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Microstructural Evolution of Aluminum-4043/Nickel-Coated Silicon Carbide Composites Produced via Stir Casting

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Authors' contributions

This work was carried out in collaboration between all authors. Author PKF designed the study, supervised over the research and wrote the first draft of the manuscript. Author SO performed the experimental work and obtained the necessary results. Author BOA managed the literature and interpreted the experimental results. Author PKF finalized the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

Aluminum-silicon carbide (Al/SiC) metal matrix composites have become a promising engineering material owing to its high strength to weight ratio. However, there is formation of aluminum carbide phase which is deleterious to the physical properties of the composites and needs to be eliminated. In this paper, the stir casting of Al-4043/Ni-coated SiC composites was investigated and reported. The Al/SiC composites were produced by melting Al-4043 lumps, injecting SiC powder with varying weight fraction into the Al melt and pouring the stirred mixture into a sand mould at 680℃. Prior to the use of the SiC powder, the powder particles were coated with 15 µm thickness of nickel to prevent dissolution of the SiC into the Al melt. The microstructure of the composites was

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characterized using optical and scanning electron microscopes to reveal the microstructural features and phases present. The result showed that composite microstructure was characterized by filament-like and particulate-like features which are uniformly distributed in the Al matrix. The volume fraction of the filaments increased with increasing SiC reinforcement fraction employed during processing. The microstructure formed is predominantly Al-SiC eutectic microstructure with uniformly dispersed SiC particles. The Ni coating has presumably prevented the formation of Al_4C_3 and promoted the formation of eutectic microstructure. A higher strength is anticipated with the filaments randomly oriented and uniformly dispersed in the Al-4043/Ni-coated SiC composites.

Keywords: Metal matrix composite; aluminum; silicon carbide; nickel coating; microstructure; stir casting; Al/Si eutectic.

1. INTRODUCTION

Aluminum alloys are known to belong to the family of light metals which are considered for weight-saving, structural applications such as aerospace, automotive, turbomachinery and other industrial applications [1]. The structural use of aluminum alloys in aircraft and automotive industries reduces fuel consumption, thus bring about energy cost saving and reduction of CO_2 and NO_x emissions which are responsible for ozone depletion and global warming. Though, the Al alloys are desirable due to their low density, their structural stiffness may not be adequate for some light weight applications. Moreover, the use of monolithic metals may not serve favorably for industrial applications where there are concerns for combinations of high temperature strength, high thermal conductivity, increased wear resistance and high structural stiffness at room temperature amongst others. Hence, there is a need for development of new materials such as metal matrix composites which could serve well industrially to ameliorate the limitations of monolithic materials.

Metal matrix composites (MMCs) are formed to synergise the thermo-mechanical properties of metal and embedded ceramic reinforcements which can be in the form of particles, fibres, or whiskers. Aluminum MMCs are developed to achieve a light-weighted material with high strength when compared to monolithic Aluminum alloys for space, aircraft, transportation, microelectronics and industrial applications [2,3]. The most frequently used ceramic reinforcement in Al alloys is silicon carbide (SiC) which is due to its low density when compared to other ceramics such as TiC, WC, etc [4]. Al/SiC composites can be produced via either solid phase processes such as: powder metallurgy, hot isostatic pressing, and rolling or liquid phase processes such as: squeeze casting, stir casting, and infiltration amongst others [5]. Amongst the various processing routes for the manufacture of Al/SiC composites, stir casting method has been considered to be of significant, low cost approach [6,7]. The stir casting method has been reported to give a better distribution of the reinforcement in the matrix which significantly and positively influence the thermo-mechanical properties of the Al/SiC composites produced.

