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Production of high performance AA7150-1% SiC nanocomposite by novel fabrication process of ultrasonication assisted stir casting

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ABSTRACT

The effect of ultrasonic vibration treatment on nanoparticle distribution was successfully investigated and developed a novel fabrication process to produce nano silicon carbide particle reinforced AA7150-1% SiC nanocomposite through a combination of the vortex, double stir casting, and ultrasonic vibration techniques. Ultrasonic frequency of 20 KHz and with a power capacity of 1000 W was used in the process. Ultrasonic probe was used for proper mixing of the nanoparticles in the molten bath. Microstructure investigation of grain formation, particle distribution, and fracture surface was analyzed through an optical and scanning electron microscope at the as-cast condition. Energy dispersive spectroscopy was used for determining chemical composition of the nanocomposite. In the novel fabrication process, the influence of sonication effect on material properties such as porosity, microhardness, tensile strength were examined and compared with double stir casted nanocomposite material as well as the base material. Mechanical properties of AA7150-1% SiC novel fabrication process were enhanced with a reported increase of 26.05% in tensile strength, and 10.85% in microhardness. 74.1% reduction in porosity as compared to the base alloy. In the double stir casting process, there was 19.6% increase in tensile strength, 2.9% of improvement in microhardness, and 46.96% reduction in porosity as compared to base material properties. The enhancement of material properties with the ultrasonic probe assisted novel fabrication process are attributed to grain refinement of composite and homogeneous distribution of SiC nanoparticles due to the acoustic streaming and cavitation effect.

1. Introduction

Ceramic particle reinforced aluminium alloy matrix composites are widely used in aerospace and automobile industries. Al-Zn-Mg-Cu alloys are used especially in upper and lower wings, fuselage and stringers of aeroplane due to high specific modulus and strength to weight ratio [27,30,32]. Silicon carbide particles are an attractive choice as ceramic reinforcement for lightweight alloy matrix due to their desirable characteristics such as high hardness, high melting point, good thermal stability, low thermal coefficient of expansion, high corrosive resistance and low density [21,29]. The strength of aluminium alloy matrix composite depends on the size of reinforcement and interparticle spacing. The micro ceramic particles, which are incorporated as reinforcement into liquid metal matrix to manufacture the composite material for significant improvement in strength of composite material but decreases the elongation. Recently nano ceramic particles were introduced to fabricate metal matrix composites and proven that the effect of size of ceramic reinforcements is playing a crucial role in the composite material properties and distinctly enhancing the base material properties while maintaining beneficial elongation as well as high resistance to temperature creep [9,12,34]. The quality of metal matrix nanocomposites fully depends on the ceramic nanoparticles distribution and dispersion into the liquid metal.

The material properties of metal matrix composites mainly depending upon the selection of production process and processing methods plays a crucial role to satisfy the demands of industries as well as required functional properties [23]. The main disadvantage of preparing aluminium metal matrix composites is heterogeneous distribution of ceramic nanoparticles in molten liquid, higher cost of nanoparticles and initial investment cost. The cost effective fabrication techniques of nanocomposites are essential for elaborate their field of applications [14]. The leading cost effective methods for fabrication of bulk metal matrix composites are stir casting, infiltration, compo casting and powder metallurgy. However, the major problem with externally adding of the nanoparticles to molten liquid is difficult due to high specific volume ratio and poor wettability between matrix and

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Table 1
Chemical composition of AA7150.

Element	Si	Mg	Mn	Cu	Fe	Zn	Zr	Al
Wt. Percentage	0.08	2.56	0.009	2.25	0.12	6.37	0.11	Balance

ceramic reinforcements [20]. These ceramic nanoparticles are easy to float on the molten liquid surface and form agglomerations in liquid metal. To overcome this problem, vortex is one of the accepted methods for introducing the ceramic nanoparticles into the dense liquid metal to avoid the floating of nanoparticles and redirect into the melt pool through mechanical stirring process. This stirrer was vigorously stirred to form a vortex in the molten liquid and then nanoparticles were incorporated at the side of vortex form [16].

Mehrdad Shayan et al. [26] fabricated hybrid Al2024-SiO_2-TiO_2 nanocomposites through stir casting technique and examined their mechanical as well as structural properties. The grain refinement was observed and increases with increase of nanoparticles content and considerable improvement of 36 and 30% in tensile strength and hardness properties of nanocomposites. Hamid Reza Ezatpour et al. [8] fabricated Al6061 based nanocomposite through stir casting technique and investigated effect of Al_2O_3 ceramic nanoparticles on mechanical as well as microstructure properties of Al6061-Al_2O_3 nanocomposites and compared with extruded sample results. Double stir casting technique (DS) is used for the distribution of ceramic particles into molten liquid. A.V Pozdniakov et al. [24] fabricated AA6063-5%B_4C composites through double stir casting process and nanoparticles were introduced with vortex technique into molten liquid. It was noticed that the particle distribution and mechanical properties enhanced with yield strength (425 MPa) as well as ultimate strength (455 MPa).

