

Effect of Refractory Oxide (CaO) on the Structure and Mechanical Properties of Al-4%Cu Alloy

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ABSTRACT

Aluminium-4%wt copper alloy being a 2xxx series alloy, exhibits moderate strength as a result of needle-like structure of the intermetallic compound ($Al_2Cu(\theta)$) formed as non-coherent precipitates as the alloy cools slowly to ambient temperature. Hence, this paper focuses on addition of a refractory oxide (CaO) to melted Al-4%wtCu alloy, in order to refine the grain size and structure, thereby improving the mechanical properties of the alloy. The objective of this work is to study the extent to which each concentration of CaO influenced the mechanical properties of Al-4%Cu alloy and the concentrations that gave an appreciable mechanical property suitable for engineering application. The experiment was carried out with refractory oxide (CaO) with concentration of 0.75%, 1%, 1.25%, 1.5%, 1.75%, 2% and 2.25% by weight to melted Al-4%Cu alloy, stirred and sand cast. The results established that the refractory oxide (CaO) can be used to improve the microstructure and mechanical properties of Al-4%Cu alloy.

Keywords: Refractory Oxide, Structure, Mechanical Properties and Al-4%Cu Alloy

I. INTRODUCTION

Aluminium alloys have been utilized for more than a century. The combination of strength, low density, formability, corrosion resistance, conductivity and abundance nature makes the material ideal for a number of applications (Nnuka, 2002). The alloys have high strength-to-weight ratio and durability which makes them useful for foil and conductor cables (Kissel and Ferry, 1995). Both the automotive and aerospace industries benefit greatly from the high strength-to-weight ratio of aluminium alloys (Wang et al, 2004). The heterogeneous nature of aluminium alloys is most evident in members of the high strength alloys of the 2xxx, 6xxx, 7xxx and 8xxx and most particularly the 2xxx series alloys where alloy additions are required to obtain the high strength to weight ratio properties of these materials (Birbilis and Bucheit, 2005; Hatch, 1984; Castillo and Lavernia, 2000).

Besides, aluminum on its own is a great thermal conductor, making it highly applicable for heat transfer systems, including heat exchangers and cooling ribbons.

It is the third most abundant element, it forms some 80% of the earth's crust, exist in the form of aluminosilicate or aluminum oxide called bauxite as a result of its high affinity for oxygen and silicates. Aluminum is ductile and malleable due to its polycrystalline structure (Kissel and Ferry, 1995). According to Cochran and Maphother (1958), it is capable of being a superconductor with a superconducting critical temperature of 1.2 Kelvin and a critical magnetic field of about 100 gauss (10 milliteslas). Owing to its resistance to corrosion, aluminum is one of the few metals that retain silvery reflectance in finely powdered form, making it an important component of silver coloured paints (Kissel and Ferry, 1995).

Al-4%Cu alloy is distributed in form of individual isolated inclusions between the dendrite cell and grain boundary (Nnuka, 1991). The increase in hardness and tensile strength is due to the interaction of the stress field around the particles with a moving dislocation and also due to physical obstruction by the hard particles to the moving dislocation (Nnuka, 2002). The extent to which strengthening is produced depends on the amount of the second phase particles, the characteristics and properties

of the second phase, the particle size, shape and distribution (Nnuka, 1991).

Aluminium-4%wt copper alloy being a 2xxx series alloy, exhibits moderate strength as a result of needle-like structure of the intermetallic compound ($Al_2Cu(\theta)$) formed as non-coherent precipitates as the alloy cools slowly to ambient temperature (Chester and Polmear, 1983; Nnuka, 1991; Bourgeois et al, 2011). Hence, this paper focuses on addition of a refractory oxide (CaO) to melted Al-4%wtCu alloy, in order to refine the grain size and structure, thereby improving the mechanical properties of the alloy. To produce Al-4%wtCu alloy with improved mechanical properties, the concentrations of lime (CaO) was varied in the order of 0.75, 1.0, 1.25, 1.5, 1.75, 2.0 and 2.25%wt, in Al-4%Cu alloy.

The extent to which each concentration of CaO influenced the mechanical properties of Al-4%Cu alloy was studied and the concentrations that gave an appreciable mechanical properties suitable for engineering application was established.

