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[1.] Emmanuel I. AKPAN, [2.] I. U. ABHULIMEN, [3.] S. O. ADEOSUN, [4.] O. P. GBENEBOR

MECHANICAL CHARACTERISTICS OF Al-$CaCO_3$/Zn COMPOSITES

[1-2,4.] Department of Materials and Production Engineering, Ambrose Alli University, Ekpoma, NIGERIA
[3.] Department of Metallurgical and Materials Engineering, University of Lagos, Lagos, NIGERIA

Abstract: Effect of $CaCO_3$ particle (100μm) addition on the mechanical properties of aluminium-0.5% Si alloy has been investigated. Samples are produced using conventional stir casting method and homogenized at 550^{0}C for four hours. Mechanical properties studied are tensile strength, tensile elongation, hardness and fracture strength. Cast samples containing 25wt%$CaCO_3$ and 5wt%Zn exhibit the highest tensile strength (134.3MPa) owing to a breakdown of grain structure caused by the dissolution of $CaAl_2$ and AlFeSi into the α-aluminium matrix. Samples containing 5wt%$CaCO_3$and 5wt%Zn have superior hardness (131.1MPa) due to high volume fraction of precipitates in the microstructure. The microstructure shows the presence of AlFeSi, Mg_2Si and $CaAl_2$intermetallics in the α- aluminium matrix.
Keywords: $CaCO_3$ particles, composites, precipitates, Intermetallics, casting

1. INTRODUCTION

Materials are the core of all technological advances, and are essential for human survival on earth. Aspects of daily life such as clothing, transportation, construction, communication are influenced by materials. The advancements of societies are tied to ability to process materials to meet needs. Mastering the development, synthesis and processing of these materials opens opportunities that are scarcely thought of few decades ago. Everyday new limits and heights are attained in engineering applications. This has forced materials science into a rapid ladder of development, from the very heavy weight machine beds to the flyweight of electronic circuit boards, high temperature performance of super alloys and the versatility of ductile steels. Engineering materials have proven to be a long chain of evolution in the history of technology [1].

Structural components made from aluminum alloys are vital to the aerospace, automobile and building industries because of light weight, high strength, good formability and high corrosion resistance. However, aluminum alloys system exhibit poor tribological properties. The desire to develop new materials with improved wear resistance and better tribological properties without compromising the strength to weight ratio, can be achieved using metal matrix composites [2, 3]. Discontinuously reinforced aluminum matrix composites are fast emerging as engineering materials and competing with common metals and alloys because of its higher specific strength, specific modulus and good wear resistance over unreinforced alloys [3]. Pure aluminum possess weak matrices, but the presence of reinforcing particles results in significant matrix strengthening. Iron – ore, steel dust, titanium oxide, aluminum oxide, iron powder, iron oxide, granulated slag and arc furnace dust have been used as reinforcement in aluminum matrix with significant improvement in stiffness and strength than its conventional alloys [4 – 11].

Previous research has shown that addition of particles or elemental powders such as iron powder, aluminium oxide, steel dust, ceramic particles and silicon carbide to aluminium alloy matrix produced significant improvement in the mechanical properties of the alloy with good combination of ductility and tensile strength which will enable its use in structural applications [2, 12-16]. Due to lower recovery, the addition of Mg to aluminium strongly increases the dislocation density, leading to an increase of the proof stress over a wide range of strain. The Mg addition, however, increases the strain corresponding to the formation of a stable microstructure and the saturated strength value. Moreover, Mg and Zn solute segregation in the grain boundary regions cause improvement in the stability of the alloy structure. Agrawal et al [17] observed that the tensile strength and hardness of the resultant alloys increase with the addition of Zinc, while magnesium also has a positive impact on the tensile strength of the alloy.

Calcium carbonate ($CaCO_3$) powder is a common filler in many industries, such as the cement industry where it is used in the production process. Natural $CaCO_3$ can be obtained from calcite and aragonite in mines. This $CaCO_3$ can be found in limestone, spot disland and other types of rock. Waste $CaCO_3$ powder is produced as a by-product in stone sawing factories with a typical particle size of 0.5 – 1μm. Addition of waste $CaCO_3$ powder particles in aluminium alloys and its effect on the alloy's mechanical properties have not been studied. However, it is noted that the presence of Calcium can cause the development of useful intermetallics in aluminium alloys. Stir casting route because of its simplicity and easy adaptability with all shape casting processes is the most promising casting route for synthesizing well-dispersed discontinuous reinforcement in the composite matrix [18]. The study of aluminum metal matrix composites in relation to reinforcement size, deformation processing and subsequent annealing is of outmost importance for strength improvement. In this study calcium carbonate particle is added to AA6063 alloy with zinc and magnesium as modifiers to assess its mechanical and microstructural responses. Experiment was designed by the authors to aid the assessment of the effect of calcium carbonate powder addition in the presence of zinc. Magnesium was added to stabilize the microstructure of the wrought aluminium alloy.

