Effects of Heat Treatment on Microstructure and the Fracture Toughness of SiC_p/Al Alloy Metal Matrix Composites

D. P. Myriounis[1-2]*, S. T. Hasan[2], N. M. Barkoula[1], A. Paipetis[1], T. E. Matikas[1]

[1]University of Ioannina, Department of Materials Science & Engineering, 45110, Ioannina, Greece
[2] Sheffield Hallam University, Materials & Engineering Research Institute, S1 1WB, Sheffield, UK

Abstract

The current study focuses on the fracture toughness behaviour of A359 aluminium matrix reinforced with 31 wt. % SiC particulates subjected to different heat treatment conditions. Unreinforced aluminium alloy fracture properties have been also determined for reference purposes. Three different heat treatment conditions have been applied to the Al/SiC_p composites and the fracture toughness values have been determined for all specimens. Infrared thermography was used to monitor the plane crack propagation behaviour of the materials and helped in verifying the validity of the fracture toughness testing. The results indicate that valid K_{IC} values were obtained, which were found to be lower than those for the unreinforced matrix alloy, as expected. However, heat treatment considerably improved the fracture toughness of the composites. In particular, the specimens heat treated under the T6 condition exhibited enhanced fracture toughness compared to the other two conditions. This behaviour can be attributed to a mechanism related to microstructural modifications at the vicinity of the interface due to the heat treatment. This mechanism is associated with precipitates accumulated at the interfacial region resulting in material hardening.

Keywords: Metal matrix composites; Particulate-reinforced aluminium, Fracture toughness; Heat treatment; Precipitation hardening; Infrared thermography

** Corresponding author. Tel: (+30) 6955 999057 Fax: (+30) 26510-97034*
E-mail address: myrioun@cc.uoi.gr

1. Introduction

Aluminium alloys are attractive base materials which, with the addition of discontinuous ceramic reinforcements, can achieve enhanced mechanical performance, i.e. elastic properties, wear resistance, strength and coefficient of thermal expansion [1-6]. The major drawback of the inclusion of the ceramic reinforcement in aluminium matrix composites is their tendency to brittle behaviour, i.e. low fracture toughness values, due to the brittle nature of the ceramic reinforcement in an otherwise ductile matrix [7-15]. The microstructure-dependent fracture mechanisms and their correlation to the macroscopical mechanical behaviour are not yet well understood in the case of particulate-reinforced metal matrix composites (MMCs). Furthermore, while performing fracture mechanics studies in such materials, it is quite difficult to satisfy specific validity criteria, and often the provisional fracture toughness, K_Q, is quoted instead of the plain strain fracture toughness, K_{IC}.

The mode I plain strain fracture toughness, K_{IC}, is a material property characterising its resistance to fracture under the following conditions: a) a sharp crack is present under tensile loading, b) in the vicinity of the crack's front triaxial plane strain conditions occur and c) the plastic region at the crack-tip is small compared with the crack size and specimen dimensions. A valid K_{IC} value provides a lower limiting value of fracture toughness, and is a key parameter in estimating the relationship between failure stress and defect size for a material in service under similar stress state conditions [16].

At present, there are no standard fracture toughness testing procedures specifically for MMCs, therefore, conventional standards for metals such as the ASTM E399 are normally used [17]. Some of the problems associated with interpreting toughness test results on MMCs have been considered by Goolsby and Austin [18] who concluded that there were very few results in the literature that satisfied the ASTM E399 validity criteria. The main reasons for failing to obtain valid K_{IC} values were largely due to excessive crack curvature, non-linearity of the load-displacement curve, or out-of-plane crack propagation.

To further understand the mechanisms involved with the fracture toughness of MMCs, microstructural strengthening mechanisms such as precipitation hardening, need to be addressed. The strengthening micromechanical mechanisms of MMCs are very complicated due to the several parameters involved. The ability of reinforcement to improve the overall material mechanical behaviour of the composite is not always successful for improving each and every mechanical property of the MMC, due to the fact that the brittle nature of the reinforcement usually diminishes some properties, such as the fracture toughness.

Some of the factors affecting significantly the fracture properties of particulate MMCs, are the particles size, interparticle spacing, and volume fraction of the reinforcement [19-21]. Furthermore, the fracture toughness values of particulate MMCs can be influenced by complex microstructural mechanisms such as precipitation hardening achieved by heat treatment processing. Using appropriate heat treatment conditions, precipitates are formed in the matrix material in a form of separate phases,

leading to an improvement of interfacial strength of the composite, thereby enhancing the overall strength of the material [22-23].

In this work the influence of the microstructure at the vicinity of the interface on the fracture behaviour of particulate-reinforced aluminium alloy matrix composites is studied. Furthermore, a novel approach is being applied to characterise the fracture behaviour of the particulate composites. Infrared thermography is being used to monitor the plane crack propagation behaviour of the materials.

2. Materials and Heat Treatment

The metal matrix composites studied in this work consisted of aluminium – silicon – magnesium alloy matrix A359, reinforced with silicon carbide particulates. Hot rolled A359 Aluminium alloy with 31% SiC particles per weight with an average particle size of 17±1 μm was used (Fig.1). Additionally, unreinforced Aluminium-Copper 2xxx series alloys have been examined for comparison purposes. The chemical compositions of the matrix alloys are shown in Table 1.

Table 1. Chemical composition of the matrix materials.

