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RESEARCH

Effect of Zircon Silicate Reinforcements on the Microstructure and Properties of as Cast Al-4.5Cu Matrix Particulate Composites Synthesized via Squeeze Cast Route

The as-cast microstructure and properties of Al-4.5Cu/$ZrSiO_4$ particulate composite synthesized via squeezed casting route was studied, varying the percentage $ZrSiO_4$ in the range of 5-25wt%. The result obtained revealed that addition of $ZrSiO_4$ reinforcements, increased the hardness value and apparent porosity by 107.65 and 34.23% respectively and decrease impact energy by 43.16 %. As the weight percent of $ZrSiO_4$ increases in the matrix alloy, the yield and ultimate tensile strength increased by 156.52 and 155.81% up to a maximum of 15% $ZrSiO_4$ addition respectively. The distribution of the brittle $ZrSiO_4$ phase in the ductile matrix alloy led to increase strength and hardness values. These results had shown that, additions of $ZrSiO_4$ particles to Al-4.5Cu matrix alloy improved properties.

Keywords: *$ZrSiO_4$; Al-4.5Cu; reinforcement, tensile strength and microstructure*

1. INTRODUCTION

Three decades of intensive research have provided wealth of new scientific knowledge on the intrinsic and extrinsic effects of ceramic reinforcement to metals and their alloys [1, 2, 3]. The successes of these various researches have stimulated application of metal matrix composite in the design of many engineering and non engineering component [1, 3].

Aluminium matrix composite (AMCs) have shown high mechanical properties such as high strength, high stiffness, wear resistance and good elevated temperature properties when compared to the unreinforced matrix alloy, which has lead to the use of aluminium matrix composite in the following; electronic heat sinks, automotive drive shaft, ground vehicles brake rotors, jet fighters, air craft firms, electronic instrument racks, satellite struts, crankshafts, gear parts brake drum cylinder block and suspension arms [4, 5].

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New researches on metal matrix composite have focus on particle reinforcement due to low cost of the ceramic reinforcement and less complex fabrication technique [1, 9]. Stirring casting route has been used successfully to synthesis metal matrix composite.

However, initial investigations employing a squeeze casting process (the application of external pressure on the molten metal) for aluminum-based MMC's have also demonstrated many advantages over the stir cast production technique, such as: (a) better compatibility between the metal matrix and the reinforcement particles, (b) a more improved structure of the matrix alloy (c) better mechanical properties and (d) pressure activation of the reinforcement-metal interface [6-10, 25]. From the onset of the space era, metal-matrix composites (MMCs), with high specific stiffness and near-zero coefficient of thermal expansion (CTE), have been developed for space applications [11]. MMCs possess such as high-temperature capability, high thermal conductivity and low CTE in addition to the above named properties. Those potential benefits generated optimism for MMCs for critical space system applications. Also joints and attachment fittings for truss structures, longerons, electronic packages, thermal planes, mechanism housings, and bushings are made from MMCs [10-11].

Apart from the use of Particles Aluminium Matrix Composites (PAMCs) in space application, PAMCs have been successfully used as components in automotive, aerospace, opto-mechanical assemblies and thermal management. PAMCs are used as fan exit guide vane (FEGV) in the gas turbine engine, as ventral fins and fuel access cover doors in gas military aircraft [7-11]. Fan exit guide vanes (FEGV) component made up of PAMCs are used in turbine engine [12]. Also PAMCs are used as rotating blade sleeves in helicopters. Flight [1, 2]

Microstructural examination of Metal matrix composites reveals an interfacial zone between the metal matrix and the reinforcement material [13-15]. This zone between these two phases (metal matrix and reinforcement) is one essential part of MMCs. Interfacial bonds in the zone are developed from physical or chemical interactions, interfacial frictional stress and thermal stresses due to differences between coefficient of thermal expansion of reinforcement and matrix. The understanding and control of the underlying interfacial phenomena governing the transmission of thermal, electrical, and mechanical properties across the whole composite might become of paramount importance when designing MMC for a particular task [14, 15].

