EFFECT OF RECIPROCATING SLIDING SPEED ON THE TRIBOLOGICAL PERFORMANCE OF NANO Al$_2$O$_3$ REINFORCED NI MMC$_S$ PRODUCED BY DC AND PULSE ELECTRO CO-DEPOSITIONS

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Abstract

In this study, Ni/Al$_2$O$_3$ metal matrix composite (MMC) coatings were prepared from a modified Watt's type electrolyte by direct and pulse current (DC and PC) plating methods, and the effect of current density is investigated. The tribological tests were performed by a reciprocating ball-on disk apparatus sliding against M50 steel ball (Ø 10 mm). The wear tests were carried out at different sliding velocities of 50 mm/s, 100 mm/s and 150 mm/s by using a constant load. Increasing sliding velocity resulted in increasing wear rate in DC produced coatings but decreasing in PC co deposited materials.

1. Introduction

Electrolytic co-deposition is widely used to obtain metal matrix composites due to its ease of preparation, low-cost and versatility. Extensive research efforts, over the past decade and a half, have concentrated on conventional D.C. electroplating, pulse plating and electroless plating, as well as co-deposition processes to produce nanocrystalline materials. Electrodeposition of composite coatings containing second phase particles dispersed in the metal matrix has been the objective of investigation for industrial applications date back to 1970 [1].

In particular, composite coatings incorporated with different kinds of particles exhibit distinctly improved properties, such as high hardness, high wear resistance and corrosion resistance, as contrasted with pure metal or alloy coatings [2-6]. Particle-reinforced MMCs generally exhibited wide engineering applications due to their enhanced hardness, better wear, and corrosion resistance when compared to pure metal or alloy [7-11]. Oxides, carbides, diamond particles, nitrides, oxometallates and oil-containing microcapsules were incorporated into the nickel matrix to improve tribological properties of these materials [12-21]. Research on electrodeposition of nano-composite coatings has been mainly focused on the determination of optimum conditions for their production; electrolysis conditions (composition of the electrolytic bath, presence of additives, pH value), current conditions (type of imposed current and values of the current density) [22-25].

The purpose of this study is to deposit a layer, composed with soft Ni and hard nanosized ceramic particles by DC and PC plating method. Since the Ni coating has poor wear

resistance, it was suggested to increase the hardness of the Ni coating and wear resistance by optimizing current density parameters. Although there are several studies on the wear of Ni-Al_2O_3 nanocomposite coatings, there is no such specific study to compare the current type (direct or pulsed) and sliding speed in Ni-Al_2O_3 coatings.

2. Experimental Procedure

The plating electrolyte for the electrodeposition of the nano particle reinforced MMCs was a Watt's-type bath. The bath composition and electrodeposition conditions are shown in Table 1. The average particle size of the α-Al_2O_3 used for the experiment as reinforcing phase is 80 nm. Prior to deposition, zeta potential of the nano particle suspended solution was measured with Malvern Zetasizer Nano Series Nano-ZS model instrument.

In the electrodeposition experiments, four different current densities of 1, 3, 6 and 9 A/dm^2 and two current types (DC and PC) were studied to obtain optimum conditions for homogeneous microstructures and wear resistance. Plating time was 2 hours for each electroplating run. In this process, substrate was polished and the electrolyte of plating bath was prepared with nano particles and then was stirring with a magnetic stirrer for 20h and then dispersed ultrasonically for 0.5h. Microstructural investigations were performed by JEOL-JSM 6060LV instrument. Rigaku D/MAX/2200/PC model device was used for X-ray analysis at speed of 1°/min and range between of 10-100°. From the XRD pattern results. The hardness of the coatings was measured by using a Vicker's microhardness indenter (Leica VMHT) with a load of 50 g.

Wear and friction tests were performed with a reciprocating ball-on disk CSM tribometer at room temperature with a relative humidity of 55–65 % under dry sliding conditions. Counterpart was a M50 steel ball (Ø 10mm) suitable to DIN 50324 and ASTM G 99-95a. The system allows measuring friction coefficient and time dependent depth profiles by using sensitive transducers. The depth transducer was vertically located on top of the sample. The tests were performed at a constant applied load of 1.0 N with a sliding speed of 50 mm/s. The amount of wear of the composites after each test was calculated by measuring the wear width and dept by using 3D surface profiler (KLA Tencor P6) and low magnification optical micrographs. These measurements were also compared with the vertical transducer depth profiles and thus, the wear rate of the composites and the steel ball were determined.