Alaneme and Aluko [2] introduced silicon carbide particles into Al-Mg-Si alloy and obtained enhanced properties as well as developed uniform distribution of silicon carbide particles throughout the composite. Yang et al. [17] introduced a new manufacturing process with ultrasonic vibration which combines the effect of acoustic streaming with cavitation. Ultrasonic vibration [18] is a widely used technique in aluminium alloy based composites to ensure uniform dispersion of micro/nano-sized particles as well as enhance wettability between the matrix to ceramic reinforcements with reduced porosity [19]. The ultrasonic vibration creates a large number of bubbles with high-intensity waves which have transient “micro-hot-spots” (temperature > 5000 °C and atmosphere > 1000 atm, heating and cooling rate of 10^{10} K/s) [28]. Due to this temperature and pressure difference, the bobble explodes and break up the clusters and acoustic streaming spreads the particles throughout the melt [4].

Nampoothiri et al. [22] introduced the ultrasonic treatment into the aluminium alloy melt to fabricate Al-4.4Cu/$2TiB_2$ nanocomposites. The results of the compressive strength of ultrasonically treated nanocomposites are two times of the base aluminium alloy. Salehi et al. [25] produced Al-SiO_2 nanocomposite foams through ultrasonic assisted stir casting method and found that the Al-0.75 wt% SiO_2 nanocomposite exhibits the highest hardness and stresses more than 3 times of base material. Absar et al. [1] developed a flow pattern to evaluate acoustic streaming on different geometrical models of ultrasonication processing cell. The better geometrical shape was obtained to produce a large acoustic stream. The validation of the model was investigated and achieved by two different tests such as processing of metal matrix nanocomposites and sedimentation. The most stable distribution of CNEs in water is achieved at 43 h. It is also shown the uniform distribution of nanoparticles at optimal parameters and effect of ultrasonic acoustic streaming flow pattern. Harichandran and Selvakumar [10], Kai et al. [13] and Wang et al. [31] fabricated hybrid nanocomposites through ultrasonic assisted stir casting by various weight percentages of nanoparticle reinforcement and observed the significant improvement of material properties and microstructure refinements due to acoustic and cavitation effect as compared to base alloy material. Zhang and Nastac [35] investigated the effect of sonication process during the fabrication of Al6061-1 wt% SiC nanocomposite and applied CFD model to analyze the degassing and refinement of grains in nanocomposite material. Kannan and Ramanujam [15] fabricated Al7075-Al_2O_3-hBN based nanocomposite material and evaluated the effectiveness of processing technique as well as heat treatment on material properties and observed the significant enhancement in microstructure and mechanical properties.

In this work, a novel fabrication process was developed by combining vortex, double stir casting and ultrasonic vibration treatment to enhance the homogeneous distribution of nanoparticles in AA7150-1% SiC nanocomposite. The mechanical as well as microstructural properties were investigated and compared with double stir casting AA7150-1%SiC nanocomposite and base material.

2. Materials and methods

Matrix material chosen was AA7150 (Al-Mg-Zn-Cu Alloy) Ingots and nano Silicon Carbide (SiC) ceramic particles having 40–60 nm average particle size and > 99.9% purity as reinforcements. The density of AA7150 and Silicon Carbide nanoparticles is 2.83 g/cc and 3.92 g/cc. Table 1 shows the chemical composition of aluminium 7150 alloys. Mg was used for improving the wettability for matrix and reinforcement, while C_2Cl_6 tablet was used for degassing aluminium alloy melt pool.

Fig. 1a and b show the schematic diagram of the fabrication methods for ceramic particle dispersion while adding by a vortex formed double stir casting (mechanical mixing) and ultrasonic

