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RESEARCH PAPER

Effects of Rapid Infrared Heating and Cryogenic Cooling on the Tensile Properties and Fracture Behavior of Al-Cu-Mg

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Abstract: *The objective of this work was to investigate the effect of rapid heating and cryogenic cooling on the fracture and tensile properties of Al-Cu-Mg samples. The specimens were subjected to three different heat treatment cycles in which the Infrared heating (IR) were used as the heating medium at the ageing stage, and the liquid nitrogen and water were used as the quenching mediums. The ageing temperature and time were 190ºC and from 2 - to 10h respectively. The results indicated that by using IR at the ageing stage, the hardening rate enhanced because the rapid heating via this method led to faster diffusion of the alloying elements. Moreover, the high density of nano-sized precipitates formed during ageing was another reason for higher strength and ductility. Cryogenic treatment had a negligible effect on both the microstructure and tensile properties. However there was an improvemnet in the ductility to some extent. Overall, the combination of a high heating rate and cryogenic treatment led to the highest mechanical properties. SEM micrographs of the fracture surface demonstrated that in Cryogenic treatment plus Artificial Ageing (CAA) condition, the surface was fully covered by deep dimples in contrast to the Cryogenic treatment plus Infrared Heating (CIR) and Water-Quench plus Infrared Heating (QIR) conditions which contained shallwer dimples. Some facets were also observed in the latter samples.*

Keywords: *Rapid heating, Cryogenic treatment, Precipitation hardening, Al- Cu-Mg, Tensile properties, Fractography.*

1. INTRODUCTION

The heat-treatable Al–Cu–Mg alloys because of their magnificent properties (high specific strength, good damage tolerance, suitable corrosion resistance, excellent fatigue strength) are the most economical and attractive alloys [1, 2].
The use of component from Al-Cu-Mg alloy instead of heavier materials such as steel or copper is in the automotive industry due to their high strength to weight ratio [2-6].
To achieve optimum mechanical properties, it is essential to control the microstructural evolution during the solution, quenching and precipitation heat treatments. Different heat treatment scenarios have been applied to the allumiunm alloys to achieve desired mechanical properties [7]. Mirzakhani et al. [8] used single and double age hardening on the mechanical properties of Al-3.7Cu-1Mg alloy to increase both strength and elongation and reduce the heat treatment time. A few researchers have studied the effects of rapid heat treatment such as Electromagnetic [9], Electro-pulsing [1, 10, 11], salt bath [12], electric field [13, 14] on the precipitation phenomena and microstructural changes. However, there is not a systematic study concerning the effect of Infrared heating on the ageing time and tensile properties of Al-Cu-Mg.
Infrared heating has many benefits, including remarkably better temperature control and high heating rates, short heat-up times and also high energy efficiency. Without any intermediary, the object is heated by the infrared ray produced from the heat source. Therefore, this paper aims to investigate the effect of a high heating rate (by infrared heating) on the age hardening process, microstructure and mechanical properties of Al-Cu-Mg alloy.

2. EXPERIMENTAL PROCEDURE

In the present research, the commercial Al-Cu-Mg alloy with the chemical composition shown in Table 1 was used in the form of a plate with 10mm thickness. In this paper, the influence of quench medium after solutionizing and high heating rate during precipitation hardening treatment on the microstructure and mechanical properties of the alloys was investigated and compared with conventional solution and age treatments.

Table 1. Chemical composition of Al alloy used in this study.

Element	Al	Cu	Mg	Mn	Zn	Fe	Si
wt. %	92.4	4.9	1.5	0.6	0.2	0.2	0.2

Tensile specimens were fabricated according to ASTM E8 standard with a gauge length of 25 mm. various kinds of heat treatments were applied to the specimens as listed in Table 2. After each of the heat treatments, the microstructural analysis, hardness and tensile test were carried out.