II. METHODS AND MATERIAL

1.1 Literature review

Micro-addition is a technique that influences the mechanical properties and the micro-structure of Al-Cu alloy. In this technique, the alloying additives go into either substitutional or interstitial solid-solution, and distort the lattice structure and offer resistance to dislocation movement. This resistance is greater with interstitial element. Micro addition is meant basically to improve the mechanical properties such as strength, hardness, ductility and toughness. It is sometimes used to improve the fluidity and other casting properties (Higgins, 1986). The high strength and hardness or reliability of aluminium-4%wt copper alloy is dependent on the percentage of copper in solution (α -solid solution of copper in aluminium matrix) and on the form, size, number and the distribution pattern of the intermetallic compound. Micro additives can enhance critical properties in aluminium-4%Cu alloys.

In Al-4%Cu alloy system, the alloying elements or impurities go into solid solution in the matrix and form intermetallic compound during the solidification of the metal. Micro alloying additions alter the properties of

Al-4%Cu alloys by changing the morphology, chemistry, structure, spartial distribution and size of precipitate (Nnuka, 1991). Micro alloying is considered to affect the formation kinetics of nano-clusters directly (Salimon et al, 2001). Micro alloying additions induce formation of second phase particles.

The effect of micro additive on the quantity and distribution pattern of the secondary phase in aluminum-copper alloy system was studied by Nnuka (1991). It was revealed that the solubility of copper in aluminum matrix increased with increasing temperature and the maximum solubility of Cu in aluminum matrix was at the eutectic temperature ($540^\circ C$), with maximum copper concentration of 5.7wt%. It was also observed that at low concentration of copper, α -solid solution was formed (dissolution of copper in the aluminum matrix), and at higher concentrations of copper, the secondary phase Al_2Cu was formed.

Chester and Polmear (1983) disclosed that the solidification sequence is influenced to a greater extent by the interaction of the alloying element. The presence of a high amount of alloying elements in Al-4%Cu alloy system complicates the solidification process, and there is a need to understand the role of each alloying addition and how it affects the microstructure, and hence the properties and the interrelationship between these additions to achieve improved properties.

Nnuka (1994) proposed a study to evaluate the effect of varying the composition of the major alloying additions (refractory oxides) within the specified limit on the microstructure, and distribution of phases in Al-4%Cu alloys. The alloying additions investigated are calcium oxide, magnesium oxide, silicon dioxide and chromium trioxide. The presence of second phase particles often causes lattice distortions which result when the precipitate particles differ in size and crystallographic structure from the host atoms.

1.2 Paper outline

The rest of this paper is organized as follows. Section 2 gives an overview of the method used for the experiment, Sections 3 describe experimental procedure, section 4 describe mechanical test while 5 discusses micro-structural examination. Section 6 and 7 discusses the result; section 8 discusses Micrographs and Quantitative

Microstructure Analysis of Studied Specimens. Section 9 concludes the work.

2.0 Methods

The refractory oxide (CaO) was added in concentrations of 0.75%, 1%, 1.25%, 1.5%, 1.75%, 2% and 2.25% by weight to melted Al-4%Cu alloy, stirred and sand cast. Subsequently, specimens obtained from the casting were subjected to machining and mechanical properties such as ultimate tensile strength, hardness, yield strength, ductility and impact strength were determined for each specimen using Mansanto tensometer, Rockwel hardness tester and universal impact testing machine, respectively. The microstructure of the samples was also studied using metallurgical microscope with photographs taken.

3.0 Experimental Procedure

Casting processes:

- 3.1 Charge preparation: The materials to be charged into the furnace were prepared according to the following processes : 357g of aluminium scrap taken as 96wt% and 14.892g of copper scrap taken as 4wt% were weighed using a portable electronic weighing balance. Similarly, for 0.75wt%, 1wt%, 1.25wt%, 1.5wt%, 1.75wt%, 2wt% and 2.25wt% of (CaO), the equivalent in grams weighed, were 2.74g, 3.65g, 4.56g, 5.48g, 6.39g, 7.30g and 8.21g respectively.
- 3.2 Mould preparation: The mould was wooden comprising of a cope and drag and the sand used was green sand, mixed with bentonite as the binder. With the aid of a cylindrical pattern, an impression was made on the sand which served as a cavity to contain the hot molten alloy to be poured for solidification. The sand was rammed, and the runners and the risers were equally made as well as vent. Eventually, the pattern was removed along with the wood used for making the risers and the mould preparation was completed.
- 3.3 Melting and casting: The weighed scraps and oxide compositions were melted in a crucible furnace in the following order : 357g of aluminium was first poured into a metallic crucible (cup), immersed in the furnace and heated in order to melt at 670°C. The melted aluminium was stirred during which 14.892g of copper was added and the copper melted

