Plasticity and microstructure of A356 matrix nano composites

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Received 5 July 2011; accepted 29 November 2011
Available online 6 December 2011

Abstract In this investigation, the effect of the volume fraction of the nano-SiC particles on the mechanical properties of the Al–Si matrix composites was studied. The yield strength and tensile strength increase, but the elongation decreases with the increase in the volume fraction of the SiC particles, indicating that increasing the volume fraction of the SiC particles can improve the strength but degrade the plasticity of the composites. For nano-SiC particle reinforced aluminum metal matrix composites, the SiC particle is the main strengthening factor. The increase in the yield strength by the introduction of the SiC particles is caused by the external applied stress transferred from the Al matrix to the nano-SiC particles. The higher the volume fraction of the SiC particles, the more apparent the grain refinement and dislocations' interaction. Microstructure evaluation revealed a uniform distribution of nano-particles, grain refinement of aluminum matrix, and presence of the minimal porosity.

1. Introduction

Aluminum alloys are promising materials in high technology fields owing to their excellent specific mechanical properties (Almajid, 2011). However, they suffer from poor elevated temperature and tribological properties. Aluminum matrix composites, in which hard ceramic particles are dispersed in a relatively ductile Al matrix, have widespread applications in the areas of ground transportation (auto and rail), thermal management, aerospace, industrial, recreational and infrastructure industries owing to functional properties that include high structural efficiency, excellent wear resistance, and attractive thermal and electrical characteristics (Shabani and Mazahery, 2011a; Zhao et al., 2008; Hassan and Gupta, 2005a). While in composites reinforced with continuous fibers, strengthening is associated with load transfer from the matrix to the fiber, it is associated with the high dislocation density in the matrix of composites reinforced with whisker and particulate (Ferkel and Mordike, 2001; Groza, 1999; Akio et al., 1999; Lan et al., 2004a; Shabani et al., 2012).

The size of particulate reinforcements in AMC generally ranges from a few micrometers to several hundred micrometers (Ma et al., 1999). Microsize Ceramic powders and fibers were widely used in the fabrication of Al-based composites to improve the ultimate tensile and the yield strengths of the metal.
However, the ductility of the MMCs deteriorates significantly with high ceramic particle concentration. A decrease of the reinforcement particle size from micrometric to nanometric scale brings a superior increase in the mechanical strength of the composite, but the tendency of particle clustering and agglomeration also increases (Fogagnolo and Robert, 2006). It is important to note that a homogeneous distribution of the reinforcing particles is essential for achieving the improved properties (Khakbiz et al., 2009; Lu and Lai, 1998).

It will be attractive to produce as-cast lightweight bulk components of MMNCs with uniform reinforcement distribution and structural integrity. It is expected that the strength of aluminum reinforced by ceramic nano-particles, would be enhanced considerably, while the ductility of the aluminum matrix is retained. However, it is extremely difficult to obtain uniform dispersion of nano-sized ceramic particles in liquid metals due to high viscosity, poor wettability in the metal matrix, and a large surface-to-volume ratio (Lan et al., 2004a,b). The production methods of MMCs can be categorized into a number of types including compocasting, thixoforging (Naher et al., 2005), in situ technique (Almajid, 2011; Zhao et al., 2008), disintegrated melt deposition (Hassan and Gupta, 2005a) mechanical alloying, diffusion bonding, powder metallurgy (Ferkel and Mordike, 2001; Groza, 1999), vortex process (Akio et al., 1999; Mazahery and Shabani, 2011b), ultrasonic method (Lan et al., 2004a,b).

The powder metallurgy, mechanical alloying and diffusion bonding generally involve the fabrication of particulate-reinforced MMCs from blended elemental powders by a number of steps prior to final consolidation (Rosso, 2006). It allows essentially a wide range of materials to be used as the matrix and reinforcement (Lloyd, 1994). Also, separation effects and intermetallic phase formations are less for these processes (Zebarjad and Sajjadi, 2007). However, in this case manufacturing route is relatively complex, lengthy, expensive, and energy consuming (Lloyd, 1994).

