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Flow stress behaviour and microstructural analysis of hot deformed Aluminium matrix composites reinforced with CuZnAlNi shape memory alloy particles

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ABSTRACT: The compressive flow stress behaviour and microstructures of hot deformed Al alloy matrix composites (AMCs) reinforced with CuZnAlNi based shape memory alloy (SMA) particles was investigated. Al-Mg-Si based alloy, reinforced with 4, 6, and 8 wt.% Cu-18Zn-7Al-0.3Ni, and 8 wt.% SiC particles, were produced by double stir casting and subjected to hot compression testing at 1.0 s^{-1} strain rate, 400 °C temperature, and ~ 60% constant global strain using a Gleeble 3500 thermomechanical simulator. The starting and as-deformed microstructures of the composites were examined using optical microscopy. The use of Cu-18Zn-7Al-0.3Ni particles as reinforcement resulted in the development of finer matrix structure compared with the use of SiC. The flow stress and hardness of the AMCs reinforced with Cu-18Zn-7Al-0.3Ni particles were generally higher than that of the unreinforced Al alloy and the SiC reinforced Al alloy. Also the flow stress, and to a large extent the hardness, increased with increase in the weight percent of Cu-18Zn-7Al-0.3Ni particles in the AMC. The improvement observed with the use of Cu-18Zn-7Al-0.3Ni alloy particles was ascribed to the combination of enhanced matrix grain refinement strengthening, interfacial strengthening, compressive residual stresses, high thermal conductivity, and damping capacity offered by the Cu-18Zn-7Al-0.3Ni alloy.

KEYWORDS: CuZnAlNi alloy; Flow stress; Hot deformation; Microstructure; Shape memory alloy reinforced Al based composites; Strengthening mechanism

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RESUMEN: *Comportamiento del flujo de esfuerzo y análisis microestructural de compuestos de matriz de aluminio deformados en caliente y reforzados con partículas de la aleación con memoria de forma CuZnAlNi.* Se investigó el comportamiento del flujo de esfuerzo por compresión y las microestructuras de los compuestos de matriz de aleación de Al deformada en caliente (AMC) y reforzados con partículas de la aleación con memoria de forma (SMA) basadas en CuZnAlNi. La aleación base Al-Mg-Si reforzada con 4, 6 y 8% en peso de Cu-18Zn-7Al-0,3Ni, y 8% en peso de partículas de SiC, se obtuvieron mediante agitación doble y se sometieron a pruebas de compresión en caliente a una velocidad de deformación de 1,0 s^{-1}, temperatura de 400 °C, y ~60% de deformación global constante, para ello se utilizó un simulador termo-mecánico Gleeble 3500. Las microestructuras iniciales y deformadas de los compuestos se examinaron utilizando microscopía óptica. El uso de partículas Cu-18Zn-7Al-0,3Ni como refuerzo dio como resultado el desarrollo de una estructura de matriz más fina en comparación con el SiC. La tensión de flujo y la dureza de los AMC reforzados con partículas de Cu-18Zn-7Al-0,3Ni fueron generalmente más altos que los de la aleación de Al no reforzada y la aleación de Al reforzada con SiC. También la tensión de flujo y, en gran medida, la dureza creció con el aumento en el porcentaje en peso de partículas de Cu-18Zn-7Al-0,3Ni en el AMC. La mejora observada con el uso de partículas de la aleación

Cu-18Zn-7Al-0,3Ni se atribuyó a la combinación del refuerzo mejorado y el refinamiento del tamaño grano de la matriz, fortalecimiento la interface, el esfuerzo residual de compresión, la alta conductividad térmica, y la capacidad de amortiguación ofrecida por la aleación Cu-18Zn-7Al-0,3Ni.

PALABRAS CLAVE: Aleación base Al reforzada con composites; Aleación CuZnAlNi; Flujo de esfuerzo; Deformación en caliente; Memoria de forma; Mecanismo de refuerzo

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1. INTRODUCTION

Light weight structural materials which can combine high strength, shock resistance and damping capacity are now the materials of choice for several industrial and load bearing applications (Anshuman, 2017; Chen *et al.*, 2019). Steel which basically has been the main stay structural material for the same applications, is gradually losing prominence on account of its high density and limited damping capacity (Alaneme *et al.*, 2019a). Several other monolithic alloys equally fall short of the desired combination of properties for modern structural and load bearing applications.

