

# Experimental Study of Ageing Behaviour of Al-Cu-Mg/Bagasse Ash Particulate Composites

*The experimental correlation of ageing behaviour for Al-Cu-Mg/Bagasse ash particulate composites with 2-10wt% bagasse ash particles produced by double stir-casting method has been investigated. Hardness values measurement and microstructural analysis were used in determining the ageing behaviour, after solution and age-hardened heat-treatment. For comparison, the ageing characteristics of the unreinforced matrix alloy with an identical processing and ageing history were also examined. The results indicate that the composite exhibits an accelerated hardening response compared to the unreinforced matrix alloy at the three selected aging temperatures of 100, 200 and 300 °C. Ageing temperature has great influence on the hardening characteristics of the Al-Cu-Mg/BAP composite. TEM observations reveal that the addition of bagasse ash particles to the Al-Cu-Mg alloy can speed up the growth rate of precipitates S' (Cu<sub>3</sub>Al<sub>2</sub>, and Al<sub>6</sub>CuMg<sub>4</sub>) phases. The accelerated precipitation of S' phases is proposed to be responsible for the enhanced age-hardening of the Al-Cu-Mg/BAP composites.*

**Keywords:** Aluminum matrix composite; Al-Cu-Mg alloy Age hardening; Bagasse ash, Precipitation; Microstructure.

## 1. INTRODUCTION

The development of metal matrix composites (MMCs) is of great interest in industrial applications for lighter materials with high specific strength, stiffness and heat resistance.

They form a new class of industrial materials [1]. In MMCs, aluminium-matrix composites (MMCs) reinforced with discontinuous reinforcements are very attractive because they give the best combination of strength, ductility and toughness and they can be processed by conventional methods such as casting, rolling, forging, extrusion and as a final process, machining [1-3].

Recently, there has been an increasing interest in composites containing low density and low cost reinforcements [4-5]. Among various discontinuous

dispersions used bagasse ash has been found to be one of the most inexpensive and low density reinforcement available in large quantities as solid waste from the sugar processing mill [3, 6]. Hence, composites with bagasse ash as reinforcement are likely to overcome the cost barrier for wide spread applications in automotive and small engine applications. It is therefore expected that the incorporation of bagasse ash particles in aluminium alloy will promote yet another use of this low-cost waste by-product and, at the same time, have the potential for conserving energy-intensive aluminium and thereby, reducing the cost of aluminium products[3, 6].

The age hardening characteristics of an alloy are generally modified by the introduction of reinforcement. These modifications are due to the manufacturing process, the reactivity between the reinforcement and the matrix, the size, morphology and volume fraction of the reinforcement. Strength increment due to ageing is necessary in aluminum alloys because it helps to develop acceptable mechanical properties [1].

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The earlier works [7-11] concluded that the addition of discontinuous ceramic particles into aluminium matrix resulted in the dislocation generation leading to different ageing kinetics compared to monolithic alloys. Thus, there is a complexity involved in the ageing process of composites when compared with that of unreinforced alloys [8]. Hence, an attempt has been made in the present investigation to study and systematically record the effects of heat-treatment parameters on hardness values of Al-Cu-Mg/Bap composite.

## 2. MATERIALS AND EXPERIMENTAL PROCEDURE

The Bagasse ash used in these study were characterized and the results shown in Table 1. Composites used in this study were A2009Al-Bagasse ash particles composites containing 2-10 Wt% Bagasse ash particles. The samples were produced using the double stir casting method [1, 3]

by keeping the percentage of copper and magnesium constant (3.7wt%Cu and 1.4wt%Mg) according to the recommended standard to produced alloy of type A2009 [3, 6] with 2-10Wt% Bagasse ash of particles size of 64µm. A control sample without the Bagasse ash was also produced with this method. After casting, the specimen was machined into hardness coupons for the purpose of determining the thermal ageing behaviour of the produced composites.