Table 1. Bath compositions and electrodeposition conditions for nano Al_2O_3 reinforced MMC production.

Nickel sulphate ($Ni_2SO_4.6 H_2O$) (g/l)	300
Nickel chloride ($NiCl_2.6 H_2O$) (g/l)	50
Boric acid (H_3BO_3) (g/l)	40
Sodyumdodecyl sulphate (g/l)	0,1
Hexadecylpyridinium bromide (HPB) (mg/l)	200
Alumina (Al_2O_3) (g/l)	20
pH	4
Temperature (°C)	45
Current density (A/dm^2)	1, 3, 6, 9
Current type	DC and PC
Duty Cycle	50%
Pulse Frequency	50 Hz
Plating time (h)	2

3. Results and discussions

3.1. Effect of current density on deposition

Fig. 1 and 2 show the effect of current density on the volume percentage of Al_2O_3 in coatings. In order to determine the current density on the microstructural and wear properties of the resultant coatings, the surfactant (HPB) and particle concentration in the electrolyte were kept constant as 200 mg/l and 20 g/l, respectively. It is observed that the volume percentage of Al_2O_3 in coatings increases initially with the current density (approximately 9 % volume fraction) and reaches to the maximum at 3.0 A/dm^2. Increasing current density in DC coated nanocomposites no important particle content increase was observed. However, in the composites that produced with PC method, the particle content in the 6.0 A/dm^2 is decreased. Increasing current density to 9 A/dm^2 resulted in a very sharp increase with the Al_2O_3 particle in the electrodeposition. Beyond this current density, the co-deposited Al_2O_3 content is almost stable. Another feature is very obvious that the homogenization of particle distribution is very outstanding in the composites produced PC method.
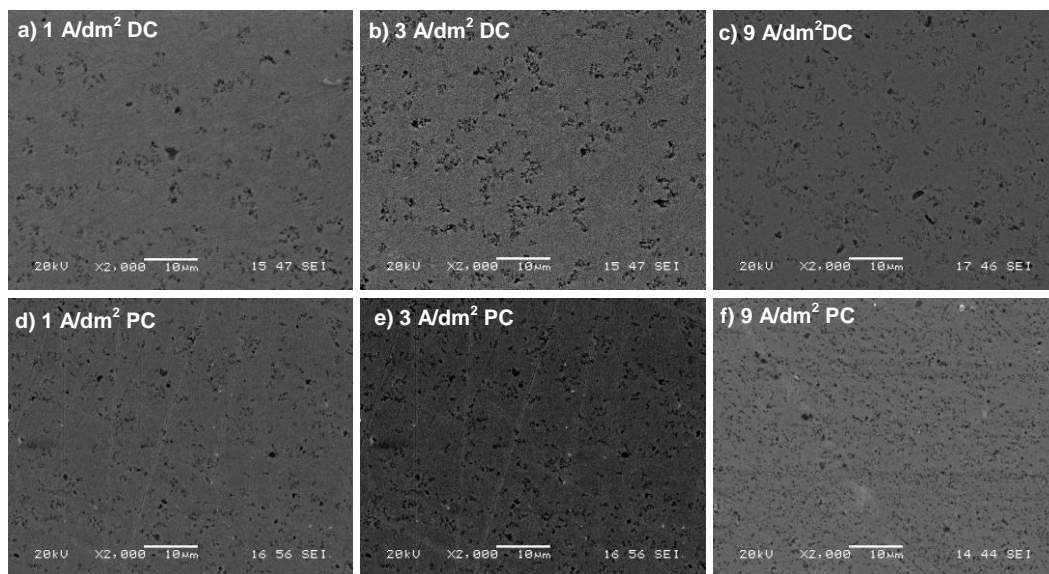

Fig. 1. Cross sectional SEM micrographs of MMCs co-depositions showing distribution of Al_2O_3 particles coated with current densities; a)1 A/dm^2 DC, b) 3 A/dm^2 DC, c) 9 A/dm^2 DC d) 1 A/dm^2 PC, e) 3 A/dm^2 PC and f) 9 A/dm^2 PC.