2. EXPERIMENTAL METHODOLOGY – MATERIALS AND PREPARATION

The aluminium alloy (25kg) used for this study is obtained from Nigeria Extrusion Company, Oshodi, Lagos, Nigeria. The spectrometer analysis of this alloy is shown in Table 1. The aluminium alloy is modified by addition of Zinc (Zn) and Magnesium (Mg) obtained from Nigeria Extrusion Company, Oshodi, Lagos, Nigeria, while $CaCO_3$ is obtained from a local quarry factory in Ikpeshi Igara, Edo state, Nigeria. Samples are cast with 5wt.% and 7 wt.% zinc in each case, with calcium carbonate powder addition ranging between 0 - 25 wt. %. 2.5 wt.% of magnesium is added to all samples. Samples are cast into rectangular shapes using Plaster of Paris (POP). Weighed samples of aluminium, magnesium and zinc are charged into a crucible and placed in a diesel fired furnace fired and operated at $700^{o}C$. The weighed calcium carbonate is added to the molten mix and stirred thoroughly before pouring into the POP mould. The cast sample is allowed to solidify before removal from the mould.

Table 1: Spectrophotometric Analysis of virgin Aluminium

Element	Fe	Si	Mn	Cu	Zn	Ti	Mg	Pb	Sn	Al
Composition	0.395	0.540	0.148	0.197	0.097	0.017	0.098	0.014	0.002	98.28

Cast samples are machined into tensile, hardness and metallographic specimens. Some specimens are tested in as-cast condition while others are homogenized in a muffle furnace at $550^{0}C$ for 4 hours and air-cooled. Tensile test is conducted using a digital Instron Universal Tensile Testing Machine (M500) with each specimen placed in the testing machine, locked at each end, and stretched by applying tension until it fractures. The loads and the extension are measured by means of a load cell and extensometer. During the application of tension, the elongation of the gauge section is recorded against the applied force.

A Vickers hardness test is carried out on the specimens using a Leco AT700 digital Micro Hardness Tester with an applied load of 490.3mN (approximately 50kgf). Each specimen is placed on the anvil of the tester and the load is applied with a dwell time of 10 seconds. Three to six readings are taken for each of the specimen and the average values are calculated from two closely related values.

The machined specimens are rough ground for microstructural analysis by first clamping each specimen on a bench vice, filling to appropriate smoothness and finely ground in succession using 60, 120, 180, 240, 320, 400, 600 and 800/2400 microns emery papers. The ground surfaces of the specimens are polished with aluminium powder kept moist by continuous application of water (to reduce friction)to remove scratches obtain during the grinding process. Each polished specimen is etched in a solution of 0.5% of hydrofluoric acid (HF). The etched specimens are finally examined using a digital metallurgical microscope at a magnification of x200 and x400.

3. RESULTS AND DISCUSSION

3.1. Tensile Strength

Figure 1 shows the tensile strength of as-cast samples with percentage weight of $CaCO_3$. The tensile strength of Al5wt%Zn composite increase with $CaCO_3$ addition to a peak value of 134 MPa at 25wt% $CaCO_3$ addition. On the other hand, Al10wt%Zn composite show appreciable increase in the tensile strength as the wt% of $CaCO_3$ addition increases, to a peak value (101 MPa) at 5wt% $CaCO_3$beyond which it declines. Figure 2is tensile strength response of homogenized samples with percentage weight of $CaCO_3$. The tensile strength of Al5wt%Zn composite increase as the addition of $CaCO_3$ increases to a peak value of 113.92MPa at.5wt% $CaCO_3$ addition and declines with further filler addition. On the other hand, Al10wt%Zn composite show an initial decrease in the tensile strength as the wt% of $CaCO_3$ addition increases to a minimum of 51MPa at 10wt.% $CaCO_3$. However, there is a rise with further addition of $CaCO_3$ to a peak of 110.67MPa at.15wt% $CaCO_3$ and thereafter declines.

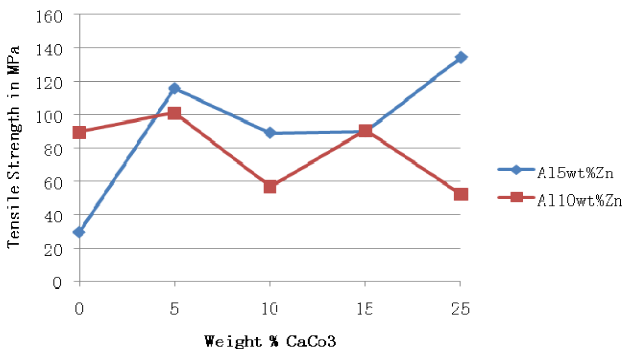

Figure 1: Ultimate tensile strength of zinc modified aluminum-0.5%Si / $CaCo_3$ composite in as-cast state

