which improves the micro hardness, wear resistance and corrosion resistance of the composite coatings. Today in industry electrodeposited coatings are gaining more importance due to their low cost, ease and simplicity of operation of composite coatings for tribological applications of cutting tools. Ni is one of the most important hard coating materials and is widely used metal matrix. Generally, composites containing dioxide and nitride (like TiO_2 and TiN) are preferred for high wear resistance; high micro hardness, improved corrosion resistance, and high temperature oxidation resistance as compared to pure metal electroplating. TiO_2 nano-particles have good mechanical properties and can be used as secondary phase to improve the hardness of Ni coatings. In this paper Ni-TiO_2-Al_2O_3 & Ni-Al_2O_3 composite coatings are employed on WC cutting tool via electro deposition. The composite layers are characterized using scanning electron microscopy (SEM), Vickers micro hardness tester and Scratch tester. The surface morphology of the coated layers shows the deposition of fine grained structures (cauli-flower like) at low currents, which increases the strain produced due to lattice defects and act as a basis for increase in hardness. The result of micro hardness and adhesion of Ni-TiO_2-Al_2O_3 coating is observed than Ni-Al_2O_3 composite coating by using Vickers Hardness tester and Scratch Tester.

Keywords: *Electrodeposition, Vickers micro hardness tester, scratch Tester*

1. Introduction

For several decades now, coating technology has found a wide array of applications. These include functional coating, decorative coating, industrial coating, thermal barrier coating, to evaluate the mechanical properties is through the adhesion strength measurements. According to the literature, there are more than 250 methods available to determine this particular property [K.L Mittal *et al*, 1995]. Various studies have shown that among those methods, indentation and scratch adhesion are among the most commendable techniques in evaluating adhesion strength, especially for hard coating applications [D.S Rickerby *et al*, 1988; Valli, J *et al*, 1987]. Electro deposition is the process of coating a thin layer of one metal on top of a different metal to modify its surface properties. Electro-deposition is one of the challenged processes for improvement of the surface [R. Balaji *et al*, 2006]. Specially, it is used for the improvement of mechanical properties such as wear and hardness properties of the coating surface. These have the large projected applications for automotive parts, aerospace, printed, electrical contacts, jewelry, musical instruments, soft

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metal gaskets, and decorative door, light and bathroom fittings[R.C Agarwala *et al*,2003].The Nano-sized particles is used by most the researchers for improving, micro hardness, corrosion resistance. In this electro deposition process Ni-TiO_2-Al_2O_3 & Ni-Al_2O_3 composite coatings are employed on WC cutting tool. [M.U. Aytac *et al*, 2013]

2. Experiment Analysis

Electro-deposition is one of the practical methods to make nanostructure materials. Figure 1 shows a simple setup of electro-deposition system. In this system the electro deposition process is controlled by Potentiostat/ Galvanostat (GAMRY Referance600) instrument. A hot plate is used to control the temperature of solution. A specially designed three electrode electro-deposition cell was used for electro-deposition of Ni-TiO_2 (Figure1) where cutting tool was working electrode, Ag/AgCl as reference electrode and pure Ni act as counter electrode (anode). Hard Nickel bath solution was prepared by adding appropriate amount of chemicals in 150 mL distilled water. The beaker was heated on a hot plate and maintained at 45-50°C with continuous magnetic stirring. The temperature was maintained by the use of a hot plate and the electro-deposition was controlled by a DC source.

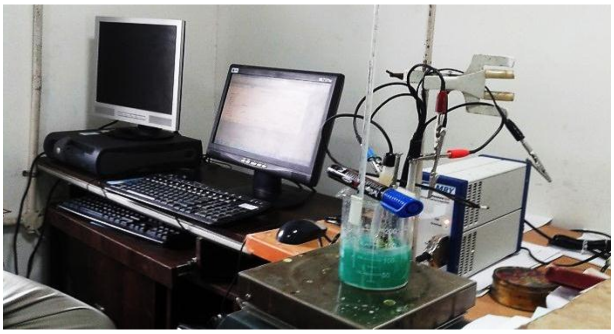

Figure1: Setup of Electro deposition process

Scanning electron microscopy (SEM) can be used for qualitative surface morphology Analysis and energy dispersive spectroscopy (EDS) is a technique to determine the chemical composition of a material non-destructive and Adhesions strength of the samples was evaluated using Scratch tester machine (TR-102).Scratch test is one of widely used, fast, and effective methods to obtain the critical loads that are related to adhesion properties of coating. The electrical current was kept a constant value of 24.4 mA for Ni-$TiO_2Al_2O_3$ coating and 23 mA for Ni-Al_2O_3 coating throughout the experiments and the corresponding voltage varied from approximately 0.7-0.85V for both coatings. A Nickel plate was used as anode whereas the prepared specimens were used as cathode. The pH of the plating solution was maintained by adding sulphuric acid (Increasing pH). The pH was maintained (pH 5.76) in both coatings.

