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Electrochemical preparation and characterization of Ni/SiC compositionally graded multilayered coatings

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ABSTRACT

In our study, Ni/SiC functionally graded coatings have been obtained by electrochemical deposition of silicon carbide microparticles (mean diameter 2 μm) from nickel Watts baths with different concentrations of SiC particles in solution. The SiC particles were characterized by electroacustics technique in order to determine zeta potential and particle size. Moreover, the effect of the concentration of SiC particles in solution on the amount of SiC deposited in the nickel layer was investigated. Further experiments showed that the degree of particle incorporation provoked changes in the texture of the nickel matrix. The characterization of the coatings proved that the Ni/SiC graded composite coatings were bright and compact, presented good adhesion and improved the hardness and wear resistance of pure nickel electrodeposits.

1. Introduction

Composite electroplating is a method of codepositing fine particles of metallic, non-metallic compounds or polymers in an electrodeposited metal matrix in order to improve material properties such as wear resistance, lubrication, or corrosion resistance [1–8]. In this sense, the codeposition of ceramic particles gives exceptional advantages in terms of mechanical properties (hardness, chemical inertia, good frictional behaviour) [9–12] as compared to alloy and pure metal electroplating.

One of the most widely applied composite coatings is the Ni/SiC system [13–16] being its major field of application in the automotive industry where it is used to reduce the wear on the inside of cylinders. The properties of this system depend on several variables, in particular on the content and nature of the SiC particles dispersed in the metal matrix. Many interrelated experimental parameters influence the codeposition process of the Ni/SiC system [4,17,18] such as electrolyte formulation, temperature, current density and electrode/electrolyte movement. However, a clear picture of the exact effect of each parameter is difficult to obtain, because often different, or even contradicting, results are reported by different authors [4,12,19–23].

On the other hand, functionally gradient material (FGM) is a novel engineering system developed in the mid-1980s. Its character is that, some characteristics like composition and structure gradually change over the volume, resulting in corresponding changes in the properties of the material [23]. Moreover, it is known that functionally graded materials reduce delamination and spallation because the transition between different types of materials is not abrupt. In the simplest FGMs, two different material ingredients change gradually from one to the other. The material ingredients can also change in a discontinuous way in a stepwise gradation. FGMs have been produced by several methods such as chemical or physical vapor deposition, thermal spray or powder metallurgy [24]. FGMs can also be obtained by electrochemical techniques. Thus, the variation of process parameters as current density, stirring rate, and particle loading in the bath causes changes in the electrodeposition process that can be used to process graded coatings [4]. Some examples include a nickel deposit with SiC microparticles [23,25–27], ZrO_2 nanoparticles [28] or Al and Al_2O_3 nanoparticles [29–30]. The major advantages of this method over the other techniques are the simplicity to control, possibility to process complicated parts and the low initial capital investment.

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In this study Ni/SiC multilayered composite coatings with a graded content of SiC particles were prepared by electrodeposition in order to obtain high-performance compositionally graded materials for specific applications. Prior to the formation of Ni/SiC FGMs, the effect of the particle concentration in the electrolyte on the amount of SiC embedded in the nickel layer and on the structure of the nickel electrodeposit were investigated. The graded multilayered coatings were electrodeposited from suspensions of SiC microparticles (d_m = 2 μm) in nickel Watts baths without additives. Previously the SiC particles suspended in the nickel Watts baths were analysed using electroacustics method in order to determine zeta potential and particle size. The graded multilayered coatings obtained were characterized from the compositional (EDX) and morphological (OM, SEM) points of view. As the mechanical behaviour of these systems is very important from the viewpoint of practical applications, some mechanical properties like adhesion, hardness, wear and friction were also evaluated.

2. Experimental

Ni/SiC composite coatings were electrolytically deposited from a nickel Watts bath without additives to which SiC was added (Table 1). α-SiC powder (99.8%, Alfa Aesar) with an average particle size of 2 μm was used for the synthesis of the composite coatings. Silicon carbide particles were used as received.

The zeta potential and the size distribution of the particles in stirred nickel Watts baths were determined by electroacustics technique (DT1200, Dispersion Technology) [31]. This technique is much more sensitive than classical electrophoretical method [32], allowing the measurement of zeta potential (as well as particle size) in concentrated suspensions with high ionic strength [33], i.e. allowing the measurement of zeta potential and particle size under real working conditions.

