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Electrodepositing $Ni\text{-}Al_2O_3$ nanocomposite layers onto drug stamping pestle by selective electroplating

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ABSTRACT

Electrodepositing $Ni\text{-}Al_2O_3$ nanocomposite layers by selective electroplating technology is a technology that is becoming more popular today. It is very It is very suitable for equipment and machines, which non-movable or portable components. Here we report a one-step process of Electrodepositing $Ni\text{-}Al_2O_3$ on Steel 09CrSi, wich is made into a drug stamping pestle. The coating surface quality, microstructure, friction coefficient and wear resistance performance of the coatings were investigated. Comprehensive characterization of the layer coating is then performed by means of SEM and EDX techniques. The results showed that the surface after plating is smoother, the organization is also more uniform, the $Ni\text{-}Al_2O_3$ nacomposite coating shows a shiny. The plated sample has a friction coefficient less than that of the unpainted sample about 30%. The optimum coating thickness is 34-35 µm which is achieved when the plating time is 7 minutes.

KEYWORDS

$Ni\text{-}Al_2O_3$ nanocomposite coatings, micro hardness, selective electroplating, wear resistance, Al_2O_3 nanoparticles;

INTRODUCTION

Corrosion resistance for machine equipment, under wear and corrosion, is a major technical problem due to the harsh corrosive conditions in chemical and mechanical abrasive environments. Currently, a lot of different coating technologies in the world appear to improve the corrosion and abrasion resistance for these equipment such as metal coating, hot dip galvanizing, special paint, electroplating, depending on the specific cases, use the methods optimally. Selective electroplating is an electrochemical process that is used to coat and polish with local electrodes on a part of products. Selective electroplating is popularly used in repairing and resizing of worn and defective parts without having to disassemble or relocate the part to be plated [1,2]. In the plating process according to the plating technology, the engineer uses his hands to cover the place to be plated with plating solution [3]. Today, selective electroplating, is thriving due to its high universal versatility, saving costs, time and money. In rubbing plating, nickel rubbing is the most popular, because the nickel layer is easy to precipitate, high hardness, wear resistance, good rust resistance….[4].

Currently, the nanocomposite coating is receiving a lot of research interest, some publications on high quality Ni-P nanocomposite coatings, applied in the field protect against wear and corrosion of steel parts. The nanoparticles of size nm in the Ni composite coating, putting very hard nanoparticles (SiC, Al_2O_3, diamond) inside the Ni-P coating, the hardness of the composite is greatly improved, through which improves abrasion resistance [5]. For SiO_2 nanoparticles, used 20 nm-sized particles dispersed in commercial plating solution, with the reducing agent NaH_2PO_2 under the help of ultrasonic waves [2,8]. The coating film has a film forming rate of 10-12 µm/hour and the nanoparticles account for about 2% of the coating volume. Some reports also showed that the composite Ni-P/nano-SiO_2 coating with particles with the size of 10-20 nm was surface modified by γ-aminopropyltrimethoxysilane[4,9]. After plating, the coatings are heat treated at 400°C for 1 hour. The resulting coating has a thickness of 17 µm, is tight and free of cracks. Another way, Ni/Al_2O_3 nanocomposite showed that the hardness of the coating increased when the density of Al_2O_3 nanoparticles increased, with the content of Al_2O_3 nanoparticle 20g/*l*, hardness, abrasion resistance of the coating is 60% higher than that of pure Ni coating.

When applying SiO_2 nanoparticles to the Ni-P coating (2% volume) the coating hardness also increased from 785 HV to 970 HV, which could reach 1340 HV [1,10]. The temperature has the greatest influence on the micro-hardness of the composite coating (hardness can be increased up to 125%) [6,11-13]. Some studies around the world show that the presence of nanomaterials such as Al_2O_3, TiO_2, SiO_2 ... has significantly improved the hardness and abrasion resistance of the nickel plating, even helping the layer. Nickel plating retains its hardness when raised to 850ºC for 20 hours [7,14-16]. Wear resistance for punching punches is a very important issue, in the article, we want to demonstrate the good impact of the coating on the wear resistance of the product. First, researching and manufacturing the coating on the drug stamping pestle according to the rubbing method to improve the micro-hardness and gloss for the details. Next we discuss the analysis of chemical molecules in the coating. Finally, we focus on the analysis and evaluation of the wear resistance of the coating on the mortar pestle.

