Gradient Nickel – Alumina Composite Coatings

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Abstract

Particle reinforced metal matrix composite (MMC) coatings have been developed due to property combinations such as increased hardness, high creep/fatigue resistance as well as superior wear and oxidation resistance. MMC coatings can be produced by various techniques, which include powder metallurgy (plasma spray and HVOF processes), liquid metal processes and electrodeposition. This paper presents the development of the electrodeposition process to obtain nickel-alumina composites. The process conditions were optimized, and based on this, gradient nickel-alumina composites were prepared. Microstructural characterization and hardness determinations were carried out. The effect of process parameters on the amount and distribution of alumina particles as well as morphology of the deposits with and without alumina gradients are presented and discussed.

Keywords: Metal matrix composites, electrodeposited composites, functionally gradient materials.

Introduction

Particle reinforced metal matrix composite coatings were developed due to a combination of properties such as high hardness, good creep and fatigue resistance, increased resistance to abrasion and strength at high temperatures. Metal matrix composite (MMCs) coatings can be produced by a variety of techniques which include powder metallurgy (plasma spray and HVOF processes), liquid metal processing and by electrodeposition. Each of these techniques has its advantages and limitations, depending on the matrix and the reinforcement, and processing drawbacks such as restrictions on size or shape of the component being coated. Electrodeposition or co-deposition of non-metallic or ceramic particles in a metallic matrix is a fabrication technique that combines the ability to deposit on both complex shapes and large components with the advantages of low cost of equipment and raw materials, simplicity of operation, possibility to incorporate significant percentages of reinforcement of varying forms and composition, and production at ambient temperature and pressure[1]. In addition, changing the volume percent of inert particle in the electrodeposited coating, critical to producing graded materials, can easily be accomplished through alteration of processing parameters[2-8]. This was observed by varying the current density and particle loading in the bath[2].

In co-deposition, the particles to be co-deposited are maintained in suspension in the electrolytic bath by agitation while the metal from the bath is being deposited. The particles can be sulfides, carbides, oxides, graphite etc., which confer properties such as abrasion resistance, fatigue resistance etc.

Electrodeposited composite coatings can be produced from acid electrolytes where in the reinforcement acquires a positive charge by adsorption of metallic ions. These adsorbed metallic ions cause the reinforcement (particles) to be attracted towards the cathode (due to electrostatic forces) and help form efficient mechanical links between the particles and the cathode surface. The particles attracted to and touching the cathode surface are constantly

covered by the growing metallic deposit[1,9]. A number of parameters control the properties of the electrodeposited composite coatings and include besides reinforcement shape, size and content, also, bath composition, current density, pH, bath temperature, additives to the bath and efficiency of agitation[10].

Nickel-alumina composites coatings have exhibited hardness in the range 300-500 VHN[11,12]. These papers also showed that there was no correlation between hardness of the ceramic particles and that of the respective composites. On the other hand, the type of bonding between the ceramic phase and the metal determine the hardness and other mechanical properties of the composites[11].

This paper presents: (a) the effect of alumina particle additions to the nickel bath and processing parameters such as bath temperature, current density, and duration of deposition on nickel-alumina composite deposit characteristics and (b) optimization of some of the parameters to obtain gradient nickel-alumina composites coatings.

Materials and Methods

A Watts bath with the following composition was used to obtain the nickel-alumina deposits.

$NiSO_4$	240 g/L
$NiCl_2$	30 g/L
H_3BO_3	40 g/L

Electrolytic nickel anodes were used and the bath conditions were as shown in Table I.

The grain size distribution of the alumina particles added to the bath was such as to have > 90% with < 2μm (Figure 1). The morphology of the alumina particles is ahown in Figures 2 and 3. AISI 304 stainless steel cathodes were used and these were ground to 400 mesh, degreased ultrasonically in trichloroethylene and activated at 2.7 A/dm^2 for 15 minutes in a bath of: $NiCl_2.6H_2O$ (240 g/L), NaF (10 g/L) and HCl (60 g/L).

Table I. Electrolytic bath conditions.

