

# Oxidation behaviour of carbon/aluminium and SiC/aluminium composites

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Isothermal oxidation of 6061 Al, reinforced with chopped carbon fibres and SiC particles, was studied to investigate the applicability of these composites in the temperature range 300–500 °C, in terms of their degradation due to oxidation. Carbon/aluminium composite suffered a tremendous loss in weight at 350 °C. The extent of the damage due to oxidation for SiC/Al composites depended upon the concentration of nucleation sites, which formed the interface between the composite and the matrix. The number of such sites depended upon the volume fraction and size of the dispersed particles. Analysis of the oxide scale was carried out using SEM/EDAX and X-ray diffraction. The deterioration in strength of the composites, due to oxidation, was determined by tensile testing of exposed specimens.

## 1. Introduction

Discontinuously reinforced aluminium matrix composites find potential applications in the automobile engine parts, such as drive shafts, cylinders, pistons, and brake rotors [1]. An investigation relating to the temperature profiles of the piston area in a diesel engine has shown that the temperature can reach as high as 400 °C in certain regions of the piston [2]. Because the piston and cylinder areas are exposed to air and fuel at high temperatures, the material used should be quite resistant to oxidation. In order to design better composite components, the performance of the composites used should be fully evaluated in terms of their degradation in oxidizing environments. With this objective, an attempt has been made to study the oxidation behaviour of 6061 Al matrix reinforced with carbon fibres and SiC particles in the temperature range 350–500 °C. The effect of particle size and concentration on the oxidation behaviour has been observed using thermogravimetric tests, scanning electron microscopy/energy dispersive analysis of X-rays (SEM/EDAX analysis), X-ray diffraction analysis and by measuring the degradation in mechanical properties.

Very little work has been reported on the oxidation behaviour of carbon- or SiC-reinforced metal matrix composites [3–5]. However, much work has been published on the SiC–ceramic composites or tungsten or alumina/Ni<sub>3</sub>Al composites, which are considered for very high-temperature applications [6]. This work, therefore, is a first attempt to study the behaviour of aluminium-based metal matrix composites (MMC) to be used for moderate-temperature applications.

## 2. Experimental procedure

### 2.1. Materials

Both C/Al and SiC/Al composites were prepared in the Metallurgical Engineering Department Laboratory of this Institute. 6061 Al, carbon fibres and SiC particles with sizes 5, 15 and 35 µm were used for preparation. Table I gives details of the specimens prepared with the type and concentration of the reinforced material, the process used for fabrication and the post-heat treatment, if any, given to the final composite samples.

### 2.2. Oxidation tests

The samples, dimensions 10 mm diameter and thickness 2 mm, were polished to 1000 grit finish using series of emery papers. The specimens after polishing were washed in acetone, dried and weighed before subjecting them to oxidation tests. Oxidation kinetics were determined by measuring the weight changes as a function of time after regular intervals of time, at a particular temperature. For C/Al, the test was carried out at 350 °C, while for the SiC/Al composite sample, oxidation was carried out at 400 and 500 °C.

Scanning electron microscopy (SEM) was used to observe the reinforced composites before and after oxidation to determine the site of oxidation and the topography and morphology of the oxide products formed. X-ray diffraction was used to identify the oxide formed.

Degradation in the mechanical properties of the composites, as a result of exposure to the oxidizing environment, was determined by conducting oxidation tests on miniature tensile specimens, prepared as

per ASTM E-8 specification, at 400 °C. Yield stress, ultimate tensile stress (UTS) and per cent elongation were determined using a Housenfield tensometer at a crosshead speed of 0.1 mm min<sup>-1</sup>

### 3. Results and discussion

#### 3.1. Microstructure of the composites

For observing the microstructure of composite samples, the samples were mounted in epoxy resin, polished to a fine finish using a series of emery papers, and finally with alumina suspension. Optical micrographs of the different samples are shown in Fig. 1. The dark regions in the matrix are the reinforced carbon fibres (Fig. 1a) or SiC particles (Fig. 1b-d).

