

Study the Effect of Electrodeposition Parameters on Ni/SiC Composite Coating

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Abstract

In this research, we successfully obtained Ni/SiC micro-composite coatings with various contents of SiC particles of particle size (10 μm), by using electrodeposition method from nickel watts bath in which the SiC particles suspend. The effects of the current density, temperature, and particle loading (PL) of SiC particles in the electrolyte on the morphology, texture, and vol% of SiC in deposit were investigated. The morphological and structural analysis show uniform distribution of SiC particles within the composite coatings. It was found that the depositing conditions affect the microstructure of deposited nickel and the SiC vol% in deposit. Furthermore, the vol% of SiC increases in deposit by increasing the particle loading (PL) in the bath, while decreased by increasing the current density. Also the higher values of SiC vol% were obtained at temperature (50°C).

Keywords: SiC, Codeposition, Composites coatings, X-ray diffraction, Electron microscopy.

1. Introduction

Many trials have been made in order to find ways for improving the properties of surface of metal parts to defend from wear, corrosion, and decreasing their costs. Electrodeposition was regarded as one of the most essential, and cost effective manufacturing techniques for making protecting coatings. The improvement of current technology needs metallic substance with superior surface properties, of wear, and corrosion resistance. So the composite coatings have been the object of studying for some decades in industry, and science [1]. Although there is many techniques to produce the composite coating such as plasma spraying, physical vapor deposition, chemical vapor deposition, electrodeposition, powder metallurgy, sol-gel [2]. When compared between the coating methods, the electrocodeposition method is more beneficial, due to the ability of low temperatures of working, ease of maintenance, lower cost, and capacity to make composite coatings with various properties just by varying the electrodeposition parameters [3]. Composite electroplating or "Electro-codeposition" is a process of co-depositing fine particles of non-metallic, metallic compounds, or polymers in an electrodeposited

metal matrix to enhance the properties of material like, corrosion resistance, wear resistance, or lubrication [4]. One of the most extensively applied composite coatings is Ni/SiC system being its main field of application in the automotive manufacturing, which it is used to decrease the wear on the inner surface of cylinders, The properties of this system are subject to a number of variables, especially the nature, and concentration of the SiC particles distributed in the metal matrix. Also, there is a related experimental factors affect the co-deposition process of the Ni/SiC system like, current density, electrolyte composition, electrode/electrolyte movement, and bath temperature. However, it is difficult to find a clear image of exact influence of each factor, because often different, or even opposing results are reported by different authors [5].

[6] prepared Ni/SiC with SiC particles of micro and nano size by use a nickel sulfamate bath, they reported that the incorporation of SiC can be increased by raising the SiC concentration in the plating bath also found that the SiC particles of nano size are more hard to co-deposit than the SiC particles of micro size. [7] Obtained Ni/SiC nano composite coatings with different concentrations of SiC nano particles, they reported that the mechanical properties of the Ni-SiC nanocomposite coatings were enhanced, and also increased with raising concentration of the SiC nano particulate in plating bath.

The aim of the present work is study the effects of chosen deposition parameters (current density, SiC particle loadings, and bath temperature) on the SiC vol% in deposit using a nickel Watts bath.

2. Experimental procedure

2.1 Specimen preparation

Specimens with approximate dimensions of (5×5×0.7) cm^3 of copper sheet were used. The substrate surface ground using SiC paper down to the 2000 grade. Only one of the two surfaces with an area of (4×4) cm^2 was used as the plating area by isolating other surfaces with suitable adhesive polymer, giving an effective plating area of (16 cm^2). Then the copper substrates were rinsed in distilled water, rinsed with alcohol, pickled in

(10% H₂SO₄) for 30s, then washed with distilled water, dried and prepare to deposition.

2.2 Codeposition

The codeposition of Ni-SiC composite coatings was done by using the Conventional Electrocodeposition CECD set up with vertical and parallel electrodes having a constant distance of (9cm) for all experiments. The electrolyte was Nickel Watt’s bath. The composition and the range of the parameters of experimental process are listed in Table(1). The electrolyte was mixed with SiC powder of particle size (10 μm), and magnetically stirred for 1 h and then ultrasonically agitated for 1 h just before electroplating to enhance the wettability of particles, and to achieve uniform dispersion of reinforcement particles in the electrolyte.