Aluminium matrix composites in recent times have been found desirable for several new-age technological applications on account of the stunning property combinations, which they can be tailored to possess. Thus, they are now commercially utilized in automobile, aerospace, defense and marine applications, among others (Prasad *et al.*, 2014; Bodunrin *et al.*, 2015). As part of measures to enhance product quality assurance and reliability, there are concerted efforts to boost the property base of AMCs, particularly their significantly lowered damage tolerance, toughness and ductility compared to the base Al alloys (Lu *et al.*, 2019; Abraham *et al.*, 2019). This is essentially due to the predominant use of ceramics as reinforcement in the AMCs (Zhao *et al.*, 2016). There have been some attempts to address this with the use of metallic materials as reinforcement (Alaneme *et al.*, 2019b). The metallic materials (in form of particles or fibres) because they are inherently tougher and more ductile than ceramics, potentially help improve the shock resistance and formability of the composites (Fathy *et al.*, 2015; Gopi Krishna *et al.*, 2018). However, the effect their selection has on the high temperature mechanical properties and damping capacity of the AMCs, has not come under scientific scrutiny.

In the present study, the use of low-cost shape memory alloys based on CuZnAl is proposed for use as reinforcement. Shape memory alloys (SMAs) are largely noted for their good thermoelastic characteristics and excellent damping properties (Jani *et al.*, 2014; Alaneme and Okotete, 2016). Being an intrinsically high damping capacity material, the use of CuZnAl as reinforcement is expected to provide the composite, capacity to dampen undesirable mechanical vibrations and wave propagation. However, its effect on the high temperature mechanical properties of the AMCs requires investigation. This is pertinent in automobile components such as engine blocks, connecting rods, and engine casing, among others; where the operating conditions induces stresses and heat generation which can instigate premature failure of the components (Hasem *et al.*, 2016). Hence the need to ascertain the high temperature strength of the CuZnAlNi reinforced AMC.

A number of studies have shown that the use of SMAs as reinforcement in AMCs help enhance the adaptive properties of the AMCs through the reversible thermoelastic martensitic transformation which engenders native sensing, high damping capacity and self-strengthening (Wei *et al.*, 1997; Ni *et al.*, 2016; Oliveira *et al.*, 2017; Huang *et al.*, 2019). They are reported to contribute to strengthening and enhanced fracture toughness due to the microscopic scale thermal residual compressive stresses they assert on the matrix, due to difference in coefficient of thermal expansions (CTEs) between them and the matrix (Huang *et al.*, 2019). However, none of these studies appraised the hot mechanical strength of the composites in comparison with that of traditional ceramic reinforced AMCs. The research questions which this study intends to provide answers to are:

- How does the flow stress behaviour of the hot deformed Al-CuZnAl base composites differ from that of the unreinforced Al alloy and the Al alloy reinforced with SiC?
- Is the flow stress behaviour dependent on the weight percent of the CuZnAl SMA?
- Can the microstructures provide useful insight on any materials phenomena influencing the observed deformation trends?
- Are the ensuing microstructures of the AMCs homogeneous all through sample geometry or heterogeneous?

The answers to these research questions will help provide better understanding of the suitability or otherwise of CuZnAl based SMA as reinforcement in the development of AMCs for stress bearing and vibration damping applications.

2. MATERIALS AND METHODS

Al-Mg-Si alloy with composition (Al-0.43Si--0.42Mg-0.1Fe-0.11Cu-0.02Mn-0.01Zn) was selected as composite matrix, while Cu-18Zn-7Al-0.3Ni based SMA developed from a previous study (Alaneme and Umar, 2018) was selected as reinforcement. The CuZnAlNi SMA was milled to average particle size of 30 μm using a conventional planetary ball mill, to have the same particle size range with SiC particles which were used to develop the AMC composition that served as control sample. AMCs containing 4, 6, and 8 wt.% of the CuZnAlNi SMA particles were developed alongside the unreinforced alloy and AMC containing 8 wt.% SiC – both of which served as control compositions. The composite production procedure followed the well-established double stir casting process (Alaneme and Aluko, 2012), and commenced with charge calculations. The Al-Mg-Si alloy was melted completely in a crucible furnace, then allowed to cool slowly to a semi-solid state (~600 ºC) before preheated CuZnAlNi particles were added to form a semi-solid metallic slurry, which manual stirring for 5 min was used to achieve a homogeneous mix. The mixture was superheated to 750 °C ± 20 °C, and the stirred again, using a mechanical stirrer, operated at 400 rpm for 10 min. The composite was then cast into sand moulds. The same process was followed for the production of the AMC composition reinforced with 8 wt.% SiC. The sample compositions produced and the sample designations are summarized in Table 1. The composites produced were all machined to cylindrical specimen configuration with dimensions 10 mm diameter and 15 mm length. Isothermal hot compression tests were then performed on the samples using a Gleeble 3500 thermomechanical simulator at 1.0 s^{-1} strain rate, 400 ºC temperature, and ~ 60% constant global strain. Prior to hot-compression testing, nickel paste and graphite foil were placed between the samples and the ISO-T tungsten carbide anvils to reduce frictional effects. Additionally, chromel-alumel thermocouple was attached to the center of the composite samples to measure the temperature of the samples during the experiment. The samples were heated to the deformation temperature of 400 °C at a heating rate of 5 °C·s^{-1} and holding at the temperature isothermally for 180 s to ensure uniform sample temperatures. The samples were then compressed until the global strain was attained, at which point the samples were immediately compressed air-cooled to preserve their microstructures. Optical microscopy (OM) using a Leica DM5000 optical microscope was used to assess the as-cast and post deformation microstructures of the AMCs. Prior to microscopy, Samples were cut parallel to the deformation axis and were mounted using a polyfast resin. The samples were ground on a silicon carbide abrasive paper with different grit sizes of 600, 800, 1200, 2400 and 4000. Thereafter, the samples were polished using colloidal silica suspension on a Struers polishing machine operated at rotating speed of 150 rpm and a counter rotation of 50 rpm for 10 min. The polished samples were then etched in Weck's reagent for approximately 18 s. Similar metallographic procedure was performed on the as-cast composite samples to obtain the initial microstructures. The microstructural examination of the deformed samples was performed by imaging the sample microstructures at the centre, edge and edge length as illustrated in Fig. 1. The phases present in the as-cast composites were determined by carrying out X-Ray Diffraction (XRD) measurement on the as-cast composites using BRUKER D2 Phaser machine. The machine was fitted with a copper anode material and operated at generator settings of 10 mA, 30 kV. The samples were scanned at 2θ = 5 – 90° at room temperature. The XRD patterns were interpreted using Panalytical Highscore software. Vickers microhardness tests were also performed on the deformed samples, which

