Microstructural evolution and mechanical properties of aluminum silicon/short carbon fiber composite fabricated by developed semi-solid thixomixing method

Ebrahim Akbarzadeh*, Josef A. Picas, M. Teresa Baile
Light Alloys and Surfaces Treatment Design Centre (CDAL), Universitat Politècnica de Catalunya (UPC),
08800 Vilanova i la Geltrú, Barcelona, Spain
Ebrahim.akbarzadeh@estudiant.upc.edu

Abstract

A novel method was developed, based on thixotropic nature of semi-solid slurry of aluminum silicon alloy, for the effective dispersion and distribution of short carbon fibers (C_{sf}) under intensive shear stress (τ) into the eutectic without physical or chemical damage. The C_{sf} were distributed homogeneously into the matrix at optimum conditions: a temperature of 576-580 °C, around 48% of solid fraction, and rotation speed of mixing was at 100 rpm. The gas entrapment and porosity was a challenge in the fabrication the aluminum composite by thixomixing, and its size increased when the samples were heat treated. The extruded and heat treated composites had around 24% improvement of mechanical strength as assessed by tensile tests. The predominant deposition of silicon and its intermetallics at the interfacial region improved the wettability and adherence to achieve good mechanical properties. The formation of some intermetallics can be helpful to overcome low wettability effects and their structural evolutions were impressive. The fractography analysis illustrated the ductile fracture with some plastic deformation as the results of dimples and some fiber pull-out in the fractured surface. Ultra-micro hardness evaluation showed good interfacial bonding after heat treatment. The thixomixing process can be developed to produce various composites with different reinforcement.

Keyword*: A. Carbon fibers; A.* Metal-matrix composites (MMCs); *B. Fibre/matrix bond; E. Thixomixing.*

1. Introduction

Aluminum composite is an important new material that has outstanding mechanical properties and lightness, which are desirable for aerospace, automobile and other industries; it is also less expensive. The polyacrylonitrile (PAN)-based short carbon fibers (C_{sf}) provide high strength, specific modulus, low coefficient of thermal expansion and low density when combined with aluminum as a metal matrix, and they are is suitable for high voltage transmission cables. Since 1960, various methods have been used to fabricate aluminum composite reinforced with carbon fibers [1, 2]. Conventional liquid stir casting and powder metallurgy are the main methods, but they are more costly and less effective for direct fabrication of aluminum carbon fiber composites. Low interfacial adherence and non-homogeneous distribution are the main challenges for the conventional metal composite manufacturing process [3]. Previous research on homogeneous distribution of reinforcement particles in an aluminum matrix have been attempted by milling of SiC_p and aluminum powders and casting at semi-solid state to improve the wettability and distribution [4, 5]. Undesirable fiber cracking occurs under high load pressure in the powder metallurgy process [6]. Metal matrix composites (MMC) are also prepared with prepreg methods using liquid metal routes such as squeeze casting or vacuum pressure infiltration, which leads to poor wettability and weak adherence at the interface [7]. Good adhesion at the fiber/matrix interface and homogeneous distribution of reinforcement are the main technical problems, which remained unsolved [8, 9]. Poor infiltration, low interfacial adherence of matrix reinforcement, undesirable intermetallic formation, good dispersion and homogeneous distribution and weak tensile strength are common problems in aluminum composite fabrication. A successful load transfer is indicated by good interfacial bonding [10]. Strong adherence is essential in a MMC with good dispersion and uniform distribution of fibers in the matrix to increase of the mechanical strength. To the best of our knowledge, the thixomixing method was not being utilized for distribution of C_{sf} into the aluminum matrix yet. In compocasting, the reinforcement particles are added to a fully liquid metal under vigorously mixing [11]. The long processing methods at high temperature are not economical and cause deleterious chemical reaction such as brittle phases at the interface of bonding [12]. Magnesium (Mg) and silicon (Si) may improve the wettability of aluminum onto carbon fiber by predominate deposition Si and formation of suitable adhesive intermetallic phases [9, 13].

In this study, a novel thixo-process was developed for the dispersion of filaments of carbon fiber and mixing of C_{sf} at semi-solid aluminum under high shear stress. Thixomixing can be classified as a method of compocasting. Basically, it is an application of high shear stress (τ) on solid particulate embedded in semi-solid slurry of metal to overcome the strength of dendritic structure. The effects of shearing and temperature were studied on the dispersion of fibers. Some of the advantages of the improved structure include relatively good adherence, less damage to the fibers, no flotation, low cost and good distribution and dispersion of fibers. Some problems associated with the reinforced MMCs, such as weak interfacial bonding, non-distributive microstructure, and deleterious interfacial reactions can be solved by this method.

