JEMMME (Journal of Energy, Mechanical, Material, and Manufacturing Engineering)
Vol.6, No. 2, 2022

ISSN 2541-6332 | e-ISSN 2548-4281
Journal homepage: http://ejournal.umm.ac.id/index.php/JEMMME

Microstructure and Mechanical Properties of a Partially Recrystallized Aluminium Alloy having Varying Cu/Mg Ratio

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Abstract

Heat treatable Al-Zn-Mg-Cu alloys are widely used in automobile industries, aerospace, and military applications. This study investigates the effects of different amounts of copper and magnesium on microstructure and mechanical properties. The as-cast alloys prepared in a permanent mold showed a dendritic microstructure and the intermetallic phase surrounded by secondary phases. As-cast microstructure was refined substantially during hot rolling processes. The ultimate tensile strength and hardness values both in hot-rolled and aged conditions along longitudinal and transverse directions were found greater for the alloy containing 1.33 wt.% copper and 1.01 wt.% magnesium, whereas strain to fracture values for alloy 01 with 1.09 wt.% copper and 1.8 wt.% magnesium. The fracture surface of the tensile sample having relatively lower amount of copper content revealed dimple rupture behavior, while higher 4.32 wt.% copper content indicated trans granular and cleavage fracture. A similar pattern was also observed along the transverse direction. Overall, copper appeared to be more effective in strengthening of the studied alloys.

Keywords*: Aluminum Alloys; 7xxx Series; Heat Treatment; CALPHAD; Fractography*

1. INTRODUCTION

Al-Zn-Mg-Cu alloys are a favoured system as a structural material for using in automotive, aerospace and military applications owing to their lightweight [1]. Over the decades, it has been known reducing weight could lead to substantial environment-friendly component due to reduced fuel consumption. Al-Zn-Mg-Cu alloy system have shown greater strength with profoundly added zinc in the system [2]. With 6.28 wt.% zinc, retrogression and re-ageing heat treatment process can successfully improve strength [3]. The precipitation reactions recognize primarily the formation and dissolution of precipitates: supersaturated solid solution (α) → coherent GP zones → semi-coherent η′ → incoherent equilibrium η [4]. Major alloying elements paying role in controlling behaviour of these alloys are zinc, magnesium, and copper. If more zinc and magnesium are added, strength can be moderately improved [5]. Addition of copper increases the amount and number of secondary phases, and at the same time improves strength [6]. High amount of zinc also perturbs the system by changing precipitation kinetics and strength can be modified by altering ageing treatments [7]. The stable precipitate in the Al-Zn-Mg-Cu alloy system is η ($MgZn_2$) [8]. There is still a lack of understanding about the behaviour of copper and magnesium altogether in this system. A good effort was obtained at a solution temperature 475 °C [9], demonstrating dominating participant is magnesium.

In the current work, a set of hot-rolled and treated alloys with different magnesium and copper ratios are studied for understanding controlling-parameters for microstructural and mechanical properties. More specifically, this work investigates the effects copper (1—4 wt.%) and magnesium (0.98—1.8 wt.%) on the microstructure and mechanical properties in as-rolled and aged conditions along longitudinal as well as traverse directions.

2. METHODS

Alloys of three different compositions were prepared by casting in a permanent metal mould. The major focus was obtaining a different copper to magnesium ratio. The ratio was planned to vary apparently from 0.6 up to 4.5. The dimensions of casting are about 215.9×63.5×50.8 mm. Chemical composition of the alloys was determined in an Optical Emission Spectroscope (Shimadzu PDA-7000), and also by appropriate wet chemical analysis method. The obtained chemical composition of the alloys is given in Table 1.

Table 1. Chemical composition of the experiment alloys (wt. %)

Name	Cu	Mg	Zn	Cu/Mg
Alloy 1	1.09	1.8	3.67	**0.61**
Alloy 2	1.33	1.01	4.24	**1.32**
Alloy 3	4.32	0.98	4.36	**4.41**

(Other elements: Fe<2.8, Si<0.95, Mn<0.05, Ti<0.015, balance Al)

As-cast alloys were homogenised at 400 °C and hold for 3 hr, and then quenched into water, prior to rolling and ageing at different temperatures.

