MICROSTRUCTURAL AND MECHANICAL PROPERTIES OF AL-REINFORCED WITH MICRO AND NANO Al_2O_3 PARTICLES USING STIR-SQUEEZE CASTING METHOD

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ABSTRACT

Composites focused on aluminium metal matrix are popular composite material which offers better mechanical properties than monolithic metal. Experimental work was carried out on Scrap Aluminium Alloy Wheels (SAAW) with micro-sized Al_2O_3 and nano-sized Al_2O_3 particles as reinforcement material. The density, percentage of porosity, microstructural and mechanical properties were evaluated. An optical micrograph shows that reinforcing is evenly distributed within matrix grain boundaries. The mechanical properties such as hardness, tensile strength and compressive strength of the composite were significantly enhanced to 57 HRB, 209 MPa and 587 MPa respectively. The SAAW reinforced composites (S1) combined with 1 wt % nano-sized Al_2O_3 particles and with 4% micro-sized Al_2O_3 particles exhibited the highest tensile, compressive and hardness properties compared to other composites.

KEYWORDS: Metal Matrix Composite, Nanoparticles, Alumina, Aluminium, Stir-Squeeze Casting, Microstructure & Mechanical Properties

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1. INTRODUCTION

Due to their enhanced mechanical properties along with high strength to weight ratio and easy processing, aluminium based Metal Matrix Composites (MMCs) find many applications in engineering industry such as automotive, aerospace, etc. Metal matrix composites are metals or alloys containing flakes, whiskers, fibers derived out of a particular ceramic material, such as SiC, Al_2O_3 , B₄C, TiB₂, ZrO₂ and Graphite, which provide unique opportunities for refining to specific design criteria. Aluminium castings are being used for about 40% of automobile parts which require lighter in weight and fuel efficiency [1]. Porsche uses cast aluminium block manufactured with a preform of silicone in porous conditions and Honda uses a kind of material that use aluminium-alumina and carbon fibers bores [2]. Pistons and cylinder liners made of graphite particles dispersed with aluminium alloy which renders the solid lubrication. A reduced coefficient of friction and wear were obtained when tested with piston and liners made of graphite -aluminium [3]. J Li et al. studied the influence of ultrasonic treatment on Al-cu hybrid composite reinforced with 10% Mg 2 Si + 1% nano SiC particles. It was reported that hybrid composited Al-cu reinforced with 10% Mg 2 Si + 1% nano SiC exhibited higher tensile strength than the base material [4]. The significance of reinforcement volume content in phase, fabrication technique, size of particle and density on both physical and thermo-mechanical properties of aluminium composite was studied by Pradeep Gudlur and others. The composite with smaller aluminium particles is found to have higher density values and

hence better elastic surfaces and lower CTE values than composite aluminium particles [5]. Singh et al. used liquid metallurgy technique to prepare Al-6061 material added with nano Al_2O_3 particles varying by weight from 1–3 %. The mechanical properties of the composite developed by the ultrasonic treatment assisted squeeze process including tensile strength, compressive strength and hardness were found to be more than that of the ultrasonically assisted stir-squeeze casting process [6]. Suresh et al. developed and evaluated LM 25 MMCs combined with micro and nano particles up to 10 wt. %. [7]. Kannan and other researchers analyzed the aluminium based nanocomposites in terms of mechanical and microstructural characteristics. They found that nano alumina particle reinforced composite obtained better properties than other composites [8]. Lei et al. researched the concentration of nanomaterials which had an important effect on the composites' thermal expansion behaviour [9]. They concluded that the concentration of nanoparticles had important effects on the composite's thermal expansion behaviour.

Although researchers have conducted experiments on alumina reinforced composite embedded with aluminium matrix, but very few research papers are available with SAAW as raw material [10,12]. Hence, an effort was formulated to determine the mechanical properties of aluminium-alumina composite using SAAW as a matrix material reinforced with Al_2O_3 nano-sized and Al_2O_3 micro-sized particles.

2. RESEARCH METHODOLOGY

2.1 Materials

The matrix material used was a scrap aluminium alloy wheels of Honda car make and reinforcing materials combination of nano alumina powder of 80 nm with 99.5 % purity imported from US Nanocomposites, USA & micro-sized alumina powder particle size of about 45 microns purchased from Merck, India. The FESEM image of micro-sized alumina powder is shown in figure 1. Table 1 presents the experimental parameters and variables pertaining to the aluminium-alumina metal matrix composites fabrication.