TABLE I Details of the materials used in the present project

Sample	Form of the composite
A	As-extruded, extrusion ratio 1:17
4% C/6061Al, rheocasting	
A1	Solution-treated, quenched in cold water and aged at 180 °C for 8 h
4% C/6061Al, rheocasting	
C	As-extruded, extrusion ratio 1:17
15% C/6061Al, rheocasting	
C2	Solution-treated, quenched in cold water and aged at 180 °C for 8 h
15% C/6061Al, rheocasting	
O	As-extruded, extrusion ratio 1:17
6061Al,	
O2	Solution-treated, quenched in cold water and aged at 180 °C for 8 h
6061Al,	
AS	As-extruded, extrusion ratio 1:17
35 u/5% SiCp/6061Al, squeeze casting	
G	As-extruded, extrusion ratio 1:17
5 u/5% SiCp/6061Al, squeeze casting	
I	As-extruded, extrusion ratio 1:17
5 u/15% SiCp/6061Al, squeeze casting	

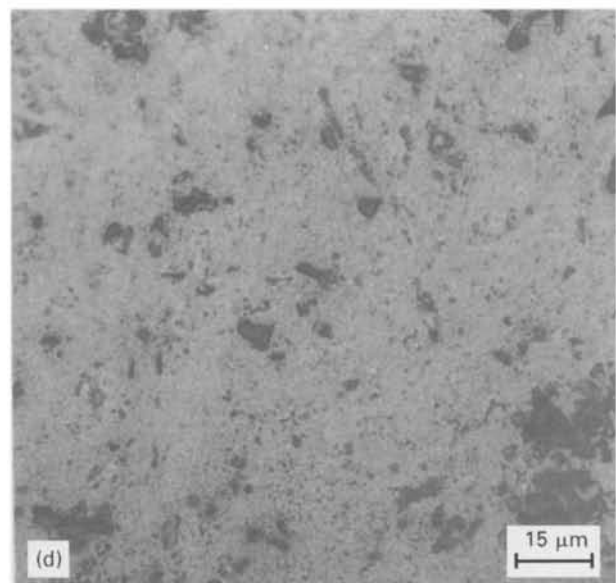
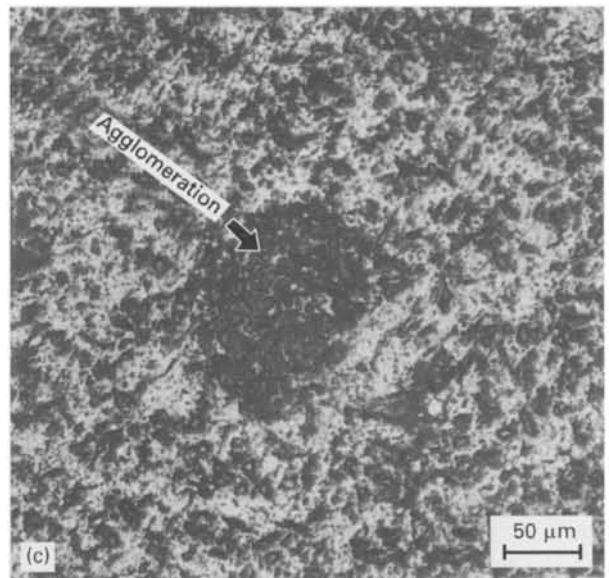
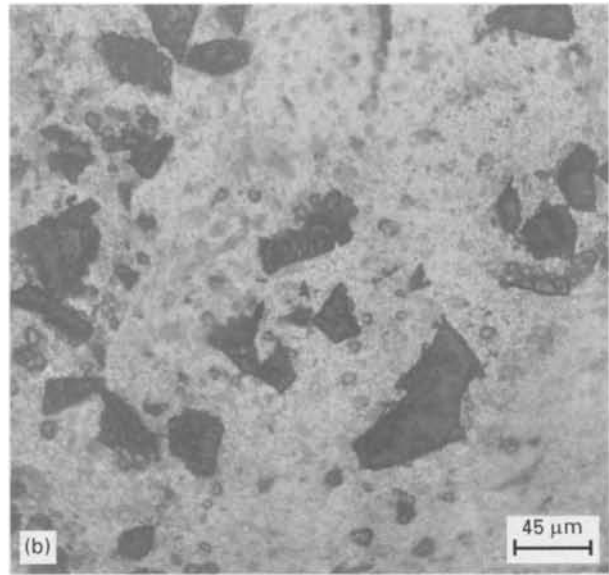
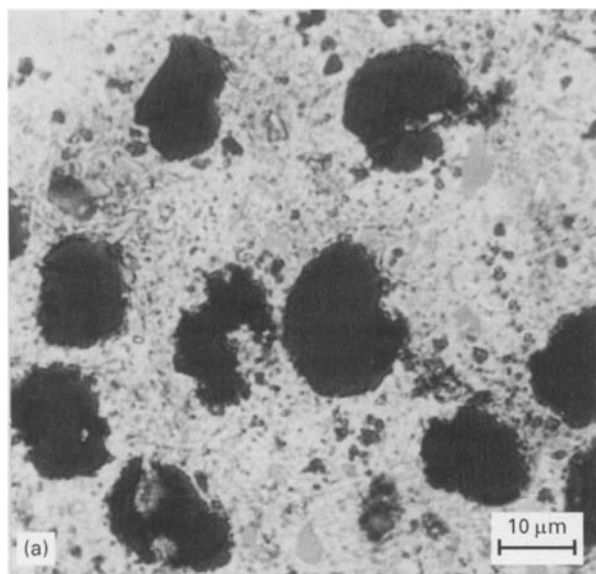