TABLE 1. Composite Composition and Sample designations

Sample Designation	Composite Composition
Al1	Unreinforced Al alloy
Al2	Al alloy + 4 wt.% Cu-17Zn-7Al-0.3Ni
Al3	Al alloy + 6 wt.% Cu-17Zn-7Al-0.3Ni
Al4	Al alloy + 8 wt.% Cu-17Zn-7Al-0.3Ni
Al5	Al alloy + 8 wt.% SiC

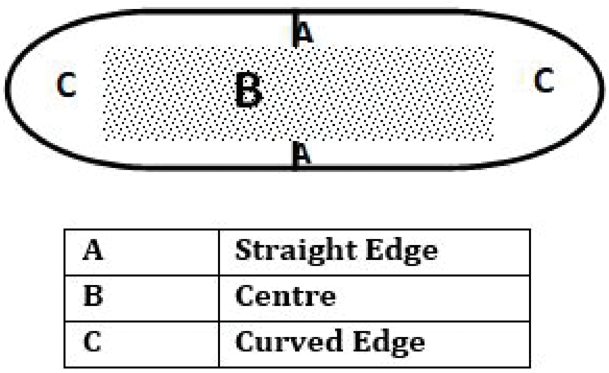

FIGURE 1. Schematic illustration of sections of the as-deformed samples examined using optical microscopy.

were polished to obtain plane parallel, smooth surfaces. The test was conducted on a Future Tech FM 700 Vickers microhardness testing machine using diamond indenter at an applied load of 300 gf for 15 s. Six repeat test measurements were taken for each sample and the average hardness determined.

3. RESULTS

3.1. As-cast microstructures and X-Ray Diffraction

The as-cast microstructures of the AMCs produced as presented in Fig. 2. It is observed that the microstructures show varied grain sizes for the composites produced. The SiC reinforced AMC (Fig. 2e) is observed to have relatively larger grain sizes compared to the Cu-18Zn-7Al-0.3Ni particles reinforced composite grades. This can be attributed to the Cu-18Zn-7Al-0.3Ni particles which offer a greater undercooling effect during solidification of the composite compared to the SiC particles. The Cu-18Zn-7Al-0.3Ni particles being inherently Cu based, have a high thermal conductivity, which favours a faster rate of cooling and hence the formation of more solidification nuclei (Guerione *et al.*, 2008). Also, the high damping capacity reported for CuZnAl based SMAs (Van Humbeeck, 2003; Crăciun *et al.*, 2016) implies the availability of a large number of heat dissipation centres during solidification unlike with the use of SiC. Thus, the microstructures suggest that the use of Cu-18Zn-7Al-0.3Ni particles offers improved grain refinement to the starting microstructure compared with the use of SiC as reinforcement. The phases present in the composites are observed in the XRD spectra presented in Fig. 3. The unreinforced Al alloy and SiC reinforced Al alloy, show peaks of Al, and Al, SiC, and Si, respectively, which are the phases expected. For the Cu-18Zn-7Al-0.3Ni reinforced AMCs, it is observed that variants of AlCu phase, depending on the weight percent of the SMA particles, are formed. For the 4 wt.% Cu-18Zn-7Al-0.3Ni reinforced Al, AlCu was the secondary phase identififed, while AlCu and Al_2Cu were the phases identified in the AMC composition containing 6 wt.% of the Cu-18Zn-7Al-0.3Ni particles. The AMC composition containing 8 wt.% Cu-18Zn-7Al-0.3Ni particles, had Al_2Cu and $Al_{0.92}Cu_{0.08}Mg$ as the identified secondary phases.