Material	Elements (wt %)					
	Si	**Mg**	**Mn**	**Cu**	**Fe**	**Zn**
A359 aluminium	***9.5***	***0.5***	***0.1***	***0.2***	***0.2***	***0.1***
Al-Cu 2xxx	**-**	***1.5***	***0.6***	***4.4***	**-**	**-**

The microstructure of the as received (AR) materials was modified using the following two heat treatment conditions [24]:

(a) T6 heat treatment: In the solution heat treatment, the alloys were heated to a temperature just below the initial melting point of the alloy for 2 hours at 530±5 ºC. Thus, all the solute atoms were allowed to dissolve to form a single-phase solid solution before being quenched in water. Next, the composites were heated to a temperature of 155 ºC for 5 hours and subsequently cooled in air.

(b) HT-1 heat treatment: In the solution heat treatment, the alloys were heated for 1 hour to a temperature lower than the T6 heat treatment that is 450±5 º C, and then quenched in water. Subsequently, the alloys were heated to an intermediate temperature of 170 ºC for 24 hours in the age hardened stage and then cooled in air.

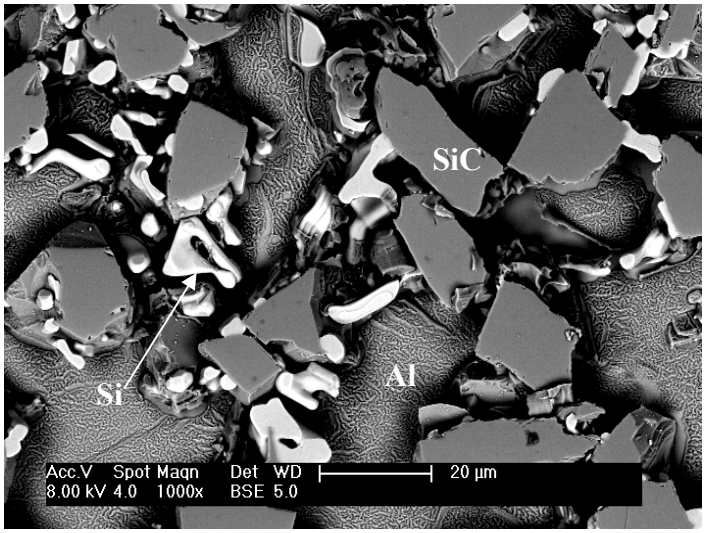

Fig.1 SEM image showing an aluminium – silicon – magnesium alloy matrix, A359, reinforced with 31 wt. % silicon carbide particles.

3. Experimental Procedure

3.1. Fracture Toughness K_{IC} Testing

The plane strain fracture toughness test involves the loading to failure of fatigue pre-cracked, notched specimens in tension or in three-point bending. The calculation of a valid toughness value can only be determined after the test is completed, via examination of the load-displacement curve and measurement of the fatigue-crack length. The provisional fracture toughness value, K_Q, is first calculated from the following equation:

$$K_Q = \left(\frac{P_Q}{BW^{1/2}} \right) \cdot f\left(\frac{a}{W} \right) \tag{1}$$

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

$$f\left(\frac{a}{W} \right) = \frac{(2 + a/W)(0.86 + 4.64a/W - 13.32a^2/W^2 + 14.72a^3/W^3 - 5.6a^4/W^4)}{(1 - a/W)^{3/2}} \tag{2}$$

Only when specific validity criteria are satisfied, the provisional fracture toughness, K_Q, can be quoted as the valid plane strain fracture toughness, K_{IC} [16].

The standard used for conducting this experiment, i.e. ASTM E399, has strict validity criteria to ensure that the plane strain conditions are satisfied during the test. These criteria include checks on the form and shape of the load versus displacement curve, requirements on specimen's size and crack geometry, and the 0.2% proof

strength values at the test temperature. Essentially, these conditions are designed to ensure that the plastic zone size associated with the pre-crack is small enough so that plane strain conditions prevail, and that the linear elastic fracture mechanics approach is applicable.

Fracture toughness tests were conducted using a 100 KN servo-hydraulic universal testing machine with data acquisition controller. The system was operated on load control for the fatigue pre-cracking stage, and on position control for the crack opening displacement (COD) testing. The fatigue test for pre-cracking was conducted at a frequency of 1 Hz, at a load ratio R = 0.25 and load range of 3.7 - 4.5 KN according to the materials' ultimate tensile strength. The COD was monitored by a clip gauge attached to the specimen with a testing rate set at 1 mm/min. Moreover, a thermal camera was set for thermographic monitoring of the crack opening displacement. Compact tension (CT) specimens were prepared for fracture toughness tests according to ASTM E399; the CT specimen geometry is shown in Fig.2. The thickness B of the specimens was 9.2 mm for the MMC, and 5 mm for the unreinforced aluminium alloys.

