Thixomixing as Novel Method for Fabrication Aluminum Composite with Carbon and Alumina Fibers

Ebrahim Akbarzadeh, Josep A. Picas Barrachina, Maite Baile Puig

Abstract—This study focuses on a novel method for dispersion and distribution of reinforcement under high intensive shear stress to produce metal composites. The polyacrylonitrile (PAN)-based short carbon fiber (C_{sf}) and Nextel 610 alumina fiber were dispersed under high intensive shearing at mushy zone in semi-solid of A356 by a novel method. The bundles and clusters were embedded by infiltration of slurry into the clusters, thus leading to a uniform microstructure. The fibers were embedded homogenously into the aluminum around 576-580°C with around 46% of solid fraction. Other experiments at 615°C and 568°C which are contained 0% and 90% solid respectively were not successful for dispersion and infiltration of aluminum into bundles of C_{sf}. The alumina fiber has been cracked by high shearing load. The morphologies and crystalline phase were evaluated by SEM and XRD. The adopted thixo-process effectively improved the adherence and distribution of C_{sf} into Al that can be developed to produce various composites by thixomixing.

Keywords—Aluminum, carbon fiber, alumina fiber, thixomixing, adhesion.

I. INTRODUCTION

THIS lightness with high strength convinced engineers to design the cost saving metal composites. Since 1960, various methods are being projected for fabrication of aluminum composite and carbon fibers [1]-[4]. Liquid metal routs and powder metallurgy as main conventional methods are most costly and less effective for aluminum carbon fibers composites. Aluminum melt casting leads to flotation and segregation of fibers due to the difference in density; moreover, less wettability and weak adherence at interface were detected [5]. High load pressing are essential in powder metallurgy process whereby undesirable cracking of fibers occurs. Good adhesion at fiber/matrix interface and homogenous distribution of reinforcement are as main technical problems, remained unsolved [6], [7]. An effective interfacial bonding between matrix and reinforcement for successful load transferring is essential in a good MMC. Another challenge for Al/C_{sf} composite has been to disperse and align fibers in order to increasing of strength was

attributed to uniform distribution. Last attempts have been reported, the agglomeration of particulates and non-dispersed induces unwanted brittle nature in composite due to as predominate crack initiation sites [8].

The long processing methods are not economical and cause to the chemical reactions like brittle phases at the interface of bonding [9]. Magnesium and silicon may improve the wettability of aluminum on carbon by formation of suitable intermetallic phase [7], [10]. To the best of our knowledge, the thixomixing method was not being utilized for distribution of C_{sf} or other fibers into aluminum matrix. Basically, it is an application of high shear stress (τ) on solid particulate embedded in liquid metal to overcome the strength of dendritic structure.

In this study, a novel thixo-process was developed to dispersion and mixing of reinforcements in aluminum at semi-solid condition by high shearing rate. The effects of shearing and temperature were studied on wettability and dispersion of fibers. The improved structure, less damages of fibers, no flotation or segregation, short process times, effective controlling on temperature, low cost and good distribution and dispersion of fiber bundles can be mentioned as advantages of this process.

II. MATERIALS AND EXPERIMENTAL

A. Samples Preparation

The polyacrylonitrile (PAN)-based carbon fiber T300 made by Japan Toray Co. Ltd and Nextel 610 alumina fiber of 3M Co. were chopped into 5±1 mm length then the agent sizing on the fibers removed by heating in 500°C for 30 minutes in aerated furnace. The physical properties of C_{sf} and alumina fiber have shown in Table I. A356 were chosen as matrix with chemical composition Al–7.12Si–0.335Mg–0.003Cu–0.05Fe–0.005Mn–0.1Zn–0.1Ti, in wt. %.

A specific mixer has been designed for thixomixing process of semi-solid of aluminum alloy and reinforcements as illustrated in Figs. 1 (a) and (c). In this study, mixer was designed near to Searle rheometer where the inner cylinder is rotating and the outer cylinder is stationary with some changes. The gap between internal wall of die and rotor was 2 mm which provides high shear rate ($\gamma°$) and shear stress. The die and rotor were made of H13 steel alloy and heat treated for better hardness and toughness, then combined with CNC machine for precision vertical movement and various rotation speed of rotor. First of the sampling, die and rotor preheated

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up to 700ºC then molten A356 poured into the die. The temperature of die, rotor and aluminum must be maintained for thermal stabilization to form semi-solid slurry at different solid fraction (0%, 48%, and 90% solid). The chopped carbon fibers added gradually to the mixer to prevent agglomeration and mixing continued at constant situation as mentioned for 5 minutes. Finally, rotor removed out the die and mixture was cooled down to the room temperature. The samples were removed out and re-melted to form as bar or ingot for further process or characterization. Specimens for microstructural characterization were cut from different positions of the sample after mixing. The morphology and distribution of fibers in the mixed and remelted samples were characterized using SEM (Jeol JSM-5600). The intermetallic and compounds in samples were identified by XRD (Siemens D500 instrument with CuKa radiation)