The observed maxima on the curve of current density versus volume percentage of Al_2O_3 in coatings can be attributed to the transition from an activation-controlled metal deposition reaction to a diffusion-controlled of particles transfer [25].
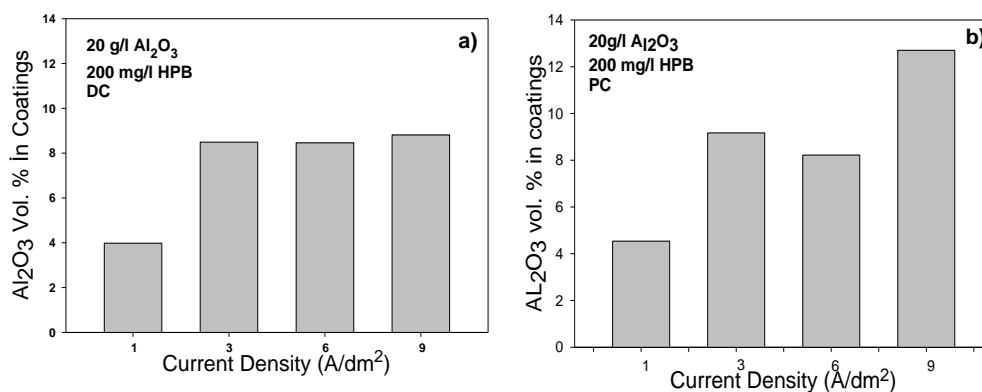

Fig 2. The volume percentage of co-deposited Al_2O_3 particles in various current densities for each current type. a) direct current and b) pulse current.

3.2. Microhardness of composite coatings

Fig. 3 compares the microhardness of unreinforced Ni, Ni-Al$_2$O$_3$ composite coatings, which are produced DC and PC methods. The microhardness of the coatings, basically, increases with increasing dispersed nano particle content. The improvement in the hardness of composite coatings is related to the dispersion hardening effect caused by nano Al$_2$O$_3$ particles in the composite matrix, which obstructs the shift of dislocation in the nickel matrix [26]. From Fig. 3, it can be seen that the microhardness of Ni–Al$_2$O$_3$ composite coating is higher than that of pure Ni coating and increases with the increase with the nano-Al$_2$O$_3$ content in coatings. There are three reasons behind the increase in hardness [13, 17, 22, 25]; particle-strengthening, dispersion-strengthening and grain refining. Particle-strengthening is related to the incorporation of hard particles and volume percent above 20%. Dispersion-strengthening is associated with the incorporation of fine particles (<1 μm) and volume fraction lower than 15%. In this case, the matrix carries the load load, the small particles hinder dislocation motion. The third mechanism is related to the nucleation of small grains on the surface of the incorporated particles, resulting in a general structural refinement. The presence of smaller grains impedes dislocation motion resulting in an increase in microhardness. The results can be explained by the second and third mechanisms. This revealed that the fine particles incorporated within the Ni matrix could restrain the growth of Ni crystals and impede the motion of dislocations, by way of grain refining and dispersive-strengthening effects.
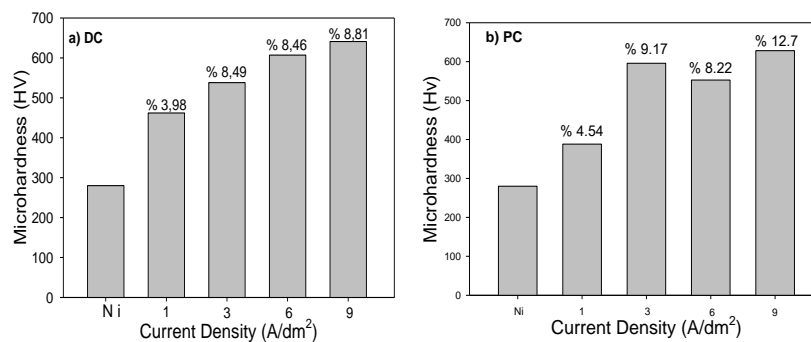

Fig. 3. Effect of current density on microhardness with produced direct (a) and pulse current (b) composite coatings.