EXPERIMENTAL PROCEDURES

Sample preparation

The drug stamping pestle set to be studied and tested here is mounted on the ZP 31 machine. The detailed drawing of the mortar set as shown in Figure 1 is the 09CrSi steel.

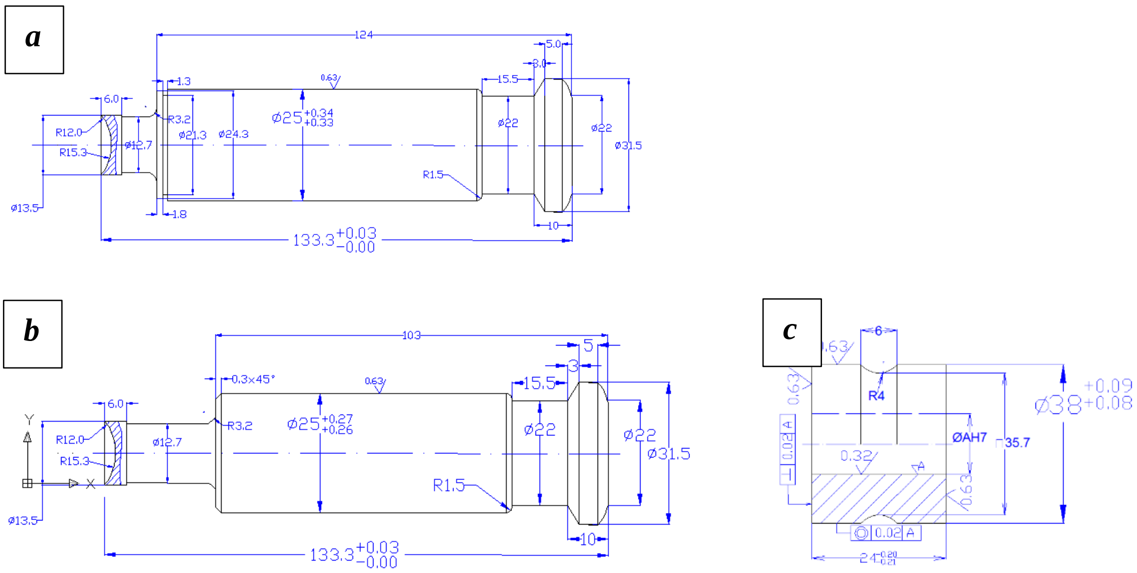

Figure 1. Drug stamping set; a: The upper brute; b: The lower brute; c: Jar

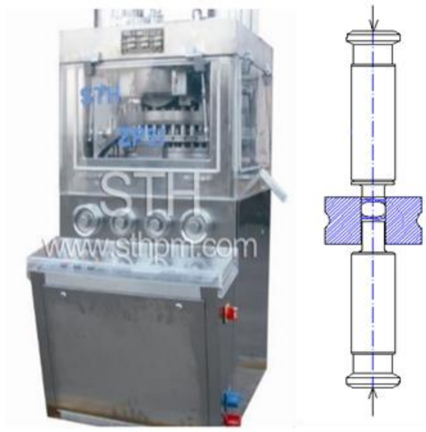

Figure 2. Structure of the ZP31 machine

Principle and working process of the sample

The diagram of the assembly of the mold and shape and structure of the ZP 31 pelletizing machine is shown in Figure 2. When the machine works, the rotary tray will carry 31 sets of rotating molds, the upper cam and lower cams are responsible for creating up and down movement for the upper and lower ram. At the blade position, the upper tibial is raised to the highest position, the lower tibial is lowered to the lowest position, where the powder is fed into the hole of the mortar and quantified by the position of the lower tibial and the brush. To the compression position, the cam mechanism will bring the upper ram to the lowest position, the lower tibial to the highest position and the compression wheel system will press on the tail of the tibial to create compression. The compression is adjusted by the eccentric shafts supporting the inner hole of the compression wheel.

Electrodepositing Ni-Al_2O_3 nanocomposite by selective electroplating

The chemicals used for Ni composite plating are listed in the Table 1. Sodium dodecyl sulfate is used as an additive to increase the surface activity of neutral particles. Neutral particles used in the experiment are Al_2O_3 with particle size ≤ 1 µm and Al_2O_3 content of 50 g/*l*.