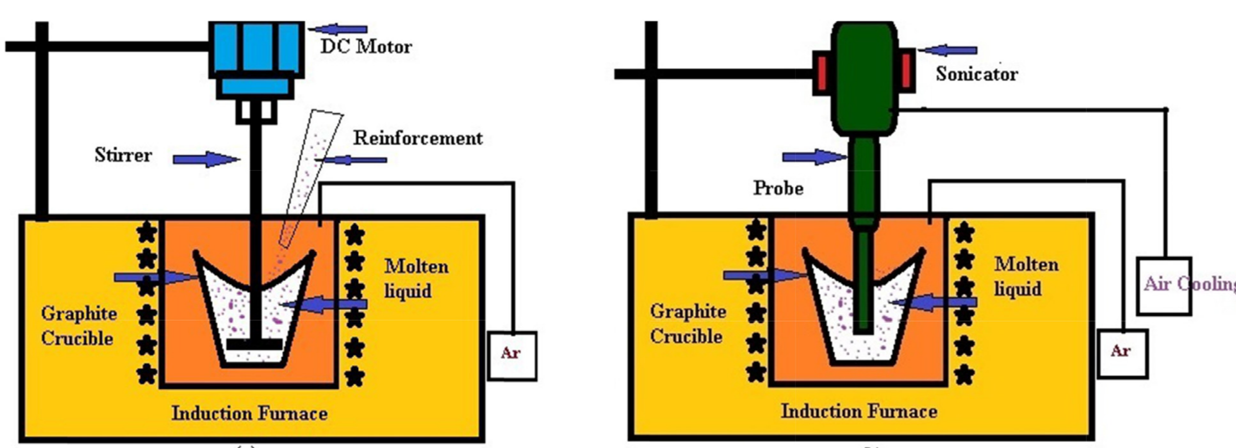


Fig. 1. Schematic Diagram of (a) Stir casting process (b) Ultrasonication process.

Table 2
Average grain size and mechanical properties of base alloy and nanocomposites.

Composition	Density (g/cc)	Porosity (%)	Hardness (HV)	UTM (MPa)	Average Grain Size (μm)
AA7150	2.6967	4.71	154	114.4	68
AA7150-1% SiC Double Stir Casting Process	2.7678	2.3	165.9	120.6	29
AA7150-1% SiC Novel Process	2.7984	1.22	170.7	144.2	24

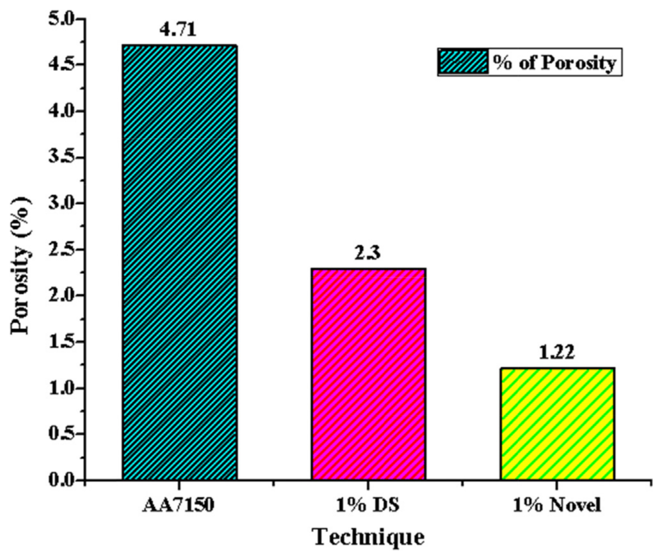
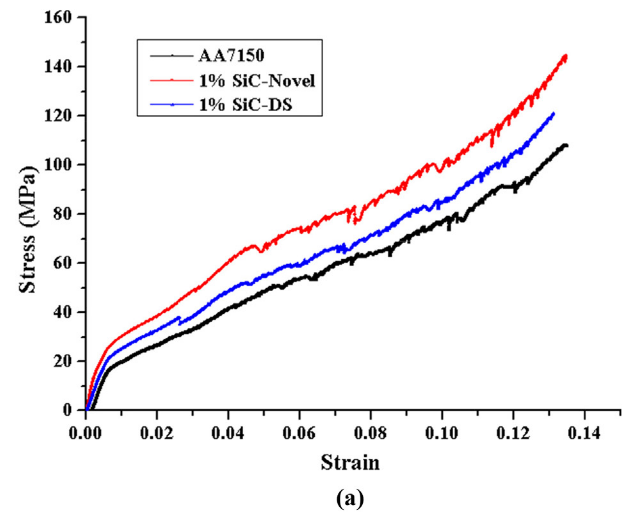


Fig. 2. Porosity graph for AA7150, DS process AA7150-1% SiC, Novel process AA7150-1% SiC.



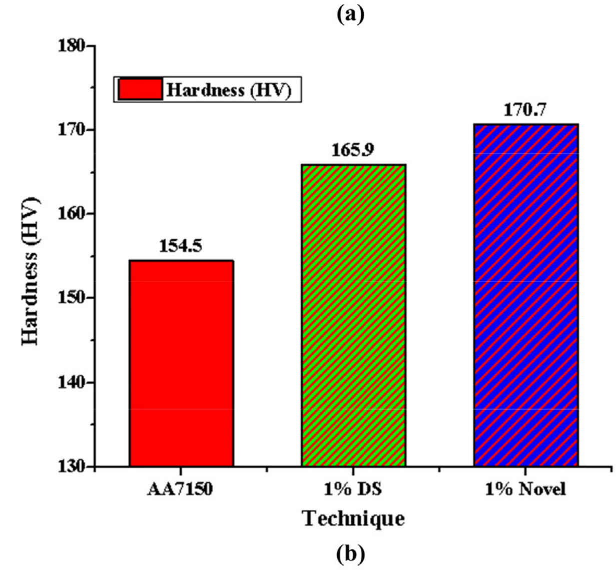


Fig. 3. (a) Stress-Strain graph (b) Hardness graph for AA7150, DS AA7150-1% SiC and novel AA7150-1% SiC.