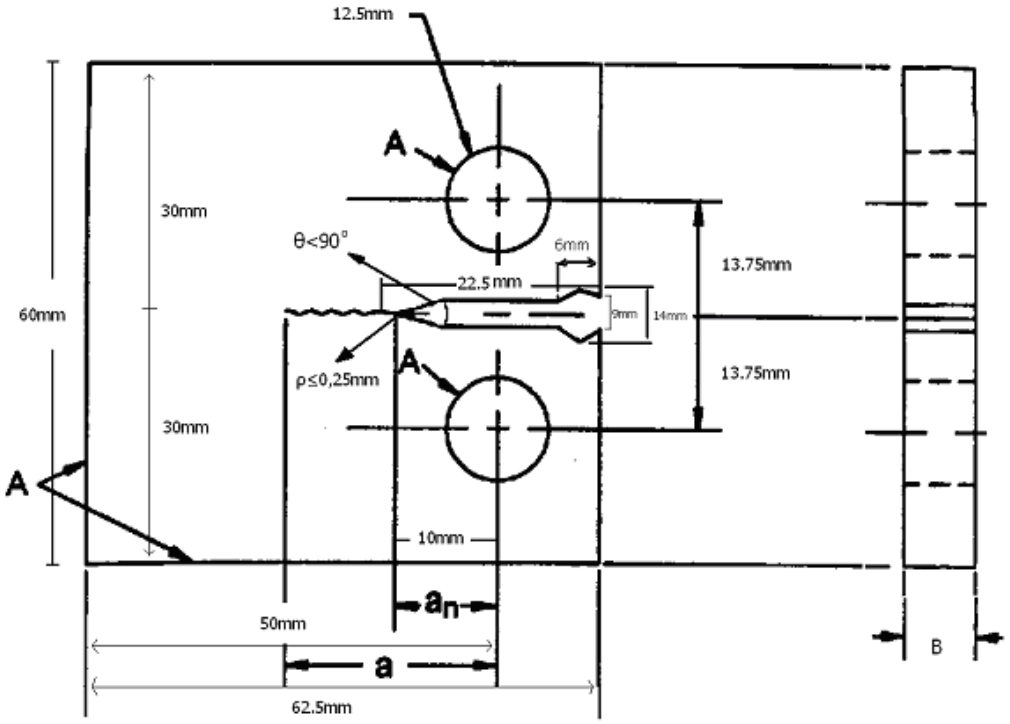

Fig.2 Fracture Toughness CT specimen geometry according to ASTM E399 standard.

3.2. Infrared Thermography

Infrared thermography was used to monitor the plane crack propagation behaviour of the materials. The deformation of solid materials is almost always accompanied by heat release. When the material becomes deformed or is damaged and fractured, a part of the energy necessary to initiate and propagate the damage is transformed in an irreversible way into heat [25-26]. The heat wave, generated by the thermo-mechanical coupling and the intrinsic dissipated energy during mechanical loading of the sample, is detected by the thermal camera. By using an adapted detector, thermography records the

two dimensional ‘‘temperature’’ field as it results from the infrared radiation emitted by the object. The principal advantage of infrared thermography is its noncontact, non-destructive character.

3.3. Fractography

The microstructure of the fractured composites was investigated in the as-received and heat-treated conditions, using a Philips XL40 Scanning Electron Microscope with a link 860 EDAX and a Philips FEI Nova Nano – Scanning Electron Microscope.

4. Results and discussion

4.1 Fracture toughness, KIC

Provisional K_Q values have been calculated according to ASTM E399 standard for all specimens according to Equations (1) and (2), where $P_q = P_{max}$. Load versus displacement curves for Al/SiCp composites and unreinforced aluminium alloys are shown in Fig.3. Fracture toughness data for Al/SiC_p and unreinforced aluminium alloys are detailed in Table 2.

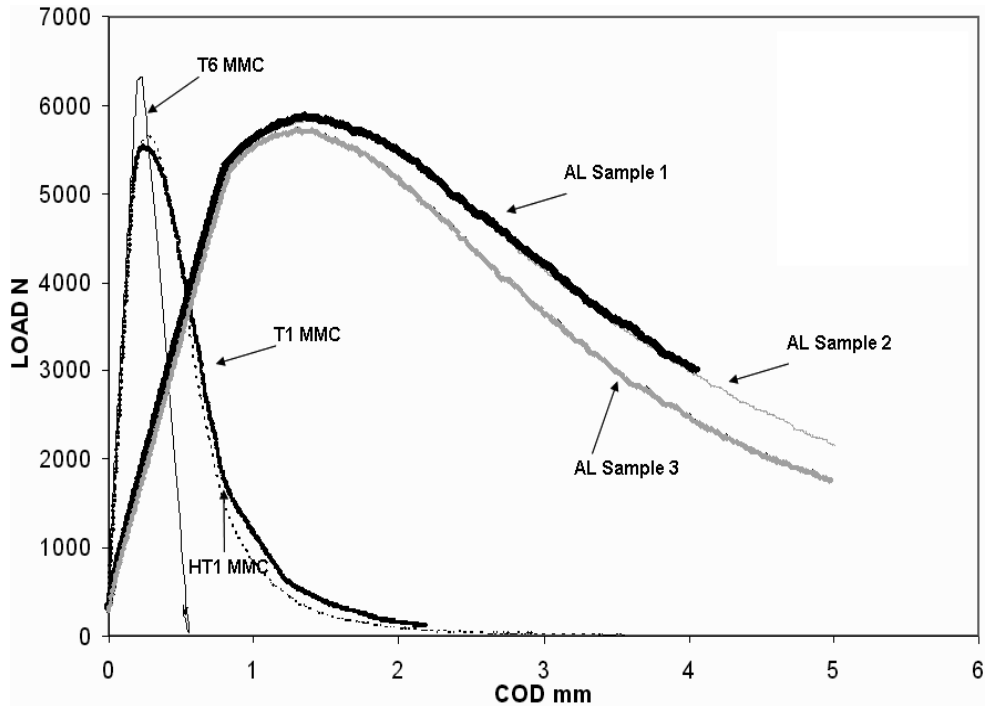

Fig.3 Load – Displacement curves for Al/SiCp composites subjected to T1, T6 and HT-1 heat treatment conditions and three unreinforced aluminium alloy samples.