Parameter	Quantity or Condition
Temperature	55-60°C
pH	4
Current density	2-4 A/dm^2
Duration of deposition	20-60 min
Alumina concentration in bath	25-200 g/L
Bath agitation	Mechanical

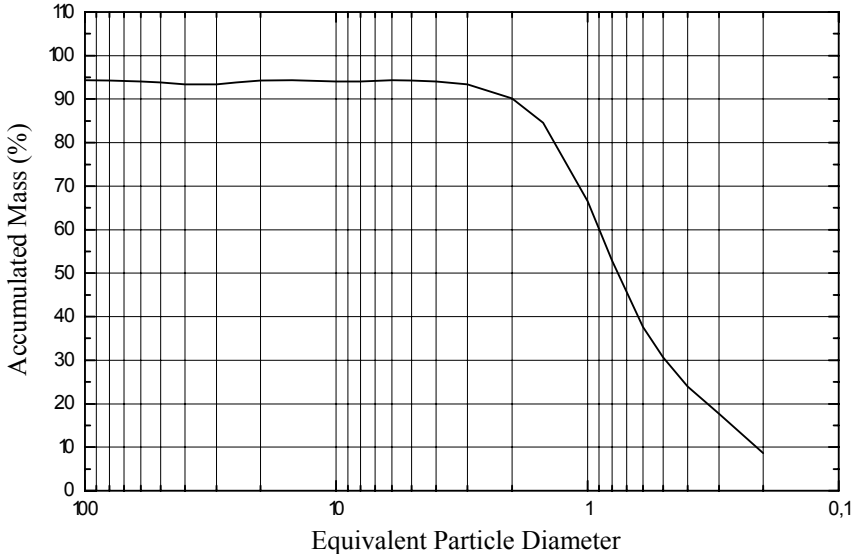

Figure 1: Alumina particle size distribution.

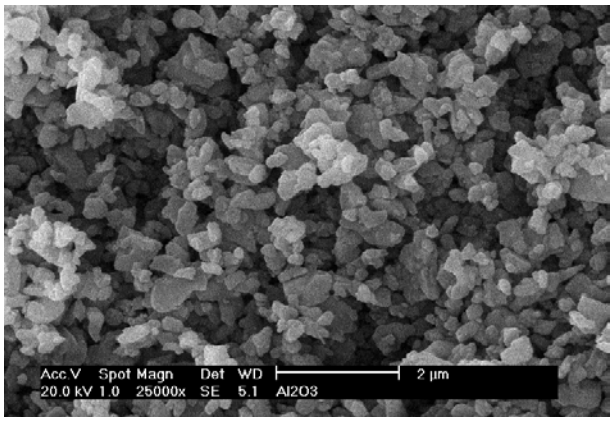

Figure 2. Scanning electron micrograph of the alumina particles used to produce nickel-alumina composites. 25.000x.

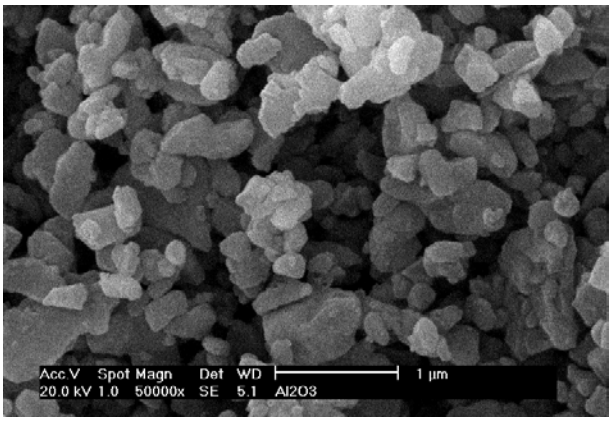

Figure 3. Scanning electron micrograph of the alumina particles. 50.000x.

Transverse sections of the deposits were examined in a scanning electron microscope, to evaluate the thickness, particle distribution and adherence of the deposit to the substrate. The microhardness of the deposit was also measured. The volumetric alumina particle content of the deposit was determined gravimetrically. The electrodeposit was initially dissolved in a warm 50% HNO_3 solution, the solution filtered and the weight of alumina determined by difference between the initial and final weight of the filter paper.