Rockwell B hardness tests were carried out at room temperature on the aged specimens, and the average of five in dentations was only reported. Tensile Tests were conducted at room temperature at a ram speed of 5 $mm.min^{-1}$ by the universal tensile test machine. The stress-strain curves, yield strength, ultimate tensile strength (UTS) and elongation of samples were determined and compared.

Finally, all of the peak-aged specimens were prepared for metallography and SEM examinations. The Keller reagent was used to etch the specimens and reveal their microstructures. The SEM and EDS were used to study precipitates and tensile fracture surface.

3. RESULTS AND DISCUSSION

3.1. Mechanical properties

The hardness data for the samples subjected to CIR, QIR and CAA as a function of ageing time are plotted in Fig. 1. The ageing temperature was 190ºC, as presented in Table 2. The results show that the rapid heating by the infrared system increases the hardness at different ageing times. Besides, Cryogenic quenching has no significant effect on the hardness of materials in comparison with the water quenching. The peak hardness values of CIR and QIR are 76HRB, respectively, which is more than CAA (66 HRB) at the peak-age stage. Also, CIR and QIR reach the maximum value of hardness after ageing for 6 hours, which is remarkable shorter than conventional T6 treatment.

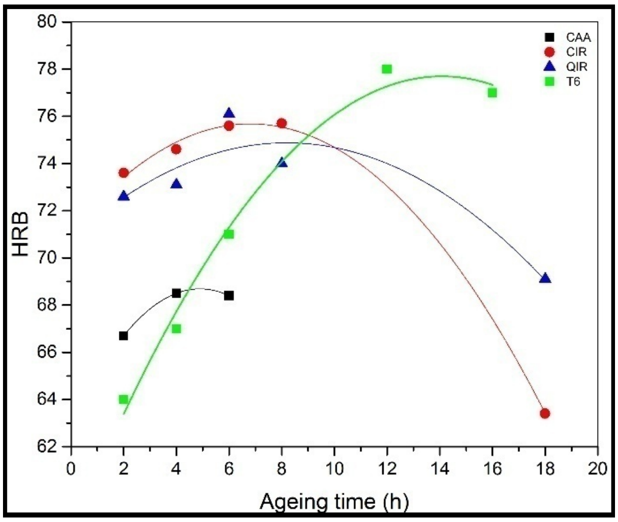

Fig. 1. Hardness vs. ageing time for the alloy subjected to four different heat treatments.

In other words, the high heating rate has enhanced the ageing kinetics. It can be due to that in CIR and QIR conditions the specimens are subjected to high heating rate, which is applied by infrared heating. This high heating rate brings about activation energy for precipitation of secondary phases, which is diffusion-controlled processes. Therefore nucleation of precipitates and their growth are accelerated significantly by high heating input [15, 16]. Meanwhile, during a high heating rate, the density of non-equilibrium vacancies raises, and as a result, the preferred nucleation sites for precipitates are enhanced, leading to the ageing kinetics increases.

Table 2. Three heat treatment cycles of age hardening

Cycle Name	Solutionizing	Quench	Ageing
CIR	490°C for 1 h	Water-Quench Immersion in liquid nitrogen for 30 min	Infrared Heating at 190°C for 2, 4, 6, 8, 18 h
QIR	490°C for 1 h	Water-Quench	Infrared Heating at 190°C for 2, 4, 6, 8, 18 h
CAA	490°C for 1 h	Water-Quench Immersion in liquid nitrogen for 30 min	Artificial Ageing in the oven at 190°C for 2, 4, 6, 8, 18 h
T6	490ºC for 1 h	Water-Quench	Artificial Ageing in the oven at 190ºC for 2,4,6,12,16 h

