Hindawi Publishing Corporation
Advances in Materials Science and Engineering
Volume 2015, Article ID 351780, 8 pages
http://dx.doi.org/10.1155/2015/351780

Research Article

The Microstructure and Thermal Conductivity of Pressureless Infiltrated SiC_p/Al Composites Containing Electroless Nickel Platings

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Received 22 March 2015; Revised 4 June 2015; Accepted 7 June 2015

Academic Editor: Jainagesh A. Sekhar

A nickel (Ni) coating was deposited on the surface of silicon carbide particles (SiC_p) through electroless plating and we characterized the morphology and phase structure of the coating and the pressureless infiltrated SiC_p/Al composites. The effect of Ni coatings on the thermal conductivity of the composites was examined and analyzed with three-dimensional video microscope, scanning electron microscope (SEM), energy-dispersive X-ray spectroscopy (EDS), X-ray diffraction microscope (XRD), and finite-element. The results show that a continuous and uniform coating with a certain thickness (around 3.5 μm) can be formed on the surface of SiC_p. With the addition of the Ni layer, there are some intermetallics Ni_3Al but no interfacial carbide Al_4C_3, which improves the wettability and the thermal conductivity of the composites. The experiments and simulations both show that Ni coatings do not substantially decrease the overall thermal conductivity of the composite, although the thermal conductivity of Ni itself is lower than Al and SiC by a factor of 1.

1. Introduction

In recent years, high volume fraction SiC_p reinforced aluminum matrix (SiC_p/Al) composites have received much attention in the aerospace, transportation, and electronics packaging industries, due to its desirable properties, such as high specific strength and specific modulus, low specific gravity, low thermal expansion coefficient, excellent wear resistance, and low cost [1–3]. Among all the available techniques for preparing high volume fraction SiC/Al composites, spontaneous or press infiltration technology is believed to be an ideal process because of its net-shape formation, low cost, and simple equipment. Biswas et al. reviewed the dynamic analysis of pressure infiltration of porous preforms by pure metals [4]. Pech-Canul and Makhlouf used starch (or dextrin) and wax emulsion as binders to form the SiC preforms (with porosity of 40%–50%) by cold press and obtained the optimum parameters for aluminum pressureless infiltration into porous SiC preforms [5].

However, there is a problem in this preparation process since SiC_p has poor wettability with molten aluminum [6]. Although the wettability between Al and SiC can be improved at an elevated temperature, the detrimental interfacial phase of aluminium carbide (Al_4C_3) easily appears and is known to be sensitive to some corrosive environments, resulting in the degradation of the properties of the composite. Certain methods need to be implemented to improve the wettability and also avoid the formation of Al_4C_3. At present, there are two methods that are routinely studied by some researchers. One of the methods to alloy the matrix is by decreasing the interfacial energy between Al and SiC and the surface energy of the molten aluminum. It has been established that adding certain Mg and Si elements in the aluminum system could be beneficial for the pressureless infiltration of the SiC preform by aluminum [7–9]. For instance, Choh and Oki [9] observed that the addition of Si to Al decreased the incubation period of wetting at temperatures between 1173 and 1373 K. The second method to increase the surface energy of the particle surface involves pretreatment of the SiC particles with several surface coating technologies, such as the sol-gel method or high temperature oxidation to obtain silica coatings [10, 11] by electroless plating [12, 13]. Among the above techniques, electroless plating is considered more effective due to its low cost, easy-to-control procedure, and large-scale production abilities. At present, the nickel platings produced by electroless deposition have already achieved remarkable results in the area of modifying weak wettability between ceramic particles and metal matrix alloys [14, 15].

From the available literature, a large number of studies have shown that Ni coatings can enhance the wettability and modify the interface bonding between SiC_p and aluminum matrix. The nickel layer has a lower thermal conductivity compared to the Al matrix and the SiC reinforcement. Furthermore, the addition of nickel may produce a new intermediate compound that may affect the thermal properties of the composites. Only few investigations have discussed the effects of Ni coatings on thermal conductivity of the composites.

In the present work, a Ni coating was applied on the surface of SiC particles using electroless deposition. The morphologies and the microstructure of the coated SiC_p and the SiC_p/Al composites were characterized, and the effect of Ni platings on the thermal conductivity of the composites was examined and analyzed.

2. Materials and Methods

2.1. Electroless Nickel Deposition on the Surface of SiC_p. Green and abrasive grade SiC particles with a purity of 98.5% and the average size of 100 μm were used as reinforcement for the composites. A nickel coating was deposited on the surface of SiC particles through electroless deposition. In the first part, pretreatment was performed by the traditional three-step method (coarsening, sensitization, and activation). First, the SiC particles were coarsened by etching in 5% wt hydrogen fluoride (HF) aqueous solution at room temperature for 30 min. Second, the particles were sensitized in silicon chloride ($SnCl_2$) solution followed by activation in silver nitrate ($AgNO_3$) solution, both for 10 min. After each step, the particles were rinsed in distilled water and dried in a vacuum oven at 75°C for 4 hours.