Gradient deposits were obtained: (a) by transferring the cathode from bath to bath with increasing alumina content and (b) by increasing the alumina content of a single bath at pre-determined intervals. The sections of the specimens thus obtained were also examined in the scanning electron microscope for particle distribution.

Results and Discussion

In order to determine the ideal conditions for electrodeposition of nickel-alumina composites, preliminary studies were carried out to determine the effects of current density and duration of deposition on deposit characteristics. The other experimental conditions were maintained constant, including bath agitation and alumina concentration. The results of this preliminary investigation are shown in Table II. The deposit thickness was measured with an Elcometer and the hardness, in a Vickers microhardness measuring apparatus with a load of 50g. It can

be seen from Table II that the hardness variation did not show any correspondence with the alumina content.

Table II. Deposit thickness, microhardness and alumina content of the nickel-alumina composites obtained from baths containing 25 vol% of alumina.

Specimen	Duration of deposition (min)	Current density (A/dm^2)	Deposit thickness (μm)	Micro-hardness $(HK)_{50}$	Alumina content (wt%)	Alumina content (vol%)
25230	30	2	42.6	306	4.66	9.86
25240	40	2	45.9	319	3.66	7.78
25250	50	2	51.0	244	-	-
25260	60	2	52.9	268	-	-
25320	20	3	35.9	298	4.99	10.53
25330	30	3	41.8	311	4.57	9.69
25340	40	3	50.2	334	4.03	8.60
25350	50	3	59.0	305	3.38	7.27
25420	20	4	54.8	-	-	-
25430	30	4	58.3	-	-	-
25440	40	4	59.3	-	-	-

The volumetric alumina content decreased with duration of deposition although the thickness of the deposit increased. This indicated that for deposition times longer than 30 minutes, particle incorporation decreased in proportion to nickel deposition. Pushpavanan reported that the alumina content increased steadily with time up to 30 minutes and thereafter remained unchanged[9]. It was also reported that the deposit thickness varied from 10-17 μm although the deposition times were much longer than those used in this investigation. It can also be observed that at current densities of 2 and 3 A/dm^2, the volumetric alumina content was the same for 30 and 40 minutes of deposition.

Based on these results, the current density was fixed at 2 A/dm^2 and the duration of deposition, at 30 minutes. Deposits were prepared from baths containing varying concentration of alumina. Data obtained from this set of experiments are shown in Table III. Figure 4 summarizes the variation in deposit thickness and alumina content of the deposit as a

function of alumina content in the bath. The thickness of the deposit was constant even though the alumina content of the baths varied. The volumetric alumina content of the deposits were higher than those reported earlier for deposits obtained under similar conditions[9,10].

Table III. Deposit thickness, microhardness and alumina content of the nickel-alumina composites obtained at 2 Adm^{-2} for 30 minutes from baths with different alumina content.

Specimen	Alumina concentration in bath (g/L)	Deposit thickness (μm)	Micro-hardness $(HK)_{50}$	Alumina content on deposit (wt%)	Alumina content on deposit (vol%)
25231	25	12.7	262	4.33	8.57
50230	50	10.4	232	6.80	12.87
75230	75	10.1	271	14.06	25.26
100230	100	9.6	227	19.04	32.66
150230	150	11.2	259	18.95	31.60
200230	200	11.8	305	17.39	26.31