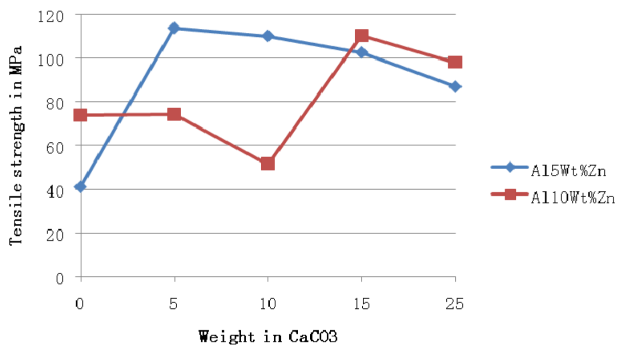

Figure 2: Ultimate tensile strength zinc modified homogenized aluminum-0.5%Si / $CaCo_3$ composite

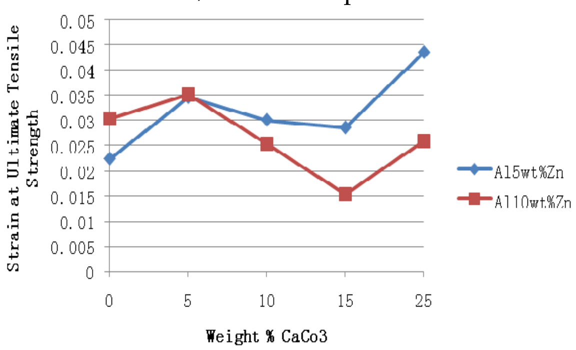

Figure 3: Strain Responses at UTS for zinc modified aluminum-0.5%Si / $CaCo_3$ composite in as-cast state

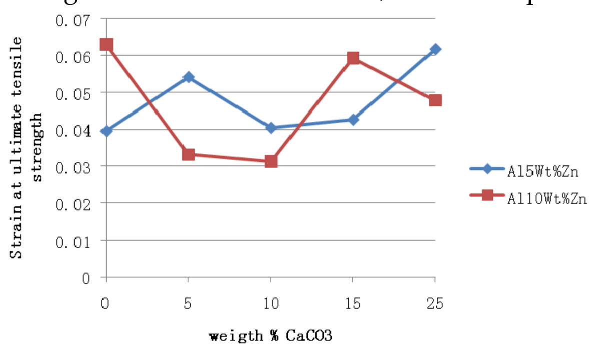

Figure 4: Strain Responses at UTS for zinc modified homogenized aluminum-0.5%Si / $CaCo_3$ composite

3.2. Tensile Elongation at UTS

The strain responses at ultimate tensile strength in relation to percentage weight of $CaCO_3$, of the aluminium alloy are shown in Figure 3. Both Al5wt%Zn and Al10wt%Zn composite show initial appreciable response to increase in percent $CaCO_3$addition with 3.464% and 3.516% elongations respectively at wt.5% $CaCO_3$. On further filler addition, there is a decrease in the elongation until at 15wt.% $CaCO_3$ where the strain rose again to 4.36% for Al5wt%Zn and 2.587% for Al10wt%Zn at

25wt.% $CaCO_3$.In Figure 4the maximum tensile strain of homogenized composite with percentage weight of $CaCO_3$addition is shown. For Al5wt%Zn based composite, increase in percentage $CaCO_3$ addition is accompanied with a rise in the maximum tensile strain to 5.41% at.5wt% of $CaCO_3$ but declines to 4.04% at 10wt% of $CaCO_3$ before it rose again to 6.17% at 25wt.% of $CaCO_3$. The maximum tensile strain for Al10wt%Zn composites decreased with increase in percent $CaCO_3$ until 10wt.% $CaCO_3$ where an increase in the maximum tensile strain of 5.93% at15 wt.% $CaCO_3$ occurred. On further additions, there is a drop in the maximum tensile strain.

3.3. Ductility

The maximum tensile strain behaviours with percent weight of $CaCO_3$, of the aluminium alloy are shown in Figure 5. For Al5wt%Zn composite, increase in percentage $CaCO_3$ addition is accompanied with decrease in the maximum tensile strain to 3.6% at 5wt.% of $CaCO_3$ and remains fairly constant within that range. The maximum tensile strain for Al10wt%Zn composite initially remained steady with increase in percentage $CaCO_3$ until at 10wt.% $CaCO_3$ where a rapid increase in the maximum tensile strain to 40%occurs. With further filler additions, a drop in the maximum tensile strain is observed. The maximum tensile strains in relation to percent weight of $CaCO_3$in homogenized aluminium alloy are shown in Figure 6. For Al5wt%Zn composite, increase in percentage $CaCO_3$ addition is followed with rise in the maximum tensile strain to 5.52% at 5wt.% of $CaCO_3$ but falls to 4.17% at 10wt.% of $CaCO_3$ before it rose again to a peak of 6.45% at25wt% of $CaCO_3$. The maximum tensile strain for Al10wt%Zn composite declined with increase in percent $CaCO_3$ until 10wt.% $CaCO_3$ where an increase in maximum tensile strain to 6.25% at wt.15% $CaCO_3$ is attained. But on further filler additions, there is a drop in the maximum tensile strain.