Alaneme and Aluko [8] investigated the production and age-hardening behavior of Al-6063/SiC composites using stir-casting method and observed a uniform distribution of the SiC particles with minimal particle agglomeration in the Aluminum matrix. It was also reported that composite hardness increased with increasing volume percent of SiC reinforcement employed. In another study, Nidhish and Sijo [9] investigated the volume fraction of SiC reinforcement in stir-cast Al/SiC composites that would yield the best fracture toughness and noted that composite with 25 wt.% SiC reinforcement gave the highest value of stress intensity factor which is indicative of high fracture toughness. Nagaral et al. [10] investigated the wear behavior of Al-6061/SiC composites produced with varying weight percent of SiC using stir casting. It was reported that there was a significant reduction in wear loss of the composites as the reinforcement fraction increases from 6 - 9 wt.% with compared to the wear loss of the monolithic Al-6061 matrix. Zakaria [2] investigated the microstructure and corrosion behaviour of Al/SiC metal matrix composites formed by varying the size and mass fraction of reinforcement using powder metallurgy production approach and observed that the reduction in SiC particle size and increasing its mass fraction resulted in decrease in corrosion rate in saline solution. It was further reported that Al_4C_3 phase is formed at the interface between the SiC particles and Aluminum matrix at temperature greater than or equal 710℃ when liquid phase processing approach is employed for the production of Al/SiC composites. The formation of Al_4C_3 has been found to be deleterious to the corrosion performance of the composites. Bhushan et al. [11] reported that frequent problems encountered when fabricating Al alloy based MMC with SiC is the formation of Al_4C_3 at the carbide/matrix interface as a result of interfacial reaction at elevated temperature. Lee et al. [12] studied the interface of SiC/Al-2014 alloy (with 1% Si in matrix) held 2 hours at 600℃, and observed an increased amount of Si in the matrix, but the presence of Al_4C_3 could not be identified by XRD. However, nanosized Al_4C_3 precipitates were observed on the surface of SiC particles by SEM. Hu et al. [13] investigated the microstructure and properties of SiC particle reinforced aluminum matrix composites using vacuum assisted high pressure die casting and noted that there was uniform distribution of SiC particles in the MMCs with decreased fraction and sizes of pores when compared to MMCs produced by gravity die casting. In another similar study, Soltani et al. [14] fabricated aluminum matrix composites with SiC as reinforcement and reported that shorter stirring period is required for proper metal/ceramic bonding at the interface and higher stirring temperature (850℃) led to improvement in the particle distribution. However, shrinkage porosity and intensive formation of Al_4C_3 were rarely observed. As notably reported by Mahesh et al. [15] that Al_4C_3 formation is promoted during the fabrication of aluminum and SiC when the processing temperature exceeds 720℃, this makes the processing of Al/SiC composites to be temperature sensitive.

The extensive study of the previous work on the production of Al/SiC composites has shown that the composites have superior properties when compared to the monolithic Aluminum alloy. The SiC reinforcement size and mass fraction are significant at influencing the thermo-mechanical and corrosion properties of the composite formed. Whilst stir casting method, a liquid phase processing technique has been widely reported to be the most economically and cost-effective technique for the production of Al/SiC composites, there is a challenge of Al_4C_3 phase formation at the interface between the SiC particles and the Aluminum matrix with this production technique employed. This interfacial Al_4C_3 phase formation has been observed to reduce the corrosion resistance of the composite and resulted in poor load transfer between the matrix and the embedded reinforcements. In the past, researchers have experimented different strategies such as preheating of the reinforcement particles and addition of some elements which include, Ni, Cu, or Mg. These are expected to slow down the rapid precipitation of the Al_4C_3 phase and also reduce surface tension of the molten Aluminum matrix to improve the wettability of the particle surfaces by the liquid Aluminum during processing.

Hence, the aim of this study is to investigate the microstructure of stir-cast composites of Al-4043 alloy and Ni-coated SiC particles which is yet to be elucidated. Whilst many of the previous researches have concentrated on the use of Al-6061 alloy, in this work Al-4043 alloy was employed as it is easy to cast and possesses excellent wettability characteristics due to its high silicon content of 4.5-6 wt.%. Thus, a good wetting of the reinforcing particles is envisaged.

