The influence of electroless nickel coated SiC on the interface strength and microhardness of Aluminium composites

V. Umasankar1, S. Karthikeyan2*, M. Anthony Xavior1
1Manufacturing Division, School of Mech. and Building Sciences, VIT University, Vellore, India.
2Surface Engineering Research Laboratory, CNBT, VIT University, Vellore, India.

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*Corresponding author: skarthikeyanphd@yahoo.co.in

Abstract
An attempt has been made to improve the interfacial strength of AlSiC composites which are widely used in defense and automotive sectors. Enhancement of interfacial strength is studied by radial crushing of composites reinforced with electroless nickel coated SiC particles and manufactured by powder metallurgy process. It was visualized that ultimate breaking load for electroless Nickel coated SiC reinforcement has increased by 40% with respect to uncoated reinforcement. XRD data confirmed the existence of Nickel in Aluminium matrix. SEM studies revealed the incorporation of Nickel with Aluminium and Silicon carbide. Because of improved interfacial strength and microhardness values the so-obtained composites can be used for heavy machineries as an alternate to monolithic alloys.

Keywords: Composites, Coatings, Interface, reinforcement

1. Introduction
Metal matrix composites are widely used in aerospace and automotive industries owing to their outstanding specific stiffness, modulus and tensile strength coupled with abrasion resistance. Pazman and his co-workers1 investigated that the formation intermetallic phases between Nickel and Al-SiC composites and the sintering temperature of 580°C favored the diffusion of Nickel into matrix. Burak Dikici1 et.al.,2 have studied the influence of Nickel on the corrosion resistance of Al-SiC composite prepared by liquid processing. Nickel coated composites have not shown significant improvement in corrosion resistance of the composite in neutral medium. According to Liang-Guang Chen and Su-Jien Lin3, the Nickel coated SiC exhibited low interfacial strength values for Fiber reinforced 7075 Aluminium composites. L.B. Li et.al.4, studied that electroless nickel coated Silicon carbide/Aluminium composites in alkaline medium increased the microhardness and adhesion. They have not claimed corrosion resistance in alkaline bath. However electroless coated reinforcement enhanced the adhesion and diffusibility between heavier particles, there is lack of information available on the improvement of interfacial strength and microhardness by coating process. Due to these facts an attempt has been made to obtain metalized reinforced composites that can be used for heavy machineries as an alternate to monolithic alloys.

As far as we know no concrete investigation has been made for obtaining electroless coated Aluminium/SiC composites with superior interfacial strength and microhardness values. Due to the poor bath stability and also lack of adhesion of Nickel on composites, a special attention is to be paid to enhance the mechanical strength of composites that can be used in machineries. At present the powder processed aluminium composites are not much used on account of its inferior interface strength especially with higher percentage of reinforcement. It is well established that higher is the percentage of reinforcement lesser is the interparticle distance which resulted in inadequate sintering. Also poor bond strength promotes void propagation and increased defect density at elevated sintering temperatures.

2. Experimental section
2.1. Materials
AA 6061 matrix used in the present study composed of Fe = 0.19%, Cu = 0.27%, Si = 0.56%, Zn = 0.03%, Mg = 0.94% and Al remainder. The interfacial strength of the uncoated and coated composite were measured by
Instron 8081 (USA) adapting ASTM 939-09 procedure with 1mm/min strain rate. The electroless bath employed for the present study consisted of Nickel methane sulphonate = 25 g/l; sodium hypophosphate = 22g/l; tri ammonium citrate =45g/l; acetyl thio uria = 0.01 mg/l; proprietary surfactant = 0.01 g/l; pH =5.2; temperature =88ºC; mechanical agitation was provided at 240 rpm using Remi mechanical stirrer, India. The plating time was 30 minutes. Before plating, the reinforcement was subjected to pretreatment such as degreasing with acetone, acid pickling in 10% HCl at room temperature, sensitization in SnCl₂ solution and then activation of powders in palladium chloride solution. Subsequently washed with water and dried at 50ºC in furnace for 30 minutes. Then the particles were introduced in to electroless plating bath.

