Preparation and Characterization of Ni-coated Aluminum Powder by Electroless Plating

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ABSTRACT: *This paper deals with the preparation and characterization of Nickel-coated Aluminum ceramic particles by Electroless Plating Technique. Aluminum powder is used for plating. The bath for Electroless Nickel Plating is prepared with some chemicals like Nickel Chloride hexahydrate, Sodium hypophosphate Monohydrate, Ammonium Chloride, Tri Sodium Citrate, Sodium Hydroxide. A bath temperature of 85°C, pH value of 8 and stirring time of 20 min is considered to carry out the deposition. And we checked for the colour change, hardness and density values have given the primary indications and the results of SEM (scanning electron microscopy) is whether convincing or not. The results shown a great change in colour change i.e, deposition of nickel is rich that indicates the coating is done on the Aluminum powder, also there is a great change in density.*

1. INTRODUCTION

Aluminum ceramics are widely used as reinforcement in many applications of modern metal matrix composite materials. However, engineering applications of a ceramic material are determined by its brittleness. The composite preparation also suffers from low wettability because of liquid metal and ceramic interactions. As the bonding between metal and ceramic plays a prominent role in deciding the characteristic of the composite, the research is focused at the methods of improving wettability. One such method is modifying the particulate-reinforcement surface using a metal coating as layer. Nickel, known for its excellent corrosion and wear resistance, is a good choice for a coating material. A few methods like co-precipitation method, sol-gel method are employed to prepare the coated powder . On the other hand, the electroless plating method is another option with an advantage in high deposition rate, simple operation and capability of mass production. Another advantage of using electroless nickel is its ability to produce the deposits with a very high degree of uniformity of thickness. The Ni deposit has a good wettability and is generally hard. The Electroless plating proposed by Brenner and Riddell has been widely used during the past two decades in many applications.

2. RAW MATERIALS

99% pure Aluminium powders of average particle size 50 nm, supplied by Aahila Plastic Processing Industry,Hyderabad are used in the present experiment. The primary characteristics of the powder are shown in Table 1.The chemicals which are required for Pre-treatment Process and Composition and Conditions of the Bath are supplied from Growwell Scientific and surgicals, Laboratory equipment supplier in Rajahmundry, India.These are clearly shown in Table 2 and 3.

Particle Size	50 nm (Avg. Size)
Purity	99 %
Density	3.9 g/cm3
Crystal Structure	FCC
Appearance	Light Grey Solid

Table:1 Characteristics of the Al_2O_3 Powders used in the Experiment

3. MECHANISM OF PLATING

The basic principle of Electroless plating is similar to Electro Plating but without many Electrical Contacts and apparatus. Charge transfer takes place through a chemical reduction process. The Electroless plating depends upon the catalytic reduction of a metallic ion in an aqueous solution containing a reducing agent and the subsequent deposition of the metal without the use of electrical energy. In this experiment, Nickel Chloride Hexahydrate (NiCl2.6H2O) is used as a source of Nickel ions while Sodium hypophosphite (NaH2PO2.H2O) is the reducing agent. The pre-treatment of the powder (Sensitization and Activation) makes the conditions favorable for plating. The nickel deposition with SnCl2 sensitization and PdCl2 activation is employed in the present work . In the sensitization process, Sn2+ ions are absorbed on the surface of the Aluminium powders, which could facilitate attracting of Pd2+ ions onto the surface of particles (Eq. 1). When the activated Aluminium powders are added to the bath, the metallic nickel could be deposited with the following reactions (Eq. 2 & Eq. 3) taking place.

$$Sn^{2+} + Pd^{2+} \rightarrow Sn^{4+} + Pd^{0} \qquad (1)$$

$$Ni^{2+} + 2H_2PO^{2-} + 2H_20 \rightarrow Ni^{0} + 2H_2PO^{3-} + 2H^{+} H_2 \qquad (2)$$

$$Pd + Ni^{2+} \rightarrow Pd^{2+} + Ni \qquad (3)$$

The Ni^{2+} ions will be reduced to metallic nickel and the metallic Pd^{0} oxidized to Pd^{2+}. That is, both oxidation and reduction reactions happen in the process.