The test coupons were polished at both the ends. The test samples were solution heat-treated at temperature of 500°C in an electrically heated furnace, soaked for 3 hours at this temperature and then rapidly quenched in water. Thermal ageing of the quenched samples were carried out at temperatures of 100, 200 and 300°C, for various ageing times until the peak ageing is exceeded [3]. The ageing characteristic of these grades of composites was evaluated using hardness values obtained from age-hardening samples.

**Table 1. Composition and properties of Bagasse ash particles [6]**

Constituent Formula	Cliftonite,(C), Quartz (SiO <sub>2</sub> ), Moissanite(SiC), Titanium Oxide(Ti6O)
Density and phase	1.95g/cm <sup>3</sup> and Solid
Refractoriness	1600°C
Appearance(color)	Black-Odorless powder
Size	64µm
Hardness values	75.05 HRB

The various diffraction peaks, inter-planar distance and phases in the alloy and composites with 8wt% bagasse ash particles were determined using a Bruker D8 θ-θX-ray diffractometer [3].

The microstructure and the chemical compositions of the phases present in the test samples were studied using a JOEL JSM 5900LV Scanning Electron Microscope equipped with an Oxford INCATM Energy Dispersive Spectroscopy (EDS) system. The polished samples were firmly held on the sample holder using a double sided carbon tape before putting them inside the sample chamber. The SEM was operated at an accelerating voltage of 5 to 20 kV. TEM observation of the aged samples was performed at 200kV using a Jeol JEM-2000FX. The hardness values of both as-cast and thermally age-hardened samples were determined according to the provisions in ASTM E18-79 using the Rockwell hardness tester on “B” scale (Frank Welltest Rockwell Hardness Tester, model 38506)

with 1.56mm steel ball indenter, minor load of 10kg, major load of 100kg and hardness value of 101.2HRB as the standard block[1, 8].

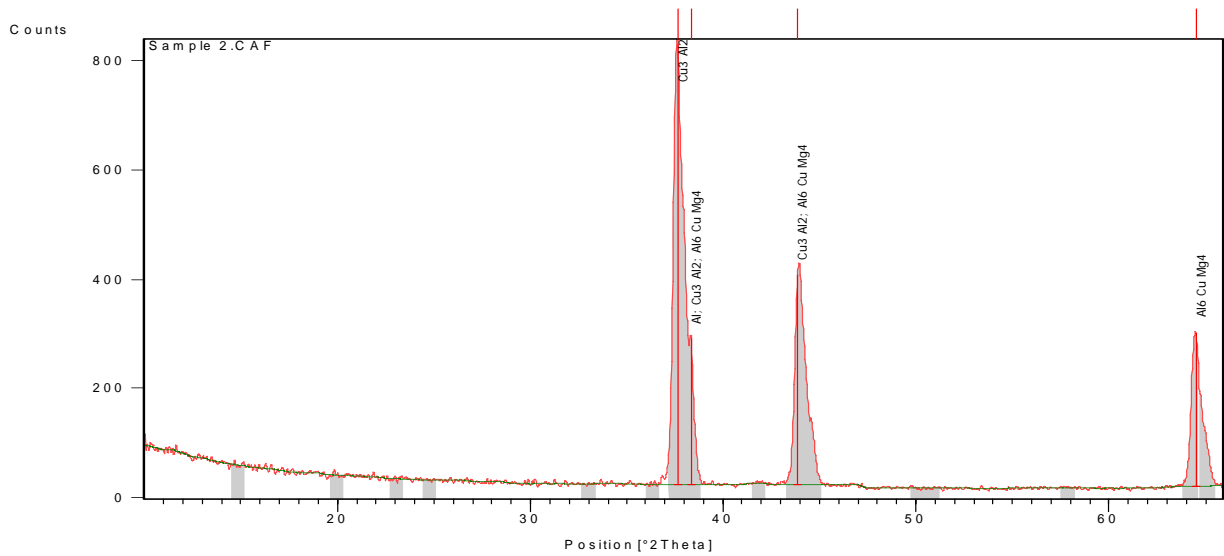
## 3. CONCLUSION RESULTS AND DISCUSSION

The XRD pattern of the unreinforced Al-Cu-Mg alloy(A2009) and Al-Cu-Mg alloy reinforced with 8wt% bagasse ash composite manufactured by double stir casting method matrix are shown in (Figures 1-2).