During the electrodeposition, a magnetic stirring was done continuously to avoid settling down of SiC. The temperature was maintained by using heating element (filament wire), which was directly dipped in the bath, and controlled by temperature controller. While the pH value was corrected by using of sodium hydroxide. The plating time was changed for various current density levels to ensure that all coatings were approximately (50 μm) thick.

Table 1- Bath Composition and Operational parameters

Bath Composition	
Nickel Sulfate (NiSO ₄ .6H ₂ O)	330 g/l
Nickel Chloride (NiCl.6H ₂ O)	45 g/l
Boric acid (HBO ₃)	30 g/l
Hydrogen peroxide (H ₂ O ₂)	0.5 ml/1h
Operational parameters	
Dispersion ((g/l)	Silicon carbide(SiC): 2, 5, 8
pH	5.3 ± 0.2
Temperature (°C)	(40 , 50 , 60) ± 2
Current density (A/cm ²)	0.01, 0.04, 0.07, 0.1, 0.2

2.3 Samples analyses

2.3.1 Morphological Test

In order to study the morphology of prepared electrocomposite coatings and investigate the SiC particle incorporations in the coatings, an optical ((LOM) MEIJI TECHNO CO. LTD) and scanning electron microscopes (SEM) (INSPECT S50 (FEI) made in Netherlands) were used. The

test samples were cleaned with distilled water to get rid of the loosely particles on the surface.

2.3.2 X-ray Diffraction

Phase analysis of the coating was done by using X-ray diffraction (XRD) using (XRD-7000, Shimadzu), with Cu Kα radiation of wavelength 1.5418 Å, and a scan speed of 8.0 (deg/min). The Crystallite size of the material of matrix was calculated for pure nickel, and Ni/SiC composite coatings from the (XRD) pattern results, and by using Scherrer’s equation[8], given by:

$$D = \frac{0.9\lambda}{B \cos \theta}$$

where *D* is the main grain size of obtained coating, *λ* is the wavelength of Cu Kα radiation (0.154nm), *B* is the full width at half maximum (FWHM) of the films in radian, and *θ* is the Bragg diffraction angle.

2.3.3 Particle incorporation investigations

The vol% of SiC particles in the Nickel matrix was assessed by dissolution analysis, which includes calculation the weight% of SiC micro-particles which located within the Ni matrix, after dissolve both the substrate and Ni matrix by using solvents do not affect the particles.

3. Result and Discussion

3.1 Morphological Test

The Morphology was examined by means of (optical, and (SEM) microscopes); the surface images show that the SiC particles are homogeneously dispersed in the composite coating. This homogeneity mainly because the uniform dispersion of the SiC particles in the plating bath, which achieved by mechanical agitation process. And the uniform dispersion of the SiC particles in the coatings is the most important role to obtain uniform mechanical properties of the composite coating.

The SiC micro-particles on the samples surface were partially incorporated in the metal matrix through growth as seen in **fig.**(1), can be observe that the particles have a rather angular form.

The Optical microscope images show that the vol% of SiC incorporation influenced by deposition parameters. The incorporation of particles decrease with increasing the value of current density, as in **fig**(2), while increase with the increasing the PLs in the bath, as in **fig**(3). In **Fig.**(4), we could observe that the higher incorporation of SiC in deposit was obtained at temperature (50°C).

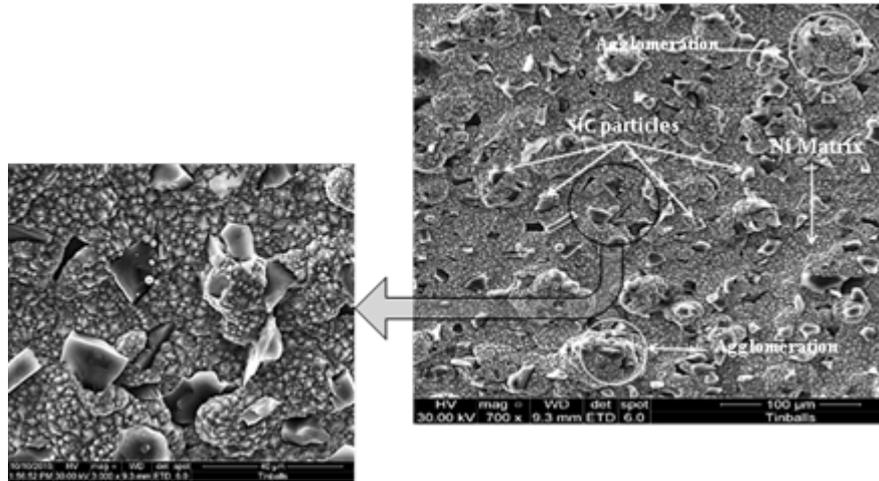


Figure (1): General topography of the Ni-SiC composite coatings for sample prepared at PL=8g/l, D=0.04, T= 50 °C

