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# Assessment of the mechanical behaviour of thermally aged B and Fe modified CuZnAl shape memory alloys

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**ABSTRACT:** The mechanical properties of unmodified, and 0.05 wt.% B and Fe modified CuZnAl alloys produced following liquid metallurgy route was investigated. The alloys were subjected to thermal ageing treatments at 200 °C and 450 °C; while mechanical testing and light microscopy were used for assessing the alloy response to the treatment. The results show that Microstructures with different structural features were observed in the unmodified-, and B, Fe modified- Cu–Zn–Al alloys, both in the unaged and aged conditions. The hardness of the unmodified Cu–Zn–Al alloy and the B modified Cu-Zn-Al alloys increased significantly with ageing at 200 °C and 450 °C, while the Fe modified Cu-Zn-Al alloy only exhibited marginal changes in hardness with thermal ageing treatment. Also, the ultimate tensile strength of the unmodified CuZnAl alloy was the most sensitive to ageing treatment performed at 200 °C, as UTS increase as high as 18.5% was compared to the 6.8 and 6.1% increases obtained for the Fe and B modified CuZnAl alloy compositions. The percent elongation of all the CuZnAl alloy compositions improved significantly with ageing treatment with peak values obtained when ageing is performed at 200 °C. It was opined that the generally marginal changes in the mechanical properties of the modified CuZnAl alloy compositions on ageing was due to the stabilizing effect of the B and Fe modifiers on the CuZn primary phase, which curtailed to some extent the tendency for precipitation of secondary phases.

**KEYWORDS:** CuZnAl; Martensite stabilization; Microalloying additions; Shape memory alloys; Transformation hysteresis; Thermal ageing

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**RESUMEN:** *Evaluación del comportamiento mecánico de la aleación con memoria de forma Cu-Zn-Al con adiciones de Boro y Hierro y tratadas térmicamente*. Se estudiaron las propiedades mecánicas de la aleación Cu-Zn-Al modificada y sin modificar con 0,05% en peso B y Fe fabricada siguiendo la ruta de estado líquido. La aleación se sometió a tratamientos térmicos de envejecimiento a 200 °C y 450 °C. Los ensayos mecánicos y de microscopía óptica se realizaron para evaluar la respuesta al tratamiento térmico. Los resultados mostraron la presencia de microestructuras con diferentes características estructurales en la aleación Cu-Zn-Al sin modificar y modificada con B y Fe, tanto en las condiciones sin envejecer como en las envejecidas. La dureza de la aleación Cu-Zn-Al sin modificar y modificada con B aumentó significativamente con los tratamientos a 200 °C y 450 °C, mientras que la aleación Cu-Zn-Al modificada con Fe solo mostró pequeños cambios en la dureza. Adicionalmente, la resistencia a la tracción final (RTF) de la aleación Cu-Zn-Al sin modificar fue la más sensible al tratamiento térmico a 200 °C, ya que el aumento de la RTF llegó hasta un 18,%, comparado con aumentos de 6,8 y 6,1% obtenidos con la aleación Cu-Zn-Al modificada con Fe y B, respectivamente. El porcentaje de alargamiento de todas las composiciones de la aleación Cu-Zn-Al mejoró significativamente con el tratamiento térmico, con valores máximos obtenidos con el envejecimiento a 200 °C. Se interpretó que los cambios, generalmente pequeños, en las propiedades mecánicas de la composición de la aleación Cu-Zn-Al modificada, se debieron al efecto estabilizador del B y del Fe en la fase primaria de Cu-Zn, que redujo en cierta medida la tendencia a la precipitación de las fases secundarias.

**PALABRAS CLAVE:** Adición microaleantes; Aleaciones con memoria de forma; Aleación Cu-Zn-Al; Ciclo de histéresis de la transformación; Envejecimiento térmico; Estabilización martensita

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## 1. INTRODUCTION

Shape memory alloys (SMAs) are known for their capacity to recover their pre-deformed shape in response to changes in temperature (shape memory effect) or stress (pseudoelasticity). Although several alloys display shape memory transformation, Cu based SMAs have been of keen interest to researchers because they are the most cheaply processable alloys which exhibit exploitable shape memory properties (Amini *et al.*, 2013). They are widely referred to as low cost shape memory alloys and considered long term replacement to NiTi based SMAs which possess better shape memory properties, albeit more expensive and difficult to process (Dar *et al.*, 2016).

