

An Experimental Study of the Effect of Nickel with SiC Codeposited on Aluminium 7075 under Direct Current and Pulse Current

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Abstract In the present work, Nickel with micro SiC composite coatings were obtained by electrochemical codeposition method from an additive – free Watts type bath. The aim of this work is to obtain Nickel with micro SiC composite coatings on Aluminium 7075 with more reinforcements. For comparison, pure Nickel deposits were also produced under the same experimental conditions. The effects of the SiC particle concentration on the bath, addition of surfactants and the coating time on the composition of micro-composite coatings were studied. The surface morphology and micro structural study were executed by means of Scanning Electron Microscope (SEM) and X-ray diffraction (XRD) analysis. Characterization experiments showed micro SiC codeposition with Nickel caused changes in the texture of the Nickel matrix. Moreover, the results clearly depict the high incorporation of micro SiC in Pulse Current codeposition when compared with conventional Direct Current.

Keywords Electro-codeposition, Micro SiC, Direct Current, Pulse Current, SEM, XRD, Aluminium 7075

1. Introduction

Applications of Aluminium 7075 gained increasing attention in aerospace and allied fields due to its inherent lightness and good strength-to-weight ratio. It replaces steel and many other metals where weight is the major factor since its density is one third of the steel. Even though it has good strength-to-weight ratio, it cannot withstand fretting wear and fretting fatigue loads as much as other metals [1-5]. Hence, when Aluminium is used under fairly high temperatures and weary conditions such as engine cylinder pistons, it needs surface treatments to increase the wear resistance and to lower the coefficient of friction. This is the fact which increased the interest on electrodeposition of Nickel on Aluminium 7075 that can minimize the problems of wear to some extent [3-7]. Therefore the process of electroplating has been carried out on applications such as engine cylinders, high pressure valves, drill fittings, aerospace, mining equipment where high wear rate can be spotted [16-20]. Electroplating of Nickel on any metal reduces wear but, when fine particles of micro size metallic or non-metallic reinforcement in Nickel plating enhances the wear resistance, lubrication and corrosion resistance than the

pure Nickel plating [6-10].

In the current scenario, research on codepositing ceramic particles such as SiC, Cr₂O₃, TiO₂, Al₂O₃, and WC along with Nickel plating finds a platform to improve the wear resistance of plated component [6-20]. Improvement of wear resistance by this codeposition could be obtained only by optimizing the parameters such as percentage of particle concentration, bath composition and temperature and further more. Electro deposition of Nickel was conventionally adapted for many decades and was successfully implemented in the industries when compared with other techniques. Hence this research focuses on the optimization of the parameters to codeposit the non metallic particles along with Nickel using the conventional electroplating method.

Recent literatures reveal that codeposition of micro size particles homogeneously in the deposit is highly difficult because of the higher tendency of micro particles to get agglomerated. Non-homogenous electro-codeposition leads to decrease the wear resistance of the coated substrate [21]. Hence more attention for research is needed to attain the homogeneous distribution of reinforced particles in near non- agglomerated form leads to the harder and more wear resistant coatings. Direct current (DC) was conventionally used to coat Nickel in industries but only few researches were carried out using Pulse Current (PC) to study the influence of it on electro-codeposition [22]. The aim of this research work is to find the suitability of electro-codeposition method by analysing the DC and PC

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parameters in order to attain the maximum incorporation and homogeneous distribution and thereby enhancing the wear resistance of the coatings.

