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Influence of fibre-matrix interactions and of matrix microstructure on the mechanical properties of Aluminium-based MMCs reinforced with Altex fibers and processed by medium pressure infiltration

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The introduction of alloying elements in an aluminium matrix may have a detrimental effect on the mechanical properties of unidirectional composites. The aim of this work was to establish a correlation between the mechanical properties of Aluminium based/Altex fibers MMCs processed by medium pressure infiltration and their microstructure, the composition of the matrix and the mechanical properties of the fibers. Al-Mg, Al-Si, Al-Mg-Si and Al-Mg-Si-Cu matrices were studied. The microstructure of the composites was characterized by optical and electron microscopy observations. Transmission electron microscopy was also used to investigate the reactivity phenomena likely to occur between the matrix and the fibers. Furthermore, the tensile properties of the fibers after introduction in the metal matrix were measured by single fiber tests, to assess the degradation of the fibers. The tensile properties of the composites were also determined both in longitudinal and transverse direction. They were interpreted referring to the microstructural state of the composites and to the tensile properties of the fibers. Lastly, the detrimental effects due to the alloying elements (solid or liquid state reactivity, presence of brittle phases, matrix hardening) were classified according to their importance.

1. Introduction

Age-hardenable alloys are generally used to ensure good mechanical properties of the non reinforced regions of real structural components partially reinforced with Aluminium-based metal matrix composites (MMCs). However, the use of an alloyed matrix may have several detrimental consequences on the mechanical properties of the reinforced region. According to the processing conditions, damage of the fibers in consequence of chemical interactions between alloying elements and fibers may occur. Moreover, the presence of brittle intermetallic precipitates at the fiber-matrix interface or in the matrix may induce a premature catastrophic failure of the material.

Thus, the aim of this work is to study the importance of the consequences of these phenomena on the mechanical properties of Al-Mg-Si/alumina-silica fiber MMCs processed by medium pressure infiltration.

2. Materials and experimental procedure

2.1. Materials

The materials investigated in this work consist of aluminium-based matrix composites reinforced with about 35 vol % alumina-silica Altex fibers. These continuous fibers are produced by Sumitomo Chemical. Their composition and main properties [1] are given in Table I.

As matrix, various aluminium alloys were employed: binary Al-Mg and Al-Si alloys and ternary Al-Mg-Si alloys. These alloys were chosen in order to separate clearly the influence of the different factors of degradation of the mechanical properties of the composite materials. The practical problem was investigated by using a 6013 industrial alloy. A pure aluminium matrix was also used to produce a reference composite. The mean chemical composition of all aluminium matrices used in this work, after the processing of composites, is reported in Table II.

The composite materials were processed using a medium pressure infiltration technique (Fig. 1) [2]. The details of this process are the following: fibers tows are rolled onto a preform mandrel which is part of the casting mold, so as to get the expected fiber volume fraction. The mold itself is placed in a sealed steel can which is inserted in the infiltration unit, where is also placed the metal to infiltrate. A dynamic vacuum of

TABLE I Composition and main properties of the Altex fibers given by Sumitomo [1]

Chemical composition	Density (g/cm^3)	Diameter (μm)	E (GPa)	σ_r (MPa)	ε_r (%)	Coefficient of thermal expansion/K
85% γ Al_2O_3-15% SiO_2	3.25	15	200	1500[a]	0.8	4.5×10^{-6}

[a]Gauge length: 100 mm.

TABLE II Mean chemical composition of the alloys used as matrix after processing

Materials	Cu (%)	Si (%)	Mg (%)	Fe (%)
Al	0.005	0.005	<0.015	0.02
Al-1wt%Mg	///	///	0.9	///
Al-1wt%Si	///	0.9	0.01	0.07
Al-3wt%Si	///	3.15	0.03	0.12
Al-1wt%Mg-1wt%Si	///	0.96	0.96	0.12
Al-4wt%Si-0.6wt%Mg (AS4G0,6)	0.002	4.2	0.54	0.12
A356 (AS7G03)	0.02	6.9	0.35	0.06
A357 (AS7G06)	0.03	6.76	0.47	0.096
6013	0.6	0.67	0.9	0.3

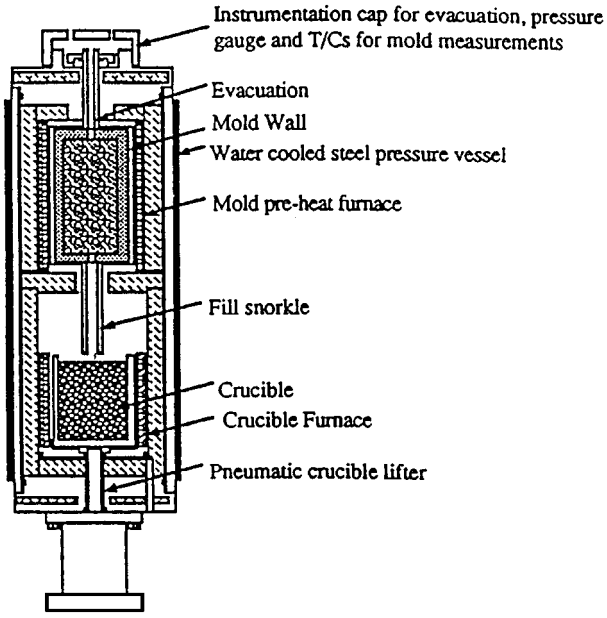

Figure 1 Principle of the medium pressure infiltration technique.

approximately 1 Torr is then established. The fiber preform and the metal are adjusted to the desired temperatures, respectively T_f and T_m. Then, the melt is lifted to cover the snorkel melt inlet. Nitrogen gas pressure is then applied to force the aluminium to infiltrate fully the preform and the mold. A directional solidification of the melt is obtained from the top to the bottom of the mold by means of a metallic plug placed at the top of the mold. Table III gives the parameters chosen for the infiltration of the preforms by the molten metals (mean temperature of the preform and of the molten metal at the beginning of the processing), as well as the contact times between the ceramic reinforcement and the liquid metal at three different locations in the composite (top, middle and bottom of the mold). The processing conditions defined in Table III lead to contact times ranging

TABLE III Processing conditions—contact times

Matrix	T_m (°C)	T_f (°C)	t_1	t_2	t_3
Al	680	660	1′10″	3′	3′
Al-1wt%Mg	714	674	1′52″	3′54″	4′34″
Al-1wt%Si	699	678	1′46″	3′18″	4′34″
Al-3wt%Si	722	689	1′52″	3′30″	3′48″
Al-1wt%Mg-1wt%Si	728	675	2′24″	4′32″	5′14″
Al-4wt%Si-0.6wt%Mg	670	650	4′	5′20″	5′20″
A356	660	640	4′20″	5′40″	5′40″
A357	640	620	3′3″	5′	5′

T_m and T_f are the mean temperature of the molten metal and of the fibers. t_1, t_2, t_3 are the contact times at liquid state between the matrix and the fibers in the top, middle and bottom of the mold.

between 1 min and about 6 min, depending on the location. Thus, they can be, in some cases, sufficiently long to lead to reactions between the fibers and the alloying elements of the matrix.

For specific studies, an Al-4,5wt%Mg alloy matrix composite was fabricated by a squeeze-casting technique, under the following conditions: temperature of the mold: 350 °C, temperature of the preform: 700 °C, temperature of the liquid alloy: 700 °C, final infiltration pressure: 70 MPa. In this case, the contact time is of the order of a few seconds.

2.2. Experimental

The microstructure of the composite materials was examined at different scales of observation.

To gain information on the infiltration of the fiber preforms by the liquid metal, on the distribution and alignment of the fibers in the metal matrix and on the presence of secondary phases in the matrix, optical and/or scanning electron microscopy (SEM) observations were performed on the polished surfaces of all materials. SEM observations were also conducted to visualize the fracture surfaces of the composites and were performed with a SEM-JEOL 840 equipped with an X-ray dispersion spectrometer (EDS).

In order to investigate the chemical reactions likely to be met in Al-Mg or Al-Mg-Si alloys reinforced with Altex fibers, a study of the reactivity of magnesium with the fibers was undertaken, both in the liquid state (during processing) and in the solid state (during solutionizing at 540 °C). This investigation was based essentially on Transmission Electron Microscopy (TEM) observations and electron diffraction, but also on thermoelectric power (TEP) measurements.

Composite specimens for TEM observations, after mechanical grinding to a thickness of around 100 μm, were dimpled to 15 μm. They were then ion-milled at 5 kV with argon ions at low temperature (100 K). These conditions were used to minimize the problems of preparation due to the difference in abrasion speed between the metal and ceramic reinforcement. Observations and analyses were made with a TEM-JEOL 200CX instrument equipped with an X-ray energy dispersion spectrometer (EDS).

