Optimization of loading factor of Nanocomposite Coatings deposited by Physical Vapor Deposition

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Abstract. The Physical Vapor Deposition (PVD) is a technique in which metal or metal alloy vapors are deposited on the surface of the tools in a vacuum chamber. The positioning of the tools i.e. fixture and loading factors (% area of the chamber filled with tools) influence the flow of the vapors in the chamber. This results in a significant effect on the coating characteristics which affect the film properties. Nano Composite Coatings i.e. AlTiN/Si\textsubscript{3}N\textsubscript{4} (nACo) or AlCrN/Si\textsubscript{3}N\textsubscript{4} (nACRo) deposited by the PVD technique show superior hardness, toughness, heat and oxidation resistance. The adhesion and diffusion of these Nano structured films on substrate depend on proper surface cleaning steps and loading factors. In present work, nACo and nACRo coatings were done by changing the loading factors (50% to 75%) for coating. These coated samples were characterized and the tribological, mechanical and wear properties were studied. It was observed that; fixture, surface cleaning steps of substrate and vacuum surface areas (loading factors) have influenced the adhesion and thickness properties of the coated films. The 75% loading factors greatly enhance the efficiency of tools coated with both nACo and nACRo structured films.

1. Introduction

Surface engineering is defined as alteration in the properties of the surface phase in order to reduce the degradation over time. This is done by making the surface robust to the environment in which it will be used. Surface engineering techniques are being used in a number of industries such as; automotive, aerospace, missile, power, electronic, biomedical, textile, petroleum, petrochemical, chemical, steel, power, cement, machine tools, and construction [1]. Titanium Nitride (TiN) and Titanium Aluminum Nitride (TiAlN) have been widely used as protective hard coatings for cutting tools. The demand for the improvement of machining technology requires higher cutting speed, better quality of the machined surface, lower consumption of lubricants and coolants. These requirements can be fulfilled by depositing wear resistant coatings on machine tools [2].

Coated materials are normally used in operations which require high hardness, wear resistance, thermal and chemical stability of the surface. Coatings deposited by physical vapor deposition of the metal vapors or metal on the surface of the stressed materials should satisfy the conditions related to maintaining sufficient quality of the coated parts during their application in practice. Thin coatings applied to the surface of machine parts and tools must show relevant requirements, such as high hardness and modulus, resistance to wear, thermal and chemical resistance, good adhesion and adequate roughness values. Determination of properties of thin PVD coatings mainly depends on the correctly selected methods for their successful application in the practice. [3]. In the present work, nACo and nACRo coatings were deposited by changing the loading factors (50%, and 75%). The tribological, mechanical and wear properties of these coated samples were studied. It was observed that fixture, surface cleaning steps of substrate and vacuum surface areas (loading factors) have influenced the adhesion and thickness properties of the coated films. The 75% loading factors greatly enhance the efficiency of tools coated with both nACo and nACRo structured films.
2. Experimental work

The textile thread guides of SS material were used for present work. In present work, nACo and nACRo coatings were developed by changing the loading factors (50%, and 75%). The samples were coated using the Physical Vapor Deposition System (PLATIT π 80).

2.1 Surface preparation

Textile thread guides were first ultrasonically cleaned by washing them in an ultrasonic bath using Carbon Tetra chloride (CCl₄) for 5 minutes. After washing, the samples were thoroughly treated with distilled water to remove the traces of CCl₄ followed by washing the samples in an ultrasonic bath containing acetone. After the removal of samples from the ultrasonic bath they were carefully dried using an air compressor. Then Samples were placed in a Cleaning solution (Clean-Gel) at 60°C for about 10 minutes.

2.2 Deposition of coating

Two types of coatings, nACo and nACRo with coating thickness of 2 µm and 2.5 µm were done on Textile Thread Guides using Physical Vapor Deposition System (Platit π 80) with two lateral rotating cathodes. The coating deposition was conducted in a flowing nitrogen atmosphere under a working pressure of 1.5 Pa. The pre-cleaned Textile Thread Guides along with test coupons were mounted on carrousel substrate holders. This rotates continuously around the vertical central axis at a speed of 12 rpm. During deposition, a negative DC bias of -70 V was applied to the substrates.

