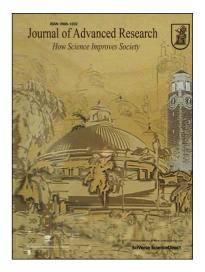
### Accepted Manuscript

### Original Article

Comparative study on the mechanical and microstructural characterization of AA 7075 nano and hybrid nanocomposites produced by stir and squeeze casting

C. Kannan, R. Ramanujam

| PII:           | S2090-1232(17)30031-0                        |
|----------------|--|
| DOI:           | http://dx.doi.org/10.1016/j.jare.2017.02.005 |
| Reference:     | JARE 515                                     |
| To appear in:  | Journal of Advanced Research                 |
| Received Date: | 5 December 2016                              |
| Revised Date:  | 13 February 2017                             |
| Accepted Date: | 28 February 2017                             |



Please cite this article as: Kannan, C., Ramanujam, R., Comparative study on the mechanical and microstructural characterization of AA 7075 nano and hybrid nanocomposites produced by stir and squeeze casting, *Journal of Advanced Research* (2017), doi: http://dx.doi.org/10.1016/j.jare.2017.02.005

This is a PDF file of an unedited manuscript that has been accepted for publication. As a service to our customers we are providing this early version of the manuscript. The manuscript will undergo copyediting, typesetting, and review of the resulting proof before it is published in its final form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

### Comparative study on the mechanical and microstructural characterization of AA 7075 nano and hybrid nanocomposites produced by stir and squeeze casting

C.Kannan<sup>\*</sup>, R. Ramanujam

School of Mechanical Engineering, VIT University, Vellore – 632014, India

Short running title: Comparative evaluation of stir cast and squeeze cast nanocomposites.

\*Corresponding author details,

| Name : | C.Kannan |
|--------|----------|
|--------|----------|

Tel. +919444788891:

Fax. : +914162243092

E-mail address: kannan.chidambaram@vit.ac.in

#### Abstract

In this research work, a comparative evaluation on the mechanical and microstructural characteristics of aluminium based single and hybrid reinforced nanocomposites was carried out. The manufacture of single reinforced nanocomposite was done with the distribution of 2 wt.% nano alumina particles (avg. particle size 30-50nm) in the molten aluminium alloy of grade AA 7075; while the hybrid reinforced nanocomposites were produced with of 4 wt.% silicon carbide (avg. particle size 5-10µm) and 2 wt.%, 4 wt.% nano alumina particles. Three numbers of single reinforced nanocomposites were manufactured through stir casting with reinforcements preheated to different temperatures viz. 400°C, 500°C, and 600°C. The stir cast procedure was extended to fabricate two hybrid reinforced nanocomposites with reinforcements preheated to 500° C prior to their inclusion. A single reinforced nanocomposite was also developed by squeeze casting with a pressure of 101MPa. Mechanical and physical properties such as density, hardness, ultimate tensile strength and impact strength were evaluated on all the developed composites. The microstructural observation was carried out using optical and scanning electron microscopy. On comparison with base alloy, an improvement of 63.7% and 81.1% in brinell hardness was observed for single and hybrid reinforced nanocomposites respectively. About 16% higher ultimate tensile strength was noticed with the squeeze cast single reinforced nanocomposite over the stir cast.

**Keywords:** hybrid nanocomposites; AA 7075; alumina; silicon carbide; squeeze casting; stir casting.

#### Introduction

Aluminium metal matrix composite (AMMC) is being preferred for numerous engineering applications like aerospace, marine, automobile and mineral processing due to their lightness associated with remarkable specific strength and thermal properties [1-5]. In aluminium composites, the properties like high toughness and ductility associated with aluminium matrix are combined with superior properties of ceramics such as high strength and elastic modulus by adding ceramic reinforcements in the base matrix [6,7]. Alumina (Al<sub>2</sub>O<sub>3</sub>), silicon carbide (SiC) and graphite (Gr) are the most common reinforcing materials [8,9] which can be incorporated in the base aluminium matrix in the form of whiskers or particles. However, manufacturing complexity and low cost favour the particle reinforced composite over whisker-reinforced [10,11].

Metal matrix nano composites (MMnC) are a new category of materials, in which the reinforcements in the range of nano-meter size are being used [12]. Increased surface area offered by nano scale reinforcements at the matrix interface leads to superior properties in composites such as increased mechanical strength, higher fatigue life and better creep resistance at elevated temperature without much compromise on ductile characteristics [13-15]. However, the end properties of MMnCs are greatly influenced by the size, shape, uniform distribution, hardening mechanism and thermal stability of nano reinforcements [16, 17]. Hybrid metal matrix composite (HMMC) is being investigated by various researchers around the world due to their enhanced properties over single reinforcements in different forms like particulates, whiskers, fibres and nanotubes or two different reinforcements of the same form. The primary and secondary reinforcements can be blended in a way to optimize the properties of hybrid composites.

Improved mechanical properties were observed with hybrid reinforced nanocomposites over single reinforced nanocomposites due to a significant reduction in meniscus penetration defect and inter-metallic component formation [18-23].

At present, the vehicle manufacturers are trying various methods to enhance the efficiency. This necessitates the automobile components to be manufactured from lightweight materials. Across the globe, the researchers are putting their efforts to develop light materials in the form of composites for aerospace and automobile applications [24,25]. Despite their efforts, limited research is available on hybrid reinforced nanocomposites that are based on aluminium alloy AA 7075, which has zinc as a primary alloying element. It has excellent strength to weight ratio. The fatigue strength of this material is comparatively better than many other aluminium alloys [26]. The limited exploration on AA 7075 hybrid reinforced nanocomposite demands further investigation. Hence, in this investigation, single and hybrid reinforced nanocomposites were manufactured with the incorporation of nano alumina and micro silicon carbide particles as reinforcements in base matrix of AA 7075. High hardness, excellent stability and better insulation are the most interesting properties of Al<sub>2</sub>O<sub>3</sub> [27]; while SiC has excellent oxidation resistance up to 1650°C and thermal shock resistance. High thermal conductivity, low thermal expansion and high strength of silicon carbide are imputed to these characteristics [28].  $Al_2O_3$ nanoparticles preheated to different temperatures were added to molten metal. This was performed to examine the influence of particle preheat temperature on its uniform distribution. In addition, a single reinforced nanocomposite was manufactured with squeeze casting to analyze the effect of squeezing pressure on the improvement of mechanical properties over a stir cast nanocomposite. The reinforcement inclusion in the molten metal and stirring for a prescribed time was followed by transferring molten metal into a die steel mold of squeeze casting

arrangement by opening the furnace valve using automatic control. This was immediately followed by the application of squeezing pressure over the solidifying composite metal. The die set up was cooled by water circulation that enhances the final mechanical properties of composites through cooling effect. The solidified nanocomposite taken out of die setup was of  $\phi$ 50 mm diameter and 300 mm length. All hybrid reinforced nanocomposites considered in this investigation were produced through stir casting. A comparative evaluation was performed on the mechanical properties of single and hybrid reinforced nanocomposites produced through different processing techniques (preheating of reinforcements prior to their inclusion in the matrix, stir casting, and squeeze casting) and presented in this work.

