



A comparative study of the effect of mechanical and ultrasound agitation on the properties of electrodeposited Ni/Al₂O₃ nanocomposite coatings

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ABSTRACT

In our study, Ni/Al₂O₃ nanocomposite coatings were obtained by electrochemical deposition of alumina nanoparticles from nickel Watts baths with different concentrations of Al₂O₃ particles in solution. A comparison of the effects of mechanical (300 rpm) and ultrasonic (24 kHz, 38 W cm⁻²) stirring on the properties of electrodeposited Ni/Al₂O₃ composite coatings was performed, observing an enhancement of the Al₂O₃ incorporation in the composite coating and an improvement of the uniformity of particles distribution in the nickel metal matrix when ultrasonic energy was applied to the process. Moreover, the characterization of the coatings also showed that for both types of agitation, the incorporation of Al₂O₃ nanoparticles led to changes in the morphology and structure of the nickel matrix leading to an improvement of its tribological properties. These properties were further enhanced when ultrasonic stirring was used during the composite coating electrodeposition.

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1. Introduction

Electrochemical composite deposition is a method of codepositing inert particles in an electrodeposited metal matrix. Different types of particles with a variety of properties, such as pure metals, ceramics and polymers, can be used. Combined with the variety of metals that can be electrodeposited, composite electroplating enables the production of a wide range of composite materials with improved properties in terms of wear resistance, lubrication or corrosion resistance [1]. One of the most applied composite coatings due to its high abrasion and heat resistance is the Ni/Al₂O₃ system, being its major field of application for engine cylinders, high-pressure valves and dies, car accessories and small aircraft and electrotechnical parts. Until now, most of the works were carried out using Al₂O₃ microparticles [2–8]. However, the recent emergence of nanotechnologies has led to scientific and technological interest on the electrodeposition of Ni/Al₂O₃ nanocomposite coatings with Al₂O₃ particles smaller than 100 nm [9–17], mainly devoted to increase the abrasion resistance of metal surfaces in microdevices. Despite rapid advances in nanocomposite coatings research, certain aspects concerning the agglomeration of particles in the electrolyte, the low content of nanoparticles in the composite coating and the non uniformity of particles distribution in the metal matrix remain to be solved. To cope with these problems, some attempts have been made, such as to use additives as a chemical dispersion agent for the particles

[18,19] or to use pulse plating in order to enhance codeposition of particles and improve homogeneity [14,20,21]. Another possibility is to apply ultrasonic energy. Thus, the deposition of metals under the influence of ultrasound has received significant attention, particularly in the applied literature, as ultrasound is thought to confer various benefits over conventional silent electrodeposition. Several researchers demonstrated that, under suitable conditions, ultrasound assisted electrodeposition leads to the improvement of the metal deposition rate, the quality of the metallic electrodeposits and the electrode surface cleanliness [22–28]. These results are attributed to the effects that ultrasonic energy can promote on the electrodeposition process [29]: (1) ultrasound greatly enhances mass transport, thereby altering the rate, and sometimes the mechanism of the electrochemical reactions; (2) ultrasound is known to affect surface morphology through cavitation jets at the electrode–electrolyte interface, which usually acts to increase the surface area and, (3) ultrasound reduces the diffusion layer thickness and therefore ion depletion. Ultrasound can also be used for dispersion and deagglomeration of particles into liquids. Thus, when sonicating liquids the sound waves that propagate into the liquid media result in alternating high- and low-pressure regions. As a result of this, bubbles are formed inside the liquid which grow and collapse with tremendous pressures provoking the vigorously agitation of the solution. Therefore, the use of ultrasound agitation during composite electrodeposition may promote the deagglomeration of particles, enhance the particle codeposition rate and improve the content and uniformity of particles in the metal matrix [30–34]. However, only a few studies have evaluated the effect of ultrasound agitation on Ni/Al₂O₃ composite coating electrodeposition [11,35,36] although this system is one of the most promising composite coatings that can find wide engineering application.

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In this study, attempts have been made to analyze the effect of mechanical and ultrasound agitation on the properties of Ni/Al₂O₃ nanocomposite coatings. The coatings were electrodeposited from suspensions of Al₂O₃ nanoparticles in nickel Watts baths without additives. Previously the Al₂O₃ particles suspended in the nickel Watts baths were analyzed using acoustics method in order to determine particle size. The coatings obtained were characterized from the compositional (EDX), morphological (OM, SEM) and structural (XRD, TEM) points of view. As the mechanical behavior of these systems is very important from the viewpoint of practical applications, some mechanical properties like hardness and wear were also evaluated.

2. Experimental

Ni/Al₂O₃ nanocomposite coatings were electrolytically deposited from a standard nickel Watts bath [37] to which Al₂O₃ nanopowder (Sigma-Aldrich) with an average particle size of 40–47 nm (according to the commercial supplier) was added. Nickel Watts bath was selected as it is one of the most popular electrolytes for functional applications. In order to facilitate characterization of the changes undergone when Al₂O₃ is present in the bath, the addition of additives was avoided. The pH of the plating solution was adjusted to 4. The detailed bath composition and experimental process parameters are shown in Table 1.

The size distribution of the particles in stirred nickel Watts baths was determined by acoustic spectrometry (DT1200, Dispersion Technology) at 45 °C (maximum operating temperature of the equipment). The acoustic spectrometer measures attenuation spectra and calculates the particle size assuming a certain model for describing the sound attenuation in terms of the physical properties of the system [38]. Traditional measurements of particle size usually involve light scattering or sedimentation techniques which require extreme dilution of the suspensions. This dilution step often changes the size distribution, thereby distorting the information being sought. Acoustic spectrometry allows the measurement of particle size in concentrated suspensions with high ionic strength, i.e. allowing the measurement of particle size under real working conditions.

