Mechanics of Advanced Composite Structures 6 (2019) 139 - 146

Semnan University

Mechanics of Advanced Composite Structures

journal homepage: http://MACS.journals.semnan.ac.ir

Effect of Aging on Fracture Toughness of Al6061-Graphite Particulate Composites

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PAPER INFO

Paper history:
Received 2018-11-06
Received in revised form 2019-02-14
Accepted 2019-04-25

Keywords:
Al6061-9% graphite
Fracture Toughness
Age hardening
CT Specimens
SEM

ABSTRACT

This article presents the investigative work conducted on the fracture toughness and microstructure of Al6061-9% graphite particulate composites. The requisite specimens for the fracture toughness testing were compact tension ones prepared using stir casting technique. The Al6061-9% graphite particulate metal matrix composite has been heat treated in the underaged condition. It is observed from the results that as aging time increases, the fracture toughness increases. This gain in fracture toughness is with the loss of ductility. Examination of the fractured surface of Al6061-graphite is done using a scanning electron microscope (SEM) which shows the brittle fracture of the composite.

1. Introduction

Fracture toughness is typically utilized as a general phrase for measures of material imperviousness to the propagation of the crack. Values of fracture toughness may likewise fill in as a premise in performance evaluation, quality affirmation and material description for representative engineering structures, together with oil and gas pipelines, aircraft, ship, and automotive structures, piping and pressure vessels, petrochemical tanks, etc. In this manner, fracture toughness investigation and assessment have been critical issues being developed for fracture mechanics technique and its engineering applications.

The most important parameters [1] used in fracture mechanics are the elastic energy release rate G (or its equivalent accomplice – stress intensity factor K), the J-integral and the crack-tip opening displacement (CTOD). To measure these parameters, many experimental techniques have been adopted to explain the material fracture toughness (KIc). Customary terminology relating to KIc testing and assessment has been defined in E399-17 [2, 3] by the American Society for Testing and Materials (ASTM). All concepts and requisites related to fracture tests utilized as a part of this work are characterized by ASTM E399. ASTM fracture test standards prescribed many types of conventional fracture test specimens. The most commonly used is the compact tension specimen. Different specimen size requirements are prescribed for different fracture test standards to get valid fracture toughness, and to restrict the effects of crack-tip limitation on that fracture toughness parameter.

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DOI: 10.22075/MACS.2019.15614.1177

Metal matrix composites (MMCs) have their applications, where they require weight savings, wear resistance, and thermal management. Considerably the majority of commonly used metal matrix composites [4] has their base material like aluminum, magnesium, and titanium alloys reinforced with silicon carbide (SiC), alumina (Al_2O_3), carbon, or graphite.

The fracture behavior of the particulate metal matrix composites was influenced by some of the variables such as inter-particle spacing, particles size, and weight fraction of the reinforcement. Additionally, some of the variables affected by the fracture toughness assessment of particulate MMCs are complex micro-structural mechanisms; for example, precipitation solidifying carried out by the heat treatment process. By utilizing a suitable heat treatment process, a fine distribution of precipitates could be obtained.

Precipitation hardening is usually applied to increase the yield strength of the structural alloys of aluminum, nickel, magnesium, titanium, and also aluminum matrix composites. In superalloys, it is known to cause yield strength inconsistency giving astounding high-temperature strength [5]. Precipitates are formed in the matrix material in the form of separate phases by utilizing proper heat treatment conditions, prompting a superior interfacial strength of the composite, subsequently upgrading the overall strength of the material. From the age hardening, the strongest aluminum alloys like (2xxx, 6xxx and 7xxx) could be produced. By appropriate heat treatment process, a fine distribution of precipitates can be obtained.

Age hardening depends on modification in solid solubility with temperature to create fine particles of an impurity phase, which block the development of disengagements, or imperfections in a crystal lattice. Since disengagements are regularly the leading carriers of plasticity, age hardening serves to harden the material. The impurities play an indistinguishable part as the particle substances in particle-reinforced composite materials. Not at all like standard hardening, alloys must be kept at elevated temperature for quite a long time to enable precipitation to happen. This time delay is called "aging".

Lumley et al. [6] studied new methods of heat treatments of age-hardenable aluminum alloys. Commonwealth Scientific and Industrial Research Organization (CSIRO) developed some new heat treatments in Australia, which offers the potential to improve alloy properties and reduce processing costs. The T6I6 heat treatment can be tailored for improvements in strength, ductility, toughness, and fatigue resistance.