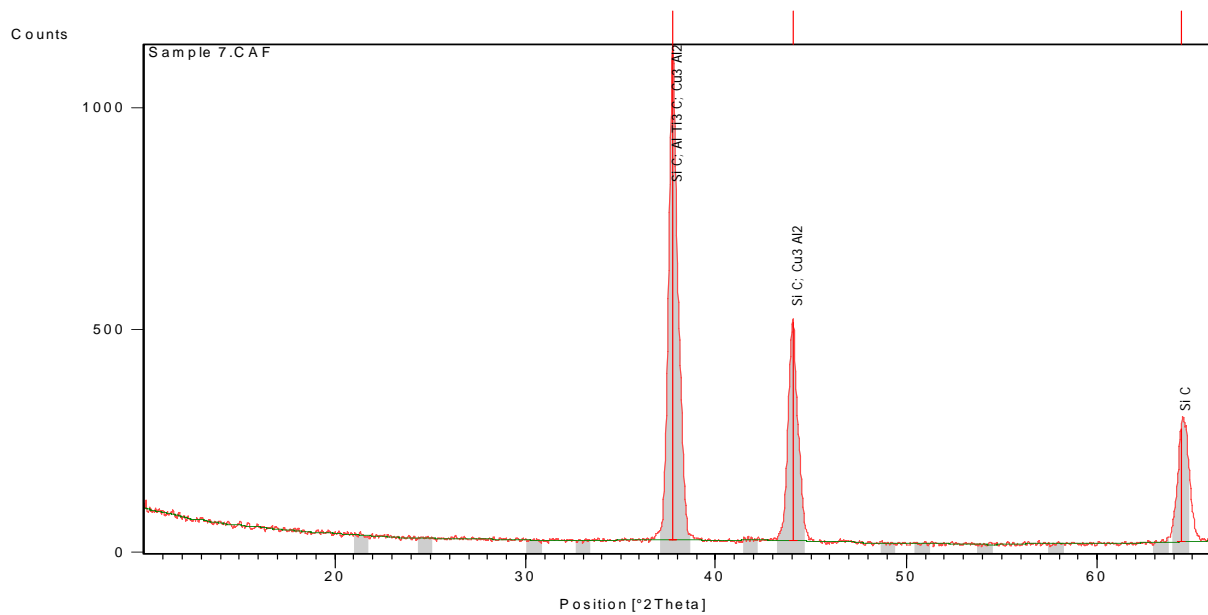
From the Figures 1-2 it was observed that, the major diffraction peaks of the unreinforced alloy are 37.6881°, 43.8769° and 64.4819° and their inter-planar distance, 2.38685Å, 2.06348Å and 1.44391Å, phases at these peaks are Aluminium (α-Al), Aluminum Copper phase(Cu<sub>3</sub>Al<sub>2</sub>) and Aluminum Copper Magnesium phase(Al<sub>6</sub>CuMg<sub>4</sub>).

The composite show three principal diffraction intensity of  $2\theta=37.7722^\circ$ ,  $44.0750^\circ$  and  $64.3863^\circ$  and their inter-planar distance are  $2.38173\text{\AA}$ ,  $2.05466\text{\AA}$  and  $1.44582\text{\AA}$ , phases at these peaks are silicon carbide (SiC), Aluminum Titanium Carbide phase( $\text{AlTi}_3\text{C}$ ) and Aluminum Copper phase( $\text{Cu}_3\text{Al}_2$ ).

In these diffractograms, one can evidently deduce the crystalline phases of the master alloy from that of the composite material. The X-ray patterns show the  $\alpha$ -aluminum, copper and magnesium presence on the matrix alloy Al-Cu-Mg and evident of the bagasse ash particles in the composite.