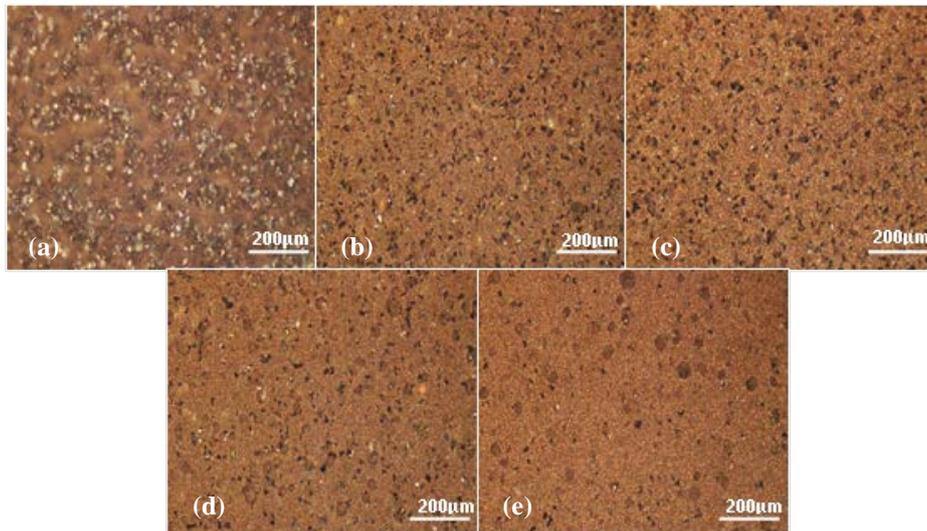


Figure (2):The morphology of Ni-SiC composite at PL=(5)g/l, and Temp(50)C⁰ with different current densities: (a)0.01, (b)0.04, (c)0.07, (d)0.1, (e)0.2

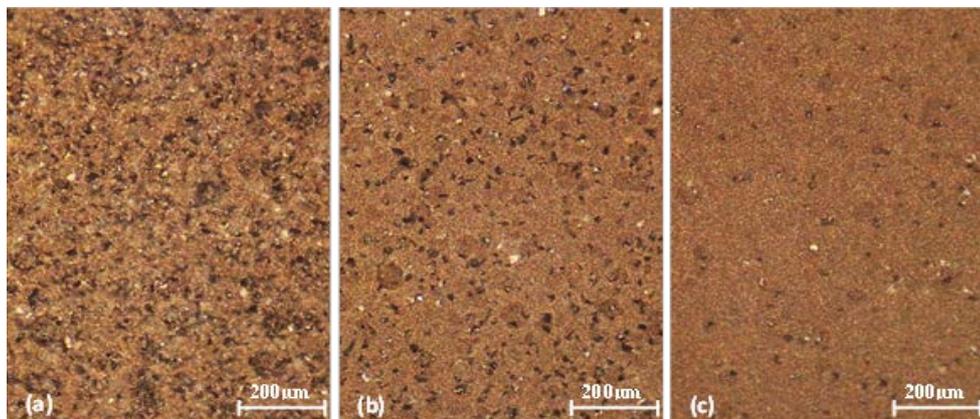


Figure (3): The microstructure Ni-SiC composite with different PLs: (a) 8 g/l (b) 5g/l (c) 2 g/l.//at D=0.04, T=50 C.