3.3. Wear and friction Properties

3.3.1. Effect of current density on wear and friction properties

The relationship between current density and composites and coefficient of friction are shown in Fig 4. From the Fig 4, it can be clearly seen that increasing current density causes to decrease in wear rate for the composites which are produced with DC plating method. (Fig. 4a) However, the composites which are produced with PC plating method the wear rate not affected directly from increases with current density. The effect of sliding speed on the wear rate of these composites produced with PC method is more impressive. When the wear rate of the coated nanocomposites between DC and PC plating method is compared it can be introduced that the wear rate decreases with producing PC method approximately from 17 x 10^{-4} mm^3/Nm to 2 x 10^{-4} mm^3/Nm at 150mm/s sliding speed. The decreasing at the wear rate was found about 8 times higher in the PC method compared with DC plating technique.
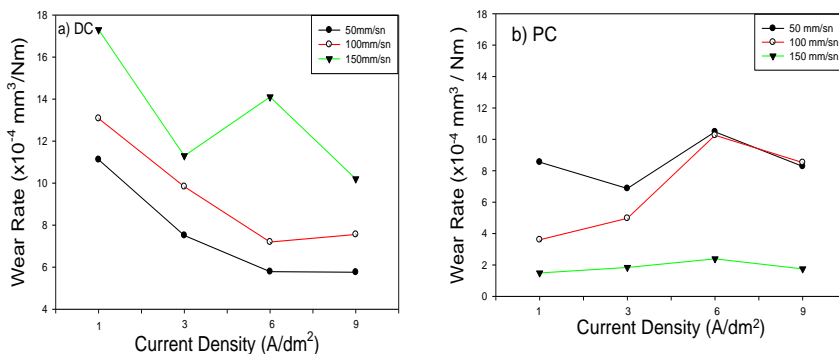
Fig. 4. Effect of sliding speed on the wear rate of Ni–Al$_2$O$_3$ composite coatings prepared with different current type and densities a) DC b) PC.

Fig. 5 shows the friction coefficient do not significantly affect from both plating methods. Since the deposition, process was carried out with the constant surfactant amount of 200 mg/l and particle concentration in the electrolyte of 20 g/l, the interfacial bonding between nano Al$_2$O$_3$ particles and Ni matrix is thought to be one of the most effective features in sliding wear process. It is known that PC current method provides not only higher amount of nano second phase particles in the electrodeposited layer but also better interface properties between matrix and ceramic particles [27]. It is already evident from our SEM micrographs that PC method provides more homogenous distribution and segregation free particle distribution. Because of this reason, increasing sliding speed in DC coated materials resulted to increase wear rate but in PC coated materials the inverse result was observed; increasing sliding speed, decreasing wear rate. This implies that the best load carrying capacity associated with tribo oxide formation governs the wear phenomena and decrease wear rate.
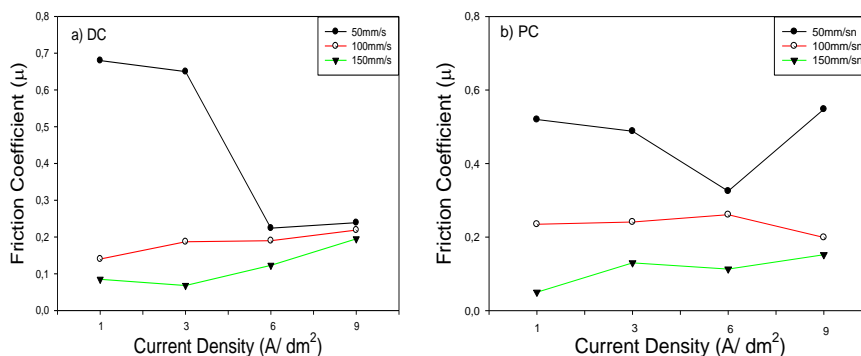

Fig. 5. Effect of sliding speed on friction coefficient of Ni–Al$_2$O$_3$ composite coatings prepared with different current current type and densities a) DC b) PC

