Research Journal of Applied Sciences 14 (4): 103-109, 2019
ISSN: 1815-932X

Effect of Particle Size and Coating Parameters on the Uniformity of Ni-P Electroless Coating Applied on SiC Particles

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Abstract: The deposition of a uniform Nickel Phosphorous (Ni-P) coating on Silicon Carbide (SiC) particles by an electroless method was investigated in this research. The electroless bath is prepared with nickel sulfate and sodium hypophosphite at pH value of 8. In this research the effect of two different particle size 10 and 80 μm and the influence of both time and temperature on (Ni-P) coating deposition were studied. (Ni-P) coating is analyzed by optical microscopy (SEM) and (EDS). Uniformity in coating were obtained for pre-treated SiC with particle size of 80 μm at bath temperature of 70°C and stirring time of 15 min. Non-uniform coating was noted in SiC of 10 μm and in SiC of 80 μm during deposition time of 5 and 10 min. Electroless bath temperature of 50, 60 and 80°C resulted in the formation of segregated clusters of (Ni-P) leading in many uncoated parts of SiC particles.

Key words: Electroless, Nickel-Phosphorus (Ni-P), SiC particle, coating, formation, deposition

INTRODUCTION

Silicon carbide ceramics are widely used as reinforcement in Metal Matrix Composites (MMCs) due to their good mechanical properties like high hardness, elastic modules and wear resistance. They are suitable for aerospace, automotive and military applications (Khosroshahi *et al*., 2014; Li *et al*., 2013). Poor interface between MMCs and non-coated SiC particles due to their low wetting can be overcome by metallic coating. Electroless Nickel (EN) coating of the reinforcement has been applied to improve wettability and the surface energy of the reinforcement. EN is a process where metal alloys are deposit from aqueous solutions onto a substrate without using an electric current. It is a chemical process which reduces metal ions in solution to metal by chemical reduction (Pazman *et al*., 2010; Kretz *et al*., 2004; Leon and Drew, 2002). Previous researchers reported on EN coating of SiC particles with particle size of 8, 14, 70 μm, the smaller the size of SiC particles the higher clustering is formed (Kretz *et al*., 2004). Others investigated the effect of EN coating parameters and ceramic particle size on the uniformity of Ni-P coating, shown that uniformity of Ni-P coating were obtained for the 80 μm SiC particles at pH value of 8 during bath temperature of 50°C for 15 min (Khosroshahi *et al*., 2014).

In the present study, SiC particles were cleaned with acetone, surface pre-treated (with sensitization and activation aqueous solutions) and Ni-P coated by electroless plating method. In the sensitization process, Sn^{2+} ions are absorbed on the surface of the SiC powders which help in attracting Pd^{2+} ions during the activation process onto the surface of particles (Eq. 1). When the activated SiC powders are added to the bath, the metallic nickel could be deposited with the following reactions (Eq. 2 and 3) taking place:

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

$$Ni^{2+}+2H_2PO^{2-}+2H_2O \rightarrow Ni^{0}+2H_2PO^{3-}+2H^{+}+H_2 \quad (2)$$

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

The Ni^{2+} ions will be reduced to metallic nickel and the metallic Pd^{0} oxidized to Pd^{2+} (Kumar *et al*., 2016). Electroless plating is the deposition of autocatalytic chemical Ni-P which doesn't require any electric current; it is a self-regulated process by kinetics of the reactions involved.

MATERIALS AND METHODS

In this research, SiC powder of two different particle size were obtained, 99.51% pure SiC of 80 μm (Fixanal-Germany) and 99.4% pure SiC of 10 μm (Zhengzhou Haixu Abrasives-China) were employed to be coated (Fig. 1a, b). SiC powders were first cleaned with

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Table 1: Composition of the Ni-P electroless bath

Role in bath	Composition	Chemical formula	Concentration
Main salt	Nickel sulfate	$NiSO_4.6H_2O$	25 g/L
Reducing agent	Sodium hypophosphite	$NaH_2PO_2.H_2O$	27.6 g/L
Complexing agent	Tri-sodium citrate	$C_6H_5Na_3O_7.2H_2O$	46 g/L
Buffering agent	Acid boric	H_3O_3	26 g/L
pH adjuster	Sodium hydroxide	NaOH	To adjust pH to value of 8
SiC powder (particle size 10 and 80 μm)			0.5 g/100 mL