Measurement of the thermoelectric power (TEP) was used to follow the microstructural evolution of the composites during solution heat treatment at 540 °C and

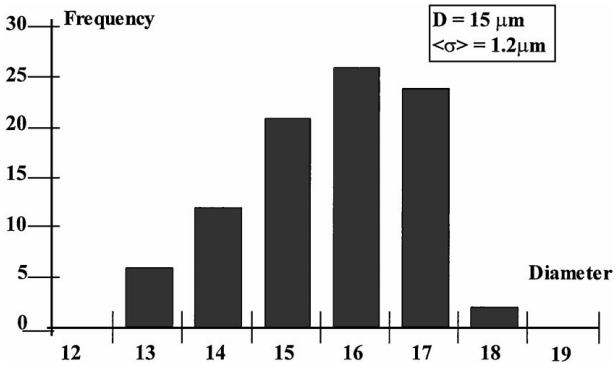

Figure 2 Distribution of diameters of the Altex fibers.

to draw conclusions on the kinetics of reaction likely to be met in Al-Mg and Al-Mg-Si alloys reinforced with Altex fibers. Indeed, this technique allows informations to be obtained on the variations in the concentration of the alloying elements in solid solution [3]. A description of the measurement method and of the apparatus has been given previously [4]. The solution heat treatment of the materials was carried out in salt baths. In order to protect the samples from these baths, they were embedded in thin aluminium foil for short treatment times (less than 15 min). They were then placed in a quartz tube for longer solutionizing times.

This microstructural study was completed by the evaluation of the tensile properties of the fibers and of the composite materials.

The mechanical properties of the Altex fibers were determined in the as-received state and after processing of the composite materials. In this last case, the fibers were extracted from the metal matrix of the composites by dissolution of the matrix in aqua regia. The Weibull parameters of the strength distribution of the fibers were determined by single fiber tests conducted on specimens with a 30 mm gauge length. To evaluate the strength of the fibers tested, we measured the real diameter of each fiber by diffraction of a LASER beam. Indeed, the distribution of diameters of the Altex fibers, determined on a population of 90 fibers, is rather widespread (Fig. 2). The corresponding mean value is 15 μm, with a standard deviation of 1, 2 μm.

Lastly, tensile tests were carried out on composite samples either in the as-cast state or treated two hours or fifteen hours at 540 °C. The composites were tested in both longitudinal and transverse directions. The testing was carried out on an Instron machine using a crosshead speed of 1 mm/min. A minimum of three specimens was tested per condition.

3. Mechanical characterization of the composite materials

3.1. Analysis of the macrostructure of materials

Optical microscopy observations performed on the transverse and longitudinal sections of the composite materials after processing allowed us to show that the infiltration of the fibers by the liquid metal was good, with no porosity or infiltration defects, whatever the type of matrix. Moreover, the distribution of orientation of the fibers in the matrix was found to be rather homogeneous and the alignment of the fibers not to be strongly disturbed by the infiltration. All these observations are clearly evidenced in Figs 3a and b which were taken from pure aluminium matrix composites. The microstructure of the materials, which concerns the presence of secondary phases in the matrix, will be discussed later.

3.2. Mechanical properties of the composites reinforced with Altex fibers

For each composite, we carried out tensile tests at room temperature. In the case of alloyed matrices, the composites were tested either in the as-cast state or in the homogeneized state after a solutionizing treatment of 2 h at 540 °C. Complementary experiments were conducted with some samples homogeneized 15 h at 540 °C, in order to study the effect of a prolonged exposure at high temperature on the tensile properties. The effect of the structural hardening heat treatment was not investigated.

Elastic modulus and rupture strength were determined in both the longitudinal and transverse directions (Tables IVa and b). For each tensile characteristic, we give the mean value, the standard deviation and the number of tests. For comparison, the tensile properties of the pure aluminium matrix composite were added.

TABLE IV Mechanical properties of the Altex fibers reinforced composites ($V_f = 35\%$): (a) longitudinal properties, (b) transverse properties

		Longitudinal properties		
Composite ($V_f = 35\%$)	Thermal treatment	σ_r (MPa)	E (GPa)	σ_r/σ_{Al} (%)
Al/Altex	2 h at 540 °C	**516**/48/5	**109**/3.5/5	**100**
Al-1wt%Mg/Altex	As-cast state	**415**/20/3	**110**/4/3	**80**
	15 h at 540 °C	**270**/20/3	**110**/5/3	**52**
Al-1wt%Si/Altex	2 h at 540 °C	**348**/6/4	**110**/5/5	**67**
Al-3wt%Si/Altex	2 h at 540 °C	**240**/16/3	**114**/6/3	**46.5**
Al-1wt%Mg-1wt% Si/Altex	2 h at 540 °C	**350**/3/3	**110**/2.5/3	**68**
Al-4wt%Si-0,6wt% Mg/Altex	2 h at 540 °C	**165**/64/4	**106**/2/4	**32**
A356/Altex	2 h at 540 °C	**214**/16/3	**109**/2/3	**41**
A357/Altex	2 h at 540 °C	**179**/28/3	**108**/1/3	**35**
6013/Altex	2 h at 540 °C	**260**/25/4	**108**/6/4	**50**
	15 h at 540 °C	**212**/10/4		**41**

		Transverse properties	
Composite	Thermal treatment	σ_r (MPa)	E (GPa)
Al / Altex	2 h at 540 °C	**92**/7/3	**101**/7/2
Al-1wt%Mg/Altex	As-cast state	**115**/8/3	**108**/9/3
	15 h at 540 °C	**115**/8/4	**109**/4/4
Al-1wt%Si/Altex	2 h at 540 °C	**133**/5/4	**115**/10/4
Al-3wt%Si/Altex	2 h at 540 °C	**129**/14/3	**130**/15/4
Al-4wt%Si-0,6wt% Mg/Altex	2 h at 540 °C	**121**/35/3	///
A356/Altex	2 h at 540 °C	**113**/36/3	///
A357/Altex	2 h at 540 °C	**136**/5/3	///
6013/Altex	2 h at 540 °C	**150**/24/3	///

Mean value/standard deviation/number of tests. ε_r : Total strain to failure.

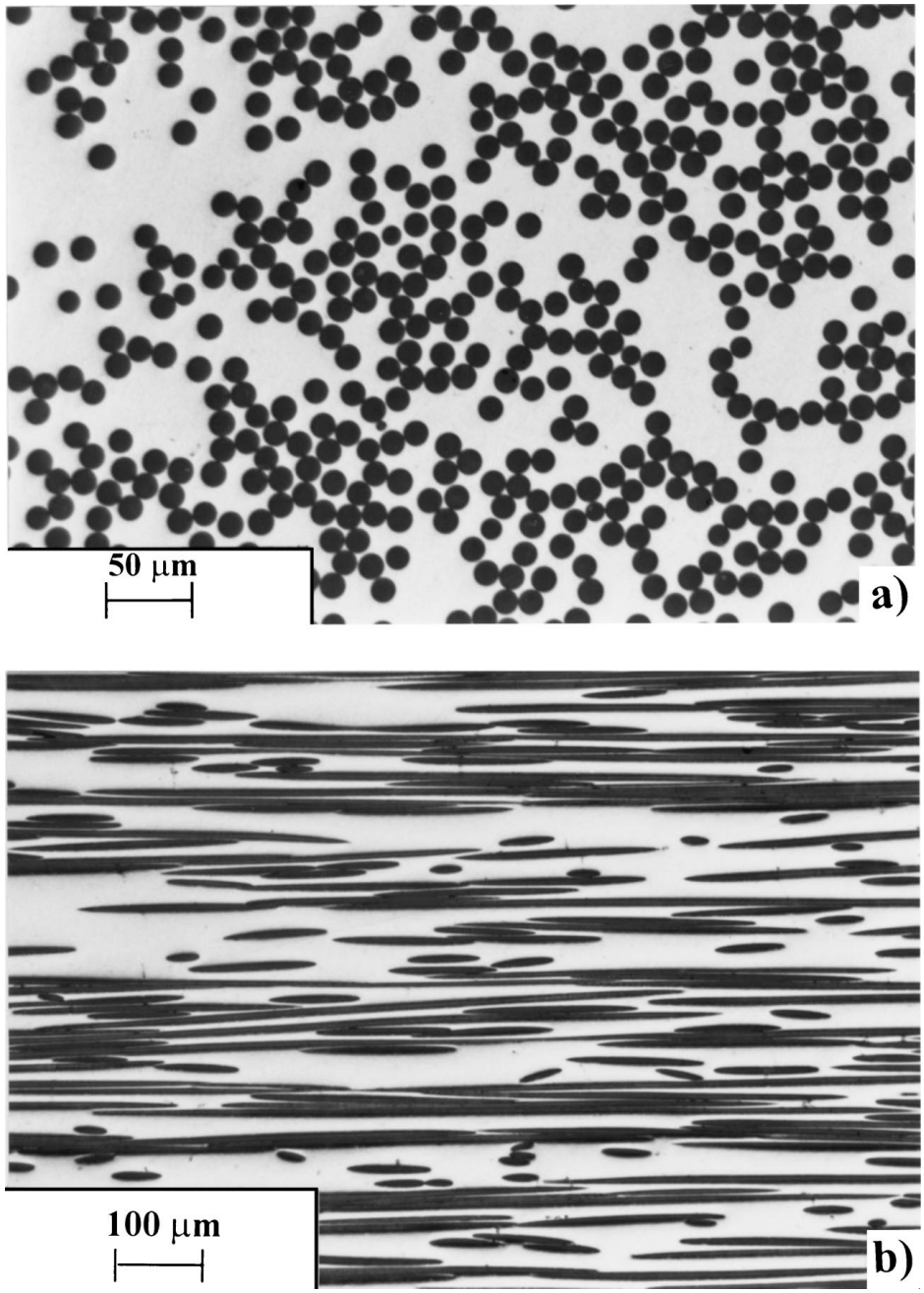

Figure 3 Optical micrograph of the pure aluminimium matrix composite reinforced with Altex fibers: (a) Transverse section; (b) Longitudinal section.