It is reported that the addition of nano-particles has brought a considerable increase in the hardness of all composites compared to the matrix alloy and 3 vol.% micrometric particles resulted in a hardness level of the composite with 1 vol.% nanometric particles (Sameezadeh et al., 2011). This increase is attributed to hard particulate reinforcements which act as a barrier to the dislocation movement within the matrix and exhibit greater resistance to indentation of the hardness tester (Abdizadeh and Baharvandi, 2009; Mazahery and Shabani, 2011c).

Compared to other routes, melt stirring process that utilizes mechanical stirring has some important advantages, e.g., the wide selection of materials, better matrix–particle bonding, easier control of matrix structure, simple and inexpensive processing, flexibility and applicability to large quantity production and excellent productivity for near-net shaped components (Kok, 2005).

When the reinforcement particles are added into the molten matrix, they float on the melt surface. This is due to the surface tension, very large specific surface area and high interfacial energy of reinforcement particles, presence of oxide films on the melt surface and presence of a gas layer on the ceramic particle surface. Mechanical stirring can usually be applied in order to mix the particles into the melt, but when stirring stops, the particles tend to return to the surface.

Stir casting is a widely used technique of producing Al matrix composites that are reinforced by microceramic particles (Mazahery and Shabani, 2011c). However, there are some problems associated with stir casting of AMCs such as: poor wettability and heterogeneous distribution of the reinforcement material. When the particles are wetted in the metal melt, they will tend to sink or float to the molten melt due to the density differences between the reinforcement particles and the matrix alloy melt, so that the dispersion of the ceramic particles is not uniform and the particles have high tendency for agglomeration and clustering (Hashim et al., 2001).

It is extremely challenging for the conventional mechanical stirring method to distribute and disperse nano-scale particles uniformly in metal melts because of the poor wettability and higher specific surface areas of nano-particles which lead to agglomeration and clustering. Moreover, several structural defects such as porosity, particle clusters, oxide inclusions and interfacial reactions arise from the unsatisfactory casting technology (Zhou and Xu, 1997).

In the present article, a mixture of nano-SiC and aluminum particles was used as the reinforcement in order to improve the incorporation of the particle into the A356 matrix. Then the experimental study was carried out on the mechanical properties of nano-SiC reinforced A356 matrix composites.

2. Experiments

The experimental Al/SiC composites were produced from A356 aluminum alloy and nano-SiC powders by Vortex technique. The chemical composition of A356 alloy was analyzed as: 7.5 Si, 0.38 Mg, 0.02 Zn, 0.001 Cu, 0.106 Fe and balanced Al in weight percents. The liquidus temperature of this alloy quantified by thermal analyzing technique is 615 °C. This alloy has been selected because of its good fluidity as well as the presence of silicon and magnesium. Since the silicon content of A356 alloy is sufficiently high, it can be maintained in the liquid state at typical casting temperatures for certain periods of time without giving rise to extensive formation of Al4C3.

A mixture of nano-SiC and aluminum particles with respectively average particle size of 50 nm and 16 μm was used as the reinforcement (Al/SiC = 1.67) and ball milled in isopropyl alcohol for 20 min. The mixture was then dried in a rotary vacuum evaporator and passed through a 60 mesh screen. The powder mixtures were cold pressed under 200 MPa into samples having 60 × 60 × 60 mm3 dimension. The compacted samples were crushed and then passed through 60 mesh screen. The presence of Mg in this alloy is thought to improve the wetting of the reinforcement by the liquid alloy. One weight percent of magnesium additive in powder form was used as a wetting agent. The purposes of Mg additions are manifold including enhancement of the wetting behavior with the SiC particles, the formation of spinel (MgAl2O4) or MgO at the interface of oxidized SiC, thus protecting the SiC particles from reaction with Al, enhancement of the interfacial bonding and strengthening of the Al matrix (by solid solution hardening).

The Vortex technique involved melting weighted amounts of the alloy in a clay-bonded graphite crucible using an electrical resistance furnace. Then the temperature of the melt was raised to about 750 °C (above the alloy liquidus temperature) and specific quantities of nano-SiC particles (0.5, 1.5, 2.5, 3.5 and 4.5) were added to the melt. The powder mixture was in-
serted into an aluminum foil by forming a packet. The packets were added into molten metal of crucible when the vortex was formed. The packet of mixture melted and the particles started to distribute around the alloy sample. The melt was subsequently stirred at 600 rev/min using a graphite impeller attached to a variable speed motor. The temperature of the furnace was kept constant at 750 °C for 15 min while stirring was continued at the same speed. There is a nitrogen supply to the crucible in order to minimize the oxidation of molten aluminum.