2. Materials and Method

2.1. The Electrolyte

Table 1. Chemical composition and the parameters of electrodeposition

Electrolyte (Watts' type)	Concentration (gL ⁻¹)
NiSO ₄ .6H ₂ O	300
NiCl ₂ .6H ₂ O	50
H ₃ BO ₃	40
Sodium Dodecyl Sulphate	0.2
Silicon Carbide (SiC)	5,10,15,20
pH	4
Temperature (°C)	55
Current Density (A/dm ²)	0.5-2
Stirring Speed (rpm)	250 -650
Plating Time (min)	60

The standard Watts' Nickel Sulphate bath was used and the composition of plating solution along with the parameters was stated in Table 1. Codeposition of micro SiC in near non-agglomerated condition is the major problem in composite coatings. To avoid agglomeration problem Sodium Dodecyl Sulphate has been added as the surfactant which activates the positive zeta potential of micro SiC particles. The positive zeta potential of inert SiC particles gains extra adhesion force with the cathode is activated [23]. During electro-codeposition, the SiC particles settle at the bottom of the plating bath which reduces the percentage of incorporation of micro SiC particles in the codeposition process [25]. Along with the surfactant and stirrer, the agglomeration of the particles were avoided by preparing a mixture by blending the required volume of SiC with little electrolyte and a small volume of surfactant as a paste. The paste is then mixed in the plating bath and stirred for a minimum of one hour before deposition process.

2.2. Substrate Preparation

The aluminium 7075 substrate does not have the tendency to get coated with Nickel deposition directly. Hence the Aluminium 7075 substrate has to undergo some preliminary process such as zincating and copper plating, before carrying out electro-deposition of Nickel with SiC. Aluminium 7075 was purchased in form of rods and was machined in the form of billets of size 25 mm diameter and 25 mm height as per the ASTM standards [6]. They were polished with sandpaper, 2000 grit size to obtain uniform and smooth surface. The aluminium substrates were first degreased before electroplating in 42 g/L NaOH solution at 65 °C for 20 seconds. Further the substrate is cleaned in distilled water and it is zincated (Zinc oxide 100g/L, Sodium hydroxide 525 g/L, ferric chloride 10g/L and Sodium tartate 1 g/L). Copper deposition is done on the zincated Aluminium substrate for

30 seconds and again washed in distilled water before the codeposition of Nickel with micro SiC is done [24].

2.3. Plating and Testing Methodology

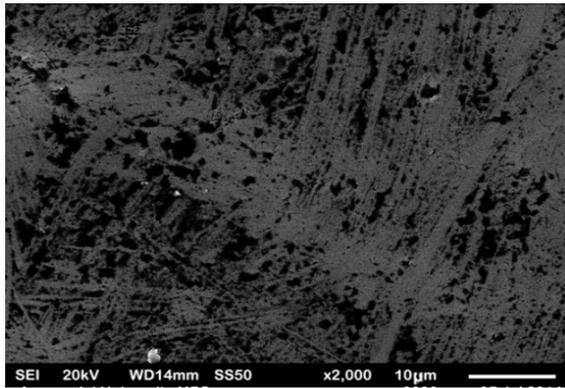
Nickel plate of 30 X 35 mm is used as anode. It was cleaned properly to remove the oxide layers. The bath which is shown in Fig. 1 was controlled by a tailor made microcontroller and it was developed for this research work through which all the parameters such as current density, voltage, plating time, temperature and stirring speed were controlled. The microcontroller was also equipped to generate DC and the PC with varying duty cycles. The microcontroller was designed in such a way that the parameters can be edited. The actual values and the edited values can be monitored through the display devices which were controlled by the microcontroller. The freshly prepared electrolyte as per the Table 1 is used and for fabrication of codeposited samples the SiC is dispersed in the form of paste and mixed to maintain the homogeneity using stirrer for minimum of one hour before the electro-codeposition. Pure Nickel and composite Nickel with SiC coatings were electrodeposited based on the conditions shown in Table 2. The commercial micro SiC powder of 99.9% pure with mean diameter of 2-3µm was added in the bath. The pH of the plating bath was maintained to 4.0 ±0.1 in all conditions. A constant distance of 3 cm was maintained between the cathode and the anode. The current density, voltage and temperature were constantly maintained and the values have been furnished in table 2 for all samples. Both the DC and PC with 50% duty cycle were employed to coat the samples and it was ultrasonically cleaned in ethanol for 10 minutes to remove loosely adsorbed particles from the surface. The coating time for each specimen was varied to maintain the coating thickness to a constant value of 50 µm. The composition of percentage weight of micro SiC was varied between 0% to 20% for both DC and PC. The samples were then prepared for SEM analysis. Morphology and crystallographic structures of the electro-codeposited samples were taken using SEM (JEOL-JSM-6610LV) machine at different magnifications. The coated samples were examined and the presence of micro SiC as reinforcement in the Nickel matrix was confirmed. The size of micro SiC incorporated in each codeposited samples were measured and it is correlated with the size of micro SiC dispersed in the plating solution.