3.2.1. Longitudinal tensile properties

From the results presented in Table IVa, it appears that the pure aluminium matrix composite has the best longitudinal mechanical properties. Its ultimate tensile strength is of the order of 516 MPa.

In the case of the alloyed aluminium matrix composites, the longitudinal properties appear to be very dependent on the matrix composition. For example, in comparison with the strength of the pure aluminium matrix composite, the ultimate tensile strength (UTS) of the as-cast Al-1wt%Mg/Altex fibers composite is not reduced sharply and is equal to 415 MPa. In the case of the binary Al-Si alloys/Altex fiber composite, the decrease in ultimate tensile strength is more pronounced and increases with the silicon content. However, it has to be noted that the lowest ultimate tensile strengths are obtained for the ternary Al-Mg-Si matrix composites. For the Al-1wt%Si-1wt%Mg matrix composite, the decrease in strength is of the order of 32%, while for the industrial 6013 matrix composite, which contains almost the same magnesium and silicon content, this decrease reaches 50%. Lastly, the strength of composites with a high silicon content never exceeds 40% of the ultimate strength of the pure aluminium matrix composite.

It is also important to note that a long thermal treatment has a great influence on the longitudinal properties and leads, in all cases, to a decrease in the ultimate tensile strength of the composites.

For all composite materials, the values of the Young's modulus are of the order of 110 GPa. This value corresponds to 90% of the theoretical value given by the rule of mixtures. This good correlation with the rule of mixtures indicates that fibers keep their elastic properties and are not appreciably disaligned during the processing of the composites.

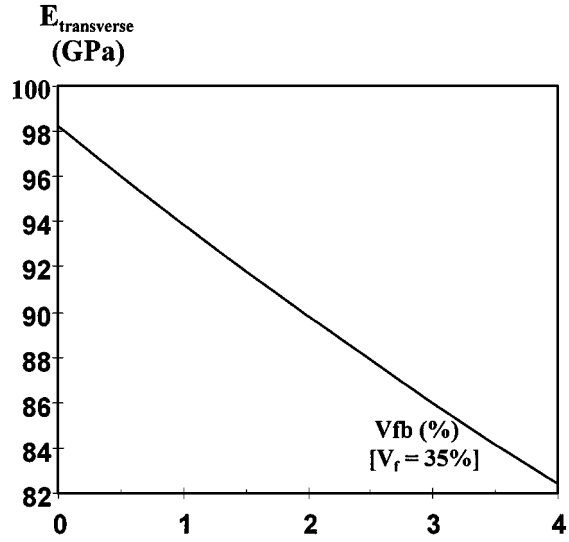

Figure 4 Evolution of the transverse modulus as a function of the volume fraction of debonded fibers (V_{fb}) assuming $V_f = 35\%$ (model of Kyono).

3.2.2. Transverse tensile properties

In the case of the transverse properties, the alloying elements (Mg and Si) lead to an increase in the value of the ultimate tensile strength. Indeed, as can be seen in Table IVb, the ultimate tensile strengths of the alloyed aluminium matrix composites are always higher than those obtained on the pure aluminium matrix composite (92 MPa) and vary between 115 and 150 MPa.

The experimental values obtained for the transverse elastic modulus confirm the absence of infiltration defects such as porosity in the composites. In effect, as was shown by Kyono [5] using an Eshelby type method [6], the presence of infiltration defects (such as interfacial porosity corresponding to debonded fibers or non infiltrated regions of the preform) should be accompanied by a drastic reduction of the transverse modulus. Using the approach developped by Kyono, we plotted, in Fig. 4, the evolution of the transverse modulus of a pure aluminium matrix composite reinforced with 35 vol % fibers as a function of the percentage of debonded fibers (V_{fb}). From this figure, it appears that the proportion of debonded fibers has a marked influence on the transverse modulus, since with $V_{fb} = 2\%$, the decrease in the value of transverse modulus is of the order of 14%. In the case of the pure aluminium matrix composite investigated in this work, a good correlation is observed between the experimental tranverse modulus (101 GPa) and the theoretical one calculated assuming $V_{fb} = 0$ (98 GPa). Thus, it is possible to conclude to the absence (or to a very low density) of infiltration defects in this composite and to the quality of the composites elaborated for this study by the medium pressure infiltration method.

3.2.3. Fracture surfaces

a) Longitudinal direction: The fracture surfaces of the composites tested in the longitudinal direction are almost identical whatever the type of matrix. An example of these fracture surfaces is shown in Fig. 5a in the case of the pure aluminium matrix composite. From the observation of this fracture surface, it appears that rupture of the composite occurs in a brittle manner

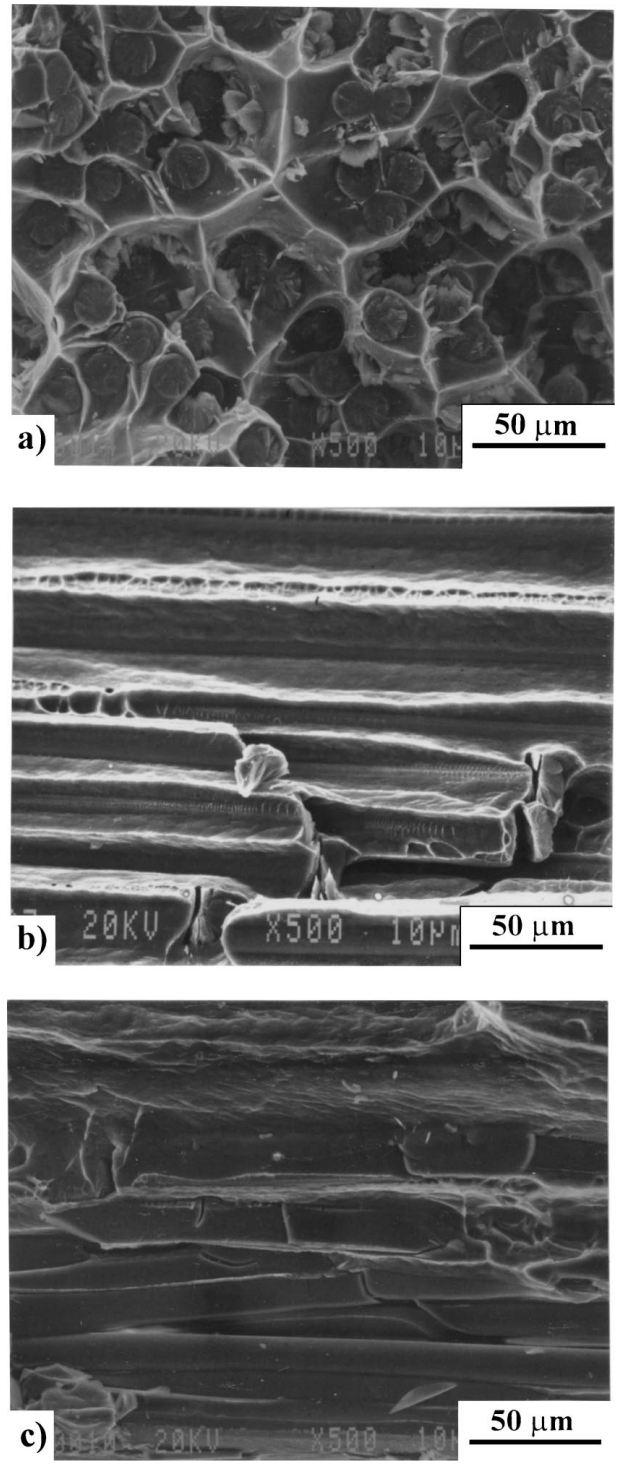

Figure 5 Fracture surfaces of: (a) the pure aluminium matrix composite tested in the longitudinal direction; (b) and (c) the pure aluminium matrix composite and the alloyed matrix composites tested in the transverse direction.

and is perpendicular to the tensile direction. However, the fracture surface is not uniformly plane and is characterized by steps corresponding to zones locally less reinforced. Lastly, it should be noted that a pronounced plastification is present in the matrix between the fibers. This plastification is accompanied by a slight decohesion of the matrix at the end of the cracked fibers.

b) Transverse direction: In the transverse direction, the fracture surfaces are different in the pure aluminium matrix composite and in the other composites, as can be seen in the micrographs of Figs 5b and c.