The percentage of phosphorus is 8.2% and nickel is 91.8%. The determination of phosphorus in the deposits involve the complete precipitation of phosphorus as ammonium phosphomolybdate, dissolving the coated SiC powders in HNO₃ and excess HNO₃ was estimated by back titration.

2.2. Methods
Before electroless plating the SiC powders were degreased with isopropyl alcohol, washed with water, sensitized with stannus chloride and then activated as described earlier. Nickel coated and uncoated SiC mixtures were compacted at 450 and 550 MPa in universal testing machine of 60 tons capacity uniaxially and then sintered at 400⁰ and 500⁰C for about one to three hours, for 5% to 15% SiC. In order to avoid the formation of protective Al₂O₃, nitrogen gas was used during sintering. The formation of intermetallic phases and surface morphology was ascertained using XRD data and SEM EDAX analysis.

3. Results and discussion
3.1. Surface morphological studies for metalized SiC incorporated Aluminium
3.1.1. XRD Analysis
This study revealed that the appearance of sharp peaks at 2Ø =39.3, 65.9 and 75.05 correspond to the existence of Nickel (Figure 1 sintering at 400⁰C) in the composite. Acute peaks appeared at diffraction angle of 45.5 and 42.2 are the characteristics of Al₂Ni and Al₂Cu as reported by J. Pázmán et al. The smaller peaks at 2Ø values 72.68 and 83.2 is the characteristic value for Ni₃P and a very feeble peaks appeared at 2Ø = 34.8, 75.77 and 103.08 indicated the presence of SiC. The peaks formed at 2Ø values of 60.02, 83.22, 97 and 99.78 confirmed the formation of Ni₂P₃. These results are in good agreement with reports published earlier.

Figure 1(a) XRD results obtained for composites sintered at 500⁰C
Figure 1.b indicated XRD results obtained for composites sintered at 500°C. The diffraction angle appeared at 45.67, 65.9, 78.95 are the significant peaks for Nickel where as those obtained at 39.47, 60.76 and 80.02 indicated the formation of Ni$_3$P. The peaks value for SiC observed at 35.58, 73.82, 95.5, 102.9. The smaller peaks appeared 60.76, 83.1, 96.59 and 99.69 confirming the formation Intermetallic phases like Ni$_3$P$_5$. The above x-ray diffractograms resemble each other in particular for Ni$_3$P and Ni$_3$P$_5$.

This could be due to the formation of precipitation of aluminium, copper, silicon and phosphorus in nickel moiety which is accounted for enhanced interfacial strength and microhardness for metalized SiC reinforced in Aluminium matrix. The XRD results observed at 600°C indicated that the crystallinity formation of composites such as Al$_2$Cu, AlNi$_3$, Ni$_3$P, Ni$_3$P$_5$ and SiC dominant Ni peaks (Fig.1c). Also it could be noticed that many peaks got extinguished due the over crowding of Ni-P in SiC which resulted sharp peaks. Because of this, the composite has failed in its performance.

![Fig. 1.(b)](image1)

![Fig. 1. (c)](image2)

XRD for sintered composites reinforced with Ni coated SiC
3.1.2. SEM studies

SEM images for metalized SiC incorporated Aluminium composites are given in figure 2.a and 2.b. Aluminium alloy particle to particle bondability is minimized (portrayed at x, 5000) due to the formation of oxide layer on aluminium.[9]. However the formation of metalized Nickel layer on SiC is visible as white shadowed structure. In the case of figure 3.b., the coverage of aluminium powders by metalized SiC is non uniform. This may be due to the emanation of aluminium starts at 500°C by sintering.

![Fig. 2 (a)](image)

![Fig. 2 (b)](image)

**SEM images of Ni coated Al-SiC composites**

3.1.3. EDAX analysis

EDAX analysis (Figure 2.c) gave an idea about the existence of Nickel in the Al-SiC matrix. The spectrum contained Al (26.53 wt %), Na (0.63 wt %), Si (5.5 wt %) and O (40.75 wt %) in definite proportions. The co-deposition of Nickel layer by electroless process on SiC favored the diffusibility of Al and SiC. The weight percentage of Ni was found as 0.53. The weight percentage of oxygen is greater than the other elements establishing that aluminium formed stable oxide layer which is not vulnerable to corrosion of composites and thus lessening the interfacial bonding. The absence of other metals like Mg and Cu indicated that the spectra are concerning the formation intermetallic phases at sintering temperature only.