at 1083°C. Digital multimeter was used for measuring the temperature during casting. Finally, 2.74g of CaO was added and further stirred for homogeneity of the mixture. As homogeneity was achieved, the cup was removed from the furnace and poured into the cylindrical cavity of a prepared green sand mould and was left there for 3 to 5 mins, for solidification to take place. Similarly, the casting process was repeated for 3.65g, 4.56g, 5.48g, 6.39g, 7.30g and 8.21g of CaO. A control sample, comprising of only 357g of aluminium scrap and 14.892g of copper scrap was equally casted. Eventually, each casting was recovered from the mould after solidification, and was allowed to cool in air.

- 3.4 Machining: The machining operation was carried out using a three jaw chuk lathe machine. The samples to be machined were firmly clamped on the machine and facing, turning and shaping operation was done on the clamped samples with the aid of a cutting tool, mounted on the tool post of the lathe machine. Eventually, the required dimensions for impact, tensile and hardness tests as well as microstructural analysis, were obtained.

4.0 Mechanical Tests

- 4.1 Tensile Test : Standard tensile test specimens, having an original gauge length of 28mm and diameter of 5mm were used for this test. The tensile test was therefore conducted using a horizontal bench - top Mansanto tensometer and the test was carried out at room temperature. The specimens (three samples on each composition) were tested to determine their ultimate tensile strength, yield strength and ductility (%EL). These properties were determined with the aid of an automatic plotted Force (N)-Extension (mm) curve and the tensile specimens, and the average results were as tabulated in table 4.1.
- 4.2 Hardness test: Hardness of the specimens was determined using a Rockwell hardness testing machine. The hardness test was conducted using TESTOR HT 1 Otto Wolpert-Werke Rockwel hardness tester, with a 15kg indentation - 100kg scratching scale. Three readings were taken for each specimen at different locations to circumvent the possible effect of any alloying element segregation and the average value was recorded. With the aid of a standard conversion table as attached in appendix

1, the Rockwell hardness test results were converted to brinell hardness test values which were tabulated as shown in Table 1.

4.3 Impact test : Impact test was carried out with a charpy impact testing machine and the test was done at standard organization of Nigeria (SON) using Samuel Denison - LS10 2DE impact testing machine. The impact test samples were machined to a dimension of (10 X 10 X 55)mm with a v-notch of depth 2.5mm at its midpoint. The samples to be tested were placed at the machine's sample post with the notch facing the hammer and the hammer was raised to an angle of 45° and released to swing through the positioned sample in order to break it. As the samples were broken by the swung hammer, the impact energy absorbed was read from the charpy impact energy scale, calibrated in Joules. Therefore, the impact energy of all the samples, as well as the control sample were captured and tabulated in Table 4.1.

5.0 Micro-structural examination:

The following steps were taken for micro-structural examination of the as-cast specimens:

- 5.1 Cutting – The specimens were cut to mounting size with the aid of a hack saw.
- 5.2 Mounting - The cut specimens were mounted on a thermosetting material.
- 5.3 Grinding- The mounted specimens were ground using P220C, P320C, P600C, and P800C grades of water-proof Silicon carbide grinding paper (sequenced from coarse to fine).
- 5.4 Polishing- The ground specimens were polished using ECOMET II polisher, after pouring diamond paste on the rotating polishing surface of the ECOMET II polishing machine. This was done to remove the fine surface scratches on the specimens in order to have mirror-like finish.
- 5.5 Etching – The polished specimens were etched with Keller's reagent to expose the specimens' surface for micro-structural examination. It was further rinsed in water and dried with METASER specimen dryer.
- 5.6 Micro-structural examination - The etched and dried specimens were finally subjected to micro-structural examination using an optical microscope, after which the micrographs of (×100) magnification were obtained with the aid of a digital camera linked to a computer, for a visual display of the snapshots.

III. RESULTS AND DISCUSSION

Table 1: Results on the effect of dopant (CaO) of 0.75% wt - 2.25%wt concentrations on mechanical properties of Al-4%Cu alloy.