One of the Cu based system whose shape memory properties have been explored extensively is Cu-Zn-Al alloy. Despite having attractive shape memory properties like wide transformation hysteresis and high strain recovery (5%); Cu-Zn-Al alloys just like other Cu based SMAs, have some limitations which lessens their use for sensing applications (Bujoreanu *et al.*, 2011). The alloys are often prone to brittleness due to coarse grain structures, a high degree of order and elastic anisotropy, and poor aging resistance which results in alteration in mechanical properties and transformation temperature (instability) hysteresis (Balo *et al.*, 2009; Alaneme and Okotete, 2016). The transformation hysteresis arises from the susceptibility of the alloys to precipitation of secondary phases (referred to as aging) during heating and cooling cycles. The precipitates results in effects such as martensite stabilization, phase hardening, stress and strain fields development within the vicinity of the precipitates during martensitic transformation. These phenomena significantly alters the martensite / austenite transformation temperatures and behaviour during heating and cooling, thus making Cu based SMAs unreliable in temperature sensitive systems (Babacan *et al.*, 2017).

Presently, there have been efforts aimed at addressing the limitations in Cu based SMAs, Cu-Zn-Al alloys inclusive (Dasgupta *et al.*, 2015; Kumar *et al.*, 2015). Some of the approaches which have been explored include the use of micro-alloying elements and thermomechanical processing (Suru *et al.*, 2016). Microalloying additions principally aid grain refinement, which helps to address the brittle tendency of these SMAs (Yang *et al.*, 2009; Alaneme *et al.*, 2017); and heat treatment/ thermomechanical processing have been explored to moderate aging response of Cu based SMAs (Stošić *et al.*, 2017). However, the effects of these interventions are not definite and appears dependent on the specific Cu based SMA and type of processing deployed (López-Ferreño *et al.*, 2013; Saud *et al.*, 2014; Yidiz *et al.*, 2015; Shivasiddaramiah *et al.*, 2016; de Castro Bubani *et al.*, 2017). The present investigation is focused on B and Fe modified Cu-Zn-Al alloys, which Alaneme *et al.* (2017) reported that the use of both cheaply sourced microalloying additions enhanced the mechanical properties of the Cu-Zn-Al alloys. However, the effect of thermal ageing on the transformation temperatures, mechanical and shape memory properties of these alloys has not received attention judging from the literature available on the subject in scientific research databases. This paper is delimited to the assessment of the effect of thermal ageing on the mechanical properties of the B and Fe modified Cu-Zn-Al alloys.

## 2. MATERIALS AND METHODS

### 2.1. Alloy and sample preparation

The study material, Cu-Zn-Al alloys containing B and F as microalloying elements were produced in accordance with Alaneme *et al.* (2017). The amounts of the base metals and the micro-alloying elements required to produce the Cu-Zn-Al based shape memory alloys were determined using charge calculations. The alloys containing 0.05% of micro-alloying elements (Fe and B) were produced using commercial purity copper, zinc, aluminium, iron and boron. A Cu-Zn-Al alloy composition without the addition of the microalloying elements was also prepared as a control composition to compare the results with those containing the microalloying elements. The Cu-Zn-Al alloys were all produced via liquid metallurgy following procedures described in details by Alaneme *et al.* (2017). The EDS analysis of the alloys produced showing the elemental composition is presented in Table 1.