TABLE I
PHYSICAL PROPERTIES OF CARBON FIBER

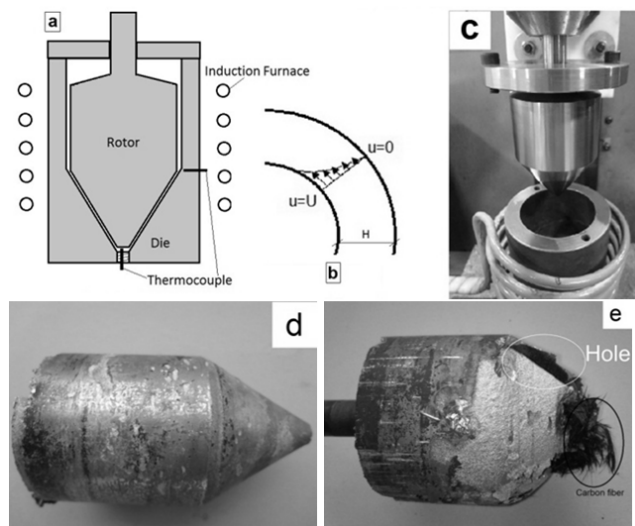| Type	Tensile Strength (MPa)	Tensile Modulus (Gpa)	Elongation (%)	Density (g/cm³)
M40JB	4400	377	1.2	1.75
Nextel 610	3077	380	-	3.88

Fig. 1 A schematic illustration of designed thixomixer, the inner part (Rotor) is rotating and the outer cylinder (Die) is stationary (a) and schematic of flow in present system in the gap between two parallel plates (b), real image of mixer (c), the successful sample mixed at semisolid with 48% (d) and unsuccessful with 90% solid content (e)

III. RESULTS AND DISCUSSION

As it mentioned, the goal of this study is developing a new method for Al composite reinforced by short carbon fiber or alumina fiber fabrication without crack of fibers and having better dispersion and distribution. In Figs. 1 (d) and (e) shown the fabricated samples, which were successful and unsuccessful (90% solid), respectively by thixomixing. The fully liquid of aluminum is not sufficient for dispersion and distribution and fibers were agglomerated at bottom of die while shear stress produced. The dark spots on the sample

showed in Fig. 1 (d) are iron oxide residue from die. Whereas, as shown clearly in Fig. 1 (e) for 90% solid, big holes was created with no infiltration of aluminum into fibers. The semisolid slurry in this case, just surrounds the cluster of fibers with no infiltration then fibers were agglomerated and cracked by solid particles. Homogeneous distribution and well dispersion are highly recommended to get the superior strength with suitable ductility.

Thixotropic supposed as gel, when sheared becomes liquid and reverts back when static. Successful dispersion of C_{sf} within the semisolid of aluminum in microscopic scale can be clearly seen in Fig. 2 (a). Nucleation of primary phase (α-Al) started in the liquid just below liquidus at lower temperature to semisolid state where a distance away from the fibers and surrounded by eutectic. In other words, the mushy flow of aluminum sheared and overcome to the agglomeration between carbon fiber filaments, so the primary Alpha can entrap the fibers and disperse them and eutectic surrounded over fibers. The Si in eutectic detected in a very fine size in this method, with an average size less around 1 μm (Fig. 2 (c)). Fiber to fiber connections and agglomeration leads to a non-homogeneity and lower macroscopic mechanical properties. The formation of such fine Si particles also has been observed before in rheo-process for ceramic composite [11]. Fine and homogeneous microstructure achieved by this method is expected to improve simultaneously the tensile strength and ductility, unlike conventional MMC fabrication methods.

The wettability of carbon fiber with Al liquid is generally poor but last attempts in semi-solid conditions approved better adherence and wettability, may referred to high silicon concentration in eutectic. The growth of α-Al phase leads to enrichment of Si content in the eutectic. The C_{sf} can act as heterogeneous nucleation sites for Silicon.

The convectional mixer with impeller may damage fibers undesirably, which are less effective for dispersion of fiber clusters. Agglomeration may occur by small particles or specious which have affinity to clump together and increase their total surface area. In this study has been found that the well dispersion and homogeneous distribution of C_{sf} can be achieved by intensive shearing of matrix associated with the production of MMCs. A novel mixer based on thixotropy provides extremely applications for mixture of various reinforcements such as ceramic particles, CNT, nano-particles with metal matrix such as aluminum, magnesium or steel. The semisolid state shows non-Newtonian or viscoplastic behavior which depends on the yield strength and the shear rate. Here, yield strength means the maximum shear stress at which the shearing begins, and mainly depends on the solid faction or temperature of the semisolid alloy [12]. In thixomixing, the semisolid slurry is sheared continuously and exhibits a complex non-Newtonian flow behavior. Laminar flow, induced in the liquid by the rotor, is associated normally with high viscosity fluids. Fiber agglomerates break-up within the high shearing, which can lead to de-agglomeration and homogenization. Optimum speed of rotor is necessary to distribute the C_{sf}, because at higher rotating speed, the solid

particles will be segregated from liquidus by the centrifugal force.