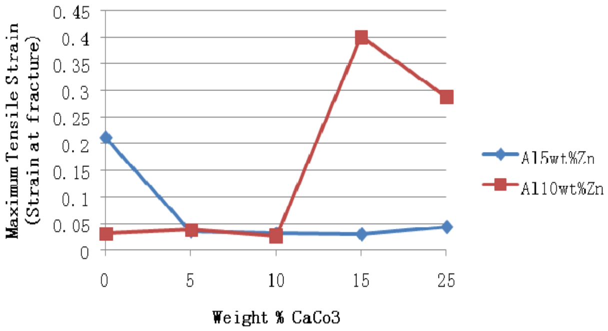

Figure 5: Maximum tensile strainsfor zinc modified aluminum-0.5%Si / $CaCo_3$ composite in as-cast state

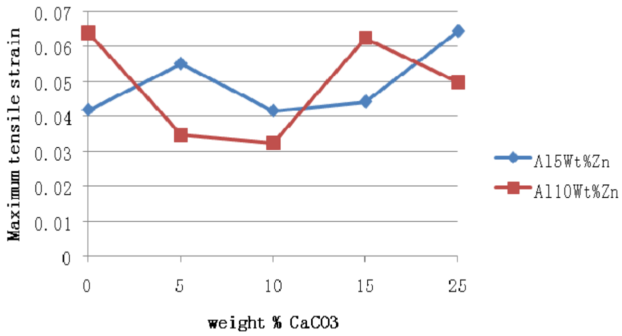

Figure 6: Maximum tensile strains for zinc modified homogenized aluminum-0.5%Si / $CaCo_3$ composite

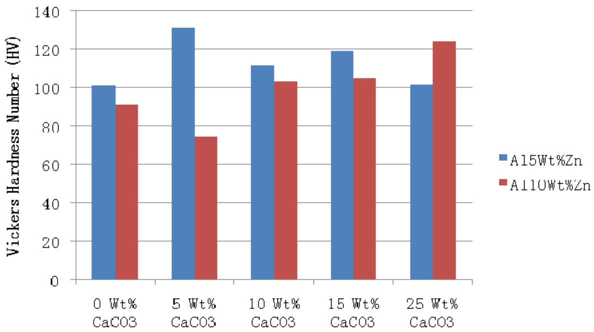

Figure 7: Hardness results (HV) of zinc modified aluminum-0.5%Si / $CaCo_3$ composite in as-cast state

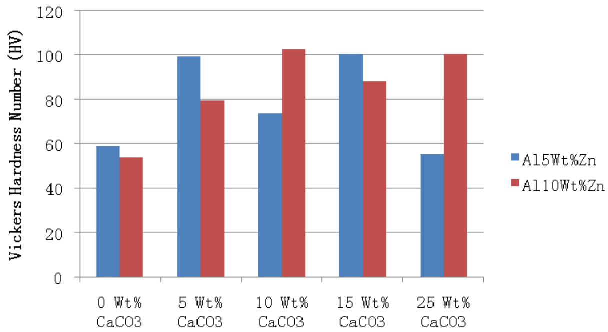

Figure 8: Hardness results (HV) of zinc modified homogenized aluminum-0.5%Si / $CaCo_3$ composite

3.4. Hardness

The hardness characteristics of Al-$CaCO_3$ composites are shown in Figure 7 in as-cast condition. Hardness values for Al5Wt%Zn tend to increase as the %wt. fraction of $CaCO_3$ increases to a maximum of 131.1Hv at 5wt%$CaCO_3$ and declines with further increase in $CaCO_3$ content. Contrary to this, Al10wt % Zn experiences increase in hardness as $CaCO_3$ content increases except for an initial decline at 5wt% filler. The highest value (123.9Hv) is seen in samples with 25wt % $CaCO_3$.Homogenization of the composites (see Table 2) promotes decrease in hardness with filler additions. All samples show decline hardness after homogeniezation. Al7wt % Zn with 10wt. % $CaCO_3$ possess the highest hardness value followed by that with 25wt.%$CaCO_3$. In most cases Al5wt % Zn samples show superior hardness values as increase in zinc content may have promote decrease in hardness responses.