Myriounis et al. [7] studied the fracture toughness behavior of Al359 matrix reinforced with SiCp subjected to different heat treatment under the T6 condition and found that heat treatment considerably improved the fracture toughness of the composites. This improvement is due to the heat treatment process, which modifies the microstructure at the region of the matrix-reinforcement interfaces.

Reis et al. [8] investigated the material properties of 2024 aluminum alloy. It was given solid solution treatments at different temperatures followed by quenching in water. It was then artificially aged at 190 and 208°C. Experiments were conducted and found improved mechanical properties as an effect of artificially aging. Radutoiu et al. [9] investigated the influence of the artificial over-ageing time to hardness and mechanical properties. Experimental results reveal that improved hardness and Young's modulus values.

Hegde et al. investigated the effect of aging heat treatment on fatigue behavior [10] and fracture toughness [11] of aluminum-silicon carbide-graphite hybrid composite. Investigative outcomes reveal that enhanced the fatigue and fracture behavior of hybrid composite. Superior fatigue resistance and fracture toughness of the heat treated hybrid composite was due to considerable changes in microstructure and precipitation of second phase particles.

Tocci et al. [12] investigated the tensile strength and impact toughness of AlSi3Cr alloy. From the experiment, improved tensile strength and poor impact toughness values were obtained. Microstructural analysis shows the presence of coarse intermetallics, which acts as crack initiation and propagation. El-Menshawy et al. [13] investigated the effect of aging time on corrosion properties of aluminum alloy 6061 in under, peak and overaged conditions in the temperature range of 140-225°C. The results of the investigation have been discussed in terms of changes in the precipitate type, size, volume fraction, and distribution.

From the literature, it could be observed that many works have been carried out on aluminum silicon carbide at as-cast and age hardened condition. A lot of scopes has been identified concerning the research on aluminum graphite MMCs. In the present work, an attempt has been made to examine the influence of the aging time and temperature on the fracture behavior of Al6061-9% graphite.

2. Materials

Al6061 as a matrix and graphite particles as reinforcement is utilized for the work. The reason to involve these materials is their density. The density of Al6061 is 2.65 g/cc, and of graphite is 2.26 g/cc [14]. The Al6061-graphite particulate composites exhibit isotropic properties and also have outstanding combinations of mechanical and structural properties.

A precipitation-hardened aluminum alloy called Al6061 and its main alloying elements is silicon (0.70 wt%) and magnesium (0.81 wt%). Physical properties [14, 15] of Al6061 are hardness = 95 BHN, Elastic modulus = 68.9 GPa, ultimate tensile strength = 315 MPa, yield strength = 275 MPa, extension = 17%. Graphite is available in the shape of fibres and particles identified as high-strength material. Physical properties of graphite are the elastic modulus of 15 GPa, yield strength of 55 MPa, Thermal Expansion Coefficient is 8.2x10-6z°C. Out of many factors which influence the fracture properties, the particle size of graphite is most important microstructural variable. The particle size of graphite is 44 µm, 300 Mesh.

Al6061 and graphite particulate metal matrix composites produced by solidification techniques present greater tribological properties such as low wear rate, low coefficient of friction, and their outstanding antifriction properties used for a range of automobile applications [16].

3. Processing

Stir casting method [16-18] was utilized to prepare the Al6061-graphite particulate metal matrix composites at 9% weight fractions of graphite. The Al6061 blocks were allowed to melt in the stir casting furnace at a temperature about 720°C. A degasifier was added to the molten aluminum to take away the gases. The requisite quantity of graphite particles was also added to the molten Al6061 while stirring with a stirrer at a speed of 500 rpm. In the split type graphite mold, molten Al6061-graphite was poured, and it was allowed to solidify. From the bars are taken out from molds were utilized for determining required properties of Al6061-graphite alloy bars.

In the microstructure, shown in Fig. 1(a), of the Al6061 graphite particulate composite, confirms the uniform distribution of the reinforcement. During the time spent the mixing, a spinning of liquid material is formed by the revolution of the stirrer through which the graphite particles are drained into the dissolve.

To determine the chemical composition of the Al6061-graphite composites, EDX measurements were carried out in the SEM on individual specimens. The corresponding EDX profile analysis shown in Fig 1(b), provides the atomic percentages of the elements found on the Al6061-graphite surface. The reaction products were also observed at metal matrix/reinforcement interfaces.