Zircon silicate is naturally occurring sand. Zirconium silicate contains mainly zirconium oxide and silicon oxide with a minor amount of potassium, gold and calcium oxide [16]. It possesses properties. Such as; Use temperatures up to 2400°C, High density, Low thermal conductivity (20% that of alumina), Chemical inertness, Resistance to molten metals, Ionic electrical conduction, Wear resistance, High fracture toughness and High hardness [17]. This has made it a good reinforcer for the production of MMCs for engineering applications.

Das [16] studied the microstructure and aging behaviour of Al_2O_3/$ZrSiO_4$ particulate reinforced Al-4.5wt%Cu matrix composite, synthesized by stir casting route. From the investigation the authors found out that the Al_2O_3 reinforced composite showed more accelerated aging properties compared to the $ZrSiO_4$ reinforced composite.

Lack of data of comparison between the reinforced and unreinforced metal alloy could negatively affect the engineering areas of applications [18-23]. Adequate understanding of the physical and mechanical properties of the metal matrix composite is important, because these properties help to correctly predict the areas of application of the material [24, 25]. The objectives of the present work are therefore to investigate the microstructure and properties of as cast Al-4.5%Cu alloy and its composites reinforced with zircon sand particles using the squeeze casting production technique.

2. EXPERIMENTAL METHODS

2.1 Specimen preparation

The Al-Cu/$ZrSiO_4$ composites used for this research were synthesized by the squeeze cast method at the laboratory of the Department of Metallurgical Engineering, Ahmadu Bello University, Zaria, Kaduna State Nigeria. Zircon sand used for this research was received from the mineral department of the National Metallurgical Development Center (NMDC) Jos, Nigeria. The composition of the zircon sand is given in the table 1. High purity aluminium electrical wires were obtained from Northern Cable Company NOCACO (Kaduna).

Table 1. Chemical composition of zircon sand particles (wt %)

ZrO_2	SiO_2	CaO	TiO_2	Cr_2O_3	MnO	Fe_2O_3	PbO	Rb_2O	Y_2O_3	Ag_2O	CeO_2	Yb_2O_3	HfO_2	Bi_2O_3	U_3O_8
65.6	21.3	.27	.076	.075	.045	1.97	.167	.059	.73	4.73	.098	.41	2.53	.37	.57

The samples were produced by keeping the percentage of copper constant while varying the percentage of aluminium and zircon sand. The zircon sand was varied in the range 5-25%wt. the high purity electrical wires free from contamination were charged in a graphite crucible kept in an electrical resistance furnace and 0.01%$NaNO_3$ powder was used as a cover for melting the alloy. The $NaNO_3$ addition was aim at reducing oxidation of aluminium and creating a protective atmosphere inside the furnace. At the beginning of melting of pure aluminium, the furnace temperature was raised to 750°C. 4.5 percent copper was introduced into the molten pure aluminium using hardener (50%Al-50%Cu). The reason was to facilitate easy melting of the charged copper. With progressive melting the furnace temperature was raised to 800°C and the melt was held at this temperature for 12minutes. Then, skimming was done to remove oxides and impurities.

For each melting 250 grams of charge material was used to produce the alloy. $ZrSiO_4$ particulate of particle size in the range of 2-15µm was preheated to a temperature of 1200°C so as to ensure good interfacial bonding between the alloy matrix and the reinforcement. Then, the molten aluminium alloy is mixed with the preheated zircon sand and the mixture is stirred thoroughly with a mechanical stirrer for 15minutes at an average stirring rate of 180rpm. In the final stirring stage the furnace temperature was controlled between 760°C and 780°C and pouring was controlled to a temperature of about 750°C. A squeeze casting machine with a press capacity 10MPa, approach speed of 50m/s, return force of 5MPa, pressing speed of between 1-10mm/s, die length of 0.18m and die diameter of 0.02m was used to prepare the test samples. The schematic diagram illustrating sequence of steps adopted during the squeeze casting process is shown in Figure 1.