In the case of the pure aluminium matrix composite (Fig. 5b), the transverse rupture is initiated in the matrix located between the fibers. This indicates a strong interfacial bond between the fibers and the matrix, the strength of which is greater than that of the matrix.

In contrast, the other fracture surfaces (Fig. 5c) are characterized by a rupture initiated in the fiber itself. As a consequence, in this case, this is the transverse mechanical strength of the fibers, which is responsible for the rupture of the composites. The reasons of this change of behaviour will be discussed later.

3.2.4. Conclusion

Fracture surfaces show that the interfacial bonding between fibers and matrix is always strong. Thus, the loss in longitudinal mechanical strength of composites due to the use of alloyed matrices can only be explained by a loss in the mechanical properties of the fibers, due to chemical reactions between the fibers and the matrix during processing or thermal treatment, or to an embrittlement of the material, due to secondary phases in the matrix or at fiber-matrix interfaces. These problems are discussed in the two following sections.

4. Study of reaction phenomena occuring during processing or thermal treatment: interaction of magnesium with the Altex fibers

4.1. Introduction

Owing to the nature of alloying elements contained in the alloys investigated in this work, magnesium is the only one which can give rise to extensive reaction phenomena with alumina or silica and lead to the formation of interfacial compounds. These compounds may be detrimental to the mechanical properties, since they may modify the nature of the interfaces and damage the fibers.

The studies performed in the literature on Al-Mg or Al-Mg-Si alloy/Al_2O_3 composites showed that, from a thermodynamical point of view, several reactions may occur in the liquid or solid state. Depending on the temperature range and on the magnesium content in the alloy, two different reaction products are expected to be observed at the interface between the matrix and the fibers: spinel ($MgAl_2O_4$) and magnesia (MgO). The two main reactions leading to the formation of these two compounds and implying the alumina reinforcement are the following:

$$3[Mg]l + 4[Al_2O_3]s \Longleftrightarrow 3[MgAl_2O_4]s + 2[Al]l \quad (1)$$

$$3[Mg]l + [Al_2O_3]s \Longleftrightarrow 3[MgO]s + 2[Al]l \quad (2)$$

As was shown by the stability diagrams established by Mc Leod [7] in an Al-Mg alloy reinforced with alumina particles, $MgAl_2O_4$ spinel is more likely to be met at low magnesium contents and at high temperatures, while the formation of the MgO magnesium oxide is enhanced at high magnesium contents and at low temperatures. From an experimental point of view, it is generally observed that for magnesium content lower than 4 wt %, the formation of $MgAl_2O_4$ prevails [8–10]. In contrast, when the magnesium content is greater than 4 wt %, it is found that the main reaction product at interfaces is MgO [11, 12].

In Al-Mg alloy/Al_2O_3-SiO_2 composites, additional reactions are expected to take place. Indeed, the silica binder is likely to be reduced, according to one of the three following reactions as suggested by Molins *et al.* [13]:

$$[SiO_2]s + \tfrac{4}{3}[Al]l \Longleftrightarrow \tfrac{2}{3}[Al_2O_3]s + [Si]s \quad (3)$$

$$[SiO_2]s + 2[Mg]l \Longleftrightarrow 2[MgO]s + [Si]s \quad (4)$$

$$2[SiO_2]s + 2[Al]l + [Mg]l \Longleftrightarrow [MgAl_2O_4]s + 2[Si]s \quad (5)$$

It is also necessary to take into account the interaction between the silicon released by the silica binder and the magnesium contained in the matrix, which leads to the formation of Mg_2Si compounds according to reaction (6):

$$2[Mg]l + [Si]s \Longleftrightarrow [Mg_2Si]s \quad (6)$$

As can be seen from the above considerations, Al-Mg and Al-Mg-Si alloys reinforced with Altex fibers are expected to be reactive systems in the liquid state, but also probably in the solid state. As no systematic investigation was undertaken on the reactivity of these materials on a microscopic scale (TEM observations), the aim of this part of the work was to determine whether reaction products can form during the processing or the solutionizing treatments of these materials.

To show clearly the reactivity of magnesium with the Altex fibers both in the liquid and solid states, we chose to investigate the microstructure of binary Al-Mg alloy matrix composites, as the interaction of magnesium with the Altex fibers can only be clearly established with this kind of matrix. Indeed, with an Al-Mg-Si alloy, the presence of the Mg_2Si crystals at the interface between the matrix and the fibers could originate from a precipitation phenomenon of the magnesium and silicon of the matrix. In contrast, with a matrix of Al-Mg type, the presence of such crystals can only be the consequence of the combination (reaction 6) of the magnesium of the matrix with the silicon released by the reduction of silica of the fibers (reactions 3–5).

4.2. Study of the reactivity of magnesium with the fibers during processing (liquid state)

This study was conducted on the as-cast Al-1wt%Mg/Altex fiber composite. TEM observations on this material allowed us to show the reactivity of magnesium with the fibers during processing by medium pressure infiltration. Indeed, two types of compounds were found in the interfacial region between the matrix and the fibers:

- Mg_2Si compounds (Fig. 6a), which formed from the magnesium of the matrix and from silicon released by the reduction of silica of the fibers, as explained above.

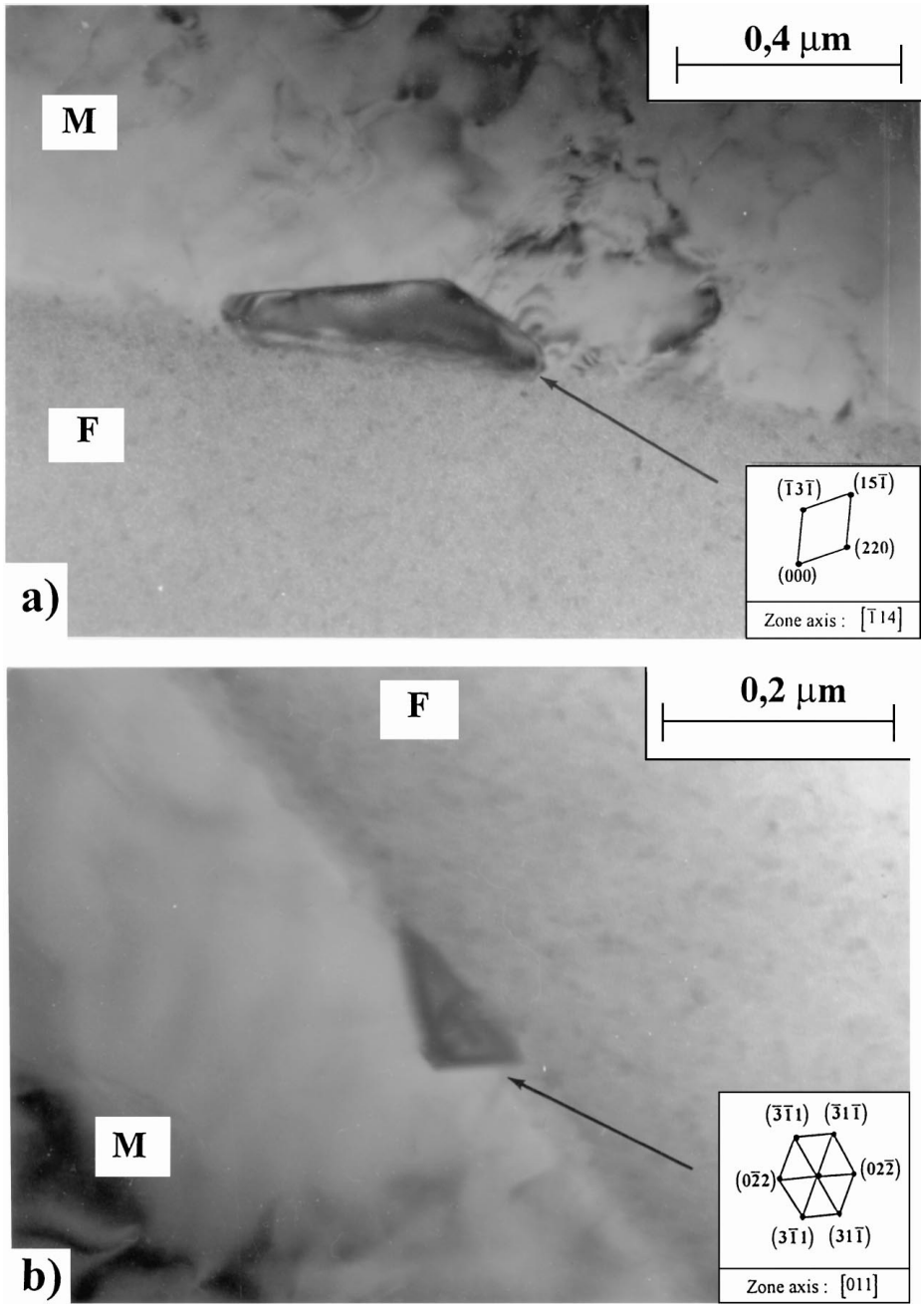

Figure 6 As-cast Al-1wt%Mg alloy matrix composite—TEM micrograph and diffraction pattern of: (a) Mg_2Si precipitates, (b) $MgAl_2O_4$ precipitates at the interface between the matrix and the fibers.