### 2.2. Thermal aging treatment

The Cu-Zn-Al alloys produced were subjected to thermal ageing treatment to ascertain the effect of aging on the mechanical behaviour of the alloys. The procedure adopted was in accordance with Abid Ali (2010); and entailed firstly, heating all the samples to 800 °C for 1 hour in a muffle furnace and thereafter quenching in water maintained at 50 °C. This step was taken to ensure the formation of martensite phase in the alloys on cooling. The solution heat-treated samples were subjected to thermal ageing at

TABLE 1. Chemical Composition of Cu-Zn-Al alloys

| Composition | Cu (wt.%) | Zn (wt.%) | Al (wt.%) | Fe (wt.%) | B (wt.%) |
|---|---|---|---|---|---|
| A | 75 | 19 | 6 | – | – |
| B | 75 | 18.95 | 6 | 0.05 | – |
| C | 75 | 18.95 | 6 | – | 0.05 |

different temperatures: a set of samples were aged at 200 °C for one hour and then quenched in water maintained at 50 °C, while the second set of samples were subjected to thermal ageing at 450 ºC for one hour and also quenched in water maintained at 50 °C. Thermal treatments and corresponding sample designation for Cu-Zn-Al alloys are presented in Table 2.

### 2.3. Microstructural characterization

A Zeiss optical microscope was used for microstructural investigation of the alloys produced with a view to assess the grain structure and phase distribution. The specimens for microstructural examination were metallographically prepared following a series of grinding and polishing process. Subsequently, the specimens were etched in a solution of 5 g ferric chloride, 10 ml HCl, and 95 ml ethanol, by swabbing for 10-20 s; after which examination of the microstructures and image analysis were performed following standard procedures.

### 2.4. Mechanical testing

#### *2.4.1 Hardness measurement*

Hardness tests were carried out on the Cu-Zn-Al alloys using Indentec Hardness Testing Machine. The SMA samples were machined and the surfaces polished using emery papers of progressively fine grit sizes in accordance with standard metallographic procedures. This was to ensure that a smooth plane parallel surface is produced which allows for reliable measurement of the hardness of the alloys. A 50 Kgf load was applied on the sample surfaces for a duel time of 10 s. Five hardness indents were made on each specimen and readings within the margin of ±2% were taken for the computation of the average hardness values of the samples. The sample preparation and testing procedure was performed in accordance with ASTM E18-16 (2016) standard.

#### *2.4.2. Tensile testing*

The tensile properties of the Cu-Zn-Al alloys produced were evaluated by tensile testing using an Instron universal testing machine. Specimens for the test were machined to tensile test specifications of 30 mm gauge length, 4 mm guage diameter, 6 mm grip diameter and grip length of 10mm. The tests were performed at room temperature, and specimens were mounted on the testing platform and pulled monotonically at a strain rate of $10^{-3}$/s until fracture. The specimen preparation and testing procedure were in accordance with ASTM E8/E8M-15a (2015) standard. For each Cu-Zn-Al alloy composition, three repeat tensile tests were performed to guarantee the consistency and dependability of the data generated.

## 3. RESULTS AND DISCUSSION

### 3.1. Microstructure

Representative optical micrographs of the unmodified and modified Cu-Zn-Al alloys in the unaged and aged conditions are presented in Figs. 1 - 3. It is observed from Fig. 1a that the unmodified CuZnAl alloy is characterized by needle-like lath grain structures which is a finger print feature of lath martensitic structures (Dasgupta, 2014; Alaneme *et al.*, 2017). The differentiating features of the microstructural changes which occurred in the unmodified CuZnAl alloy on ageing at 200 ºC and 450 ºC, were not distinctively discernable at the resolution of the optical micrographs, as can be confirmed from Fig. 1b. Abid Ali (2010) reported that ageing of CuZnAl alloy with an initial lath martensitic microstructure at temperatures of 450 ºC, results in the precipitation of $\gamma$ - type phases, typically FCC structured (Suru *et al.*, 2016). The microstructures of the Fe and B modified CuZnAl alloys have been reported to be characterized by round/elliptical grain edges (Alaneme *et al.*, 2017). However, from Figs. 2 and

TABLE 2. Ageing temperatures and Sample designation after heat treatment

| Heat Treatment | Composition | Sample Designation |
|---|---|---|
| 850 °C/1hr/$H_2O$ at 50 °C | Cu-Zn-Al | A1 |
| | Cu-Zn-Al-0.05Fe | B1 |
| | Cu-Zn-Al-0.05B | C1 |
| 850 °C/1hr/$H_2O$ at 50 °C + 200 °C/1hr/ $H_2O$ | Cu-Zn-Al | A2 |
| | Cu-Zn-Al-0.05Fe | B2 |
| | Cu-Zn-Al-0.05B | C2 |
| 850 °C/1hr/$H_2O$ at 50 °C + 450 °C/1hr/ $H_2O$ | Cu-Zn-Al | A3 |
| | Cu-Zn-Al-0.05Fe | B3 |
| | Cu-Zn-Al-0.05B | C3 |