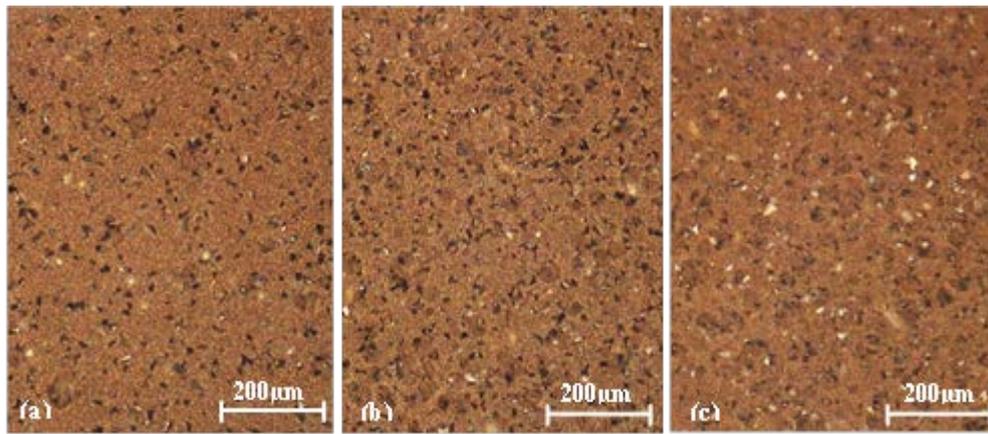


Figure (4): The morphology of Ni-SiC composite with different Temperatures: (a) 40, (b) 50, (c) 60, at $D=0.04$, $PL = 5 \text{ g/l}$

3.2 Volume fraction of SiC in Deposit

The Vol.% of ceramic particles reinforced MMCs prepared by electrodeposition method cannot be simply controlled or fixed the required vol.%. But it form in the simple range which can be controlled the vol.% of SiC by controlling the deposition variables.

Influence of plating condition on the SiC volume fraction in deposit:

I. Current density:

Fig.(5) shows that when the current density increases, the vol.% of SiC particles in deposit is decrease. In composite electrocodeposition, when the particles fill the available sites for Ni deposition, the real current density is consider higher than the obvious one; this may due to the reduced the real available area for nickel deposition[9]. Thus, it is desirable to use a reasonably low current density in order to get coatings with the wanted properties. Also that when the current density is low (0.01 A/cm^2),the required time to get the wanted thickness of the coating is longer when compared to the time at higher current density (0.1 A/cm^2). Thus, the probability of entrap the particles at lower current density is more[10]. While the current density increases, the movement of Nickel ions from the electrolyte to the surface of cathode was faster than that of the SiC particles. So the Nickel deposition rate increases and this results in obvious decreasing in the incorporation of SiC.

II. Bath Temperature:

Generally nickel baths are worked in the temperature range of $40\text{--}60 \text{ }^\circ\text{C}$ to increase the deposition rate of nickel. Fig.(6) shows the variation of SiC particles vol.% with current density at different bath temperature. It can be seen that the vol.% of SiC particles increased with increasing the bath temperature up to $50 \text{ }^\circ\text{C}$, and then will be decreased. Below 50°C , the activity of particles increases with raising the bath temperature. When the bath temperature is higher

than 50°C , the thermodynamic motion of ions is greatly improved, which results in increasing the kinetic energy of particles, and hence to decrease the over potential of the cathode and the electric field[9], which is may be more difficult for the particles to be incorporated in the metal matrix and then leads to a reduction in the SiC vol% in the deposit.

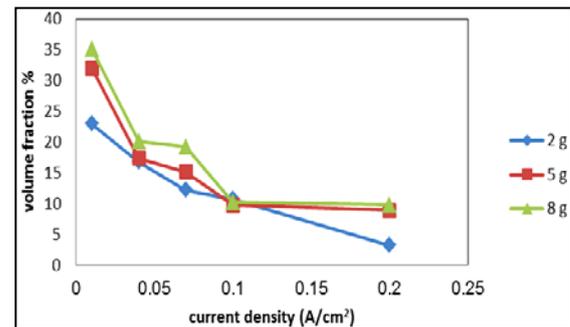


Figure (5): Variation of the SiC volume% in coating with current density for different PLs in the bath, at Temperature = 50°C .

III. particle loading:

The SiC Vol.% in deposit increased with increasing the PLs in the plating bath up to around (8 g/l) as shown in Fig.(5). The increasing may be owing to increase the amount of SiC particles reaching the surface of cathode with increasing the concentration of SiC particles in plating bath. Only the particles which stay adsorbed on the surface of cathode for an enough time are effectively codeposited into the growing Nickel matrix [11]. Since the amount of particles approaching the surface of cathode increases with increase the SiC concentration in the plating bath. So a higher particle loading increases the adsorption,thus resulting in a higher SiC vol% in deposit.