#### Materials and methods

Aluminium alloy of grade AA 7075 was selected as the base matrix and it was melted in the resistant heating furnace that has an integral stirrer. Nano size (30-50nm) Al<sub>2</sub>O<sub>3</sub> and micron size (5-10 $\mu$ m) SiC particles were added as reinforcements for the current investigation. The chemical composition of AA 7075 and the properties of reinforcing materials are listed in Table 1 and Table 2 respectively. The UV-visible spectrometric and transmission electron microscopic (TEM) analysis of nano Al<sub>2</sub>O<sub>3</sub> particles is shown in Fig. 1(a) and 1(b) respectively. The absorbance of light, while passing through a sample was measured using UV-visible double beam spectrophotometer (Hitachi model U-2800) in the spectrum of 380 to 600nm. In the spectrometric analysis, the absorbed light peak at a wavelength of 400nm showed the presence of alumina particles. The size and morphology of nano alumina particles were determined using TEM (Philips CM12 Transmission Electron Microscope, Netherlands). About 10mg/L of Al<sub>2</sub>O<sub>3</sub> nanoparticles was plunged in acetone solution which was preceded by 5 min ultrasonic

treatment. The dispersed particles were then deposited onto the lacey-carbon-coated copper grid. Nearly spherical particles of 30-50nm were observed from the micrograph. During the melting of base matrix, about 10 grams of sodium aluminium hexafluoride ( $Na_3AlF_6$ ) was added to the melt to prevent slag formation and to improve the efficiency of the casting process. This was done prior to the addition of reinforcements in the molten metal. Reinforcement preheating was attempted by several researchers [29-31] to remove surface impurities, alter the surface composition and for desorbing the gases. Previous research works performed with other aluminium alloys considered the reinforcement preheating temperatures in the range of 200-800°C. Based on the consideration of existing literature and the capacity of available equipment, the reinforcement preheating temperatures of 400, 500 and 600°C are going to be adopted in this work. Regardless of the base matrix, several weight proportions of the reinforcement (0-3.5 wt. %) were being tried by previous researchers [32-35]. Existing literature revealed that uniform distribution of nano reinforcements in the melt could be achieved, keeping their weight fraction not exceeding 2%. In most cases, a declining trend in the mechanical properties was observed, when this weight fraction exceeded. Thus, three single reinforced nanocomposites were produced by stir casting with the inclusion of 2 wt.% nano Al<sub>2</sub>O<sub>3</sub> particles, which were preheated to 400°C, 500°C and 600°C prior to their inclusion. This was done to investigate the influence of reinforcement preheat temperature on the mechanical characteristics of composites, thus produced. Another single reinforced nanocomposite was produced with squeeze casting to perform the quantitative comparison of mechanical characteristics with that of stir cast composite. The hybrid reinforced nanocomposites could be developed keeping the weight fraction of secondary reinforcement either one-half or same as that of primary reinforcement to investigate the influence of secondary reinforcement on the end properties of the composites.

Thus, two hybrid reinforced nanocomposites were developed through stir casting with the incorporation of 2 wt.% and 4 wt.% nano  $Al_2O_3$  with 4 wt.% micro SiC particles in the melt. Based on preliminary studies accomplished on stir cast composites, optimized processing parameters such as stirrer speed of 600 rpm, reinforcement flow rate of 5g/min and stirring time of 4 minutes were adopted for the fabrication of all composites [30,36].

### Fabrication Procedure

The bottom type stir casting set up used for manufacturing of single and hybrid reinforced nanocomposites is shown in Fig. 2. About 1.2 kg of AA 7075 was melted in a graphite crucible, which was heated to a temperature of 850°C. When the melt temperature was about 30°C above the pouring temperature, the preheated stirrer was introduced in the melt. The stirrer was made to run at 600 rpm for two minutes. While the agitation is being continued, the preheated reinforcement or mixture of reinforcements was introduced into the melt. Al<sub>2</sub>O<sub>3</sub> reinforcement was maintained at 2 wt.% in single reinforced nanocomposite, while it was varied as 2 wt.% and 4 wt.% for hybrid reinforced nanocomposites. The secondary reinforcement in these hybrid nanocomposites is 4 wt.% SiC. The stirring was continued for another 4 minutes to ensure the proper mixing of the matrix and the reinforcement. The molten metal was then poured into the preheated steel moulds to obtain the castings. The adopted design of experiments (DOE) for the fabrication of single reinforced nanocomposites is presented in Table 3.

Test specimens were fabricated from these castings using a wire-cut electro discharge machine (WEDM). The notation for the test samples and description of their processing methods are listed in Table 4. Mechanical characterisation tests such as hardness, porosity, tensile strength, impact strength and microstructural evaluation were performed on these test specimens. Whilst Archimedes principle was adopted to measure the experimental density; the tensile

strength of the composites was determined using the universal testing machine. Optical Brinell hardness testing machine was used to observe the hardness. The microstructure and distribution of reinforcements in the base matrix were examined using an optical microscope and scanning electron microscope.

#### **Results and Discussion**

#### **Density & Porosity**

The theoretical and experimental density of single and hybrid reinforced nanocomposites under investigation are shown in Fig. 3. The theoretical density of a nanocomposite was determined using the rule of mixtures and can be represented as

$$\rho_{\text{theoretical}} = \rho_m \varphi_m + \rho_r \varphi_r \qquad \dots (1)$$

where  $\varphi_m$  and  $\varphi_r$  represent wt. fraction of matrix and reinforcement;  $\rho_m$  and  $\rho_r$  represent density of matrix and reinforcement;  $\rho_{\text{theoretical}}$  represents the theoretical density of a composite. The rule of mixtures was adopted to compute the theoretical density of a nanocomposite; whilst Archimedes principle was employed to determine the experimental density [37-40]. Nano Al<sub>2</sub>O<sub>3</sub> and micro SiC particles used as reinforcements in this investigation have density values of 3970 kg/m<sup>3</sup> and 3210 kg/m<sup>3</sup> respectively. Due to the higher density of these reinforcements over the base matrix, the theoretical density of a nanocomposite was found to increase in proportion with wt.% of reinforcements. The experimental density of all developed single and hybrid reinforced nanocomposites was found to follow the trend of theoretical density, which indicated the successful fabrication of these composites through stir and squeeze casting. The hybrid nanocomposite reinforced with 4 wt.% nano Al<sub>2</sub>O<sub>3</sub> and 4 wt.% SiC was found to have the highest density among all samples. This might be imputed with high density Al<sub>2</sub>O<sub>3</sub> particles. For

the same level of nanoparticle reinforcement (2 wt.%), the squeeze casting results in much higher density over the stir casting, which can be clearly inferred from sample 7 in Fig. 3.

The procedure of determining the theoretical and experimental density of a composite through the respective utilization of rule of mixtures and Archimedes principle was subsequently followed by porosity computations. It was found that porosity of both single and hybrid reinforced nanocomposites was higher than unreinforced alloy. This might have been associated with issues such as poor wettability characteristics, particle agglomeration, clustering and pore nucleation at the interface with inadequate mechanical stirring [41,42]. Generally, the agglomeration of reinforcement and subsequent clustering provides a hindrance to the liquid metal flow. The preheating of reinforcement could reduce the wettability issues imposed by nanoparticles and lead to better distribution in the molten metal [43]. The influence of preheating temperature (400°C, 500°C and 600°C) and effect of squeezing pressure on the percentage porosity of nanocomposites was studied. The percentage porosity was calculated for all composites using the relation

$$\% \ porosity = \frac{\text{Theoretical density} - \text{Experimental density}}{\text{Theoretical density}} X \ 100 \qquad \dots (2)$$

The porosity in the metal matrix composites is instituted due to the improper interfacial reaction between the ceramic reinforcements and the matrix. This interfacial reaction is principally influenced by the factors such as free energy at the interface, convection properties and temperature gradient that exists between particles and matrix during solidification in addition to other parameters viz. stirring speed, melt viscosity, clustering, the density difference between melt and particles [44]. With an invariable reinforcement (2 wt.%) in single reinforced nanocomposites, the particles preheated at 500°C was found to result in low porosity over the

other preheating temperatures, 400°C and 600°C. This might have been resulted due to the favourable convection properties and temperature gradient that established with the particle preheated temperature of 500°C. The porosity of this single reinforced nanocomposite was further scaled down to 0.7% through squeeze casting. This is due to the fact that the plastic working induces the pore closing [45]. The calculated porosity of single and hybrid reinforced nanocomposites is shown in Fig. 4. An appreciable amount of porosity (~4.6 %) was observed with a hybrid reinforced composite, which possessed 4 wt.% nano Al<sub>2</sub>O<sub>3</sub> and 4 wt.% micro SiC particles. The calculations revealed that hybrid reinforced nanocomposites were found to possess higher porosity when compared to single reinforced nanocomposites. Increased weight fraction of nanoparticle raises the ratio of agglomeration that might have resulted in this appreciable increase in porosity; which can be reduced through squeeze casting.