4. EXPERIMENTAL PROCEDURE

The plating process involves two steps, Viz. Pretreatment (Surface preparation) and Bathing. Initially, the powder was treated with acetone using Ultrasonic cleaner and later on with different chemicals to complete the pretreatment process. Xu *et al.*, reported Ultrasonic agitation promotes better nucleation and deposition of nickel than mechanical agitation in case of nickel-coated graphite [10]. The detailed description of the chemicals and their purposes used for experimentation are mentioned in the Table 2. The powders are rinsed with distilled water and are dried in each step.

The pretreated powder is sent to the electroless nickel deposition bath. The alkaline solution bath was adapted for the advantage of having low phosphorous content [7]. The bath elements used here are less in quantity and have an economical advantage than those proposed by pang *et al.*, [4]. The deposition bath for plating was prepared using the formulations given in the literature [8] and is indicated in Table 3

In this experiment, in addition to adopting the $SnCl_2$ sensitization and $PdCl_2$ activation process in Ref. [7], rinsing of the sensitized Aluminium powders was intentionally applied for the Ni electroless deposition. Firstly, the Aluminium powders were immersed in acetone solution with ultrasonic agitation for 20 min to clean the surface. Secondly, the powders were sensitized in an aqueous solution of 5 g/L $SnCl_26H_2O$ and 15 ml/L HCl for 10 min. Finally, the Aluminium powders were activated in an aqueous solution of 0.125 g/L $PdCl_2$ and 1.5 ml/L HCl for 10 min. The powders were rinsed with distilled water in each step and dried in oven after the pretreatment processes. In order to investigate the effect of $Sn^{2þ}$ absorption behaviors on electroless nickel plating, different $SnCl_2$ sensitization conditions were controlled to prepare powders.In the elecroless deposition process, a NaOH solution (30 wt%) was used to adjust the pH to a value of about 8. The powders were electroless deposited for 20 min at a temperature of 85^0C, followed by thoroughly rinsing and drying for characterization.

Chemicals and Conditions	**Concentration**
Nickel chloride, hexahydrate ($NiCl_2.6H_2O$)	15 g/L
Sodium hypophosphite Mono Hydrate ($NaH_2PO_2.H_2O$)	12.5 g/L
Ammonium Chloride (NH_4Cl)	25 g/L
Tri Sodium Citrate ($Na_3C_6H_5O_7$. $2H_2O$)	20 g/L
Sodium Hydroxide (NaOH)	To maintain the required pH
Bath Temperature	85 ± 2^0C
pH	8
Time	20 Min

Table:2 Pretreatment Process

Process	**Chemicals**	**Concentration**	**Time**	**Purpose**
Ultra Sonic Cleaning	Acetone		20 Min	Removes Volatile compounds from the surface and makes the particle surface clean
Etching	HNO_3	5ml/L	15 Min	Microscopic Roughening
Rinsing	Distilled Water		5 Min	
Sensitization	$SnCl_2$. $6H_2o$ + HCl	5 g/L 15 ml/L	15 Min	Breaks the surface integrity and makes ready for activation
Rinsing	Distilled Water		5 Min	
Activation	$PdCl_2$ + HCl	0.125g/L 1.5ml/L	15 Min	Activate the pores and prepare the surface for plating
Rinsing	Distilled Water		5 Min	

Table:3 Composition and Conditions of the Bath

Fig:1 Aluminium powder mixed with 5ml Nitric acid and half litre distilled water

Fig:2 Vaccum filter which is to be used for the for filtering of powder from water

Fig:3 Heating upto 85^0C in water bath and checking temperature by thermometer

Fig:4 Ni-coated Aluminium powder

5. CHARACTERIZATION OF POWDERS

The surface morphology of the Aluminium powders before and after electroless Ni deposition was characterized by scanning electron microscope (SEM, JSM-6460LV) equipped with an energy dispersive spectroscopy (EDS) at Andhra Univeristy ,Visakhapatnam,India.