3.2. Compressive stress-strain behaviour and hardness

The flow stress – strain profiles of the AMCs are presented in Fig. 4. It is observed that the flow stress of the AMCs reinforced with Cu-18Zn-7Al-0.3Ni particles were generally higher than that of the unreinforced Al alloy and the Al alloy reinforced with SiC. It is also noted that the flow stress increased with increase in the weight percent of Cu-18Zn-7Al-0.3Ni particles in the AMC. This trend is largely consistent with the hardness trend of the as-deformed AMCs presented in Fig. 5. The increased flow stress and hardness of the AMCs containing Cu-18Zn-7Al-0.3Ni can be attributed partly to matrix strengthening due to grain refinement, as evidenced in the micrographs presented in Figs. 6–8. The greater pinning effect to dislocation motion due to the much finer deformation structures formed in the Cu-18Zn-7Al-0.3Ni reinforced AMCs compared to that of the unreinforced Al and the SiC reinforced AMC, contributed to improved strength observed. This position is specifically supported by the microstructures (Figs. 6–8), where it can be seen that the SiC reinforced AMCs have larger grain sizes compared to the AMC compositions reinforced with Cu-18Zn-7Al-0.3Ni particles. Thus the microstructure points to matrix strengthening as a factor responsible for the improved strength exhibited in the hot compressed Cu-18Zn-7Al-0.3Ni particle reinforced AMC in comparison to the AMC composition reinforced with SiC. In AMCs, strengthening is often ascribed to direct and indirect strengthening effects – the direct being the contribution from load transfer from the matrix to the particles, and the indirect from the additional dislocations created during cooling on account of the difference in CTEs between the matrix and the reinforcement (Chawla and Shen, 2001). In the case of the Cu-18Zn-7Al-0.3Ni particles reinforced AMCs some additional factors could be advanced for the improved strength compared to that of the SiC reinforced AMC - the likely improved interface bonding between the particles and matrix, and the compressive type residual stresses reported to be asserted by SMA particles on the matrix on cooling (Lee and Taya, 2004; Kotresh *et al.*, 2018). Metallic reinforcements are reported to offer better interface bonding to metallic matrices compared to ceramic particles (Selvakumar *et al.*, 2017; Alaneme *et al.*, 2019c). This improved bonding results in enhanced interface strength, which allows for effective load transfer between the matrix and the particles (Huang *et al.*, 2018). Furthermore, the compressive type residual stresses asserted by the particle on the matrix on cooling, is reported not to be relieved by heattreatment (Kotresh *et al.*, 2018), and hence effective even when the composite is exposed to elevated temperatures. The corollary is that despite the relatively lower melting point of the Cu-18Zn-7Al-0.3Ni alloy compared to the refractory SiC, the SMA provides higher mechanical strength at temperatures within the hot working confines of the AMC, which is typical for most automobile engine working temperatures. That is, the use of Cu-18Zn-7Al-0.3Ni SMA will provide improved high temperature strength for the composite. The reasons for this could be summarily ascribed to improved interfacial strength, thermoelastic properties, high