In the second part, electroless plating was performed in a nickel electroless bath. The bath contains nickel sulfate ($NiSO_4{\cdot}6H_2O$) as the main salt, sodium hypophosphite ($NaH_2PO_2{\cdot}H_2O$) as the reducing agent, sodium citrate ($C_6H_5O_7Na_3{\cdot}2H_2O$) and sodium acetate ($CH_3COONa$) as the complexing agent and buffer, thiourea (CH_4N_2S) as the stabilizer, and activated SiC particles. In order to reduce the agglomeration of particles and to form a fine and uniform coating, the particles were dispersed uniformly in the bath by an agitator at a certain speed. In addition, to control the quality of the plating deposition, the source of nickel, pH, and the temperature were adjusted in this experiment. The pH of the plating solution was adjusted and maintained to the desired levels with aqueous ammonia solution ($NH_3{\cdot}H_2O$). The temperature of the bath solution was carefully controlled to point values during the plating. The main controlling electroless parameters were listed in Table 1.

Table 1: Main controlling electroless parameters and their levels.

Factors	The change of their values
$NiSO_4{\cdot}6H_2O$	6 g/L, 7.5 g/L, 10 g/L, 15 g/L
pH	3.5, 4.0, 4.5, 4.8, 5.1
Bath temperature	70°C, 75°C, 80°C, 90°C

In the third part, based on the optimal electroless parameters, the morphologies and microstructure of the coated SiC deposits were characterized and analyzed with a three-dimensional video microscope, scanning electron microscope (SEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction microscope (XRD). The thermal stability of Ni-plated SiC particles was also analyzed.

2.2. Preparation and Characterization of the SiC_p/Al Composites. The electroless nickel-plated SiC particles and the original nonplated particles were used as reinforcement. ZL101 (Al, 92.45 wt%, Si, 7.2 wt%, and Mg, 0.35 wt%) was used as the Al matrix. The SiC_p/Al composites were fabricated by pressureless infiltration technology and the specific process is as follows. First, put certain SiC particles into a ceramic mold and some Al pieces into another graphite crucible. Then put the mold and the crucible together into the heating furnace. When the heating temperature reached 850°C and Al was melted completely, poured the Al liquid into the molding quickly and kept it warm at 920°C for 2 hours.

For testing other properties, some specimens were prepared by wire cutting. Each specimen was subjected to an annealing treatment (300°C × 2 h, air-cooled) before the test. The phase constituents and the microstructure of the composites were characterized by XRD, SEM, and EDS. Thermal diffusivity of the sample (sample size: a diameter of 12.65 mm and a thickness of 2.0 mm) was measured by the Netzsch LFA447 Laser Flash machine at room temperature. The thermal conductivity was specifically calculated as the product of thermal diffusivity, specific heat, and density. Meanwhile, the finite-element method (FEM) was implemented to determine the effective thermal conductivity of the composites with Ni-plated SiC_p. Based on stereology Deless law [16], the volume fraction of each phase is characterized by its area percentage. The models of the composites with Ni-plated particles were established as shown in Figure 1. The temperature load is applied by the ANSYS thermal analysis module. The temperature at the nodes along the left surface is described as T_{hot} (=35°C) and, on the right surface, the temperature is described as T_{cold} (=15°C). The ambient temperature is 25°C. For two other parallel surfaces, the direction of the heat flow to all the surfaces is assumed to be adiabatic. These temperatures are obtained with the help of a finite-element program package ANSYS, and the temperature field and local heat flux are analyzed by a program computing the effective thermal conductivity.

3. Results and Discussion

3.1. Coating Characterization. Figure 2 shows the macroscopic morphologies of the coated SiC_p obtained by

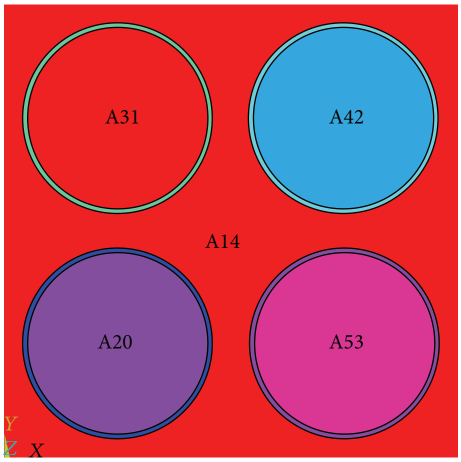

FIGURE 1: Model of the composites with Ni-plated SiC_p.

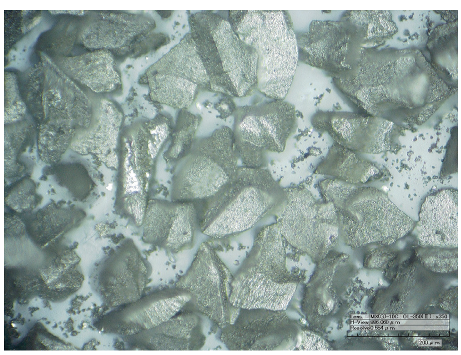

FIGURE 2: Macrographs of coated SiC particles.

the optimal electroless parameters ($NiSO_4 \cdot 6H_2O$ 7.5 g/L, pH 4.8, and bath temperature 80°C). It can be observed that the SiC particles are almost entirely coated with platings, and the platings are nearly uniform and continuous.