Overall, it can be concluded that high heating rates result in the generation of different defects in the matrix. Thus, high heating rates play a significant role in the age hardening process of Al-Cu alloy [17]. Indeed, CIR and QIR conditions enhance the diffusion rate of the solute atom through dislocations pipes and vacancies. Therefore, the fine precipitates with more uniform distribution form and consequently peak-age time declines. Whereas the precipitation is a diffusion-controlled process, it is practical to use the following equation:

$$D = D_0 \, . \exp\left(\frac{-Q}{R.T}\right) \quad (1)$$

where D_0, Q, R and T are diffusion constant, activation energy, gas constant and temperature, respectively. The vacancies are essential for the diffusion of matrix and substitutional solute atoms in metals. By using rapid heating, a high amount of energy induced in the material at a short time which increases the vacancy density in comparison with the regular heating rate. According to the above equation, it is reasonable to claim that high heating rates would cause non-equilibrium vacancies in addition to the equilibriumones. Thus, the higher vacancy concentration, the higher the diffusion rate. It means that the activation energy decreases for rapid heating in ageing treatment of QIR and CIR. Razavi et al. [16] proposed that a high heating rate adds another term in Fick's law, which can be employed to explain the higher diffusion rate in QIR and CIR conditions.

Fig. 2 represents the engineering stress-strain curves of the alloy at different heat treatment conditions. The CAA sample has the highest elongation (43%), and the CIR sample has the highest Ultimate Tensile Strength (470 MPa). From the microstructural point of view, Al-Cu-Mg alloy is a heat treatablealloy. Therefore, it can be strengthened through precipitation hardening mechanisim.

The main strengthening phase in Al-Cu-Mg alloys are rod/lath-shaped S-type precipitates, which are formed in the following sequence [18]:

$$\text{SSS} \rightarrow \text{GPB} \rightarrow S'' \rightarrow S' \rightarrow S$$

Because of S-type precipitates features such as arrangement, thickness, diameter, etc., these precipitates are considered as a non-shearable phase [19-22] thus, and the strengthening is the result of the Ashby-Orowan mechanism [23]. The Orowan mechanism stems from the fact that at low temperatures when dislocations meet un-shearable barriers, they cannot pass through the barriers. When the applied stress increases, the shear stress in the slip system raises and dislocations interact with obstacles leading to bowing of dislocations around them and leaving dislocation loops, called Orowan loops. These loops make difficulty for mobile dislocations movement and reduce the average distance between the particles. Also, by forming these loops, the total length of dislocations will enhance, and as a result, the dislocation density will increase [24-28]. The outstanding result observed in Fig. 2 is that CIR and CAA samples have a uniform and non-uniform elongations. It might be deduced that cryogenic cooling applies compressive residual stresses, which decreases the crack propagation rate.

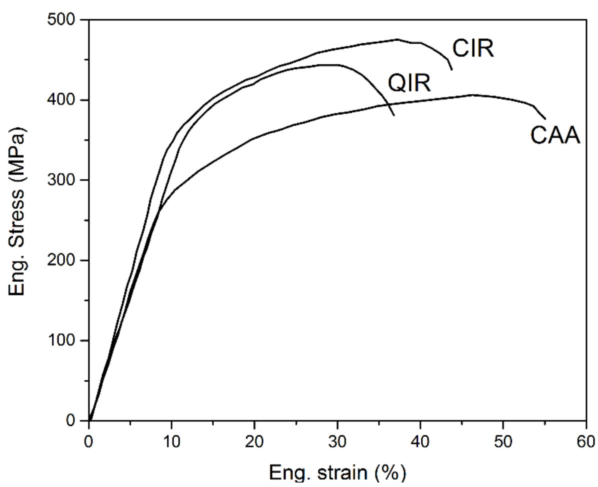

Fig. 2. Engineering Stress-strain curves for different heat treatment conditions.

3.2. Microstructure

Fig. 3 shows the microstructure after CIR, QIR and CAA treatments. The microstructures contain α-Al matrix and secondary particles, distributed within the grains and grain boundaries, which have been formed as a result of different heat treatment conditions. Moreover, all the grains elongated at the rolling direction, and in some cases, abnormal grain growth has happened. The grains size in all three conditions are approximately the same, and there is not a notable difference between them. Therefore, that would be compelling to claim that grain size strengthening (Hall-Pech equation) has a negligible contribution in the increment of the tensile strength.