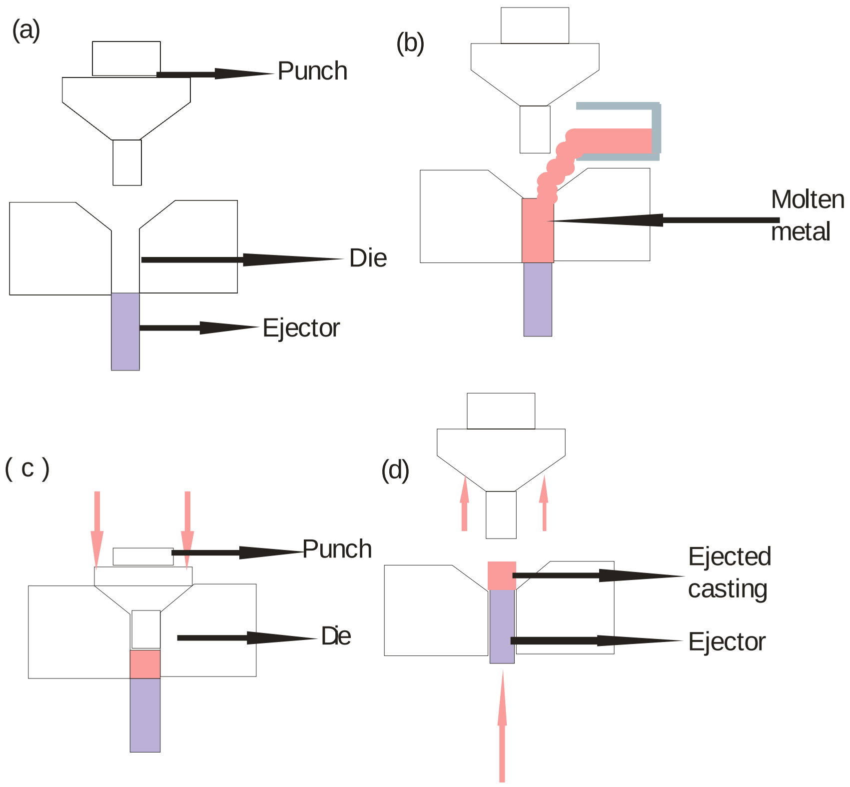

Figure 1. Schematic, showing the sequence of steps during squeeze casting operation (a) Pre-heated die and punch. (b) Molten metal poured into the die cavity. (c) Application of squeeze pressure. (d) Ejection of solidified casting

2.2 Microstructural examination

Samples for metallographic analysis were cut from the as cast unreinforced and reinforced cast samples of the Al-Cu/$ZrSiO_4$ particulate composite. The cut samples ere then mounted in bakelite and mechanically grounded in two stages: (a) rough grinding using grades of SiC impregnated emery paper (60-320) grit size and (b) fine grinding using grades of above mentioned paper of grits size (380-600). During grinding water was used as coolant and 90°C change in the grinding direction was done for shift from lower grit size to a higher one. The grinded samples were then polished using 1µm and 0.5µm size alumina polishing powder suspended in distilled water. Etching of the polished samples was done using Keller's reagent. The structure obtained was recorded using an optical microscope with an inbuilt camera.

2.3 Determination of apparent porosity

The as cast test samples were washed several time with distilled water and dried in oven at 120°C for 20minutes to obtain weight W_{WM}. The specimen was then suspended in distilled water and boiled in a hot plate for 30minutes. While, in the hot plate after boiling cold water was added to displace the hot water and the weight W_{IM} was measured on a digital weighing balance hinged on a tripod stand. The test sample was removed from the water and extra water wiped off using a slightly wet towel and the weight W_{AS} of the sample was measured. Hence the apparent porosities were calculated using the formula below:

$$P_a = \frac{W_{AS} - W_{WM}}{W_{AS} - W_{IM}} \text{ x } 100\ (\%)$$

Where:
P_a = Apparent porosity
W_{AS} = Already wiped weight
W_{WM} = weight without moisture
W_{IM} = Weight when immersed in moisture

2.4 Determination of hardness values

Hardness values of the samples were determined using the B scale of Rockwell hardness testing machine. The indenter used was a 1.56mm steel ball. Minor load of 10kg and major load of 100kg was applied. Hardness of 101.2HRB standard block was used.

2.5 Determination of impact strength

The Avery Denison impact testing machine with a test capacity of 300 Joules was used to carry out this test. The mass of the hammer was 22.7kg and the striking velocity was 3.5m/s. Charpy impact tests were conducted on notched samples. Standard square impact test samples measured 50mm x 10mm x 10mm with notch depth of 2mm and a notch tip radius 2mm at angle of 45° was used for this research.