2. Materials and Experimental

2.1. Materials and fabrication

The PAN-based carbon fiber T300 made by Japan Toray Co. was utilized in this study and the physical properties have been illustrated in Table 1. The fibers were chopped into 5±1 mm length then the pyrolytical sizing agent on the fibers removed by heating in aerated furnace at 500 ºC for 30 minutes. Two aluminum silicon alloys, A356 and A357 were chosen as matrix alloy with chemical composition tabulated in Table 2.

A novel specific mixer has been designed for process named thixomixing. The short carbon fibers, C_{sf} embed in semi-solid slurry of aluminum silicon alloy as matrix by continuous shearing of slurry which is schematically shown in Fig. 1. The mixer contains two main parts, the rotary part called rotor and the stationary part as die with a 2 mm gap provided between them. This space among two walls provides high shear rate $(\dot{\gamma})$ and shear stress (τ) by semi-solid slurry at the appropriate temperature and rotating speed. Both die and rotor were made of H13 steel alloy and heat treated for better hardness and toughness, then joined with CNC machine for precision vertical movement and various rotating speed. Prior of mixing, die and rotor were preheated to 600 ºC then the molten alloy poured into the die, so the temperature of system must be maintained for thermal stabilization to form semi-solid slurry at desirable solid fraction. Three levels of solid fractions have been selected for this study as 0%, 48%, and 90% and the speed of rotating of mixer was varied at 100 and 300 rpm. The values of solid fraction for each alloy were extracted from the diagram of solid fraction versus temperature of each matrix alloy. The chopped carbon fiber was added by a metal tweezers gradually into the mixer to prevent agglomeration when the slurry of aluminum was sheared continuously. The process of mixing takes around 10 minutes for all samples. Finally, the rotor removed out from the die and the composite was cooled to the room temperature by quenching in water then it extracted from mixer (Fig. 1c).

2.2. Extrusion and Heat treatment

For further characterization such as tensile test, it was needed to re-shape the as-mixed composite by extrusion to the bars. The tool used to perform the extrusion is made of H13 steel alloy and the extrusion ratio was 9 (18/6) which has a convergent length of 6 mm. The slugs have a length of 20 mm and a diameter of 18 mm. The extrusion temperature was selected at 490 °C, to avoid fir-tree cracking on the extruded composite occurs when the surface temperature became so high. Extrusion speed was at 0.5 mm/s and the ceraspray used for lubricating and better finish surface. The T6 heat treatment was carried out on some of the successful extruded samples. The solutionizing was carried out at 540 ºC for 30 minutes and following quenched up to 25 ºC in water and then artificially aged at 170 ºC for 3 hours and finally cooled under flow of air [14].

2.3.1. Density and porosity

The Archimedes' principle has been used to measure the density by the difference between measured values and the theoretical densities of thixomixed Al/C_{sf} composite. The theoretical density can be calculated by rule of mixture. The volume fraction was measured by extraction of embedded short carbon fibers from samples by dissolution of matrix. Three pieces of each sample firstly weighted with an accurate digital balance, and then dissolved in diluted hydrochloric acid (1 M) at room temperature. The residual fibers has been filtered, dried and weighted by an accurate digital balance. The volume fraction of these three samples calculated and got average.

2.3. Mechanical and microstructural evaluation on Al/C_{sf} composite

The successful thixomixed extruded specimens with and without T6 heat treatment were used for the tensile test at parallel path of the extrusion. The tensile tests were carried out by hydraulic ZWICK Model Z100 test machine at the crosshead speed of 1 mm/s at room temperature. The gauge length of the specimen was 15 mm and a diameter of 4 mm. The fractured surfaces of specimens were examined by SEM.

The ultra-microhardness equipment (FISCHERSCOPE HCU) has been conducted to study the interfacial micro-mechanical properties of manufactured composite. Both composite before and after T6 heat treatment were subjected to ultra-microhardness tests which the indentation points were following the lines across fibers under 0.5 mN load with 3 μm distances between marks. This evaluation has been repeated three times for different fiber and position and got average, finally the hardness values have been plotted at fiber/matrix interface and evolution properties throughout the matrix region near to the fiber. This method has been used by Ureña et al. to investigate the interfacial properties between matrix and reinforcement particle [15].