2.2 Manufacturing of Composites

Aluminium-alumina metal matrix composites were manufactured using stir-squeeze casting system equipment, as shown in Figure 2. The stir casting equipment made up of conical-shaped graphite crucible which avoid in react at elevated temperature with aluminium. A crucible of high ceramic alumina is put in a muffle. In this process, the Scrap Aluminium Alloy Wheels (SAAWs) of adequate size is heated up to 900°C in an electric furnace. Current varies from 30 and 70 A, a constant voltage of 220 V was used. 1%wt of Mg was introduced into the melt for the improvement of wettability of the aluminium and the alumina. The preheating was done separately for both nano-sized Al_2O_3 and micro-sized Al_2O_3 alumina particles, then poured into the melt. Ultrasonic treatment was performed for 60 sec, 120 sec and 180 sec in addition to 5 minutes mechanical stirring. Before pouring the molten metal into the preheated die, the temperature was eventually lowered to make the semi liquid. Then squeezing operation was done at 100 MPa during solidification process. Based on the literature, the process parameters of squeeze casting were selected meticulously to attain better mechanical properties, namely tensile, compressive, hardness and wear resistance. The volume % of micro, nano alumina and SAAW are presented in Table 1.

Figure 1: FESEM Image of Alumina (micron) Powder from Merck, India.

2.3 Density and Porosity

A polished specimen of size 13mm diameter and 25mm height was used for measurement of the density of S1, S2 and S3 composites. An electronic weighing scale was used for the calculation of the mass of S1, S2 and S3 composites. Then, specimen height and diameter determined with the help of vernier calliper having the least count of 0.001mm to find out the volume. Finally, dividing mass by volume of the composites resulted in experimental density of the specimen. The percentage of porosity estimated by the below way.

$$\text{Percentage of porosity} = \frac{\text{density(theoretical)} - \text{density(experimental)}}{\text{experimental density}} \times 100$$

Also, theoretical density was determined by $0.95 * \text{density of aluminium} + 0.5 * \text{density of alumina}$.

2.4 Microstructural Studies

Metallographic specimens were prepared from S1, S2 and S3 composites by usual processes. Standard size of the specimen is cut out of the part, positioned by casting the specimen in a bind able plastic material. The aim of the mounting of the specimen was to secure the part to be ground and polished better to protect the specimen's edges and to be easier to fasten into automated machines or to protect it manually. Besides, specimen orientation can also be easily controlled by mounting. In a microscopic examination of aluminium-alumina composites material, grey silicon carbide used as an abrasive material for polishing to create a flat, defect-free surface and texture. Finally, the specimen was immersed in Keller's etchant. After etching, the specimen was examined by an optical microscope.

Table 1: Summary of Experimental Parameters and Variables

Variables	Experiments		
	S1	S2	S3
Al ₂ O ₃ (μ m)wt%	4	7	7
Al ₂ O ₃ (nm)wt%	1	3	1
Amplitude (% of power drawn)	60	60	80
Pulse time(sec)	60	180	120

2.5 Mechanical Properties Assessment

Tensile test was conducted on aluminium-alumina composites using an automatic loadmaster series-III as per ASTM E8 standard. The standard sub size specimen used for the tensile test prepared in accordance with ASTM B557-14 is given in figure3. A compression test was performed on UTM equipment TUE make, C 1000 model in accordance with ASTM E9-09 using a 13mm diameter and 25mm height specimen to evaluate the compressive behaviour, strength and fracture pattern of the aluminium- alumina composites.

Figure 2: Stir-Squeeze Casting Set up.

As per ASTM E10-07, the hardness of aluminium-alumina composites was measured on a macro examination sample at 30 °C. A ball indenter of 1.588 mm size and load of 10 kg was used in the Brinell hardness tester for 15 seconds. Average hardness values were taken from different locations for fair values.

Figure 3: Sub-Standard Size of Tension Specimen.

3. RESULTS & DISCUSSIONS

3.1 Microstructural Analysis – Optical

The optical micrographs of aluminium-alumina composites S1, S2 and S3 are shown in figure4 at 300X magnification. In the picture, both alumina reinforcement and eutectic silicon phase are represented by grey particles, aluminium matrix is indicated by white regions and porous defects are indicated by black regions. A flake-like microstructure observed in the sample of aluminium-alumina composites and nanoparticles distributed throughout the aluminium matrix because of the squeeze pressure applied, where the grain boundaries provided the appropriate area to fill the reinforcement particles. As the ultrasonic vibrator used to mix the molten metal in a solidification process, ultrasonic heat extraction and a variation on temperature decrease in the solidification front, leading towards the instable interface between liquid / solid [11]. The dendrites are broken with the help of ultrasonic flow by the similar force on the arm, that becomes an artificial nuclei rich enough site. In the meantime, the wettability has improved among nanoparticles and matrix due to clustered particles by tremendous force of transient cavitation and gas layer removed from the nanoparticles surface.

Figure 4: Microstructure for Three Types of Composites.

A part of broken cluster particles becomes active and involved a solidification nuclei point. Hence, the ultrasonic vibration caused for the fine grain with equiaxed microstructure [12]. Reconfirmation of this finding was done with pore size and distribution of S1, S2 and S3 aluminium-alumina composites.