Figure 1 Optical micrographs showing the microstructures of various composites: (a) 15% C/Al; (b) 15%, 35 μm SiC/Al; (c) 15%, 5 μm SiC/Al; and (d) 5%, 5 μm SiC/Al.

Second phases are also visible (light-grey region). In a few samples, some agglomerations were also found (Fig. 1c).

### 3.2. Oxidation results

#### 3.2.1. Carbon/aluminium composite

Fig. 2 shows the weight change versus time plots for the oxidation of C/Al composite at 350 °C. Curves (a) and (b) represent the oxidation behaviour of the matrix alloy in the as-extruded condition and in the heat-treated condition, respectively. A small weight gain observed is due to the formation of an alumina layer, confirmed by X-ray diffraction analysis.

Other samples, as-extruded, 15 vol % carbon composite (c), and heat-treated (d), as well as extruded, 4 vol % carbon composite (e), and heat-treated (f),

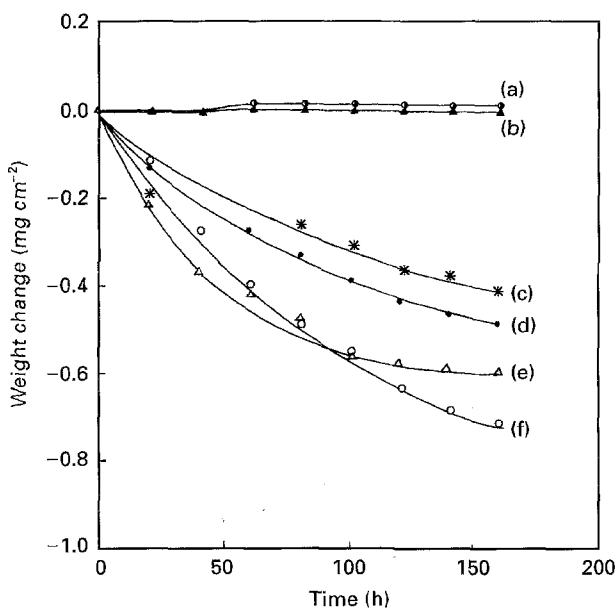
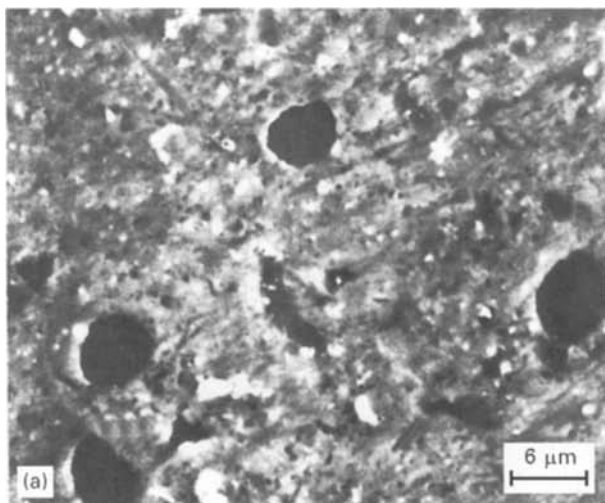