2.6 Determination of tensile properties

Tinus-Olsen tensile testing machine was used to test the tensile properties of the as cast samples at a strain rate of $.002S^{-1}$, adopting the standard tensile test specification of 12.5mm original diameter and gauge length of 50mm . The yield and ultimate tensile strength, percentage elongation and reduction in cross-section of the samples were determined.

3. RESULTS AND DISCUSSION

3.1 Results

The various microstructures developed from the different $ZrSiO_4$ addition are shown in the Figures 2-7. The result of the porosity of the various cast compositions are shown in Figure 8. The effect of $ZrSiO_4$ addition on the hardness, impact and tensile properties of Al-Cu alloy are shown in Figures 9-12.

3.2 Discussion

The microstructure of the unreinforced matrix alloy is shown in Figure 2. The structure reveals uniform grain size separated by thin 70%black grain boundaries with even faint grayish precipitate of $CuAl_2$. Figures 3-7 shows the microstructure of the reinforced alloy with $ZrSiO_4$ particles. The structure reveals an increase in the dark portion as the zircon sand addition was increased. The ceramics phase is shown as dark phase, while the metal matrix phase is grayish in colour. Figure 5 show a uniform distribution of zircon sand particles in the Al-4.5Cu matrix, as dark spots. Pronounce segregation and agglomeration was observed in Figures 5-6 was attributed to high proportion of $ZrSiO_4$ particle in the structure. The structures are in line with those obtained in [1]

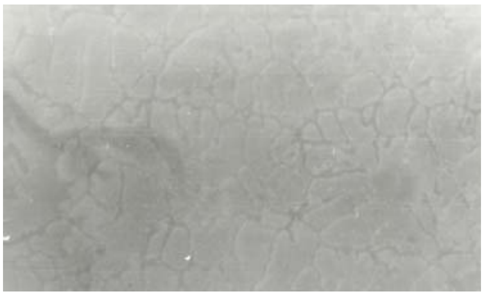

Figure 2. Microstructure of the unreinforced Al-4.5Cu Alloy (grey) magnification x 125

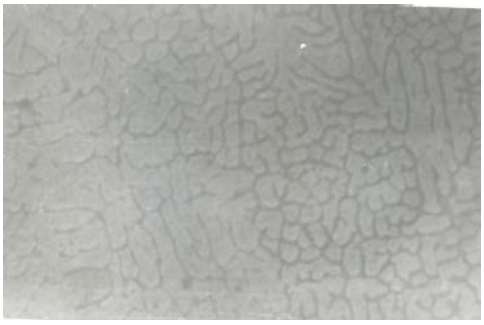

Figure 3. Microstructure of the reinforced Al-4.5Cu alloy (5% $ZrSiO_4$). Finer grains and thicker grain boundary with little Patches of $ZrSiO_4$ magnification x 125

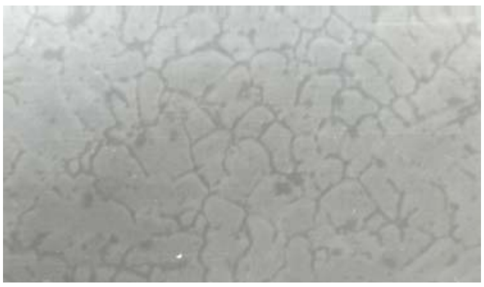

Figure 4. Microstructure of Al-4.5Cu alloy reinforced with 10% $ZrSiO_4$. More enlargements of the grain boundaries with larger patches of $ZrSiO_4$ (black) magnification x 125

Figure 5. Microstructure of Al-4.5Cu alloy reinforced with 15%$ZrSiO_4$. Uniform distribution of $ZrSiO_4$ (black) particles. Magnification X 125