- Small $MgAl_2O_4$ spinel crystals (Fig. 6b), which formed either from the decomposition of silica according to reaction 5 or from a direct chemical interaction between magnesium and alumina contained in the fibers according to reaction 1. In fact, it has to be pointed out that reaction 1 is unlikely during processing, referring to the calculation of Hallstedt [14] on the reaction kinetics in the Mg-Al_2O_3 system. Moreover, most of the spinel crystals were found to be located in the region near the Mg_2Si compounds, which supports that they were probably obtained according to reaction 5.

From these TEM observations, we could conclude that in the case of the Al-1wt%Mg/Altex fibers composites, the reactivity of magnesium with the fibers is effective, but not very extensive during processing, as only a very low density of interfacial compounds was detected along interfaces.

4.3. Study of the reactivity of magnesium with the fibers during solutionizing treatment at 540 °C (solid state)

In order to gain further understanding on the reactivity of Al-Mg alloy matrix composites, we conducted a study on the microstructural evolutions which may occur during long-term exposure of these composites at the solutionizing temperature of the Al-Mg-Si alloys (540 °C) and may influence the mechanical properties of the materials. However, it should be noted that it is possible to detect solid state reactivity only: (i) if no reaction between the fibers and the matrix has occured during processing, (ii) if the reaction is sufficiently

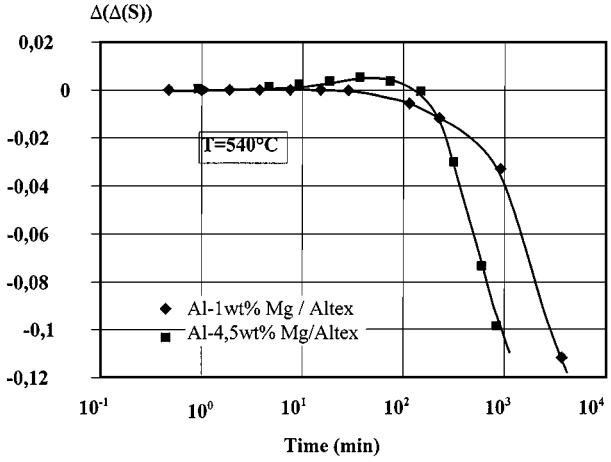

Figure 7 TEP evolution during treatment at 540 °C of the Al-1wt%Mg and Al-4,5wt%Mg matrix composites.

extensive in the solid state to give rise to easily detectable reaction products, that is to say if the magnesium content is high enough.

To satisfy these two conditions, Al-4,5wt%Mg/Altex composites were processed by squeeze-casting. As the rapid cooling rate associated with this technique limits the contact time between the molten alloy and the fibers to about 20 s, no liquid state reactivity occurs during processing, as was also confirmed by TEM observations.

For comparison, we also studied the microstructural evolution which takes place in the Al-1wt%Mg/Altex fiber composite during exposure at 540 °C.

4.3.1. Preliminary study: TEP evolution of the Al-1wt%Mg and Al-4,5wt%Mg/ Altex fibers composites during solutionizing treatment at 540 °C

As preliminary work on the solid state reactivity, we followed the evolution of the thermoelectric power (TEP) of the Al-1wt%Mg and Al-4,5wt%Mg/Altex fibers composites during isothermal solution treatment at 540 °C (Fig. 7). In the curves of Fig. 7, the TEP of the non-homogenized state (ΔS_i) of each sample was taken as a reference and the quantity $\Delta(\Delta S) = \Delta S_t - \Delta S_i$ (where ΔS_t is the TEP of the sample at time t) was reported as a function of time.

The two curves have the same general aspect. For short treament times, no TEP variation can be dectected but a TEP decrease appears after a two hour treatment. As magnesium is the only alloying element in the matrix of the two composites, the TEP variations observed can only be attributed to the variations in the concentration of this element in solid solution. As magnesium in solid solution has a strong positive effect on the TEP of pure aluminium [3], the decrease in the relative TEP of the sample corresponds to a loss of magnesium in solid solution. This loss of magnesium can only be attributed to a reaction in solid phase between magnesium and the fibers. To confirm this assumption and determine the reactions responsible for the loss of magnesium, TEM observations were conducted on these materials after fifteen hour solution treatment at 540 °C. This treatment was chosen to enhance the reactivity phenomena.

4.3.2. Evolution of the microstructure of the Al-Mg/Altex fibers composites during solutionizing treatment at 540 °C

After a fifteen hour solutionizing treatment at 540 °C, two different microstructural evolutions were observed in the two composites.

In the case of the Al-4,5wt%Mg/Altex fiber composite, two types of compounds were observed at the interfaces: Mg_2Si precipitates (Fig. 8a) and $MgAl_2O_4$ spinel crystals (Fig. 8b). As these composites did not show any evidence for a reaction during processing by squeeze-casting, these compounds formed by reaction in the solid state.

In the Al-1%Mg/Altex fiber composites processed by medium pressure infiltration, the Mg_2Si interfacial precipitates initially present in the as-cast conditions were no longer detected and it was found that these precipitates disappeared at the expense of $MgAl_2O_4$ spinel crystals (Fig. 9). Indeed, these spinel crystals were much more numerous and much larger than in the as-cast state.

From these observations, it appears that the stability of the Mg_2Si precipitates in the composites, resulting from the reaction between magnesium and silicon released by the fibers, is very dependent on the magnesium content of the matrix alloy. These precipitates, which result, in the Al-1%Mg alloy from the chemical interaction of the melt and the fibers, are no longer stable at 540 °C in the solid state and tend to dissolve. In contrast, when the magnesium content is high (Al-4.5%Mg alloy), these precipitates are stable in the solid state and thus can develop.

Concerning the $MgAl_2O_4$ crystals, their growth mechanism during the solutionizing treatment can result from reaction 1 between magnesium of the alloy and the alumina of the fibers or from reaction 5 which leads to the reduction of silica of the fibers. In this last case, the silicon released by the decomposition of silica is likely to form Mg_2Si precipitates which are stable in the Al-4.5%Mg matrix but not in the low alloyed 1%Mg matrix.

5. Microstructural study of the matrix and of the matrix-fiber interface in Al-Si and Al-Mg-Si alloy matrix composites: influence of intermetallic phases and interfacial reactions

5.1. Al-Si alloy matrix composites

With silicon, no reaction is expected to take place. However, this element can play a role on the microstructure of the composites. Its effect was clearly established on the optical micrographs of the Al-1wt%Si and Al-3wt%Si/Altex fibers composites. The microstructure of the Al-1wt%Si matrix composite was found to be identical to that of the pure aluminium matrix composite (Fig. 3), indicating that in this material, silicon is in solid solution. In contrast, in the Al-3wt% Si matrix composite, this element was found to contribute to the formation of acicular secondary phases clearly visible in Fig. 10. The identification of these phases by SEM-X-ray analysis showed us that they mainly consist of eutectic silicon. In addition, a low proportion of