Figure 2 Weight change versus time plot for the oxidation of aluminium and various carbon-reinforced aluminium composites in air at 350 °C. (○) 4% C/Al as-extruded, (△) 4% C/Al heat treated, (●) 15% C/Al as-extruded, (★) 15% C/Al heat treated, (◐) 6061 Al as-extruded, (▲) 6061 Al heat treated.



respectively, show loss in weight. This can be attributed to the loss of carbon as a result of oxidation. The sample containing 4 vol % C fibre, shows larger weight loss than the sample containing 15 vol % C. This can be explained if one observes the microstructures of the oxidized samples. Fig. 3 shows the microstructures of the samples containing 4 and 15 vol % C as the reinforced phase after 10 min oxidation at 350 °C. Two important conclusions can be made from these observations:

(i) a lower extent of oxidation on the sample having 4 vol % C than on the sample having 15 vol % C;

(ii) oxidation has dominated mostly at the interface between the matrix and the reinforced material. The sample having a larger concentration of reinforced material has larger numbers of such sites and therefore oxidizes more and has a relatively thicker scale on the matrix material. Thus a larger weight gain due to the thicker scale formation is compensated by the large weight loss due to carbon loss during oxidation.

In the case of the 4 vol % sample, weight loss due to carbon is more predominant compared to weight gain due to scale formation. Hence the overall weight loss is much larger compared to the sample containing 15 vol % C where, inspite of the fact that the weight loss due to carbon loss should be more, it is partially compensated by larger weight gain of matrix due to oxide layer formation.

Further, it was found that the oxide scale cracked severely and the carbon fibre oxidized, leaving behind pores or voids. This is shown in Fig. 4 for the 15 vol % C sample, oxidized at 350 °C for 150 h.

#### 3.2.2. SiC/Al composite

Fig. 5 shows the weight gain versus time plot for the oxidation of SiC/Al composites. Oxidation of the matrix alloy in the as-extruded condition (curve a) is compared with the oxidation of composites: curve b for 5 vol % and 5 μm SiC particles, curve c for 15 vol % with 5 μm SiC particles, and curve d for 15 vol % and 35 μm SiC particles, at 400 °C. As can be seen,

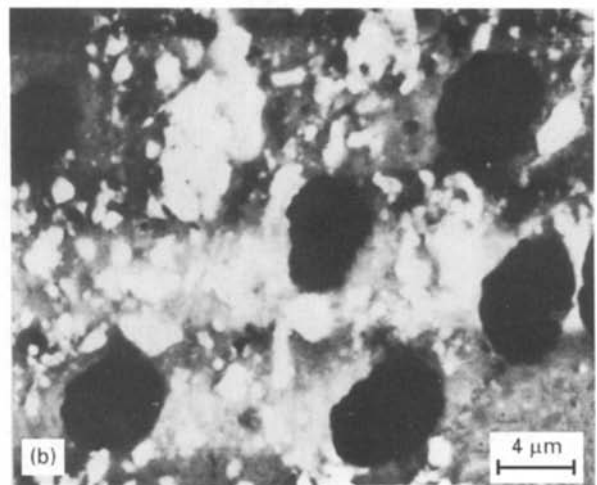


Figure 3 Scanning electron micrographs showing the morphologies of (a) 4% C and (b) 15% C aluminium composites after oxidation in air at 350 °C for 10 min.

