# TENSILE TESTS IN SEM OF 6061/SiC COMPOSITES

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#### **ABSTRACT**

The fracture behaviour of 6061/SiC composites was investigated by providing dynamic observations of the crack initiation and propagation mechanisms during in-situ SEM tensile test. It has been used an industrial 6061 alloy reinforced with 10 % by volume of 30  $\mu$ m silicon carbide particulates; four types of SiC particulates have been employed, each of them having 0.3 vol.% of amorphous oxide coating of Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, SiO<sub>2</sub> (hydrophilic), SiO<sub>2</sub> (hydrophobic), in addition to the uncoated particulates.

Our work evidences that the fracture process is matrix controlled, cracked or decohered particulates being found only along the principal crack in a narrow zone.

#### **KEYWORDS**

Tensile tests, oxide coated particulates, metal matrix composites.

#### INTRODUCTION

A serious drawback of MMC materials lies in their relatively low tensile ductilities and fracture toughness values.

Typically, additions of SiC to an aluminium alloy can decrease its strain to failure by an order of magnitude, even at low volume fraction levels.

Attempts to find detailed failure mechanisms have indicated that several factors including the reinforcement matrix interface, the cracking of reinforcements and the presence of intermetallic compounds and microstructural inhomogeneities play important roles in composite failure.

Even for the same global mechanical properties, different mechanisms have been observed operating in metal matrix composite materials, as a function of: the dimensions of particulates, the thermomechanical processing route, the intermetallic particle properties, the particulate/ matrix interface / interphase chemical composition and properties, etc.

In particular, in the case of Al/SiC composites, chemical reactions between Al alloys and pretreated SiC particulates can produce some chemical compounds at the particle / matrix interphase which may reduce the interfacial strength and allow an interface decohesion during loading. So, an improvement of ductility has been obtained by oxidizing the SiC particulates of a 356 + 10 % SiC composite (Ribes et al., 1990).

Taking into account this result and the different influence of SiC oxide coatings (Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, SiO<sub>2</sub>) on the degradation of SiC particulates by liquid aluminium during Al / SiC composite fabrication (Thanh and Suéry, 1991), the authors have proposed to study the influence of these different oxide coatings, present on the surface of SiC particulates, on the failure process, by providing dynamic observations of the crack initiation and propagation mechanisms in a 6061/SiC particulate composite during *in-situ* SEM tensile tests.

## MATERIALS AND EXPERIMENTAL METHODS

#### Materials

In this investigation a 6061 industrial alloy reinforced with 10 % by volume of 30µm SiC particulates has been studied.

The determined matrix chemical composition and the standard chemical composition of the 6061 alloy are given in Table 1.

Table 1 - The 6061 alloy chemical composition (wt %)

	Si	Mg	Fe	Cu	Mn	Zn
experimental	0.53	1.24	0.30	0.50		0.056
standard	0.15 - 0.80	0.80 - 1.20	0.70	0.15 - 0.40	0.15	0.250

To assess the effects of particulate coating on the damage evolution during tensile tests, four types of SiC particulates have been employed, each of them having 0.3vol. % of amorphous oxide coating: Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, SiO<sub>2</sub> (hydrophilic), SiO<sub>2</sub> (hydrophobic) and also particulates without coating.

For all the composites the fabrication method has been composating, followed by rapid complete remelting of the alloys before pressurised solidification at 100MPa. The mechanical stirring duration, in the semi-solid state, has been 10 min.

After casting, from each 5 ingots, disks with a 3 mm thickness have been mechanically cut. The tensile specimens for in-situ SEM tensile tests were machined by electroerosion from these disks, in the as - cast and heat-treated conditions. The geometry of the tensile specimens is presented in Fig. 1 and the heat treatment conditions are the following:

O - 603 K / 180 min / air;

T4 - 803 K / 120 min / water;

T6 - 803 K / 120 min/ water + 448 K/480 min/air.

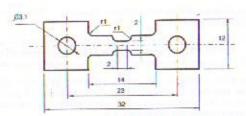


Fig.1 - Tensile specimen geometry

Before tensile tests, both flat specimen surfaces were prepared by metallographic techniques for optical and scanning electron microscopies i.e., they were polished with emery paper (300 - 1200) and down to  $1\,\mu\mathrm{m}$  diamond particulates.