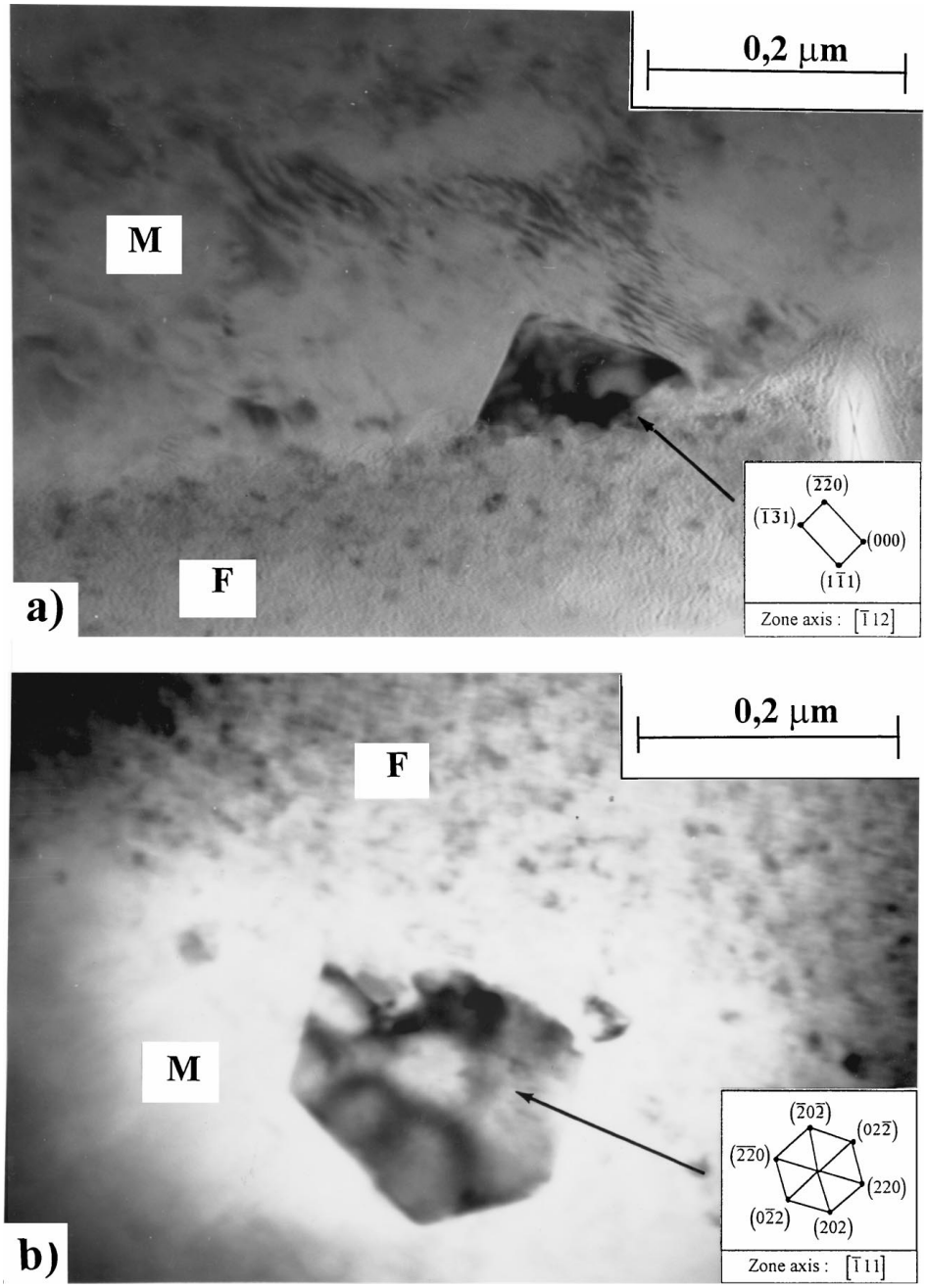

Figure 8 Al-4,5wt%Mg matrix composite treated 15 h at 540 °C—TEM micrograph and diffraction pattern of: (a) Mg_2Si precipitates; (b) $MgAl_2O_4$ precipitates at the interface between the matrix and the fibers.

intermetallic compounds including impurities such as iron was detected at interfaces.

5.2. Al-Mg-Si alloy matrix composites:

5.2.1. Study of composites with a low silicon content (wt % Si < 3): case of the Al-1wt%Si-1wt%Mg and 6013/Altex fibers composites

The Al-1wt%Si-1wt%Mg and 6013 matrix composites are characterized by the fact that their magnesium and silicon content are nearly the same. The only difference between these two composites comes from the presence, in the 6013 alloy, of alloying elements other than magnesium and silicon (such as copper, iron and manganese).

Using optical microscopy, we could clearly provide evidence that after a two hour treatment at 540 °C, no secondary phase is present in the Al-1wt%Si-1wt%Mg matrix, while two different phases, located either in the matrix or at the interfaces, can be detected in the 6013 matrix.

A further study based on TEM observations performed on the Al-1wt%Si-1wt%Mg and 6013 matrix composites allowed us to show that after a two hour solutionizing treatment at 540 °C, the fibers are covered by Mg_2Si compounds (Fig. 11). As the matrix of these two composites contain silicon, the formation of these compounds can result:

- either during the solidification process, from a segregation of the magnesium and silicon of the matrix in the form of β-Mg_2Si precipitates at interfaces,
- or during processing, from liquid state reaction between magnesium and silicon released by the reduction of the silica contained in the fibers.

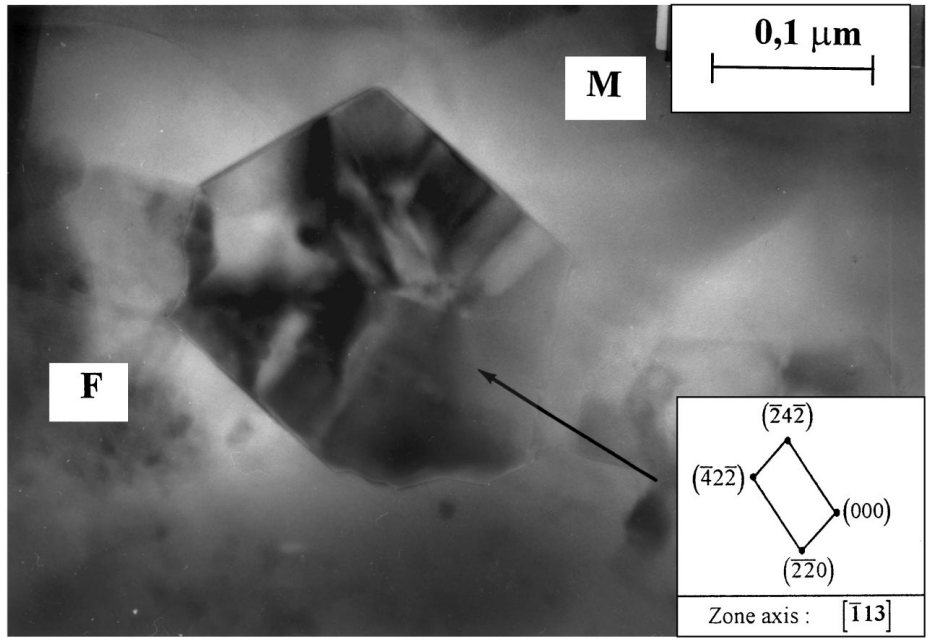

Figure 9 Al-1wt%Mg matrix composite treated 15 h at 540 °C—TEM micrograph and diffraction pattern of $MgAl_2O_4$ precipitates at the interface between the matrix and the fibers.

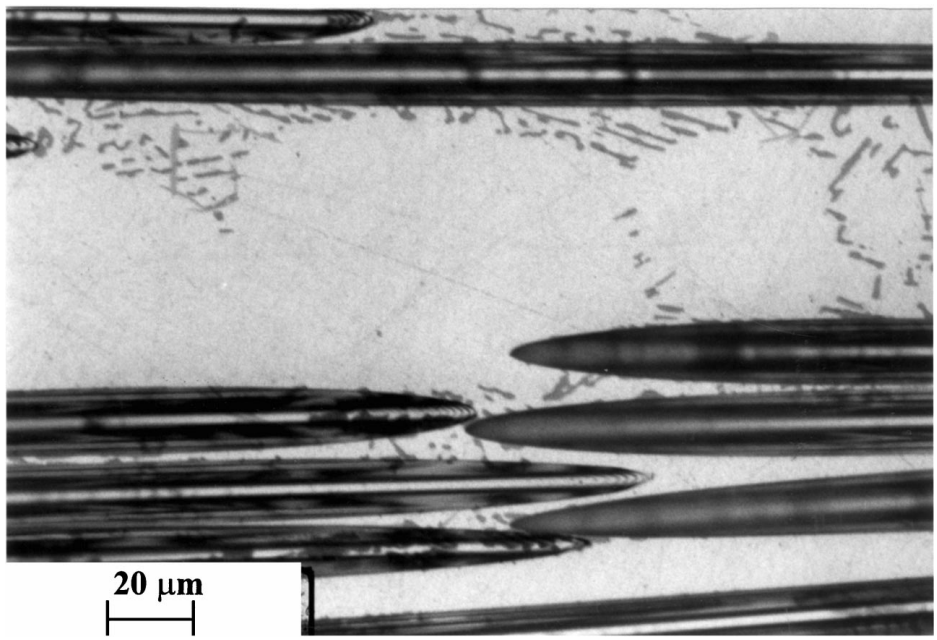

Figure 10 Optical micrograph of the Al-3wt%Si matrix composite.