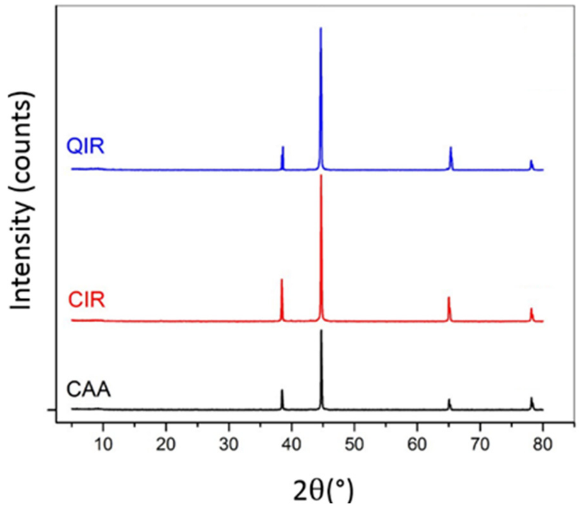

Fig. 3. XRD pattern of CAA (red), QIR (blue) and CIR (grey) at the peak-aged condition in Fig. 1

In Fig. 4, the distribution of intermetallic phases is shown in SEM images. Fig. 4-a shows that in the CAA condition these particles have not been uniformly distributed and they have been formed mostly on the grain boundaries, on the contrary, in the CIR and QIR conditions are distributed more uniform in the interior of the grains. Hence, infrared heating caused in more uniform distribution of intermetallic phases.

Fig.5 shows the XRD patterns of QIR, CIR and CAA age treatments. It is clear that the Al2CuMg phase is the dominant phase which is precipitated during ageing.

The XRD results obtained according to the Joint Committee on Powder Diffraction Standards (JCPDS): 35-0741. However, there are some differences between the intensity of XRD patterns of these three conditions.

The peak intensity in the CIR and QIR conditions is approximately double of the CAA condition, which means that the volume fraction of the S´-phase precipitates in CIR and QIR is higher than CAA. Therefore, the flow stress of samples subjected to rapid ageing and cryogenic cooling is higher than conventional ageing as illustrated in Fig. 4. Because of the more S phases, the less average distance between precipitates and consequently, the more barriers for dislocation motion. Therefore, the strength will increase. That is another reason why CIR and QIR have a higher yield and tensile strength than CAA.

Furthermore, there is no difference between the yield strength of CIR and QIR conditions. In other words, immersion in liquid nitrogen for 30 min has not affected the volume fraction of the second phase particles in both conditions. However, elongation at fracture in CIR is higher than QIR, which will discuss later.

Another point of view is that the high heating rate applies thermal gradient through the thickness of the specimens leading to plastic strain generation. Hence, that would be compelling to say that dislocation density in CIR and QIR is more than CAA. Since S-type precipitates likely to nucleate heterogeneously on dislocations [18, 19, 22, 29, 30], it is reasonable to claim that increasing dislocation density results in increasing the volume fraction of S-type precipitates.

Engineering stress-strain curves in Fig. 4 shows that cryogenic cooling after solutionizing has an impact on the ductility of the material. As it isevident in Fig. 4 the ductility of material after CAA and CIR treatments are greater than QIR. The increase in ductility is attributed to two microstructural changes: (i) the decrease in dislocation density, (ii) the highervolume fraction of nano-sized S´-phase precipitates.