#### Tensile Tests in SEM

The tensile tests are performed thanks to a loading stage type TS 250 inserted into the JEOL JSM 6400 vacuum chamber, replacing the standard sample stage. The maximum stage load capacity is 2.5kN and the load cell showed a good linearity during calibration experiments, up to the maximum load. All the tensile tests have been performed discontinuously and with a 11.4  $\mu$ m /s velocity.

## RESULTS AND DISCUSSION

In all three heat treatment conditions the matrix microstructure consists in a  $\alpha$  phase (solid solution of Si in Al), with an average grain size of 20  $\mu$ m and an eutectic phase disposed at the grain boundaries and sometimes on the surface of SiC particulates. The eutectic phase width varies between 2 and  $10\,\mu$ m and the EDX analysis shows mainly the presence of Fe, but also of Si, Cu, Cr. In the case of T4 and T6 specimens Mg<sub>2</sub>Si phases are also present on the grain boundaries, Fig. 3.

In all the studied composites the distribution of SiC particulates is relatively homogeneous (Fig. 2) and the particulate density on the specimen surface varies between 300 and 360 particulates/mm<sup>2</sup>.

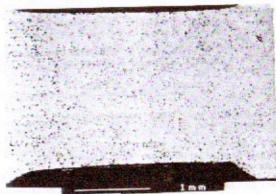


Fig. 2 - The flat surface of a 6061/SiC composite specimen before in-situ SEM tensile testing

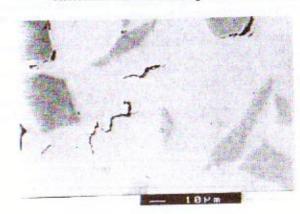


Fig. 3 - Intergranular crack in a T4 specimen

The microhardness HV of the solid solution had been determined with a 147 N load applied during 15 seconds and the obtained values are comparable with other 6061/SiC composites, with a similar heat treatment (45 - 47 HV for O, 67.79 HV for T4, 102 - 114 HV for T6 specimens. Due to the reduced specimen dimensions the mechanical characteristics (U.T.S., elongation) obtained during the tests are not representative of the analysed MMC.

However, from these results one can notice the independence of the strength and elongation values on the particulate coating compositions except for the elongation of the composite reinforced with Al2O3 coated particulates which is much greater than the other composite elongations. The reason for this is not known at present.

Also, the microstructural properties and damage evolution during the tests are not influenced by the particulate oxide coating. Consequently, the fracture evolution process will be analysed independently of particulate coating.

By studying the tensile properties of a similar MMC (6061-10 % SiC), but using tensile specimens cut from extruded bars, Lloyd, 1990, had observed that SiC particulate fracture occurs throughout the gauge length and the number of fractured particulates increases with strain. In our experiments, as previously mentioned by You et al., 1987, the composite fracture is matrix controlled, and moreover, the cracked or decohered particulates are found only in a narrow zone, with an average width of 200µm along the principal crack.

The number of cracked particulates (Nc) is greater for T4 specimens (Nc = 10 - 17) in comparison with O (Nc=3-14) specimens. Considering, as before, that one can find cracked particulates only along the principal crack, we obtain the following densities for the three heat treatment conditions: 26 particulates/mm<sup>2</sup> for T4, 15 particulates/mm<sup>T</sup> for T6 and 5 particulates /mm<sup>2</sup> for O specimens.

In the case of decohered particulates (Nd) their number does not depend on the heat treatment conditions and has an average value of Nd = 2 - 7.

So, in our experiments for all three heat treatment conditions the intergranular fracture characteristics of the fracture process are prevailing, Fig.3. The first crack appears on the specimen surface, by coalescence of voids nucleated near the particulates at a  $2-10 \,\mu m$  distance (Fig. 4), either by matrix shearing, or by cracking of Mg2Si precipitates, or by cracking of eutectic phase.

Sometimes, the voids nucleated on the SiC particulate boundaries, and also the cracking of the eutectic phase present on these boundaries is the principal reason for void nucleation (Fig. 5).