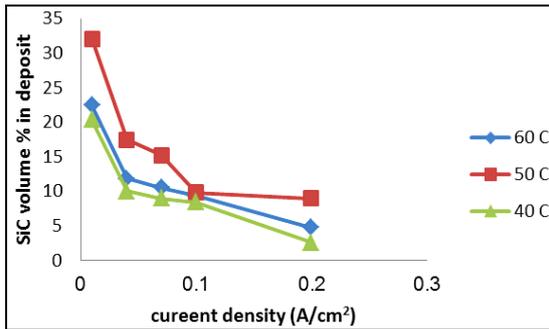


Figure (6): Variation of SiC volume.% in coating with current density for different temperatures, with PL= 5g/l

3.3 X-Ray Diffraction

The structure of both pure and reinforced Nickel coatings and the effect of the SiC particles on the structure were studied by using the (XRD) diffraction. The diffractions of pure Ni coating in

Fig.(7) show five peaks of changed intensities features of a (f.c.c) structure and it is clearly seen that the intensity of (111) crystal plane was upper than those of other crystal planes for pure Nickel

coatings.

The diffractions of Ni-SiC composite coatings reflect variations in the micro-structure which were certified on the particles concentration in the plating bath. Where the intensity of the diffraction peaks decreases with increasing the PLs of SiC in the plating bath. This refers to that the volume% of SiC in the coating increases with increasing the PLs, and this agree with the data found by vol% analysis.

The crystalline size of Ni was determined for pure Nickel and Ni-SiC composite coatings by means of Scherrer's equation, as mentioned before. From the obtained results as shown in **Table (2)** it is worth noting the change and reduction of grain size by imbedding and increasing the amount of SiC in the deposit. As seen pure Ni coating showed a crystalline size of 36.7 nm, while the codeposition of SiC (16.8 vol.%) decreased the crystalline size to 22.3 nm. Also more incorporation of micro-SiC in a nano-crystalline nickel matrix has resulted in more reduction in crystalline size.

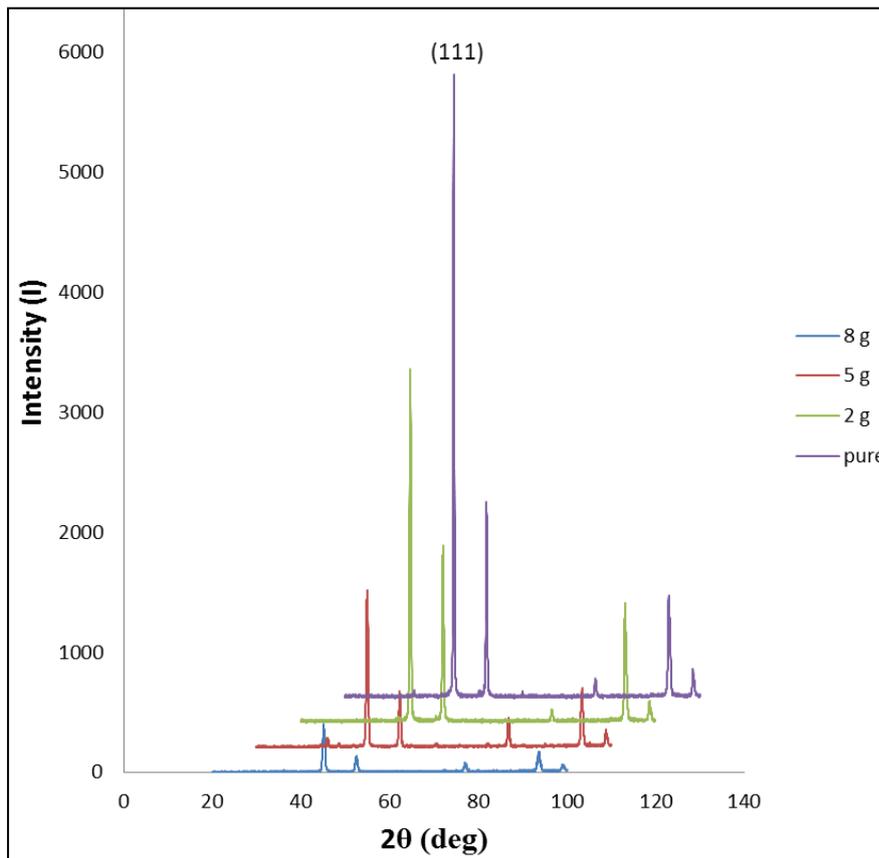


Figure (7): (XRD) pattern of pure Ni coating, and Ni/SiC composite coatings with different PLs

The explanation of the reduction in the crystal size of Nickel is that, through the electro co-deposition, the SiC particles could absorb cations in the electrolyte and would be adsorbed onto the surface of cathode. This would leads to [12,13]:

The occurrence of the new nuclei because the adsorbed micro-particles restricted the growth of the original crystal grains, and (2) an increase in electro crystalline potential because of the reducing electrical surface of cathode caused by the adsorbed particles.