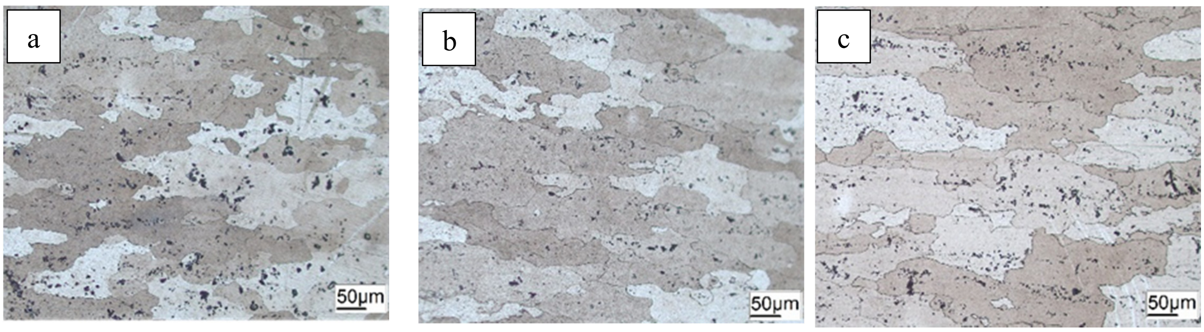

Fig. 4. The optical microstructure for different heat treatment conditions CIR (a), QIR (b), CAA (c).

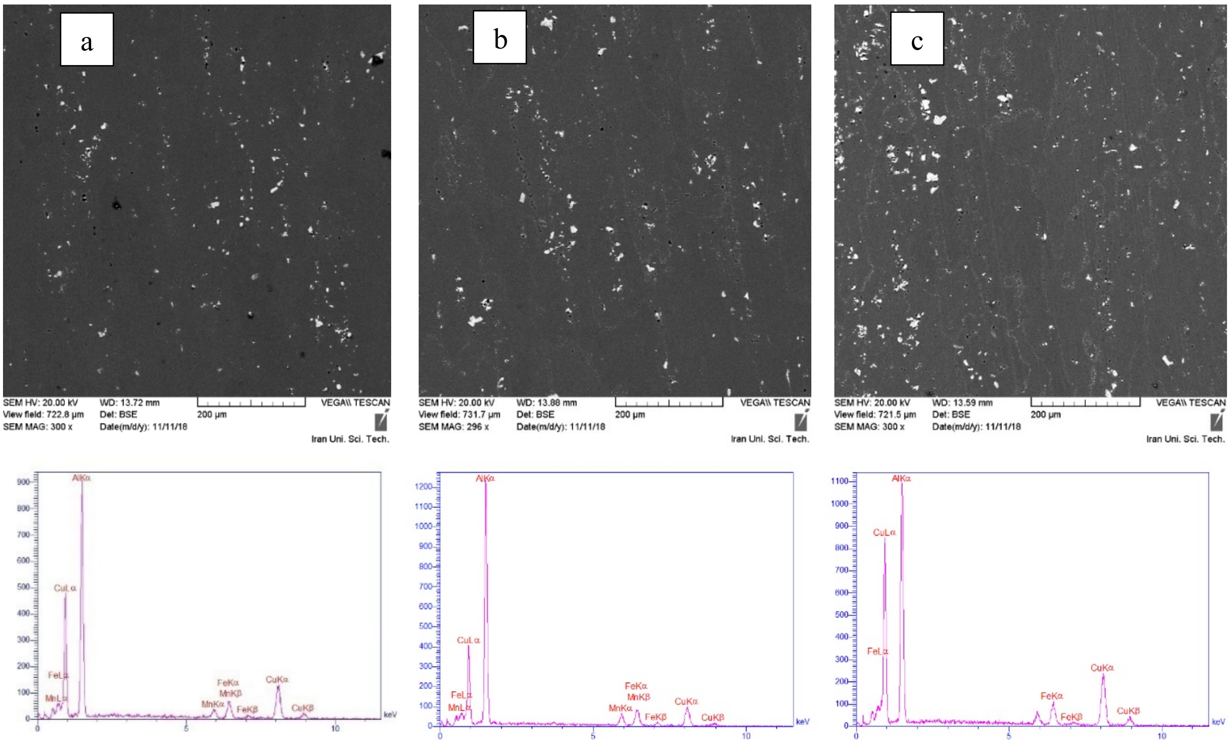

Fig. 5. SEM BSE micrograph and EDS analysis at the peak-aged of CIR (a), QIR (b), CAA (c).