It is evident from the Fig. 6 that the delamination cracks and smeared wear debris were found, which confirm that the wear process of Ni-Al$_2$O$_3$ composites is governed by the combining effect of abrasion and adhesion mechanisms. At low current density, because of the low amount of particle co-deposition, the appearance of worn surface (Fig. 6a) exhibits mainly the adhesion wear characteristics due to detachment of fragment from the nickel matrix surface. At high current densities, because of deformation disaccord between nano sized reinforcement phase and matrix, the tip of reinforcement phase gives rise to the stress concentration, and small debris are produced and expanded by the particle content increment. Increasing current density caused to form more wear debris (Fig 6b and c).
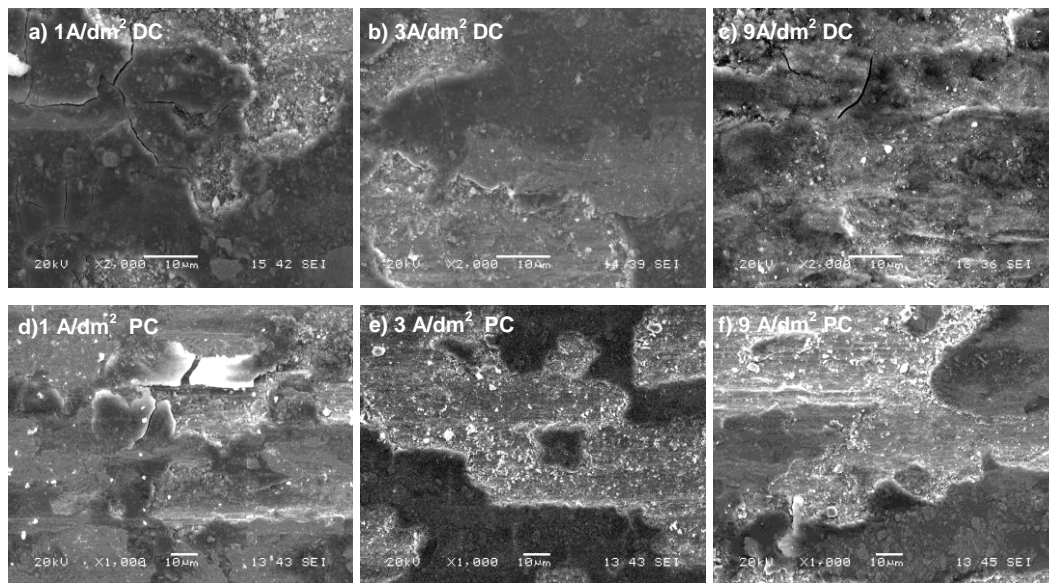
Fig. 6. SEM morphology of the wear tracks of composite coatings prepared with different current type and current densities for 50 mm/s sliding speed; a)1 A/dm^2 DC, b) 3 A/dm^2 DC, c) 9 A/dm^2 DC d) 1 A/dm^2 PC, e) 3 A/dm^2 PC and f) 9 A/dm^2 PC.

In Figure 6d, e and f the worn surfaces of the composites produced with PC method are presented after 50 mm/s sliding speed. As can be seen from Fig6. the worn surfaces of the coatings produced by PC method are comparably different from the worn surfaces of DC coated samples. The surfaces of the PC coated materials were experienced to be smoother than that of DC coated samples. Moreover, more amounts of very small de ris were also detected in PC coated materials compared with DC samples. The wear mechanism of the PC coated material is seen to be occurred starting by adhesive and continuing surface hardening and then fatigue that produces very small wear debris. The surfaces of the worn samples were also analyzed by EDS, and it was detected that increasing sliding speed in DC coated materials showed increasing amount of Al, and components transferring from the steel ball. In PC coated materials more amount of oxygen was detected, especially at the dark regions that seen in Fig. 6e and f.