The hot rolling was carried out after keeping the samples in a furnace for 1 hr at 500 °C, and then rolled in the rolling mill with equal true strains. Between intermittent passes, re-heating was carried out for 10 minutes.

The microstructure of investigate alloys was observed in a metallurgical microscope - Olympus BH2 – after sample preparation following standard metallographic procedure. The samples were machined from as-cast and rolled alloys, and subsequently ground and polished until mirror-like surface appeared. Subsequently, the samples were etched by Keller reagent. The reagent preferentially reacted with the grain boundaries. Under light microscope observation, this region became dark, and the grain interior regions were white.

The hot rolled sheets were solution treated for 4 hr at approximately 490 °C, followed by water quenching prior to artificial ageing. The hardness measurements were carried out on a Vickers Hardness Tester (model FV-800), with 3 kg_f load and dwell time of 10 s.

The fracture surface of the tensile samples was investigated by scanning electron microscopy (SEM), with an operating voltage of 25 kV. SEM was performed with energy dispersive X-ray analysis system as well. Images were taken both in secondary and backscattered mode since backscattered mode gives better contrast of some phases.
Dog-bone like specimens (approximate dimension 20×6×2.9 mm) were made from both hot rolled sheets and aged sheets. Tensile test was performed in a Universal Testing Machine, along longitudinal (L) and transverse (T) directions of the rolling direction.

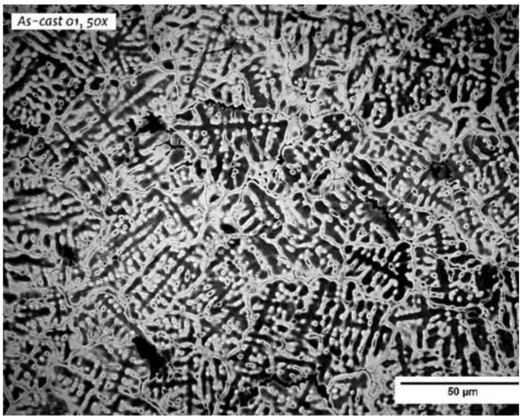
3. RESULT AND DISCUSSION

3.1 AS-Cast Microstructure Evaluation

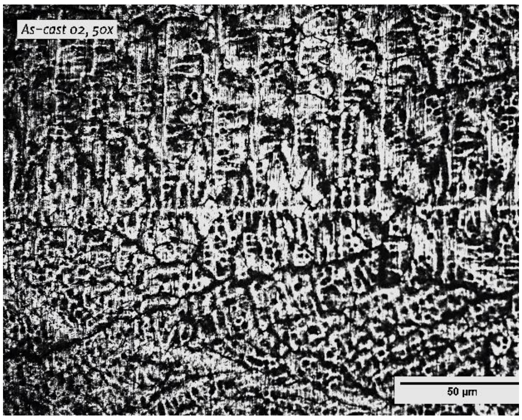
The solidification process occurs after pouring of the liquid metal into the mold. Multi-branched shapes often grow in many crystalline materials. These branches form a geometrical array that are directly related to the structure of the crystal. The branching in crystal leads to a tree-like appearance, defined as dendrites. The shape, size and orientation of the dendrites have profound effects on the properties of as-cast alloys. The presence of secondary phases at the inter-dendritic regions is evident in the micrographs, Figure 1. It is obvious that the structure is severely cored, as the dendrites were bordered by inter-dendritic secondary phases.

The microstructures of the samples are approximately similar in nature – primary aluminum grains (the light areas) and the precipitates (grey). The preferential morphology of primary grains is surrounded by colonies of secondary phases. The secondary phases appeared to be acicular in shape. As such, mechanical properties would not be at a satisfactory level, and industry usage are not appreciable in as-cast conditions.