2. MATERIALS AND METHODS

2.1 Materials

The materials used for the fabrication of the Al-SiC composites were aluminum (Al-4043) alloy lumps, as shown in Fig. 1., obtained from System Metal Industries Ltd. Aba, Nigeria and silicon carbide powder with angular morphology, typical of water atomization and having a particle size range of 10 – 60 µm, was supplied by Logitech Materials Company, UK. The elemental composition of the Al-4043 alloy can be found in Table 1.

Fig. 1. Aluminum Al-4043 alloy lumps used in the study

2.2 Nickel-coating of Silicon Carbide Particles

In order to prevent interfacial reaction which could result in the formation of Al_4C_3 phase, the SiC powder particles were coated with nickel using a model-12A4D vacuum coating machine, made by Hind High Vacuum Company Limited, Bangalore as shown in Fig. 2. The SiC particles were cleansed using acetone and a 10 g/l H_2SO_4 acidic solution for 10 mins and 7mins respectively. Thereafter, the SiC particles were immersed in a solution of 0.5 g/l $PdCl_2$ and 3ml/l HCl for 15 mins to activate their surfaces. The cleansed and dried SiC particles were gently dispersed in the vacuum chamber of the machine. The nickel coating was carried out in the vacuum at temperature range of 80-86℃. The coating took place for 75 mins to achieve an approximately 15 µm Ni-coating thickness on SiC particles in the vacuum chamber which is anticipated to be sufficient to prevent rapid dissolution and formation of the deleterious Al_4C_3 phase around the particles in the Aluminum matrix.

Table 1. Elemental composition of the Al-4043 alloy used in this study

Element	wt.%
Aluminum, Al	94.80
Silicon, Si	5.20

Fig. 2. A model-12A4D vacuum coating machine employed for Ni-coating of SiC particles

2.3 Stir casting of Al-4043/Ni-Coated SiC Composites

Aluminum alloy (Al-4043) was used as matrix reinforced with Ni-coated SiC particles. The MMCs were fabricated by melting the aluminum (Al-4043) alloy in an improvised furnace (black smith hearth furnace) then the reinforced Nickel coated SiC particles were added and stirred slowly. Thermocouple was utilized during the melting process to measure temperature. Aluminum (Al-4043) alloy was first preheated to 450℃ for 45 mins before melting and Ni-coated SiC powder were preheated to 900℃ for 30 mins to remove moisture in the powder for better wettablility. The furnace temperature was first raised above the melting point of aluminum to 720℃ in order to melt the matrix completely and then it was allowed to decrease to temperature below the melting temperature (540℃) to keep the slurry in a semi-solid state. At this stage, the preheated SiC particles were added into the aluminum slurry and mechanically agitated. The composite slurry was then reheated to a fully liquid state and stirred for 8 mins to ensure the formation of a homogenous mixture. The furnace temperature was controlled within 720±10℃. After melting, slurry mix was poured into a prepared mould, as shown in Fig. 3. with pouring temperature maintained at 680℃.

Fig. 3. Cast preparation by pouring the Al/Ni coated SiC slurry from the crucible into the mould

2.4 Cast Characterisation

Samples were cut from the cast product of the Al/Ni-coated SiC composites. These were mounted in a conductive resin, ground using abrasive SiC papers with grit size range of P150 – 1000, and polished to 1 µm Ra finish using a diamond paste smeared cloth. In order to reveal the microstructural details of the cast, the polished surfaces were etched with HF acidic reagent for less than 5 sec. The microstructure of the cast samples were evaluated using a Maker 0524011 optical microscope and a Phenom Prox 800-07334 scanning electron microscope (SEM) with energy dispersive X-ray (EDX) capability for elemental composition analysis.

