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Production and microstructural characterization of Ni matrix composite electrodeposits containing either micro- or nano-particles of Al

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ABSTRACT

Composite coatings electroplating could be a valid and relatively low cost production method to codeposite either micro- or nano-particles of Al in Ni matrix. The aim of this work is the production of Ni matrix composite deposits containing either micro- (3–4 μm) or nano- (130 nm) particles of Al using parallel plate geometry composite plating and their characterization both prior and after heat treatments at different temperatures by means of microstructure and microhardness. The obtained electrodeposits both before and after heat treatments at different temperatures have been observed by SEM in cross section both prior and after metallographic etching and analysed by XRD in order to evaluate the formation of Ni/Al phases. After heat treatment at 600 °C the micro-composite coating consisted of a biphasic γNi and γ′Ni_3Al system, while after heat treatment at 800 °C of a solid substitutional solution of Al in the γNi. The diffusion of Al in the metal matrix hindered the recrystallization of the Ni matrix and the change of the preferential orientation. The more uniform distribution of the Al nano-particles and the smaller dimensions lead to an advance of the diffusion at lower temperature (400 °C). The heat-treated nano-composite deposits consist of γNi and present a columnar structure with narrow ad short columns as the fast diffusion of Al blocked the recrystallization of the Ni matrix.

1. Introduction

Electrolytic codeposition is widely used to produce composite metal matrix coatings, as it is a relatively low cost and low temperature technique and does not require expensive additional equipment for the industrialization. Several metals have been used as matrix; nickel is undoubtedly the most used due to is hardness, corrosion and wear resistance. These properties have been further improved by the addition of different amount and sizes of ceramic particles such as SiC, Al_2O_3, WC, TiO_2, CeO_2 etc. [1–7]. The intrinsic properties of the embedded particles and the microstructural modification of the metal matrix caused by the electrodeposition allow the production of unique deposits with improved mechanical, physicochemical and electrochemical properties for targeted applications. Some works report that the presence of ceramic particles such as SiC [8] or La_2O_3 [9] improves also the high temperature oxidation resistance of pure Ni electrodeposits by limiting the thickness of the NiO scale. The NiO scale, however, cannot be considered fully protective for the underneath composite nickel matrix coatings. Hence, a different approach has been taken into consideration in the last years and considers the codeposition of metallic particles such as Cr, Al [10–20] or Ti [18] which improves the oxidation resistance of the Ni coatings due to the tendency to form a continuous Cr_2O_3, Al_2O_3 or TiO_2 layer on the coatings surface at high temperatures. The choice of adding Al or Ti in the form of particles is due to the fact that the deposition of these elements is not possible from aqueous electrolytes.

It has been reported that Ni matrix deposits containing Al micro-particles, obtained by sediment codeposition, a technique allowing the codeposition of high particles amounts, lead to the increase of the oxidation resistance by decreasing the thickness of the NiO scales due to the formation of a continuous and protective Al_2O_3 film on the coatings surface [10]. In order to form a continuous Al_2O_3 layer a critical amount of Al in the coating is necessary. The critical amount of codeposited Al is decreased noticeably by the codeposition of Al nano-particles instead of Al micro-particles [11–12]. Moreover, an annealing treatment in vacuum at 600 °C for 4 h can further increase the oxidation resistance of the Ni-Al nanocomposite coatings due to the formation of γ′Ni_3Al and γNi containing Al phases [13]. On the other hand, the pores which are formed due to fast diffusion of Al in the Ni matrix can negatively affect the oxidation resistance of the composites [14]. In this case, a high amount of Al (7–11%) decreases the oxidation resistance in the initial stage if compared to deposition with an Al amount less than 7% due to the formation of large pores. Anyway, more detailed studies on the oxidation kinetics on Ni-Al micro-composites produced using a parallel plates geometry and high amounts of particles both in the plating bath and in the produced deposits confirmed the beneficial effect of the Al particles on the oxidation resistance at both 800 and 1000 °C [15–17]. The same studies confirmed the formation of different intermetallic phases between Ni and Al after

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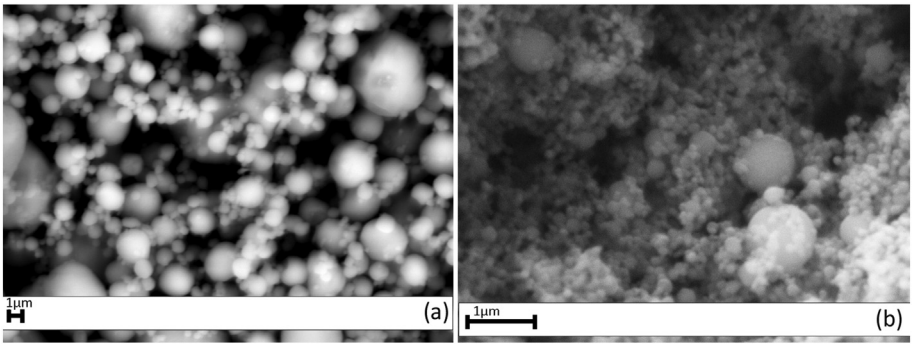

Fig. 1. SEM micrographs of the Al micro- (a) and nano- (b) particles.

heat treatments at different temperatures based on the amount of Al and the heat treatments temperature.