The microstructure was investigated by optical microscopy (Prior N334), scanning electron microscopy (SEM, Zeiss DSM 962, 20 kV, EDX) and transmission electron microscopy (TEM, Philips CM20T, 200 kV, EDX).

The specimens were prepared by grinding through 120, 400, 600, and 800 grit papers followed by polishing with 6 µm diamond paste, and etched with Keller’s reagent (2 ml HF (48%), 3 ml HCl (conc.), 5 ml HNO₃ (conc.) and 190 ml water). TEM specimens were machined to 0.5 mm thickness and cut using a wire electro discharge machine. The samples were then ground down (350–1200 grit) and perforated using double spew with methanol solution.

The amount of porosity in the cast alloy and the composite was determined by comparing the measured density with that of their theoretical density. Hardness measurements were carried out on a Brinell hardness testing machine (Eseway DVRBM), using an indenter ball with 2.5 mm diameter at a load of 31.25 kg, and the mean values of at least five measurements conducted on different areas of each sample were considered in order to eliminate possible segregation effects and get a representative value of the matrix material hardness.

The tension tests were conducted on tensile test machine (Instron 1195) in order to assess the mechanical behavior of the composites. The tensile specimens were machined from composite rods according to ASTM.B 557 standard. For each volume fraction of SiC particles, five samples were tested. In order to study the effect of nano-particles on the fracture mechanisms during tensile loading of the samples, fractography was performed on the fractured surfaces of composite specimens. The fracture surfaces were analyzed by SEM.

3. Results and discussions

At present, particulate-reinforced composites are being produced by different methods, such as stir casting, powder metallurgy, and spray deposition technique. Among these methods, stir casting is considered to be easily adaptable and economically viable due to its low processing cost and high production rate. An additional benefit of this process is the near-net shape formation of the composites. In comparison with Al matrix microcomposites, the research on Al matrix nano-composites are still limited. The key reason is perhaps related to the difficulty in synthesizing Al matrix nano-composites due to the higher agglomeration and clustering of particles.

A number of reports are available on Al alloys + SiC composites. However, the effects of nano-SiC particles on the microstructure and mechanical properties are meager. Therefore, the objective of the present study is to develop a stir casting process to produce nano-SiC reinforced cast Al alloy matrix composites, and to investigate their microstructure and mechanical properties. The microstructure and mechanical properties of the as-cast composite materials are compared with unreinforced materials.
The typical optical and SEM micrographs of composite samples are presented in Fig. 1. The microstructure of the A356 matrix composites contains primary $\alpha$-Al dendrites and eutectic silicon, while particles are separated at inter-dendritic regions and in eutectic silicon. The dendritic microstructure of the Al matrix is the result of casting process. During solidification of composites, because of lower thermal conductivity and heat diffusivity of second phase (particles) in comparison with the metal matrix, particles are cooled down more slowly than the melt and so is the equilibrium concentration of C with both SiC and the reaction product $\text{Al}_4\text{C}_3$. For a given temperature, the formation of $\text{Al}_4\text{C}_3$ will go ahead to keep the carbon content at its thermodynamic equilibrium concentration, until the Si equilibrium concentration in the Al matrix has been reached. Thermodynamic and kinetic conditions in favor of $\text{Al}_4\text{C}_3$ formation are provided during the long period of time that constituents are in the liquid Al phase. Mg is quite a common alloying element in Al/SiC composites, which has an indirect effect on the formation of $\text{Al}_4\text{C}_3$. Mg acts solely as solid solution hardening element; at contents below 4 wt.% it does not form any intermetallic phase with Al. Mg is prone to react with SiC and its surface oxide $\text{SiO}_2$, if present and, thus influencing the interfacial bonding strength. The addition of Mg is expected to increase the Al matrix strength and to enhance interfacial bonding between Al and SiC.