4. MICROSTRUCTURAL ANALYSIS

Microstructures of cast Al-Zn-$CaCO_3$ composites show the presence of three phases namely AlFeSi (light brown), Mg_2Si (dark) and $CaAl_2$ (grey) in α-aluminium matrix. Due to lack of X-ray diffractometer to identify these phases a standard method of etching was used so that the colour of the precipitates are used to identify the phases in comparison with standards (Brandes, 1998). As-cast sample of Al5wt%Zn alloy (see Plate 1a) shows clusters of AlFeSi precipitates at the grain boundaries with few Mg_2Si crystals littering the α-Al matrix surface. As-cast Al5Wt%Zn/5 wt. % $CaCO_3$composite sample (see Plate 1b) shows discontinuous coarse crystals of AlFeSi and Mg_2Si in the matrix with grain structure appearing smaller in size than Al5wt%Zn alloy matrix structure but with increase in volume fraction of precipitates. There is a decline in grain boundary clustering. The volume fraction of AlFeSi tends to remain the same while the amount of $CaAl_2$ increase.

Table 2: Comparative Vicker's hardness number for all samples

Sample	Wt.% $CaCO_3$	HV (As-cast)	HV (Homogeniezed)
Al5Wt%Zn	0	100.9	58.8
	5	131.1	99.4
	10	111.5	73.8
	15	119.2	100.4
	25	101.6	55.4
Al7Wt%Zn	0	91.3	54.1
	5	74.4	79.5
	10	103.4	102.6
	15	104.9	88.4
	25	123.9	100.6

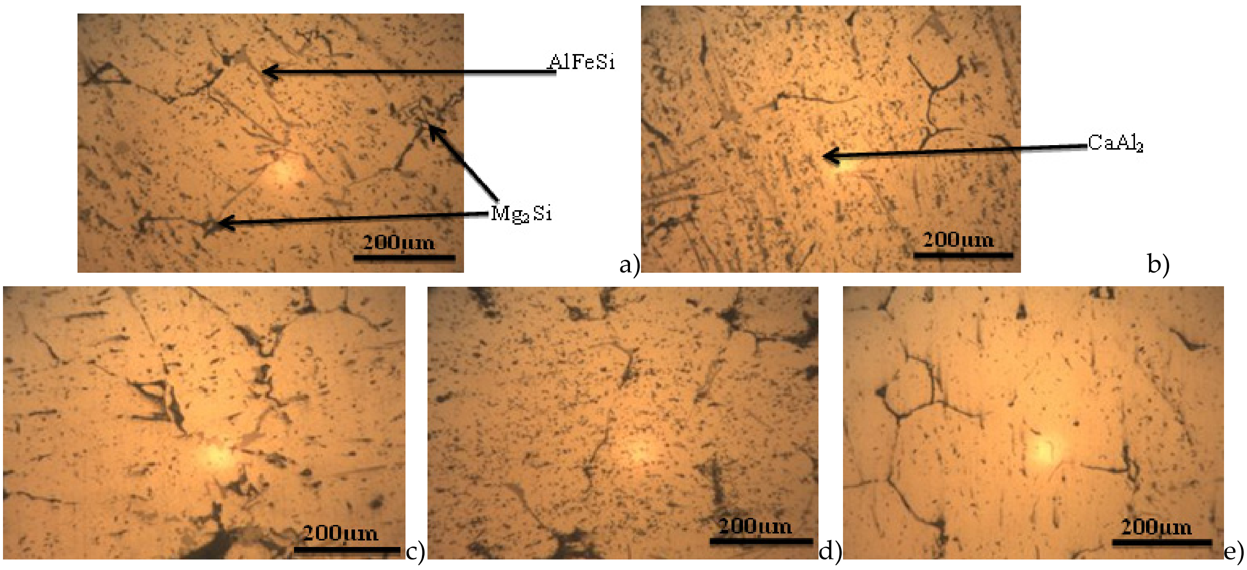

Plate 1: Microstructures of as-cast Al5Wt%Zn
(a) without $CaCO_3$ (b) 5 wt. % (c) 10 wt. % (d) 15 wt. % (e) 25 wt. % $CaCO_3$

For Al5Wt%Zn with 10 wt. % $CaCO_3$in as-cast state (see Plate 1c), the structure shows Mg_2SiPrecipitates along the grain boundaries and AlFeSi is also present in the matrix. AlFeSi and $CaAl_2$ precipitates are found dissolved in the α-Al matrix leaving Mg_2Si in the grain surface. Blow holes are also seen on the surfaces. Large cluster of AlFeSi is present in the grain boundaries with random distribution of AlFeSi in the as-cast Al5Wt%Zn/15 wt. % $CaCO_3$(see Plate 1d). There is distribution of AlFeSi along the grain boundaries inas-castAl5Wt%Zn/25 wt. % $CaCO_3$composite (see Plate 1e) with Mg_2Si sparsely distributed in the matrix.