Open vessels with volumes of 200 mL were used for the codeposition experiments. The pH of the plating solution was adjusted to 4, and the temperature was maintained at 55 °C by an automatic controller. Coatings were deposited on brass cathodes of 3 cm^2. The anode was a pure nickel foil positioned on the side of the vessel. Silicon carbide was added to the nickel Watts electrolytes and the suspensions were intensively stirred for at least 24 h at room temperature before the codeposition experiments. 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}. Magnetic stirring was employed at the cell bottom of the cell in order to maintain a uniform particle concentration in the bulk solution (ω = 300 rpm). After electrolysis, the samples were ultrasonically cleaned in ethanol for 1 min to remove loosely adsorbed particles from the surface.

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

Component	Concentration (g L^{-1})
Bath composition	
$NiSO_4{\cdot}6H_2O$	250
$NiCl_2{\cdot}6H_2O$	45
H_3BO_3	40
SiC (d_m = 2 μm)	5, 15, 25, 50
Parameters	**Value**
Deposition conditions	
pH	4.0
Temperature, T (°C)	55
Current density, j (A dm^{-2})	5.0
Electrodeposition time, t (min)	30
Stirring speed (rpm)	300 (magnetic)

The morphology of the surface and cross-section of the coatings were examined by optical (OM) and scanning electron microscopy (SEM). The weight percentage of SiC incorporation was determined using an energy dispersive X-ray (EDX) microanalyser coupled to the SEM. The amount of SiC was examined at five different locations of each coating and the average weight percentage of these particles was calculated. The microstructure of the deposits was investigated by means of X-ray diffraction (Panalytical X'Pert PRO MRD) using Cu Kα radiation.

Roughness of the coatings was determined by profilometer analysis (Form Talisurf 120, Taylor Hobson) on 16 mm^2 selected area. Friction and sliding wear tests were conducted under ASTM G99-95a using a pin-on-disc tribometer constructed at MERL. All samples were run against a 5-mm diameter 440C stainless steel (Grade 100, 60 Rc) ball indenter at 10 N normal load at the speed of 0.3 m s^{-1}. The sliding distance was 250 m. The tests were conducted under dry contact conditions at room temperature. Mass material loss was determined by weighting both the pin and the sample before and after wear testing using an analytical balance. In order to compare wear tests, the wear was expressed as a mass wear factor (kg N^{-1} m^{-1}). This wear factor was calculated by dividing the mass material change by the total sliding distance and the applied load. The hardness of multilayered Ni/SiC coating was determined with a Fischerscope H100 microindentator with an applied load of 10 mN. Five readings were taken from each area of the deposit and the values were then averaged.

3. Results and discussion

3.1. Particles characterization

Electroacustics measurements were used in order to determine the size distribution of the SiC particles suspended in the nickel Watts bath. As can be seen in Fig. 1, the maximum of the peak is located at a particle diameter of 0.8 μm but the average particle size calculated from the data of the curve shown in the diagram is 1.8 ± 0.6 μm (d_{84} = 2.8 μm). This value is comparable to the 2 μm given by the supplier as the average particle size of the SiC powder, considering that different methods of measurement were used in

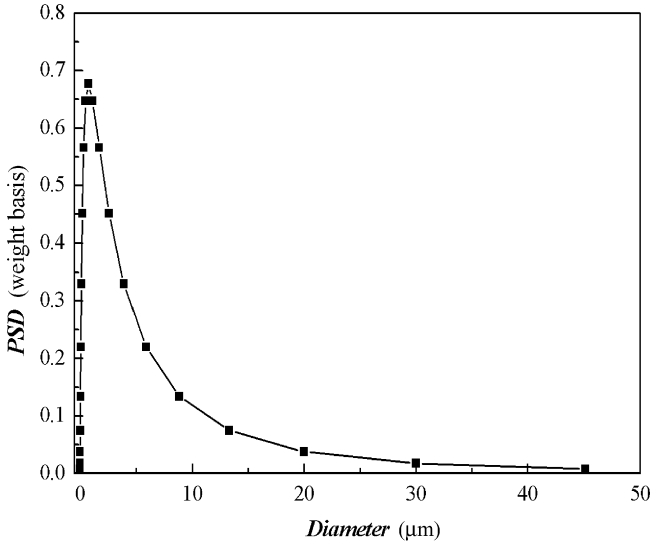

Fig. 1. Particle size distribution (PSD) of SiC particles suspended in a nickel Watts bath (50 g L^{-1} SiC). The experiment was carried out at 45 °C.