ALLOY COMPOSITION	UTS (Mpa)	YS (Mpa)	HB (Kg/mm ²)	IMPACT (CHARPY) (J)	EL (%)
Al-4%Cu	170	140	80	6	10.0
Al-4%Cu-0.75%CaO	201	134	89	6	6.7
Al-4%Cu-1.00%CaO	218	154	85	8	8.4
Al-4%Cu-1.25%CaO	254	166	84	18	11.0
Al-4%Cu-1.50%CaO	297	83	12	12.0	
Al-4%Cu-1.75%CaO	310	195	80	10	12.8
Al-4%Cu-2.00%CaO	330	210	76	8	13.8
Al-4%Cu-2.25%CaO	345	219	72	6	14.7

Note: EL – Elongation, HB- Brinell hardness, UTS – Ultimate tensile strength and YS – Yield strength.

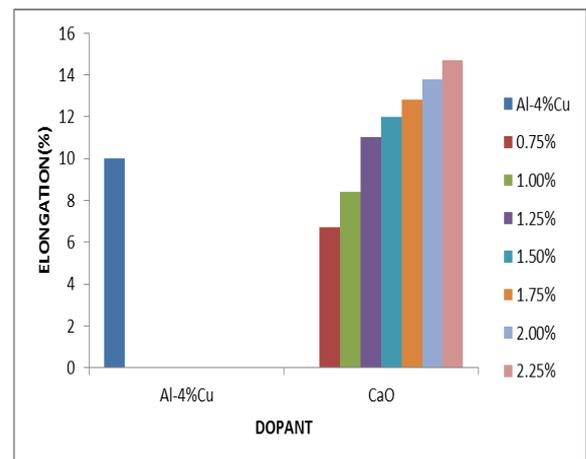


Figure 1: The effect of Dopant on percentage elongation of Al-4%Cu alloy.

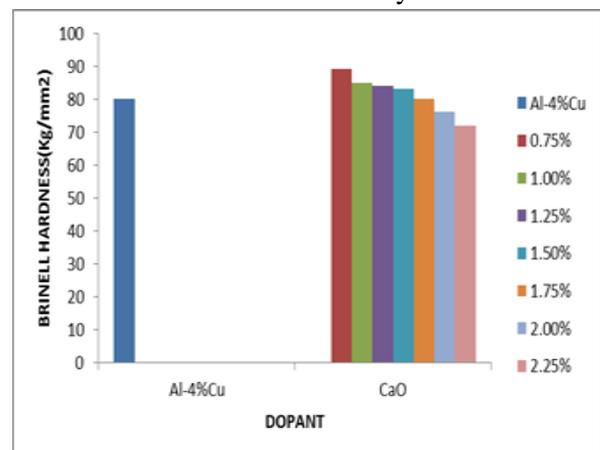


Figure 2: The effect of Dopant on Hardness of Al-4%Cu alloy.

From figure 1 it was observed that the percentage elongation of the control specimen was lower than that produced when it was doped with some of the concentrations of the dopant (CaO). It was also noted that the percentage elongation increased along with increased concentration of the dopant (CaO) beyond 1%wt. It was equally noted that CaO with 1.25% wt to 2.25% wt greatly improved the ductility of Al-4%Cu alloy.

Figure 2 shows the effect of dopant (CaO) on the hardness of Al-4%wtCu. It was observed that the hardness value of Al-4%wtCu alloy improved reasonably when the alloy was doped with 0.75%wt-1.5%wtCaO. Hardness was seen to decrease with increased in concentration of CaO beyond 1.5% wt.

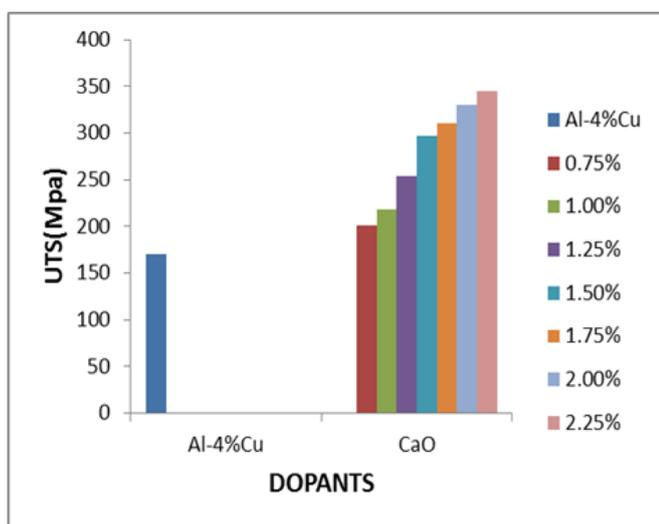


Figure 3: The effect of Dopant on Ultimate Tensile Strength (UTS) of Al-4%Cu alloy