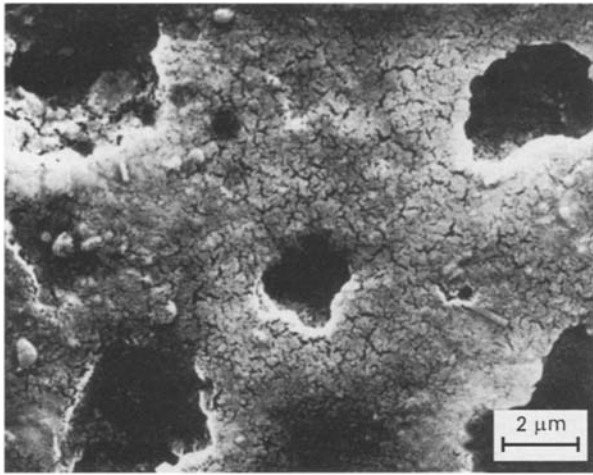


Figure 4 Scanning electron micrograph showing the cracking of the scale formed on 15% C/Al composite, after oxidation at 350 °C in air for 150 h.

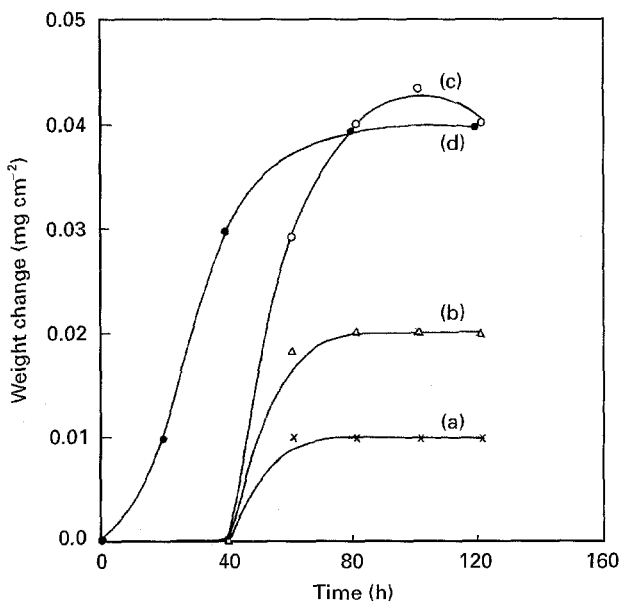


Figure 5 Weight change versus time plot for the oxidation of aluminium and various SiC reinforced aluminium composites in air at 400 °C, (●) 15%, 35 μm SiC-Al, (○) 15%, 3 μm SiC-Al; (Δ) 5%, 5 μm SiC-Al; (x) 6061 Al as-extruded.

the presence of a second phase as dispersed particles significantly changes the oxidation rate. Both the size and concentration of dispersed particles affect the oxidation behaviour. A significant weight change on the 5 vol % sample with 5 μm SiC particles occurred only after a sufficient time, which is required perhaps for the nucleation to occur at the interface. Once the concentration increases to 15 vol %, the weight increases more rapidly; however, nucleation still takes the same time. By increasing the particle size, a significant weight change is observed right from the early stage of oxidation.

Fig. 6 compares the surface morphologies of the unoxidized and the oxidized samples of 15 vol % SiC (35 μm) composite after oxidation at 400 °C for 120 h, and Fig. 7 depicts the significant scale formation on the matrix material during the oxidation of the composite at 400 °C for 120 h.