From the results shown in Table 2, it becomes obvious that Al/SiC_p composites exhibit lower provisional K_Q values than the unreinforced aluminium alloys examined. In addition, heat treatment processing, and especially T6 treated specimen, have the highest K_Q values compared to the other two heat treatment conditions. According to the load-displacement curves in Figure 3, composites clearly show more brittle

behaviour than the unreinforced aluminium alloys. T6 heat treated composites have the highest strength, but the lowest ductility compared to the other materials. Although these results provide some insight regarding the fracture behaviour of the materials examined, specific validity criteria have to be satisfied in order to obtain K_{IC} values.

Particular attention was paid to the influence of the specimen's thickness and other validity criteria such as crack curvature and in-plane crack propagation, since these are the most common reasons for a test to be invalid. In this work the most important validity criterion related to the crack curvature was found to be satisfied both for the Al/SiC_p composite specimens as well as for the unreinforced aluminium alloys. Figure 4 shows the optical examination of the crack curvature for various specimens tested having different thickness. Moreover, for CT specimens, the fracture toughness standard requires that the surface crack length should not differ from the effective crack length by more than 15%. The effective crack length a_{eff} was calculated as the mean value of the crack lengths at the centre and quarter thickness positions [16]. These validity criteria considerations are reflected in Table 2.

Table 2. Fracture toughness data for Al/SiCp and Al alloys and test validity.

Material	**Heat Treatment**	**E (GPa)**	**$Rp_{0.2}$ (MPa)**	**B (mm)**	**a/W**	**α_{eff} (mm)**	**K_Q (MPa √m)**	**Valid**	**Reason**
2000 series Al	AR	71	75	5.10	0.552	27.62	55,36	No	2**
2000 series Al	AR	71	78	5.13	0.555	26.76	56,00	No	2**
2000 series Al	AR	71	72	5.00	0.558	28.43	58,48	No	2**
A359/SIC/31p	T1	108	158	9.20	0.456	20.79	19,28	Yes	-
A359/SIC/31p	T6	116	290	9.21	0.462	20.12	22,05	Yes	-
A359/SIC/31p	HT1	110	155	9.20	0.467	21.33	20.75	Yes	-
A357/SIC/20p [16]	-	-	215	-	-	-	18.60	-	-
A359/SIC/10p [16]	-	-	300	-	-	-	17.40	-	-

**Validity criteria:
1 Excessive crack curvature
2 Thickness criteria not satisfied
3 Excessive plasticity
4 a/W out of range
5 Non-symmetrical crack front
6 In plane crack propagation

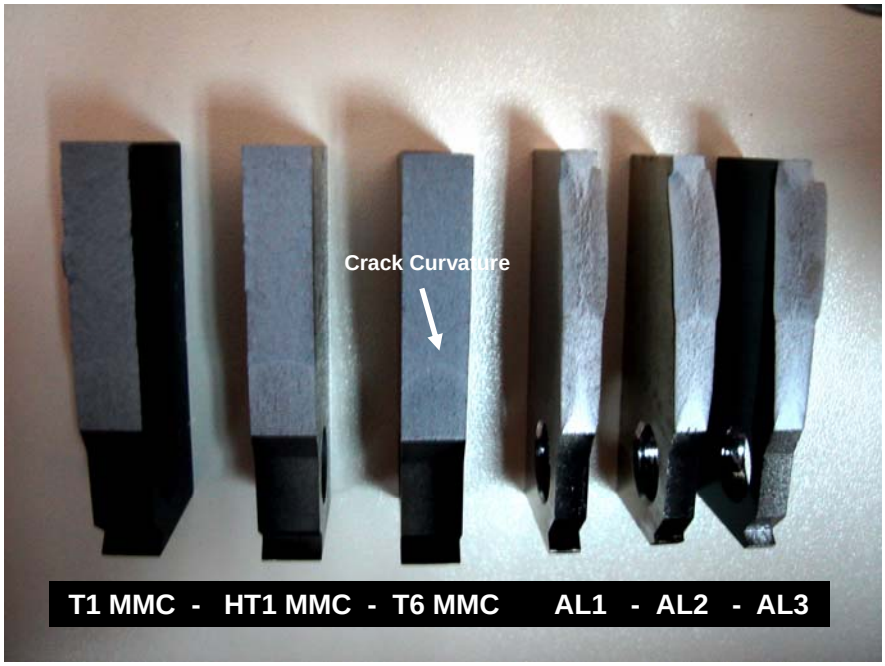

Fig.4 Variation in crack curvature with specimen thickness.

Next to the crack curvature, another important validity criterion is the plane crack propagation. It is very important to have a crack that propagates through the specimen in plane strain conditions with limited plasticity, in a straight line preferably. Especially for MMCs reinforced with brittle particles there was a high probability that a crack deflects by the hard particles, and then propagates out of plane. All specimens showed, however, valid plane crack propagation behaviour; thermograhic monitoring provided this evidence shown in Figures 5a, b, and c.

In summary, in the tests performed for all MMC specimens, heat treated in three different conditions, all validity criteria were met. Therefore, K_Q values could be considered as K_{IC} valid fracture toughness values. However, the aluminium alloy CT specimens did not meet the thickness validity criterion. Hence, in this case, the K_Q values were kept for comparison purposes. As expected, fracture toughness values of the composites were lower than those of unreinforced aluminium alloys, however, heat treatment significantly improved K_{IC} of the composites; especially, T6 condition had more effect in improving the fracture toughness values than the HT-1 condition. Also, the K_{IC} values for all heat treatment conditions were higher than other MMC values documented in the literature, even having lower weight percentage of silicon carbide particles.