Table 1. The chemicals used for Ni- Al_2O_3 composite

Ingredient	Unit (g/*l*)	Ingredient	Unit (g/*l*)
$NiSO_4.7H_2O$	325	Al_2O_3	75
$NiCl_2.6H_2O$	45	Zn	0,1
H_3BO_4	36	Fe	0,5
Na_2SO_4	25	Độ pH	4,5

The experiments in this study use a 100A coating system with flat electrodes made of graphite. The pH of the plating solution is adjusted to 4,5; for aduration of 7 min at 60°C. Sodium dodecyl sulfate is used as an additive to increase the surface activity of neutral particles. Neutral seed used in the experiment is Al_2O_3 with particle size ≤ 1 µm and Al_2O_3 content of 50g/*l*. The seeds have uneven size due to self-clumping into large seeds shown on SEM image (Figure 3). The Au peaks on EDX analysis are the result of Au coating on Al_2O_3 particle samples.

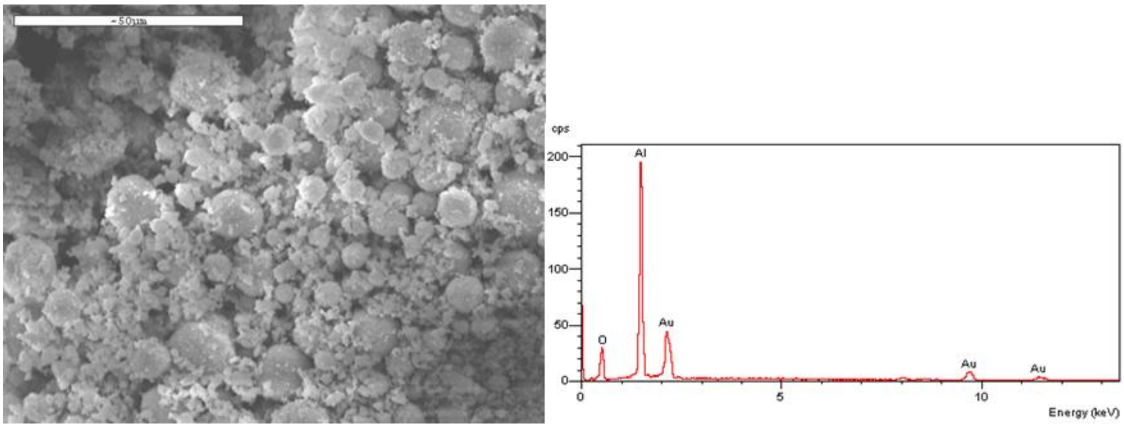

Figure 3. SEM, EDX image of neutral particles Al2O3

Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX).

Scanning electron micrographs of Ni-Al_2O_3 nanocomposite surfaces were recorded on a SEM TESCAN-BRUGER at10 kV in secondary electron imaging mode. The setup was also equipped with energy dispersive X-ray (EDX) detection system. EDX images were recorded on the surface of 1 µm2 using Pico SPM LE (Agilent Technologies/Molecular Imaging) at a scan velocity of 0.5 µm/s.

System stirring Ni-Al_2O_3 nanocomposite plating solution

The plating process uses a stirring tank with heating up to 90^0C and a stirring speed of 800 rpm used to heat the solution and stir to disperse Al_2O_3 particles into the plating solution (Figure 4).

Figure 4. System stirring Ni-Al_2O_3

Evaluation of resistance to abrasion and friction

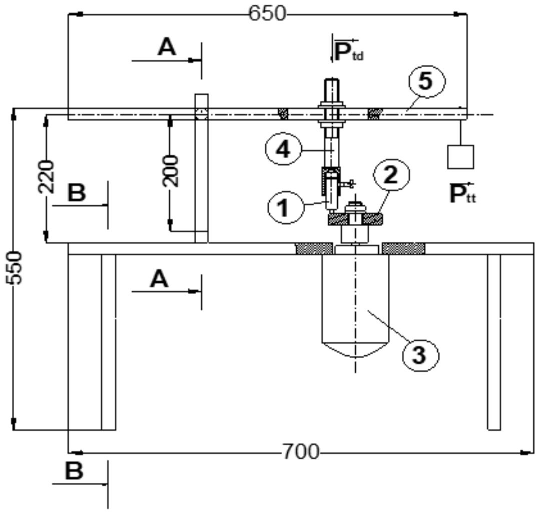

Figure 5. Diagram of POD machine

The wear resistance and friction reduction of Ni-Al_2O_3 nanocomposite layers (layer coating) was measured by a POD machine (Figure 5) working on the principle of sliding friction between the pin surface and homemade disc at room temperature 26 - 28^0C and a relative humidity of 50–60% under dry sliding conditions. Stainless steel balls with a diameter of 4 mm were used as the counterparts, and all the sliding tests were conducted at a load of 2,0 N and sliding speed of 10 mm/s.