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 is shear stress, 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 [13]. 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 [14]. 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 (3)

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

where, η_a is apparent viscosity that can be calculated.

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

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

The alumina fiber is more fragile than carbon fiber and was shown that the filaments of alumina fibers have been broken by high intensive shearing of semisolid slurry. Fig. 3 has shown the microstructural of aluminum composite which short alumina fibers have been embedded into the matrix, but the fibers have been broken and small particles of alumina distributed in the matrix. The fragility of alumina fiber was observed clearly and shows that the thixomixing with high shearing is not sufficient for distribution of fragile fibers or particles.

SEM images at high resolution have shown relatively good adherence at the interface of Al/C$_{sf}$ for fibers are embedded in Al matrix in most of the samples (Fig. 2 (c)). The thixomixing process under intensive shearing resulted in absence of chemical reactions between the fibers and matrix due to short time involved in mixing process. Prohibition of forming the fragile intermetallics such as aluminum carbide (Al_4C_3) is highly desirable from the mechanical property viewpoints. The alloys with at least 7 wt.% Si content is needed to prevent or delay the formation of aluminum carbide at temperatures up to 750°C while aluminum exposure with carbon [15].
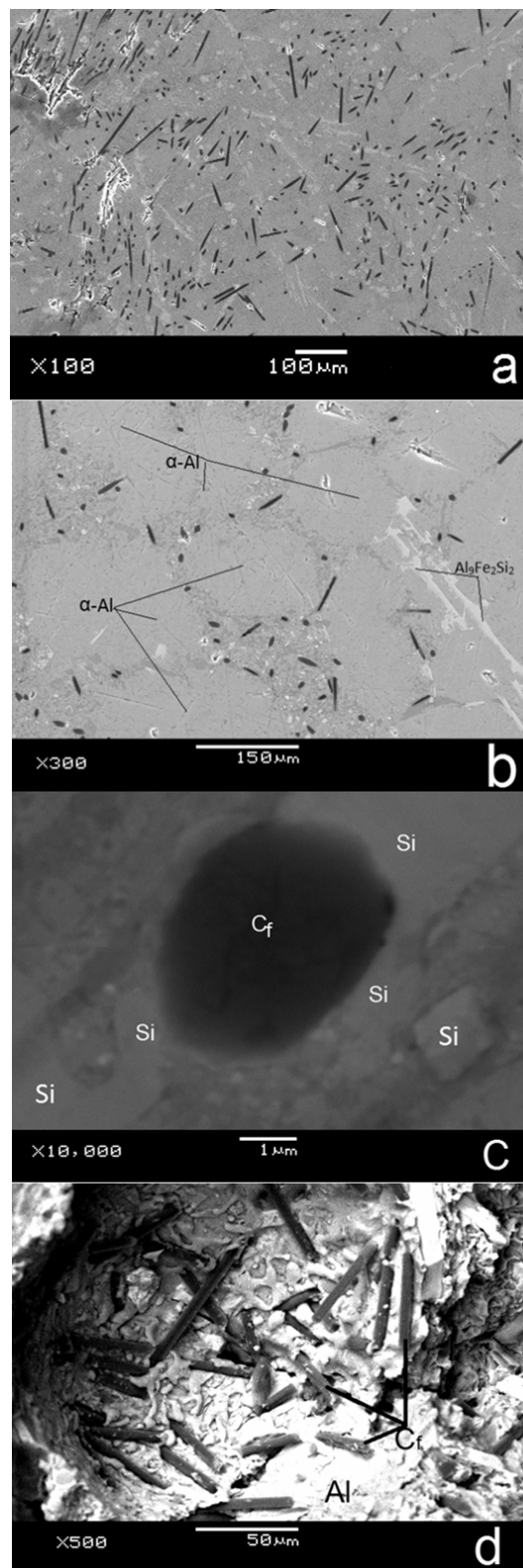