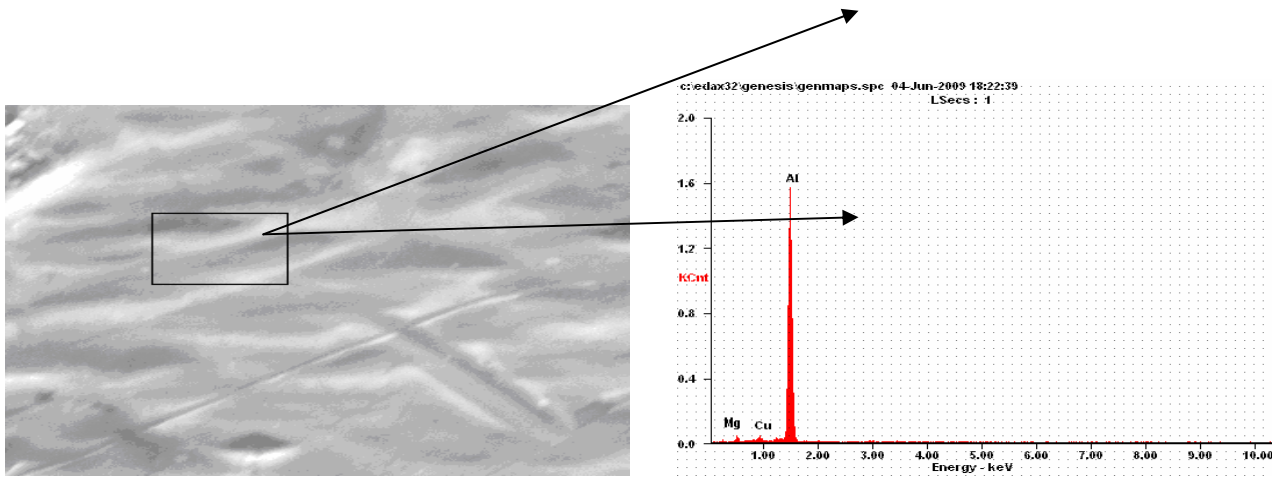
**Figure 1.** XRD pattern of the aluminium alloy (A2009)



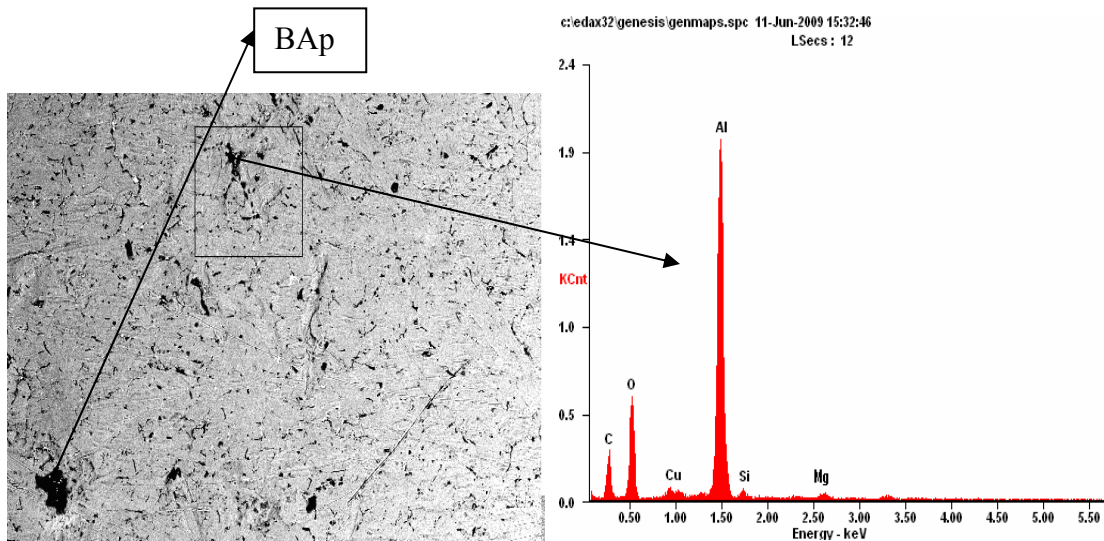
**Figure 2.** XRD pattern of the aluminium alloy (A2009) reinforced with 8wt% bagasse ash