#### Brinell hardness

The hardness of single and hybrid reinforced nanocomposites was determined according to ASTM E10-07 at room temperature of 30°C. Brinell hardness tester with a 10 mm ball indenter and 500 kg was used for 30 seconds. The measurements were taken at five different locations on each sample to acquire an average hardness value. The hardness variation for different composite samples is shown in Fig. 5. Increased hardness values were observed with an increase in weight percentage of nano  $Al_2O_3$  particles. Maximum hardness was observed with sample 3 (preheated nanoparticles ~500°C) amidst the single reinforced stir cast nanocomposites. This is due to the uniform distribution of nanoparticles in the base matrix. In the case of hybrid reinforced nanocomposites, higher hardness was observed with sample 5. In spite of increased weight fraction of nano alumina particles, sample 6 was found to possess lower hardness than sample 5, which might be due to agglomeration of nanoparticles (Fig. 9-d). Among all the

investigated composites, the squeeze cast nanocomposite (sample 7) that composed of 2 wt.% nano Al<sub>2</sub>O<sub>3</sub> particles was found to possess the highest hardness. This might be attributed to lowest porosity and extreme grain refinement in the case of squeeze cast nanocomposite over other composites. In general, when the cast composites are cooled to the room temperature, the ceramic reinforcements viz. nano Al<sub>2</sub>O<sub>3</sub> and micro SiC particles considered in this investigation tend to strengthen the matrix due to their mismatch in thermal expansion coefficient (CTE) of the alloy matrix. This, in turn, induced the mismatch strains at the interfaces of reinforced nanoparticles and matrix which hinder the dislocation movement and resulting in improved hardness of the composites. Higher hardness was observed with the hybrid reinforced nanocomposites due to stronger Al<sub>2</sub>O<sub>3</sub>-SiC interface in comparison to Al  $-Al_2O_3$  interface in the case of single reinforced nanocomposite, which can be inferred from Fig. 5. In comparison with the base alloy, an improvement of about 63.7% and 90.5% in hardness was observed for single reinforced nanocomposites that were produced through stir casting and squeeze casting.

#### **Tensile Strength**

The tensile tests were conducted on the test specimens according to ASTM E08-8 standards. Prior to their loading, the specimens were first polished with silicon carbide abrasive papers in grit size ranges from 220-800 in order to remove the surface defects on the sample. The universal testing machine (UTM-INSTRON 4000) loaded with 10 kN load cell was used to conduct the tensile test. The tensile strength was evaluated at the cross head speed of 0.5 mm/min. The dimension of the tensile test sample is shown in Fig. 6(a). The true stress – strain curves obtained for the investigated single and hybrid reinforced nanocomposites is shown in Fig. 6(b). The variation in ultimate tensile strength (UTS) for the investigated single and hybrid reinforced nanocomposites is shown in Fig. 6(c). When compared to base aluminium alloy, the

nanocomposites were found to possess higher tensile strength. Early researchers proposed different strengthening mechanisms for composites such as grain refinement, particle strengthening, load sharing and thermal mismatch strengthening imposed by nanoparticles. Out of these mechanisms, the influence of load sharing effect is minimal [46] and enhancement in tensile strength is mainly due to grain refinement according to Hall-Petch theory and the restricted movement of dislocations in the matrix due to nanoparticles according to Orowan mechanism [47]. Increased strength of nanocomposites could also be attributed to the difference in CTE of matrix and nanoparticles when it is cooled to room temperature [48]. While these nanocomposites were subjected to squeeze casting, further grain refinement and porosity reduction were achieved. This might have increased the ultimate tensile strength of squeeze cast nanocomposites over the stir-cast nanocomposites. In hybrid reinforced nanocomposites, about 8.5% improvement in the ultimate tensile strength was achieved with the inclusion of a secondary reinforcement (4% SiC) over the single reinforced nanocomposites, ensuring the uniform distribution of primary and secondary reinforcements in the matrix. About 16.35% higher UTS was observed with the squeeze cast single reinforced nanocomposite over the stir cast. Lower ductility was observed with single and hybrid reinforced nanocomposites over the base alloy and comparatively, it is the lowest in hybrid reinforced nanocomposites. This might be due to hard ceramic reinforcements (Al<sub>2</sub>O<sub>3</sub> and SiC) introduced into the matrix. These reinforcements might have introduced the brittleness and this lowered the ductility of developed composites. However, the ductility behaviour of squeeze cast nanocomposite was superior among all categories of composites under investigation. Under the influence of squeezing pressure, the space between the dendrites was continuously getting reduced and as a

consequence, more fine grains and homogeneous microstructure was obtained with squeeze casting process. This is inferred from Fig. 8(g). The nucleation rate (N) can be calculated by [49]

$$N = a. e^{\frac{-b}{(d+p)^2}} e^{-cp}$$
...(3)

Where a, b and c are functions of temperature; while p is the squeezing pressure. N is getting increased with an increase in p and thus grain refinement is achieved which improves the ductility. Thus, in the category of single reinforced nanocomposite, about 31.6% improved ductility was observed with squeeze cast nanocomposite over stir cast. From Fig. 9(c), it is evident that ductility can be improved with squeeze casting without any compromise on strength characteristics of nanocomposites.

#### Impact Strength

The impact strength of single and hybrid reinforced nanocomposites was determined using Izod impact testing machine according to ASTM E23-07a standards. Digital impact testing machine (Fine Testing Machines, Model-FIT - 300 D) was used to determine the impact energy absorbed by the specimens. The dimensions of an impact test sample and the impact strength variation for the single and hybrid reinforced nanocomposites are shown in Fig. 7(a) and 7(b) respectively. When compared to base aluminium alloy, the impact strength of single and hybrid reinforced nanocomposites were found to be marginally lower. This might be due to the fact that the impact strength of a material follows the same trend of ductility. However, the squeeze cast nanocomposite was found to have the highest impact strength of all samples. The squeeze cast process can reduce the pores and defects to a higher magnitude than stir casting. Moreover, it ensures the stronger bond between matrix and reinforcements and grain refinement [50]. All these effects collectively result in more ductile material that might have increased the impact

strength of the squeeze cast nanocomposite. The impact strength of squeeze cast nanocomposite was 106.3% higher than base alloy that was produced through stir casting.