Figure 3 provides the XRD spectra of SiC_p before and after electroless Ni plating. The SiC diffraction peaks appear in the two samples. However, the SiC diffraction peaks of the uncoated particles (Figure 3(a)) are much stronger than those of the coated ones (Figure 3(b)). This is mainly because there is partial absorption of the incident and reflected X-rays by the as-deposited coating. It can also be mentioned that the XRD pattern of the coated SiC particles exhibits a new pattern that is tentatively designated as X-ray amorphous at 2θ ranging from 40° to 50° in Figure 3(b).

Figure 4 presents the SEM morphologies of the coating. It can be seen from Figure 4(a) that the entire surface of the SiC particles is nearly covered with a continuous film. Energy-dispersive X-ray spectroscopy (EDS) analysis, as shown in Figure 4(b), demonstrates that the Si, Ni, and P accompanying O are detected on the sample after plating, and it reveals a weak signal in the Si peak, which may indicate that a thin film has already formed on the surface of the SiC particles. Moreover, the appearance of the O signal in the EDS indicates that there could be a reaction involving oxygen during plating, which is most likely due to Ni oxides or hydroxides.

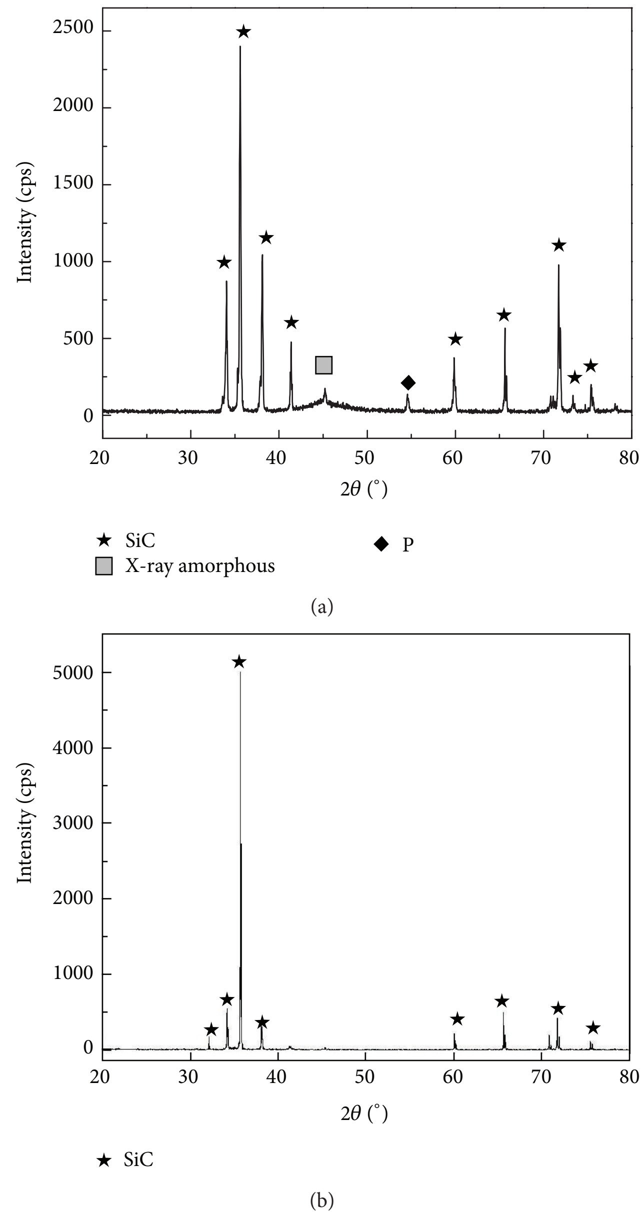

FIGURE 3: XRD patterns of SiC_p: (a) after and (b) before electroless Ni plating.

The high-magnification micrograph in Figure 4(c) indicates that the final coating is constituted with irregular nodules that are squeezed against each other to form a compact layer. Besides, as shown in Figure 4(c), there are no obvious cracks, porosity, and other defects on the surface of the coating. The higher-magnification graph in Figure 4(d) reveals that the size of the nodules is approximately 700 nm and there is a good bonding among these nodules.

In order to analyze the thickness of the coating, the cold mounting method was used in the experiment. Figure 5 is the cross-sectional SEM photograph of the coated SiC particle. It can be seen from Figures 5(a) and 5(b) that the coating exhibits a uniform layer with a thickness of approximately 3.5 μm.