2.3 Characterization of the coating

nACo and nACRo coatings were characterized for their mechanical and tribological analysis, which includes the following techniques:

2.3.1 Coating thickness measurement

The coating thickness was measured with the help of Calo-tester as shown in figure 1. A rotating ball of 20 and 30 mm diameter was pressed on the coating surface with a preselected load. The position of the sphere relative to the sample and the contact load were kept constant. Abrasive slurry was added to the contact zone and a depression with the shape of a spherical cap was abraded into the coating and the substrate. The resulting impression was viewed under the image analyzer system at 100X magnification and values of X and Y was recorded.

![Figure 1. Principle of calo-tester [4]](image1)

2.3.2 Cohesion and adhesion behavior

Adhesive behavior of the coating was investigated using Rockwell Hardness Tester (C scale with an applied load of 150 Kg). The resulting impression was investigated under the Image Analyzer System and comparison was done with the chart shown in the figure 2. The acceptable adhesion depends on consumer application:
Normal HSS tools: HF1-HF4 acceptable  
Normal HM tools: HF1-HF3 acceptable  
High performance tools: HF1-HF2 acceptable

Figure 2. Standard coating adhesion chart [5]

2.3.3 Tribological study
Tribological behavior of the coating was investigated using Atomic Force Microscope (AFM) in the contact mode. Si₃N₄ Tip was used and a 10 µm scan rate was adopted for all the coatings. For each coating, three points were investigated and average values of Roughness (Ra and Rq) were reported.

2.3.4 Mechanical properties
Hardness and Modulus values of the coatings were measured using CSM Nano Indentation Testing equipped with a berkovich diamond indenter (a three-sided pyramid). The Oliver and Phar method was used for hardness calculation [6]. The applied load was 10 mN, while maximum indentation depth was set at 250 nm, which is less than one tenth of the coating thickness. Thus the substrate effect on the measured hardness can be neglected.

3. Results & discussion

3.1 Adhesion testing
Two batches of nACo with coating thickness of 2 and 2.5 micron were deposited. The standard cleaning procedure was adopted on the substrate before coating. Rockwell hardness test was used to access Rockwell adhesion. This test method uses a standard Rockwell hardness tester causing damage adjacent to the boundary of the indentation. The damage to the film was compared with a defined adhesion strength quality. The adhesion values are measured in HF values. HF1-HF4 is defined as good adhesion while HF5-HF6 is defined as poor adhesion. The adhesion values in the range of HF1 were obtained as sown in figure 3 (a and b). These values are showing an excellent adhesion of nACo with coating thickness of 2 and 2.5 microns.
3.2 Coating Thickness Measurement

The thickness of coatings deposited on cutting tools is one of the most important characteristic for their practical application as it affects the cutting life of these tools. Rapid and simple determination of coating thickness is carried out by a Calotester. The results of coating thickness values of nACo and nACRo batches are in acceptable range. Table 1 depicts that thickness values are somewhat lower than the values which are set in the recipe during the coating. This loss in thickness can be understood as some of the coating material sticks to the chamber walls and sample holders as well; resulting in slight decrease of the thickness values.

By measuring the parameters X and Y, the thickness of the coating can be calculated by a simple geometrical equation.

\[
d = \frac{X^2 - Y^2}{4 \times D \times 1000}
\]

Where;
- \(d\) = Coating thickness (in microns)
- \(X\) = Diameter of the outer circle
- \(Y\) = Diameter of the inner circle
- \(D\) = Diameter of Ball used in the experiment
Table 1. Coating Thickness Values of all the four batches

|                        | X (mm) | Y (mm) | D (mm) | d (µm) |
|------------------------|--------|--------|--------|--------|
| nACo 2µm               | 563.30 | 429.50 | 20     | 1.66   |
| nACo 2.5µm             | 474.10 | 280.90 | 20     | 1.82   |
| nACRö 2µm              | 560.10 | 430.50 | 20     | 1.60   |
| nACRö 2.5µm            | 860.20 | 693.50 | 30     | 2.15   |

3.3 Tribological Properties

Tribological behavior of nACo and nACRö batches was studied on AFM. Table 2 shows the roughness values of these coatings.