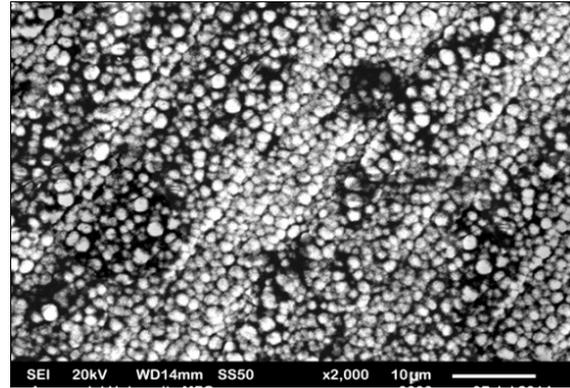
Figure 1. The Experimental Setup

Table 2. Conditions for electro-codeposition of samples

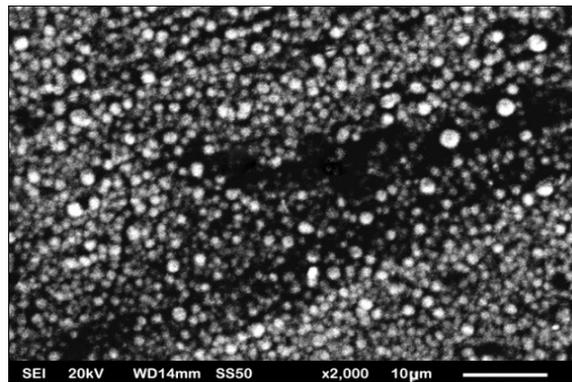
Sample Number	DC/PC	% Wt of Micro SiC of Size 2-3 μm	Voltage in volts	Current Density in A/dm^2	Temperature in $^{\circ}\text{C}$	Stirring Speed in RPM
1	DC	0%	2	2	55	250
2		5%	2	2	55	250
3		10%	2	2	55	250
4		15%	2	2	55	250
5		20%	2	2	55	250
6	PC	0%	2	2	55	250
7		5%	2	2	55	250
8		10%	2	2	55	250
9		15%	2	2	55	250
10		20%	2	2	55	250



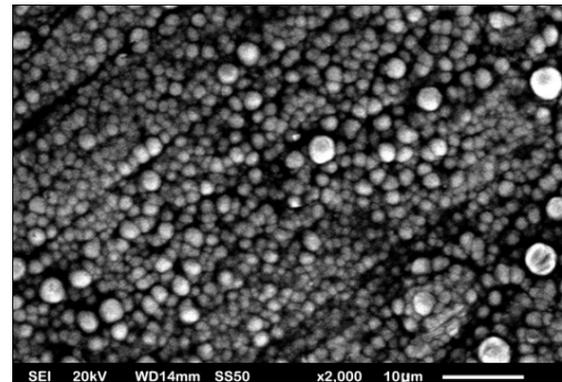
(a)



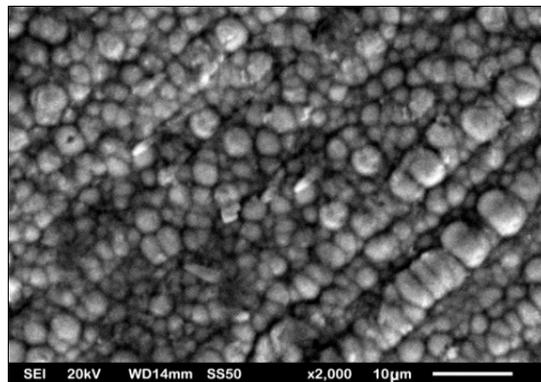
(b)