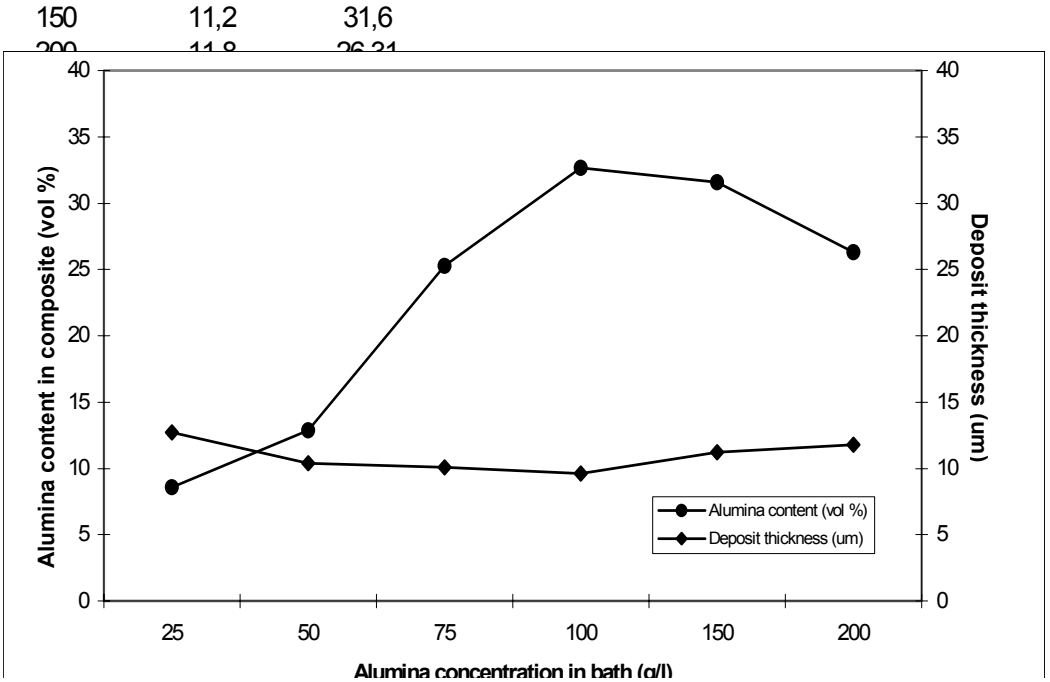

Figure 4. Effect of alumina content in electrolytic bath on deposit thickness and alumina content in the nickel-alumina composite.

A number of mechanisms have been proposed to explain the co-deposition of ceramic particles in a metallic matrix. One of these proposes that the co-deposition of alumina depends on effective collision of particles on the cathode surface. Based on this, an increase in alumina content in the deposit would be expected with increase in alumina content of the bath. Data shown in figure 4 indicates that this tendency exists to some extent and that the alumina content in the deposit reaches a maximum at 100 g/L of alumina in the bath. Further increase in bath alumina content did not increase deposit alumina content and also resulted in ineffective agitation of the bath. The scanning electron micrograph of a section of a deposit obtained from a bath with 25 g/L of alumina is shown in Figure 5 and reveals uniform particle distribution.

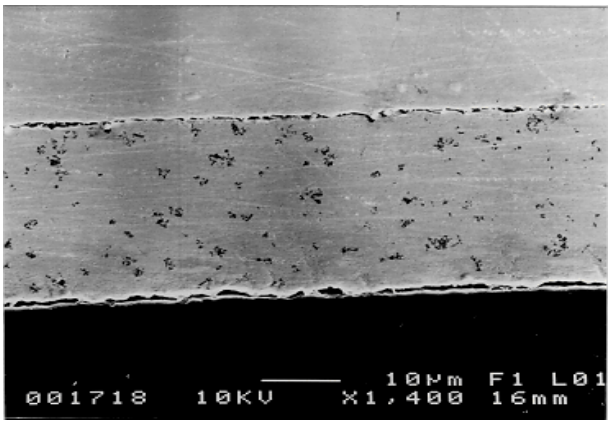

Figure 5. Scanning electron micrograph of a section of specimen 25230, obtained from a bath containing 25 vol% alumina at 2 A/dm^2 for 30 minutes.