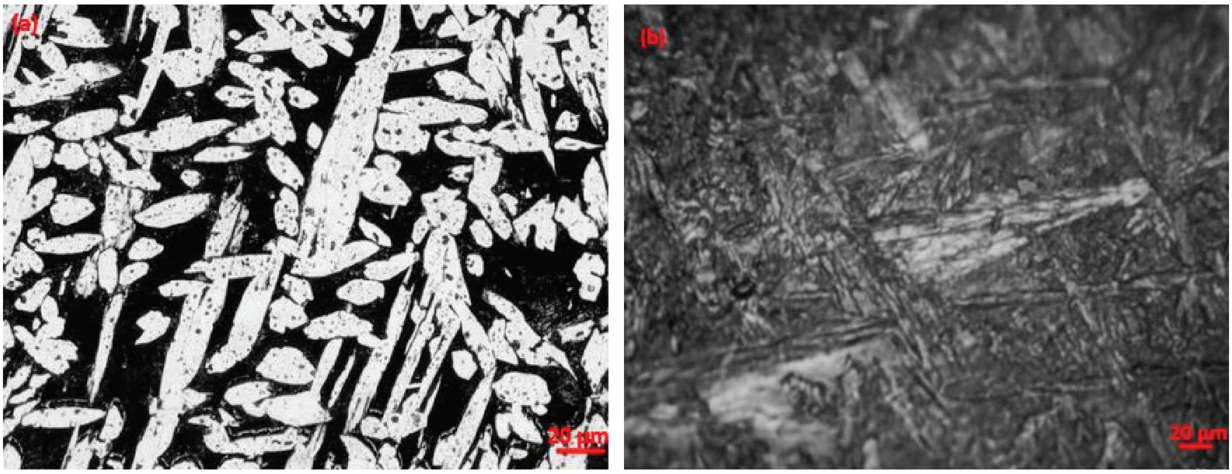


FIGURE 1. Representative optical micrographs of (a) Unmodified Cu-Zn-Al alloy, and (b) Unmodified Cu-Zn-Al alloy aged at 200 °C.

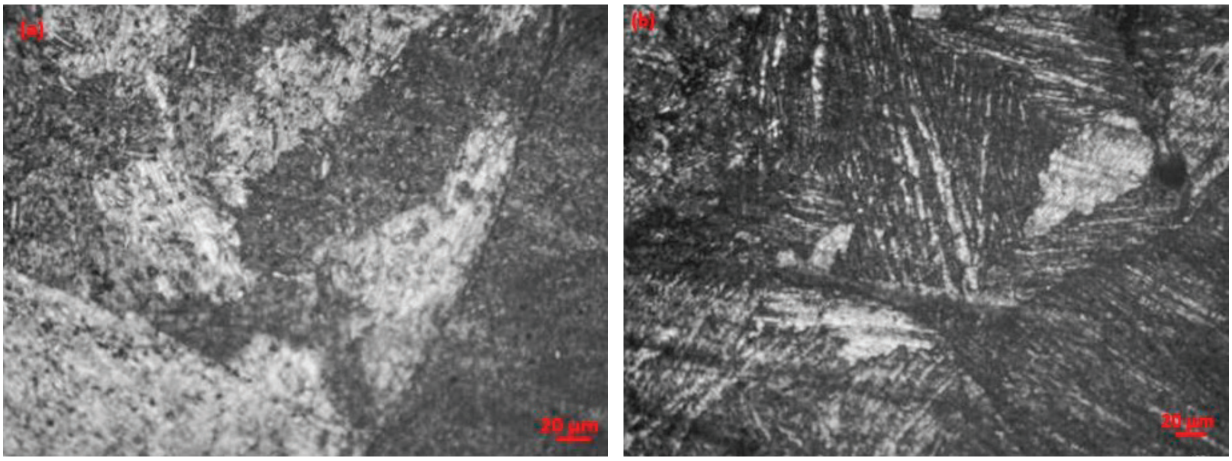


FIGURE 2. Representative optical micrographs of (a) 0.05% Fe modified CuZnAl alloy and (b) 0.05% Fe modified CuZnAl alloy aged at 450 °C.