Effects of increase in zinc content on the microstructures of the as-cast composites are shown in plate 2. Mg_2Si and $CaAl_2$ are randomly distributed around the matrix in Al10Wt%Zn alloy (see Plate 2a) and AlFeSi crystals are scattered over the matrix. Al10Wt%Zn / 5 wt.% $CaCO_3$(see Plate 2b) shows Mg_2Si along the grain boundaries while crystals of AlFeSi and $CaAl_2$are scattered in the matrix. In Al10Wt%Zn / 10 wt.%$CaCO_3$(see Plate 2c), a loss arrangement of crystals is shown with

Mg_2Si still distributed along the grain boundaries while Al10Wt%Zn /15 wt.% $CaCO_3$ sample (see Plate 2d) shows a cluster of Mg_2Siprecipitates present along the grain boundaries with distributions of AlFeSi in the matrix. Mg_2Siprecipitates clustered at the grain boundaries with AlFeSi and $CaAl_2$ precipitates dispersed all over the α-Al matrix inAl10Wt%Zn / 25 wt.%$CaCO_3$ sample (see Plate 2e).

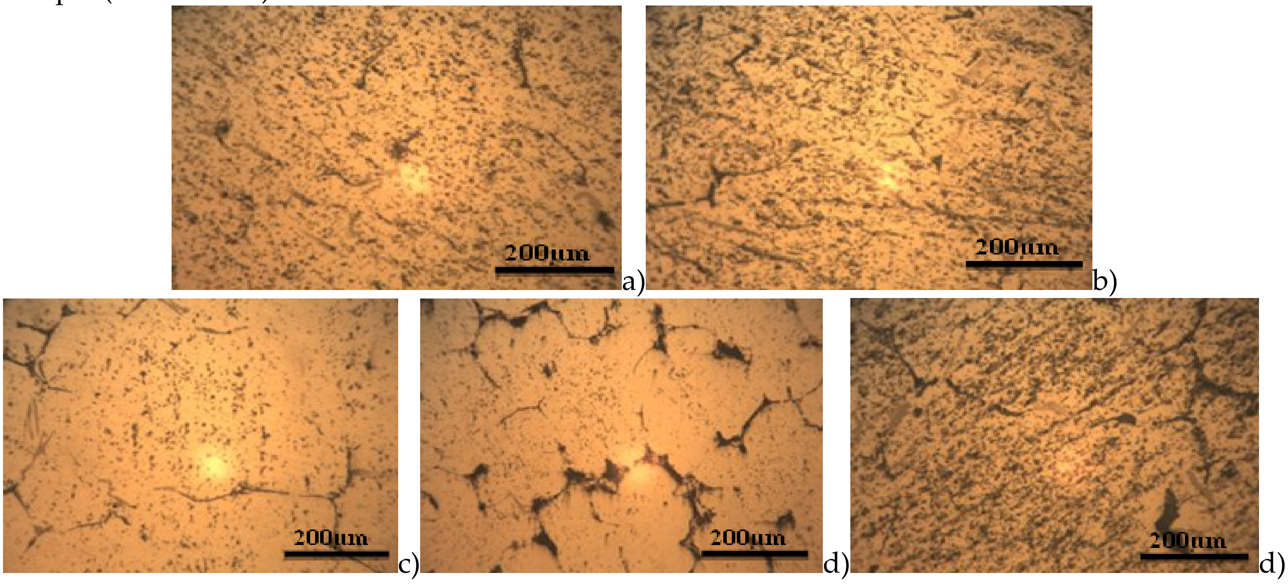

Plate 2: Microstructures of as cast Al10Wt%Zn
(a) without $CaCO_3$ (b) 5 wt. % (c) 10 wt. % (d) 15 wt. % (e) 25 wt. % $CaCO_3$

In Plate 3 the effect of homogenization on the structural features of Al-$CaCO_3$ composites is shown. Al5wt%Zn homogenized sample (see Plate 3a) shows a distribution of Mg_2Si along the grain boundaries with scattered AlFeSi precipitates present in the matrix. Plate3 b and c show the microstructure of5wt. % and 10 wt.% $CaCO_3$ modified composites with Mg_2Siprecipitates evenly distributed in the grain boundaries and around the matrix and crystals of AlFeSi scattered all over the surface. Al5Wt%Zn/15wt.% $CaCO_3$(see Plate 3d) homogenized sample exhibits a sparsely distributed AlFeSi crystals within the matrix with Mg_2Siprecipitates clustered along the grain boundaries. ForAl5Wt%Zn /25wt.% $CaCO_3$ sample (see Plate 3e), Mg_2Sicrystals littered the α-Al matrix surface with AlFeSi present in the matrix.