The codeposition of Al or Ti particles does not affect only the oxidation resistance of the produced deposits but also the microstructure of the Ni matrix by influencing the nucleation and growth processes. Parameters such as the plating bath chemical composition, the stirring rate, the particles concentration in the plating bath, the size and type of metallic particles, the deposition current density influence the amount of codeposited particles, their distribution in the obtained deposit and the microstructure of the matrix, as occurs in every metal matrix composite system. As mentioned before, sediment codeposition allows the incorporation of higher amounts of particles even of large dimensions. According to Naploszek et al., Ti particles can be deposited in higher amounts than Al particles using the same particles loading in the plating bath and the same current densities. The optimum conditions are different for the two systems, indicating than the type of particles strongly influences the deposition [18]. Other researchers [19] compared the deposition of micro- and nano-particles of Al using the same particles load in the plating bath and the same current density and observed that the use of nanoparticles leads to a more uniform distribution in the metal matrix, in comparison to the use of micro-particles, and to a noticeable grain refinement of the Ni matrix microstructure [19]. The amount of codeposited particles decreases by increasing the current density, keeping stable all other plating parameters, and the preferential orientation of the obtained deposits changes from (200) to a non-oriented one. This influences also the residual stresses of the composite deposits which thus decrease by increasing the current density.

The presence of Al particles does not only influence the oxidation resistance of the obtained deposits but could also influence the mechanical properties of the bulk deposit at high temperatures due to the formation of the intermetallic Ni-Al phases.

The aim of this work is the production and characterization of relatively thick Ni-Al deposits containing either micro- or nano-particles. To this aim the composite deposits have been produced using plating baths with low Al load and parallel plates geometry in order to make easier the industrialization of the process in the future. To this aim a high speed plating bath has been used and the plating parameters have been kept stable and close to the values used for the pure nickel deposition. The effect of the codeposition of micro- or nano-particles on the structure and hardness of the obtained deposits after heat treatments at different temperatures has been also evaluated.

2. Experimental

2.1. Specimens production

Three types of coatings have been produced: specimens coated with pure nickel (Pure Ni), used as reference, and specimens coated with Ni containing either micro- (Ni/μAl) or nano-particles (Ni/nAl) of Al.

The electroplating bath used was a High Speed nickel sulfamate plating bath containing: 500 g/L Ni $(SO_3NH_2)_2 \cdot 4H_2O$, 20 g/L $NiCl_2 \cdot 6H_2O$, 25 g/L H_3BO_3, 1 mL/L surfactant $(CH_3(CH)_{11}OSO_3Na$ based industrial product). The pH of the plating bath was 4.30.

The depositions were carried out using a plate parallel cell geometry with an electrolyte volume of 1,5 L at 45 °C, under galvanostatic control at 4 A/dm^2, and continuous stirring (500 rpm). The deposition time was 2 h in order to obtain an about 80 μm thick deposit.

For the production of the Ni/μAl composite coatings an Al micrometric powder with a mean size of 3–4,5 μm (mkNANO MKN-Al-4500 Lot #08/459, purity 97,5%) was used. For the production of the Ni/nAl composite coatings an Al nano-metric powder with a mean size of 130 nm (Skyspring nanomaterial 130 nm, purity 99,7%) was used.

Both Al powders were analysed by SEM in order to control shape and size. Both particles presented a spherical shape, the micro-particles dimensions correspond to the declared size (Fig. 1a), while some micrometric particles with a mean diameter of 1 μm were observed in the Al nano powder (Fig. 1b).

In order to produce the composite coatings, 40 g/L of either micro or nano-particles were added into the electroplating bath and dispersed by

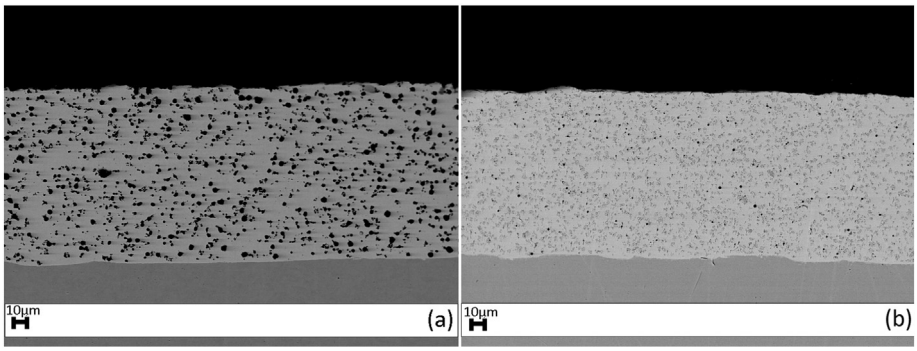

Fig. 2. SEM micrographs at cross section of the as plated micro-composite (a) and nano-composite (b) coatings.