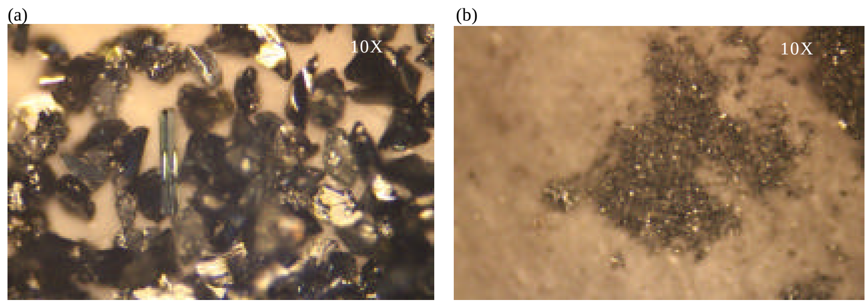

Fig. 1: Optical microscope of uncoated SiC particles: a) 80 μm and b) 10 μm

acetone to remove impurities then pre-treated in three steps followed by cleaning with distilled water after each step. Pre-treatment solutions included etching in HF (40%), sensitization in acidic solution of $SnCl_2$ followed by activation in acidic solution of $PdCl_2$. Powders then were dried for 2 h at 90°C then coated with Ni-P by an electroless method then dried for 2 h at 90°C (Khosroshahi *et al*., 2014). Pre-treatment procedures were carried out by magnetic stirrer while coating procedure for SiC were by magnetic stirrer and ultrasonic as an agitation process.

Electroless Nickel (EN) bath was formulated based on Nickel salts (Ni^{2+}), reducing agent (NaH_2PO_2) and additives that control the pH, complexing agent and the addition of other compounds to ensure quality of the participated coating (Table 1). Continuous, uniform deposition of Ni-P is achieved when Hydrogen atom (H) produced in the bath from reducing agent (NaH_2PO_2) by cathodic reduction.

Optical microscope, Scanning Electron Microscope (SEM) and Energy-Dispersive Spectroscopy (EDS) were employed to study the uniformity of the deposited coating on SiC particles. The difference in SiC powder mass before and after coating has been calculated and represented by the term weight gain.

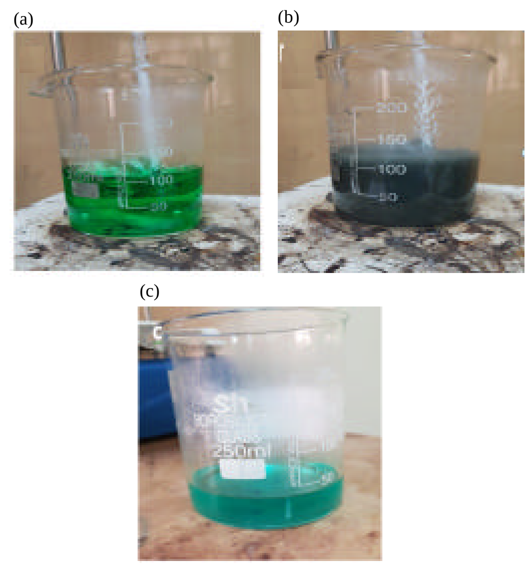

Fig. 2: Electroless nickel plating: a) Before adding SiC powder; b) SiC powder was added and Ni-P coating has formed and c) EN bath after coating and filtration processes

RESULTS AND DISCUSSION

When SiC powder is added to the heated bath, bubbles start forming (releasing hydrogen gas) accompanied by discoloration of the aqueous bath due to nickel reduction (Kumar *et al*., 2016). Bath color changes from deep green to light green which resample's the formation of Ni-P while SiC particles becomes darker in color which is more noticeable after passing it through filtration paper (Fig. 2).

The effect of two different particle sizes on Ni-P deposition represented in Fig. 3. EN bath for all Samples (S) at temperature of 70°C. Samples of 80 μm particle size S1 and S2 were mechanically agitated by magnetic stirrer and ultrasonic, respectively, both showed great weight

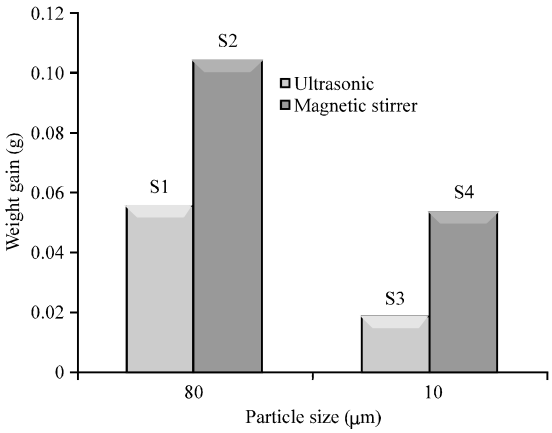

Fig. 3: The effect of different particle size (80 and 10 µm) and mechanical agitation (magnetic stirrer and ultrasonic) on coating uniformity