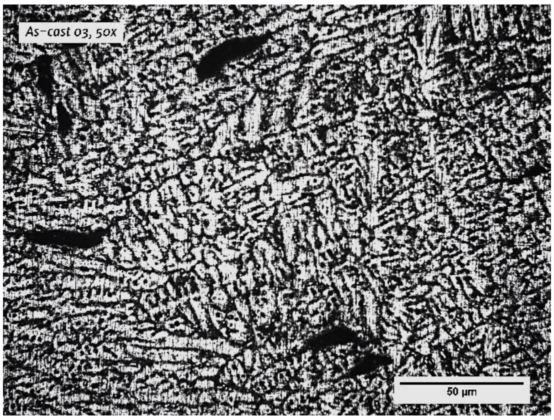
Vol.6, No. 2, 2021 doi: https://doi.org/10.22219/jemmme.v6i2.16587



(a)



(b)



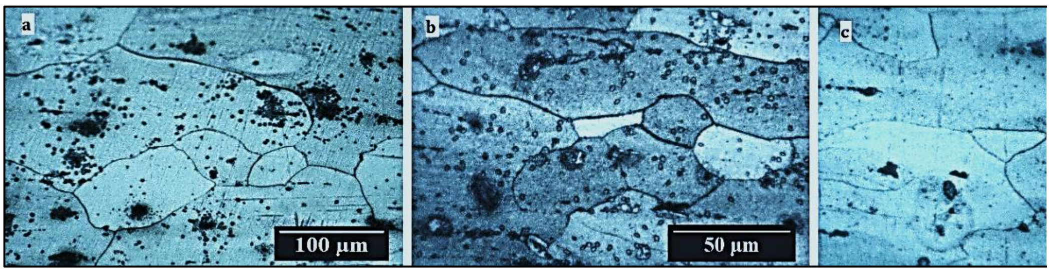
(c)

Figure 1. As-cast microstructure of a) Alloy 1, b) Alloy 2 and c) Alloy 3

3.2 AS-Rolled Microstructure

During hot rolling, the thickness of as-cast plates was reduced from 12 mm to approximately 2.9 mm. The grain shape reformed as an elongated pancake type morphology owing to rolling. Stringers are also formed due to the presence of greater amounts of secondary phases. Both dynamic recovery and recrystallization are expected to occur – as evident from Figure 2. Following recrystallization, nucleation and growth of relatively defect-free grains within deformed grains, where growth occurring through the movement of high-angle grain-boundaries. The formation of new grains in the parent grains were observed in the microstructure of three alloys. The recrystallized grains are distributed along the grain boundaries of parent grains.

Hot rolled alloys comprised of a partially recrystallized grain structure with particles, whereas average grain size was about 15-25 µm.



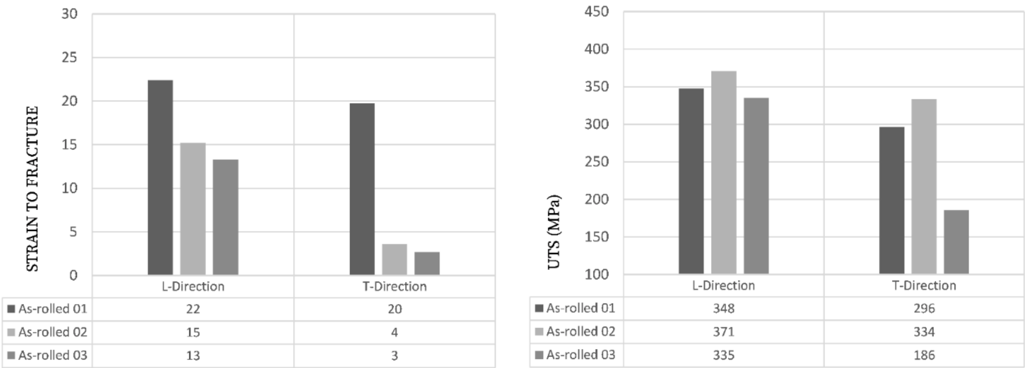
Rolling Direction⟶

Figure 2. Microstructure of (a) alloy 01, (b) alloy 02 and (c) alloy 03 as-rolled condition

As-cast dendritic structure was disintegrated by hot rolling and formed a microstructure with varying grain sizes. During the preheating (between intermittent passes), temperature before rolling high copper content promotes the formation of coarse particles, which act as nucleation sites for particle stimulated nucleation of recrystallization. This is also clear in Figure 2b. More recrystallized grains were found for Alloy 2.