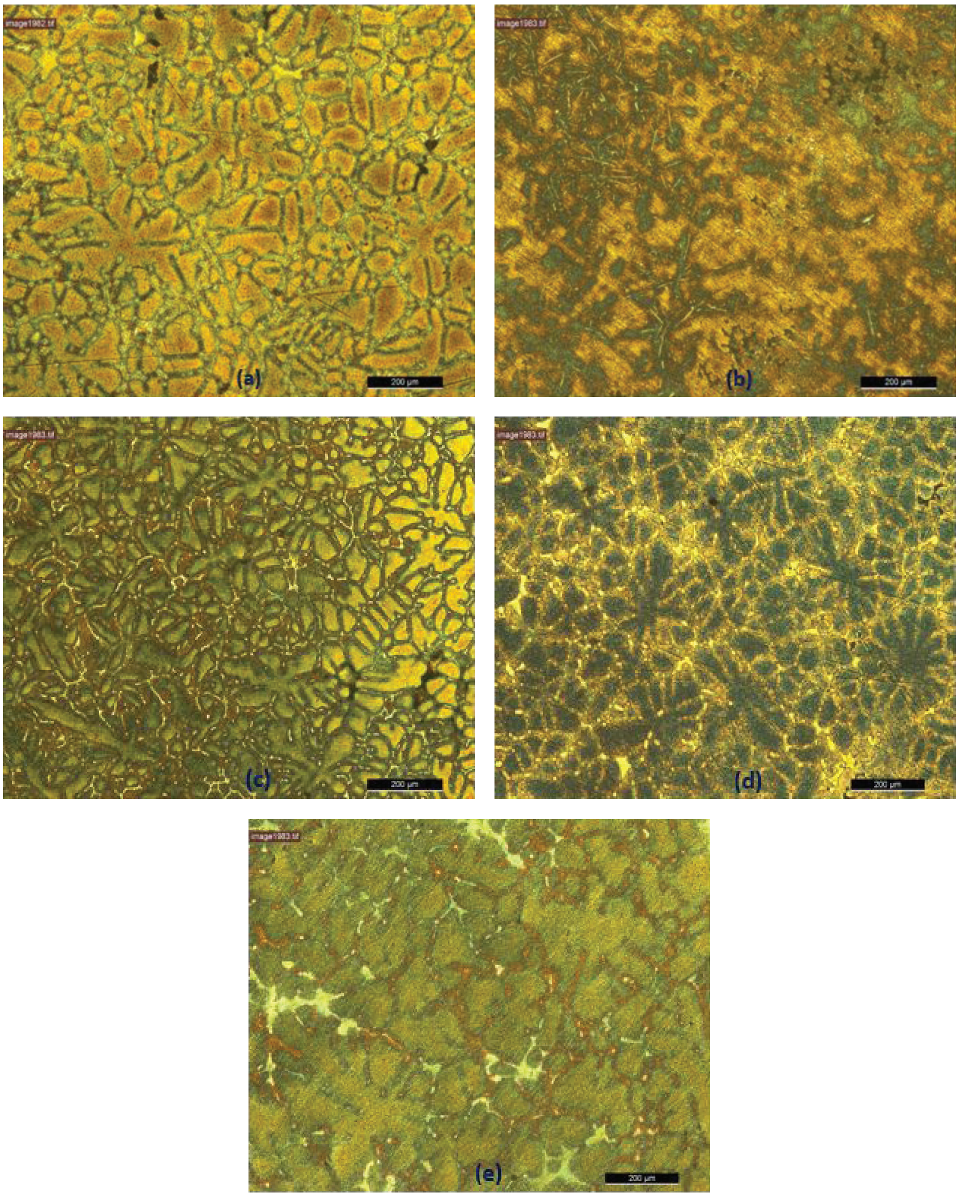

Figure 2. (a) as-cast microstructure of unreinforced Al alloy, (b) as-cast microstructure of Al alloy reinforced with 4 wt.% Cu-18Zn-7Al-0.3Ni, (c) as-cast microstructure of Al alloy reinforced with 6 wt.% Cu-18Zn-7Al-0.3Ni, (d) as-cast microstructure of Al alloy reinforced with 8 wt.% Cu-18Zn-7Al-0.3Ni, and (e) as-cast microstructure of Al alloy reinforced with 8 wt.% SiC.

thermal conductivity and damping capacity of the Cu-18Zn-7Al-0.3Ni alloy.

3.3. Microstructural variation in deformed samples

The microstructures of the as-deformed composites (Figs. 6 – 8), are noted to show the nature of strain distribution during hot deformation of the composites. It is observed that for all the AMCs investigated, the deformation bands are more conspicuous at the centre of the sample than at the edges, where the grains appear close to equiaxed in shape. At the centre of the sample, the grains are observed to be elongated, with orientation perpendicular to the loading direction. This should be noted is reflective of uneven strain distribution as the deformation strains are more intense at the centre compared to the outer fringes (edges) of the samples. This behaviour is very much consistent with observations reported in literatura (Poletti *et al.*, 2009; Fan *et al.*, 2017). It is also noted that the SiC reinforced AMC had more significantly enlarged grains oriented along the deformation axis compared to that of the Cu-18Zn-7Al-0.3Ni