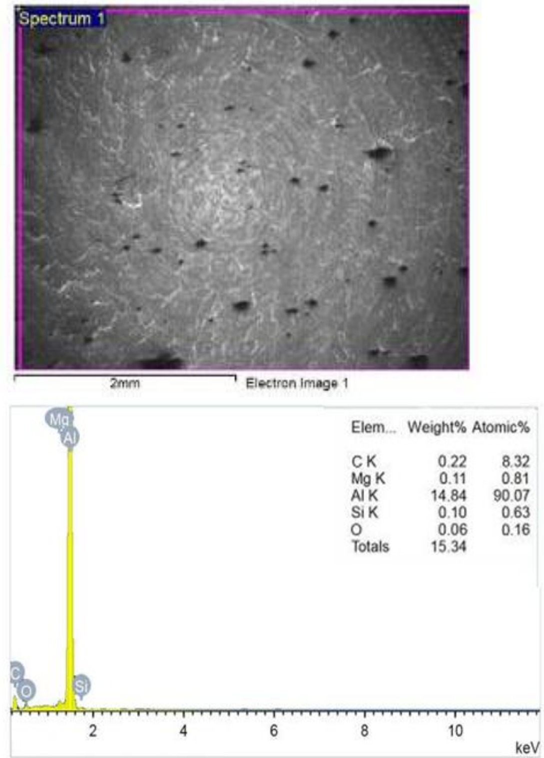

Fig. 1. a) SEM graph showing a distribution of graphite particles in Al6061-9% graphite. b) EDX profile analysis for the surface of Al6061-9% graphite

The table in Fig 1(b) summarizes the atomic percentages of silicon, magnesium, carbon, and aluminum. These outcomes specified that the chemical composition of the Al6061-9% graphite is uniform. The atom percentage of carbon is higher than that of silicon and magnesium. The presence of carbon indicates the addition of graphite reinforcement in the Al6061 matrix. The content of Silicon (0.63 wt%) and Magnesium (0.81 wt%) indicates the presence of Si and Mg in the Al6061 alloy. From the EDX analysis (Fig 1(b)), it is found that Al6061-9% graphite MMCs are rich in both Si and Mg. The existence of MgAl2O4 at interfaces was confirmed in a detailed study [19] on the interfaces in discontinuously reinforced metal-matrix composites. In all the compositions of Al6061-graphite, oxygen (O) content was obtained. The content of O is due to the formation of Al_2O_3 on the top of the pits as the main compound on the surface.

From Fig. 2(a), it is clear that there exists a good bonding between the aluminum matrix and graphite particles. In addition, it is found that there is no indication of extensive segregation or void formation at the matrix-particle interface. The great interfacial bonding and homogeneous distribution of graphite particles in the matrix have a direct effect on the mechanical behavior of a composite material. Due to the good bonding and uniform distribution of graphite particles in the aluminum matrix, Al6061-graphite particulate composites have greater tribological properties and their outstanding antifriction properties. Fig. 2(b) shows the particle size of graphite in the aluminum matrix.

4. Experimentation

Using the following heat treatment process, the microstructure of the Al6061-9% graphite material was changed. The said MMC had been heated to the temperature as solutionising temperature which is above the solvus line temperature of the base alloy for 2 hrs followed by quenching in water. In this way, all the coarse intermetallic phase dissolve into the matrix during the solution process. Artificial aging was done at 155°C, for periods ranging from zero to 7 hrs [8] and subsequently cooled in the air.

hgCT specimens were prepared and heat treated to test the fracture toughness as per the ASTM E399 standard; the CT specimens are shown in Fig. 3. The thickness (B) of the Al6061-9% graphite specimens is considered as 10 mm. The fracture toughness experiments are conducted, at room temperature, in the servohydraulic testing machine with 250 kN load capacity. All the surfaces of the experimental samples are maintained as per the standards and crack length was measured by a visual method. Specimens are fatigue pre-cracked under mode 1 loading to an a/W=0.45. All specimens were subjected to a load ratio of 0.1. Cyclic loading was applied by maintaining the frequency of 5 Hz.