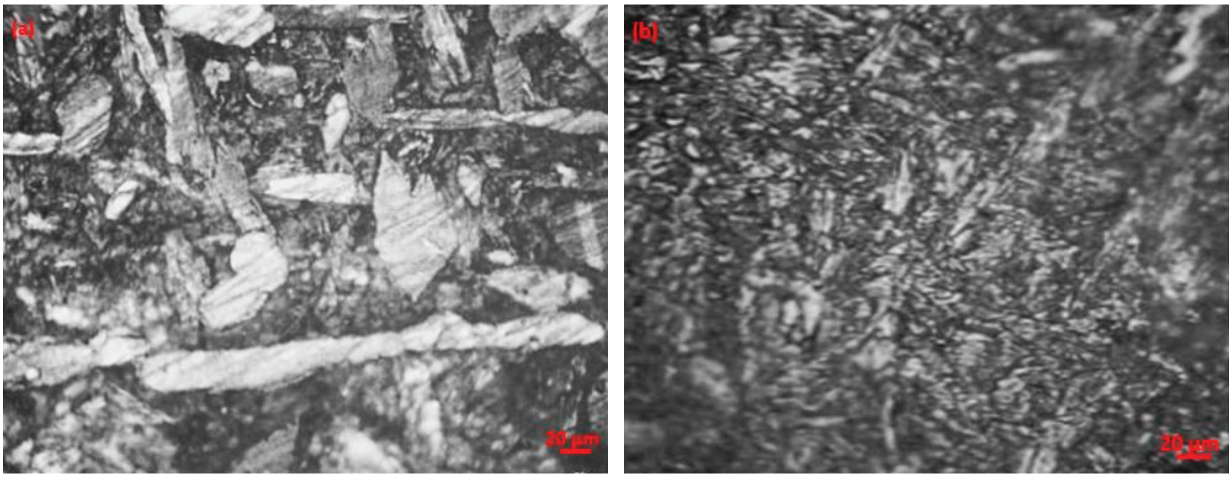


FIGURE 3. Representative optical micrographs of (a) 0.05% B modified Cu-Zn-Al alloy, and (b) 0.05% B modified CuZnAl alloy aged at 450 °C.

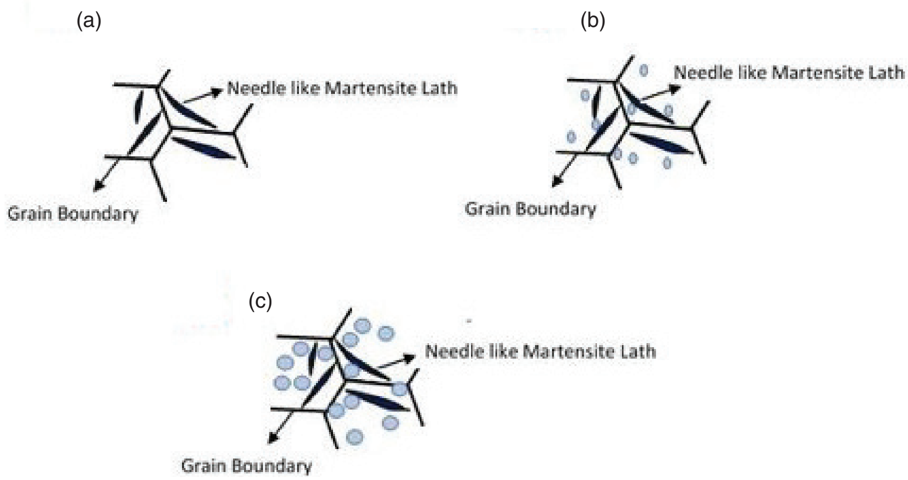


FIGURE 4. Idealized precipitation dynamics for Unmodified Cu-Zn-Al alloy from (a) unaged condition, (b) aged at low temperature, and (c) aged at relatively high temperature.