(c)



(d)



(e)

Figure 2. SEM micrographs of the samples prepared under DC (a) pure Nickel. (b). 5% codeposited SiC with Nickel. (c).10% codeposited SiC with Nickel. (d). 15% codeposited SiC with Nickel. (e). 20% codeposited SiC with Nickel

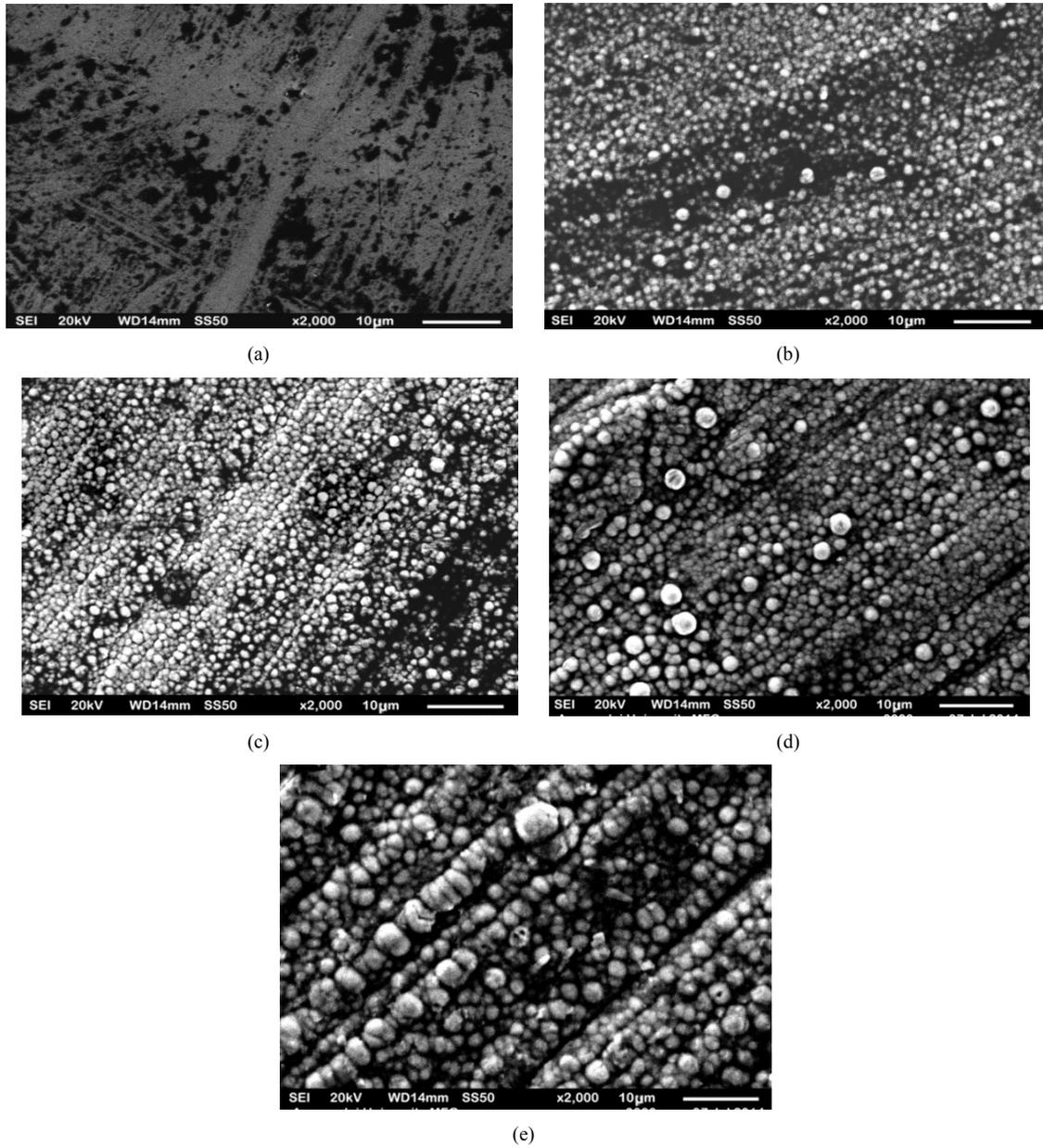


Figure 3. SEM micrographs of the samples prepared under PC (a) pure Nickel. (b). 5% codeposited SiC with Nickel. (c).10% codeposited SiC with Nickel. (d). 15% codeposited SiC with Nickel. (e). 20% codeposited SiC with Nickel