The microstructure of the as-cast of the master alloy and that of the reinforced alloy with bagasse ash at 2wt% and 8wt% were analyzed using Scanning Electron Microscopy (SEM)/Energy Dispersive Spectrometer (EDS). The microstructure of the as-cast unreinforced alloy is shown in Micrograph 1. The structure reveals the eutectic phase containing  $\text{Cu}_3\text{Al}_2$ , and  $\text{Al}_6\text{CuMg}_4$  in  $\alpha$ -aluminium matrix (see Figure 1.).

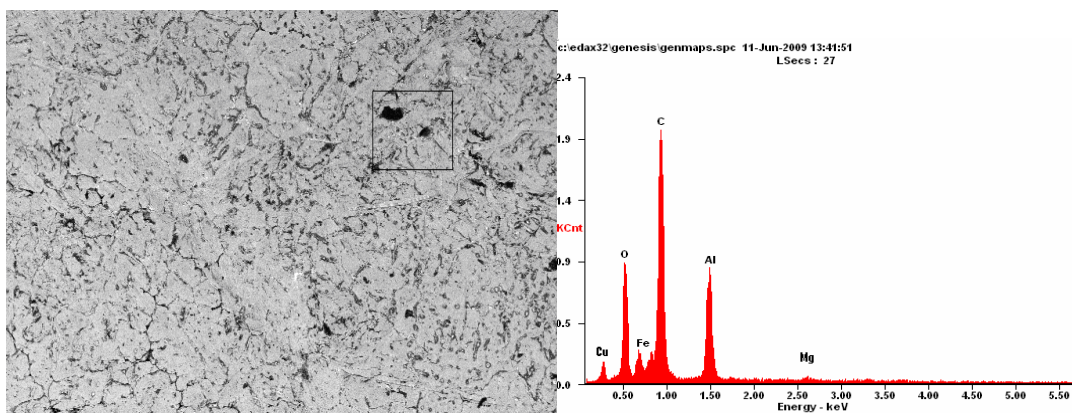
Micrographs 2-3. Show the microstructure of the as-cast reinforced alloy with bagasse ash particle additions at 2wt% and 8wt%. The microstructure reveals that there are small discontinuities and a reasonably uniform distribution of bagasse ash particles in the metal matrix. The ceramic phase is shown as dark phase, while the metal phase is white (see Micrographs 2-3).



**Micrograph 1:** of SEM Microstructure of the unreinforced Al-Cu-Mg alloy/EDS spectrum(x400). The structure reveals the presence of  $\text{Cu}_3\text{Al}_2$ ,  $\text{Al}_6\text{CuMg}_4$  phases in  $\alpha$ -Al-matrix(white)



**Micrograph 2:** SEM Microstructure of the reinforced Al-Cu-Mg alloy with 2wt% Bagasse ash/EDS spectrum) (x400). The structure reveals the dissolution of the  $\text{Cu}_3\text{Al}_2$ ,  $\text{Al}_6\text{CuMg}_4$  phases and slight distribution of bagasse ash(black) in  $\alpha$ -Almatrix(white) matrix



**Micrograph 3:** SEM Microstructure of the reinforced Al-Cu-Mg alloy with 8wt% Bagasse ash/EDS spectrum(x400). The structure reveals the dissolution of the  $\text{Cu}_3\text{Al}_2$ ,  $\text{Al}_6\text{CuMg}_4$  phases and uniform distribution of bagasse ash(black) in  $\alpha$ -Al matrix(white)