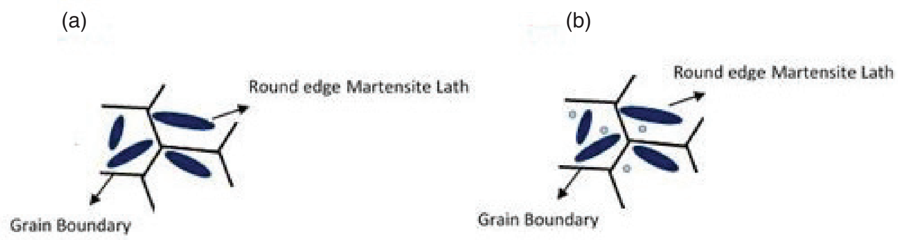


FIGURE 5. Idealized precipitation dynamics for modified Cu-Zn-Al alloy from (a) unaged condition, (b) aged condition.

3 (as in the case with the unmodified CuZnAl alloy), the precipitates potentially formed during the ageing process cannot be resolved by the resolution limits of light microscopy. Hence the effect of the ageing process on the precipitation and transformation behaviour was not discussed in this study. For unmodified Cu based alloys, the precipitation dynamics has been reported to be largely influenced by the ageing temperature – finer precipitate sizes are generally formed at lower ageing temperatures while larger precipitate size and quantity normally occurs at relatively higher ageing temperatures (Bhuniya *et al.*, 2005; Suresh and Ramamurty, 2007; Sari *et al.*, 2011). This precipitation order is idealized in the pictorial description presented in Fig. 4. Thus it is expected that the precipitates formed at 200 °C will be finer in size compared to those formed at 450 °C. The presence of the modifiers is opined will stabilize the CuZn primary phase and make it less susceptible to precipitation on ageing as idealized in Fig. 5. This line of thought is supported by the mechanical properties results presented in the succeeding section.

## 3.2. Mechanical Properties

### *3.2.1. Hardness*

The average hardness values of the unaged and thermally aged Cu-Zn-Al alloys produced are presented in Fig. 6. It is observed that the hardness values of the unaged samples (A1, B1, and C1) were within the same range with very marginal difference (< 2%). It is however observed that the hardness of the unmodified Cu-Zn-Al alloy (A series) and the B modified Cu-Zn-Al alloys (C series) were sensitive to ageing treatment as the hardness increased significantly with ageing at 200 and 450 °C. This can be attributed to the formation of precipitates, which have been reported to be principally $\gamma$ precipitates which help in hardening of the alloy (Pons and Portier, 1997). It was also observed that the highest hardness for the unmodified and B modified CuZnAl SMAs was obtained for samples aged at 200 °C. This may be linked to finer precipitate sizes which increases the number of dislocations-particles interactions (Orowan strengthening) and contributes to

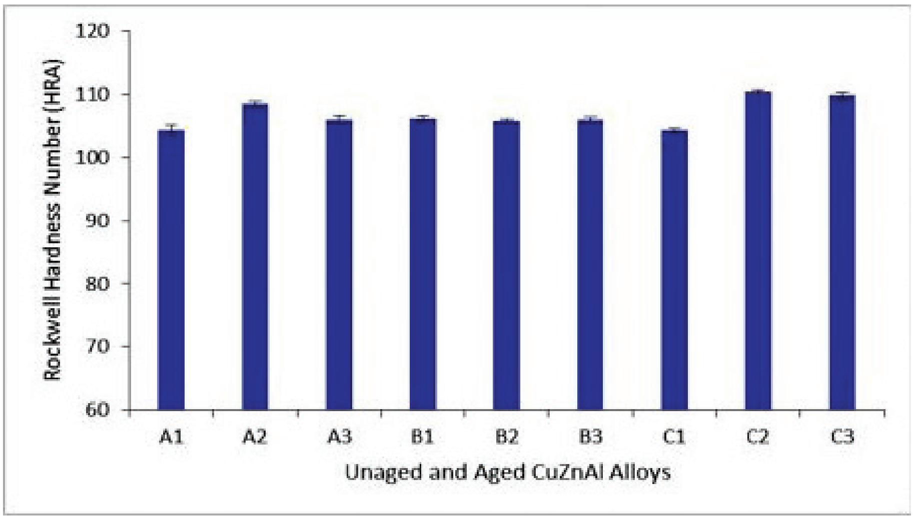


FIGURE 6. Hardness of aged and unaged Cu-Zn-Al based alloys.