3. Result analysis

3.1 Micro hardness Measurement

Micro hardness values measured at different points on the surface shows that the Micro- hardness readings are homogeneous. Table 1 shows the variation of micro hardness values measured on the Ni- TiO_2 Al_2O_3 coated surface 66.3HRC and for Ni- Al_2O_3 coated surface 55.8 HRC. From the experiment it is clear that with increase in TiO_2 in Nickel bath concentration the micro hardness value increases. For addition of TiO_2 also similar trends were observed. Surface mechanical property is the outcome of particle co-deposition along with Ni grain size and texture obtained by the deposition process. From both the figure it is clear that Ni- TiO_2 Al_2O_3 composite coating exhibits more hardness as compare to the Ni- Al_2O_3 coating.

Table 1: Microhardness measurement

Material	Rockwell Hardness
Titanium dioxide and Alumina oxide	66.3HRC
Alumina oxide	55.8 HRC

3.2 Scratch Tester Result

The scratch testing method is a comparative test in which critical loads at which scratch Appear in the samples are used to evaluate the relative cohesive or adhesive properties of a Coating or bulk material. The effect of the scratching load on the failure behavior was investigated in this study. It can be observed from the graph that Coating Ni-$TiO_2Al_2O_3$ is better in terms of adhesion strength.

Figure 2: Comparision between Traction force and stroke for three scratches of Ni-TiO_2 Al_2O_3 Coated specimen

During the test, scratches are made on the sample with a generally Rockwell C diamond, tip radius ranging from 20 to 200μm which is drawn at a constant speed across the sample, under a progressive load with a fixed loading rate. Series of scratch tests are performed with constant normal loads on a coating to obtain a load where the coating exhibits failure. Each scratch is examined with an optical microscope. Figure2 shows about the comparision of traction force for three different scratches of Ni-$TiO_2Al_2O_3$ coated specimen. The three scratch shows almost maximum value of traction force. It means adhesion strength is more in coating. The second scratch shows that the maximum values of traction force in distance 2.29 mm stroke which means maximum adhesion strength in this area of specimen.

The scracth Tester method is applied on the Tungesten carbide tool bit coated with Ni-Al_2O_3.The figure 3 shows that the comparision between Traction force and Stroke for scratch 3 of Ni-Al_2O_3 coated specimen. The value of traction force curve goes negative at the starting of stroke parameters. It means Al_2O_3coating fail in starting the stroke (mm). The result show that the adhesion strength of Ni-$TiO_2Al_2O_3$coated specimen is more than the Ni-Al_2O_3 coated specimen.

Figure3: Comparision between Traction force and Stroke for scratch 3 of Ni-Al_2O_3 coated specimen

3.3 Scanning electron microscopy and EDS Result

Microstructure analysis on the coated surface as well as on the cross section was carried out with scanning electron microscope (SEM).