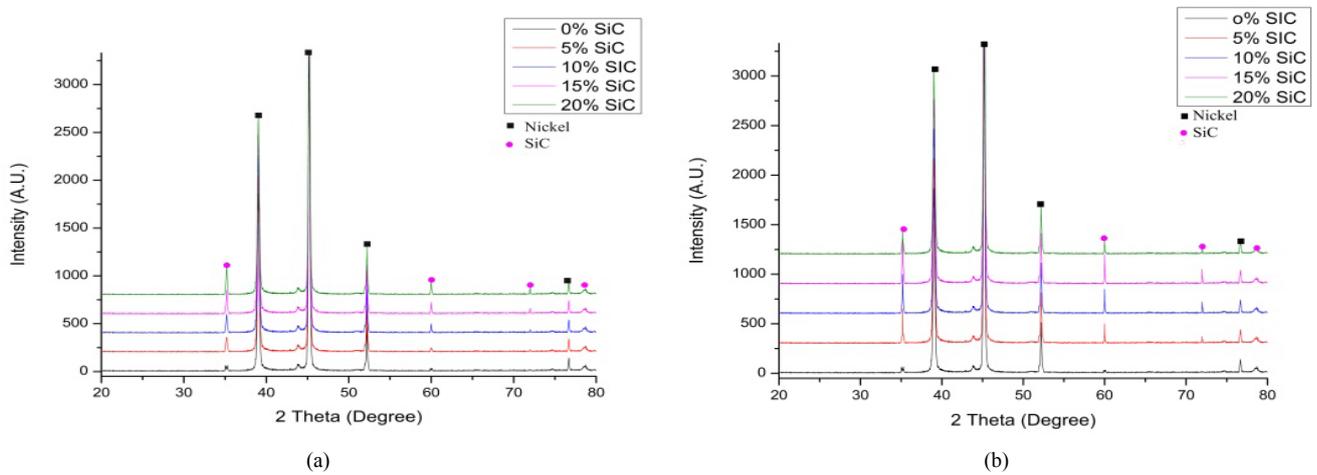


Figure 4. XRD patterns of (a) Direct Current (b) Pulse Current

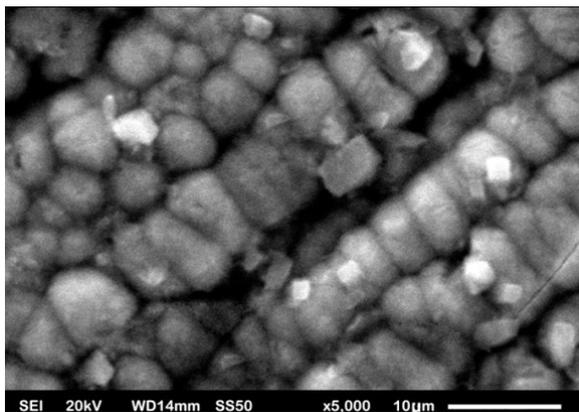
3. Results and Discussion

The optimum values of the variables of the bath such as current density, voltage, coating time, percentage volume of the surfactant, stirring speed and the distance between cathode and anode were determined after conducting several experiments on trial basis. The smooth codeposited coatings as well as the codeposition of SiC were achieved under the condition of 4.0 pH, 55 °C temperature, 2 A/dm² current density, 0.2 g/L surfactant and 2 V voltages. The surface morphology of the samples prepared by DC and PC were shown in Fig. 2(a-e) and Fig. 3(a-e). Fig. 4 (a and b) shows the XRD analysis result which was carried out with a Rigaku D/Max/2200/PC model X-ray diffractometer at a scanning speed of 1°/min in the 2 theta range of 20-80. The Fig. 2 (a) shows the pure Nickel coating and Fig. 2 (b), (c), (d), (e) shows the codeposition of SiC with Nickel in 5%, 10%, 15%, 20% under the application of DC. Fig. 3 (a) shows the pure Nickel coating, Fig. 3 (b), (c), (d), (e) shows the codeposition of micro SiC with Nickel in 5%, 10%, 15% and 20% under the application of PC. It is apparent from the Fig. 2 (b), (c), (d) and Fig. 3 (b), (c), (d) that the composite coatings which were obtained by 5-10% of SiC by both Direct and PC method is smooth with uniformly distributed micro SiC particles. Whereas, the agglomeration and irregular microstructure of the Nickel and micro SiC particles can be seen in Fig. 2(e) and Fig. 3(e).

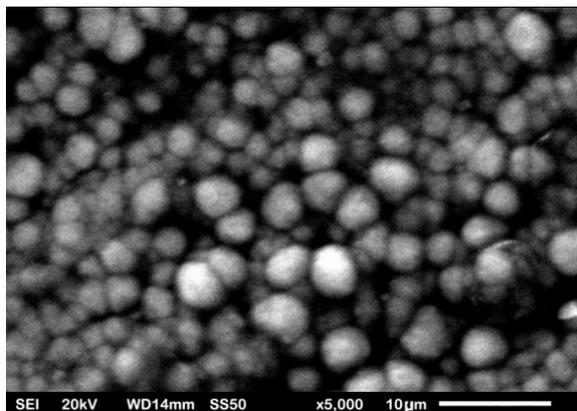