vibration. The mixing of nanoparticles is very difficult through conventional mechanical rotary impeller due to the high surface area to volume ratio of nanoparticles. Another challenge is the floating of nanoparticles and cluster formation before mixing into molten liquid due to the poor wetting property of ultra-fine particles with dense liquid. To overcome these challenges during the process, nanoceramic particles are incorporated at 750 °C of aluminium melt through mechanical rotary impeller by vortex method. Stirrer speed has been adjusted to form a vortex and it may depend on the dimensions of stirrer blade and molten liquid temperature. Good quality composites can be produced by this method if there is proper selection of process parameters such as pouring temperature, stirring speed and preheating temperature of reinforcement. The preheated temperature of reinforcement nanoparticles leads to reduction in oxidation and cluster formation.

Double stir casting is meant to improve the efficiency of the stir casting process. The process involves two step stirring process with 20 min. Step-I: Molten liquid is stirred (650 °C) for 10 min and following that, molten liquid is allowed to attain semisolid state at low temperature and then reheated to above liquidus temperature. This semisolid state reduces cluster formation and settles the nanoparticles. Step-II: 10 min of the mechanical rotary stirring process is designed to enhance the dispersion of ceramic nanoparticles by breaking the sub-micron clusters, and agglomerations to improve wettability. The wettability of nanoparticles to matrix and flowability of metal liquid also increases as temperature increases [3,5]. This process is conducted at a liquidus temperature of over 750 °C; therefore this temperature leads to enhanced fluidity of a molten liquid and wettability property. At this temperature, metal liquid is allowed to infiltrate ceramic nanoparticles, clusters, and agglomerations, which leads to reduction in force (shear force, which is created by mechanical rotary impeller while mixing the ceramic nanoparticles) required to break the ceramic nanoparticle agglomerations/clusters and spread around the melt.

After the double stir casting process, ultrasonic vibrator (probe) is used to distribute the ceramic nanoparticles into the metal pool for homogeneous mixing. The probe is placed at 3/4th depth of liquid and is allowed to vibrate. These vibrations create high energy waves, which form a large number of cavitation bubbles in the molten pool. These cavitation bubbles contain high atmospheric pressure (> 1000 atm) and high temperature (> 5000 °C) which explode within microseconds due to the pressure difference and break the clusters, and agglomerations as well as spread the agitated nanoparticles homogeneously throughout the liquid metal. The processed metal liquid is poured into preheated (500 °C) die steel mold to avoid stress concentration and it is allowed to cool at room temperature. Cast aluminium blocks were prepared for tensile and hardness test as per ASTM E8M and ASTM E92 standards.

Microstructure analysis was investigated through an optical microscope for grain formation and its refinement. Nanoparticle distribution was studied through a scanning electron microscope. Elemental analysis was made through Energy Dispersive X-ray Spectroscopy (EDS) for