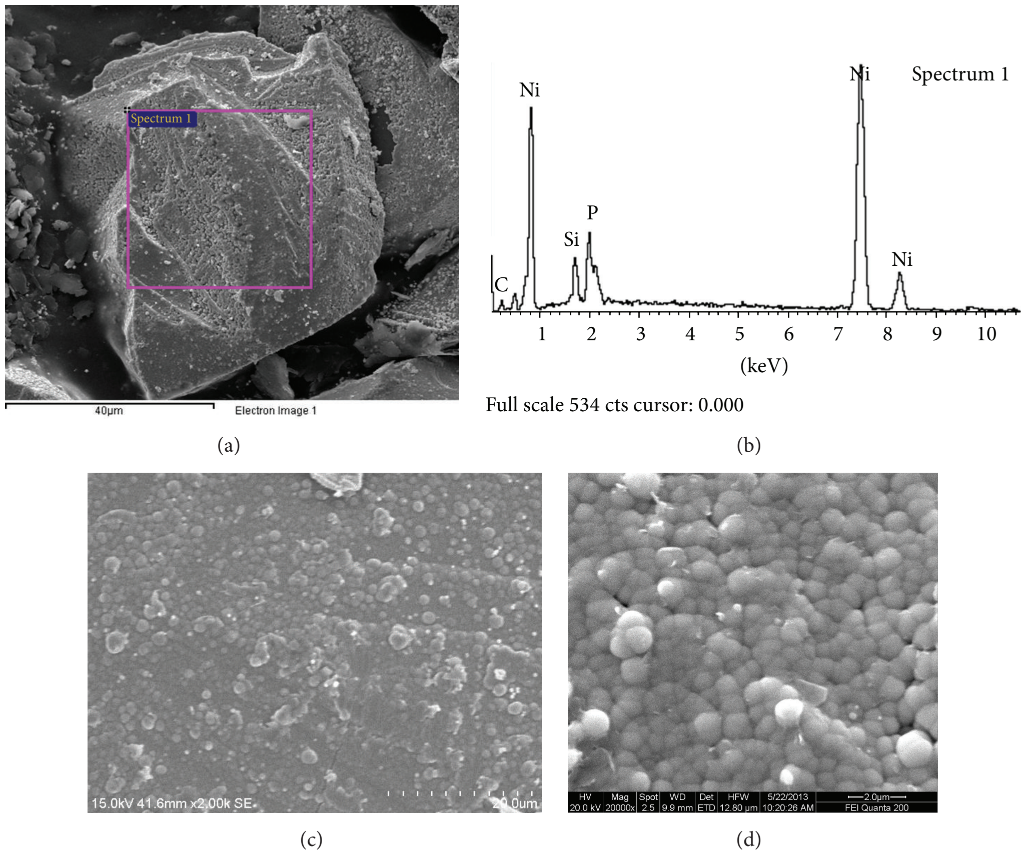

Figure 4: SEM morphologies and EDS of the plating: ((a), (c), (d)) SEM, (b) EDS.

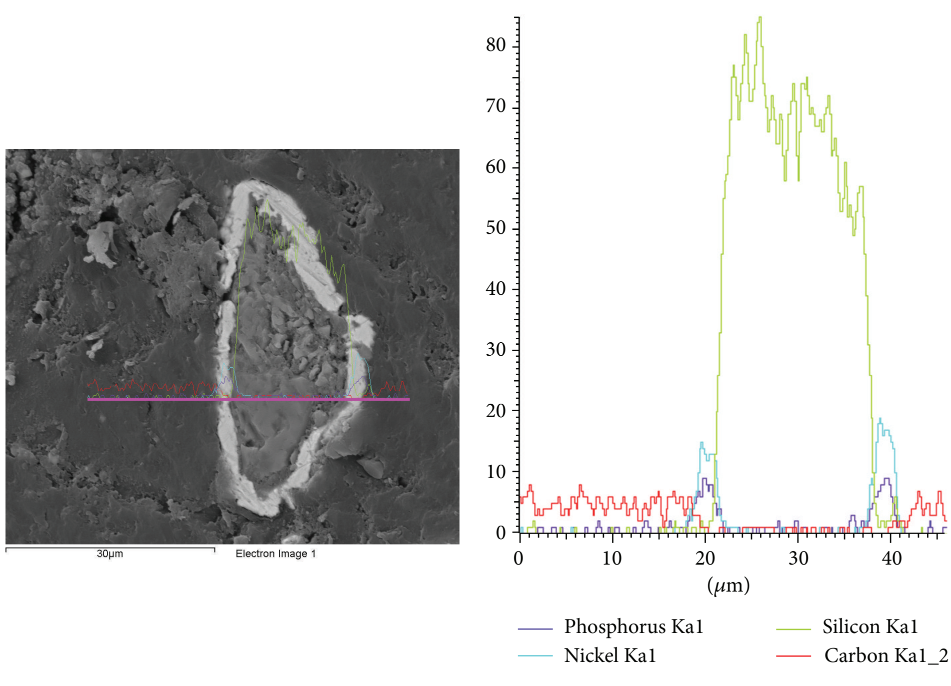

Figure 5: SEM morphology and EDS spectrum of the cross section of plating: (a) SEM, (b) EDS.