To reveal and make a better comparison between the wear mechanisms of the DC and PC coated materials some selected worn nanocomposite surfaces were scanned with 3D profilometry. The results are presented in Fig. 7. For brevity, only the nanocomposites tests at 150 mm/s are chosen. It can be seen from the Fig. 7a that belongs to the worn surface of the DC plated composite produced at 1.0 A/dm^2 there is very rough surface and shows a very high amount of plastic deformation occurred and a large, deep valley was revealed. The rough surface is evidence that significant amounts of wear products were smeared on the surface, including agglomerated Al$_2$O$_3$ nano particles (Fig 7a). In the case of PC coated worn surface a very smooth surface was obtained (Fig 7b). Increasing current density resulted in decreasing, smearing and scuffing on the wear surface. It is probably because of increasing particle content in the deposited layer by increasing current density in DC method (Fig 7c). Applying PC current and then wear testing for 9.0 A/dm^2 current density yielded smoother surface and fewer protruded areas compared with the sample produced by DC plating. These results also prove PC plating produced better interfaces between nanocomposite constituents and provide better tribological behavior.
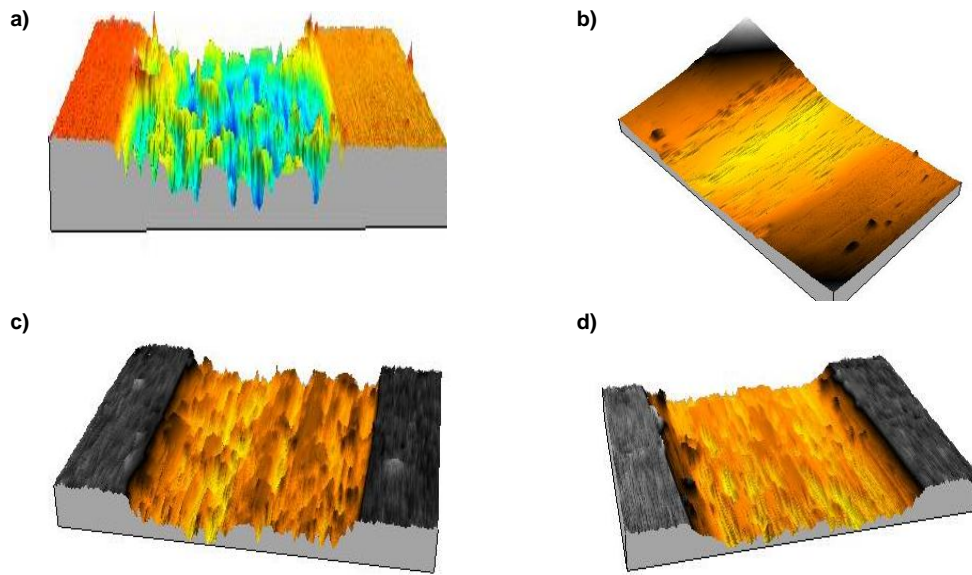
Fig. 7. 3D profilometry results f composite coatings prepared with different current type and current densities and wear tested at 150 mm/s; a)1 A/dm^2 DC, b) 1 A/dm^2 PC, c) 9 A/dm^2 DC d) 9 A/dm^2 PC

4. Conclusions

Ni matrix reinforced with nano Al$_2$O$_3$ (80 nm in size) MMC coatings were successfully produced by DC and PC method. A comprehensive investigation was aimed to accomplish to optimize the process parameters of current type and current density on the microstructural and tribological properties. The following results have been pointed out for the produced composite coatings;

1. Increasing the current density resulted in increasing of Al$_2$O$_3$ vol. % from 8.81 % which is produced with DC method and to 12.7, which is produced with PC method.
2. The hardness values of the nano Al$_2$O$_3$ reinforced electrodeposited coatings yielded as high as with DC plating method 641 Hv and with PC plating method, 628 Hv hardness values because of unique dispersion effect.
3. It is observed that the volume percentage of Al$_2$O$_3$ in coatings increases with the current density and reaches maximum values at about 8.81 vol. % at 9.0 A/dm^2 with DC plating method. It is observed that the volume percentage of Al$_2$O$_3$ in coatings, which is produced with PC plating method increases with the current density and reaches maximum values at about 12.7 vol. % at 9.0 A/dm^2.
4. The best wear rate was obtained with the 150 mm/s sliding speed in the coatings which is produced with PC plating method. When compared DC and PC plating method the wear rate decreases 8 times that is obtained 150 mm/s sliding speed.
5. The friction coefficient showed to be increased with process parameters except, current density, which caused to decrease from 0.5 to 0.2 for unreinforced Ni and the composite produced with 9.0 A/dm^2, respectively.

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