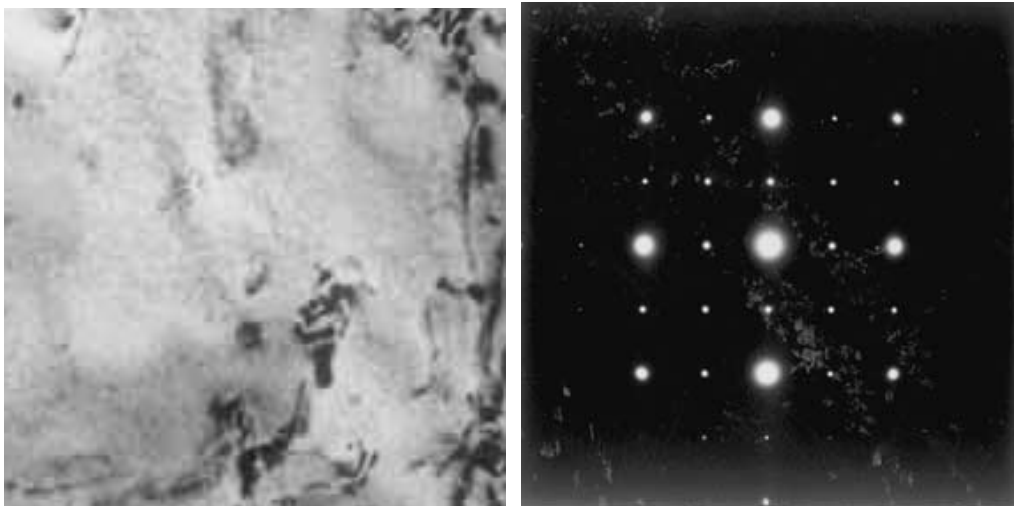
The TEM investigation was carried out on samples that were solution heat treated and water quenched aged artificially at 200°C for peak ageing time for A2009 alloy and peak aged and over-aged for reinforced alloy at 8wt% bagasse ash. After peak ageing S' precipitates were detected in samples of the monolithic A2009 alloy and the reinforced alloy at 8wt% bagasse ash.

Micrograph 4. show the bright field (BF) image of S' precipitates observed in the matrix of the A2009 alloy. Generally, the S' precipitates was distributed in the matrix at the grain boundaries and in the vicinity of the bagasse ash particles. The BF images of S' precipitates observed in the reinforced alloy at 8wt% bagasse ash are shown in Micrograph 5. There corresponding selected diffraction pattern (SADPs) in the  $[112]_{\text{matrix}}$  direction is also shown. The S' precipitates in the sample of peak aged of the composite generally gave stronger diffraction than the one observed in the samples of the

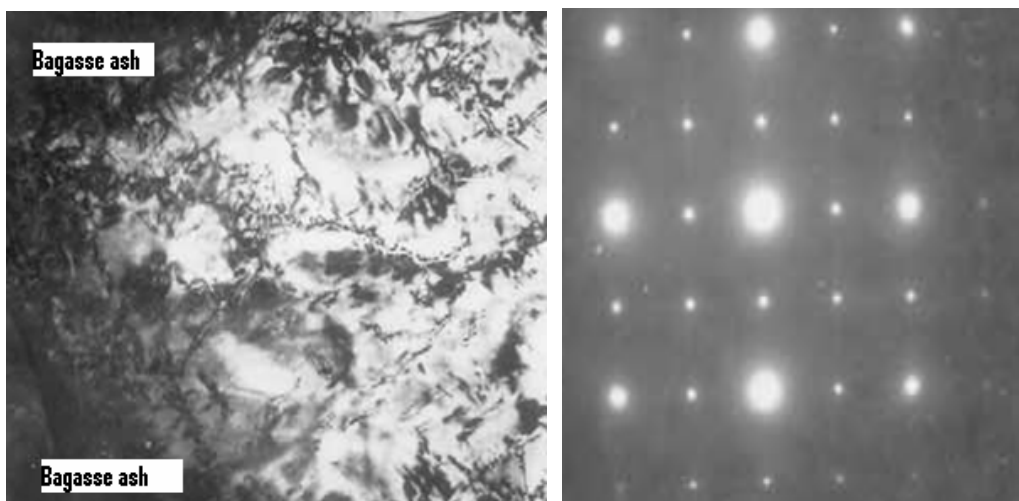
unreinforced alloy given the same level of heat treatment.