![Fig. 2 (c) EDAX spectra taken for Ni coated Al/SiC composite](image)
3.2 Influence of EN coated SiC on the interfacial strength of the composite

Table 1 gives the ultimate breaking load of the hollow cylindrical specimen measured for understanding the interfacial strength of the coated and uncoated SiC reinforced Aluminium composites. At 10% by weight reinforcement the possibility of SiC and SiC particles distance reduces and due to the lower sintering temperatures with respect to the melting temperature of SiC bonding is non existent and resulting in week interface areas which have detrimental effect on the strength of the composite. With Nickel incorporation on SiC it facilitates better interface bonding and preventing harder ceramic particles connecting each other through formation Nickel Silicide phases as described earlier. Radial compression testing is advocated and predominantly used by the PM industry to evaluate the sintering performance of PM compacts and hence used at present for assessing the influence of Nickel coating. The improvement in strength may also be attributed to reduction in porosity and defects during sintering. In the present investigation the breaking load increased by 40% with respect to uncoated composites due to establishment of non contacts between the SiC to SiC by Nickel layer which helps interdiffusion and intermetallic phase formation of composites at 500 °C as evidenced from the results XRD. This sintering temperature was reported as nearer to the eutectic temperature of Al-SiC (524°C). Increase in strength can also be attributed to prevention of Al₄C₃ formation during sintering due to presence of Nickel layer on the hard SiC particles.

Table 1. Performance of coated Vs uncoated composites under radial crushing

<table>
<thead>
<tr>
<th>Wt.% of SiC</th>
<th>Compacting Pressure MPa</th>
<th>Sintering Temperature °C</th>
<th>Ultimate Breaking Load Newtons</th>
<th>% Increase</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Uncoated</td>
<td>Coated</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>450</td>
<td>500</td>
<td>1814.65</td>
<td>21</td>
</tr>
<tr>
<td>10</td>
<td>550</td>
<td>400</td>
<td>1545</td>
<td>19</td>
</tr>
<tr>
<td>15</td>
<td>550</td>
<td>500</td>
<td>1428.36</td>
<td>41</td>
</tr>
</tbody>
</table>

3.3 Microhardness measurements

The results of Vickers microhardness measurements are given in Table 2. The hardness value is improved for coated SiC due to the formation of intermetallic phases at 500°C. The higher hardness is obtained by the precipitation hardening reaction between Nickel and phosphorus embedded in SiC, Al and Cu lattices. The combined effect of hard SiC and precipitation hardening by metallic atoms could enhance the hardness of the resultant composite to a greater extent.

Table 2. Assessment of Microhardness of coated and uncoated reinforcement composite

<table>
<thead>
<tr>
<th>Wt.% of SiC</th>
<th>Compacting Pressure MPa</th>
<th>Sintering Temperature °C</th>
<th>Microrhardness HV₀.₅</th>
<th>% Increase</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Uncoated</td>
<td>Coated</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>450</td>
<td>500</td>
<td>55.1</td>
<td>72.7</td>
</tr>
<tr>
<td>10</td>
<td>550</td>
<td>400</td>
<td>65.3</td>
<td>203.8</td>
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<tr>
<td>15</td>
<td>550</td>
<td>500</td>
<td>72.2</td>
<td>191.7</td>
</tr>
</tbody>
</table>

Conclusion

The improvement of interfacial strength was observed for coated SiC in aluminium matrix. The coating enhanced diffusion of SiC –Al particles. Because of improved interfacial strength and microhardness values the so obtained composites can be used for heavy machineries as an alternate monolithic alloys. XRD and SEM studies validated the performance of composites prepared through a coating process.

References


(2014); [http://www.jmaterenvironsci.com](http://www.jmaterenvironsci.com)