Fig.4 - Voids nucleated near SiC particulates

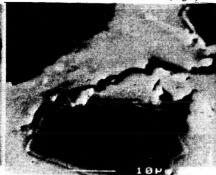


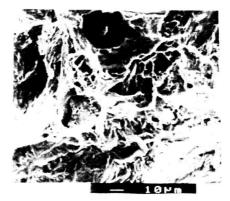
Fig.5 - Voids nucleated by cracking of eutectic phase (bright in the figure)

Previously, a preferential void nucleation in regions of high volume fraction of particulates (Lloyd and Morris, 1990, Hunt et al., 1987) had been observed but in our experiments most of the cracks propagate between particulates placed at a  $4-20\,\mu\mathrm{m}$  distance and not inside the particulate clusters. The distance between the particulates along the crack path decreases as microhardness increases from O to T6, suggesting the influence of shear band nucleation near particulates, this can be the first step towards crack nucleation.

The role of shear bands on the crack nucleation has been mentioned by Ribes et al., 1990, in the case of tensile tests performed by using MMC (A 356 + 10 % SiC) specimens in as cast condition but has not be mentioned by the other authors which have used specimens machined from deformed products (extruded, rolled).

The dominant role of intergranular fracture to the damage evolution during the tensile tests has been confirmed by the fracture surface analysis.

On these surfaces the cracked or decohered SiC particulates are surrounded by an intergranular or shear fracture zone. In Fig. 6, 7, the rare dimples with a 5 -  $10 \mu m$  diameter, which can be noticed on the fracture surface are related to the fracture of precipitates of  $1-3\mu m$ .



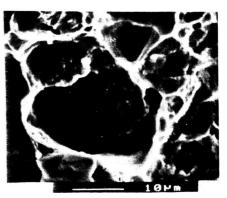


Fig. 6 - Shear and intergranular fracture in a T4 Fig.7 - Decohered SiC particulates and small dimples in a T6 specimen

Having in view the comparison of our results to Lloyd's results we have used the following two ratios, the value of which are given in Table 2:

Total particle length along the fracture Fracture length

and

Total particle length along a matrix line Matrix length

Table 2 - The densities of the particulates in the matrix and on the fracture surface.

COMPOSITE	F	P	MP		
	(a)	(b)	(a) '	(b)	
6061 + 10 % SiC, T4	0.15	0.18	0.17	0.13	
6061 + 10 % SiC, T6	0.14	0.18	0.18	0.13	

(a) - our results:

(b) - Lloyd et al., 1990, results.

It is obvious from the above results that in the case of the present work, the particulates density on the fracture surface in both heat treatment conditions is less than in the matrix, conversely to the Lloyd results which indicate a higher particulate density on the fracture surface in the aged condition and comparable in the solution treated condition.

## SUMMARY

The following conclusions can be drawn:

- During in-situ SEM tensile tests the intergranular fracture characteristics prevail; consequently
  the influence of particulate coating on the fracture process has not been noticed.
- . The crack nucleation and propagation is not connected to particulate clusters.
- The cracked or decohered particulates are found only near the main crack in a narrow zone of 200 m average width. Inside this zone the number of cracked particulates is greater for T4 depend on the heat treatment and particulate coating composition.
- The total number of particulates is higher on a polished surface than on the fracture surface, in spite of relatively large particulates (30  $\mu$ m) which have been used in experiments.

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## REFERENCES

HUNT W.N.Jr., RICHMOND O., YOUNG R.D., (1987), \* Fracture initiation in particle hardened materials with high volume fraction\* In: ICCM & ECCM (cd. Mathews F.L., Bushell M.C.R., Hodgkinson J.M., Morton J.) 2, Elsevier Applied Science, pp. 2209 - 2223.

LLOYD D.J., MORRIS P.L. (1990), 'Tensile fracture of 6061 - 15 % Al<sub>2</sub>O<sub>3</sub> particulate', ASM conference, Montreal, Canada, pp. 241 - 250.

LLOYD D.J. (1991), "Aspects of fracture in particulate reinforced metal matrix composites", Acta Metall, Mater., 39, pp 59 - 71.

RIBES H., Da SILVA R., SUÉRY M., BRETHEAU T., (1990), "Effect of interfacial oxide layer in Al-SiC particle composites on bond strength and mechanical behaviour", Materials Science and Technology, 6, pp. 621 - 628.

THANH L.N., SUÉRY M., (1991), "Influence of oxide coating on chemical stability of SiC particles in liquid aluminium" Scripta Metall. Mater. pp. 2781 · 2786.

YOU C.P., THOMPSON A.W., BERNSTEIN I.M., (1987), "Proposed failure mechanism in discontinuously reinforced aluminium alloy", Scripta Metall. 2L, pp.181 - 185.