Specimens were cut from different samples and positions for microstructural evolution and distribution of fibers. The morphology and distribution of fibers in the mixed and extruded samples and the fractured surface of tensile specimens were characterized using SEM (Jeol JSM-5600) and optical microscopy (Leica optical microscope).

3. Results and Discussion

3.1. The effect of temperature and speed of mixing on Thixomixing process

The main aim of this study was to develop a new method for fabrication of the Al/C_{sf} composite without damaging the fibers to have better dispersion and distribution in the matrix along with good adherence at the interface. The convectional mixers with impeller may damage the fibers undesirably, which due to its sharp edges of conventional impeller. Thixomixing process with no impeller protects the fragile carbon fiber from damaging whereas; this method is simple and consists of dispersive and distributive mixing under intensive shear load prior to the shaping. Thixotropic state supposed as a gel, when sheared becomes liquid and reverts back when static. Si concentration increases in the eutectic by a severe refinement caused to pushing silicon, when the alpha particles is growing initially in the molten. Temperature determines the solid fraction of slurry and various amount of viscosity. Three different levels of solid fraction 10%, 48% and 90% were selected to study the relationship between the solid fraction and speed of mixing at 100 and 300 rpm. The effects of the solid fraction and rotational speed of mixing on the success of dispersion, distribution and infiltration ratio was examined and shown in Fig. 2 for A356 as matrix. The solid particles segregate from liquidus by the centrifugal force at high speed that reduces the viscosity and infiltration. The marks provided in Fig. 2 indicate the successful (○) and unsuccessful (×) thixomixed samples with the values of infiltration ratio attached to them. It shows that the composite samples which the reinforcement have been distributed homogeneously in the matrix were fabricated under a specific fraction of solids in semi-solid slurry and at the appropriate rotational speed of mixer. Both 10% and 90% solid fraction regardless of mixing speed were unsuccessful, and C_{sf} did not infiltrated effectively into the matrix due to very low and too high shear stress, respectively. The slurry with 90% solid fraction was too stiff and just surrounds the cluster of fibers with no infiltration then fibers were agglomerated and cracked by solid particles. In the other point of view, semisolid slurry with 10% solid fraction does not provide sufficient viscosity and shear rate as required for dispersion of short fibers, so a few infiltration with more agglomeration was occurred. Both high and low solid fractions in slurry cannot be suggested for thixomixing process for dispersion and distribution of fibers or other type of reinforcement. The mushy state of slurry is necessary to provide shearing stress on fiber clusters and dispersion of them to being well distributed in the eutectic. Semi-solid thixomixing method can be assumed for alloys which have a long range of freezing. Homogeneous distribution and well dispersion are highly recommended to get the superior strength with suitable ductility. Fiber to fiber contacts and agglomeration leads to a non-homogeneity and caused the lower mechanical properties. It has been found that, the best dispersion and distribution of C_{sf} achieved at 48% solid fraction at 100 rpm as fabricating speed for both alloys. The mushy flow of slurry sheared and overcome to the agglomeration between carbon filaments, so it can entrap through the fibers and surrounds over fibers.

Theoretically, it can be interpreted that the semisolid aluminum slurry shows non-Newtonian or viscoplastic behavior which depends on the yield strength and the shear rate. Commonly, the shearing begins when the shear stress reaches to yield strength and it is mainly depends on the solid faction which proportionally related to temperature of alloy [16]. Laminar flow, induced in the liquid by the rotating of mixer, is associated normally with high viscosity fluids. The agglomerates of fibers break-up within the high shearing, which can lead to deagglomeration and homogenization. The Herschel–Bulkley model can be considered for shear stress (τ):

$$\tau = \left[\frac{\tau_0(\lambda)}{\dot{\gamma}} + K\dot{\gamma}^{n-1}\right]\dot{\gamma} \qquad (1)$$

Where, τ_0 is yield stress, $\dot{\gamma}$ is shear rate, λ is structural parameter, K is the consistency index and n is the power law index [17]. Burgos et al. represent the K as time dependent factor in semisolid behavior, when all the solid particles are connected, K=1 and none of the particles are connected, K=0 [18]. Based on the predicted temperature, the yield stress (τ_0) for A356 alloy is calculated as:

$$\tau_0 = 4.0 \times 10^{49} \times \exp(-0.181 \times T) \qquad (2)$$

which is corresponding variation of τ_0 with temperature. The rheological behavior of the A356 alloy in semisolid state is represented in Eq. 4

$$\tau = \eta_a\dot{\gamma} \qquad (3)$$

where, η_a is apparent viscosity that can be calculated (Eq. 5)

$$\eta_a = \left[\frac{\tau_0(\lambda)}{\dot{\gamma}} + K\dot{\gamma}^{n-1}\right] \qquad (4)$$

As the apparent viscosity increases sharply with a small increase of solid fraction and is a function of shear and cooling rates.