3.2 Mechanical Properties Analysis – Porosity, Hardness, Tensile and Compression Strength

The properties of materials are affected by porosity depends on many characteristics. Figure 5 illustrated the pore

distribution corresponding to the produced S1, S2 and S3 composite materials. It is understood that the complete avoidance of porosity during aluminium casting is impracticable due to the reduction resulting of contraction in volume [13].

The reasons of this phenomena are because of liquid-form gas solubility of solid metal. Shrinkage and gas evolution increase the pore forming sites by adding reinforcement particles; thus, composites exhibited more porosity than pure matrix content. Moreover, a composite material with lower porosity, even with some available empty spaces will be stronger. The high pressure within which the squeeze casting process used during solidification, lead to the minimal defects in casting particularly porosity less casting component. It is easier and faster and relatively efficient to achieve an experimental density very closely to the theoretical density by using the squeeze casting process [14]. As the relative density of the composites decreases by agglomeration therefore the micro-hardness and strengths, setting up spaces around agglomerations for inducing the crack initiation from pores. The addition of nanoparticles with higher stirring time would lead to a higher porosity level. In addition, the contact surface between aluminium and nanoparticles is mostly related which in effect will raise the level of porosity as well as the agitation in molten composites due to the long stirring process[15].

The grain distribution of the aluminium-alumina composites materials S1, S2 and S3 is shown in figure 6. In addition to the effect of agglomeration, the grain boundaries would be filled with nano particles if its material reached a critical value [16]. This forms a grain boundary with brittle phase, through the boundaries with grain and weakens the strength of a composites. This indicates that over the mechanical properties of the strengthening mechanisms prevails the agglomeration and hence making of grain boundaries surrounded with brittle phase.

Figure 5: Pores Distribution for Three Types of Composites.

Figure7 presents the hardness values of S1, S2 and S3 composites. The hardness values achieved for three composites S1, S2 and S3 did not vary many degrees. The S1 composite obtained a highest hardness than others. The main reason for higher hardness values of S1 was because of the small size alumina particles compared to others. Also, it can be confirmed that the small grain size with limited pores exhibited in figure 5 a) and 6 a).

Figure 6: Grain Size Distribution for Three Types of Composites.

The ultimate tensile strength values of S1, S2 and S3 aluminium-alumina composites was observed and shown in figure 8. Further grain refinement and reduced porosity is accomplished in cases of squeezed nano-composites. With the squeeze casting process, the squeezing pressure effect, the space between the dendrites continuously decreased, likely to result in a finer grain, and homogeneous microstructure. It could have strengthened the ultimate tensile strength of nanocomposites casted over stir-cast nanocomposites by squeezing. The properties of MMCs depend on the distribution of reinforced particle and interface bonding of particles and matrix, not only on the particles and their volume fractions as well. It has been observed that owing to an inhomogeneous distribution and agglomeration of the nano-size reinforcement. However, the change in the reinforcement does not impact mechanical properties in Al–Al₂O₃ nanoparticles more than 2 vol.%. [17].

Figure 7: The Hardness of Three Types of Composites.

The overall tensile strength and yield strength of the composites increased at the same time as that of the matrix material. It could be related to combined grain refinement factors and high thermal interface stress related to coefficients of thermal expansion between the matrix of Al₂O₃, and reinforcement. Also, effective transition of tensile lead to uniform distribution of nano particles. Figure 8 also exhibited the compressive strength of S1, S2 and S3 aluminium-alumina composites. The compression test was carried until the fracturing stage, although all samples budged, deformed, in barrel shape and the sample not detached in the diagonal plane after a fracture. The increased strength of S3 compression is attributed to the greater percentage of silicon compounds in the matrix: S3 composites. The morphological study has improved mechanical properties by the combination of acicular form on the grain boundaries with eutectic silicon and alumina reinforcement.

Figure 8: Mechanical Properties of Three Types of Composites.

Nevertheless, at grain boundaries, the intergranular distribution had higher mechanical properties and hindered failure along grain boundaries. Also, the mechanical properties enhanced with a matrix grain size, particularly with alumina reinforcement. Further, silicon eutectic phase formed by many nucleation sites through the small size of alumina reinforcement. Thus, it can be concluded that the specific size of the form of reinforcement greatly affected the mechanical properties.

4. CONCLUSIONS

The aluminium-alumina metal matrix composites were successfully made via the stir-squeeze casting process.

The main findings are summarized below:

- The metallographic examination revealed that all three composite S1, S2 and S3 had a uniform alumina particle distribution with flake-like microstructure throughout the sample.
- The observed porosity of composites in micrographs correlated with the percentage of porosity calculated.
- The highest hardness (57HRB) was obtained for S1 composites followed by S3 and S2
- The composites S3 exhibited the optimum compressive strength and ultimate tensile strength of 382MPa and 121MPa respectively. Next, higher values were obtained for composites S1 followed by S2.
- A major improvement of aluminium-alumina composites was observed in particular with ultimate tensile and compressive strength because of the development of alumina combined with silicon eutectic phases, which exhibits a marginally acicular, mainly spherical structures with certain blunted and needle-like forms.

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