First of all, the reduction in dislocation density after ageing can be considered as a result of the fast recovery process due to rapid heating during ageing providing an excess area for dislocation accumulation before saturation during tensile testing. Moreover, the high density of nano-sized S´-phase precipitates can effectively trap the dislocations formed during tensile testing. Since a high work-hardening rate has an intense effect on the ductility, the above two factors increase the work-hardening rate, which results in incrementing ductility [31-32].

3.3. Fractography

Fig. 6 reveals the SEM fracture micrograph of the CIR, QIR and CAA specimens after tensile tests. It can be seen that the ductile fracture is the dominant fracture mechanism in all three heat treatment conditions as tensile curves in Fig.4 demonstrated. Classical void growth model mainly explains the ductile fracture. This model describes that ductile fracture starts because of voids nucleation. Then voids grow, and at the final stage, they link together resulting in ductile fracture. In all heat treatment conditions, two types of dimples are distinguishable. The First type is coarse dimples attributed to the formation of voids around large particles and cracked-intermetallics. The second type of dimples is small dimples in a honeycomb structure that has the size of about 500 nm. However, there are some differences between the fracture surfaces of CIR, QIR and CAA, which can be directly related to their tensile behaviour. In the CAA condition (Fig.6 (a, b, c)) the entire fracture surface has been covered by dimples representing a complete ductile fracture. Moreover, at the bottom of the dimples, there are cracked-second phases. On the one hand, these cracked-second phases are the result of the crack growth enhancement at the final stage of the crack after dimples' formation. On the other hand, cracked-second phases would cause stress concentration, which results in crack formation. It is worthwhile to note that in the CAA treatment the dimples are deeper in comparison with the other two conditions, which results in more ductility. Fig.6 (d, e, f) illustrates the fracture micrograph in the CIR condition. The shallow dimples demonstrate ductile transgranular fracture that occurred in the interior of the grains. In this condition, voids nucleation has occurred as a result of particle-matrix decohesion (yellow arrows) and represents the highest strength among these conditions. Also, transgranular cracks (red circles) are also observed in the images. SEM images of QIR condition are illustrated in Fig.6 (g, h, i). Red circles show the facets of the fracture surface of tensile specimens. These smooth surfaces and tear ridges indicate the local quasi-cleavage fracture.