The TEM observations revealed that the principal strengthening precipitates in the Al-Cu-Mg / Bagasse ash composite and the unreinforced matrix alloy is S' ( $\text{Cu}_3\text{Al}_2$ , and  $\text{Al}_6\text{CuMg}_4$ ) phases. There are no S' precipitates either within grains or at subgrain and grain boundaries in the peak aged samples.

The precipitation of fine needle shape S' precipitates was found in the composite after peak ageing (see Micrograph 5.). Coarser S' precipitates was developed in the composite after over-aged (see Micrograph 6). The TEM results suggest that the precipitation kinetics of S' phase is accelerated in the Al-Cu-Mg/BAP composite and high density of S' precipitates can be observed in the matrix of the composite.



**Micrograph 4.** TEM/ SADP showing S' precipitates in the unreinforced matrix alloy peak aged

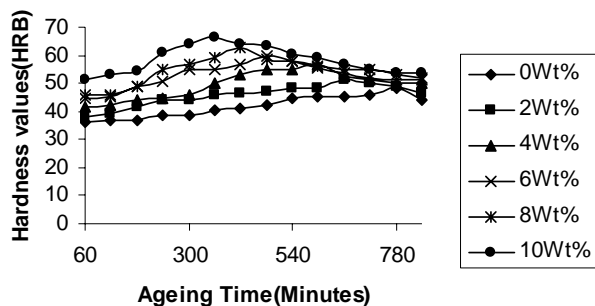


**Micrograph 5.** TEM/ SADP showing S' precipitates in the composite peak aged



**Micrograph 6.** TEM/ SADP showing S' precipitates in the composite over-aged

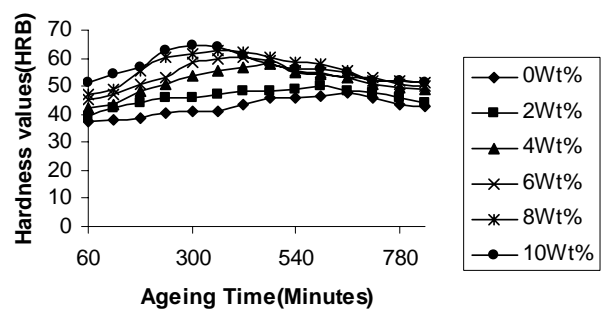
The high density dislocations in the Al- Cu- Mg/BAP composite can provide additional heterogeneous nucleation sites for S' phase, especially in the early stage of ageing. Nucleating of S' phase on dislocations and grain boundaries significantly reduces the critical energy for nucleation, resulting in accelerated precipitation of S' phase. It can be seen that the incubation time for S' nucleation is shorter in the composite than in the matrix alloy at the same ageing temperature and time, indicating the faster precipitation of S' phase in the composite, as a result of higher density dislocations. This precipitation form may depend on [3, 12]: (i) the extra **interfacial area** - and hence energy - between precipitate and matrix; (ii) the possible creation of an **anti-phase boundary** (APB) within an ordered precipitate and (iii) the change in separation distance between dissociated dislocations due to different stacking fault energies of matrix and particles. Similar behaviour of increased precipitation at interfaces was reported by Hassan et al [8] for age-hardened aluminum alloy reinforced with silicon carbide particle.