3.2 Microstructure

Fig. 3 shows the distribution and dispersion of C_{sf} in the composite at different resolutions for both alloys with different volume fraction under optimum condition of thixomixing in absence and presence of T6 heat treatment. Fig. 3a and 3b were taken at perpendicular plane to the extrusion direction and short fibers were aligned to it. Both images exhibited a good distribution of short fibers in the aluminum matrix which are well dispersed with few agglomerations, but contain some porosity. The grain size in reinforced samples were shown smaller than which was non-reinforced because of good distribution and dispersion of fibers in the matrix and provides a lot of heterogonous nucleation site [19]. The chopped fibers contain air which was arrested through them so when added to the semisolid slurry of aluminum while mixing, the gases were released into matrix and the porosity formed. Some of the pores may come from the air which was drawn in during the thixomixing process. The porosities can be reduced or removed by further processes such as extrusion or forging especially at semi-solid state. The iron intermetallic compounds have been detected as main intermediate compounds in all samples due to the Fe, an average atomic concentration which exits from matrix alloy or slightly dissolving from the steel body of die. As substantiated by Taghiabadi et al. with the concentration of Al, Fe and Si formed the needle-like β-Al_5FeSi and $Al_9Si_2Fe_2$ shown in Fig. 3a and EDAX results illustrated in Fig. 3d [20]. Most of aluminum silicon alloys are sensitive to the presence of iron, because of its low solubility in alpha phase at solid solution and form Fe-containing compounds which is harmful for mechanical properties and ductility [21]. As illustrated in Fig. 3c, the fibers has been embedded in the eutectic with higher concentration of silicon; whereas the primary spherical α-phase was nucleated initially prior of stirring. The dendritic structures of primary α-phase were broken under high shear stress and converted to a globular shape as it can be seen in Fig. 3c, and surrounded by the eutectic. During the process, the mixture contains the eutectic, short fibers and globular α-phase is being sheared continuously to get a homogeneous mixture. The α-phase has been solidified initially in the semisolid slurry in the shape of globular and participates with short fibers in the mixing process under high shearing load in the molten eutectic. As silicon in eutectic and the iron intermetallics solidify relatively late in the process, they have been deposited at the fiber/matrix interface. The lower temperature and short time involved in thixomixing process under intensive shearing prohibited or delayed the undesirable chemical reactions such as fragile aluminum carbide (Al_4C_3) at the interface of fibers and matrix. In additions, the high cooling rate such as quenching in water decreased the interfacial reactant and formation of Al_4C_3, remarkably. As demonstrated formerly, aluminum carbide which formed locally at the interfacial surface of matrix/fiber as needle-like affected the surface quality of C_{sf}, thermal conductivity and mechanical properties are due to the low capability of load transferring from matrix to fibers. Silicon content in aluminum alloy (having at least 7 wt.%) is a dual action element which improves the adherence and prevents the formation of aluminum carbide especially at temperatures up to 750 °C [22]. In this method, the high concentration of silicon in the eutectic might be helpful to overcome the problem in order to the lack of wettability between the carbon fiber and aluminum and will be discussed later. Consequently, the silicon layer was deposited in form of iron intermetallic, improving the wettability and inhibiting undesirable reactions, thus the extra coatings mentioned [23] such as copper or nickel are not needed. The size of Si particles in this method was very fine with an average size around 1 µm (Fig. 3c). During solidification the formation of such fine

Si particles has previously been observed before in rheo-process for ceramic composite due to uniform dispersion of particles in the matrix which provides a lot of heterogeneous nucleation sites (in this case, C_{sf} as nucleation sites) [7]. The fine and homogeneous microstructure achieved by this method is expected to improve simultaneously the tensile strength and ductility, due to that the agglomeration of particulates and non-dispersed reinforcement induces unwanted brittle nature in composite by predominant crack initiation sites [10]. The globular α-phase was illustrated in Fig. 3c has been changed to small grain size with different morphology after extrusion. The aluminum matrix composite developed by thixomixing have a homogeneous and uniform microstructure throughout the whole area and in different cross-sections. It is notable that the majority of C_{sf} are at the eutectic regions and in the grains boundaries, and represent good wettability, which was improved by silicon or intermetallics.