Table IV. Experimental parameters used to obtain gradient nickel-alumina composites

Alumina concentration in the bath (g/L)	Deposition time(min)		
	specimen 50-3	specimen 75-4	specimen 100-5
50	5	-	-
75	5	5	-
100	5	5	10
150	5	10	10
200	10	10	10
Total deposition time	30	30	30

Composites with an alumina concentration gradient were obtained by electroplating specimens under conditions shown in Table IV. Figures 6-8 show the distribution of alumina in the deposits obtained under the conditions shown in Table IV. In Figure 6, a gradient in alumina concentration is evident. Up to mid-thickness of the deposit, the alumina content increases with alumina content of the bath. Beyond mid- thickness, the alumina content even though high, did not vary across the deposit section. This reconfirmed the observations made earlier from Figure 4 where at bath concentrations > 100g/L, the alumina concentration in the deposit did not change significantly. This behavior was also observed in Figure 8, where specimen 100-5 was obtained from baths with 100, 150 and 200 g/L of alumina.

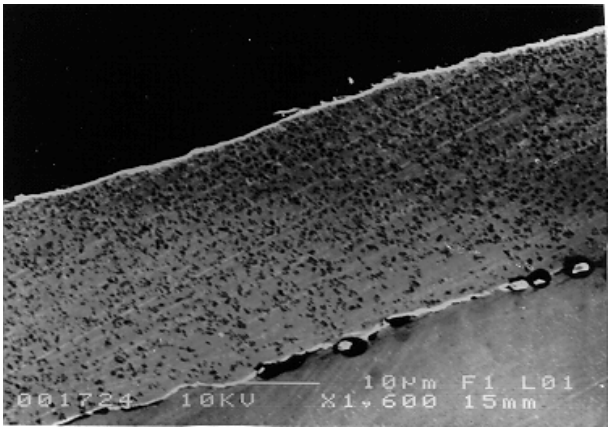

Figure 6. Scanning electron micrograph of a section of specimen 50-3, obtained under conditions shown in Table IV.

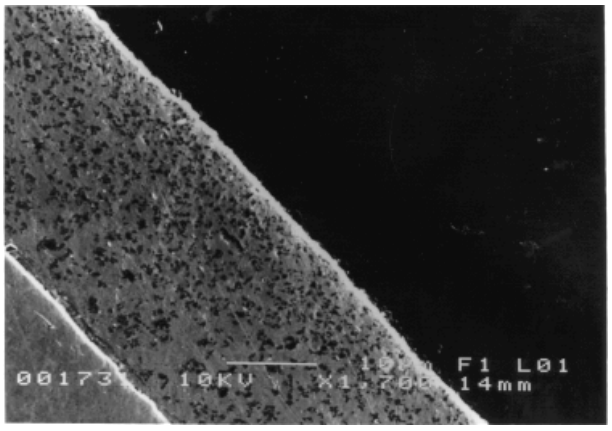

Figure 7. Scanning electron micrograph of a section of specimen 100-5, obtained under conditions shown in Table IV.

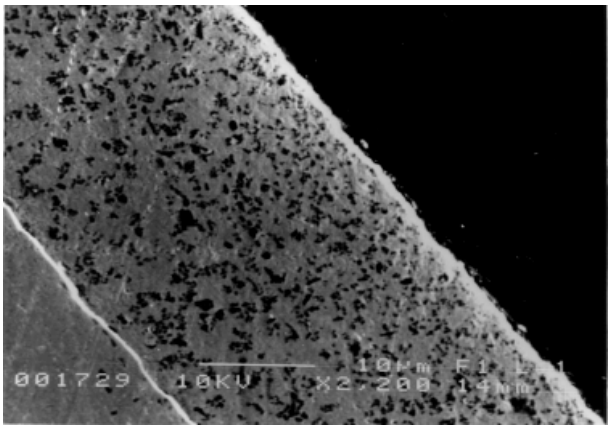

Figure 8. Scanning electron micrograph of a section of specimen 100-5, obtained under conditions shown in Table IV.

Conclusions

1. The co-deposition of alumina particles from an agitated Watts type nickel-plating bath containing suspended alumina particles has permitted nickel-alumina electrodeposited composites to be obtained.
2. The bath parameters were optimized to obtain uniform and thick composite deposits with homogeneous alumina particle distribution.
3. The effect of alumina content in the electrolyte on alumina content in the deposit as well as deposit thickness was determined.
4. Nickel-alumina deposits with an alumina concentration gradient were obtained by electrodeposition from a series of baths with increasing alumina concentration from 0 to 100 g/L.

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