#### **Microstructural Examination**

Fig. 8(a-d) shows the micrographs of AA 7075 base alloy and nanocomposites reinforced with 2 wt.% nano Al<sub>2</sub>O<sub>3</sub> particles produced with stir casting at three different reinforcement preheat temperatures 400°C, 500°C and 600°C respectively. The micrograph of hybrid reinforced nanocomposites with 2 wt.% and 4 wt.% nano Al<sub>2</sub>O<sub>3</sub> mixed with 4 wt.% SiC content is shown in Fig. 8(e) and 8(f). More uniform distribution of reinforcements was established in the hybrid reinforced composite that contained 2 wt.% nano alumina and 4 wt.% SiC particles. This is depicted in Fig. 8(e). Keeping the same silicon carbide content, when nano alumina particles were increased from 2 wt.% to 4 wt.% enhanced grain refinement was observed. This is shown in Fig. 8(f). Improved tensile strength and hardness as observed in single and hybrid reinforced nanocomposites can be attributed to grain refinement that was achieved through near uniform distribution of reinforcements in the matrix. The micrograph of a single reinforced nanocomposite developed through squeeze casting is shown in Fig. 8(g). From this micrograph, it can be inferred that ultra-level grain refinement is possible with squeeze casting than stir casting, even with the same level of nano reinforcement.

The scanning electron microscope (SEM) image of aluminium alloy AA7075 (as cast condition) is shown in Fig. 9(a), while the energy dispersive spectroscopy (EDS) analysis of this alloy is shown Fig. 9(b). The SEM image of single reinforced nanocomposite produced through stir casting with 2 wt.% nano  $Al_2O_3$  particles that were preheated to the temperature of 500°C is shown in Fig. 9(c). The nano  $Al_2O_3$  reinforcements in the base matrix were identified through the utilization of higher magnification. The EDS analysis also confirmed the presence of  $Al_2O_3$ 

nanoparticles in the matrix. This is presented in Fig. 9(d). It is well proven that for aluminium metal matrix composites, improved mechanical properties principally depend upon the uniform distribution of the second phase in the final composite. From SEM images, it was evident that nanoparticles were almost uniformly distributed in the base matrix for the composites under investigation. It could be inferred from Fig. 9(e), a hybrid reinforced nanocomposite with 2 wt.% nano Al<sub>2</sub>O<sub>3</sub> and 4 wt.% micro SiC established the uniform distribution of reinforcements in the base matrix. The presence of both primary and secondary reinforcement in the base matrix was confirmed through EDS analysis. EDS of the secondary reinforcement (silicon carbide) is shown in Fig. 9(f). While the weight fraction of primary reinforcement was increased beyond 2%, agglomeration of both primary and secondary reinforcements was observed. This is shown in Fig. 9(g). The SEM image of single reinforced nanocomposite produced by squeeze casting is shown in Fig. 9(h). The SEM images of fractured tensile test samples of 2 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposite (stir cast), 2 wt.% Al<sub>2</sub>O<sub>3</sub> and 4 wt.% SiC hybrid reinforced nanocomposite (stir cast) and 2 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposite (squeeze cast) are shown in Fig. 9(i), 9(j) and 9(k) respectively. The SEM image taken over the fractured surface of single reinforced squeeze cast nanocomposite was exposing some fine dimples and cleavages, which represented the respective ductile and brittle fracture modes (refer Fig. 9(k)).

#### Conclusions

This paper addressed the comparative study on mechanical and microstructural characterization of AA 7075 based single and hybrid reinforced nanocomposites produced through stir and squeeze cast methods with different preheating temperatures. The composites are prepared with reinforcement of 2, 4 wt.% nano alumina particles and 4 wt.% silicon carbide particles. The hybrid nanocomposite is produced with reinforcing nano alumina and silicon

carbide particles. The mechanical properties such as density, porosity, hardness, tensile strength and impact strength are evaluated and compared. The significant findings of this investigation are as follows:

- An increase in hardness and tensile strength is observed for single and hybrid reinforced nanocomposites with increasing Al<sub>2</sub>O<sub>3</sub> content and found to be higher than base aluminium alloy.
- In comparison to base alloy, hardness is getting improved by 63.7% and 81.1% for single and hybrid reinforced nanocomposite (stir cast), while an improvement of 90.5% is observed with single reinforced nanocomposite (squeeze cast). An increase in the ultimate tensile strength with magnitudes of 60.1%, 73.8% and 92.3% is observed with the same sequence of these composites over the base matrix.
- The microstructure and SEM analysis revealed the uniform distribution of particles in the base matrix provided that the weight fraction of nano reinforcement is limited to 2%.
- Among the different reinforcement preheat temperatures adopted for fabrication of nanocomposites, 500°C is witnessed to produce more uniform distribution and prevents agglomeration of particles, while the weight fraction of nano reinforcement is not exceeding 2%.
- From the mechanical characterisation tests, it is inferred that the density, hardness and ultimate tensile strength of single and hybrid reinforced nanocomposites are superior to base alloy. However, when nano reinforcements are increased beyond 2%, agglomeration of nanoparticle in the base matrix is inevitable, which deteriorates the mechanical characteristics of hybrid reinforced nanocomposites.

• On the implementation of secondary material processing such as squeeze casting, even single reinforced nanocomposites own improved properties over hybrid reinforced nanocomposites that are produced through stir casting. The mechanical and microstructural characterization of hybrid reinforced nanocomposites by squeeze casting is still to be carried out.

From this experimental investigation, it is concluded that both squeeze cast single reinforced nanocomposite and stir cast hybrid reinforced nanocomposite exhibit superior mechanical properties over the base alloy, AA 7075. Due to this fact, these composites can be employed as candidate materials in aerospace and automotive sectors, where quality is not a compromise.

M

\*

### References

- [1] Mazumdar S. Composites manufacturing: materials, product, and process engineering.
  CRC press; 2001.
- [2] K.K. Chawla, Composite materials: science and engineering. Springer-Verlag, New York; 1998.
- [3] Surappa MK. Aluminium matrix composites: Challenges and opportunities. Sadhana-Acad P Eng S. 2003; 28(1-2):319-34.
- [4] Miracle DB. Metal matrix composites-from science to technological significance. Compos Sci Technol. 2005; 65(15):2526-40.
- [5] Rosso M. Ceramic and metal matrix composites: Routes and properties. J Mater Process Tech. 2006; 175(1):364-75.
- [6] Bhushan RK, Kumar S, Das S. Fabrication and characterization of 7075 Al alloy reinforced with SiC particulates. Int J Adv Manuf Tech. 2013; 65(5-8):611-24.
- [7] Karabulut S, Gokmen U, Cinici H. Study on the mechanical and drilling properties of AA7039 composites reinforced with Al<sub>2</sub>O<sub>3</sub>/B<sub>4</sub>C/SiC particles. Compos Part B - Eng. 2016; 93:43-55.
- [8] Baradeswaran A, Perumal AE. Study on mechanical and wear properties of Al 7075/Al<sub>2</sub>O<sub>3</sub>/graphite hybrid composites. Compos Part B - Eng. 2014; 56:464-71.
- [9] Baradeswaran A, Perumal AE. Wear and mechanical characteristics of Al 7075/graphite composites. Compos Part B Eng. 2014; 56:472-6.
- [10] Yigezu BS, Mahapatra MM, Jha PK. Influence of reinforcement type on microstructure, hardness, and tensile properties of an aluminum alloy metal matrix composite. J Miner Mater Charact Eng. 2013; 1(4), Article ID:33948,7 pages

- [11] Das DK, Mishra PC, Singh S, Pattanaik S. Fabrication and heat treatment of ceramicreinforced aluminium matrix composites-a review. Int J Mech Mater Eng. 2014; 9(1):1-5.
- [12] Tjong SC. Recent progress in the development and properties of novel metal matrix nanocomposites reinforced with carbon nanotubes and graphene nanosheets. Mat Sci Eng R. 2013; 74(10):281-350.
- [13] Ahamed H, Senthilkumar V. Experimental investigation on newly developed ultrafinegrained aluminium based nano-composites with improved mechanical properties. Mater Design. 2012; 37:182-92.
- [14] Mazahery A, Abdizadeh H, Baharvandi HR. Development of high-performance A356/nano-Al<sub>2</sub>O<sub>3</sub> composites. Maters Sci Eng A-Struct. 2009; 518(1):61-4.
- [15] Su H, Gao W, Feng Z, Lu Z. Processing, microstructure and tensile properties of nanosized Al<sub>2</sub>O<sub>3</sub> particle reinforced aluminum matrix composites. Mater Design. 2012; 36:590-6.
- [16] Cao G, Konishi H, Li X. Mechanical Properties and Microstructure of Mg/SiC Nanocomposites Fabricated by Ultrasonic Cavitation Based Nanomanufacturing. J Manuf Sci E-T ASME, 2008; 130(3):031105.
- [17] Casati R, Vedani M. Metal matrix composites reinforced by nano-particles—a review. Metals. 2014; 4(1):65-83.
- [18] Sharma P, Khanduja D, Sharma S. Production of Hybrid Composite by a Novel Process and Its Physical Comparison with Single Reinforced Composites. Mater Today: Proceed. 2015; 2(4):2698-707.