RESULTS AND DISCUSSION

Evaluation of the coating microstructure through SEM technology

The results of the experiment were were analyzed by SEM TESCAN-BRUGER with the maximum magnification 10,000 times. The results of microscopic analysis show that the surface of 09CrSi steel substrate before and after plating is as shown in Figure 6. The surface before the plating is more rugged, the surface after plating is smoother, the organization is also more uniform, the Ni-Al_2O_3 nacomposite coating shows a shiny, beautiful image with high aesthetics.

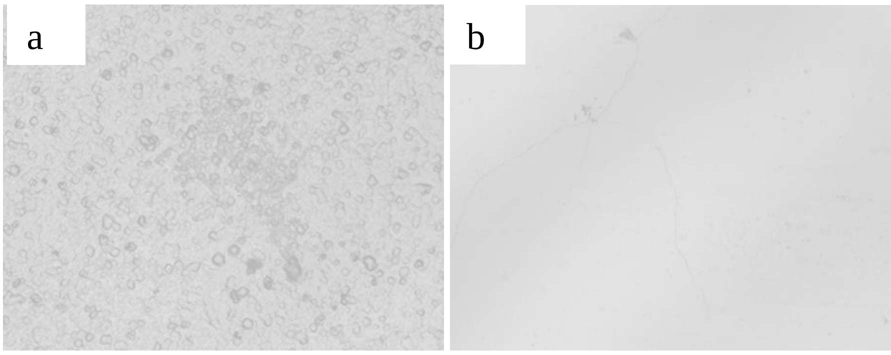

Figure 6. Sample surface analyzed by SEM (a) Before plating, (b) After plating

The SEM magnification image with magnification 1500 times in Figure 7 shows that the Ni-Al_2O_3 nacomposite coating of the experimental process has good adhesion on the substrate. After 7 minutes of coating time, the coating thickness shown on the TESCAN is about 34µm, the thickness of the coating is evenly distributed on the surface of the substrate even at the detail angle. Also on the Figure 7. we can be seen that the adhesion of the composite coating to the substrate is quite good (no gaps). However, the angle of the part has a poorer adhesion to the substrate, but is still good. After 10 minutes of plating, the coating still adhere well to the surface of the part and the thickness of the coating reaches about 80-90 µm (Table. 2).

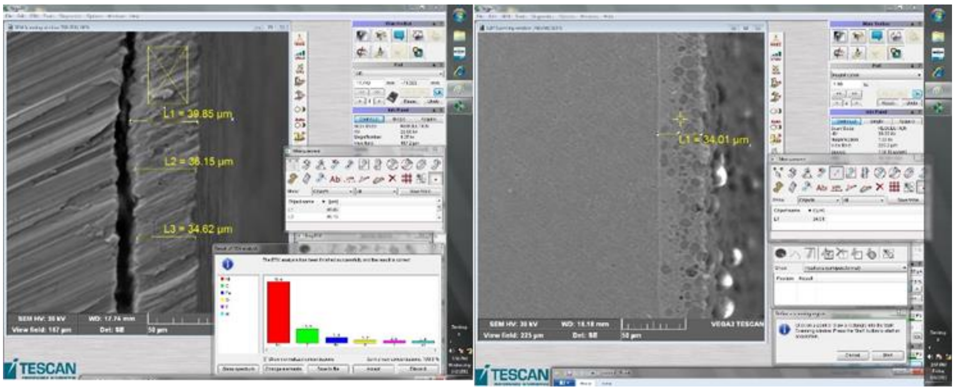

Figure 7. Image of SEM analysis of the coating thickness

Table 2. The coating thickness depends on the plating time

Time (minutes)	*05*	*06*	*07*	*08*	*09*	*10*
Thickness (µm)	*15*	*26*	*34*	*45*	*55*	*80*
Quality	*Good*	*Good*	*Good*	*Good*	*Good*	*Good*