Fig. 2 Microstructure of successful A356/C$_{sf}$ composite showing random orientation of fibers in the aluminum matrix (a) ×100, (b) in ×300, microstructure of interface of C$_{sf}$ and aluminum matrix (c), Fractured surface of composite which shown fibers in matrix (d)
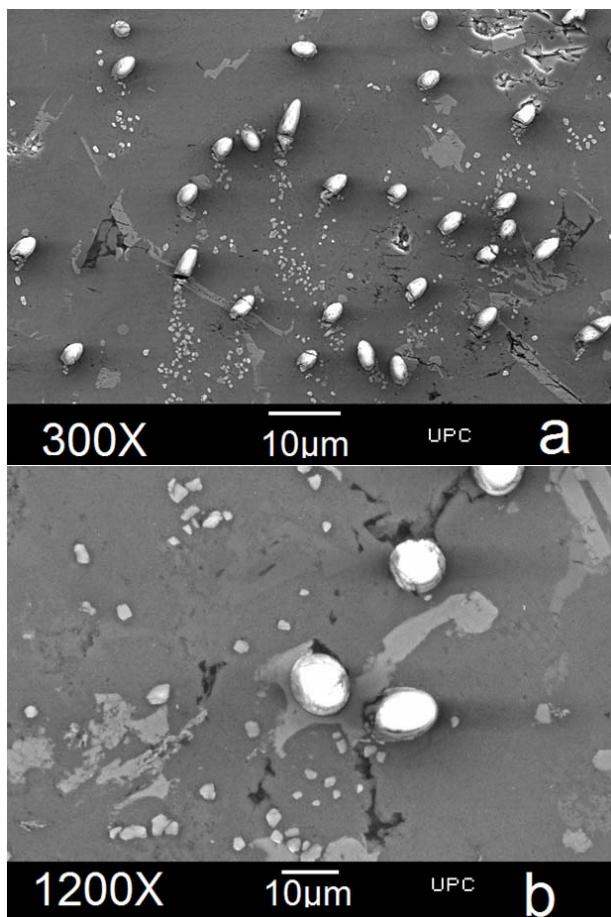
Fig. 3 Microstructure of successful Al/alumina fiber composite showing breakage of alumina fiber in aluminum matrix (a) ×300, (b) ×1200

In Fig. 2 (d) has shown the fractured surface of composite that fibers embedded in eutectic. Some plastic deformation investigated as good adherence of aluminum with carbon fibers. The relative surface area of composite can be taken equal to the volume fraction of the C_{sf}, so the statistical image analysis used for several SEM images provided that the average volume fraction of C_{sf} in successful composites is 8 ± 1 vol. %. High level of porosity was observed in samples, may be resulted from air bubbles which arrested between filaments in bundle or air sucked during mixing into the melt. The porosities will be reduced or removed by further processes such as extrusion or forging.

The XRD pattern of fabricated composite by thixomixing was shown in Fig. 4 revealed that the formation of Al_4C_3 has not been occurred while the iron intermetallic ($Al_9Fe_2Si_2$) formed as result of reaction of aluminum with steel die. It can be controlled by coating or material selection for die in further experiments. In most common alloys of aluminum the presence of iron is harmful for mechanical properties and especially ductility due to the low solubility of Fe in the α-Al solid solution phase and its affinity to form Fe-containing compounds [16]. The ternary Al–Fe–Si phases, such as monoclinic β-AlFeSi and the hexagonal α-AlFeSi, are very common intermetallic compound in Al–Si based alloys. The presence of Mn can be helpful to improve the ductility of the alloys by forming an equilibrium quaternary phase which stabilizes and consumes Fe.
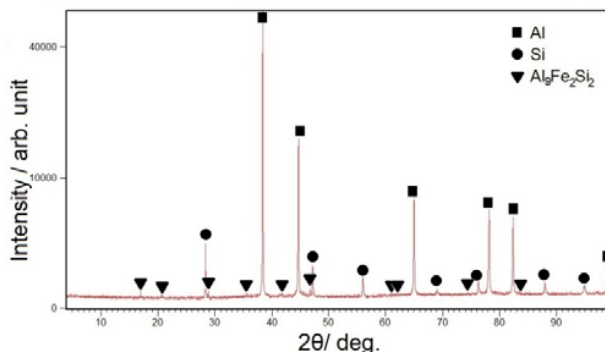

Fig. 4 XRD plots for A356 reinforced with short carbon fiber showing iron-containing intermetallic from thixomixer during process and not contains Al_4C_3

IV. CONCLUSION

Thixomixing process has been innovated to fabricate metal composite of A356 with 8 ± 1 vol. % C_{sf} by shearing semisolid slurry. It was demonstrated that high shear stress and laminar flow of aluminum semi-solid dispersed the bundles of C_{sf} and distributed them into matrix thus leading to a uniform microstructure. Additionally, mixing at above condition seems better wettability, few damages on the fibers, prohibition of aluminum carbide formation and improvement on microstructure of matrix. The SEM images satisfied the dispersion and distribution of reinforcement into matrix with good adherence. Alumina fiber was cracked during the process by high shear load. The XRD analysis carried out to demonstrate the intermetallic which can be formed during the process for further specifications.

A novel thixomixing opened a new rout for metal composite production so further characterizations and developments are essentially to be studied in future.

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