- [19] Rajmohan T, Palanikumar K, Ranganathan S. Evaluation of mechanical and wear properties of hybrid aluminium matrix composites. T Nonferr Metal Soc. 2013; 23(9):2509-17.
- [20] Poovazhagan L, Kalaichelvan K, Rajadurai A, Senthilvelan V. Characterization of hybrid silicon carbide and boron carbide nanoparticles-reinforced aluminum alloy composites. Procedia Eng. 2013; 64:681-9.
- [21] Prasad DS, Shoba C, Ramanaiah N. Investigations on mechanical properties of aluminum hybrid composites. J Mater Res Technol. 2014; 3(1):79-85.
- [22] Jiang J, Wang Y. Microstructure and mechanical properties of the rheoformed cylindrical part of 7075 aluminum matrix composite reinforced with nano-sized SiC particles. Mater Design. 2015; 79:32-41.
- [23] Ahmed K. Hybrid composites prepared from Industrial waste: Mechanical and swelling behavior. J Adv Res. 2015; 6(2):225-32.
- [24] Immarigeon JP, Holt RT, Koul AK, Zhao L, Wallace W, Beddoes JC. Lightweight materials for aircraft applications. Mater Charact. 1995; 35(1):41-67.
- [25] Cole GS, Sherman AM. Lightweight materials for automotive applications. Mater Charact. 1995; 35(1):3-9.
- [26] Cobden R, Banbury A. Aluminium: physical properties, characteristics and alloys. Talat Lecture. European Aluminium Association. 1994; 1501:56.
- [27] Kopanda JE, MacZura G, Hart LD. Alumina Chemicals Science and Technology Handbook. 1990.
- [28] Harris GL (Ed.). Properties of Silicon Carbide, INSPEC, London. 1995.

- [29] Juang SH, Fan LJ, Yang HP. Influence of preheating temperatures and adding rates on distributions of fly ash in aluminum matrix composites prepared by stir casting. Int J Precis Eng Manuf. 2015; 16(7):1321-7.
- [30] El-Mahallawi IS, Shash AY, Amer AE. Nanoreinforced Cast Al-Si Alloys with Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and ZrO<sub>2</sub> Nanoparticles. Metals. 2015; 5(2):802-21.
- [31] Kok M. Production and mechanical properties of Al<sub>2</sub>O<sub>3</sub> particle-reinforced 2024 aluminium alloy composites. J Mater Process Technol. 2005; 161(3):381-7.
- [32] Mazahery A, Ostadshabani M. Investigation on mechanical properties of nano-Al<sub>2</sub>O<sub>3</sub>reinforced aluminum matrix composites. J Compos Mater. 2011; 45(24):2579-86.
- [33] Li X, Yang Y, Cheng X. Ultrasonic-assisted fabrication of metal matrix nanocomposites. J Mater Sci. 2004; 39(9):3211-2.
- [34] Su H, Gao W, Feng Z, Lu Z. Processing, microstructure and tensile properties of nano-sized Al<sub>2</sub>O<sub>3</sub> particle reinforced aluminum matrix composites. Mater Design. 2012; 36:590-6.
- [35] Akbari MK, Baharvandi HR, Mirzaee O. Investigation of particle size and reinforcement content on mechanical properties and fracture behavior of A356-Al<sub>2</sub>O<sub>3</sub> composite fabricated by vortex method. J Compos Mater. 2014; 48(27):3315-30.
- [36] GG S, Balasivanandha P, VSK V. Effect of processing parameters on metal matrix composites: stir casting process. J Surf Eng Mater Adv Technol.2012; 2(1), Article ID: 16992, 5 pages.
- [37] Prasad DS, Shoba C, Ramanaiah N. Investigations on mechanical properties of aluminum hybrid composites. J Mater Res Technol. 2014; 3(1):79-85.

- [38] Kumar GV, Rao CS, Selvaraj N, Bhagyashekar MS. Studies on Al6061-SiC and Al7075-Al<sub>2</sub>O<sub>3</sub> metal matrix composites. J Miner Mater Charact Eng. 2010; 9(01):43.
- [39] Boopathi MM, Arulshri KP, Iyandurai N. Evaluation of mechanical properties of aluminium alloy 2024 reinforced with silicon carbide and fly ash hybrid metal matrix composites. Am J Appl Sci. 2013; 10(3):219.
- [40] Agrawal A, Satapathy A. Effects of aluminium nitride inclusions on thermal and electrical properties of epoxy and polypropylene: an experimental investigation. Compos Part A-Appl S. 2014; 63:51-8.
- [41] Sevik H, Kurnaz SC. Properties of alumina particulate reinforced aluminum alloy produced by pressure die casting. Mater Design. 2006; 27(8):676-83.
- [42] Vencl A, Bobic I, Arostegui S, Bobic B, Marinković A, Babić M. Structural, mechanical and tribological properties of A356 aluminium alloy reinforced with Al<sub>2</sub>O<sub>3</sub>, SiC and SiC+ graphite particles. J Alloy Compd. 2010; 506(2):631-9.
- [43] Singh J, Chauhan A. Characterization of hybrid aluminum matrix composites for advanced applications–A review. J Mater Res Technol. 2016; 5(2):159-69.
- [44] Priyadarshi D, Sharma RK. Porosity in aluminium matrix composites: cause, effect and defence. Mater Sci: An Indian Journal. 2016; 14(4):119-29.
- [45] Hangai Y, Kitahara S, Amada S. Pore defect control in die casting by compression loading. Mater Trans. 2006; 47(9):2363-7.
- [46] Sajjadi SA, Ezatpour HR, Parizi MT. Comparison of microstructure and mechanical properties of A356 aluminum alloy/Al<sub>2</sub>O<sub>3</sub> composites fabricated by stir and compo-casting processes. Mater Design. 2012; 34:106-11.

- [47] Zhang Z, Chen DL. Consideration of Orowan strengthening effect in particulate-reinforced metal matrix nanocomposites: A model for predicting their yield strength. Scripta Mater. 2006; 54(7):1321-6.
- [48] Ezatpour HR, Sajjadi SA, Sabzevar MH, Huang Y. Investigation of microstructure and mechanical properties of Al6061-nanocomposite fabricated by stir casting. Mater Design. 2014; 55:921-8.
- [49] ZHAO Zu-de. Solid-Liquid forming theory and technology of composites. Metallurgical Industry Press, Beijing; 2008.
- [50] Deshmanya IB, Purohit GK. Development of models for predicting impact strength of Al7075/Al<sub>2</sub>O<sub>3</sub> composites produced by stir-casting. J Compos Mater. 2012; 46(26):3247-53.