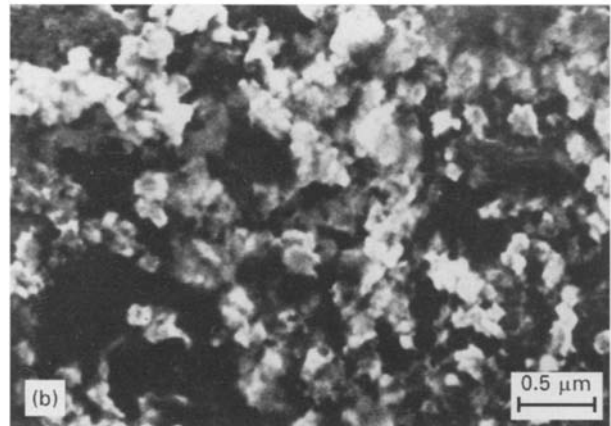
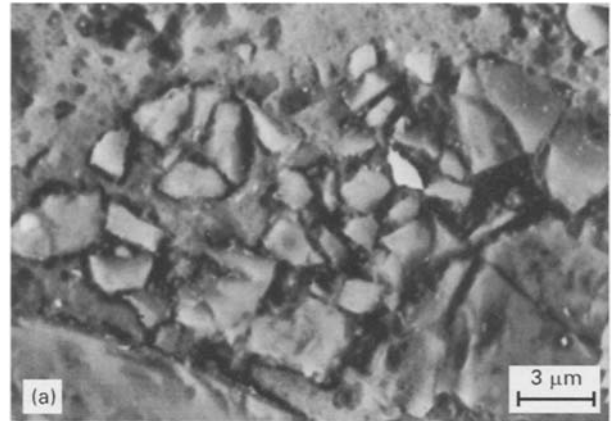


Figure 6 Scanning electron micrographs showing the microstructures of (a) unoxidized and (b) oxidized 15%, 35 μm SiC/Al at 400 °C in air for 120 h.

The extent of reaction increased as the temperature of oxidation was increased to 500 °C. Fig. 8 shows the oxidation of the three composites at 500 °C, maintaining the same trend as that at 400 °C, confirming the role of both the concentration and the size of the dispersed phase in influencing the oxidation rate. Secondly, there was no incubation period for nucleation, which was instantaneous even for 5 μm sized particles. The overall weight gains were an order of magnitude more than that at 400 °C.

### 3.2.3. Effect of oxidation on the tensile properties

Table II lists the various values obtained for UTS, yield stress and per cent elongation at fracture for two composite specimens which had particles of same size, i.e. 5 μm but had different composition, namely 5 and 15 vol % SiC. These were exposed for 50 and 100 h at 400 °C in air. For the 5 vol % sample, the value of UTS decreased from 211 MPa for the unoxidized sample to 173.5 and 157.8 MPa after exposure for 50 and 100 h, respectively, and for the 15 vol % sample, this drop was from 237.8 MPa for unoxidized sample to 205.1 and 189.4 MPa after exposure of 50 and 100 h, respectively.

This can be explained in the light of oxidation results discussed above (Fig. 5). It was seen that no significant weight change occurred during the first

50 h exposure at this temperature. Therefore, a significant decrease in UTS in the first 50 h exposure could be due to the overageing of 6061 Al alloy. It is a precipitation-strengthened alloy (ageing temperature and time being 175 °C and 8 h, respectively). The subsequent decrease in strength can be attributed to a combined effect of ageing as well as oxidation.

Fractographic analysis of the failed specimens after the completion of the test showed a ductile failure of the 15 vol % sample, exposed for 50 h. Voids initiated at the particle matrix interface (Fig. 9). Mixed failure could be seen for the same sample oxidized for 100 h (Fig. 10). This can be attributed to the increase in