All the CT specimens of thickness (B) = 10 mm are tested to find the fracture toughness. Fracture toughness and fracture characteristics of the aluminum-graphite particulate with 9% weight will be determined using a universal testing machine (UTM) as per ASTM standard testing procedure. For each test, the P_Q value and crack opening displacement is measured and fracture toughness KIc is determined using the empirical Eq. 1 [2, 3]: Fracture load and the calculated value of fracture toughness of the Al6061-9% graphite composites for different specimen thickness are listed in Table 1.

$$K_{Ic} = \frac{P_Q}{B\sqrt{W}} f\left(\frac{a}{W}\right) \tag{1}$$

where, P_Q is the load corresponding to a defined increment of crack length, B is the specimen thickness, W is the width of the specimen, and $f(a/W)$ is a geometry dependent factor that relates the compliance of the specimen to the ratio of the crack length and width, expressed as follows [20]:

$$f\left(\frac{a}{W}\right) = \frac{\left(2+\frac{a}{W}\right)}{\left(1-\frac{a}{W}\right)^{3/2}} \left(\begin{array}{l} 0.886 + 4.64\left(\frac{a}{W}\right) - \\ 13.32\left(\frac{a}{W}\right)^2 + 14.72\left(\frac{a}{W}\right)^3 - \\ 5.6\left(\frac{a}{W}\right)^4 \end{array} \right) \tag{2}$$

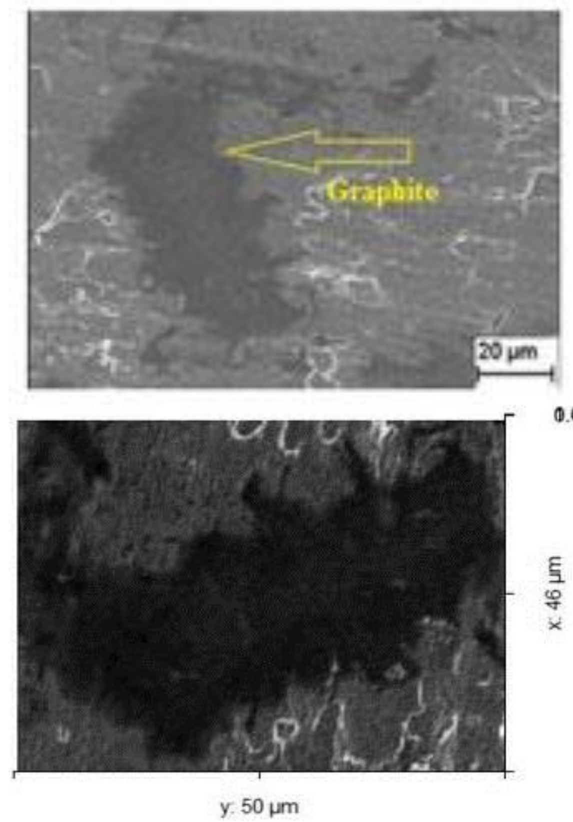

Fig. 2. a) SEM of Al6061-9% graphite demonstrating interface between the matrix and reinforcement. **b).** SEM of Al6061-9% graphite demonstrating an average particle size of graphite in an Al6061 matrix

Fig. 3. CT Specimens with different aging time

Table 1 presents the fracture toughness values for the different aging times. As-cast specimens are not subjected to heat treatment where as zero hr aging specimens are solution treated but not aged. It is clear from the results that as aging time increases, the fracture toughness increases. The optimum value of the fracture toughness was found at 6 hrs of aging time. Semi or fully coherent precipitates are responsible for increasing the fracture toughness values. Furthermore, this gain in fracture toughness is with the loss of ductility. However, a decrease in the fracture toughness of the composite has been observed at 7 hrs of aging. This decrement in the fracture toughness value is maybe due to increased brittleness of the material, which leads to faster crack propagation. The nature of load vs. displacement plot (shown in Fig. 4) shows the brittle failure of age hardened specimens, whereas as-vast Al6061 specimen shows the ductile fracture.

Doddamani et al. [18] utilized compact tension (CT) specimens of various weight fractions of aluminum graphite and tested for their fracture toughness values. The specimen preparation and experiments are carried out according to ASTM-E 399 standard for three different crack length to width (a/W) ratios. Results of the experiments and comparisons are shown in Fig. 4.

5. Results and Discussions

Data obtained from the experiment were used to plot the load versus crack opening displacement graph. Fig. 4 shows the load versus crack opening displacement (COD) curves for thickness (B) = 10 mm, width W=40 mm and various age hardened conditions. In Fig. 4, the curve follows the Type–III curve [18]; therefore, the maximum value of the load itself will be the critical load (PQ). As compared to as-cast specimens, as age hardened specimens will have more load carrying capacity for the less crack opening displacement. Hence, more would be the fracture toughness. Additionally, this increase in fracture toughness is achieved with the loss of ductility.