3.3. Microstructure evolution and heat treatment

The short fibers were distributed homogeneously in the eutectic by mixing in semi-solid state. The initial morphology represented as-mixed, was globular and made up of fragments of dendritic structure of primary α-phase with carbon fibers embedded in eutectic region around them. Heat treatment was applied to obtain an expanded α-phase which contains C_{sf}. It was also used to study the effect of morphology microstructure on the mechanical properties of composite. Fig. 4 shows the optical microscopy images of ax-extruded sample before and after T6 heat treatment which were etched by 5% vol. hydrofluoric acid. The effect of T6 heat treatment on the morphology and size of eutectic silicon was demonstrated from a planer to a fibrous shape. The needle-like iron intermetallic (β-Al_5FeSi) was identified easily in the eutectic region and appeared as small size and grey color under optical microscopy in the composite sample before heat treatment (Fig. 4a). The microstructure has been totally changed after T6 heat treatment as shown in Fig. 4b. The α-phase was grown and its size increased; this encompassed the carbon fibers and formed the homogeneous structure, so the C_{sf} were no longer located solely in the eutectic region, even in the spherical alpha phase. The silicon particle size was increased and became very regular in shape but the size of iron intermetallic compounds decreased during solutionizing. The silicon growth and re-shaping to a regular shape was illustrated after heat treatment. The micro-porosity was changed and converted to macro-porosity. The T6 heat treatment changes the acicular silicon to separated and rounded particles. The size of pores increased while the micro-pores fused; a further weakening of mechanical properties.

Fig. 5 shows SEM micrographs of a heat treated composite and some details of identified intermetallic phases at higher resolutions. As mentioned, the fibers were relocated in a uniform matrix especially in α-phase in contrast with microstructure of as-mixed composite due to growing of initial α-phase. Both Figs. 5a and 5b were captured when focusing on single filament at high resolution for samples with and without heat treatment, respectively. The interfacial adherence seems promising in the case of heat treated composite with well distributed intermetallic compounds particularly around or in contact with the fiber (Fig 5a). Heat treatment expanded initial α-phase and provides a uniform structure which the carbon fibers, silicon particles and intermetallic compounds were surrounded by expanded alpha phase as matrix. As suggested by other authors, in aluminum alloys with Mg content (0.5-2%) improve the wettability of aluminum on carbon fiber [2, 3]. Therefore, the localized high content of silicon and magnesium in the molten eutectic which was being sheared at semi-solid provides relatively strong adherence. The advantages of thixomixing compare to other conventional liquid compo-casting are high shearing load for dispersion and distribution of fibers and good adherence with lack of extra coating. The weaker adherence could be indicated in Fig. 5b at the fiber/matrix interface even with silicon deposition around the fiber. As shown, both silicon and iron intermetallics have not attached to carbon fiber perfectly and a tiny gap can be observed. This leads to a weak interfacial bonding. Thixomixing altered the dendritic form of alpha to globular under high shearing stress and also provide extra heterogeneous nucleation sites caused by rotation of the mixer. The eutectic contains acicular and tiny particles of Si and irregular shape of iron intermetallic. It has been assumed that, the heat treatment can be done to growing of alpha grains which encompassed C_{sf} with good interfacial bonding, but it had some deleterious impact on mechanical properties. The T6 heat treatment changed the morphology when the matrix hardening took place and led to higher hardness and strength and reduced the ductility as well due to the eutectic phase changed. The amount of microporosity contents was reduced by heat treatment and joined to form larger pores which are caused to lower mechanical properties.

Generally the wettability of carbon fiber with molten aluminum is poor but high local concentration of silicon in eutectic facilitated an early nucleation and precipitation over the carbon fibers [24]. Fig. 6 shows the EDAX mapping of A357/C_{sf} composite fabricated by thixomixing before and after heat treatment. Thermal conductivity of C_{sf} is higher than aluminum so leads to loss of heat in the fiber and its temperature becomes lower than that of surrounding molten eutectic, precipitation of silicon or intermetallic solidification occurred surrounding the fibers. The surface of C_{sf} could be heterogeneous nucleation sites for Si, when it nucleates in the eutectic compared to Al. The chemical compositions of the intermetallic were detected using EDAX. The presence of silicon and in some cases iron have been illustrated in the images and proved the hypothesis which described above. Silicon and magnesium were solidified on the C_{sf} outer surface in eutectic and the wettability of the molten aluminum on carbon was improved remarkably. This predominate disposition provides better adherence than fully liquid infiltration and prohibits deleterious formation of aluminum carbide. Poor wettability and weak adherence in other conventional methods such vacuum pressure infiltration, liquid casting or squeeze casting were improved by thixomixing at semi-solid state. The morphology of needle-like β-Al_5FeSi before T6 was changed, and reduced the deleterious effect of Fe on mechanical properties. However, the brittle intermetallic causes a reduction of tensile properties of Al/C composite because of the crack which might be propagate into an intermetallic and then propagates into matrix or adhering compounds on fibers and causes a premature fracture. Consequently, the formation and quantity of intermetallic formation must be controlled during fabrication of high strength Al/C_f composite.