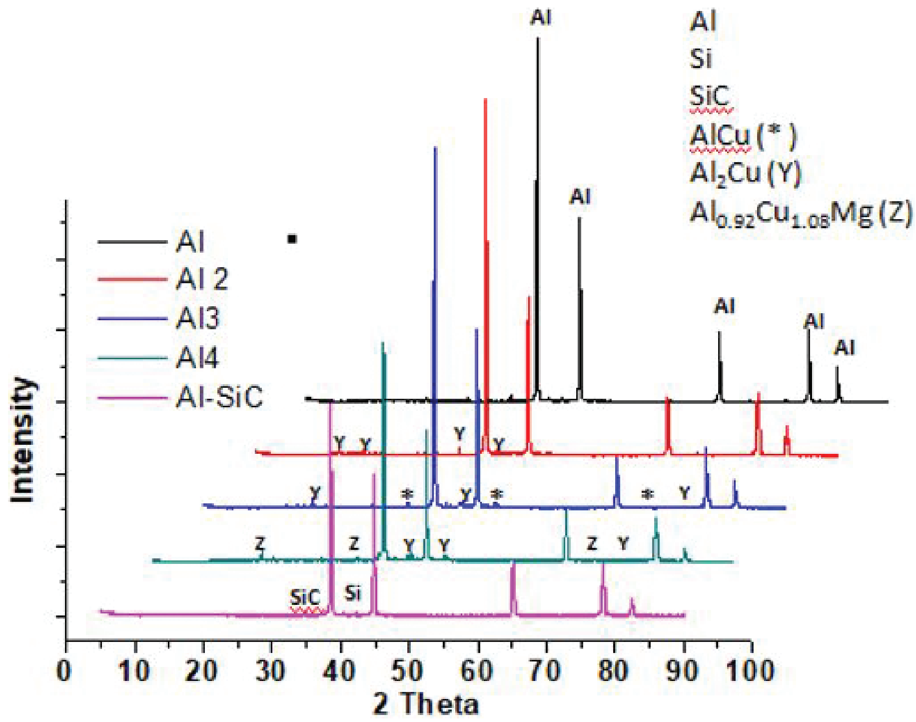

FIGURE 3. XRD profile of the Al alloy and Al based composites produced.

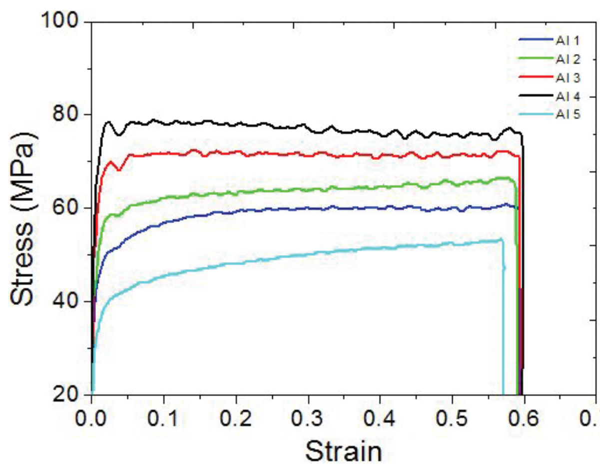

FIGURE 4. Flow stress – strain curves of the Al alloy and Al based composites produced.

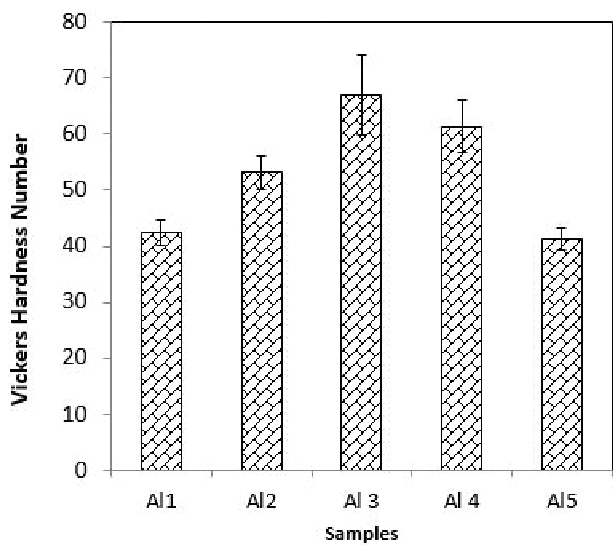

FIGURE 5. Vickers hardness number of the Al alloy and Al based composites produced.

reinforced AMCs. This is a pointer to matrix softening on account of reduced boundary strengthening, which can explain the low flow stress and hardness values observed compared with the Cu-18Zn-7Al-0.3Ni composite grades investigated.

4. CONCLUSION

The compressive stress behaviour and microstructural analysis of hot deformed Aluminium matrix composites reinforced with Cu-18Zn-7Al-0.3Ni shape memory alloy and SiC particles was investigated in this study. From the results, the following conclusions are drawn:

- The use of Cu-18Zn-7Al-0.3Ni particles resulted in the development of finer matrix structure compared with the use of SiC as reinforcement in the AMCs produced, which was attributed