Figure 3 shows the effect of dopant (CaO) on the ultimate tensile strength of Al-4%Cu alloy. From figure 3, addition of the dopant (CaO) improved the ultimate tensile strength of Al-4%Cu alloy. The improved ultimate tensile strength was noticed as the concentration of CaO increased from 0.75% wt – 2.25% wt CaO. This was generally because CaO forms dissolve solid particle which opposed the dislocation motion in the Al-4%Cu alloy matrix.

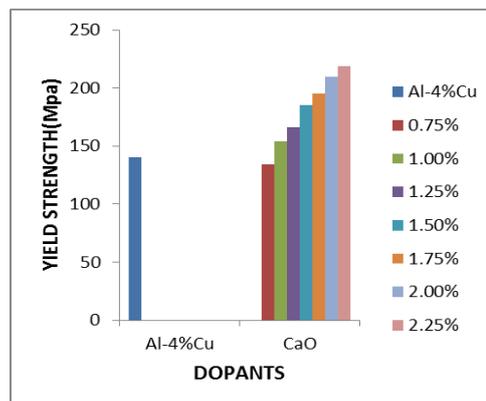


Figure 4: The effect of Dopant on yield strength of Al-4%Cu alloy.

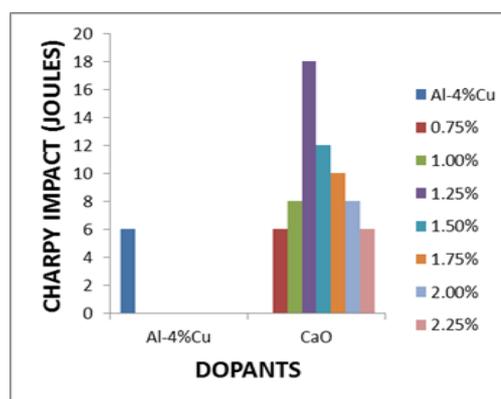


Figure 5: The effect of Dopants on Impact Energy of Al-4%Cu alloy

Figure 4 shows the effect of dopant (CaO) on yield strength of Al-4%Cu alloy. The introduction of calcium oxide (CaO) with increasing concentration beyond 0.75%wt improved the yield strength of the Al-4%Cu alloy. It was noticed that CaO dopant from 1% wt – 2.2% wt concentration, offered Al-4%Cu alloy the best yield strength.

Figure 5 shows the effect of the dopants on energy absorbed (impact energy) by Al-4%wtCu alloy. It was observed that CaO with 1% - 2% concentrations improved the impact strength of Al-4%wtCu alloy. CaO with 0.75% and 2.25% concentrations were observed to share similar impact energy with the control sample. It was observed that the composition of CaO that offered Al-4%Cu alloy the best impact strength was 1.25% wtCaO.

Plates 1-8 represent the micrographs of Al-4%Cu alloy and Al-4%Cu alloy doped with calcium oxide of 0.75% wt -2.25% wt concentrations.

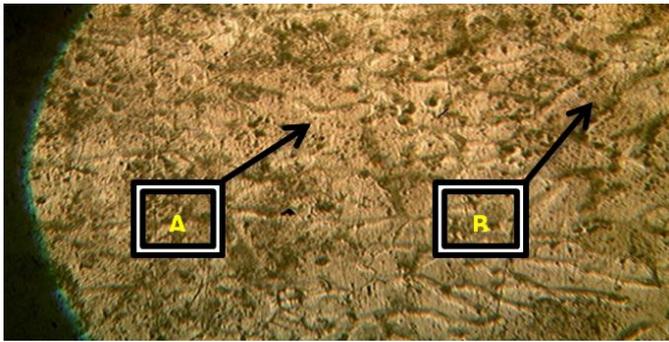


Plate 1: Al - 4%wtCu (X100)