3.3 Rolling Effects on Tensile Properties

During hot rolling, recrystallization is the most powerful tool to achieve grain refinement [10]. For Al-Zn-Mg-Cu alloys, softening of the stress-strain curves detects the recovery period. This is followed by the nucleation of new strain-free grains at grain boundaries. Warmed metal above recrystallize temperature (rolled at 500 °C) passed through two rolled to reduce cross-sectional area with uniform thickness. Hot rolling broke the as-cast microstructure and obliterates the grain boundaries giving rise to form new structure having a set of equiaxed new grains. Elongation of the grains along rolling direction occurred during the hot rolling process. To investigate the hot rolled effect on microstructure, the mechanical properties (tensile strength and per cent elongation to failure) along both longitudinal and transverse directions were analyzed in as rolled and ageing conditions.



(a) *(b)*

Graphic 1. (a) Strain to fracture values, (b) UTS values as-rolled condition

Graphic 1 shows the strain to fracture values of 22, 15, 13 (%) along longitudinal and 20, 4, 3 (%) along transverse directions; the ultimate tensile strength (UTS) 348, 371, 335 MPa along longitudinal and 296, 334, 186 MPa along transverse directions as-rolled condition for alloys 01, 02 and 03, respectively.

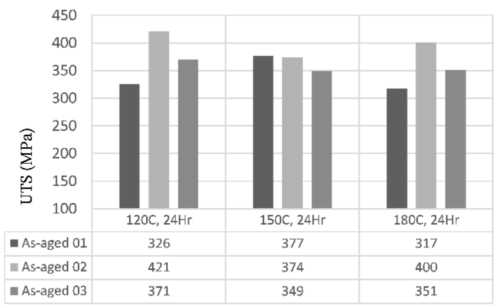
3.4 Ageing Treatment

Solution treatment of the alloys was carried out at 490 °C for 4 hr. The single-phase region was predicted by CALPHAD modelling method using aluminium thermodynamic databases [11]. All solute atoms and phases are supposedly get dissolved to form a single-phase solution. Solution treatment alone lowers the hardness of these alloys. The following water quenching to promote a super saturated solid solution that remained in a metastable state. The lowering of temperatures prevents the diffusion. Finally, the supersaturated solution was heated to an intermediate predetermined temperature for a pre-defined holding time in order to obtain a distribution of secondary phases.

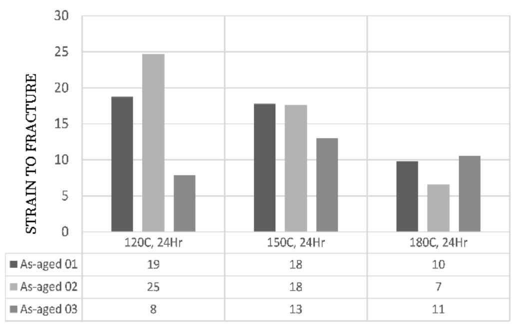
During ageing process, the super-saturated solution obtained by solution treatment and quenching, is heating up to an intermediate temperature so as to induce precipitation and held at that temperature for a specific amount of time, called temperature-time cycles. This ageing cycles induce precipitation of hardening phases that inhibit movement of dislocations. Thus, the inhibition of dislocation movement enhances the mechanical properties – tensile strength and hardness.

Three ageing cycles were performed on alloys 1, 2 and 3. Figure 4 presents three different ageing conditions: (a) aged 120 °C at 24 hr, 150 °C at 24 hr, and 180 °C at 24 hr.