3. RESULTS AND DISCUSSION

3.1 Microstructure

The microstructure of the polished surfaces of the cast samples was observed using the optical microscope. Fig. 4. shows the optical micrographs of the cast samples with varying percentage weight of SiC reinforcement in the Aluminum matrix and a micrograph of cast Al-4043 alloy without any reinforcement was also displayed. In all the micrographs for the Al/SiC composites, filaments and particulate features were observed and the area density of the filaments and particles present in the micrograph increases with increasing weight percent of the SiC reinforcement employed during cast preparation. The randomly oriented filaments and particles were uniformly distributed in the Aluminum matrix which indicates that the mechanical agitation introduced during cast preparation is adequate to produce a homogenous Al/SiC composite.

Moreover, few reinforcement agglomeration sites were observed in the composite, and prevalence of agglomeration increases with increasing SiC reinforcement fraction employed. This may be inevitable and can be overcome through the optimization of agitation speed during cast preparation. Furthermore, few pores were observed in the composites under optical microscope which may have been formed as a result of gas entrapment in the molten state of the composite. Owing to increasing solubility of gases in molten metal, gases dissolve in the metal in liquid state and as solidification and cooling begin, the gas molecules are entrapped and try to escape, thus leaving behind pores observed in the composite. This seems to be one of the challenges of casting process as full densification of cast may not be easily achieved, however, post processing techniques such as hot isostatic pressing (HIP), full densification of cast components is guaranteed.

3.2 Phase Identification

The Al/SiC composites were further investigated using SEM to examine different microstructural features and identify their probable phases. Fig. 5. shows the SEM micrograph of Al-4043/10 wt.% SiC composite in backscattered electron (BSE) mode which enable the identification of the phases based on brightness and contrast. The background was distinctly identified with the dark grey colour which is definitely the Aluminum solid solution matrix. Moreover, light grey whisker-like or filament-like features which presumably have precipitated out of the Aluminum matrix during cooling and solidification. These features are similar to the filaments observed in the composite microstructure under the optical microscope as shown in Fig. 4. Whitish and angular features were observed in the composite microstructure under SEM and few pores were also observed in the micrograph as tiny dark spots.

The occurrence of the observed features can be explained by considering the thermal history of the composite production process. Firstly, the Al-4043 alloy was melted to liquid state at 720℃, and a preheated Ni-coated SiC powder was charged into the Al alloy in a semi-molten state at 540℃. However the slurry of Ni coated SiC powder and the Al alloy was reheated and pouring temperature was approximately maintained at 680℃ which is a safe temperature zone to prevent formation of Al_4C_3 [15]. The cast was also made using a sand mould. Though a chilling effect may be inevitably experienced by the cast at its surface as cooling rate would be higher at the surface as the mould surface serves as a heat sink, however, the interior part of the cast from which the samples were taken for microstructural analysis is expected to have undergone a slower cooling. Hence, with a lower and steady cooling rate, a near-equilibrium microstructure is envisaged to be formed in the interior part of the cast Al-4043/Ni-coated SiC composites. This developed near-equilibrium microstructure is presumed to follow the relevant phase diagram as the microstructure formed is dependent on the cast chemistry and the steady and lower cooling rate.

It has been known that Al-4043 alloy is a hypoeutectic alloy as it contains a maximum value of 6 wt.% Si, as indicated on a binary Al-Si phase diagram in Fig. 6. According to the phase diagram, as cooling takes place and the cast temperature decreases below 650℃ with a silicon content of 6 wt.%, the Al-4043 liquid solution phase began to transform into Aluminum solid solution dispersed in a Si-rich Al liquid phase as shown in equation (1). It is envisaged that there would be sufficient time for the