Hence these SEM images clearly depict that the addition of SiC particle from 5% - 15% shows a linear increase in incorporation and homogeneity and further increase in SiC addition drags down the codeposition of SiC particles in the Nickel matrix. This is due to the fact that the SiC particles get agglomerated when the volume of SiC is increased to 20% and hence it decreases the wettability of SiC particles [21]. The wettability of SiC particles decrease the tendency of adsorption with Nickel ions which leads to poor codeposition of SiC with the Nickel Metal Matrix Composite (MMC) coatings. The grain sizes of the coated metal matrix composite coatings were affected not only by the percentage volume of suspended SiC particles but also by the DC and PC coatings. It is evident from the Fig. 4a and Fig. 4b that the pure Nickel coating exhibit only three intense reflection peaks as reported by H.Gul et al. [28]. When SiC particles were introduced in solution the XRD patterns reflects more number of intensity peaks which were similar to the reference peaks of SiC [29,30]. Both in DC and PC coatings the SiC incorporation percentage increases linearly from 5g/L to 15g/L then start decreasing when the coatings were performed at 20g/L. This is attributed to the fact that introducing different amount of SiC particles in Nickel matrix resulted to change nucleation and growth kinetics of the Nickel matrix deposited [28]. Hence the XRD patterns shown in Fig. 4a and Fig. 4b clearly state that the embedding of SiC particles in the Nickel matrix modifies the texture from soft mode to mixed preferred mode [6, 27]. The DC codeposition of Nickel with SiC particle has a fine grain size when compared to the PC codeposition. However, the