Alumina was added to the nickel Watts electrolytes and the suspensions were intensively stirred by a magnetic stirrer (300 rpm) for at least 24 h at room temperature before the codeposition experiments. Immediately before the electrodeposition process, the suspensions were submitted to a 10-min ultrasonic pretreatment to avoid agglomeration. The ultrasonic irradiation was given by means of UP200S ultrasonic processor horn type (24 kHz, maximum nominal power 180 W, Hielscher Ultrasonics GmbH) equipped with a sonotrode S14 (14 mm tip diameter) adjusted at 55% vibration amplitude. The ultrasonic

processor was connected to an ultrasonic power meter for measuring ultrasonic power emitted. The ultrasonic power intensity applied was 38 W cm⁻² in continuous mode.

Cylindrical glass cells with volumes of 200 mL were used for the codeposition experiments. Coatings were deposited on flat commercial mild steel (grade St37) cathodes of 3 cm². The anode was a pure nickel foil (3 cm²) positioned on the side of the vessel, face to face to the cathode. The distance between anode and cathode was 4 cm. The substrates were degreased, rinsed in cold distilled water, activated in a 15% HCl solution (1 min) and rinsed in distilled water again. Electrocodeposition was carried out at a constant current density of 5.0 A dm⁻² for 30 min using a DC power supply (5 A/30 V, BLAUSONIC FA-350). In order to maintain a uniform particle concentration in the bulk solution, two agitation methods were used during the electrodeposition process: mechanical agitation by a magnetic stirrer ($w = 300$ rpm) located at the bottom of the cell, and ultrasonic energy by an ultrasonic probe (24 kHz, 38 W cm⁻²) which was directly immersed into the solution from above and accurately positioned between the working and counter electrodes in a way that there was no shielding. The sonotrode was immersed deep enough into the bath to avoid foaming. The distance between the ultrasonic horn and the working electrode was 2 cm and the distance between the ultrasonic tip and the bottom of the cell was 4 cm. This experimental configuration does not coincide with the conventional face-on and side-on configurations used for sonochemical studies as the purpose of using the ultrasonic energy during the electrodeposition process in this study was to maintain a good dispersion of the nanoparticles in the solution and avoid agglomeration, not to directly act on the chemical/electrochemical reactions that take place at the working electrode. The electrolyte temperature was maintained at 55 °C using a thermostat (Polyscience, 9106 model). Temperature was measured before and after each experiment. Temperature increase due to ultrasonic energy did not exceed 2–4 °C. The experimental set-up is depicted in Fig. 1. After electrolysis, the samples were ultrasonically cleaned in ethanol for 1 min to remove loosely adsorbed particles from the surface.

The morphology of the surface and cross-section of the coatings were examined by scanning electron microscopy (SEM). The weight percentage of Al₂O₃ incorporation was determined from cross-sections photomicrographs using an energy dispersive X-ray (EDX) microanalyzer coupled to the SEM. The amount of Al₂O₃ was examined at five different locations of each coating and the average weight percentage of these particles was calculated. The microstructure was characterized using TECNAI G² SuperTWINN FEG (200 kV) transmission electron microscope (TEM) equipped with High Angle Annular DARK Field (HAADF) detector. The thin foils for TEM observations were cut with Quanta 3D Focused Ion Beam (FIB) equipped with Omniprobe lift-out system. The phase determination of the deposits was investigated by means of X-ray diffraction (Panalytical X'Pert PRO MRD) using CuK_α radiation. The hardness of Ni and Ni/Al₂O₃ composite coatings was determined on polished cross-sections using a Fischerscope H100 microindenter with an applied load of 30 mN. Five readings were taken from each area of the deposit and the values were then averaged. Wear tests were performed using a ball-on-disk tribometer (CSM, model THT) at room temperature. A 100Cr6 steel ball with a diameter of 6 mm was used as a counterface material. The wear tests were conducted under a normal load of 1 N, with a rotation diameter of 3 mm at a sliding distance of 3500 m and a sliding speed of 17 cm s⁻¹. After testing, section profiles of the wear tracks on the coating were performed by a contact profilometer (Taylor Hobson, model TALYSURF INTRA 50 MM). From this information, maximum depth penetration and wear section area were determined. The volumetric wear factor of the different coatings, K , was calculated using the following equation:

$$K = 2\pi rA/Nd \quad (1)$$

Table 1
Bath composition and deposition conditions for Ni/Al₂O₃ composite coatings.

Bath composition	
Component	Concentration/g L ⁻¹
Nickel sulfate (NiSO ₄ ·6H ₂ O, Sigma-Aldrich ACS reagent 99%)	250
Nickel chloride (NiCl ₂ ·6H ₂ O, Sigma-Aldrich 99.9%)	45
Boric acid (H ₃ BO ₃ , Sigma-Aldrich ACS reagent 99.5%)	40
Alumina (γ-Al ₂ O ₃ , Sigma-Aldrich, ∅ = 40–45 nm)	25, 50, 100
Deposition conditions	
Parameters	Value
pH	4.0
Temperature, T/°C	55
Current density, j/A dm ⁻²	5.0
Electrodeposition time, t/min	30
Agitation system	Mechanical stirring (300 rpm) Ultrasonic stirring (24 kHz, 38 W cm ⁻²)

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