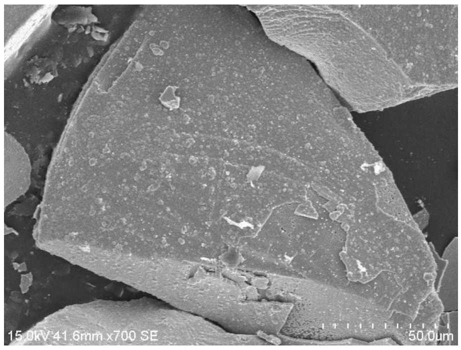

FIGURE 6: Micrographs of coated SiC particles after thermal shock.

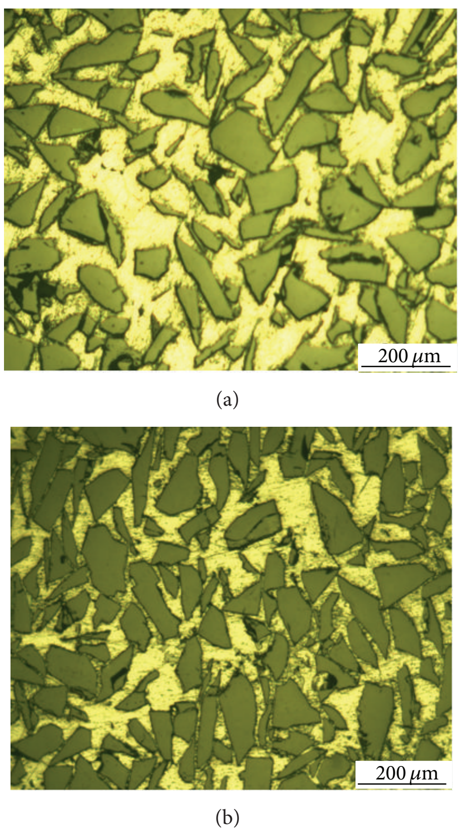

FIGURE 7: Optical microstructures of the composites: (a) without, (b) with Ni plating.

To evaluate the adhesive strength between the coating and SiC_p, thermal shock was applied to the experiment, and the detailed process was as follows. The coated particles were first heated to 400°C and held for 1 h and then quenched in water. Figure 6 describes the micrograph of the coated particles after thermal shock, and it can be seen from Figure 6 that the overall binding between the coating and the particles is close, although the coating has a small partial loss.

3.2. Microstructural Analysis of the Composites Containing Ni Coatings. Figure 7 displays the optical microstructures of the composites without and with the Ni coating. It can be seen that there are a small number of holes at the boundary between the particles and the aluminum matrix in Figure 7(a), while there are an even distribution of particles and no obvious segregation and holes in the composites in Figure 7(b).

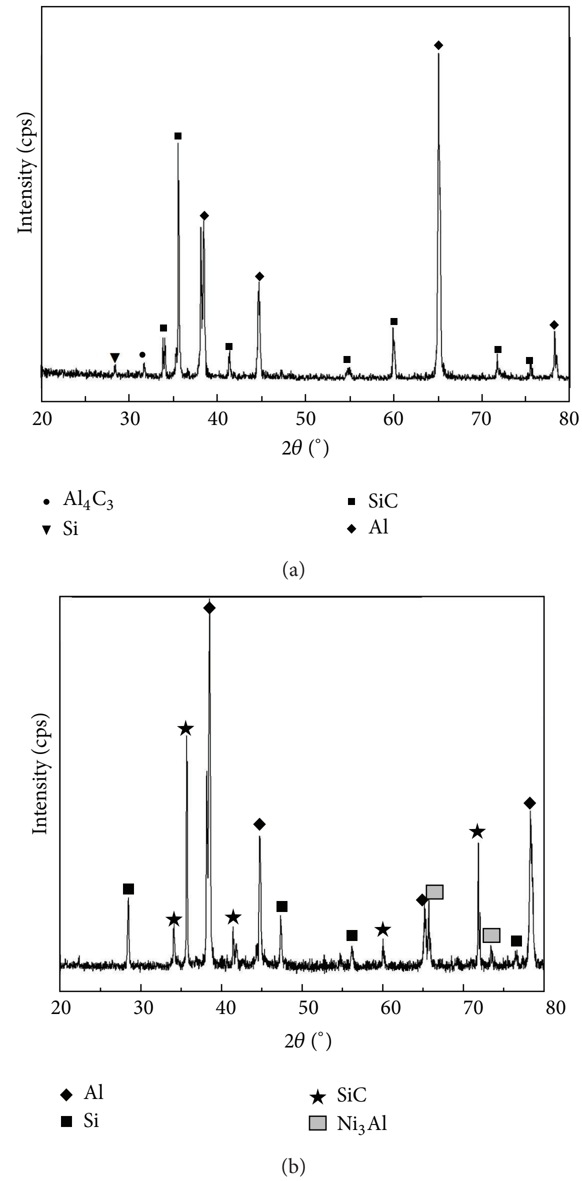

FIGURE 8: XRD patterns of the composites: (a) without, (b) with Ni plating.