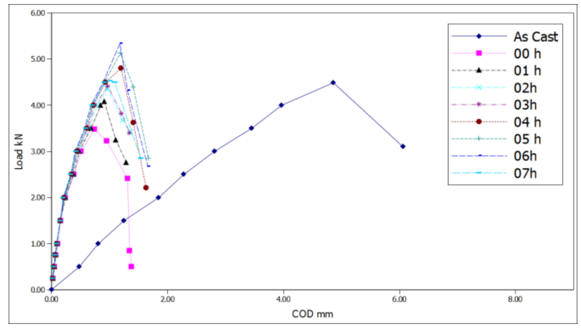

Fig. 4. Load vs displacement plot of Al6061-9% graphite at as-cast and age hardened conditions

From the results shown in Fig. 4, it becomes noticeable that age hardened Al6061-9% graphite composites exhibit higher fracture toughness values than the untreated Al6061-9% graphite composites. As indicated by the load-displacement curves in Fig. 4, Al6061-9% graphite composites noticeably demonstrate more brittle behavior than the as-casted specimens [18].

From the outcome, it has been observed that as aging time increases, the fracture toughness increases. Fracture toughness of particulate metal matrix composites is depending on particle size, interfacial strength levels, and the ductility of the matrix and reinforcement materials. At zero hr of age hardening, obtained a fracture toughness of 14.54 MPa√m, this may be due to the brittleness of the material obtained by water quenching after heating to 530ºC. From 1 hr to 6 hr of aging fracture toughness value increases with aging time and the maximum value of 22.26 MPa√m obtained at 6 hr.

Load versus displacement plot of Al6061-9% graphite at as-cast and different age hardened conditions is shown in Fig. 4. From the results of the comparison, it is observed that as-cast Al6061-9% graphite has more ductility as compared to the age-hardened condition. All the age-hardened specimens show the brittle fracture, whereas as-cast Al6061-9% graphite has demonstrated more elongation to the applied load.

Table 1. KIc of Al6061-9% graphite at as-cast and different aging time

Specimen	Thickness (B) mm	Width (W) mm	Fracture Load (P_Q) kN	f (a/w)	K_{Ic} (MPa√m)
as-cast [16]	10	50	4.49	8.34	16.74
00 Hrs Aging	10	40	3.48	8.34	14.51
01 Hrs Aging	10	40	4.08	8.34	17.01
02 Hrs Aging	10	40	4.35	8.34	18.14
03 Hrs Aging	10	40	4.42	8.34	18.43
04 Hrs Aging	10	40	4.80	8.34	20.47
05 Hrs Aging	10	40	5.13	8.34	21.39
06 Hrs Aging	10	40	5.34	8.34	22.26
07 Hrs Aging	10	40	4.53	8.34	18.89

Maximum fracture toughness obtained for the as-cast specimen is KIc = 16.74 MPa√m [18], whereas for 6 hrs of age hardening KIc = 22.26 MPa√m. In addition, it is observed that, in comparison with as-cast specimens, the load-bearing capacity of the material increases as age hardening time increases.

The micrographs from SEM of Al6061-9% graphite for different aging conditions are shown in Fig. 5 (a-h). Scanning electron micrograph (SEM) images have been obtained at a magnification of 2μm, working distance = 4.5 mm. From Fig. 5, it is clear that a strong homogeneous microstructure is obtained between aluminum and graphite after age hardening.

In Fig. 5 (a), at zero hrs, i.e., solution heat treatment at 530ºC for 2hrs followed by water quenching has some micro-voids and cracks visible, whereas at 1 and 2 hr of aging at 155ºC followed by air cooling have some refined microstructure.

Inspection of microstructure of the Al6061-9% graphite reveals that heat treatment clearly improves the fracture properties of the composite. This is related to a precipitation hardening mechanism mainly due to the accumulation of precipitates Mg2Si particles at the interfacial region. At 3 to 6 hours of aging, accumulation of precipitates near the interface of matrix and reinforcement has been obtained, which facilitates in the load interchange from the reinforcement to the surrounding matrix. Similarly, crack propagation happens in the composite by means of the reinforcement and not along with the interface. A strong matrix and reinforcement interface enables the graphite particles to set up themselves into the matrix logically, improving the crack resistance. In 5 and 6 hours of aging time have optimum values of fracture toughness which clarifies from the SEM micrograph (Fig. 5).

Fig. 6 shows the SEM images of the fractured surfaces of the CT specimens. After heat treatment and different aging conditions, the material becomes harder and shows the brittle fracture.