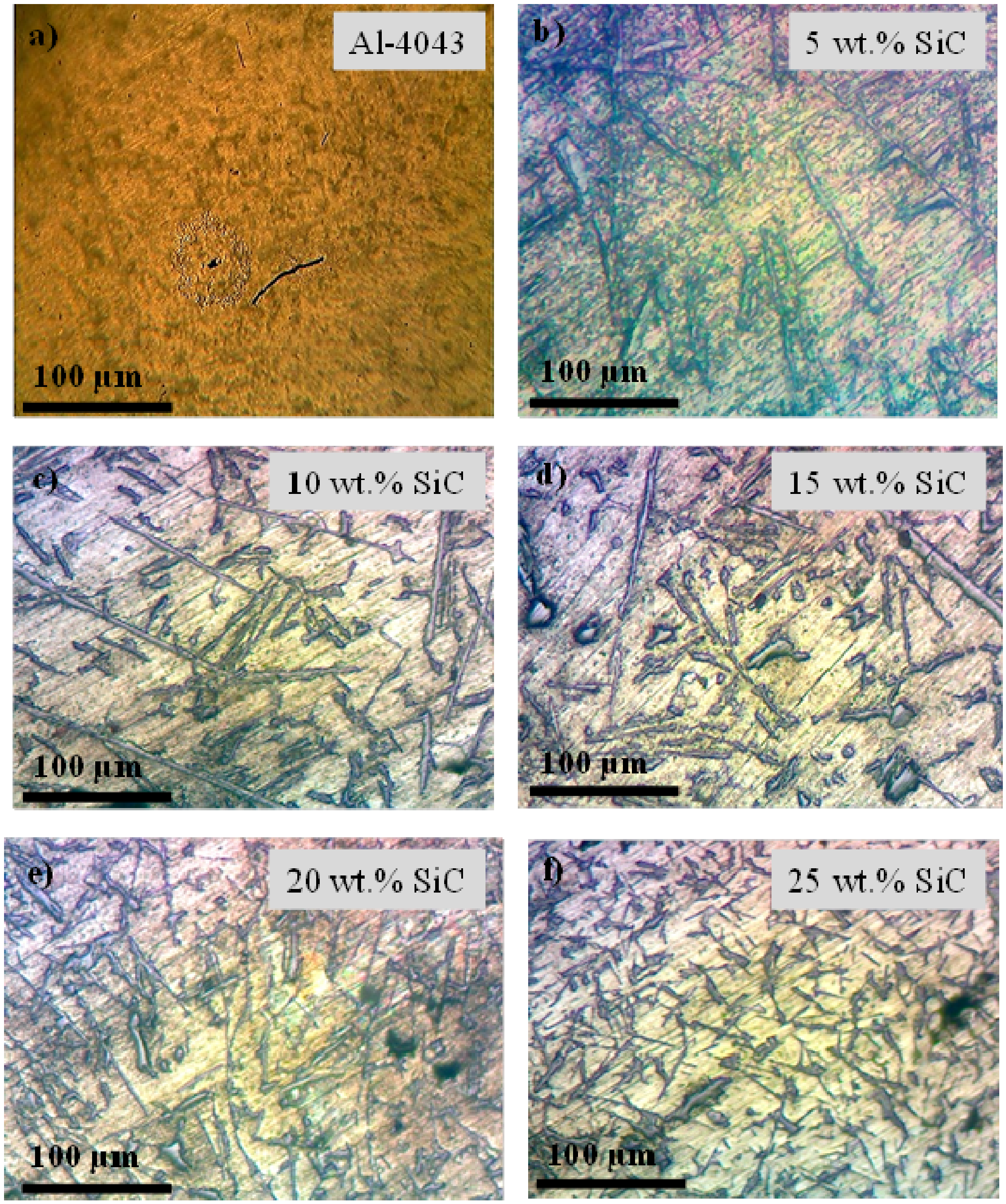

Fig. 4. Optical micrographs of the Al-4043/Ni coated SiC composites with varying wt.% reinforcement

Aluminum solid solution to nucleate and grow, also as the temperature approaches the eutectic isotherm at 577^{o}C, Al-Si eutectic phase began to form in the Si-rich Al liquid phase (equation (2)). Hence, with an assumption that the solidification follows the phase diagram, the expected microstructure to be formed is a Al-Si eutectic in a primary Aluminum solid solution matrix.