3.4. Tensile properties

The tensile flow curves of A356 and A357 aluminum/C_{sf} reinforced metal matrix composites fabricated by thixomixing at 48% solid fraction were shown in Fig. 7a and 7b, especially before and after T6 heat treatment. The values of tensile properties such as yield stress, UTS and elastic modulus of the composite and matrix alloy samples along the direction of extrusion were tabulated in Table 4. Both matrix alloys were processed by the thixomixing without C_{sf} at the same parameters as the reference for comparison. At high speed of mixing (300 rpm), the strength of samples was decreased due to increasing of volume of porosity or more agglomeration of fibers which segregated at high rotating speed. However, different factors can be influenced by strengthening the materials. The tensile properties have differences between theoretical and practical values due to the high volume of pores which were entrapped in the composite and reduce the tensile strength especially when heat treated. The values of tensile tests were improved by increasing the volume fraction of fibers in the composite through the same fabrication process. It can be seen that the significant improvement in tensile strength was achieved and UTS marginally increased when the samples were heat treated. The differences in the thermal expansion coefficient between aluminum and carbon fiber can produce residual stresses during post fabrication treatment. The quenching process to the heat treatment can build-up residual stresses and high dislocation density strengthening by accumulation of dislocation next to the reinforcement particles or at the interface. The resulting mechanism leads to a strengthening effect by increasing the dislocation densities, especially at the interface of fiber/matrix or near to reinforcement particles. Dislocation density was increased by the cooling rate and related to the size and the volume fraction of reinforcement particles [25]. The strengthening mechanism in reinforced composite materials pertained to the load transferring from matrix to fiber which provided a load-bearing effect. The subsequent load transferring from the matrix to the carbon fiber pertained to the interfacial mechanical quality or adherence which improved by silicon deposition onto the fiber in thixomixing. The results of tensile testing in this study showed further increase in C_{sf} content. The increase in strength for both matrix alloys might be related to more agglomeration of fibers and more porosity. This is especially so when the samples were heat treated but this resulted in large difference between the measured and theoretical values. The strength values and elongation decreased for both A356 and A357 composite samples which fabricated at 300 rpm of rotational speed compare with other samples at lower speed of mixing. In the present study, the heat treated samples have shown higher tensile properties compared with those of composites which were not done. Furthermore, T6 heat treatment increases the grain size of matrix and increases elongation of composite. The strength, elongation and toughness might be improved by grain refinement in the metallic matrix.

The A357/C_{sf} composite exhibited better tensile properties than A356/C_{sf} in most of the samples, especially heat treated, which contains higher content of magnesium and so improves the wettability and provides stronger adherence due to the quality of the interfacial region. Some previous attempts have been conducted to improve the interfacial adherence by additional coating such as Cu, Ni or alumina onto the carbon fiber surface in aluminum matrix composites, whereas the properties of coated Al/C_{sf} composites were lower than the uncoated composite, although the extra coatings may possess lower mechanical properties [9, 23, 26, 27]. In the stress-stain flow curve shown in Fig. 7 both alloy composites demonstrated high fracture toughness with increasing volume fraction of C_{sf}, particularly when increased from 0 to 4.2 vol.%, which caused the composite to be ductile. The heat treatment declined the fracture toughness relative to the samples without heat treatment. In fact, shear banding debonding, fiber pull-out or fiber breakage consumed the fracture energy during matrix deformation, on the other hand C_{sf} functioned as a barrier which increased the fracture energy [28, 29].