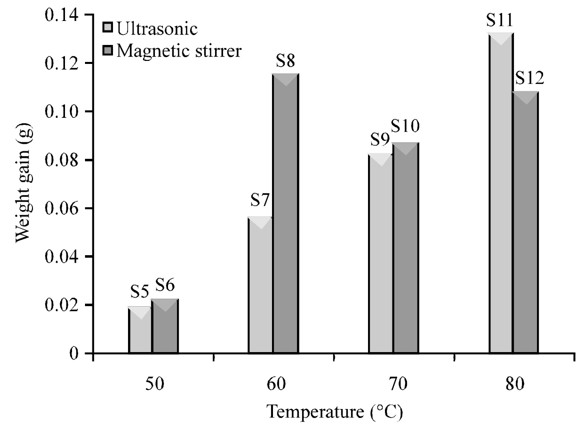

Fig. 5: Bath temperature (50-80°C) and mechanical agitation (magnetic stirrer and ultrasonic) effect on coating uniformity

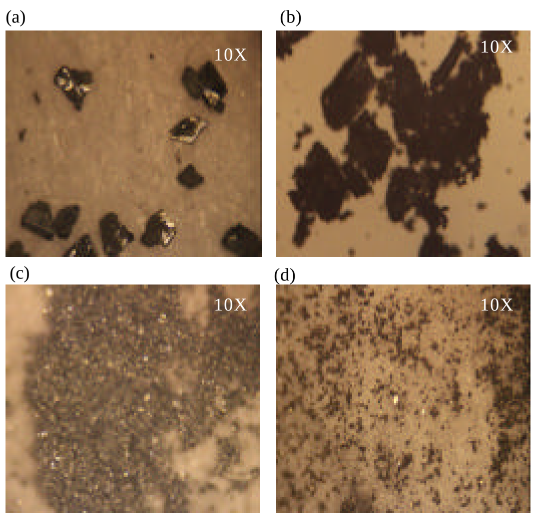

Fig. 4: a, d) Optical microscope for SiC particle size of 80 µm (S1) ultrasonic, (S2) magnetic stirrer and 10 µm (S3) ultrasonic, (S4) magnetic stirrer

gain increase in comparison with samples of 10 µm particle size S3 and S4 who were agitated by magnetic stirrer and ultrasonic, respectively. The use of magnetic stirrer during coating in both sizes (80 and 10 µm) showed more increase in weight gain than when using ultrasonic. Optical microscope images for all samples (Fig. 4), showed that almost all SiC particles surfaces in S2 are coated and has uniform plating. Although, sample S1 has good weight gain but optical microscope shows that most of SiC particles were uncoated this could be due to the use of ultrasonic which cause separation of the coat. In S3 and S4 samples of 10 µm particle size were not highly coated and many nickel-free SiC surface regions could be seen (Fig. 4). It seems during the coating of EN of these samples there was segregation of nickel clusters and small surface areas of particle were coated.

Figure 5 show the effect of using different bath temperatures (50-80°C) and mechanical agitation (magnetic stirrer and ultrasonic) in EN deposition on SiC of 80 µm particle size represented in it can be observe that the overall weight gain increases as temperature gets higher. For magnetic agitation samples (Fig. 6) shows that sample S10 with bath temperature of 70°C has better nickel coverage and growth on the SiC surfaces than that achieved with other temperatures despite for the high increase of weight gain during other temperature. S6 and S8 with bath temperature of 50 and 60°C, respectively, showed that particles are mostly coated with small exposed surface areas. The S12 of 80°C resulted in small formation of coating on SiC with large globules of nickel separated from particles. Meanwhile all ultrasonic samples S5, S7, S9 and S11 with bath temperature of 50-80°C, respectively, seems to have weight only that nickel coating is separated from the particles leaving SiC surface exposed. Figure 7 SEM images for S10 temperature of 70°C and S12 temperature of 80°C was taken for comparison. The S12 shows a large number of exposed SiC particles where nickel coating is separated in some particles the deposed coating seems to be flaking off. As for S10 it shows even deposition with some of the formed nickel clusters attached to SiC, however, there are some particles where coating has not formed on it. The reason for unfinished coating in S10 might be related to the absence of Sn^{2+} ions absorbed during sensitization are partly washed away during