$$L = L + Al\ (650^oC,\ 6\ wt.\%\ Si) \quad (1)$$

$$L = Al + Al\text{-}SiC\ (577^oC,\ eutectic) \quad (2)$$

It is worthy to note that Al-Si eutectic is sensitive to impurities [16], and in this study, Ni could have possibly acted as impurity which promotes the formation and precipitation of Al-Si eutectic as maximum solubility of Si in Al at eutectic temperature is observed to be 1.65 wt.% from the Al-Si phase diagram. There is possibly partial dissolution of the Ni coated on the SiC particle surface to inhibit the formation of aluminum carbide, owing to the thermal history of the cast process. Hence, the light grey filament-like features can be considered as the Al-SiC eutectics which had precipitated into the primary Aluminum solid solution. This is in agreement with the result presented by [17]. The whitish and angular features are considered to be the retained SiC powder particles embedded in the Al-4043 matrix. Since, there is possibility of Ni (1,455°C, melting point) dissolving into the matrix, SiC (2,730°C, melting point) particles could as well partially dissolved after the Ni layer has been fully removed and provided the Aluminum melt temperature supports the SiC particle dissolution. However, the increasing volume fraction of the Al-SiC eutectic precipitates as the weight percent of SiC powder employed to reinforce the matrix increases, suggests that there is a high possibility that the melt is more enriched with Si which then led to the increased volume fraction of Al-SiC eutectic precipitates formed in the composite as shown in Fig. 4. In both optical and SEM micrographs (Figs. 4 and 5), the primary Al phase with distinct grain boundaries were not observed in this study when compared to the microstructure observed for a typical hypoeutectic Al-Si alloy with Ni addition [18], as Al-Si eutectic microstructure dominates the cast composites.