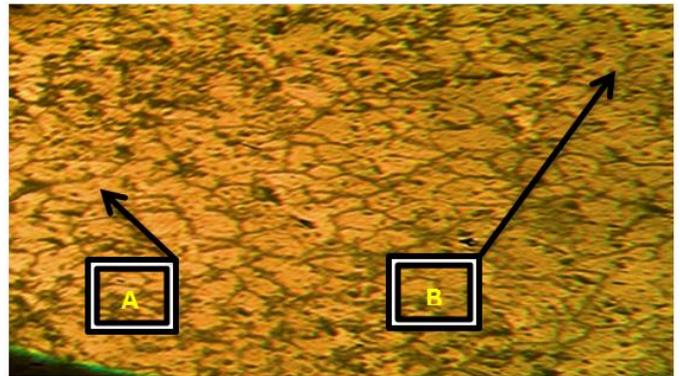


Plate 7: Al-4%Cu – 2%wtCaO (X100)

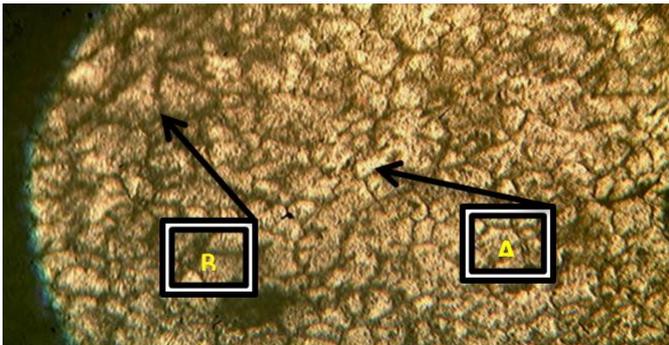


Plate 2: Al-4%Cu – 0.75%wtCaO (X100)

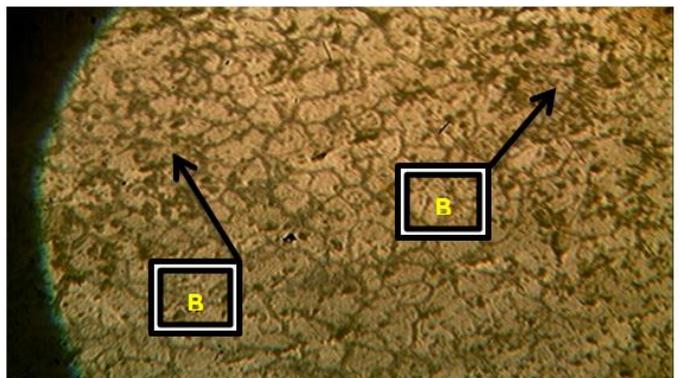


Plate 8: Al-4%Cu – 2.25%wtCaO (X100)

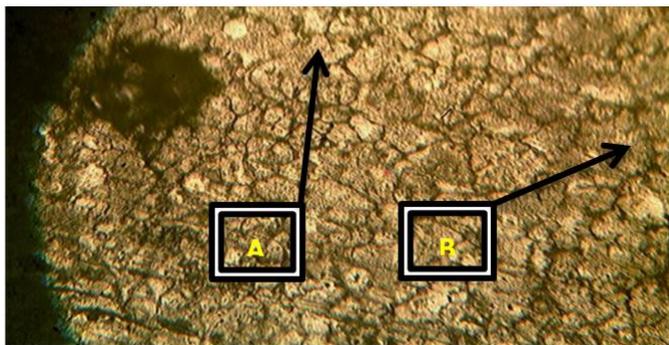


Plate 5: Al-4%Cu – 1.5%wtCaO (X100)

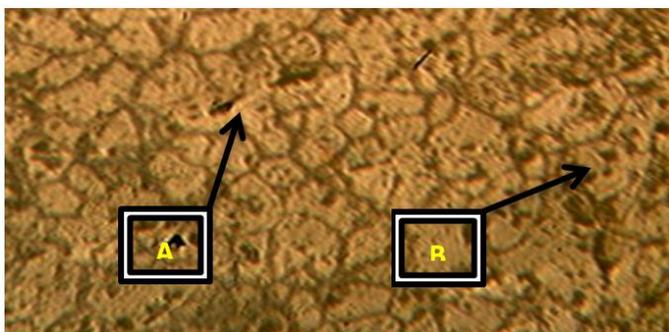


Plate 6: Al-4%Cu – 1.75%wtCaO (X100)

Where: **A** – α -solid solution, **B** – intermetallic compound.