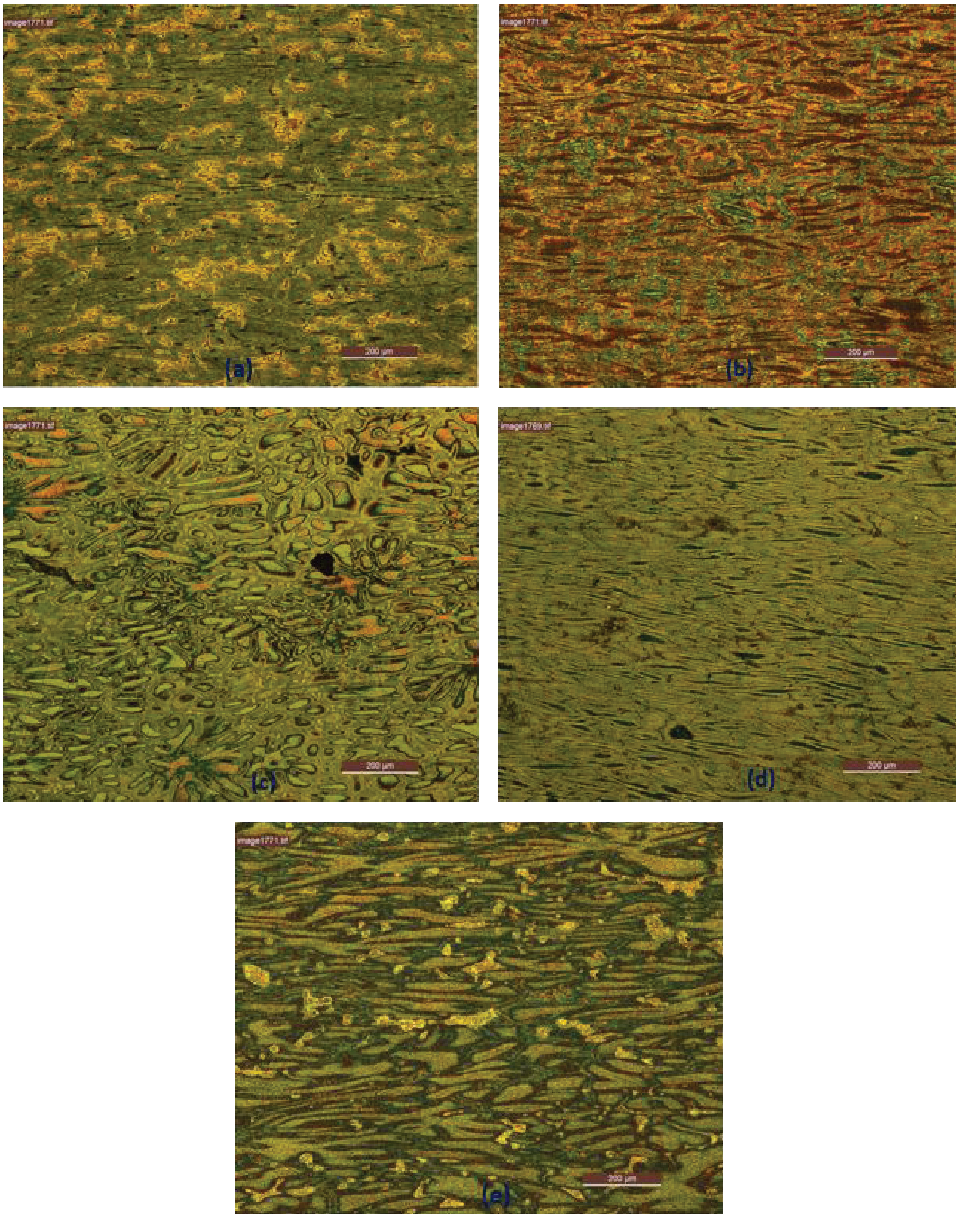

Figure 6. (a) as-deformed microstructure of the Al alloy taken at sample centre, (b) as-deformed microstructure of the Al based composite reinforced with 4 wt.% Cu-18Zn-7Al-0.3Ni taken at sample centre, (c) as-deformed microstructure of the Al based composite reinforced with 6 wt.% Cu-18Zn-7Al-0.3Ni taken at sample centre, (d) as-deformed microstructure of the Al based composite reinforced with 8 wt.% Cu-18Zn-7Al-0.3Ni taken at sample centre, and (e) as-deformed microstructure of the Al based composite reinforced with 8 wt.% SiC taken at samples centre.

to higher thermal conductivity and damping capacity of the Cu-18Zn-7Al-0.3Ni particles over SiC.

- The flow stress and hardness of the AMCs reinforced with Cu-18Zn-7Al-0.3Ni particles were generally higher than that of the unreinforced Al alloy and the Al alloy reinforced with SiC. It is also noted that the flow stress, and to a large extent the hardness, increased with increase in the weight percent of Cu-18Zn-7Al-0.3Ni particles in the AMC.
- The improved flow stress and hardness in the AMCs reinforced with Cu-18Zn-7Al-0.3Ni alloy particles over that reinforced with SiC was ascribed to the combination of improved matrix grain refinement strengthening, interfacial strengthening, compressive residual stresses, high thermal conductivity, and damping capacity offered by the Cu-18Zn-7Al-0.3Ni alloy.
- Strain inhomogeneity which is characteristic of hot deformed metallic based materials were also observed in the AMCs, as the deformation