Figure 8 shows the XRD spectra of the composite with and without Ni plating, respectively. XRD examination in Figure 8(a) displays that Al_4C_3 is present in the composite without the Ni layer, while Al_4C_3 is not found in the composite with the Ni layer in Figure 8(b). In addition, it can be noted that the XRD pattern of the composite brings out new Ni_3Al intermetallics in Figure 8(b). This is because when SiC_p is coated, the coating acts as a barrier between molten aluminum and the ceramic, resulting in a decreased activity of Al at the interface, and retards the formation of carbide.

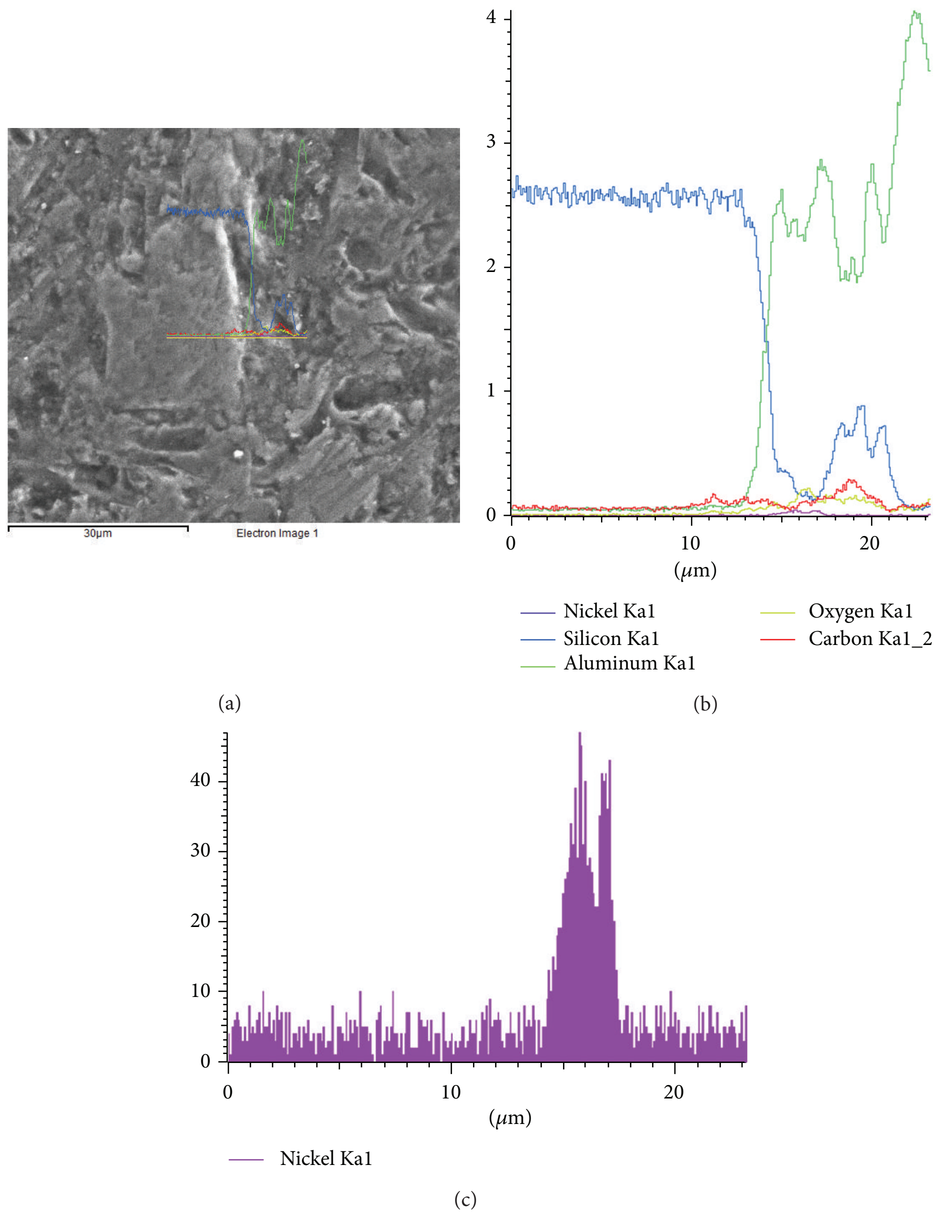

Figure 9: SEM morphology and EDS spectrum of the composites with Ni plating: (a) SEM, ((b), (c)) EDS.

This is in agreement with the results, as suggested by Ip et al. [17].

Figure 9 reveals the SEM morphologies and the EDS analysis of the composite containing the Ni-plated layer. It can be observed from Figure 9(a) that the coated SiC_p combines well with the Al matrix at the interface. Furthermore, only Si, C, O, Al, and Ni elements can be found in the EDS, as shown in Figure 9(b), and there are no new elements, which may indicate that the Ni layer is indeed acting as a certain barrier to aluminum. It can be observed in Figure 9(c) that there does exist a certain thickness (about 3.2 μm) of the intermediate Ni element layer at the interface of the composites.

3.3. Effect of Ni Coatings on Thermal Conductivity. Figure 10 depicts a variation of the equivalent thermal conductivity of the composites with and without the Ni-plated layer as well as a comparison of the values obtained from the rule of mixing (ROM) model and FEM analysis, respectively.