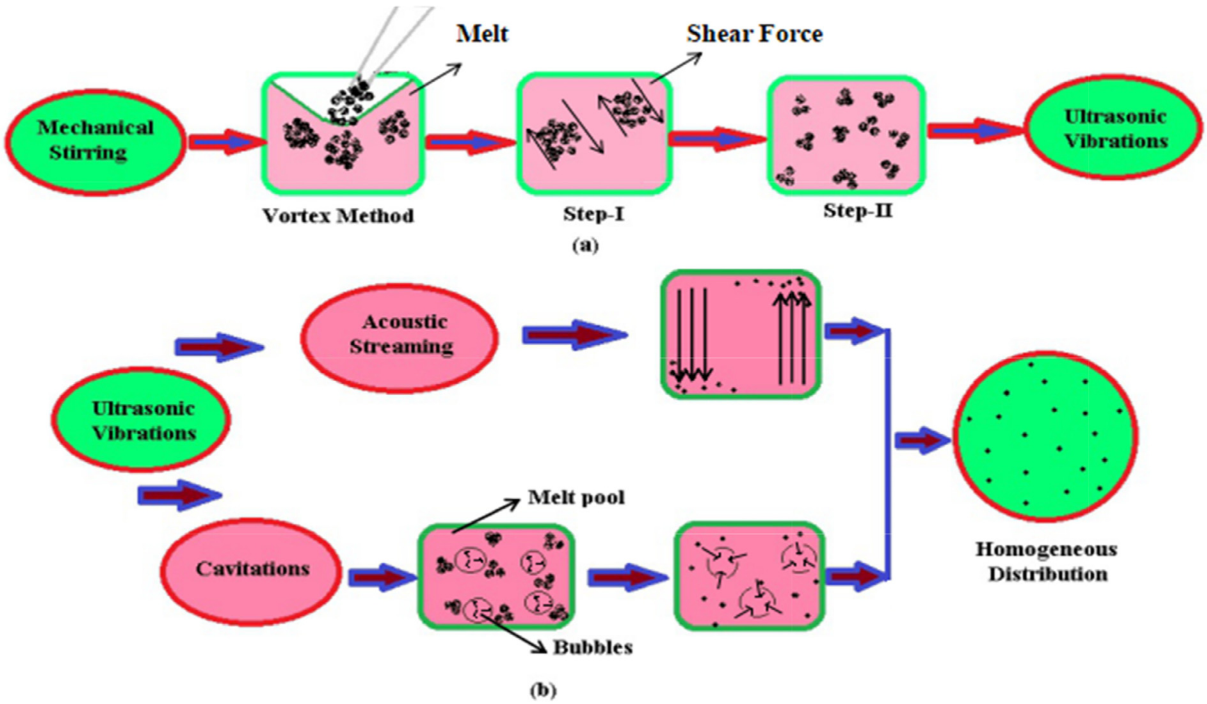


Fig. 4. Sketch of novel fabrication process.

matrix and nanocomposite. The mechanical properties such as hardness and tensile strength were investigated through Vickers hardness tester and uni-axial tensile machine. The specimens were machined from cast blocks using turning lathe: the tensile sample had a diameter of 9 mm and gauge length of 45 mm. The tensile fracture surface of failure surfaces was analyzed using scanning electronic microscope.

3. Results and discussions

3.1. Material properties

3.1.1. Density and porosity

The experimental density was obtained by Archimedes' Principle: "$\rho = \frac{m_1}{m_1 - m_2}$"; where, m_1 is the sample weight in the air, m_2 is the sample weight in water. The percentage of porosity was calculated using the equation below [18]:

$$porosity = \left[1 - \frac{\rho_{mc}}{\rho_m\left(1 - \frac{w\rho_{mc}}{\rho_p}\right) + w\rho_{mc}}\right]*100$$

where ρ_m, ρ_{mc} are the matrix and composite densities, ρ_p is SiC particle density.

Table 2 represents the measured density, and porosity of the matrix material, and double stir casting process composite as well as novel fabrication process composites. The density of the novel fabrication process AA7150-1% SiC composite increased by 3.67% while porosity decreased by 74.1% due to the effect of ultrasonic degassing [33] as compared to the base material. Double stir casting process AA7150-1% SiC composites increased in 2.53% while porosity decreased by 51% as compared to the base material. The corresponding bar chat for porosity is represented in Fig. 2.

3.1.2. Hardness and tensile strength

Fig. 3 shows the mechanical properties of monolithic and 1% SiC reinforced nanocomposite with different techniques. The uni-axial load was applied gradually and the corresponding results were generated. This load was gradually transferred from particle lacking place to particle-rich place in nanocomposite due to the hard phase silicon carbide particles, because of which load-bearing capacity of the composite increases. The microhardness and strength of novel fabrication process composite were more than that of the counterparts. These improvements were due to the decrease in porosity and grain refinement. The reduction of porosity and increase in grain boundary density which reduces the dislocation movement resulting in better strength of the composite.

3.1.3. Novel fabrication process

Fig. 4 shows the sketch of the novel technique process. Fig. 4a represents vortex method, which redirects ceramic particles into a molten pool with the help of mechanical rotary impeller. Step-I: Mechanical stirring action was performed for 10 min at 650 °C and it helps to reduce the cluster formation due to high viscosity at low temperature, leading high friction between particles and matrix and resulting in break up of clusters/agglomerations [7] and then the liquid is allowed to semi-solid state. Step-II: performed above liquidus temperature (750 °C) to enhance the distribution of ceramic particles and broken micro clusters due to low viscosity and high flowability at higher temperatures. Ultrasonic vibration was performed for homogeneous distribution of nanoparticles. During the sonication process, a large amount of cavitation bobbles were created with high pressure (> 1000 atm), high temperature (> 5000 °C) and this resulted in the process of formation, growth, and the collapse of bubbles. The bubbles collapse due to transient pressure and temperature differences and break up nano clusters as well as spread SiC nanoparticles with acoustic wave streaming in the melt pool; this which encouraging the homogeneous distribution of nanoparticles as shown in Fig. 4b.