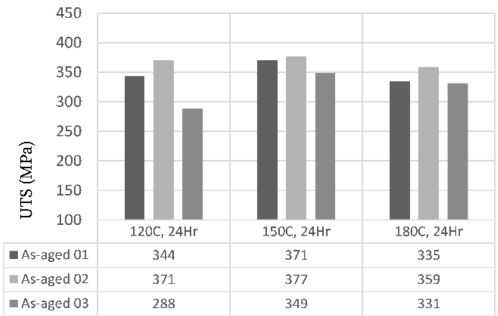
In the first condition, the UTS values 326, 421 and 371 MPa were determined along longitudinal and 344, 371 and 288 MPa were along transverse directions. The strain to fracture values were 19, 25 and 8 % along longitudinal and 18, 8 and 5 % along transverse directions.



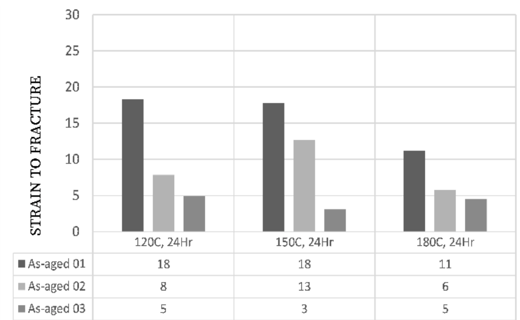
(a) UTS values along L-direction



(b) Strain to fracture values along L-direction



(c) UTS values along T-direction

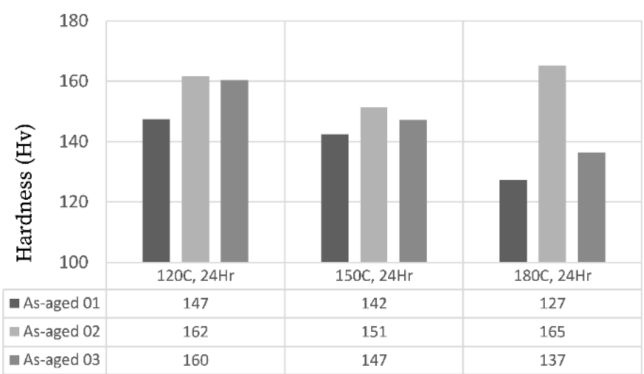


(d) Strain to fracture values along T-direction

Graphic 2. UTS and strain to fracture values along longitudinal and transverse directions as-aged at different temperature-time cycles

In the second condition. Ageing 150 °C at 24 hr cycle, the UTS values 377, 374 and 349 MPa were found along longitudinal, and 371, 377 and 349 MPa along transverse directions. The elongation to fracture values along longitudinal are 18, 18 and 13 % and along transverse directions are 18, 13 and 3 %.

In the final ageing condition, 180 °C at 24 hr cycles, the UTS values are 317, 400 and 351 MPa along longitudinal, and 335, 359 and 331 MPa along transverse directions. The strain to fracture values are 10, 7 and 11 % along longitudinal direction and 11, 6 and 5 % along transverse direction.



Graphic 3. Vickers hardness for alloys 1, 2, and 3 aged at 120 °C /24 hr, 150 °C /24 hr, and 180 °C /24 hr

Figure 5 shows the Vickers hardness aged at 120 °C /24 hr, 150 °C /24 hr and 180 °C/24 hr, among them the better hardness values is 162, 151 and 165 Hv. It is obtained for alloy 2.

In short, strengthening is found to be greater for copper to magnesium ratio higher than 1. The main benefits adding magnesium to aluminum alloys are to increase strength by solution treatment and quenching. Artificial aging further increases in strength, but substantial sacrifice in tensile elongation. The addition of copper and magnesium to aluminum alloys increase in hardness and strength [12]. In any ageing cycle, the strength behavior was supported by more copper and magnesium in the system. Increasing the ageing temperature was not much beneficial. On the other hand, elongation to failure values evidently dipped extensively with higher ageing temperature. This is partly attributed to the coarsening of secondary phases and grain growth.