### List of Tables

Table 1: Chemical composition of AA 7075

Table 2: Properties of reinforcements

Table 3: Design of Experiments (DOE) for the fabrication of single reinforced nanocomposites

Table 4: Processing methods of single and hybrid reinforced nanocomposites

### **List of Figures**

Fig. 1 Spectrometric (a) and TEM analysis (b) of nano Al<sub>2</sub>O<sub>3</sub> particles

Fig. 2 Bottom type stir casting set up with squeeze casting attachment

Fig. 3 Theoretical and experimental densities of single and hybrid reinforced nanocomposites

Fig. 4 Porosity of single and hybrid reinforced nanocomposites

Fig. 5 Hardness of AA 7075, single and hybrid reinforced nanocomposites (as cast condition)

Fig. 6(a) Dimensions of a tensile testing sample

Fig. 6(b) True stress – strain curves for single reinforced and hybrid reinforced nanocomposites

Fig. 6(c) Tensile strength and ductility variation of single and hybrid reinforced nanocomposites

Fig. 7(a) Schematic and photographs of impact testing samples

Fig. 7(b) Impact strength of single and hybrid reinforced nanocomposites

Fig. 8 Optical micrographs of base aluminium alloy (a), stir cast single reinforced nano composites (b-d), stir cast hybrid reinforced nanocomposites (e-f) and squeeze-cast single reinforced nanocomposite (g) [Magnification: 50 X]

Fig. 9 SEM images of base Al alloy (a), stir cast nanocomposite with 2 wt.%  $Al_2O_3$  (c), stir-cast hybrid nanocomposite with 2 wt.%  $Al_2O_3$  and 4 wt.% SiC (e), stir-cast hybrid nanocomposite with 4 wt.%  $Al_2O_3$  and 4 wt.% SiC (g) squeeze cast 2 wt.%  $Al_2O_3$  nanocomposite (h) EDS of Al alloy (b), nano  $Al_2O_3$  particles (d) SiC particles (f) Fractographs of stir cast nanocomposite with 2 wt.%  $Al_2O_3$  (i) Fractograph of stir-cast hybrid nanocomposite with 2 wt.%  $Al_2O_3$  (i) Fractograph of stir-cast hybrid nanocomposite with 2 wt.%  $Al_2O_3$  (i) Fractograph of stir-cast hybrid nanocomposite with 2 wt.%  $Al_2O_3$  (k)

| Si   | Fe   | Cu   | Mn   | Mg   | Cr   | Zn  | Ti   | Al   |
|------|------|------|------|------|------|-----|------|------|
| 0.13 | 0.42 | 1.42 | 0.12 | 2.42 | 0.21 | 5.4 | 0.11 | Bal. |
|      |      |      |      |      |      |     |      |      |

| Table 1 Chemical composition of AA 7075 [8,9] |
|---|
|---|

| Property  | Al <sub>2</sub> O <sub>3</sub> | SiC   |
|---|--------------------------------|-------|
| Particle size   | 30-50nm                        | 2-5µm |
| Color   | White                          | Black |
| Density (g/cm <sup>3</sup> )                                | 3.97                           | 3.1   |
| Elastic Modulus (GPa)                                       | 375                            | 410   |
| Melting point (°C)  | 2055                           | 1650  |
| Thermal conductivity (W/mK)                                 | 35                             | 83.6  |
| Coefficient of thermal expansion (x $10^{-6}/{}^{\circ}C$ ) | 8.4                            | 4.3   |
| Rectification   |                                |       |

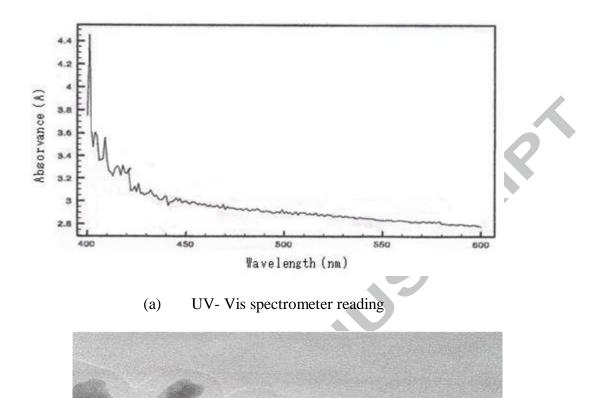
### Table 2 Properties of reinforcements [26,27]

| Type of                                  | Preneating te | inperature of remi | forcements (°C) | Squeezing FI | ressure (MPa) |
|--|---------------|--------------------|-----------------|--------------|---------------|
| Composite                                | L ouv loval   | Medium level       | High loyal      | Low level    | High layal    |
|  | Low level     |                    | High level      |              | High level    |
|  | (-1)          | (0)                | (+1)            | (-1)         | (+1)          |
| Single reinforced                        | 100           |                    | <00             | 0            |               |
| nanocomposite                            | 400           | 500                | 600             | 0            | 101           |
| (2 wt.% Al <sub>2</sub> O <sub>3</sub> ) |               |                    |                 |              |               |
|  |               |                    |                 | 5            |               |

| Table 3 Design of Experiments | (I)()E) for the tebrication | of single rainforced | noncompositor  |
|-------------------------------|-----------------------------|----------------------|----------------|
|                               | (DOE) IOI HIE IAUI CALIOI   |                      | nanocomposites |
|                               |                             |                      |                |

| 1<br>2<br>3<br>4 | Base matrix: AA 7075<br>AA 7075 reinforced with 2 wt.%<br>nano Al <sub>2</sub> O <sub>3</sub> particles<br>AA 7075 reinforced with 2 wt.%<br>nano Al <sub>2</sub> O <sub>3</sub> particles<br>AA 7075 reinforced with 2 wt.%<br>nano Al <sub>2</sub> O <sub>3</sub> particles | - Al <sub>2</sub> O <sub>3</sub> nanoparticles preheated to<br>400°C<br>Al <sub>2</sub> O <sub>3</sub> nanoparticles preheated to<br>500°C<br>Al <sub>2</sub> O <sub>3</sub> nanoparticles preheated to<br>600°C | Stir casting       |
|------------------|---|--|--------------------|
| 3                | nano Al <sub>2</sub> O <sub>3</sub> particles<br>AA 7075 reinforced with 2 wt.%<br>nano Al <sub>2</sub> O <sub>3</sub> particles<br>AA 7075 reinforced with 2 wt.%<br>nano Al <sub>2</sub> O <sub>3</sub> particles   | 400°C<br>Al <sub>2</sub> O <sub>3</sub> nanoparticles preheated to<br>500°C<br>Al <sub>2</sub> O <sub>3</sub> nanoparticles preheated to   | Stir costing       |
| 3                | AA 7075 reinforced with 2 wt.%<br>nano Al <sub>2</sub> O <sub>3</sub> particles<br>AA 7075 reinforced with 2 wt.%<br>nano Al <sub>2</sub> O <sub>3</sub> particles  | $Al_2O_3$ nanoparticles preheated to<br>500°C<br>$Al_2O_3$ nanoparticles preheated to  | Stir costing       |
| 4                | nano Al <sub>2</sub> O <sub>3</sub> particles<br>AA 7075 reinforced with 2 wt.%<br>nano Al <sub>2</sub> O <sub>3</sub> particles  | 500°C<br>Al <sub>2</sub> O <sub>3</sub> nanoparticles preheated to   | Stir costing       |
| 4                | AA 7075 reinforced with 2 wt.%<br>nano Al <sub>2</sub> O <sub>3</sub> particles   | Al <sub>2</sub> O <sub>3</sub> nanoparticles preheated to  | Stir costing       |
|                  | nano Al <sub>2</sub> O <sub>3</sub> particles   |  | Stir costing       |
|                  | -   | 600°C  |                    |
|                  |   | 000 C  | Still Casting      |
| _                | AA 7075 reinforced with 2 wt.%  | Both Al <sub>2</sub> O <sub>3</sub> and SiC particles  |                    |
| 5                | nano $Al_2O_3$ particles and 4 wt.%   | preheated to 500°C   |                    |
|                  | micro SiC particles   |  |                    |
|                  | AA 7075 reinforced with 4 wt.%  |  |                    |
| 6                | nano $Al_2O_3$ particles and 4 wt.%   |  |                    |
|                  | micro SiC particles   |  |                    |
| 7                | AA 7075 reinforced with 2 wt.%  | Al <sub>2</sub> O <sub>3</sub> nanoparticles preheated to  | Squeeze casting    |
| /                | nano Al <sub>2</sub> O <sub>3</sub> particles   | 500°C  | pressure of 101 MP |
| C                |   |  |                    |