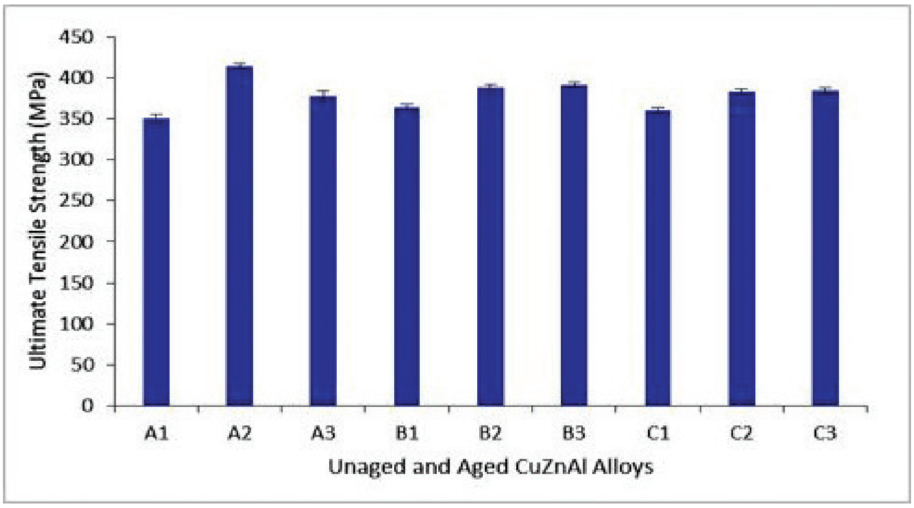


FIGURE 7. Ultimate Tensile Strength of Unaged and Aged Cu-Zn-Al based alloys.

dislocations immobilization (de Castro Bubani *et al.*, 2017). The Fe modified Cu-Zn-Al alloys are however observed to show the least sensitivity to ageing as the hardness values is somewhat invariant to the thermal ageing treatment. This suggests that the presence of Fe greatly stabilizes the CuZn primary phase (matrix) and inhibits precipitation of new phases to some extent.

### 3.2.2. *Ultimate Tensile Strength*

The ultimate tensile strength (UTS) of the unaged and thermally aged Cu-Zn-Al alloys produced are presented in Fig. 7. It is observed that for the unaged CuZnAl alloys, the unmodified Cu-Zn-Al alloy composition has lower UTS value compared to the Fe and B modified Cu-Zn-Al alloy compositions. Alaneme *et al.* (2017) described this trend to be on account of change in grain morphology from sharp edged (for the unmodified CuZnAl alloy) to round/elliptical grain edge (for the modified Cu-Zn-Al alloys), which minimizes the effect of stress concentration and gives it more stress bearing capacity. From the results, it is also observed that the aged samples (unmodified and modified) have higher UTS values compared to samples which were not subjected to ageing heat treatment. The percent increase in UTS for the unmodified Cu-Zn-Al alloy is 18.5 and 7.6% for samples aged at 200 and 450 ºC, respectively. The higher UTS values observed on aging of the unmodified Cu-Zn-Al alloys may be linked to the higher number of precipitates formed in the alloys. The precipitates contribute to strengthening on account of particles/dislocations interaction (Orowan strengthening) and the pinning effect precipitates have on dislocations by serving as dislocation movement barrier (Saud *et al.*, 2015; de Castro Bubani *et al.*, 2017).