Evaluation of the coating's microstructure through technology

X-ray energy scattering spectroscopy is a technique of analyzing the chemical composition of a solid body based on recording the X-ray spectrum emitted by a solid object due to interactions with radiation (which are mainly electron beams are of high energy in electron microscopes). In the scientific literature, this technique is often abbreviated as EDX or EDS derived from the name Energy-dispersive X-ray spectroscopy [7]. Figure 8 shows the EDX analysis of Ni-Al2O3 coating, the results show that the Ni content is 72%; C content is 18%; 5.1% is the content of O; the remaining 3.5% Al, 0.6% Fe, are metallic impurities mixed in the Ni plating. The chemical composition of the coating has a higher Ni content of more than 2/3, in addition the O and C substances create covalent bonds between the metal and the plastic substrate, the total of Ni, O and C substances accounts for 95, 1% coating content.

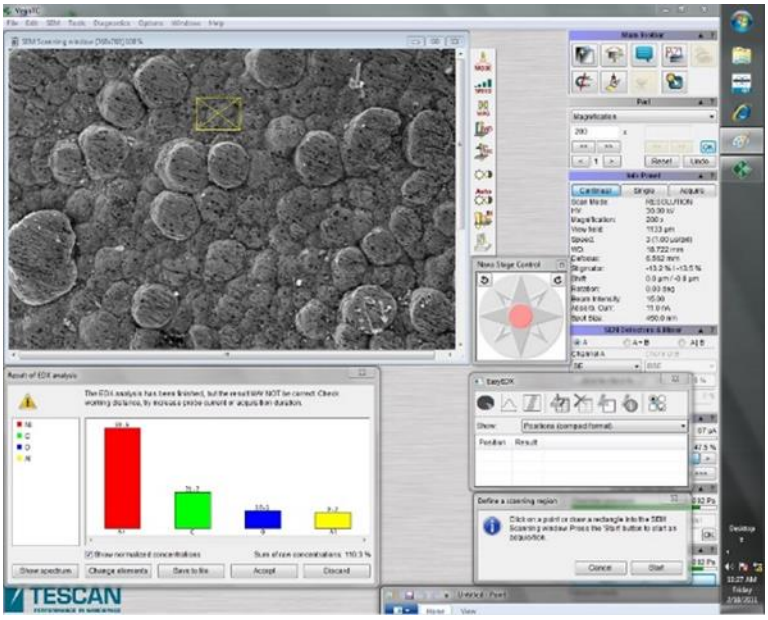

Figure 8. Image of EDX analysis of Ni-Al_2O_3 layer coating

Wear resistance of the nanocomposite coating

Wear and friction coefficient tests are conducted for different types of surface materials, respectively. Test results for the test samples with the 09CrSi alloy steel material surface, the sample surface is cleaned by ultrasonic method, and Al_2O_3–Ni plated after 7 minutes and 4 samples are not plated. The experiment was done with 2 pressure levels of 10N and 20N applied from the pin to the plate, equivalent to the load hanging on the lever arm is 3.65N and 7.3N. The wear assessment is tested on a rotating electric motor at 1450 rpm every 34 minutes (ie, equivalent to a rotating disc of 50,000 cycles), the sample is weighed once on a scale electronic scale. 1/10 gram then calculate the amount of lost material in the sample mm3. The fixed load hanging on the lever arm is 30 N. To reduce the effects of vibration and temperature, the POD machine is fastened to the floor by screws, the minimum rest time after each experiment is 15 minutes. After calculating the relationship between the wear amount of the sample and the contact time is redrawn as shown in Fig. 9. It can be seen from the graph of friction coefficient in Fig. 9 that the plated sample has a friction coefficient less than that of the unpainted sample about 30%. After about 5 test cycles (each cycle ≈34 minutes), because the tested sample is smoothed, the coefficient of friction reaches a stable value. The clad sample is 0.01 and the clad pattern is about 0.027.