Table 2. Roughness values of nACo(2 & 2.5 µm) and nACRö coatings (2 & 2.5 µm)

|        | nACo (2 µm) | nACo (2.5 µm) | nACRö (2 µm) | nACRö (2.5 µm) |
|--------|-------------|---------------|--------------|----------------|
| Ra     | 25.22 nm    | 27.45 nm      | 29.52 nm     | 34.06 nm       |
| Rq     | 32.45 nm    | 35.37 nm      | 50.56 nm     | 58.99 nm       |

The results show that the average roughness (Ra) values of nACo coatings (both 2 and 2.5 microns) are 25.22 nm and 27.45 nm simultaneously while; the root mean square roughness (Rq) are 32.45 nm and 35.37 nm. The Ra values of nACRö coatings (both 2 and 2.5 microns) are 29.52 nm and 34.06 nm simultaneously while; Rq values are 50.56 nm and 58.99 nm. This comparison shows that both Ra and Rq values of nACo coatings are lower than nACRö Coatings. Figures 5 & 6 explain the 3D analysis of nACo with coating thickness of 2 & 2.5 µm while figures 7 & 8 depict the 3D images of nACRö with coating thickness 2 & 2.5 µm. The 3D images of nACo coatings clearly showing the fineness of surface having lower roughness values than the 3D images of nACRö coatings.
3.4 Mechanical Properties

Table 3 is showing the hardness ($H_{IT}$) and modulus of elasticity ($E_{IT}$) values for nACo and nACRo coatings with 75% loading factor. The applied load was 10mN to ensure that penetration depth should not exceed 200 nm.

|          | nACo (2 microns) | nACo (2.5 microns) | nACRo (2 microns) | nACRo (2.5 microns) |
|----------|------------------|--------------------|-------------------|---------------------|
| $H_{IT}$ | 45.10 GPa        | 46.10 GPa          | 48.73 GPa         | 49.88 GPa           |
| $E_{IT}$ | 543.29 GPa       | 553.42 GPa         | 466.56 GPa        | 476.58 GPa          |

The results show that the hardness ($H_{IT}$) values of nACo coatings (both 2 and 2.5 microns) are 45.10 GPa and 46.10 GPa simultaneously while; the values of modulus of elasticity ($E_{IT}$) are 543.29 GPa and 553.42 GPa. The $H_{IT}$ values of nACRo coatings (both 2 and 2.5 microns) are 48.73 GPa and 48.73 GPa simultaneously while; $E_{IT}$ values are 466.56 GPa and 476.58 GPa. This comparison show that the hardness values increases as the thickness of coating increases whereas the hardness values of nACRo are greater with lower modulus of elasticity than the values of nACo coatings.

4. Conclusions

Nano composite coatings produced a hard layer on the surface of textile guides; certainly these layers will improve the life and other properties of tool which are required in textile industry. Both coatings enhance the efficiency of cutting tool but mechanical evaluation confirms that nACRo coatings are better than the nACo Coatings in performance. In present work, nACo and nACRo coatings were deposited by changing the loading factors (50% to 75%) during vacuum processing. It was observed that fixture; surface cleaning steps of substrate and vacuum surface areas (loading factors) has influenced the adhesion and thickness properties of the coated film. The 75% loading factors greatly enhance the efficiency of tools coated with both nACo and nACRo structured films.

References

[1] Advanced Thermally Assisted Surface Engineering Processes’ Kluwer Academic Publishers, MA, USA (now Springer, NY), 2004, ISBN 1-4020-7696-7, E-ISBN 1-4020-7764-5
[2] R Chattopadhyay, “Surface Wear- Analysis, Treatment, & Prevention”, ASM-International, Materials Park, OH, USA, 2001, ISBN 0-87170-702-0
[3] Mária HAGAROVÁ, “Experimental Methods of Assessment of PVD Coatings Properties”, Journal of Metals, Materials and Minerals. 2007 17(2), pp.29-35
[4] http://www.csm-instruments.com/new/contenus/e.shtml/calotest.shtml
[5] http://platit.com/periphery/quality-control-system?page=0%2C1
[6] W.C. Oliver, and G.M. Pharr, “An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments”, J. Mater. Res., vol. 7(6), 1992, pp. 1564-1583