From the results presented in Figure 10, it can be noted that the equivalent thermal conductivity of the composites containing the Ni layer is slightly higher than the composites without the Ni layer. The experimental values of thermal conductivity of the composites with the Ni layer are closer to the results obtained from the ROM model and FEM analysis, while the value of the composites without the Ni layer is visibly lower than that of the simulated and calculated value.

It is known that the thermal conductivity of Ni itself is about 90 W/m·K and is lower than that of SiC_p (147 W/m·K) and aluminum (180 W/m·K) in this experiment. If only the thermal conductivity of the Ni layer is considered based on

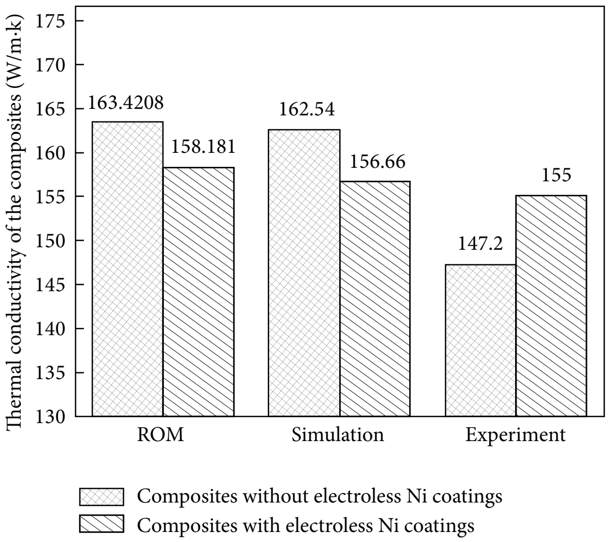

FIGURE 10: Comparison of the thermal conductivity of the composites with and without Ni plating obtained from different methods.

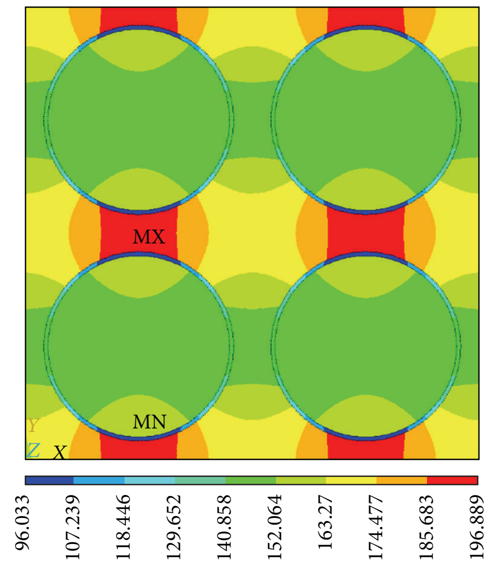

FIGURE 11: Heat flux distribution of the composite with 3.5 μm Ni plating.

ROM, with the addition of Ni in the composites, the thermal conductivity of the composite should have been lower than that of the composite without the Ni layer. However, the value is higher. The main reason for this phenomenon is that when the surface of the particles is being plated with Ni, the wetting contact angle between the particles and aluminum is decreased, as reported by Leon and Drew. The contact angle is only 12.2° for Ni-plated SiC_p and aluminum [18], which can improve the wettability and bond performance between the reinforcement and the matrix. On the other hand, when a coated Ni layer is surrounded by SiC particles, it can effectively retard the thermal decomposition temperature and enhance the thermal stability of the SiC particles [19], which results in inhibiting or avoiding harmful reaction products. This can be confirmed from the previous results, as shown in Figures 8(b) and 9. Based on these two reasons above, it will be beneficial to reduce the interface thermal resistance and provide a more continuous heat conduction path between the particles and the matrix.

In order to further evaluate the influence of the nickel layer on the thermal conductivity of the composites, it is assumed that the Ni layer was considered as an ideal interlayer that bonded well with the particles and the matrix. The heat flux of the composites with Ni coating obtained from FEM analysis is exhibited in Figure 11. It can be seen from the distribution chart that there are two kinds of colors within the SiC particles. The value of the heat flux density in the middle area of the particles varies from 140.858 to 152.064, which is very close to the eigenthermal conductivity of SiC_p (147 W/m·K), while the value in the minority area on either side of SiC_p is in the range of 152.064–163.27, which is a little higher than 147. From the above results, it is clear that the effect of the Ni layer acting as a thermal-barrier layer in reducing thermal conductivity is relatively minor and is far lower than the reduction resulting from the above-mentioned poor interface bonding. In other words, even though Ni itself has a lower thermal conductivity, it only slightly reduces the thermal conductivity of the composite. Meanwhile, Ni can improve the adhesion between the particles and the matrix, which enhances the thermal conductivity. Thus, the thermal conductivity of the composite with the Ni layer is higher than that of the composite without the Ni layer.