During processing of Al matrix reinforced with SiC particles, SiC usually dissolves in molten Al to form atomic C and Si. As dissolution proceeds, the concentration (i.e. the activity) of both C and Si increases in the Al matrix. However, the solubility of C in liquid Al is very low and so is the equilibrium concentration of C with both SiC and the reaction product $\text{Al}_4\text{C}_3$. For a given temperature, the formation of $\text{Al}_4\text{C}_3$ will go ahead to keep the carbon content at its thermodynamic equilibrium concentration, until the Si equilibrium concentration in the Al matrix has been reached. Thermodynamic and kinetic conditions in favor of $\text{Al}_4\text{C}_3$ formation are provided during the long period of time that constituents are in the liquid Al phase. Mg is quite a common alloying element in Al/SiC composites, which has an indirect effect on the formation of $\text{Al}_4\text{C}_3$. Mg acts solely as solid solution hardening element; at contents below 4 wt.% it does not form any intermetallic phase with Al. Mg is prone to react with SiC and its surface oxide $\text{SiO}_2$, if present and, thus influencing the interfacial bonding strength. The addition of Mg is expected to increase the Al matrix strength and to enhance interfacial bonding between Al and SiC.

Comparison of the measured density of the cast alloy and the composites with that of their theoretical density determined the amount of porosity. In this calculation, density of SiC was taken to be 3.2 g cm$^{-3}$. The densities of matrix alloy as well as composites were measured, using the Archimedes principle, to quantify the volume fraction of porosity. Fig. 5 shows the variation of porosity with the volume fraction of nano-SiC particles. Higher degree of defects and
Micro-porosity is observed at higher SiC content which is the result of increase in the amount of interface area (Suresh et al., 2003; Chang and Kamio, 1998; Shabani and Mazahery, 2011b). 

Hardness tests were performed using a Brinell hardness machine. In order to obtain the average values of hardness, areas predominant in the soft matrix or the hard reinforcing phase should be avoided so that the average values of hardness are attained from these measurements. The variation in hardness with volume fraction for Al/nano-SiC composites is summarized in Fig. 6.

It has been observed that the hardness of composites is invariably higher than the monolithic alloys. This effect is attributed to the presence of hard SiC particles, which aid to the load bearing capacity of the material and also restricts the matrix deformation by constraining dislocation movement (Mazahery and Shabani, 2011c). The hardness increment can also be attributed to reduced grain size. As shown, hardness increases with the amount of SiC present particles. It is believed that since SiC particles are harder than aluminum alloy, their inherent property of hardness is rendered to the soft matrix (Cooke, 1991; Mondal et al., 2006).

There is a difference between the hardness of micro- and nano-composite which is attributed to larger amount of dislocations generated due to fine size particles. The geometrically necessary dislocations increase for fine size particles compared to that for larger ones. This can be explained as at a constant volume fraction of reinforcement, by considering the interparticle distance that decreases with decrease in particle size. This results in more inhibition sites for the movement of dislocations leading to increased hardness value. However, the result indicates that the matrix hardness of Al based composite is remarkably high compared to monolithic Al. The higher dislocation density is due to the significant difference in coefficient of thermal expansion of pure A356 and A356-alloy with SiC particulates (Mazahery and Shabani, 2011c). This indicates that the increased dislocation density in composite would have caused the increase in the hardness. Other possibility is the encounter of hard SiC particle below the indentation. However, for large particle size, this possibility could be ignored as the particle number density would be small and that hardness value is the average of several indentations.

The tensile test was used for measurement of the strength according to ASTM B557 standard. Table 1 presents the results of composites yield strength and UTS. The respective figures for the mechanical properties were obtained from averaging over five measurement values. It is noted that the strength of the composites depends on the volume fractions of the composites’ constituents.

It is believed that the great enhancement in tensile strength observed in these composites is due to good distribution of the nano-SiC particles and low degree of porosity which leads to effective transfer of applied tensile load to the uniformly distributed strong SiC particulates. The grain refinement and strong multidirectional thermal stress at the Al/SiC interface are also important factors, which play a significant role in the high strength of the composites. SiC particles have grain-refined strengthening effect, since they act as the heterogeneous nucleation catalyst for aluminum which is improved with increase in the volume fraction (Abdizadeh and Baharvandi, 2009; Watson and Cline, 1992).