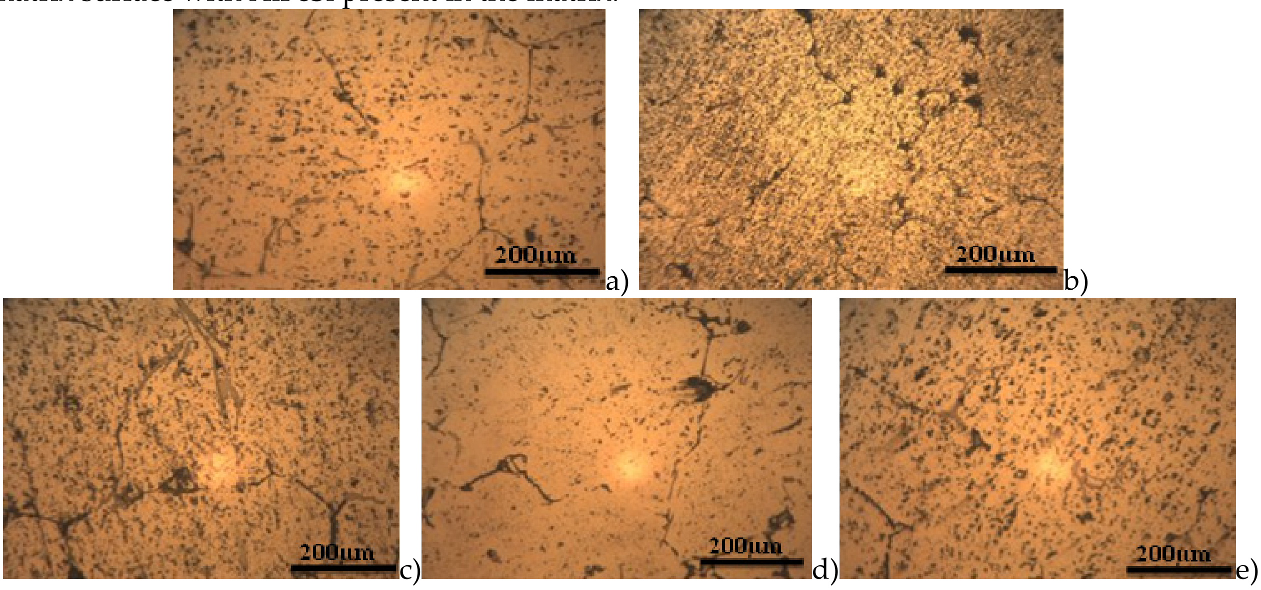

Plate 3: Microstructures of homogenizedAl5Wt%Zn
(a) without $CaCO_3$ (b) 5 wt. % (c) 10 wt. % (d) 15 wt. % (e) 25 wt. % $CaCO_3$

Homogenized Al10Wt%Zn alloy (see Plate 4a) shows an increase in AlFeSi and Mg_2Si precipitates distribution in the matrix while 5wt.% $CaCO_3$modified composite (see Plate 4b)shows a loss structure in the precipitates distribution. Furthermore, homogenized10wt.% $CaCO_3$modified composite (see Plate 4c) matrix indicates the presence of Mg_2Siprecipitates clustered at the grain boundaries with AlFeSi crystals litters the matrix surface. With Mg_2Si crystals in the grain boundaries of homogenized 15wt.% $CaCO_3$ composite (see Plate 4d), there occur random arrangement of AlFeSi Phase in the matrix. In 25wt.% $CaCO_3$ modified composite(see Plate 4e), AlFeSi phase is poorly distributed with Mg_2Sifound along the grain boundaries.