The micrograph of the control specimen (Al-4% wtCu) shown in plate 1, reveals that the microstructure of the specimen comprised of the eutectic α -solid solution (the region where copper formed a solid solution with the aluminium matrix) and the intermetallic compound (Al_2Cu) precipitates. The intermetallic compound existed in the form of coarse needle-like precipitate from the α -solid solution by the grain boundaries.

Plates 2-8 show the micrographs of Al-4%wtCu doped with calcium oxide of concentrations 0.75% wtCaO to 2.25% wtCaO. The plates show that there was an observable increase in intermetallic compounds from the grain boundaries, as the concentration of calcium oxide increased. This revealed the possibility of lattice distortion in the alloy system. Hence, a stress field was set up in the alloy matrix and it interacted with the stress field caused by the impeded dislocation, thereby giving rise to an increase in ultimate tensile strength and a decrease in hardness beyond 1.5%wt, as the concentration of CaO increased. The plates equally revealed relatively large grains in the matrix, which

explains why ductility was improved beyond 1% wtCaO concentration.

IV. CONCLUSION

The results showed that the addition of the refractory oxide at certain concentrations, improved the mechanical properties of Al-4%Cu alloy. The mechanical properties were improved because the refractory oxide (CaO) could not melt along with aluminium and copper, but remained as dissolved solid particles in the alloy (Al-4%Cu) and serves as nuclei on solidification, thereby refining the grains, modifying the microstructure of the alloy and also restricting the motion of dislocation in the alloy.

V. REFERENCES

- [1] Birbilis N., and Bucheit R. G.(2005) Electrochemical characteristics of intermetallic phases in aluminium alloys, an experimental survey and discussion, *Journal of the Electrochemical Society*,152(4):B140-B151.
- [2] Bourgeois L., Dwyer C. M., Weyland J. F., Nie and Muddle B. C.(2011)Structure and energetics of the coherent interface between θ' precipitate phase and aluminium in Al-Cu *Acta Mater.*, 59:7043-7050.
- [3] Castillo L. D., and Lavernia E.J.(2000) Microstructure and mechanical behaviour of spray deposited Al-Cu-Mg(Al-Mn) alloys, *Metallurgical and Materials Transactions A*, Vol.31A, pp.2287.
- [4] Chester R. J., and Polmear I. J. (1983) *Light Alloys: Metallurgy of the Light Metals* (3rd ed.), Institute of Metals, London, pp75-81
- [5] Cochran J.F., and Mapother .D.E. (1958) Superconducting transition in aluminium, *Physical Review*, 111(1):132-142.
- [6] Hatch J. (1984) *Aluminium Properties and Physical Metallurgy*, Metals Park, OH: American Society for Metals, Vol 6, pp. 165-187.
- [7] Higgins R. A. (1986) *Engineering Metallurgy* (Part II), Edward Arnold, London, Vol. 3, pp. 111-126.
- [8] Nnuka E. E. (1991) The effect of micro additives on the quality and distribution pattern of the secondary phase in aluminium-copper alloy system, *The Nigerian Engineer*. Vol.26, No3, pp 30-37.
- [9] Nnuka, E. E. (1994) Effect of Dopants on the structure and properties of Aluminium and some of its Alloys, *JAST*. Vol.4. No. 2, pp 145-153.
- [10] Nnuka, E. E. (2002) Atomic sub-structure and mechanical properties of aluminium alloys with Zinc and copper, *Global Journal of Mechanical Engineering* Vol.3, No.2.,pp. 13-22.
- [11] Salimon A. I., Korsunsky A. M., Shelekhov E. V., Sviridova T. A., Kaloshkin S.D., and Tchrdyntse (2001) Crystallo-chemical aspects of solid state reactions in mechanically alloyed Al-Cu-Fe quasi-crystalline, *Acta material*, 49(10): 1821-1833.
- [12] Wang S. Q., Schneider M., Ye H. Q., and Gottstein G. (2004) First principle study of the formation of Guinier-Preston Zones in Al-Cu alloys. *Scripta Materialia*, Vol. 51, pp. 665-669.