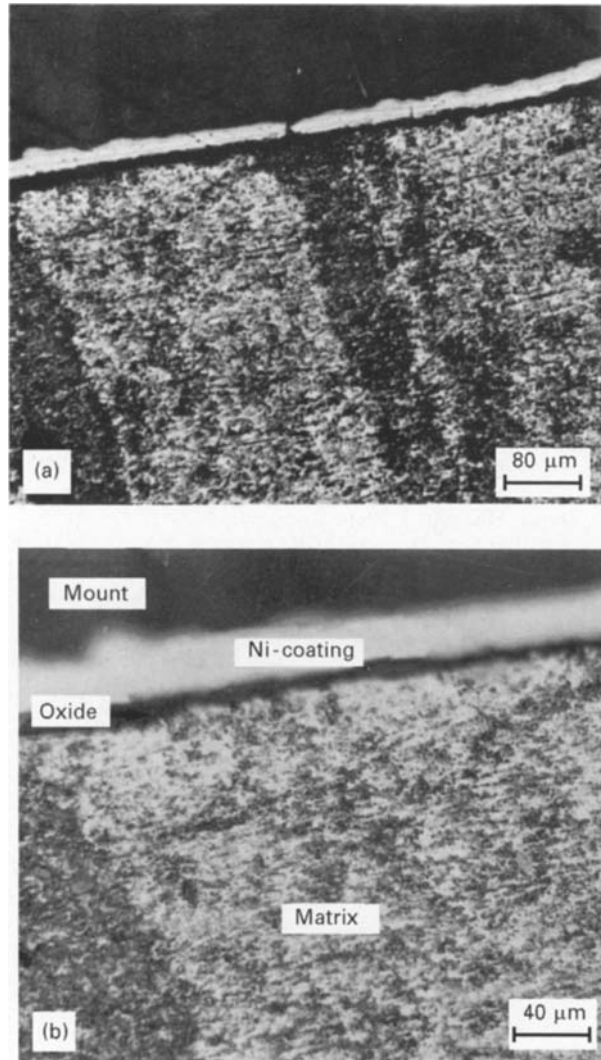


Figure 7 Scanning electron micrographs showing the scale cross-section at two different magnifications for 15%, 35 μm SiC/Al sample oxidized at 400 °C in air for 120 h.

oxidation rate and resistance to plastic deformation by the high volume fraction of the reinforcement.

#### 4. Conclusions

The preliminary work, which was an attempt to investigate the deterioration of metal matrix composites, used for several applications at ambient temperatures, showed that oxidation at relatively higher temperatures caused deterioration which was due to the reaction of the reinforced material, for example in the case of C/Al composite or due to enhanced reaction at the interface (in the case of SiC/Al composite). Some of the conclusions derived from this work are as follows.

1. The interface between the reinforcement and the matrix was found to be the main site for nucleation.
2. In C/Al composite, the reaction of carbon, which is the main strengthening element, took place with the oxygen, resulting in a loss of weight. This composite, therefore, is not suitable for a temperature of 300 °C or above.
3. SiC reinforcement was more stable; however, the extent of oxidation depends upon the size and concentration of SiC particles. Oxidation rate increased with increase in both size and number of dispersed particles. This was as a result of increased interface boundary, resulting in the increased nucleation sites.

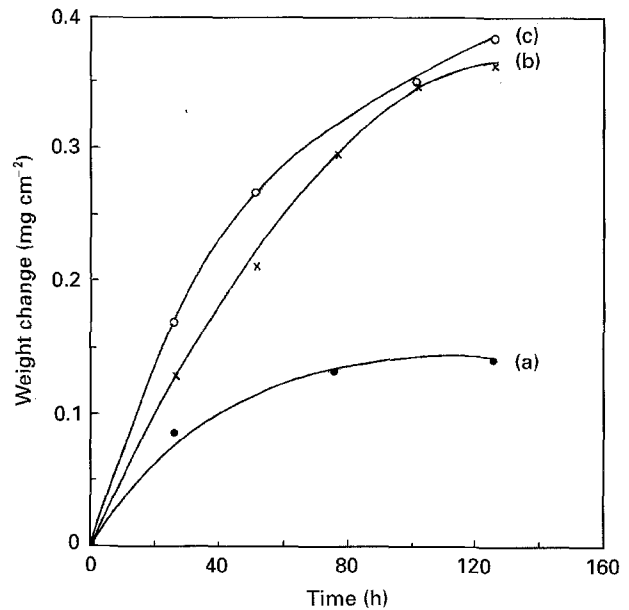


Figure 8 Weight change versus time plot for the oxidation of various SiC reinforced aluminium composites in air at 500 °C. (●) 5%, 5 μm SiC-Al; (×) 15%, 5 μm SiC-Al; (○) 15%, 35 μm SiC-Al.