4. Conclusion

Ni coating on the surface of SiC_p was performed by electroless nickel deposition. A continuous and uniform coating with a certain thickness (around 3.5 μm) can be attained by optimal electroless parameters ($NiSO_4 \cdot 6H_2O$ 7.5 g/L, pH 4.8, and bath temperature 80°C). The final coating is constituted with irregular nodules in the approximate size of 700 nm. The composites with and without the Ni layer were successfully prepared by pressureless infiltration. With the addition of Ni as a coating, there are some intermetallics Ni_3Al but no interfacial carbide Al_4C_3 in the composites. The experiments and simulations both show that even though Ni itself has lower thermal conductivity than that of SiC and Al, the addition of the Ni layer can benefit the improvement of wettability between the reinforcement and the matrix. Addition of the Ni layer does not substantially decrease the overall thermal conductivity of the composite.

Conflict of Interests

The authors declare that they have no conflict of interests regarding the publication of this work.

Acknowledgments

The paper is funded by Natural Science Foundation of China (Grant no. 51166011), Space Foundation (Grant no.

CASC201106), Aviation Science Foundation (Grant no. 2012ZF56024), and Key Laboratory for Microstructural Control of Metallic Materials of Jiangxi Province (Nanchang Hangkong University) (no. JW201423003).

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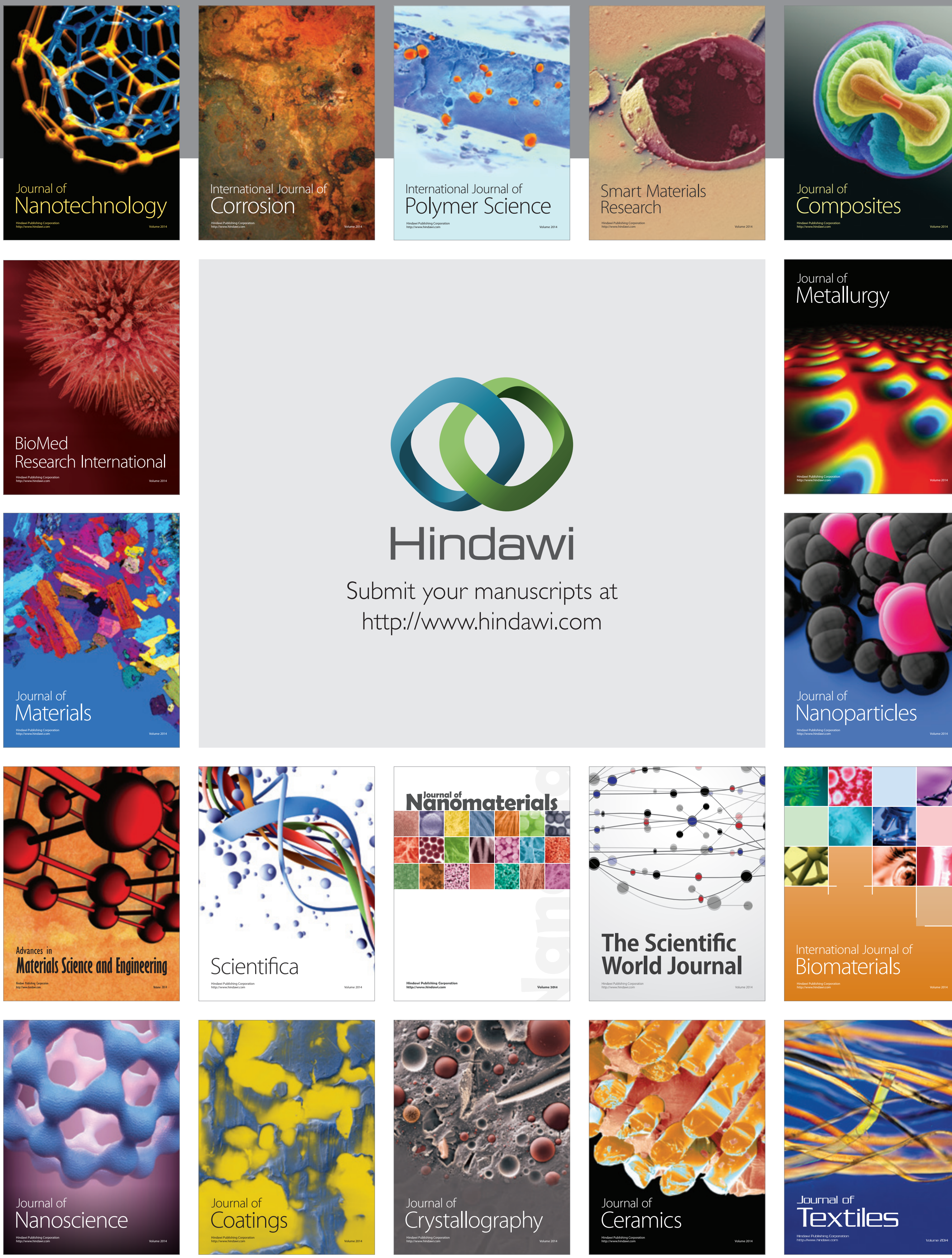

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