6. RESULTS AND DISCUSSIONS

From the literature, the reduction of nickel is always accompanied by the evolution of hydrogen gas [7]. The reaction of hypophosphite (Eq. 2) is the main reason. When the Samples B and C were added to the bath for plating at the conditions specified, the hydrogen gas was evolved in Samples C (Figure 15b) and liberation was very poor in the Sample B (Figure 15a). The color change may justify the presence of Nickel in Samples C.

(a) Sample B **(b) Sample C**

Figure 5. Evolution of Hydrogen Gas during Plating Process

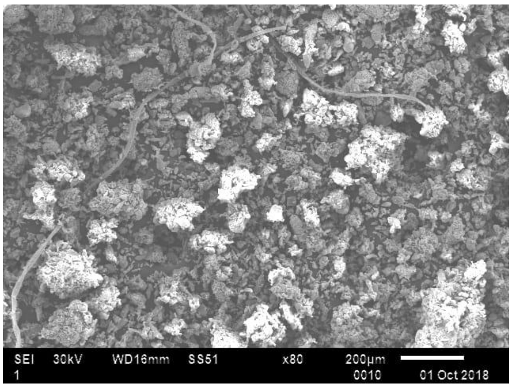
The photographs of Sample A, B and C after plating were shown in fploowing figures. The color of the powders was darker in sequence. The color of Sample A is light grey, while the color of Sample B is thick grey and Sample C is black. . The darker the color the rich in deposition of Ni[7]. These results indicated the possible signs of good Ni deposition in Sample C. The reason for less color change in Sample B may be due to the absorbed Sn^{+2} ions on the surface of Aluminium particles during sensitization process being rinsed out because of washing more times [7]. So it does not acquire enough strength to attract nickel from the bath. Clearly shown in figures

a) Sample A **b)** Sample B

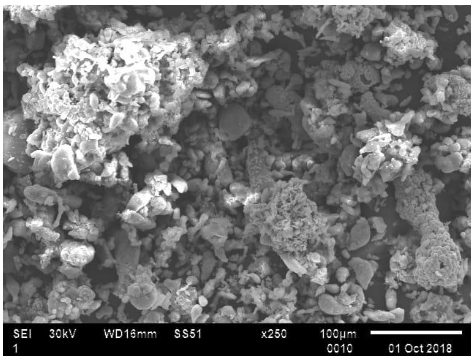
c) Sample C

Figure 6. Optical Photographs of Raw and Electroless Ni Deposited Powders under Different Conditions

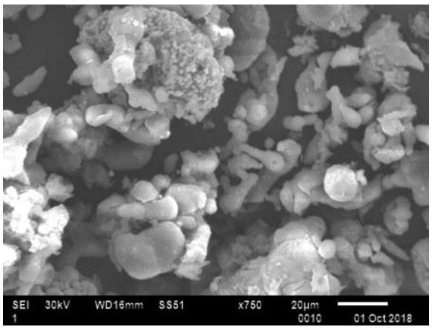
Figure 7 shows the SEM photographs of Aluminium powders before and after electroless deposition. It can be seen that the surface of raw powders were smooth and featureless [Fig. 7(a)]. After electroless nickel deposition, uniform and nanosized nickel particles instead of continuous Ni layers were observed adhering onto the surface of Aluminium particles for sample B [Fig. 7(b)]. Furthermore, it can be seen that the surface of powders of sample C were completely covered with large amount of nickel particles [Fig. 7(c)].



a) Sample A



b) Sample B



c) Sample C

Figure 7. SEM images of the eletroless Ni deposited powders.

7. CONCLUSONS

The present work shows a possibility to deposit different forms of Ni on surface of Aluminum particles. It is found that the $SnCl_2$ sensitization process and the subsequent $PdCl_2$ activation process monitored the formation of Ni layer on aluminium particles. The $Sn^{2þ}$ ions are weakly adsorbed on the surface of aluminium particles, and can be rinsed out by distilled water after sensitization process. The rinsing will lead to less/ no Pd on particle surface, while the structure of Ni layer is determined by the distribution structure of Pd around the Aluminium particles during the activation process. The size of nickel particles adhering to the surface of the Aluminium particles were about 50–80 nm.

8. REFERENCES

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