4.2 Thermography

A rectangular area on the specimen, located just in front of the initial pre-cracking region, was selected, as shown in Fig. 5a. The development of fracture was monitored in that area using infrared thermography. To accomplish this, the mean temperature in this area versus time during crack growth was constantly measured. As the specimen is stretched in tension, stresses are accumulating in the specimen, hence the temperature increases as a function of time. When the accumulated energy becomes sufficient to

propagate the crack, it results in crack growth, which results in stress relief. This corresponds to a pick in the temperature-time curve followed by a sudden decrease in temperature. This behaviour has several repetitions, as shown in Fig. 5b, 5c and 5d. In these figures the thermographic monitoring of Aluminium 2xxx alloy, Al/SiCp T6 composite, and Al/SiCp HT1 composite samples is presented respectively. These representations show the different stages of crack growth up to the specimen's final fracture of each material. As it can be observed, just prior to fracture the plasticity zone is clearly delineated on the specimen's surface as a heated region, which may be readily attributed to local plastic deformation. Furthermore, in all Figures, crack propagation shows valid in-plane crack propagation throughout the experiment.

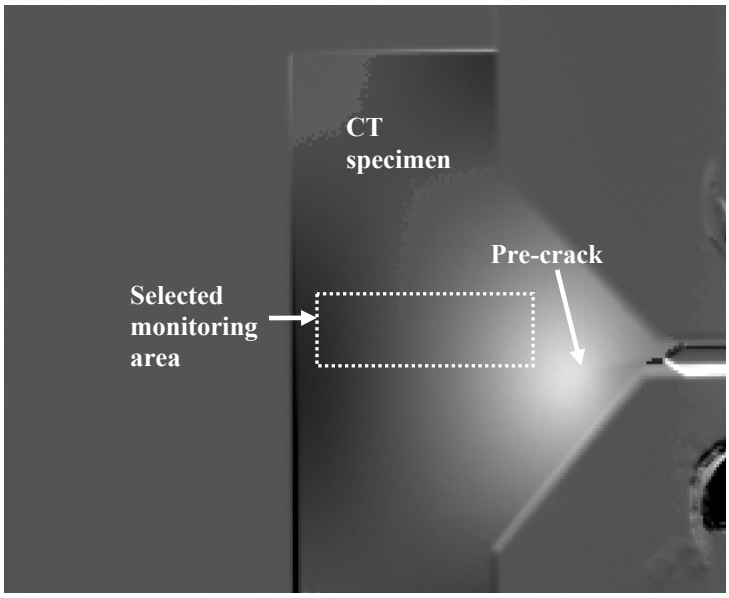

Fig.5a CT specimen showing the selected area for thermographic monitoring.

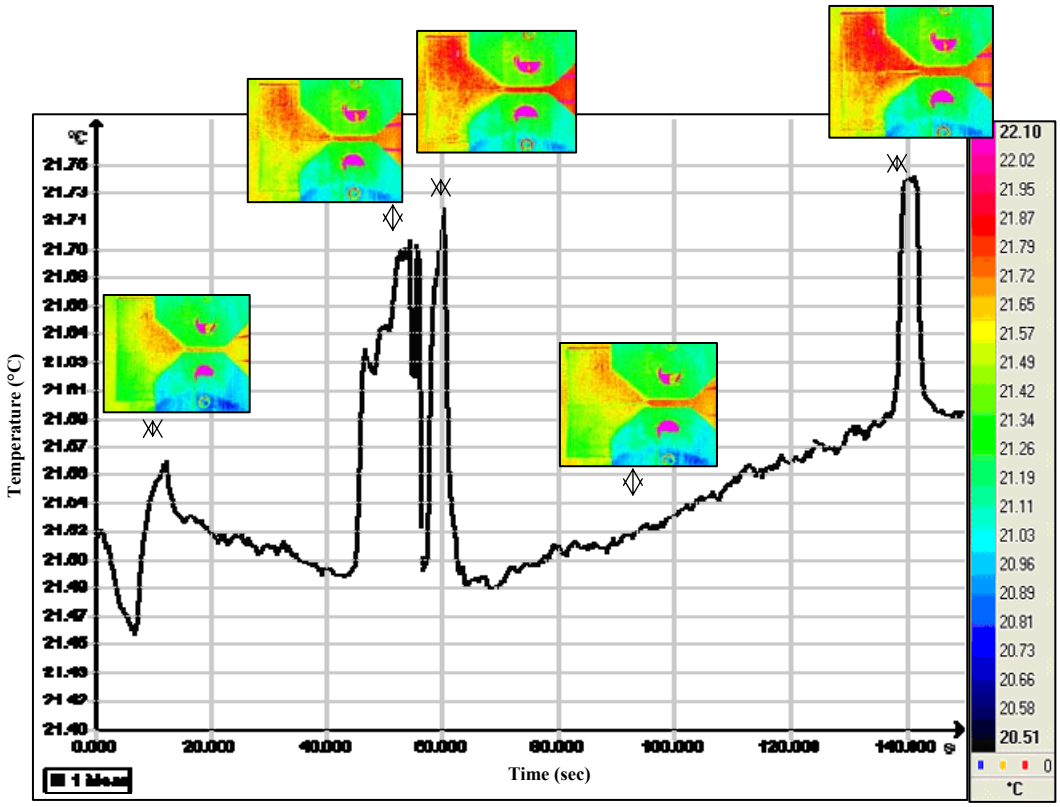

Fig.5b Thermographic monitoring of Aluminium 2xxx CT sample showing the different stages of crack growth up to the specimen's final fracture.