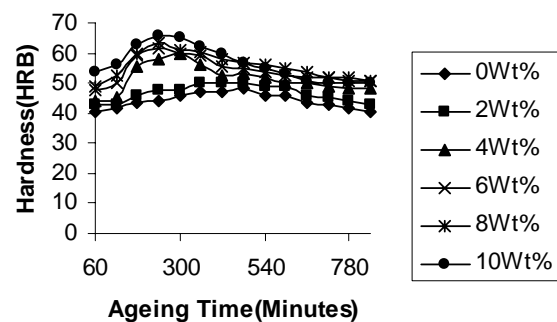
**Figure 3.** Variation of the Hardness values with Ageing Time at Ageing Temperature of 100°C

They attributed this to increase in dislocation density at interfaces. Increase in dislocation density

strain hardens the metal-matrix locally and provides heterogeneous nucleation sites for precipitation, thereby accelerating the ageing response [9]. The results obtained for the ageing behaviour of these composites are presented in Figures 3-5.



**Figure 4.** Variation of the Hardness values with Ageing Time at Ageing Temperature of 200°C



**Figure 5.** Variation of the Hardness values with Ageing Time at Ageing Temperature of 300°C

From Figures 3-5, it can be seen that there is step rise in hardness values of each grade of the composite at initial stages for all ageing temperatures and then fell after reaching the various peak ageing time, corresponding to over ageing. However, at higher ageing temperature the materials developed peak hardness at shorter ageing

time, because the rate of precipitation of the second phase materials is faster and hence increases in hardness values. The time to obtain peak hardness decreases when temperature increases (see Figures 3-5).

The thermal age hardening behavior of the Al-Cu-Mg/BAp particulates composites are similar to Al-Cu/SiC particulates as reported by Suresh et al[9] i.e. hardness continuously increases to a maximum during thermal ageing and then decreases later due to over ageing. It is interesting to note that in the reinforced aluminium alloy metal-matrix, as the volume fraction of bagasse ash particle increase to 10wt% in the aluminium alloy, there is a monotonic reduction in the time required to reach peak hardness (see Figure 5).

The 10Wt%BAP addition yielded the highest hardness value. As far as hardening behavior of the composites is concerned, particle addition in the matrix alloy increases the strain energy in the periphery of the particles in the matrix and the formation of the dislocation at the boundary of the ceramic particles by the difference in the thermo-expansion coefficient between the matrix and ceramic particles during solution treatment and quenching since a lot of dislocations generate in the main matrix/particle interface (see Micrographs 4-6) [9-10]. Thus, dislocations cause the hardness increase in composite as well as residual stress increase because of acting as non-uniform nucleation sites in the interface following the age treatment. It is thought that the higher the amount of the ceramic particles in the matrix, the higher the density of the dislocation, and as a result, the higher the hardness of the composite [8, 11, 12].

The hardness variation of the precipitation hardened alloys during ageing after the solution treatment is a result of nucleation and growth of precipitates. Similarly, the hardness variation of bagasse ash reinforced precipitation hardened aluminum alloy composite reflects the evolution of precipitation in the composite. From the age-hardening results in Figures 3-5, it is concluded that the Al-Cu-Mg composite shows a faster hardening response than the unreinforced matrix alloy at all ageing temperatures and time, which indicates the enhancement of aging kinetics, resulting from the introduction of bagasse ash particles. On the other hand, the TEM investigation demonstrates that the incorporation of bagasse ash particles does not substantially alter the precipitating phases in the Al-Cu-Mg alloy, but accelerates the nucleation or growth rate of the precipitates during ageing.

Therefore, the faster nucleation or growth of strengthening precipitates would be responsible for the higher age-hardening kinetics observed in the Al-Cu-Mg/BAP composite.

#### 4. CONCLUSIONS

From the investigation the following conclusions can be made:

1. Age-hardening has been observed in Al-Cu-Mg/BAP composites, and the ageing kinetics, in terms of time to peak hardness, is accelerated in the presence of bagasse ash particles.
2. The hardening response of the Al-Cu-Mg/BAP composites is sensitive to the artificial ageing temperature. The higher the ageing temperature, the shorter the peak aging time.
3. The addition of bagasse ash particles does not alter the principal precipitating phases of the Al-Cu-Mg alloy. Compared to the unreinforced matrix alloy having the identical ageing history, the growth rate of phase is higher and the incubation time for S' precipitation is shorter in the composite.

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