The difference between the coefficient of thermal expansion (CTE) values of matrix and ceramic particles generates thermally induced residual stresses and increase dislocations density upon rapid solidification during the fabrication process. The interaction of dislocations with the non-shearable nano-particles increases the strength level of composite samples. According to the Orowan mechanism, the nano-SiC particles act as obstacles to hinder the motion of dislocations near the particles in the matrix. This effect of particles on the matrix is enhanced gradually with the increase of particulate volume fraction (Mazahery and Shabani, 2011c; Habibnejad-Korayema et al., 2009).

Optimizing the mechanical properties of the SiC reinforced aluminum alloy composites attracted continuous interest during the last several decades. SiC nano-particles have been added to the Al 356 alloy using an ultrasonic method (Lan et al., 2004b). Experimental results showed more than 50% improvement in yield strength of A356 alloy only with 2.0 wt.% of nano-sized SiC particles.

![Figure 6](image-url)  
**Figure 6** Hardness versus the nano-SiC content.

<table>
<thead>
<tr>
<th>Vol.% SiC</th>
<th>Tensile YS (MPa)</th>
<th>UTS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Un-reinforced</td>
<td>122</td>
<td>145</td>
</tr>
<tr>
<td>0.5</td>
<td>127</td>
<td>220</td>
</tr>
<tr>
<td>1.5</td>
<td>134</td>
<td>238</td>
</tr>
<tr>
<td>2.5</td>
<td>142</td>
<td>257</td>
</tr>
<tr>
<td>3.5</td>
<td>145</td>
<td>283</td>
</tr>
<tr>
<td>4.5</td>
<td>137</td>
<td>240</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Vol. % SiC</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Un-reinforced</td>
<td>6</td>
</tr>
<tr>
<td>0.5</td>
<td>3.8</td>
</tr>
<tr>
<td>1.5</td>
<td>3.77</td>
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<tr>
<td>2.5</td>
<td>3.73</td>
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<tr>
<td>3.5</td>
<td>3.6</td>
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<tr>
<td>4.5</td>
<td>3.3</td>
</tr>
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</table>
Zhao et al. (2008) characterized the properties and deformation behavior of (Al$_2$O$_3$ + Al$_3$Zr) np/Al nano-composites produced by magneto-chemical melt reaction. It is reported that elongation, ultimate tensile strength and yield strength of nano-composites are enhanced with the increase of particle volume fraction, and are markedly higher than that of Al composites synthesized by microsize particles.

According to the results of this experiment, quite significant improvement in strength is noted initially when particles are added; however, further increase in SiC content leads to reduction in strength values. The weakening factors of mechanical properties might be responsible for this including particles clusters and porosity. Hereby, it is believed that strengthening and weakening factors of mechanical properties could neutralize the effect of each other and thus, the composite containing 3.5 vol.% SiC exhibits maximum tensile strength. Usually the introduction of micro-SiC particles increases the elastic modulus, yield stress and UTS but decreases the ductility and toughness of the composites (Reddy, 2003; Kang, 2004; Mazahery and Shabani, 2011a; Maik et al., 2007).

It is of interest to use nano-sized ceramic particles to strengthen the metal matrix, while maintaining good ductility (Mussert et al., 2002). It is inferred from Table 2 that the addition of nano-particles deteriorates the ductility of A356 alloy. The stir casting method that is used in the present work to produce the nano-composites can most probably create different interfaces between nano-particles and matrices and thus, encourage crack initiation and propagation (Abdizadeh and Baharvandi, 2009). It is also noted that the elongation remain rather constant with the addition of nano-particles. This is consistent with the findings of Hassan and Gupta (2006, 2005b).

The appearance of pores in the matrix degrades the plasticity of the composites since pores are one of the microcrack initiators during deformation, such as the SiC particles.