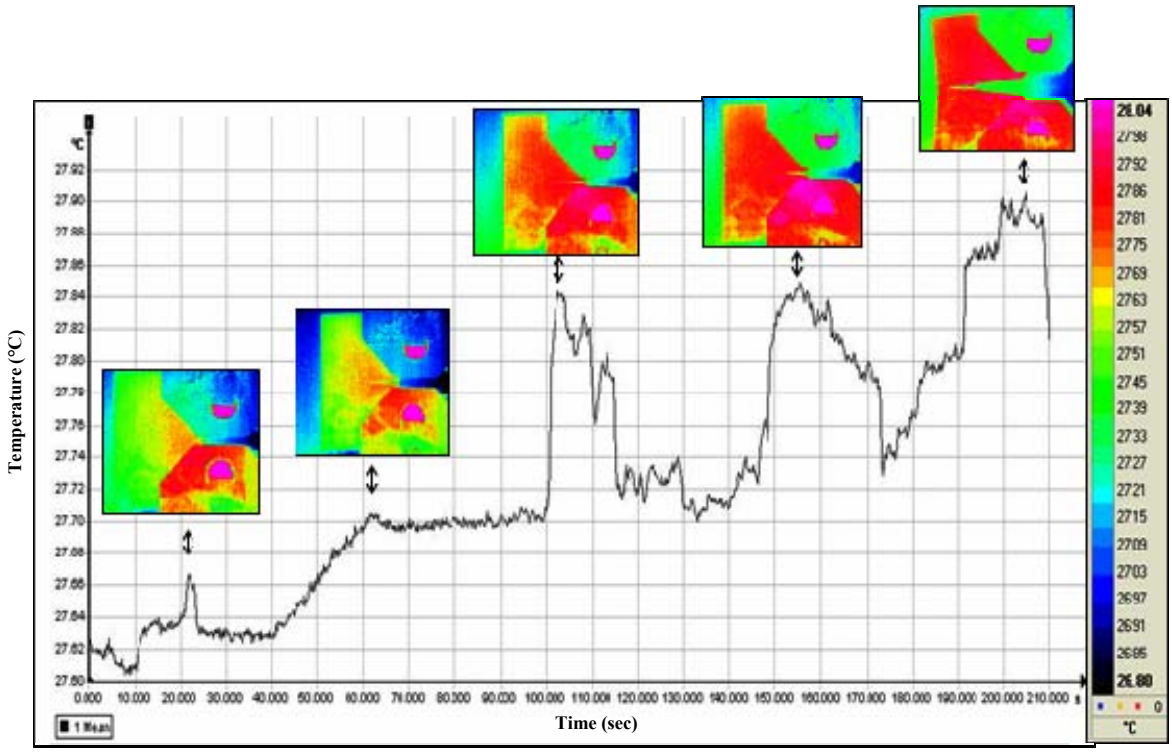

Fig.5c Thermographic monitoring of Al/SiCp T6 composite CT specimen showing the different stages of crack growth up to the sample's final fracture.