Fractography of the fracture surface in Al6061-9% graphite specimens showed particle fracture, for the most part in the aging. In the age hardened condition, graphite particles appear to be cracked yet not debonded (Fig. 6) demonstrating a decent interfacial bonding. It is generally the bigger particles those breaks have the great possibility of finding an imperfection of basic size and furthermore because of the reality of those bigger particles, during fabrication, may have been cracked. From the fractographic study of microstructure in the aluminum matrix, composite materials become apparent that the heat treatment process enhances fracture properties of the composite. This is identified with a precipitation hardening method because of the collection of precipitates at the interfacial region.

At age hardened condition of the Al6061-9% graphite MMC, fracture toughness (KIc) was estimated experimentally. Experimentation was conducted and found that fracture toughness increases as aging time increases.

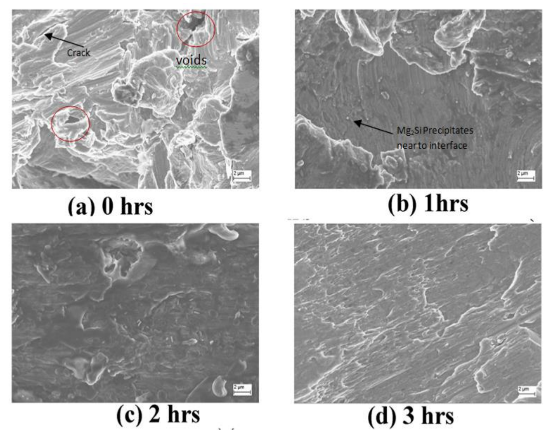

Fig. 5. SEM images at various age hardened conditions at 2μm magnification

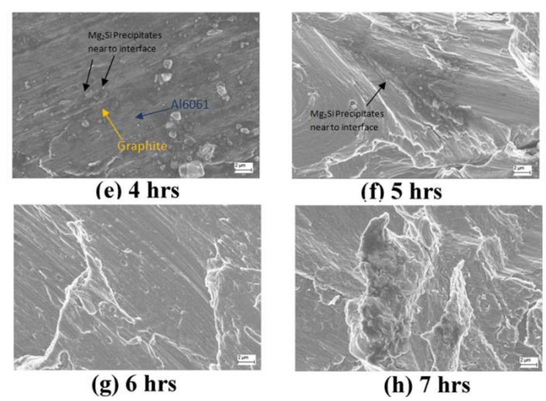

Fig.6.SEM images at various age hardened conditions at 2μm magnification

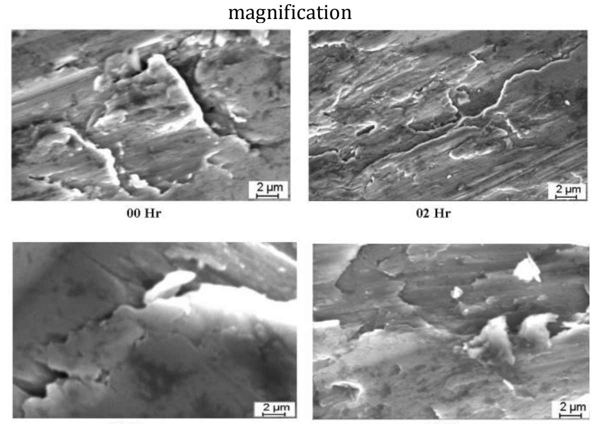

Fig. 7. SEM images showing voids, cracks in the fractured surfaces of CT specimen of various age hardened conditions

6. Conclusions

The CT specimens of the thickness (B) = 10 mm were utilized for the age hardening. The Al6061-9% graphite particulate MMC has been heat treated. In this way, all the solute molecules were permitted to dissolve to form a single-phase solid solution. It is clear from the results that as aging time increases, the fracture toughness increases. The optimum value of the fracture toughness was found at 6 hours of aging time. 5 and 6 hours of aging time have an optimum value of fracture toughness, i.e., 22.26 MPa√m. However, the decrement in the fracture toughness value at 7 hours is maybe due to increased brittleness of the material, which leads to faster crack propagation. From the fractographic study of microstructure in the aluminum matrix, composite materials become apparent that fracture properties of the composite are enhanced by the heat treatment process. In addition, this gain in fracture toughness is with the loss of ductility.

Nomenclature

K_{Ic}	Fracture Toughness MP√a
P_Q	Load at Peak
B	Thickness of Specimen respectively
W	Width of Specimen respectively
a	Crack length

Funding

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

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