The observation in the transvers direction followed the similar trend, through the values were much lower compared to those values obtained from the longitudinal direction. The foremost reason for such difference is attributed to the incomplete recrystallization of the grains.

3.5 Fractography Analysis AS-Aged Alloys

The surface of tensile fracture in the samples both longitudinal and transverse directions was characterized using SEM to determine the type of fracture. The fracture surfaces of tensile specimens demonstrate three classic fracture mechanisms. Depending on alloy composition these mechanisms are distributed in various portions of the surfaces– (1) fracture or decohesion of coarse constituent particles (C model), ductile intergranular (or inter-sub-granular) fracture (I model, characterized by relatively smooth surfaces), and ductile trans granular fracture (T model, characterized by dimples) [13]. Figures 6 to 8 show the SEM fractography of the alloys. Higher ductile dimple density indicates the better refinement of grains. This higher dimple density along longitudinal direction demonstrates the better elongation to fracture than transverse direction. Figure 6 shows SEM image of alloy 2 aged for 120 °C at 24 hr cycle, the dimple density along longitudinal direction indicates better ductility property (elongation to fracture value 25) than transverse direction.

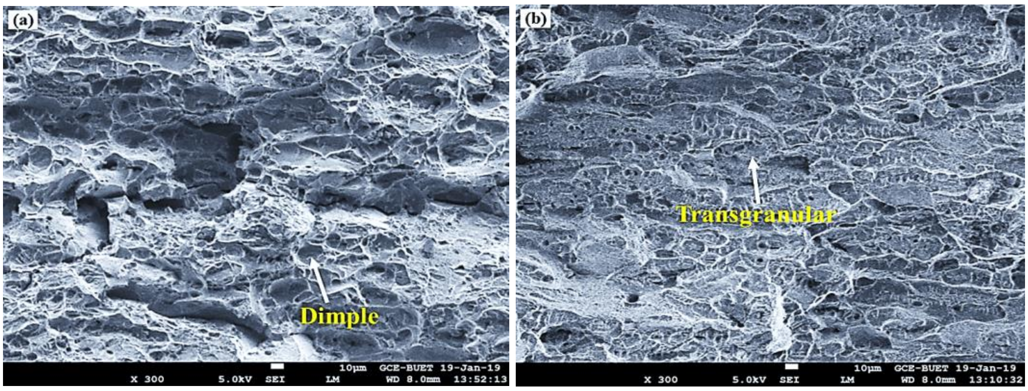


Fig. 6 SEM image of fracture surface along (a) longitudinal-alloy 02, and (b) transverse directions-alloy 02 as-aged at 120^{O}C/24hr

For the aged 150 °C at 24 hr sample, Fig. 7 shows the SEM image of alloy 01 along longitudinal and transverse direction alloy 02. Dimple density is better for alloy 01, stress to facture value along longitudinal direction is 18 %. The cleavage pattern is observed for alloy 02 along transverse direction, whereas strain to fracture value 13 %. These cleavage microstructures present its columnar grain structure of alloy 2.

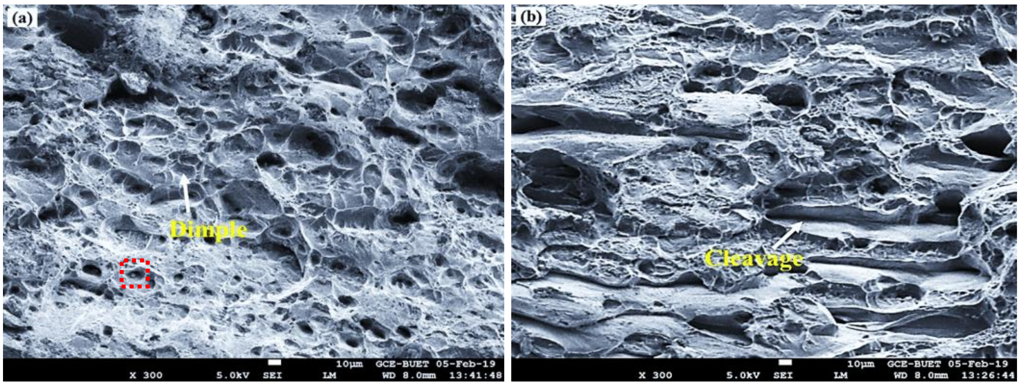


Fig. 7 SEM image of fracture surface along (a) longitudinal- alloy 01, and (b) transverse directions-alloy 02 as-aged at 150^0C/24hr.