3.5. Fracture behavior

The morphology of the fractured surface of fabricated composites which were conducted under standard tensile test show in Fig. 8. The quasi-plastic deformation is indicated in the matrix of both samples in Fig. 8a and 8b with dimple and no flat fracture. The fracture mechanism predominates for all of the samples in this study revealed as non-flat or ductile fractures with few fiber pull-outs. Conversely, a brittle structure might be produced, if the carbon fibers were not dispersed or distributed effectively. The agglomeration can be seen, in Fig. 8c due to the higher speed of mixing, cracks (indicated by pointer in figure) initiated locally from the colony of fibers and grow through the matrix and phases until the composite is broken. This phenomenon will happen also for porosities and the brittle phases such as Al_4C_3 which function as crack initiation sites. The good adherence of aluminum with carbon fibers caused plastic deformation. Thus, a few C_{sf} deboned and pulled-out were illustrated (Fig. 8d) due to better adherence and wettability by silicon. Some plastic deformation occurred around the fibers.

Fig. 9 illustrates the fracture morphology of thixomixed composites, with and without heat treatment at different magnifications. Neither alloy before heat treatment showed macro-pores. The samples containing the big holes were grown during heat treatment; this is due to the amount of trapped gas in the matrix. The micro-pores and air bubbles likely formed due to the volume fraction of carbon fiber and the high rotation speed of the slurry [30]. The hot extrusion process did not impact the porosity. Short carbon fiber might be cracked undesirably under intensive shearing load in the thixomixing process. The optimum length for short carbon fiber must be kept throughout the whole process of manufacturing [31]. Fig. 9a reveals the fracture surface with some fiber pull-out whilst some amount of fibers did not orient along the extrusion direction and cracked. The probability of composite failure is a function of filament length when loaded under specific stress [32]. As demonstrated by the illustration in Fig. 9b, the micro-cracks were initiated from the interface of matrix/reinforcement and the matrix deforms near the fibers and fibers are pulled-out relatively. Therefore it demonstrates when the load transmits from matrix to C_{sf} by interfacial bonding [26]. Silicon and intermetallic compounds' precipitation on the fiber leads to concentration of the stress, and micro-cracks form predominately near to fibers. These compounds strengthen the bonding, favoring plastic deformation. Many dimples and tearing ribs appeared for T6 heat treated A356/C_{sf} and A357/C_{sf} composite, as Fig. 9c and 9d show, with some short pull-out fibers. Big holes can be detected easily in both alloy composites after heat treatment which reduced the mechanical properties and tensile strength. The good interfacial bonding could be a barrier to passing the crack across the fiber, so some pull-out occurred in the fractured surface. The micro-cracks and cracked fibers can be seen in Fig. 9c due to high applied load during fabrication process and this was repeated across all tested samples. The severe plastic deformation occurred in A357/4.2vol.% C_{sf} where T6 caused the dimples around fibers and tearing ribs, marked by pointers in the figure.

3.6. *Nanoindentation analysis*

Ultra micro-hardness or nanoindentation was utilized across the line passing through the carbon fiber in different points (with an equal distance between them) in order to study the interface of matrix/fiber and to measure the hardness of matrix close to short carbon fiber. The hardness represents the capability of materials against deformation, particularly at the interface. The values for the above characterization were illustrated in Fig. 10 for the composite samples with and without heat treatment. It is clearly shown that the heat treated samples are stronger against deformation and the interfacial bonding adherence increased marginally. When the C_{sf} embedded in A357, the magnesium intermetallic was formed and also improved the hardness. This indicates that silicon and the intermetallic compounds precipitation improved the hardness of interface. The measured values of hardness at the distances far from C_{sf} increased markedly. Composite samples without heat treatment have a small impact on the hardness at the matrix/fiber interface; values in the range of 1800-2100 MPa as indicated in Fig. 10. This hardening effect at zones close to the fibers could be related to the favored deposition of alloying elements in the matrix during thixomixing or improvement of interfacial adherence associated with the thermal evolution of compounds after heat treatment. The interfacial bonding was improved by thixomixing due to the solidification mechanism caused by segregation of alloying elements between initial alpha phase and eutectic. This predominate deposition was modified for further heat treatment as shown in hardness values of nanoindentation.