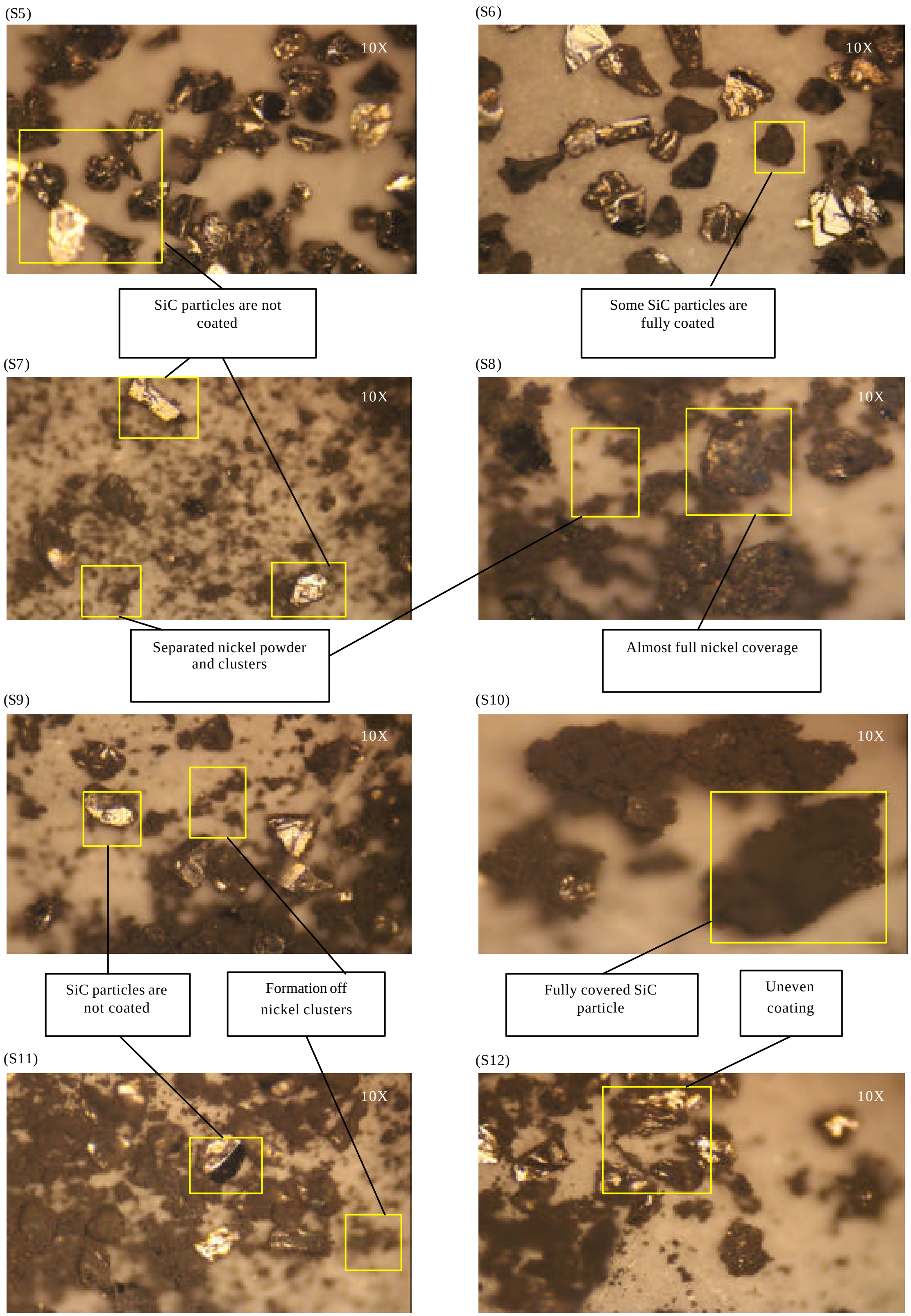

Fig. 6: The effect of bath temperature (50-80°C) and mechanical agitation

rinsing processes (Kumar *et al*., 2016). The EDS spectra also conforms the existence of Ni in sample S10 (Fig. 8).