TABLE II Effect of exposure time on the tensile properties of SiC/Al composites

Composite	Exposure time (h)	UTS (MPa)	YS (MPa)	Elongation at fracture (%)
G (5%, 5 μm SiC/Al)	0	211	100.25	11.2
	50	173.58	78.9	5
	100	157.8	78.9	2.75
I (15%, 5 μm SiC/Al)	0	237.85	144.5	1.98
	50	205.14	104.15	1.25
	100	189.36	—	—

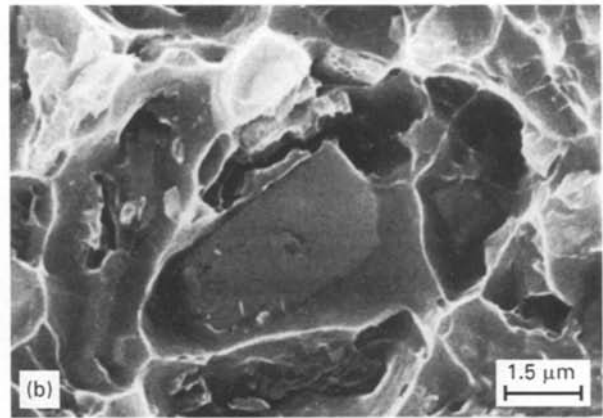
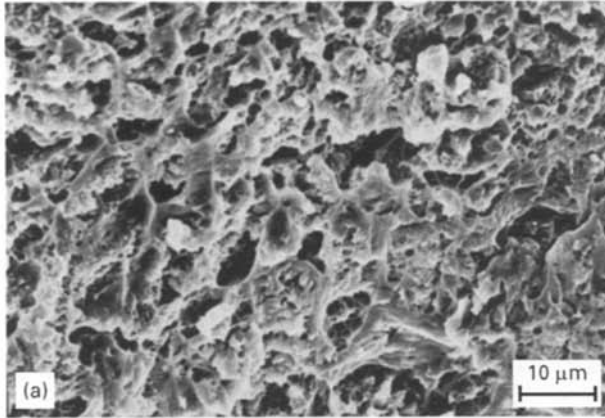


Figure 9 Scanning electron micrographs showing (a) the fractured surface and (b) voids at the particle/matrix interface, of 15% SiC/Al composite, oxidized at 400°C for 50 h.

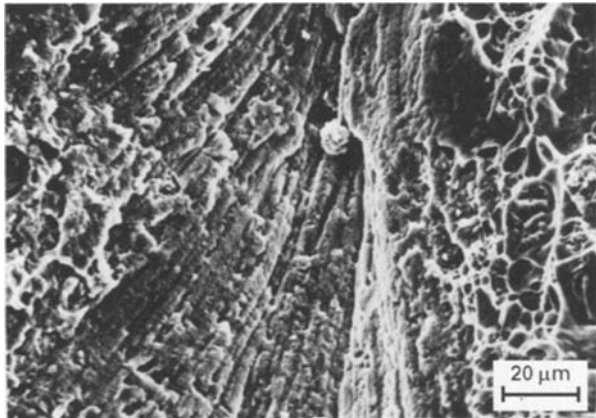


Figure 10 Scanning electron micrographs showing the mixed mode fracture observed after oxidation of 15% SiC/Al composite, at 400°C for 100 h.

Oxidation can be prevented using a suitable coating material.

4. The oxidation rate of SiC/Al composite increased by an order of magnitude at 500°C compared to that at 400°C.

5. The overall strength of the SiC/Al composites decreased with increase in exposure time. This can be attributed to the overageing at the beginning, followed by oxidation at a later time.

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Received 15 April 1994  
and accepted 21 February 1995