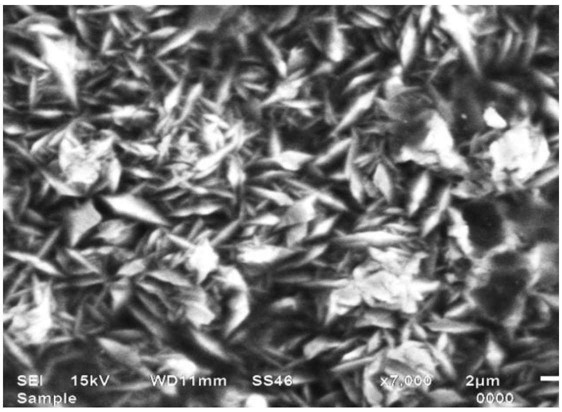

Figure 4: SEM micrograph of Tool Bit coated with Ni-$TiO_2Al_2O_3$

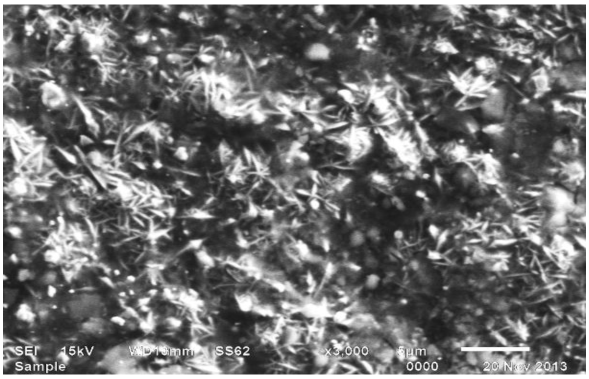

Figure 5: SEM micrograph of Tool bit coated with Ni-Al_2O_3

Figure 4, 5 shows SEM micrographs of sample deposited with Ni- $TiO_2Al_2O_3$ and the Ni-Al_2O_3.The particle size of the Ni-$TiO_2Al_2O_3$ coating is fine as compare to the Ni-Al_2O_3 coating and its look like a lotus flower and cauli-flower like (dirt –repellent).

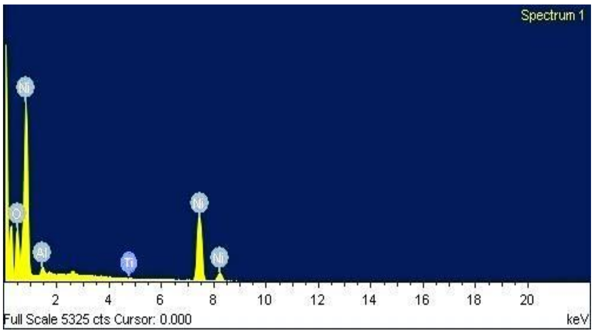

Figure 6: EDS of Ni-$TiO_2Al_2O_3$ coated specimen

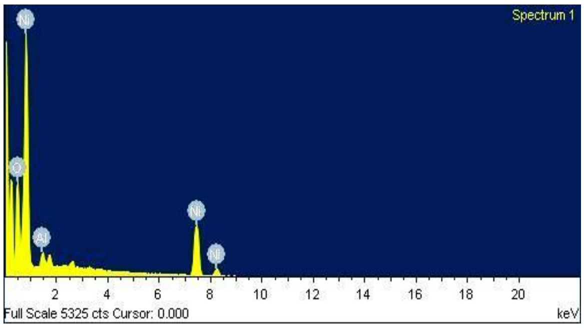

Figure 7: EDS of Ni-Al_2O_3 coated specimen

To determine the chemical composition of the different phases observed in Figure 6, 7 energy dispersive spectroscopy (EDS) study was performed on different region on the coated surface. The deposition of TiO_2 & $Al_2O_3$with Ni is confirmed by Energy Dispersive Spectro-photograph (EDS).

Conclusion

Results of this study have shown that there are different failure mechanisms when Ni-Al_2O_3 coatings are subjected to micro-scratch testing. The failure behavior in this study was categorized into three stages depending on the dominant failure mechanisms. For the as deposited coatings, the initial failure was trackside cracking followed by trackside delamination and tensile cracking and finally by delamination along the scratch path. In this case, track- side cracking was not observed. The resulting failure mechanism and behavior are believed to depend on a number of factors which include machine or testing dependent factors (e.g. loading rate, scanning speed, etc.) and coating and substrate property related factors (e.g. hardness, surface roughness, etc.). In conclusion, the resulting failure behavior of the Ni-Al_2O_3 coatings is influenced by the interaction of the factors mentioned. The result of this study may be useful in under- standing the Ni-Al_2O_3 coating failure behavior. The scratch tester is found more adhesion strength in Ni-$TiO_2Al_2O_3$Coating as compared to Alumina oxide coating. The result shows that Excessive addition of titanium can increase more amount of hardness. The surface morphology of the coated layers studies by SEM shows the deposition of fine grained structures (cauli-flower like) at low currents, which increases the strain produced due to lattice defects and act as a basis for increase in hardness. The deposition of TiO_2 & Al_2O_3 with Ni is confirmed by Energy Dispersive Spectro-photograph (EDS). The hardness of the resultant coatings is found to be 66.3HRC for Ni-$TiO_2Al_2O_3$coating and 55.8 HRC for Al_2O_3 coating depending on the particle volume in the Ni matrix. The SEM shows that the particle size of the Ni-$TiO_2Al_2O_3$ coating is fine as compare to the Ni-Al_2O_3 coating and its look like a lotus flower and cauli-flower (dirt-repellent).

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