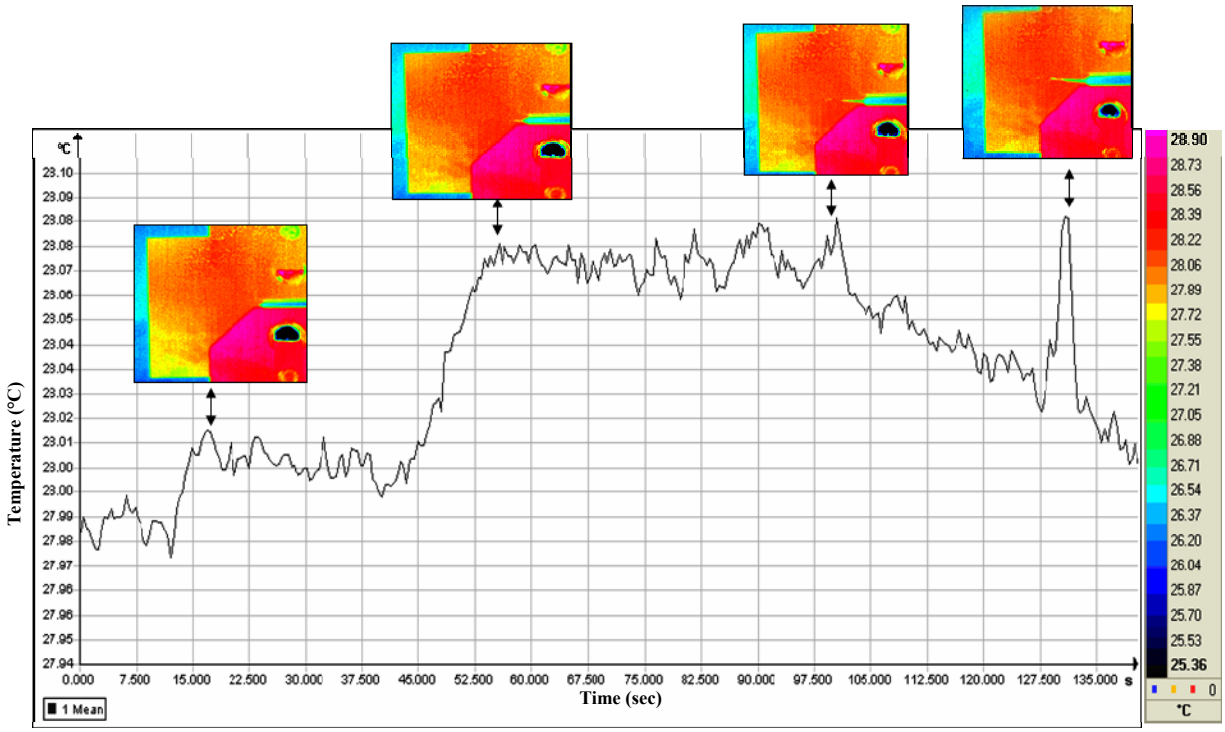

Fig.5d Thermographic monitoring of Al/SiCp HT1 composite CT specimen showing the different stages of crack growth up to the sample's final fracture.

A comparison of the thermography graphs in Figs. 5b, 5c, and 5d leads to the conclusion that different crack propagation behaviour exists for the aluminium alloy and the Al/SiC_p composites. For the aluminium alloy, the temperature versus time curve in Fig.5b shows some extended plasticity behaviour before final fracture occurs. This behaviour is evidenced by the constant increase in temperature between the temperature picks at the 60^{th} and 140^{th} seconds. The small specimen thickness may be the reason for this behaviour. However, for the T6 heat treated composite material in Fig.5c, fracture occurs in a more stable manner where elastic behaviour appears to be dominant as indicated by the multiple temperature picks. Also, plasticity is formed in a more balanced way regarding the overall fracture process. It was also observed that T6 heat treated composites exhibited fewer picks compared to the HT1 heat treated specimens (Fig. 5d). This is due to the presence of stronger interface in the T6 material due to accumulation of precipitates near the interface, resulting in improvement of the fracture toughness property of the material.

It can therefore be concluded that the T6 heat treated composite demonstrates steady elastic crack propagation and this may be attributed to the microstructural strengthening mechanism, such as precipitation hardening, where precipitates dispersed in the interfacial region of the composite sustain stresses introduced during loading of the sample providing balanced fracture behaviour.

4.3 Microstructural examination

Fractography of the rapid overload fracture region in MMC specimens tested did show some particle fracture, mostly in the T6 heat treatment. This is in accordance with previous observations [27]. In the T6 condition, SiC particles seem to be cracked but not

debonded (Fig.6a) indicating a good interfacial bonding. It is usually the larger particles that break because of the higher probability of finding a flaw of critical size and also due to the fact those larger particles may have been cracked during fabrication. In the HT-1 heat treatment condition, shown in Fig.6b, cracking was identified through the interface region. This is not the desired propagation route since interface has to remain uncracked to sustain the stresses arising from the crack. The as received condition T1, shown in Fig.6c, shows some coalescence microvoids, evidence of ductile behaviour. From the examination of microstructure in these materials becomes evident 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 at the interfacial region.

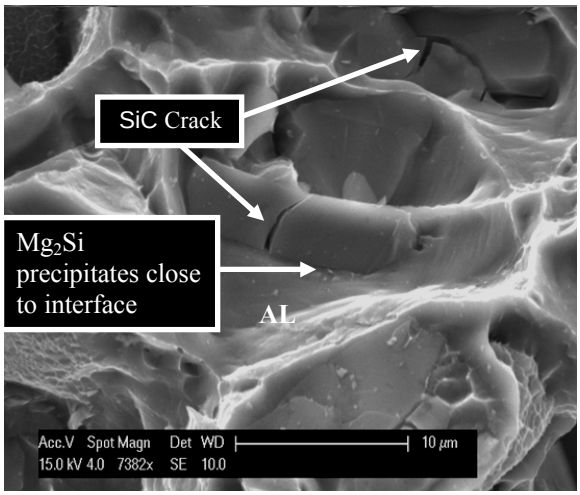

Fig.6a T6 heat treatment condition: SiC particles cracked but not debonded.

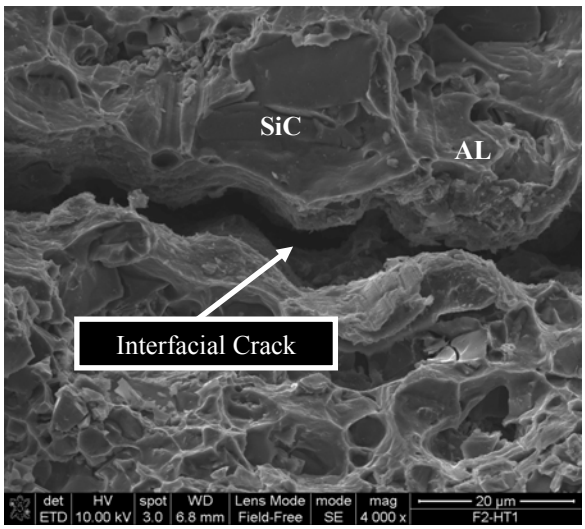

Fig.6b HT-1 heat treatment condition: cracking through interface.