3.2. Microstructure analysis

3.2.1. Optical microscope analysis

Fig. 5 shows the optical microscope images of unreinforced alloy matrix and reinforced composites developed by double stir casting as well as novel fabrication methods. Novel process AA7150-1% SiC nanocomposite was evident from the image (Fig. 5c) that the grains were significantly refined when it compared to unreinforced aluminium and

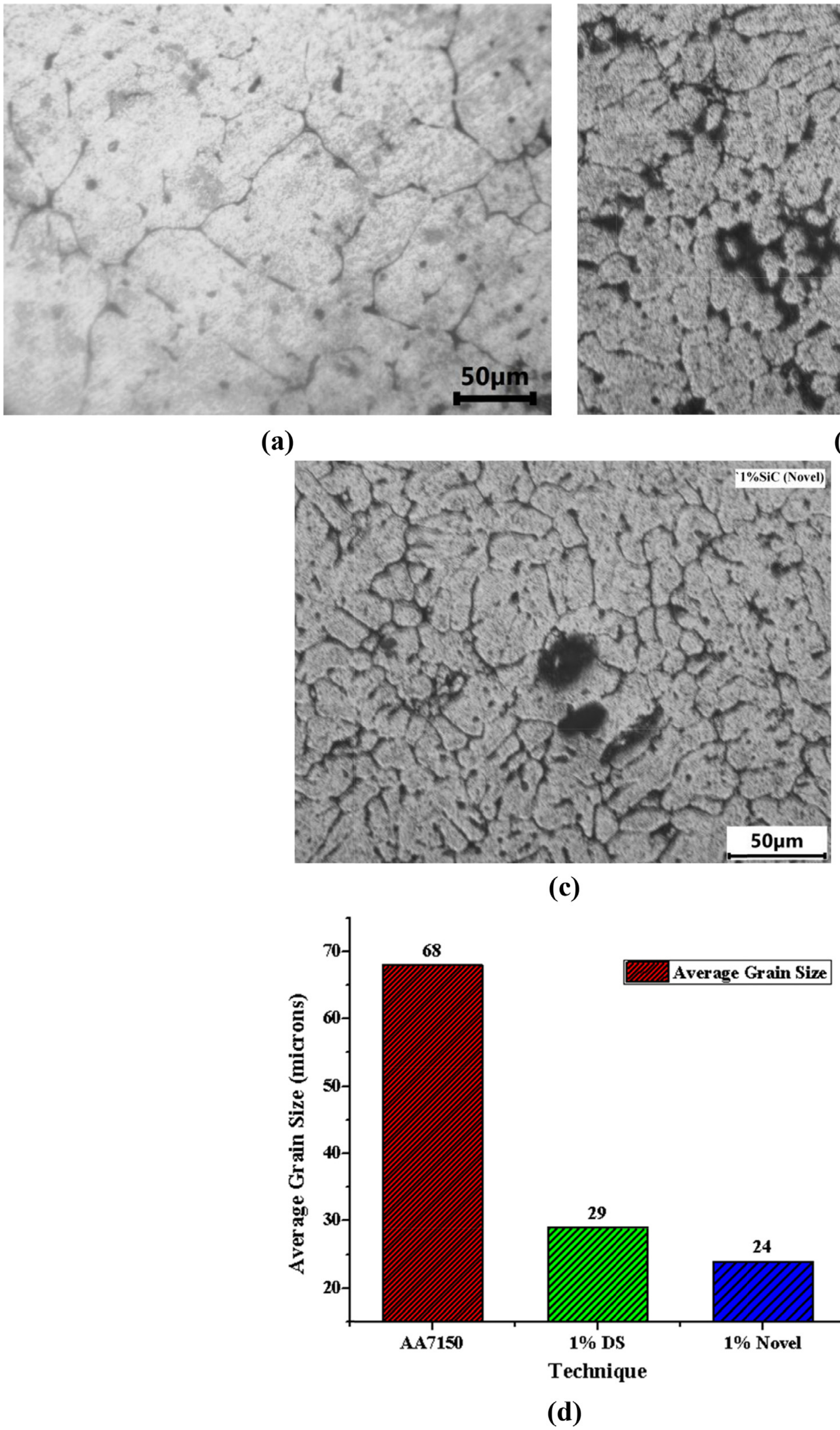
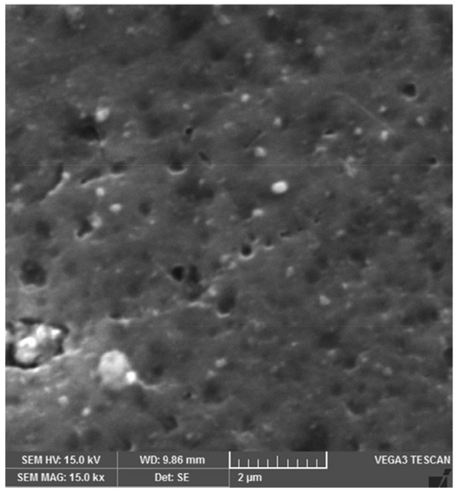