3.7. X-ray diffraction

The XRD pattern of fabricated composite by thixomixing was shown in Fig. 11 and revealed that the iron intermetallic ($Al_9Fe_2Si_2$) formed. The presence of Al_4C_3 and β-Al_5FeSi compounds cannot be detected by X-ray due to very few amount of concentration and the similarity and lower intensity of peaks related to them. The iron intermetallic might be a result of the reaction of aluminum with thus steel body of the die, or with the iron content in the initial alloy. It is possible to control the intermetallic formation by coating or selecting with sustainable and suitable material for the die in further experiments. The ternary Al–Fe–Si phases, such as monoclinic β-Al_5FeSi, are very common intermetallic compound in Al–Si based alloys. The Manganese in the alloy consumes iron and stabilizes by form an equilibrium quaternary phase which can be helpful to improve the ductility [33]. The intensity of peaks which corresponds to silicon and the intermetallic compounds was reduced after T6 heat treatment relatively to the same peaks belong to A356 as shown in spectra.

4. Conclusion

- Thixomixing process has been innovated to fabricate aluminum silicon/short carbon fiber composites for dispersion and distribution of reinforcement by intensive shearing of semisolid slurry. Microstructural evidence clearly indicated that C_{sf} dispersed and distributed homogeneously in the matrix with good interfacial bonding by predominate precipitation of alloying elements. Additionally, mixing under the above conditions produced good wettability, few damages on the fibers, inhibition of aluminum carbide formation and improvement on microstructure of matrix.

- The gas entrapment and porosity occurred under thixomixing, and post fabrication processes such as semi solid extrusion or casting could provide reasonable solutions. In contrast, the hot extrusion which was done in this study had no effective impact on random distributed C_{sf} to be aligned across the extrusion direction as represented in microstructure of composite samples and fractured surfaces.
- Heat treatment on both alloys modified the microstructure and improved the interfacial bonding between fiber and matrix. The intermetallic compounds were also modified to achieve better mechanical properties. The SEM images satisfied the dispersion and distribution of reinforcement into the matrix with good wettability and better adherence.
- Tensile strength and mechanical properties marginally improved via the increase of volume fraction of reinforcement and the heat treatment process. The ultimate tensile strength of T6 heat treated samples was improved about 20-24% rather than those not heat treated. However, the fiber increased the modulus of the composite by 5-10%.
- In the XRD analysis carried out to demonstrate the intermetallic which can be formed during the process for further specifications, no evidence was found for the presence of aluminum carbide formed by thixomixing.
- The thixotropic mixer can be useful for fabrication of various metal composites, but future studies of further characterizations and developments are essential.

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Table 1 Physical properties of PAN based M40JB carbon fiber

Specification	Value
Diameter of fiber (µm)	5.2
Number of filament	12000
Tensile strength (MPa)	4400
Tensile modulus (Gpa)	377
Elongation (%)	1.2
Mas per unit length tex (g/1000m)	454.5
Density (g/cm^3)	1.75

Table 2 Chemical composition of the A356 and A357 alloys (weigh %)

Alloy	Si	Mg	Cu	Fe	Ti	Zn	Mn	Al
A356	7.12	0.335	0.0025	0.0218	0.104	0.1	0.0049	Balance
A357	6.76	0.513	0.0011	0.1431	0.123	0.1	0.0056	Balance

Table 3 Experiment condition of thixomixing method with hardness and porosity values

Test number	Alloy	Solid fraction (%)	Speed of mixer (rpm)	C_{sf} fraction (%)	Hardness (HBW)	Porosity (%)
1	A356	48	100	0	73	2.8
2	A356	48	300	8.1	64	5.7
3	A356	48	100	4.2	72	3.6
4	A356	48	100	8.1	75	4.4
5	A356-T6	48	100	4.2	87	3.8
6	A356-T6	48	100	8.1	84	4.1
1	A357	48	100	0	76	2.7
2	A357	48	300	8.1	66	6.1
3	A357	48	100	4.2	73	3.1
4	A357	48	100	8.1	71	4.7
5	A357-T6	48	100	4.2	88	4.2
6	A357-T6	48	100	8.1	91	4.8

Table 4 Tensile properties of composites and aluminum alloy

Alloy	Yield stress (MPa)	UTS (MPa)	Elastic modulus (GPa)
A356	178	223.4	71.2
A356/8.1% C_f-300	164	176.8	69.1
A356/4.2% C_f	147	183.4	66.5
A356/8.1% C_f	158	191.6	69.0
A356/4.2% C_f +T6	176	220.1	81.4
A356/8.1% C_f +T6	188	238.2	83.3
A357	182	219.1	72.3
A357/8.1% C_f-300	124	160.2	65.7
A357/4.2% C_f	158	208.2	66.2
A357/8.1% C_f	167	222.4	72.1
A357/4.2% C_f +T6	198	240.3	85.2
A357/8.1% C_f +T6	224	261.8	86.4