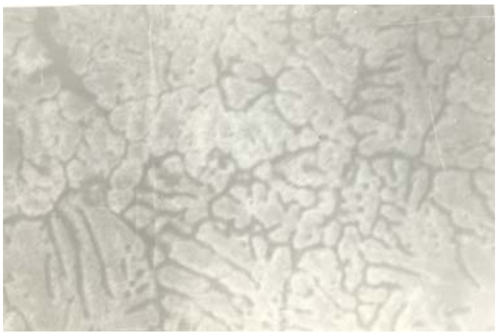

Figure 6. Microstructure of Al-4.5Cu alloy reinforced 20% $ZrSiO_4$. $ZrSiO_4$ particles (black) magnification x 125

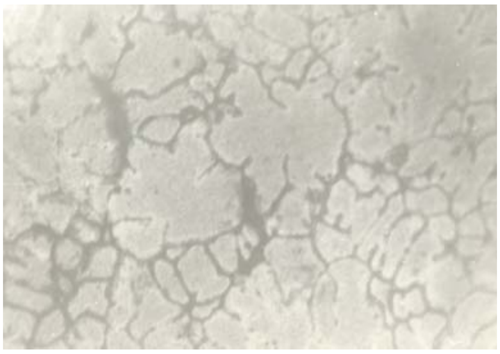

Figure 7. Microstructure of Al-4.5Cu alloy reinforced with 25% $ZrSiO_4$. ZrSiO4 particles (black) magnification x 125

The apparent porosity values of the MMCs reinforced with $ZrSiO_4$ slightly increased with percentage $ZrSiO_4$ addition shown in figure 8. the low values of the porosity obtained in this research work was as a result of the squeezing casting production method, which enhanced good wettability and interfacial bond, attributed to rapid heat extraction and compacting of the cast sample by the steel based casting section and instantaneous application of pressure to the molten casting by the squeeze casting respectively. Hence the reinforcement remained in the melt till cooling is completed rather than float to the top surface.

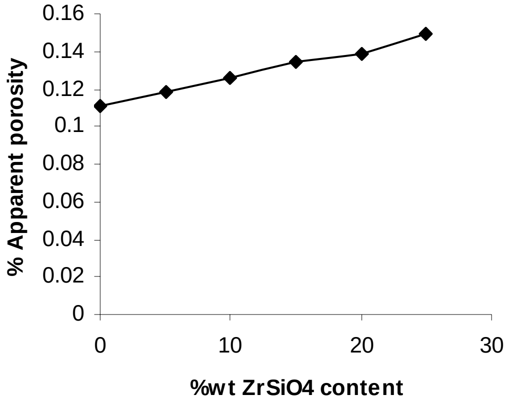

Figure.8. Variation of percentage of apparent porosity with weight percentage of $ZrSiO_4$ content

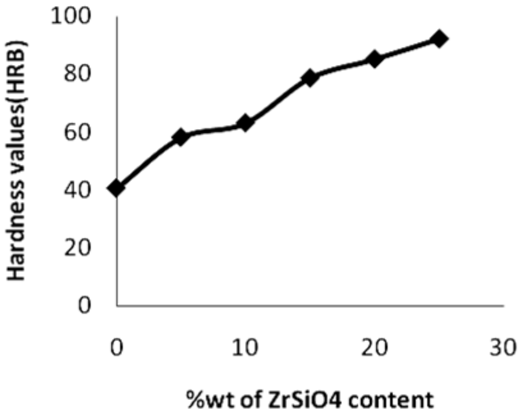

Figure 9. Variation of hardness with weight percentage of $ZrSiO_4$ content

The hardness value increases as the percentage of $ZrSiO_4$ addition increases in the alloy figure 9. This is due to high proportion of the hard and brittle phase of the zircon sand in the alloy. The zircon sand addition to the matrix alloy results to elastic and plastic incompatibility due to differences in the coefficient of thermal expansion in the hard reinforcing and soft matrix alloy, which causes high dislocation density [1]. The high dislocation density also contributed to high hardness value.

The yield strength and ultimate increased with increasing % $ZrSiO_4$ addition up to maximum values of 168.10 and 231.48N/mm^2 at 15% $ZrSiO_4$ addition respectively. Then, decreases to 123.281 and 189.394 N/mm^2 at 25% $ZrSiO_4$ addition (see Figure 10). The highest strength observed at the 15% $ZrSiO_4$ addition was attributed to a more uniform distribution of the reinforcement while above 15% $ZrSiO_4$ segregation of the particles along the grain boundary grew high, resulting to a decrease in the bonding strength along the grain boundary (see Figures 6 and 7).