homogeneous distribution of SiC in the coating is attained in better manner by PC. The higher gravimetric incorporation of micro SiC can be attained by PC rather than DC since, the micro SiC particles were deposited as inter-crystalline mechanism at the borders and edges of Nickel ions [27]. During DC deposition, the constant current density is adopted for codeposition of metal and SiC particles and hence the particle transfer is always slower than the metal ions which lead to decrease of the concentration of the adsorbed particles at the cathode surface, the adsorption become weak. However, in PC, during the off time, the micro SiC particles can be supplied when the Current Density is low or zero, which results in the enhanced amount of SiC particles in the coating. Thus the efficiency of the particle transfer in PC is higher than DC [26]. The experimental results revealed that the concentration of micro SiC particles in the electrolyte influences the addition of SiC in the codeposited Nickel matrix. SiC particles are absorbed by the Nickel ion in the electrolyte and this leads to the codeposition of composite coatings. The increase in SiC particles dispersion in the electrolyte increases the adsorption of Nickel ion by the micro SiC and therefore the codeposition percentage increases. The coating time shows a variation in coating a constant thickness over varying the concentration of the SiC in the electrolyte. The codeposition of SiC in matrix increases linearly when the concentration of SiC in the electrolyte increases whereas it decreases when the concentration is reduced. This is attributed to the fact that Nickel ions with lower throwing power finds difficult in carrying heavier SiC particles towards cathode. Hence the current density along with % volume of SiC suspended in electrolyte also plays the role on codeposition which varies the throwing power of Nickel ions. The throwing power of Nickel ions increases when the current density is increased. As S.M. Lari Baghal et al. [24] stated that an increase in current density leads to the increase in the weight percentage of SiC particles in Ni/SiC deposits. This is due to the fact that the adsorption process undergoes three steps: firstly, the particles loosely adsorbed on the substrate by hydrodynamic and electrophoretic force. Secondly, when current applied, the free ions and the ionic cloud formed by SiC particles are attracted towards cathode by electrophoresis force. Finally, particles in ionic cloud loose itself from it and strongly adsorbed at cathode [24]. As H.Gul et al. [28] stated, the entrapment of the SiC particles into the growing Nickel MMC coatings depend on both the rate of SiC particle and the Nickel ion approaching the cathode surface. The SEM micrographs of the samples shows increasing incorporation of SiC from 5g/L to 15g/L and started to decrease when coated with 20g/L. The same phenomena were even observed by H.Gul et al. [28]. However, higher the current density the coating will be rougher with burns on the surface. The higher current density also leads to the situation in which the Nickel ions with higher throwing power moves rapidly towards cathode without adsorbing the heavier SiC particles and hence leads to lower percentage incorporation of the SiC particles. The distance between the cathode and the anode

can also be varied so as to avoid higher throwing power. Hence the current density and the distance between the cathode and the anode are adjusted to a suitable level during coating to enhance the hassle free adsorption mechanism thereby avoiding the roughing and burning. When the concentration of the micro SiC is increased over the optimum level, the particle gets agglomerated in spite of all other parameters. Fig. 5 (a) shows the microstructure of an irregular codeposition of micro SiC in Nickel matrix. These kinds of agglomerations can be avoided by the addition of surfactants. It was confirmed in the Fig.5 (b) which shows the microstructure of the uniformly codeposited SiC with the addition of surfactant for the same operating parameters of the Fig. 5 (a). Addition of surfactants such as sodium Dodecyl Sulphate improves the stability of the suspension by reducing the surface tension and also enhances the electrostatic adsorption of the SiC particles on the cathode surface by increasing the positive charge.



(a)



(b)

Figure 5. SEM micrographs of the (a). Agglomerated SiC particles (b). Uniformly codeposited SiC particles under same operating parameters

4. Conclusions

In this work, Nickels reinforced with micro sized (2-3 μ m) SiC particles were successfully codeposited by DC and PC electroplating. The influence of the concentration of SiC particles in the bath on its codeposition mechanism along

with Nickel was investigated. The following observations have been made.

1. The SEM micrographs were captured and it inferred that the PC electroplating is the best route to codeposit micro SiC with much higher weight percentage when compared with the DC electroplating.
2. The increase in concentration of SiC in the bath increases the codeposition in Nickel matrix and it reduces beyond the optimum level.
3. The addition of surfactant increases the codeposition and maintains the homogeneity of coating by improving the zeta potential.
4. Optimization of the parameters such as current density, temperature, pH value, % volume of SiC dispersed in the bath also leads to the uniform coating and maximizes the codeposition.

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