Table 4 Processing methods of single and hybrid reinforced nanocomposites



(b) TEM analysis

Fig 1. Spectrometric and TEM analysis of nano Al<sub>2</sub>O<sub>3</sub> particles

100

×C'

nm

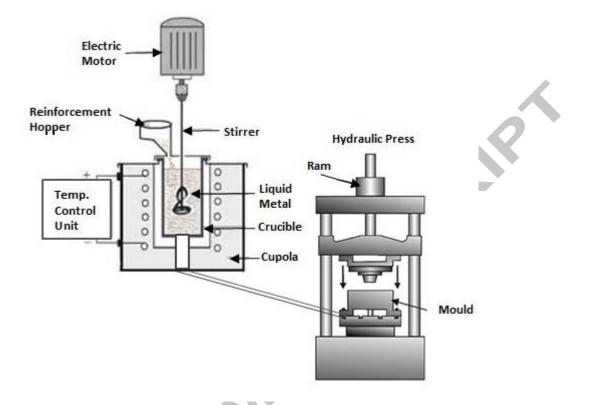


Fig 2. Bottom type stir casting set up with squeeze casting attachment

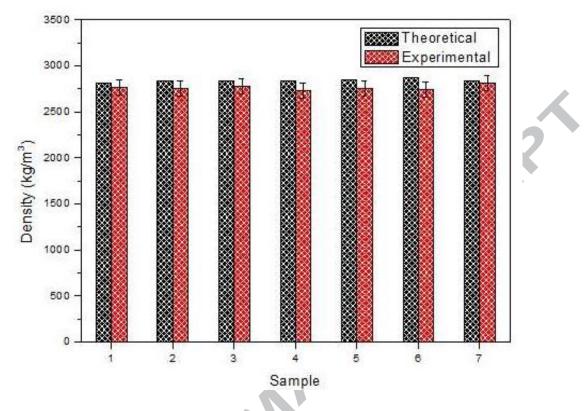


Fig 3. Theoretical and experimental densities of single and hybrid reinforced nanocomposites

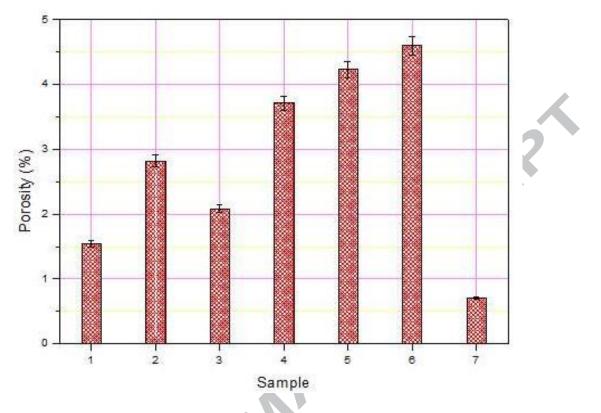


Fig 4. Porosity of single and hybrid reinforced nanocomposites

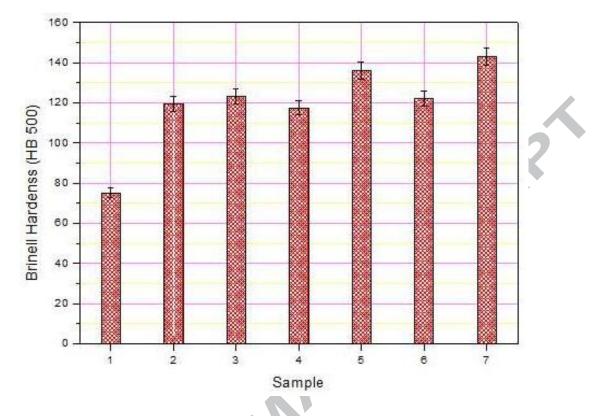


Fig 5. Hardness of AA 7075, single and hybrid reinforced nanocomposites (as cast condition)

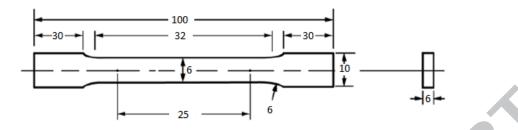


Fig 6(a). Dimensions of a tensile testing sample (All dimensions in mm)

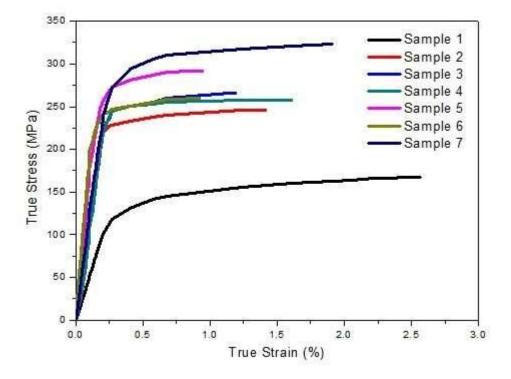


Fig 6(b). True stress – true strain curves for single reinforced and hybrid reinforced nanocomposites

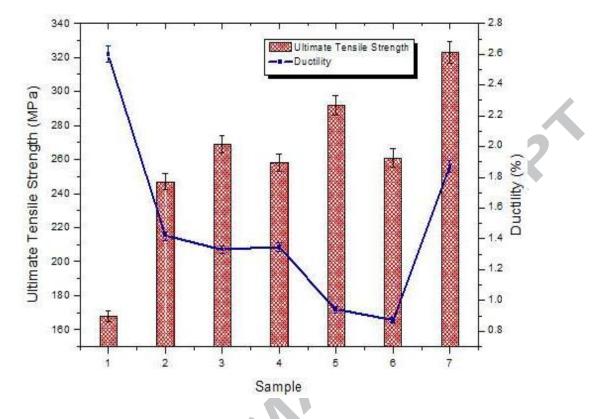


Fig 6(c). Tensile strength and ductility variation of single and hybrid reinforced nanocomposites

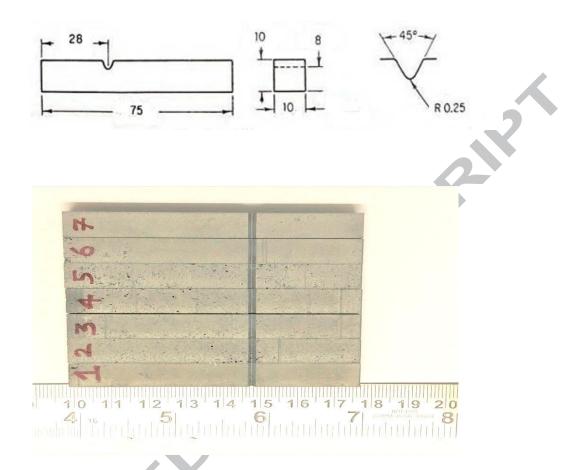


Fig 7(a). Schematic and photographs of impact testing samples (All dimensions in mm)