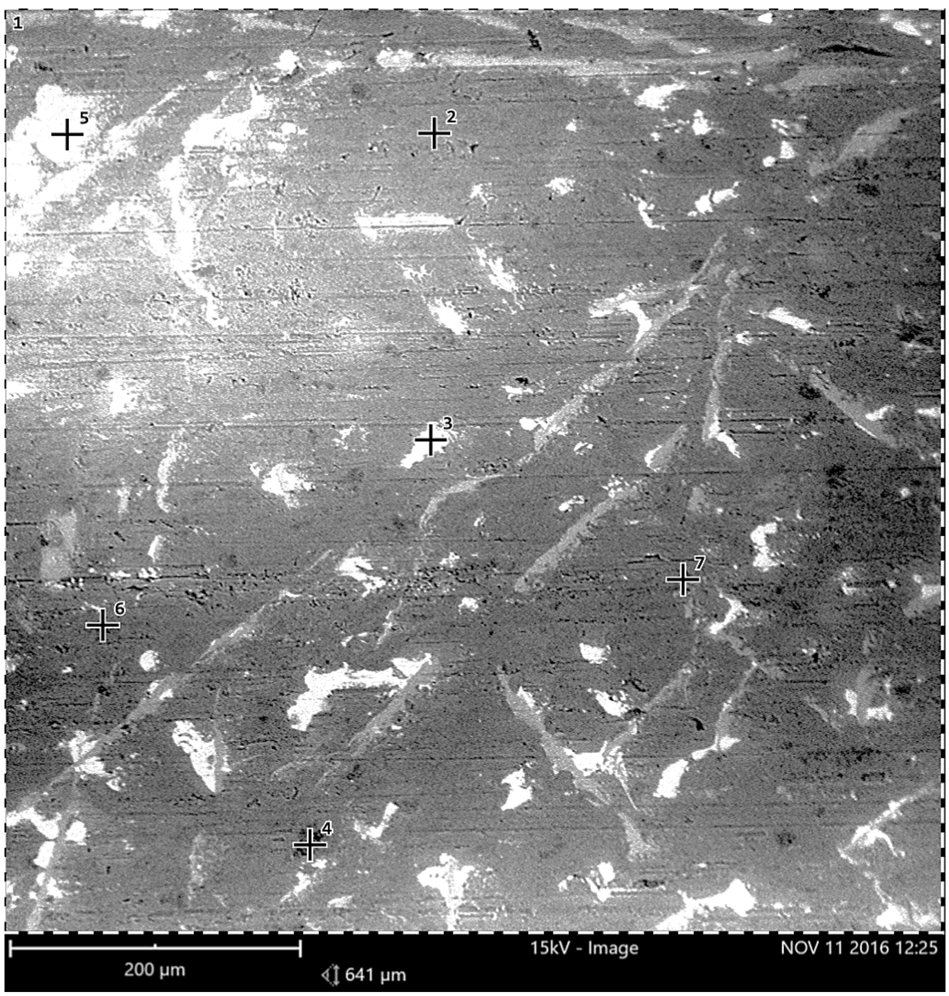

Fig. 5. BSE-SEM image of the Al-4043/10 wt.% Ni coated SiC composite

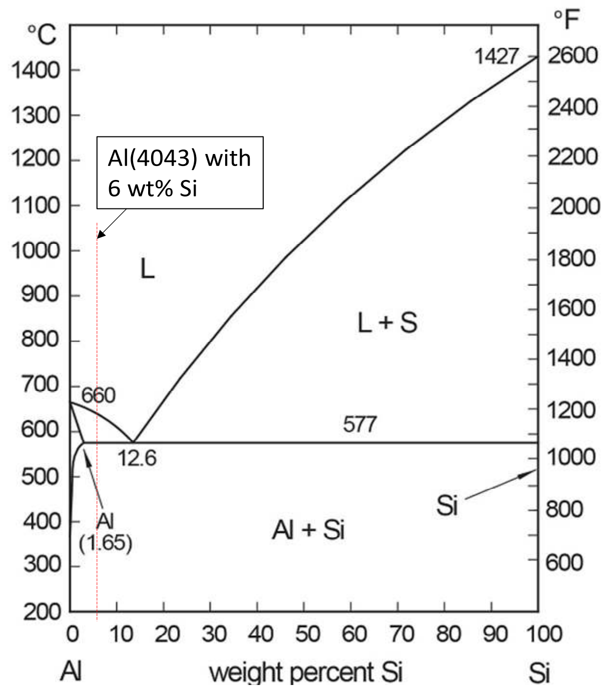

Fig. 6. Al-4043 alloy located on the binary Al-Si phase diagram

As there is possibility of partial dissolution of Ni and SiC into the molten Aluminum alloy during processing, it is certain that the molten Aluminum alloy will be enriched with carbon during the casting process. Though the aluminum carbide (Al_4C_3) was not observed in the microstructure of the composites, there is possibility of the carbon present forming an interstitial solid solution in the primary Aluminum phase or in the Al-Si eutectic phase, provided the carbon solubility in solid aluminum of 0.015 wt.% has been exceeded [19].

4. CONCLUSION

The stir casting of Al-4043/Ni-coated SiC composites has been successfully carried out and their microstructure reported. The microstructure of the hypoeutectic Al alloy with SiC reinforcement was characterized to possess filament-like and particulate-like features which are uniformly distributed in the Al matrix, thus suggesting adequate stirring during the cast process. The volume fraction of the filaments increased with increasing SiC reinforcement fraction employed during processing. The microstructure formed is predominantly Al-SiC eutectic microstructure with SiC particles dispersed as the primary Al phase with distinct grain boundaries was not observed. The Ni coating has presumably prevented the formation of Al_4C_3 and the formation of eutectic microstructure and it is expected that an interstitial solid solution with carbon would have been formed in the eutectic microstructure. A higher strength is anticipated with the filaments randomly oriented and uniformly dispersed in the Al-4043/Ni-coated SiC composites.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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