From the results obtained on the Al-1wt%Mg/Altex fiber composites, it is clear than in Al-Mg or Al-Mg-Si alloys reinforced with Altex fibers, one cannot exclude a reaction between the silica of the fibers and the magnesium of the alloy. Thus, the Mg_2Si compounds can originate from such a reaction.

However, it has to be noted that the Mg_2Si precipitates found in the Al-1wt%Si-1wt%Mg and 6013/Altex fiber composites were much more numerous than those observed in the Al-1wt%Mg/Altex fiber composites and were located extensively along interfaces. Moreover, their size was about 5 to 10 times greater, as can be seen by a comparison between Fig. 6a and Fig. 11. As the matrices of these three composites have almost the same magnesium content, it is probable that in the Al-1wt%Si-1wt%Mg and 6013/Altex fiber composites, the reaction between magnesium and silica is not the only phenomenon responsible for the presence of Mg_2Si precipitates at the interfaces and that the segregation process mentioned above is effective. This last assumption is supported by the fact that the presence of Mg_2Si located in the interfacial region is a relatively general phenomenon in Al-Mg-Si alloy matrix composites, even when the reinforcement is inert from a chemical point of view. Such precipitates were, for example, found in a 6061 alloy reinforced with pure α-alumina platelets [15].

In the particular case of the 6013/Altex fiber composite, additional intermetallic compounds were observed at the interfaces (Fig. 12). They probably formed during processing by segregation of the numerous alloying elements contained in the 6013 alloy. In some cases, their

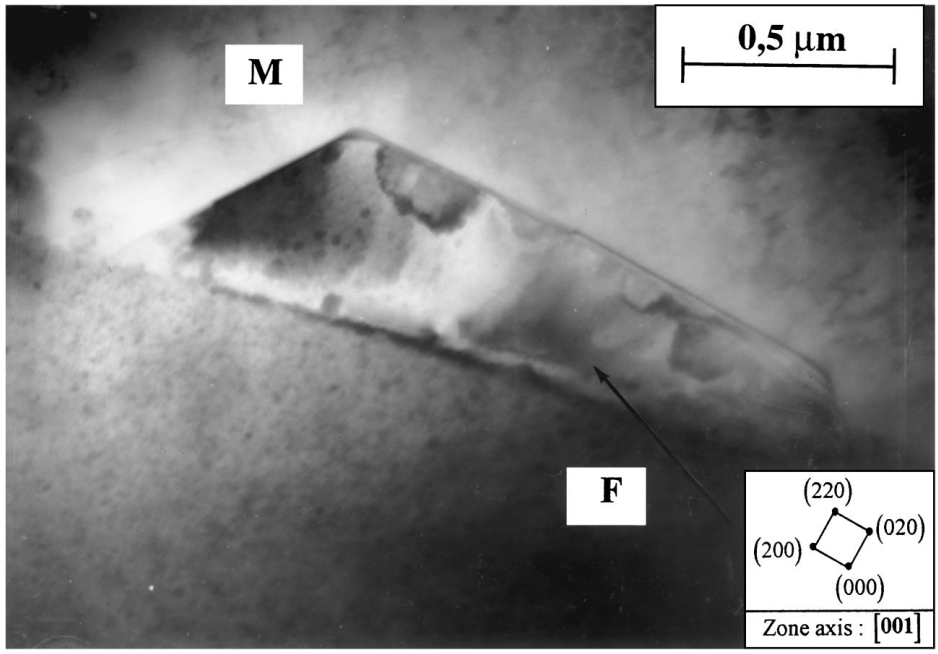

Figure 11 Al-1wt%Mg-1wt%Si or 6013 matrix composites treated 2 h at 540 °C—TEM micrograph and diffraction pattern of Mg_2Si precipitates at the interface between the matrix and the fibers.

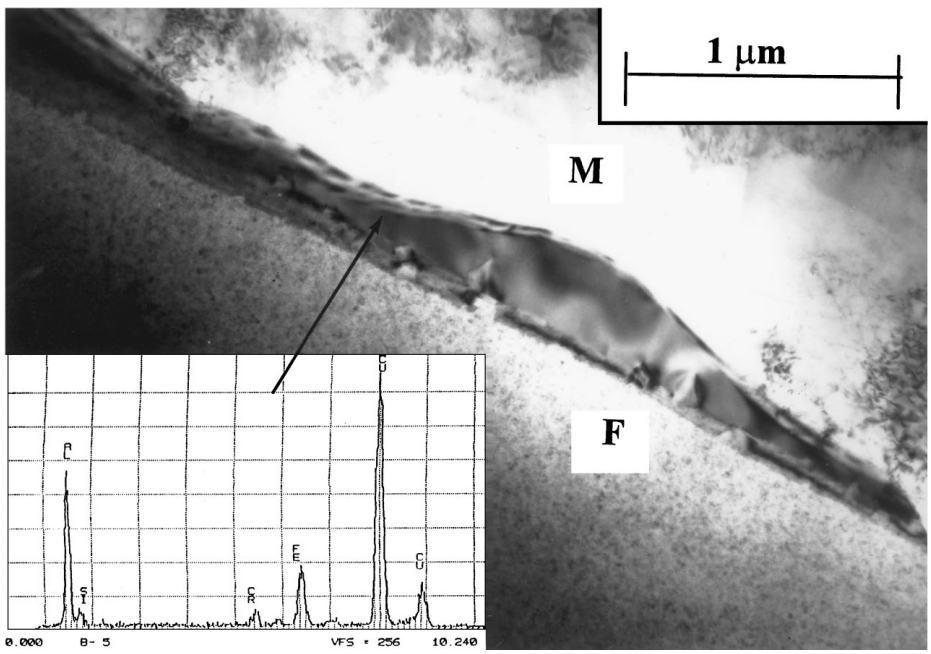

Figure 12 6013 matrix composite treated 2 h at 540 °C—TEM micrograph and X-ray analysis of intermetallic compounds at the interface between the matrix and the fibers.

size was found to reach appreciable dimensions of the order of a few microns.

Lastly, we investigated the microstructural state of the 6013/Altex fiber composite after a fifteen hour treatment at 540 °C. It was found to be very similar to that observed in the case of the Al-1wt%Mg/Altex fiber composite. Indeed, our TEM observations allowed us to highlight that the Mg_2Si precipitates initially detected were no longer present at the interfaces after 15 h at 540 °C. It is supported by the fact that in the 6013 alloy, the low magnesium content makes these precipitates unstable at this temperature. However, two types of precipitates were met in the interfacial region: intermetallic compounds and $MgAl_2O_4$ spinel crystals. The intermetallic compounds were already present after processing and did not dissolve at 540 °C. The $MgAl_2O_4$ crystals probably formed in the solid state.

5.2.2. Study of composites with a high silicon content (% Si > 3)

The study of the microstructure of the Al-4wt%Si-0.6wt%Mg, A356 and A357 matrix composites by optical microscopy allowed us to show the presence of a high density of phases in the matrix of these materials, as can be seen in Fig. 13 (case of the A357 matrix composite). These precipitates were observed both in

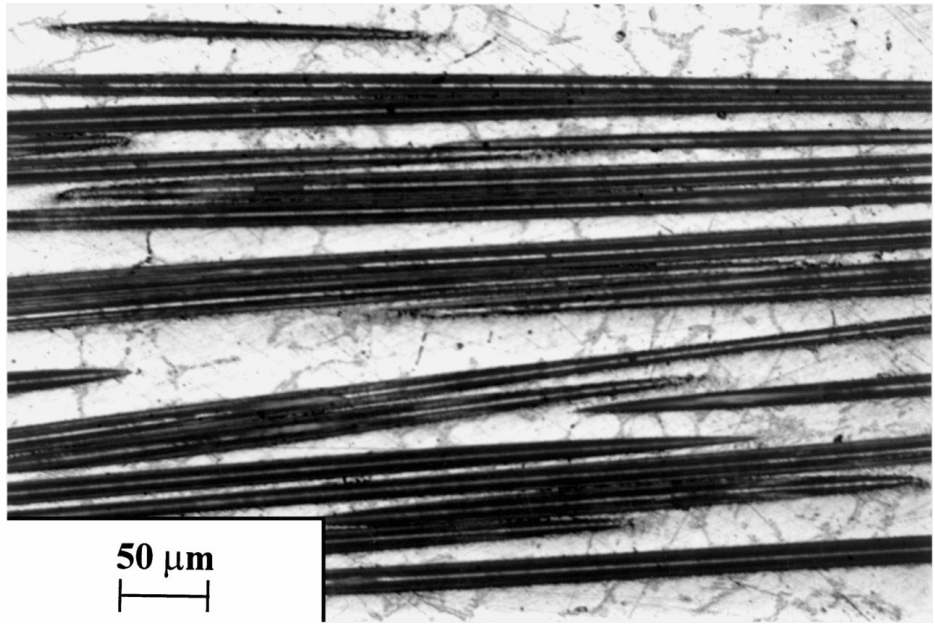

Figure 13 Optical micrograph of the A357 matrix composite.

the as-cast state and after the solutionizing treatment at 540 °C of these materials. As was shown by X-ray analysis in the SEM, these precipitates are mainly eutectic silicon and also, at a lesser extent, intermetallic compounds.