Figure 9 shows time effect on coating uniformity, SiC of (80 μm particle size) were EN coated in bath temperature of 70°C for 5, 10 and 15 min . It is apparent that mass gain

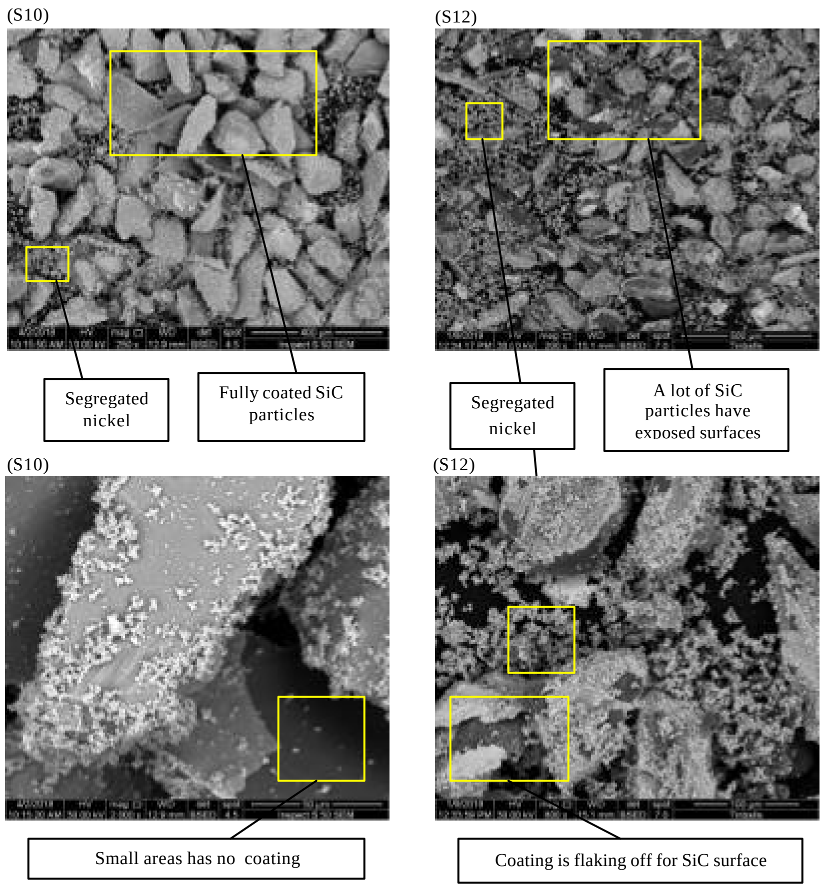

Fig. 7: SEM for SiC partical size of 80 μm, S10 (temperature of 70°C) and S12 (at temperature of 80°C)

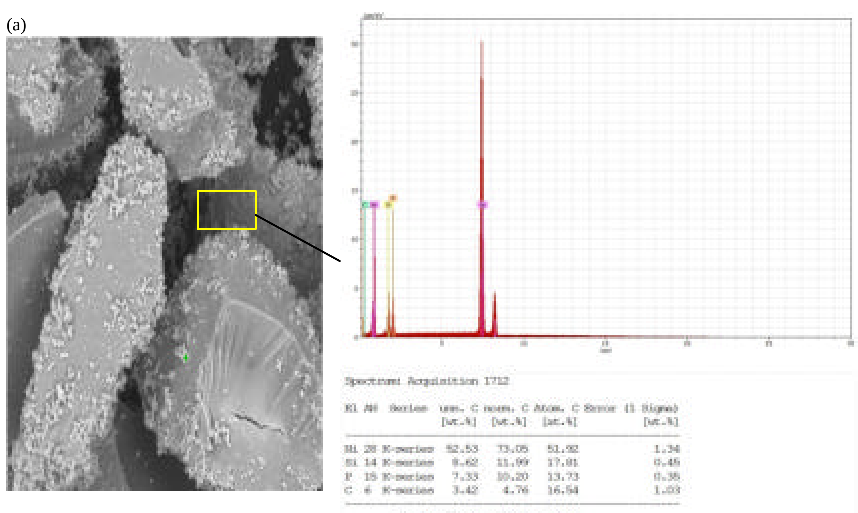

Fig. 8: Continue

(b)