Rock

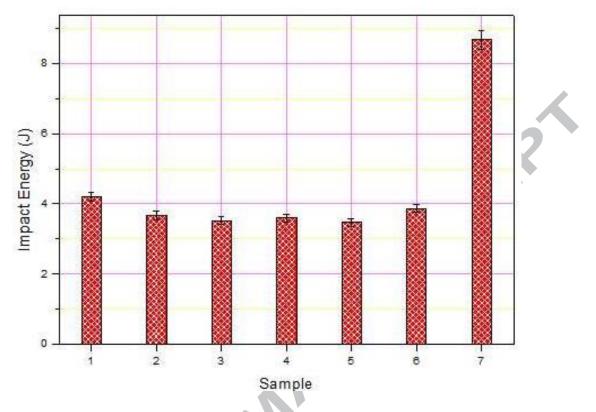


Fig 7(b). Impact strength of single and hybrid reinforced nanocomposites

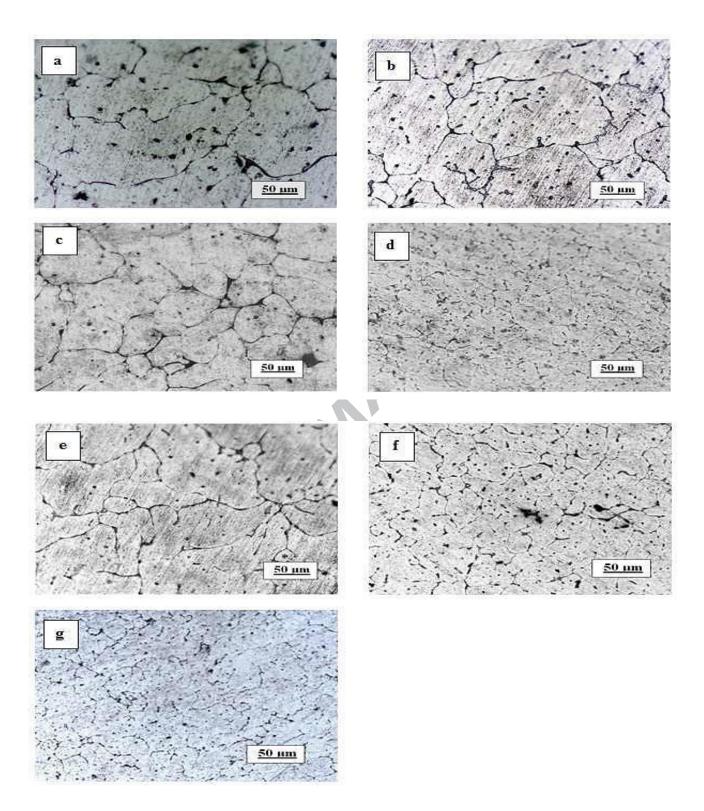
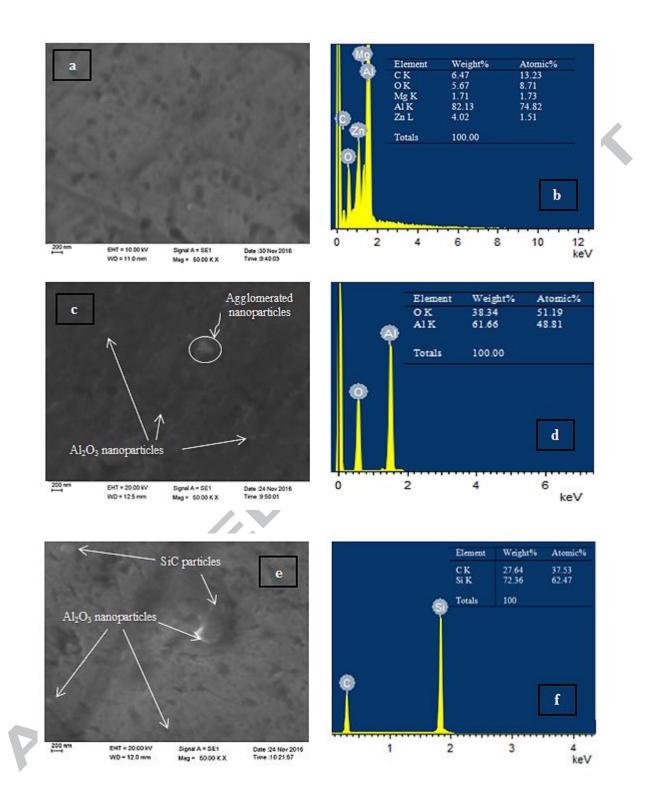


Fig. 8 Optical micrographs of aluminium alloy (a), stir cast single reinforced nano composites (b-d), stir cast hybrid reinforced nanocomposites (e-f) and squeeze-cast single reinforced nanocomposite (g)



39

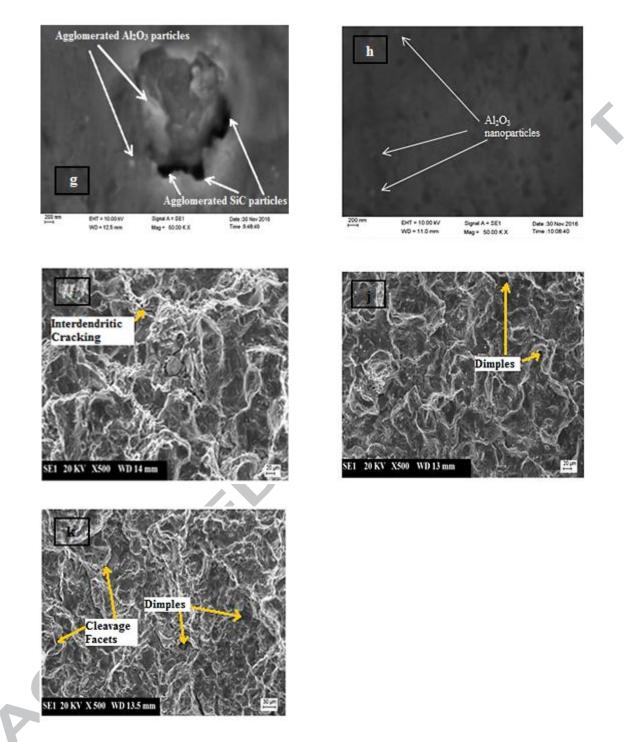


Fig 9. SEM images of base aluminium alloy (a), stir-cast 2 wt.%  $Al_2O_3$  nanocomposite (c), stir-cast hybrid nanocomposite with 2 wt.%  $Al_2O_3$  and 4 wt.% SiC (e), stir-cast hybrid nanocomposite with 4 wt.%

 $Al_2O_3$  and 4 wt.% SiC (g) squeeze cast 2 wt.%  $Al_2O_3$  nanocomposite (h)

EDS of aluminium alloy (b), nano Al<sub>2</sub>O<sub>3</sub> particles (d) SiC particles (f)

 $\label{eq:Fractographs} Fractographs of stir-cast nanocomposite (i) \ Fractograph of stir-cast hybrid nanocomposite with 2 wt.\%$ 

Al<sub>2</sub>O<sub>3</sub> and 4 wt.% SiC (j) Fractograph of squeeze cast nanocomposite (k)

### **Compliance with Ethics Requirements**

This article does not contain any studies with human or animal subjects

C.Kannan

Corresponding author

### **Conflict of Interest**

The authors have declared no conflict of interest

C.Kannan

**Corresponding author** 