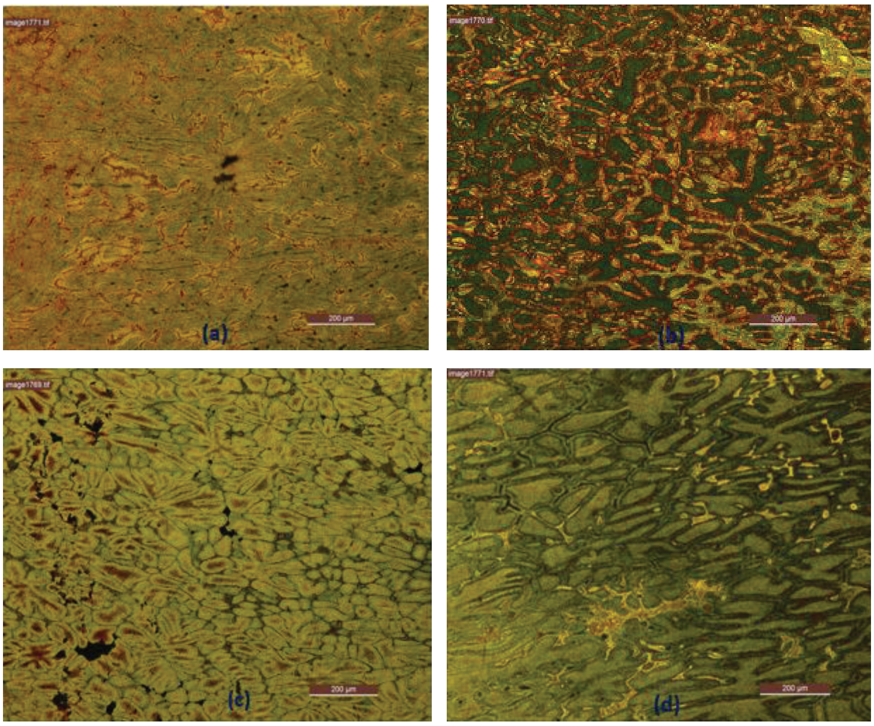

Figure 7. (a) as-deformed microstructure of the Al alloy taken at edge of the sample, (b) as-deformed microstructure of the Al based composite reinforced with 4 wt.% Cu-18Zn-7Al-0.3Ni taken at edge of the sample, (c) as-deformed microstructure of the Al based composite reinforced with 8 wt.% Cu-18Zn-7Al-0.3Ni taken at edge of the sample, and (d) as-deformed microstructure of the Al based composite reinforced with 8 wt.% SiC taken at edge of the sample.

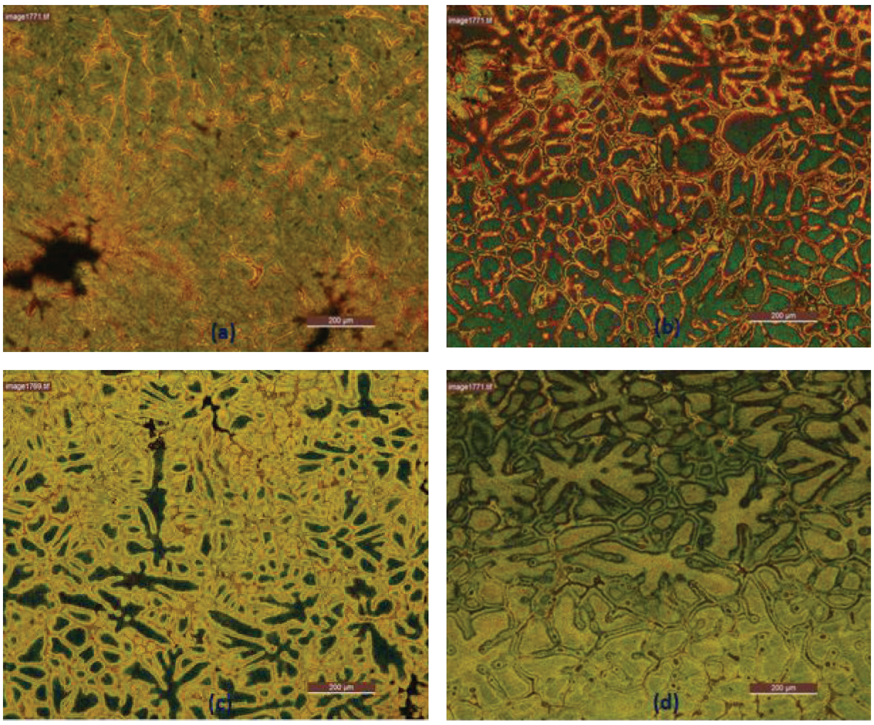

Figure 8. (a) as-deformed microstructure of the Al alloy taken at edge length of the sample, (b) as-deformed microstructure of the Al based composite reinforced with 4 wt.% Cu-18Zn-7Al-0.3Ni alloy taken at edge length of the sample, (c) as-deformed microstructure of the Al based composite reinforced with 8 wt.% Cu-18Zn-7Al-0.3Ni alloy taken at edge length of the sample, and (d) as-deformed microstructure of the Al based composite reinforced with 8 wt.% SiC taken at edge length of the sample.

bands were more conspicuous at the centre of the samples where grain elongation perpendicular to the loading direction are visible compared to the edges and edge length where the grains appear close to equiaxed in shape.
- The Cu-18Zn-7Al-0.3Ni SMA particles were established to be suitable in improving the high temperature strength of Al based composites.

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