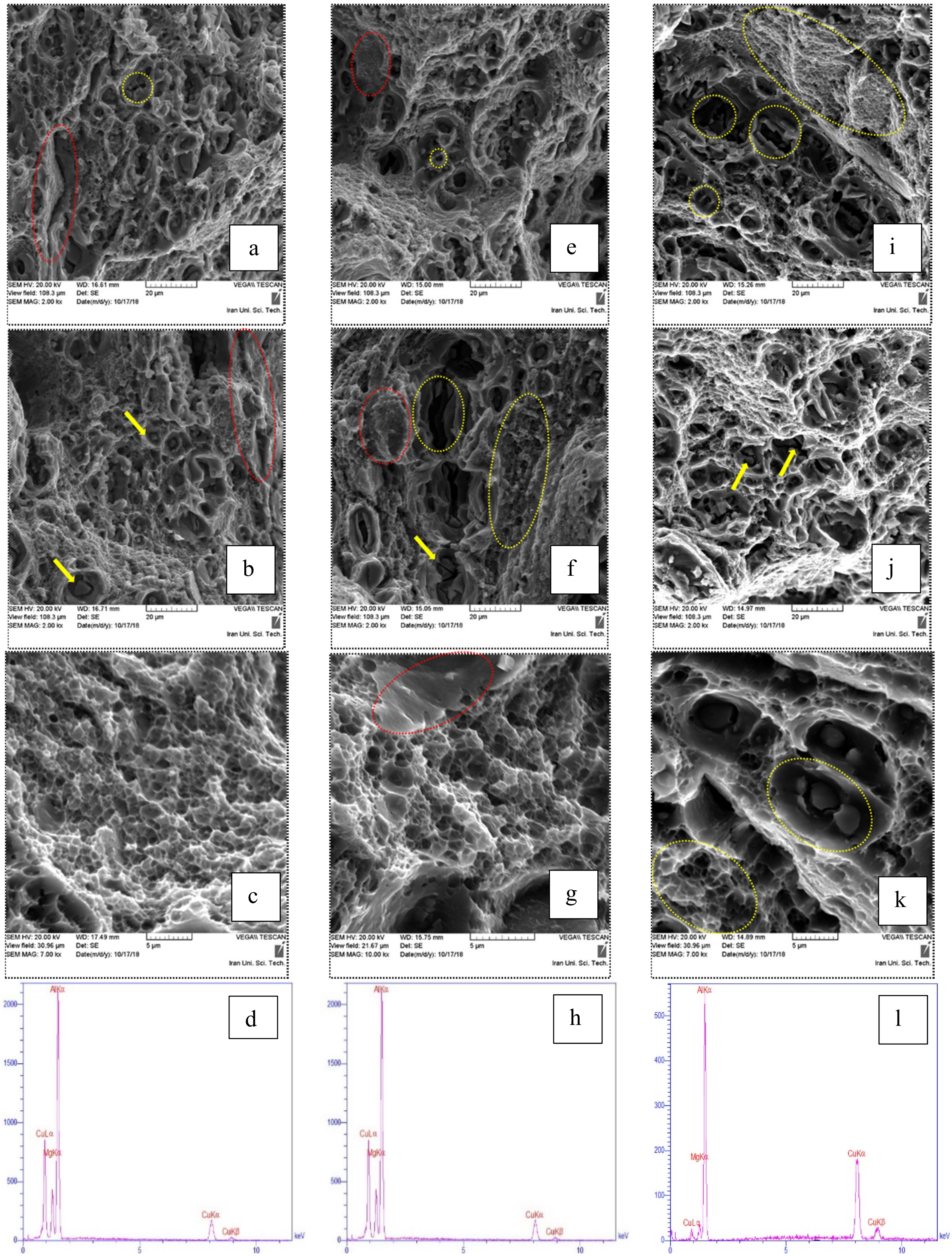

Fig. 6. SEM micrographsand EDS analysis of the fracture surface of specimens at different heat treatment conditions: CIR (a, b, c, d), QIR (e, f, g, h), CAA (i. j. k. l)

Therefore, it is expected that the ductility must be lower than the other two conditions, which it is according to the tensile tests. The shallowness of the dimples is also another reason for lower ductility. Furthermore, most of the particles at the bottom of the dimples have cracked and have been elongated because of lathy precipitates. the chemical composition of the particles at the bottom of the dimples has been examined by EDS analysis and the results are shown in Fig.7. It is clear that in all three conditions, Al2CuMg (S-phase) is the main strengthening phase and because they are non-shearable particles, particle-matrix decohesion is the dominant mechanism for voids formation.

4. CONCLUSIONS

In this work, the effect of infrared heating at the ageing stage and cryogenic cooling after solutionizing on the tensile properties, microstructure and fracture behaviour of Al-Cu-Mg samples were investigated. The following conclusions may be drawn form the results obtained:

1- Cryogenic cooling immediately after solution treatment improves ductility and ageing by infrared heating increases the strength due to the high volume fraction of nano-sized strengthening precipitates.
2- Infrared heating enhances the diffusion rate of the atoms, and as a result, the peak hardness occurs in a short ageing time.
3- When infrared heating and cryogenic treatment are applied simultaneously, the overall mechanical properties improves in comparison with the T6 treatment.

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