Fig. 5. Optical Microscope Images of (a) AA7150 (b) Double stir casting AA7150-1% SiC (c) Novel AA7150-1% SiC (d) Average grain size of different materials.

double stir casted AA7150-1% SiC nanocomposites. This refinement phenomenon was in nanocomposites mainly due to weight fraction and particle size of reinforcement. The grain size of nanocomposite decreases with increase of weight fraction of nanoparticles or decrease of nanoparticles size. Fig. 5c is also evident that the agglomerations and clusters are minimized in novel process nanocomposite as compared to double stir casting process nanocomposite due the ultrasonic effect which leads break the clusters and agglomerations. The average grain size of unreinforced and reinforced materials was calculated with liner intercept technique. The average grain size is the ratio of linear length of line to number of grains passing line. Length of line was calculated through "ImageJ Software" and the values of average grain size of each



(a) (b)

Fig. 6. SEM photographs for particles distribution of (a) DS AA7150-1% SiC (b) Novel AA7150-1% SiC.

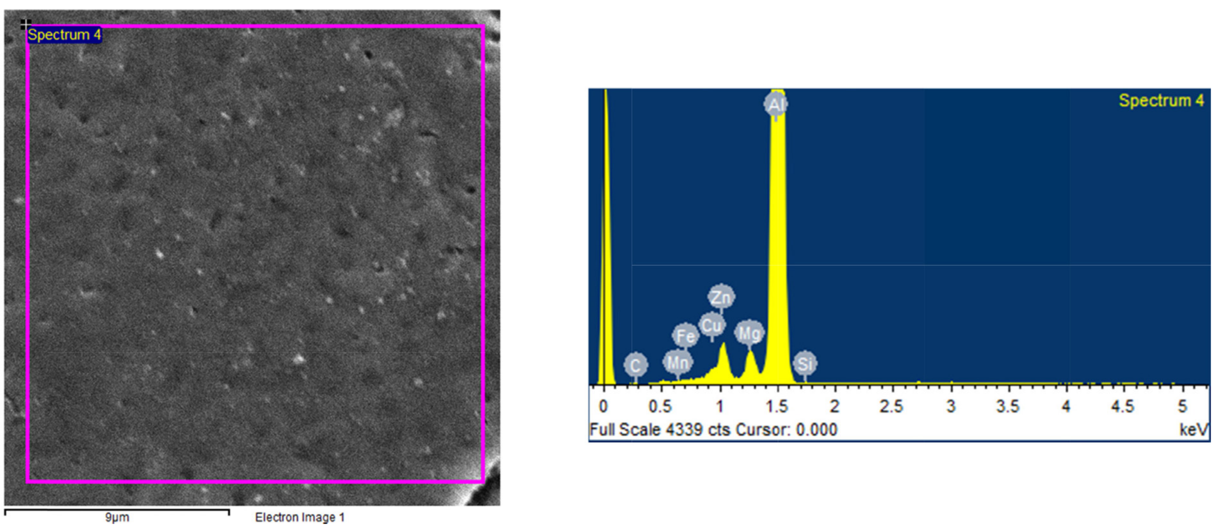


Fig. 7. EDS spectrum for Elemental analysis at AA7150-1% SiC.

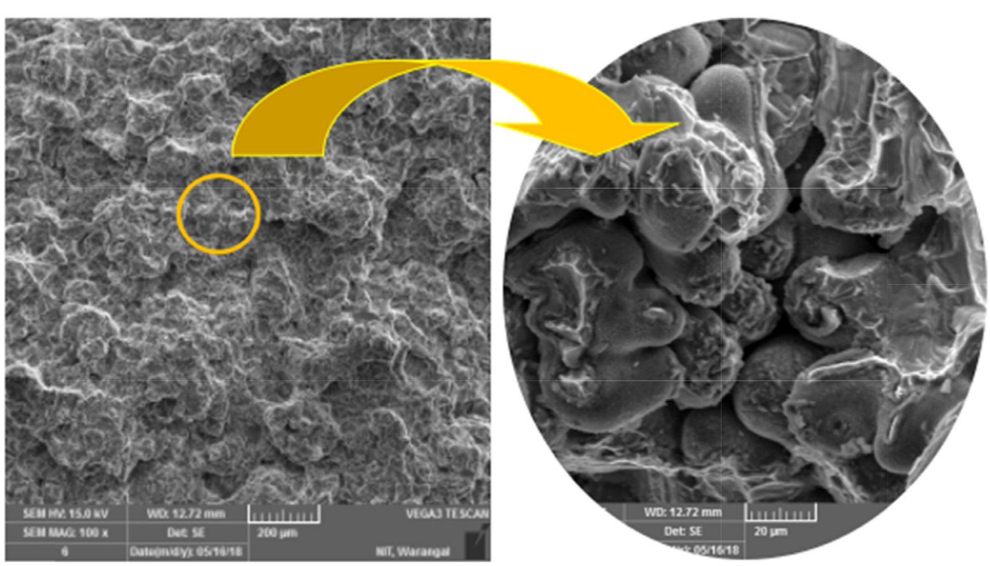


Fig. 8. Fracture surface of AA7150-1%SiC nanocomposite.