In general, the increase in the strength of the composites by the introduction of SiC particles is accompanied by the decrease in the plasticity, since SiC particles are the microcrack initiators during deformation. As a ceramic phase, SiC is brittle and has high strength. During deformation, two types of the microcracks will be initiated by the SiC particles. First, if the interfacial cohesion between the SiC particles and matrix is
strong, the SiC particles will fracture to nucleate microcracks when the local strain and dislocation density reach the critical values by the high stress concentration. Second, if the interfacial cohesion between the SiC particles and matrix is weak, decohesion between the SiC particles and matrix will happen to nucleate microcracks before the SiC particles are fractured. Thus, a strong interfacial cohesion can improve both the strength and plasticity of the composites since weak interfaces will nucleate microcracks at a rather low external applied stress.

The fracture modes of AMCs can be controlled by a number of material and processing parameters such as the type, shape, volume fraction and distribution of the particles, as well as the matrix and interface properties which may include the solute segregation, precipitation effect, porosity amount, interfacial bonding strength, original sample surface roughness, etc. Most of these parameters will be strongly influenced by the processing and thermal treatment history. Failure in particulate-reinforced AMCs is believed to be due to three different sources, namely, the matrix/reinforcement interfacial decohesion, reinforcement fracture, and failure in the matrix. To determine the fracture mechanism(s) in samples with minimum and maximum volume fraction of reinforcement particles, microscopic observations were made on the fractured samples. Typical SEM micrograph of tensile fracture surfaces for composites is shown in Fig. 7. Fracture surface observations of the samples show that the main controlling fracture mechanism is inter-dendritic cracking. This failure mode is identical for the A356 unreinforced alloy which has been recently investigated (Tahamtan and Boostani, 2010). During solidification of the composite, the nano-particles and alloys elements (mainly Si), are rejected to the solid/liquid interface and segregate to the inter-dendritic regions (Li et al., 2004).

The microcracks propagate along inter-dendritic aluminum–silicon eutectic and silicon particles resulted in failure of the specimen which implies that the fracture of this composite is dominated by failure of the matrix alloy.

However, some areas of the composites fracture surfaces consist of dimples which may be a result of the void nucleation and subsequent coalescence by strong shear deformation and fracture process on the shear plane. The dimpled rupture occurs mostly by voids initiation at eutectic silicon particles.

It is reported in the literature (Zhao et al., 2008; Lan et al., 2004b) that all the fracture surfaces of the composites consist of numerous dimples in the matrix and fragmentation and decohesion of the particles from the matrix. The dimples should be a result of the void nucleation and subsequent coalescence by strong shear deformation and fracture process on the shear plane, while the fracture and decohesion of the particles can be explained by work-hardening and the fragmentation of the ceramic phase caused by high stress concentration (Tahamtan and Boostani, 2010). The main difference of the fracture surfaces is that increasing the volume fraction of the SiC particles decreases the ductile fracture feature.

Since the strength and hardness of alloys mainly depend on their microstructure, a lot of efforts have been made for refining the microstructure of castings in order to improve the mechanical properties of A356 matrix composites. Refinement of primary dendrite arm spacing (DAS) and SDAS is the common method, which, in general, is adopted by many researchers (Li et al., 2004) [42, 43]. Figs. 8 and 9 show grain size and the length of silicon rod as a function of cooling rate. It is observed that the addition of nano-particles refines the microstructure of the Al matrix. Fig. 10 shows the effect of cooling rate on tensile strength of the nano-composite. It is observed that the refinement of the microstructure improves the strength of the composite.

4. Conclusions

1. The stir casting technique can be used to produce A356 aluminum alloy/nano-SiC particulate composites in which the SiC particles are distributed uniformly within the matrix alloy.
2. There is no obvious effect of nano-particles content on the ductility of the composites. In contrast to the plasticity, the composites’ strength and hardness are clearly influenced by the effect of the volume fraction of nano-SiC.
3. Compared to the unalloyed reference composite, the addition of 4.5 vol.% of SiC raises the UTS from 145 to 240 MPa, i.e. by about 65%. Different strengthening mechanisms contributed to the obtained strength improvements including Orowan strengthening, grain refinement, accommodation of CTE mismatch between the matrix and the particles, and the load bearing effects.
4. Regarding the fracture surface of the test specimens, two fracture modes have been distinguished for the investigated composite systems. Effective interfacial debonding between the matrix alloy and the SiC particles has not been observed in any case. Some remaining thin matrix material layer is always found to adhere to the nano-SiC particles’ surface.

References