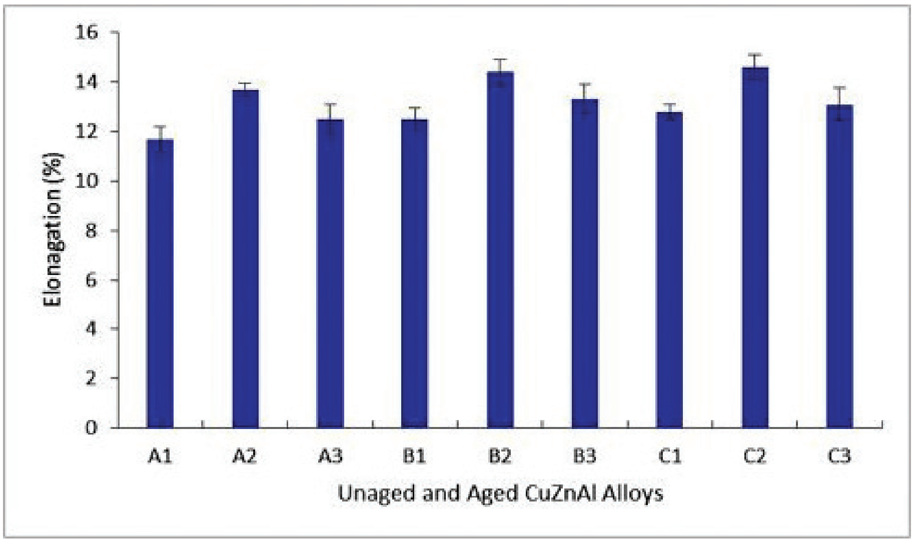


FIGURE 8. Percent elongation of Unaged and Aged Cu-Zn-Al based alloys.

For the modified Cu-Zn-Al alloy compositions, the percent increase in UTS is less significant compared to the unmodified alloy composition. In the case of the Fe modified composition, 6.8 and 7.6% increase in UTS was obtained, while 6.1 and 6.7% increase was obtained in the B modified alloys for ageing at 200 and 450 ºC, respectively. The lesser sensitivity to ageing observed in the modified alloys may be due to the stabilizing effect of Fe and B microalloying additions on the primary CuZn phase which inhibits precipitation of secondary phases to some extent.

### 3.2.3. Elongation

The percent elongation results of the unaged and thermally aged Cu-Zn-Al alloys produced are presented in Fig. 8. It is observed that the percentage elongation of the CuZnAl alloy compositions was sensitive to thermal ageing temperature. The samples subjected to thermal ageing had higher percent elongation values compared to the unaged CuZnAl alloys, and the values were higher for the alloys aged at 200 °C. This indicates that the thermal ageing treatment which leads to the likely precipitation of secondary phases, does not have an adverse effect on the ductility of the alloys, and generally results in improved mechanical properties.

## 4. CONCLUSIONS

The mechanical behaviour of thermally aged unmodified and B-, Fe- modified Cu–18Zn–6Al alloys was investigated. From the results, the following conclusions are drawn:

- Microstructures with different structural features were observed in the unmodified-, and B, Fe modified- Cu–Zn–Al alloys, both in the unaged and aged conditions. However, the effect of the thermal ageing on the microstructure was difficult to establish in details because of the resolution limit of light microscopy used for the characterization.
- The hardness of the unmodified Cu–Zn–Al alloy and the B modified Cu-Zn-Al alloys increased significantly with ageing at 200 °C and 450 °C while the Fe modified Cu-Zn-Al alloys were the least sensitivity to ageing as the hardness values changed marginally with thermal ageing treatment.
- The ultimate tensile strength of the unmodified CuZnAl alloy was the most sensitive to ageing treatment performed at 200 °C, as UTS increase as high as 18.5% was compared to the 6.8 and 6.1% increases obtained for the Fe and B modified CuZnAl alloy compositions aged at the same temperature.
- The percent elongation of all the CuZnAl alloy compositions improved significantly with ageing treatment with peak values obtained when ageing is performed at 200 °C.
- Generally the ageing phenomena did not show any adverse effect to the mechanical properties.
- It was opined that the B and Fe modifiers may have stabilizing effect on the CuZn primary phase, which curtails to some extent precipitation of secondary phases on ageing, justifying the marginal changes observed in mechanical properties of the modified CuZnAl alloy compositions in the unaged and aged conditions.

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