material are reported in Table 2. The fine grain refinement was observed with 24 μm (i.e., 64.7% reduction) in novel process AA7150-1% SiC nanocomposite as compared with base material and 29 μm (i.e., 17.2% reduction) with double stir cast AA7150-1% SiC nanocomposite (DS is 57.3% reduction with base material). The graphical representation of grain refinement is as shown in Fig. 5d.

3.2.2. Scanning electron microscope and EDS analysis

Fig. 6 shows the SEM photographs of 2 μm magnification. Novel fabrication process AA7150-1% SiC nanocomposite shows uniform distribution of nanoparticles and low porosity as compared to double stir casting process AA7150-1% SiC nanocomposite. The particle distribution depends on the viscosity of liquid metal. At temperature lower than 650 °C, the viscosity of the molten liquid is high and the friction between ceramic reinforcements to matrix material is more while mechanical rotary is stirred. The viscosity of melt decreases with the increase of temperature which reduces the friction between molten metal and ceramic nanoparticles. Therefore, mechanical stirring at lower temperature increases particle distribution and reduces agglomerations/cluster size due to more friction. Ultrasonic vibration treatment at high temperature provides better distribution of mixture due to less viscosity leading to transmission of high energy ultrasonic waves. These waves break the nanoclusters/ agglomerations as well as spread the nanoparticles homogeneously throughout the melt. The sketch of the novel fabrication process and its effects on particles dispersion is shown in Fig. 4.

Fig. 7 shows the scanning electron microscope photographs with EDS spectroscopy for monolithic as well as 1% SiC nanocomposite at 9 μm magnification level. The EDS spectroscopy confirms the chemical composition of major elements such as Al, Mg, Zn, Cu for AA7150 and Al, Mg, Zn, Cu. Si, C for 1% SiC nanocomposite.

3.2.3. Fracture surface analysis

The fracture surface of the novel fabrication process AA7150-1% SiC nanocomposite is shown in Fig. 8. An analysis of fracture surfaces reveals that the failure mechanism is a combination of naturally occurring locally ductile and brittle fracture in the nanocomposite due to soft and ductile nature of aluminium matrix as well as hard phase ceramic SiC particles. Failure of the composite because of microcracking, voids, and decohesion is evident on the fracture surface. The behaviour of the fracture is governed by ceramic silicon carbide particle mismatch to matrix alloy and it deference the strain carrying capacity among the hard phase particles to soft aluminium alloy matrix. Therefore, it promotes stress concentration effect of silicon carbide nanoparticles and clusters/agglomerations from the next alloy matrix.

Based on the analysis of results and discussions, it can be observed that silicon carbide nanoparticle distribution also plays a vital role to improve the mechanical properties. Generally, particle clusters are the initial source of crack formation which leads to load failure of composite materials. The existence of clusters/agglomerations in the composite material is necessary for crack initiation and propagation resulting in reduction of mechanical properties [6,11]. Therefore, the novel fabrication process has been introduced successfully to produce AA7150-1% SiC nanocomposite with uniform distribution and better mechanical properties as compared to double stir casting process.

4. Conclusions

AA7150-1% SiCp nanocomposite was successfully fabricated through a novel fabrication process by combining vortex, double stir casting, and ultrasonic vibration techniques. Microstructure and mechanical properties were investigated for novel fabrication process and compared with double stir casting process and base material. The following conclusions are stated below.

1. Grain refinement was observed more in the novel fabrication process than any other process and the average grain size of novel process was 24 μm (64.7% reduction) while it was 29 μm (57.3% reduction) for double stir casting process when compared to the base material (68 μm).
2. Double stir casting of AA7150-1% SiC nanocomposite showed 5.42% increase in tensile strength, 7.73% increase in microhardness and 64.7% reduction in porosity as compared to base alloy.
3. Novel fabrication process shows improvement in mechanical properties of AA7150-1% SiC with 144.2 MPa (↑26.05%) in tensile strength, 170.7 HV (↑10.85%) in microhardness and 74.1% reduction in porosity as compared to base alloy.
4. SEM photographs confirm the novel fabrication process led to uniform distribution of nanoparticles and minimum porosity (47% reduction) as compared to double stir casting process.
5. SEM photographs of fracture surface of nanocomposites confirm the mixed kind of ductile and brittle failure mechanism.

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