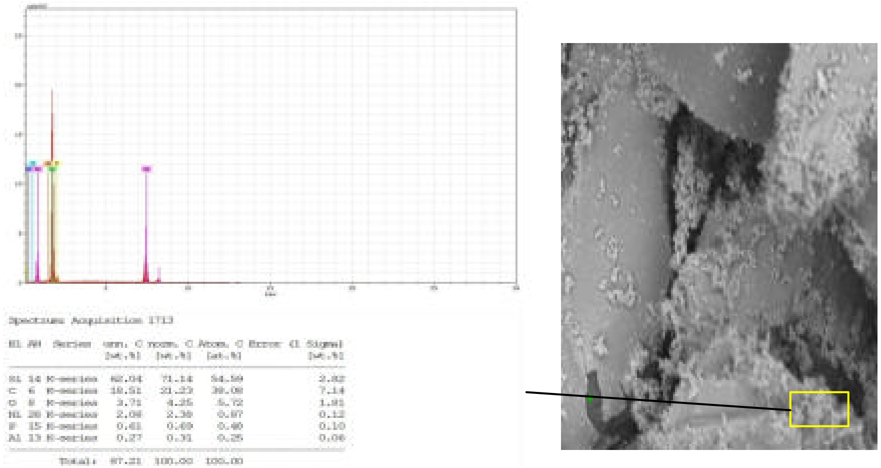

Fig. 8: EDS analysis for S10 of 80 μm and bath temperature of 70°C

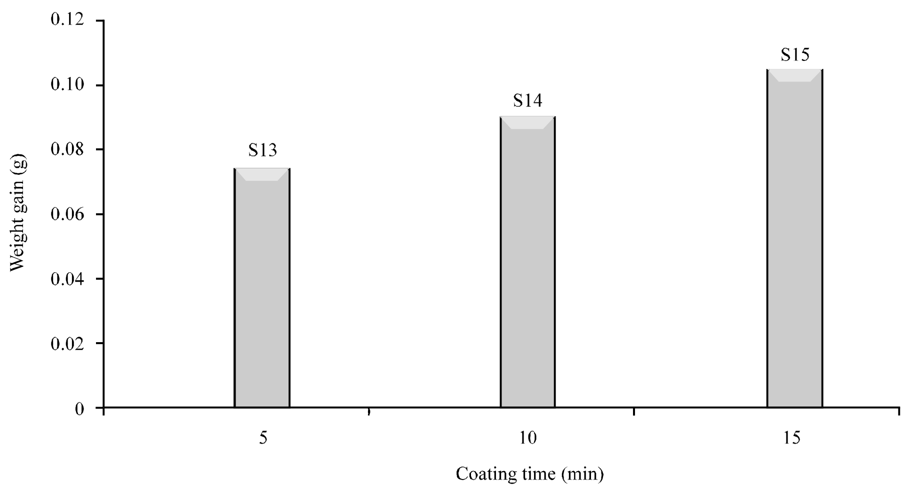

Fig. 9: Coating time effect on weight gain of SiC (particle size 80 μm at temperature of 70°C)

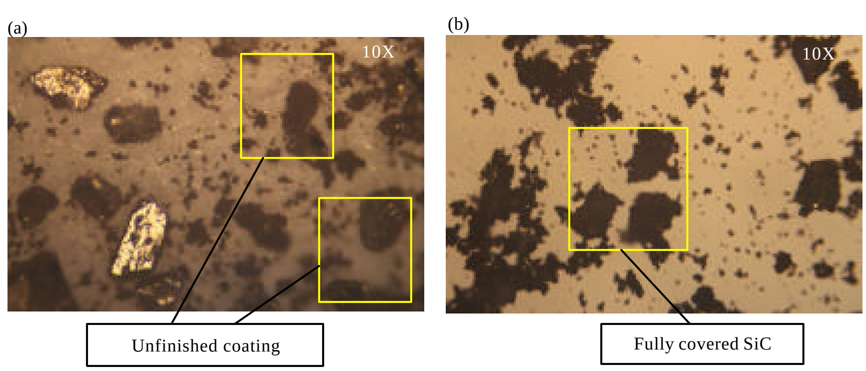

Fig. 10: Coated SiC powders (particle size of 80 μm and bath temperature of 70°C) for 5 min (S13) and for 15 min (S15)

increases with time where coating has more time to form and deposited on SiC surface resulting in a uniform coating with the formation of clusters attached to it as seen in S15 during coating time of 15 min (Fig. 10). S13 with coating time of 5 min shows larger surface area of SiC particles has no coating on them.

CONCLUSION

The aim of this research is to study the effect of particle size, bath temperature and deposition time on the uniformity of Ni-P coating.

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