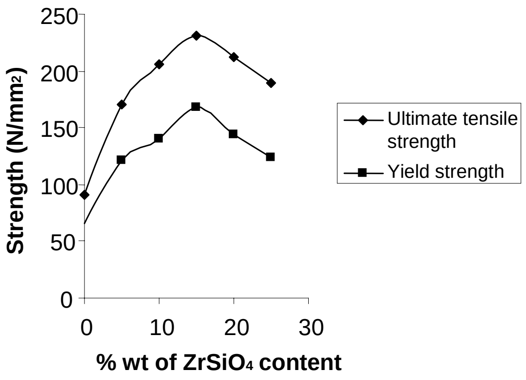

Figure. 10. Variation of ultimate tensile and yield strength with weight percentage of $ZrSiO_4$ content

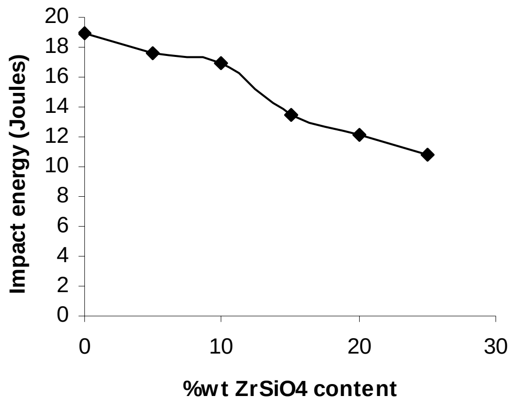

Figure 11. Variation of impact energy with weight percentage of $ZrSiO_4$ content

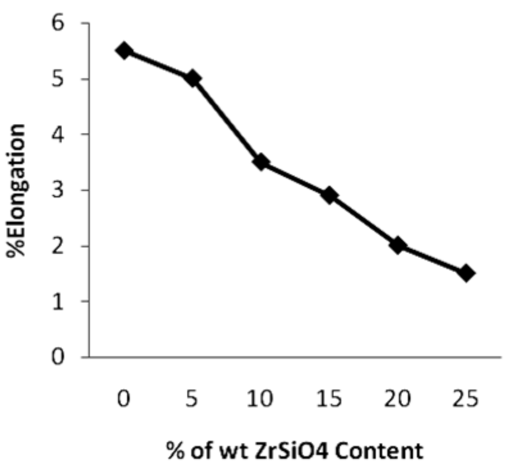

Figure 12. Variation of percentage elongation with weight percentage of $ZrSiO_4$ content

The impact energy and percentage elongation shown in figures. 11 and 12 decreased as the percentage of $ZrSiO_4$ addition increases in the alloy. The brittle nature of the reinforcing agent led to the reduction in the impact energy of the composite. Also results show that 5% $ZrSiO_4$ reinforced composite has the higher toughness value than that of 10-25% reinforced composite which is attributed to smaller proportion of the brittle reinforcement on the matrix alloy.

Hence in toughness inverse relationship with brittleness was also confirmed. It is therefore clear that an over all improvement in the mechanical properties is observed by reinforcement, which is in agreement with the result obtained by [1, 10].

4. CONCLUSION

From the result of the investigation in this research work it could be concluded that addition of $ZrSiO_4$ particles using Al-4.5%Cu alloy increased both the strength and hardness and an over all reduction in toughness and density. Also, little increase in the apparent porosity of the composite with percentage increase in $ZrSiO_4$ addition was observed. From the result, maximum service performance of the Al-4.5Cu/$ZrSiO_4$ particulate composite synthesis via squeeze casting should not exceed 15% in order to develop balance in the necessary properties.

Pronounce increase in hardness value was observed by reinforcing the matrix alloy with 5-25% zircon sand. Al-4.5Cu/15%$ZrSiO_4$ particulate composite could be appreciable in automobile industries (brake drum, crankshafts, values and suspension arms), recreational products (golf club shaft and head, skating shoe, bicycle frames and base ball shaft) and in construction company (truss structure).

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