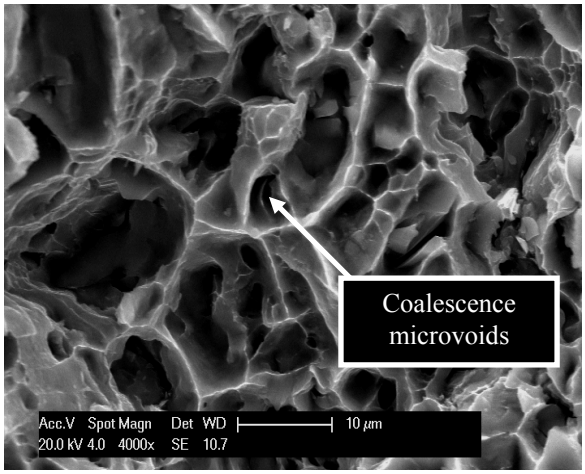

Fig.6c As received condition: Presence of coalescence microvoids indicating ductile behaviour.

5. Conclusions

The determination of valid plane strain fracture toughness (K_{IC}) for particulate-reinforced aluminium matrix composites subjected to different heat treatment conditions has been achieved by satisfying all the validity criteria as per ASTM E399 standard. Infrared thermography was used to monitor in real-time the various stages of crack growth up to the specimen's final fracture, in order to demonstrate that linear elastic fracture mechanics approach was satisfied and support the validity of fracture toughness measurements.

It was found that K_{IC} values reported in this paper are still lower than unreinforced aluminium alloys, but higher than other MMC values documented in the literature, even with lower weight percentage of silicon carbide particles.

Heat treatment processing is the key to this improvement, with the T6 heat treated composite to convene the highest fracture toughness value. This can be attributed to a dominant mechanism associated to microstructural changes in the composite. This mechanism relates to the precipitates appearing in the microstructure of the composite at the vicinity of the interfacial region, which results to the composite's hardening.

Thermographic examination of the materials show that heat treated composite samples exhibit regular crack propagation behaviour. Stress concentration, due to the presence of particle reinforcements, produces controlled crack growth and higher stresses, which are related to regular energy release by the material during fracture, indicative of brittle fracture behaviour. However, the aluminium alloy shows large plastic deformation during the test with few stress-picks.

Acknowledgements

Support of this work by MC-21 Inc, Carson City NV, USA and in particular Dr. David Schuster, is gratefully acknowledged.

References

[1] J.K. Shang, W.Yu, R.O. Ritchie, Mater Sci Eng. A102 (1988) 181–92.
[2] C.R. Crowe, R.A. Gray, D.F. Hasson, in Proc. Int. Conf on 'Composite materials', ICCM-V, (ed. W. C. Harrigan et al.), (1985) 843-866 New York, AIME.
[3] M. Manoharan, J.J. Lewandowski, Scripta Met. 23 (1989) 301-305.
[4] D.L. Davidson, Mat. Sci. 24 (1989) 681—687.
[5] M. Manoharan, J.J. Lewandowski, Int. J. Fract. 40 (1989) R31-R34
[6] M. Manoharan, J.J. Lewandowski, Acta Met. 38 (1990) 489-9.
[7] B. Roebuck, J. Lord, Materials Science & Technology. 6 (1990) 1199-1209.
[8] A.C. van Gorp, K.M. Mussert, M. Janssen, A. Bakker, S. van der Zwaag, J. Test & Eval. 29 (2001) 146-154.
[9] B. Roebuck, J. Lord, Materials Science & Technology. 6 (1990) 1199-1209.
[10] J.E. lpina Perez, A.A. Yawny, R. Stuke, Oliver.C. Gonzalez, Materials Research. 3 (2000) 74-78.
[11] A. Mortensen, Proc. Int. Conf. Fabrication of Particulate Reinforced Metal Composites, Quebec, Canada, (1990) 217-23.
[12] J.E. Allison, G.S. Cole, JOM. 45 (1993) 19-25.
[13] J. Doychak, JOM. 44 (1992) 46-51.
[14] T.L. Anderson, Fracture Mechanics-Fundamentals and applications, CRS press LLC, 1995.
[15] T.W. Clyne., P.J. Withers, An Introduction to Metal matrix Composites, Cambridge University Press, 1993.
[16] J. Lord, Fracture Toughness Test Methods For PRM, MMC ASSESS EU Network MMC-Assess – Thematic Network.
[17] Annual Book of ASTM Standards, ‘Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials’, ASTM E399-90, 1997.
[18] R.D. Goolsby, L.K. Austin, Proc. 7th lnt. Conf on 'Fracture', ICF7, Houston, TX, University of Houston. 4 (1989) 2423-2435.
[19] R.J. Arsenault, S. Fishman, M. Taya, Progress in Materials Science. 38 (1994) 1-157.
[20] J.N. Hall, J.W. Jones, A.K. Sachdev, Materials Science and Engineering: A. 183 (1994) 69-80.
[21] P.M. Singh, J.J. Lewandowski, Met. Trans. A. 24A (1993) 2451-2464.
[22] D.P. Myriounis, S.T. Hasan, T.E. Matikas, Advanced Composite Letters 17 (2008).
[23] D.P. Myriounis, S.T. Hasan, T.E. Matikas, Composite Interfaces Journal. 15 (2008) 495-514.
[24] D.P. Myriounis, S.T. Hasan, T.E. Matikas, J. ASTM Int. 5 (2008).
[25] X.V. Maldague, Theory and practice of Infrared technology for nondestructive testing, John Wiley&Sons, Inc, New York, 2001.
[26] ASM Handbook, Nondestructive Evaluation and Quality Control, 17 (1989).
[27] R.J. Zhang, Z. Wang, C. Simpson, Materials Science and Engineering: A. 148 (1991) 53-66.