Furthermore, the incorporation of SiC particles in Ni matrix has also caused an increase in the hardness (It will be mentioned in other research).

As seen in Fig.(7), the intensity of sample prepared at D=0.04 is lower than that of sample prepared at D=0.1. This indicates the reverse effect of current densities on the incorporation of SiC in deposit.

Table (2): Information obtained from XRD diffraction

D (A/cm ²)	Temp (°C)	PL (g/l)	2 θ (deg)	(FWHM) deg	Crystalline size(nm)
0.04	50	0	44.4887	0.23420	36.702
0.04	50	2	44.6819	0.38370	22.398
0.04	50	5	45.1686	0.50000	14.447
0.04	50	8	44.9473	0.51470	16.7214
0.04	40	5	45.1724	0.56640	15.193
0.1	50	5	44.8684	0.48600	17.703

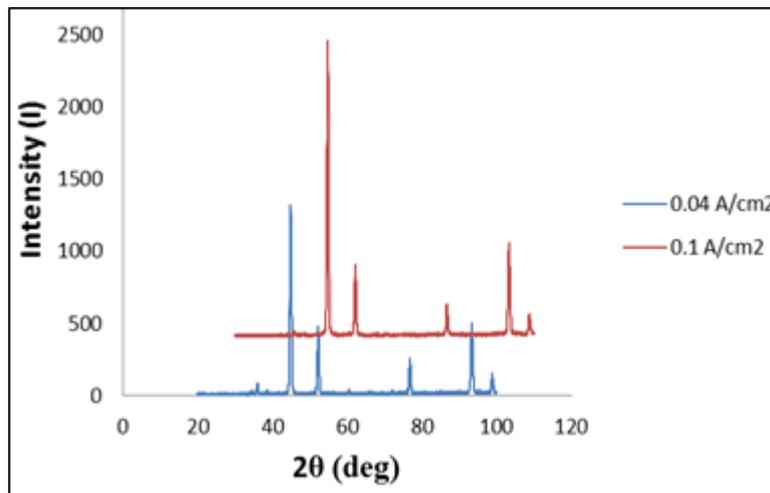


Figure (7); XRD pattern of Ni/SiC composite coating prepared at different current densities

4. Conclusions

Based on experimental results presented in this work, it was possible to draw the following conclusions:

-The deposition parameters (current density, PL, and bath temperature) exhibited a strong and direct relation to the final microstructure and morphology of the composites.

-The SiC incorporation in Ni matrix influenced by deposition parameters, an increase of the vol% of SiC in composite deposit is found by increasing the PL, and decreasing the current density, and higher vol% of SiC is obtained at temp = 50 °C.

-The crystals size and Ni microstructure of composite coatings were changed compared with pure nickel coatings.

5. References

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دراسة تأثير شروط الترسيب الكهربائي على طلاء مترابكات Ni/SiC

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الخلاصة

في هذا البحث تم تحضير الطلاء المترابكة Ni/SiC بنسب مختلفة من دقائق كاربيد السيليكون داخل حمام الطلاء ذات الحجم الحبيبي (10 μm) باستخدام طريقة الترسيب الكهربائي وباستخدام حمام واتس الذي تنشئت داخله دقائق SiC. حيث تمت دراسة تأثير كل من كثافة التيار، درجة حرارة حمام الطلاء، وتركيز الدقائق داخل حمام الطلاء على كل من مورفولوجيا السطح، التركيب، والنسبة الحجمية للدقائق داخل الطلاء. ان التحليل التركيبي للطلاء اظهر التوزيع المتجانس للدقائق داخل الطلاء. ووجد بأن شروط الترسيب لها تأثير على التركيب المايكروبي لطلاء المترابكات وكذلك على النسبة الحجمية للدقائق داخل الطلاء. علاوة على ذلك، فإن النسبة الحجمية للدقائق داخل الطلاء تزداد بزيادة تركيز الدقائق في حمام الطلاء، بينما تقل بزيادة كثافة تيار الترسيب. كما ان اعلى قيمة لاندماج الدقائق كانت عند درجة حرارة 50 °C.