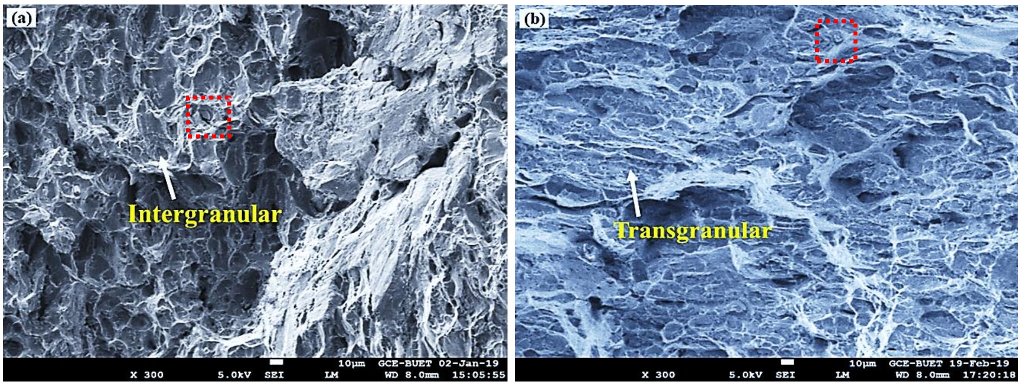


Fig. 8 SEM image of fracture surface along (a) longitudinal- alloy 02, and (b) transverse directions-alloy 02 as-aged at 180^0C/24hr

Figure 8 demonstrates the SEM image of alloy 2 both longitudinal and transverse directions aged for 180 °C at 24 hr cycle. The intergranular pattern is predominant along longitudinal, elongation to fracture drop to 7 % and transgranular microstructure along transverse direction 6 %. Intergranular feature illustrates the coarse grain structure that reduces the strain to fracture value, whereas transgranular microstructure for limited ductility. The dimples were larger in size.

4. CONCLUSION

Tensile properties of Al-Zn-Mg-Cu alloy system was investigated for different copper to magnesium ratios. Properties were studied in both longitudinal and transverse directions. Initial microstructure of the as-cast alloys showed slight difference in dendritic structures due to different ratio of copper to magnesium. Hot rolling and solution treatment accomplished the dissolution of other phases into the matrix at approximately 500 and 490 °C. The ultimate strength values were better when the ratio is greater than 1 (1.32 – copper 1.33 to magnesium 1.01 wt.%). If the alloys contain more copper, the contribution to strength is largely attributed to this element. Apparently, magnesium is not very effective in strengthening for the conditions used in this work. The trend was similar in both longitudinal and transverse directions. The strain to fracture values decreased in both longitudinal and transverse directions as the elevation of ageing temperature.

SEM fractography revealed deep dimples on the specimen surfaces, when particles from the stringers along with elongated grains were pulled out. A large number of dimples and tearing indicates the fracture mechanism was primarily by initiation of coalescence of micro-voids coupled with intergranular fracture. Trans- and inter-granular fractures appeared by greater presence of the second phases. This fracture behaviour is due to the reduction of undissolved coarser phase and the increase of precipitated particles. Coarse dimples were observed at the highest ageing temperature, correlating the coarsening effect of the particles, and hence the drop in strength. Ostensibly, in transverse direction of testing fracture mode did not include any dimple formation.

ACKNOWLEDGEMENT

The authors are grateful to the department of Materials and Metallurgical Engineering, Bangladesh University of Engineering and Technology, for funding and providing laboratory facilities. Materials were used from, fund obtained through HEQEP Sub-Project CP 3117.

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