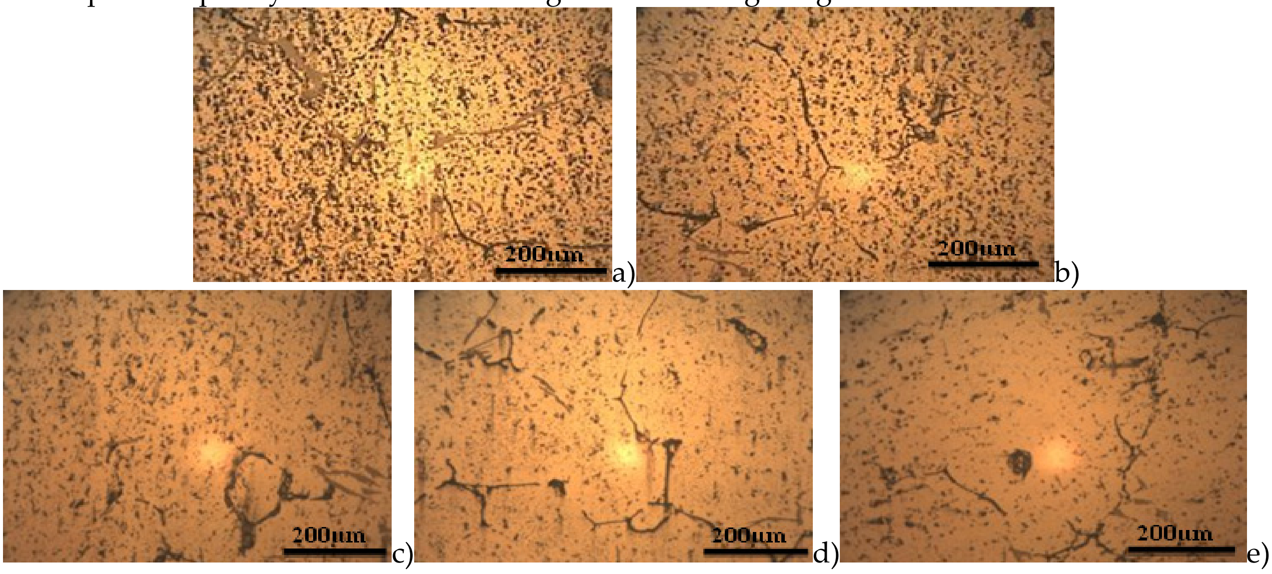

Plate 4: Microstructure of homogenizedAl10Wt%Zn
(a) without $CaCO_3$ (b) 5 wt. % (c) 10 wt. % (d) 15 wt. % (e) 25 wt. % $CaCO_3$

5. DISCUSSION

The addition of calcium carbonate powder, zinc and magnesium to wrought aluminium alloy show marked effect on the tensile strength, ductility, and hardness properties of the alloy. The UTS result of these processed samples show significant improvement over the conventional 6063 aluminium alloy. This can be attributed to combined effect of grain refinement propelled by the presence of zinc, magnesium and calcium content and the formation, precipitation, and distribution of hard intermetallic compounds (such as AlFeSi, Mg_2Si and $CaAl_2$) at matrix surface [2, 12, 19-21]. Samples containing 25wt%$CaCO_3$ and 5wt%Zn are found to exhibit the highest tensile strengths compared to all other samples in the as-cast condition. Microstructures of these samples (see Plate 1b and e) show that intermetallics of $CaAl_2$ and AlFeSi dissolved in the matrix with a breakdown of the grain structure. This is different from sample Al10wt%Zn/ 25 wt.%$CaCO_3$, which contain the same amount of $CaCO_3$ but 10wt%Zn. Microstructure of this sample (see Plate 2e) shows that formation of $CaAl_2$ and Mg_2Si intermetallics which are responsible for strength of the cast material are retarded by the increase in zinc content resulting in reduce strength compared to Al5Wt%Zn/ 25 wt.%$CaCO_3$. This phenomenon also occurs in the tensile elongation responses.Al5Wt%Zn/25 wt.%$CaCO_3$ (0.0436) sample has superior tensile elongation to Al10Wt%Zn/25 wt.%$CaCO_3$ (0.02587), which is probably due to the difference in the volume fractions of Mg_2Si and AlFeSi phases which has been noted by several authors as the precipitates that influences tensile elongation to a large extent. Sasaki et al. [22] and Zajac et al. [23] earlier explained that the small volume fraction of AlFeSi crystals support elongation. These samples also possess appreciable hardness values which may be attributed to the presence of hard precipitates (such as Mg_2Si) in the microstructure. However, Al5Wt%Zn/5 wt.%$CaCO_3$ possess the highest hardness value (131.1) this is evident from the micrograph which shows a higher volume fraction of precipitates in the grains. Moreover, these precipitates clusters in the matrix surface with few clustered around the grain boundaries. This distribution of precipitates may be the reason for the high strength value (115.7MPa). Solution treatment of these samples lead to a breakdown of the cast dendritic structure with a transformation of precipitates of AlFeSi and Mg_2Si with increase in size and reduction in volume fractions promoting decline in strength with corresponding increase in tensile elongation.

6. CONCLUSION

From the study the following deductions can be drawn:

- ✓ Increase of $CaCO_3$ has a marked influence on the mechanical properties of wrought aluminium alloy. Tensile strength is dependent on the size, volume fraction and distribution of AlFeSi and $CaAl_2$intermetallics in the α-Al matrix.
- ✓ Intermetallics of $CaAl_2$, AlFeSi and Mg_2Si are formed and precipitated in the α-Al matrix.
- ✓ Solution treatment of these alloys lead to remarkable change in their mechanical properties. This may be due to increase precipitation. Tensile elongation is improved by solution treatment but lower than that of the as-cast samples. Four hours holding time may not have been sufficient for precipitation and transformation of intermetallics.
- ✓ At higher amount of Zinc increase in $CaCO_3$caused decrease in tensile strength with an increase in hardness.
- ✓ The presence of AlFeSi and $CaAl_2$ are detrimental to tensile elongation.

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