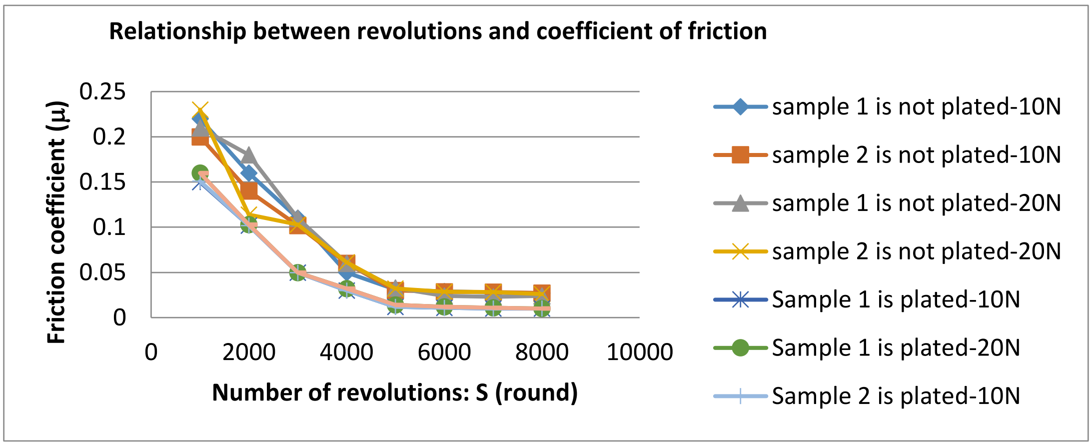

Figure 9. Relations S -μ

The resulting in Figure 10 is very similar to that of theoretical machine parts. It is worth noting that with the No. 4 plating sample, it can be seen that when a large number of turns is reached, the wear value increases very slowly at this time, which is of interest to develop wear resistance for materials. Technicians can check the mechanical properties of the pin and disc surfaces now, and then analyze them to come up with ideas for fabricating similar surfaces to increase the wear resistance of the product.

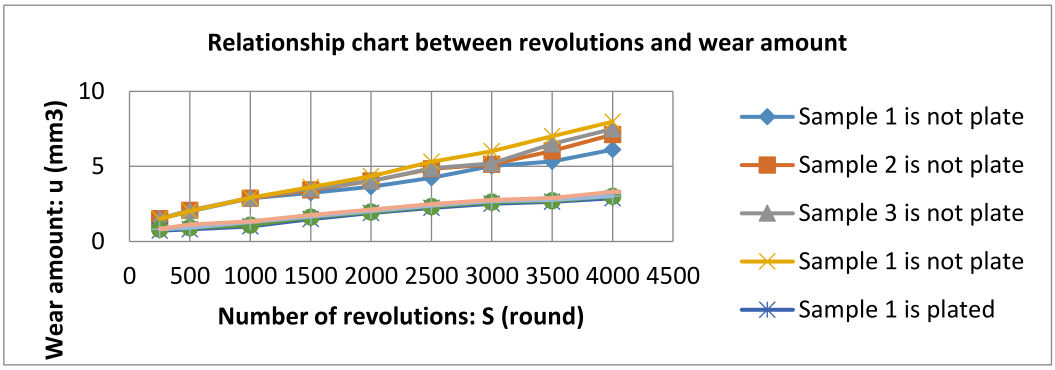

Figure 10. Relations S - u

CONCLUSIONS

In this work, we have reported a study about selective electroplating onto drug stamping pestle. In the experiments, we choose the plating theory is Selective electroplating Ni-Al_2O_3 on the material 09CrSi steel. From the experiments and analyses performed, some conclusions can be that the surface after plating is smoother, the organization is also more uniform, the Ni-Al_2O_3 nacomposite coating shows a shiny, beautiful image with high aesthetics. After 7 minutes of coating time, the coating thickness shown on the TESCAN is about 34µm, the thickness of the coating is evenly distributed on the surface. The plated sample has a friction coefficient less than that of the unpainted sample about 30%. This can be considered as the optimal thickness for the wear resistance of the Ni-Al_2O_3 nacomposite coating on the material steel. When a large number of turns is reached, the wear value increases very slowly at this time, which is of interest to develop wear resistance for materials

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REFERENCES

[1] L. Du, B. Xu, S. Dong, H. Yang, and Y. Wu, “Preparation, microstructure and tribological properties of nano-Al2O3/Ni brush plated composite coatings”, Surface and Coatings Technology, Vol. 192, No. 2-3, Pp. 311-316, 2005.

[2] T. Rabizadeh, and S.R. Allahkaram, “Corrosion resistance enhancement of Ni–P electroless coatings by incorporation of nano-SiO2 particles”, Materials & Design, Vol. 32, No. 1, Pp. 133-138, 2011.

[3] T.T. Do, T.Q. Le, D.T. Le, and A.D. Nguyen, “Research on fabrication of wear-resistant Ni/Al2O3 nanocomposite coating by selective electroplating”, The 5th National Science and Technology Conference on Mechanics - VCME 2018, Vietnam, Pp. 316-324, 2018.

[4] D. Dong, X.H. Chen, W.T. Xiao, G.B. Yang, and P.Y. Zhang, “Preparation and properties of electroless Ni–P–SiO2 composite coatings”, Applied Surface Science, Vol. 255, No. 15, Pp. 7051-7055, 2009.

[5] A. Sadeghzadeh-Attar, G. AyubiKia, and M. Ehteshamzadeh, “Improvement in tribological behavior of novel sol-enhanced electroless Ni-P-SiO2 nanocomposite coatings”, Surface and Coatings Technology, Vol. 307, Pp. 837-848, 2016.

[6] Q.T. Phan, D.B. Nguyen, and M.D. Tran, “Effects of process parameters on the micro-hardness of composite coating”, Journal of Science and Technology, VNU University, No. 78, Pp. 3-7, 2011.

[7] B. Wu, B.S. Xu, B. Zhang, and Y.H. Lü, "Preparation and properties of Ni/ nano-Al2O3 composite coatings by automatic brush plating", Surface and Coatings Technology, Vol. 201, No. 16-17, Pp. 6933-6939, 2007.

[8] X.T. Le, P. Viel, P. Jégou, A. Garcia, T. Berthelot, T.H. Bui, and S. Palacin, "Diazonium-induced anchoring process: an application to improve the monovalent selectivity of cation exchange membranes", Journal of Materials Chemistry, Vol. 20, No. 18, Pp. 3750-3757, 2010.

[9] J. Pinson, and F. Podvorica, "Attachment of organic layers to conductive or semiconductive surfaces by reduction of diazonium salts", Chemical Society Reviews, Vol. 34, No. 5, Pp. 429-439, 2005.

[10] A. Mesnage, S. Esnouf, P. Jégou, G. Deniau, and S. Palacin, "Understanding the redox-induced polymer grafting process: A dual surface-solution analysis", Chemistry of Materials, Vol. 22, No. 23, Pp. 6229-6239, 2010.

[11] D. Xu, E.T. Kang, K.G. Neoh, Y. Zhang, A.A.O. Tay, S.S. Ang, and K. Vaidyanathan, "Selective electroless plating of copper on (100)-oriented single crystal silicon surface modified by UV-induced coupling of 4-vinylpyridine with the H-terminated silicon", The Journal of Physical Chemistry B, Vol. 106, No. 48, Pp. 12508-12516, 2002.

[12] M. Charbonnier, M. Romand, Y. Goepfert, D. Leonard, F. Bessueille, and M. Bouadi, "Palladium (+ 2) reduction: A key step for the electroless Ni metallization of insulating substrates by a tin-free process", Thin Solid Films, Vol. 515, No. 4, Pp. 1623-1633, 2006.

[13] Q. Xu, Z. Liu, X. Hu, L. Kong, and S. Liu, "Interaction of adenine with palladium chloride, determination of adenine via resonance Rayleigh scattering method", Analytica chimica acta, Vol. 707, No. 1-2, Pp. 114-120, 2011.

[14] S.T. Chen, and G.S. Chen, "Nanoseeding via dual surface modification of alkyl monolayer for site-controlled electroless metallization", Langmuir, Vol. 27, No. 19, Pp. 12143-12148, 2011.

[15] Y. Shacham-Diamand, T. Osaka, Y. Okinaka, A. Sugiyama, and V. Dubin, "30 years of electroless plating for semiconductor and polymer micro-systems", Microelectronic Engineering, Vol. 132, Pp. 35-45, 2015.

[16] P. Zhang, M. Wang, Y. Yang, T. Yao, H. Han, and L. Sun, "Electroless plated Ni–Bx films as highly active electrocatalysts for hydrogen production from water over a wide pH range", Nano Energy, Vol. 19, Pp. 98-107, 2016.