6. Discussion

6.1. Influence of the processing conditions and reactions on the longitudinal mechanical properties of the fibers and composites

6.1.1. Introduction

As the mechanical properties of unidirectional composites are strongly related to those of the fibers used as a reinforcement, it is necessary to determine their tensile characteristics after inserting them into the metal matrix. Two main causes can lead to a reduction in the tensile strength of the fibers: (i) the processing procedure, (ii) the reactions between the fibers and the alloying elements contained in the matrix. Thus, the aim of this section is to assess the decrease in tensile strength of the fibers resulting from these two factors. To this end, after determining the Weibull parameters of the strength distribution of the as-received fibers, these parameters were measured on fibers extracted either from a pure aluminium matrix composite or from a 6013 matrix composite, in order to evaluate and to dissociate the contribution of the processing technique and the reactivity phenomena on the loss of properties of the fibers.

To describe the brittle behaviour of the Altex fibers, we used a two parameter Weibull model. In this case, the probability of rupture for an applied stress ranging between 0 and σ is given by: $F_q(\sigma) = 1-\exp[-(\sigma/\sigma_L)^m]$, where m is the Weibull modulus and σ_L is a scale factor depending on the gauge length.

6.1.2. As received state

Fig. 14 represents the results of the tensile tests in the Weibull plane $(\ln(-\ln(1-F_q)) = f(\ln\sigma))$ on as-received fibers. The experimental results plotted in this manner are straight lines and lead to a Weibull modulus (m) equal to 4,3 and a scale factor (σ_L) equal to 2016 MPa.

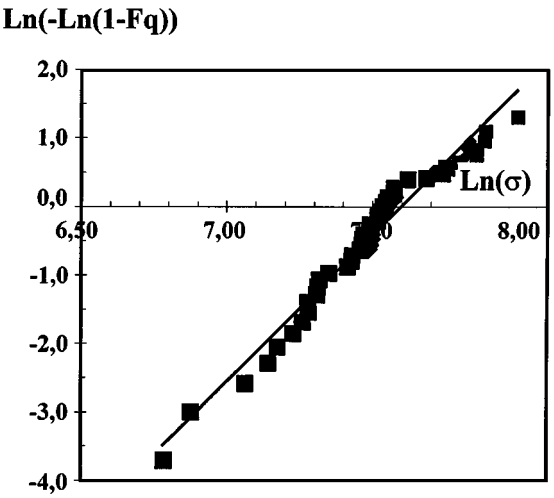

Figure 14 Weibull statistic of the as-received Altex fibers.

Using the relation $\langle\sigma_r\rangle = \sigma \times \Gamma(1+1/m)$, where Γ is the gamma function, it is possible to calculate a statistical value of the mean ultimate tensile strength $\langle\sigma_r\rangle$ of the fibers. This value was found to be 1824 MPa and is very close to that obtained by averaging the ultimate tensile strengths (1817 MPa with a standard deviation of 471 MPa).

6.1.3. Mechanical properties of the fibers extracted from the pure aluminium matrix composite

In order to quantify the loss of properties due to the mechanical degradation of the fibers during the processing of the composite, a Weibull analysis was conducted on fibers extracted from the pure aluminium matrix

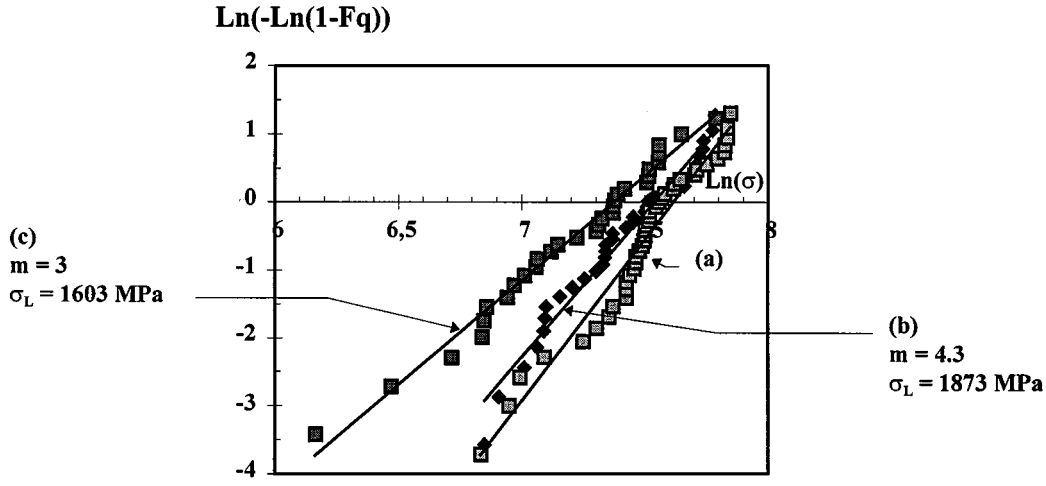

Figure 15 Weibull statistics of the Altex fibers: (a) as-received state, (b) as-received state + treatment in aqua regia, (c) after extraction from the pure aluminium matrix composite.

composite. Indeed, with such a matrix, the variation in the tensile properties of the fibers can only arise from the processing mode, no chemical degradation being expected.

Fig. 15 shows the Weibull statistics obtained on these fibers, as well as those of the as-received fibers and of the as-received fibers submitted to the same exposure in aqua regia as the extracted fibers. Table V summarizes the Weibull parameters of these three populations of fibers. From these data, it appears that the treatment in aqua regia aimed at dissolving the metal matrix does not excessively deteriorate the fibers. Indeed, this treatment leads to a decrease in the strength of the fibers of the order of 6% without modifying the Weibull modulus. In contrast, the extracted fibers are characterized by a lower Weibull modulus and by a strength about 22% lower than that of the as-received fibers. Thus, in addition to the loss of strength resulting from the dissolution of the matrix in aqua regia (6%), a decrease in strength is due to the damage of the fibers during their processing history (16%).

From the mean ultimate tensile strength of the fibers extracted from the pure aluminium matrix (1432 MPa), it is possible to evaluate the strength of the composite using the rule of mixtures. This value for a volume fraction of fibers of 35% is found to be of the order of 540 MPa and is close to the experimental value (516 MPa).

In fact, the simple two-parameter Weibull distribution cannot completely describe the distribution of the rupture strength of the fibers. Indeed, Weibull curves of Fig. 15 are not perfectly straight and local variations are observed in their slope. These deviations from the two-parameter Weibull model are due to the existence of several populations of defects with their own Weibull characteristics. These several populations may either pre-exist in the as-received fibers, or be due to the mechanical degradation during treatment in aqua regia or due to mechanical degradation during the elaboration of the composite. This explains the observed increase in deviations from the simple Weibull law from curve 15a to curve 15b. The deconvolution of σ-ε plots to determine the Weibull characteristics of each population of defects is possible in the case of a bimodal distribution of defects [16–18]. However, in this case, it is necessary for the σ-ε law at low and high strains to be only a function of the characteristics of a single population. Therefore, it is only possible to determine the Weibull characteristics of each population accurately by using the bundle test technique which the number of tested fibers is high. For evident reasons, it is impossible to perform this test on extracted fibers.

The values of the Weibull modulus m and of the mean srength $\langle\sigma_r\rangle$ of the fibers given above can be considered as correct for the evaluation of the mean mechanical properties of the fibers. However, they do not allow us to give a precise description of their mechanical behaviour.

TABLE V Mechanical properties of the Altex fibers deduced from Weibull statistics

	m	σ_L (MPa)	$\langle\sigma_r\rangle$ (MPa)
As-received fibers	4.3	2016	1824
As-received fibers treated in aqua regia	4.3	1873	1695
Fibers extracted from pure aluminium	3	1603	1432
Fibers extracted from a 6013 matrix treated 2 h at 540 °C	4	683	615

6.1.4. Mechanical properties of the fibers extracted from a 6013 matrix composite

In order to show the effect of the alloying elements on the tensile properties of the reinforcement, fibers were extracted from a 6013 matrix composite treated for 2 h at 540 °C. The corresponding Weibull statistics are plotted in Fig. 16 and compared with those of the fibers extracted from the pure aluminium matrix composite. Table V gives the Weibull parameters of these fibers.

From these results, it appears that the mechanical properties of the fibers extracted from the 6013 matrix are considerably reduced compared with those of the fibers extracted from the pure aluminium matrix. This

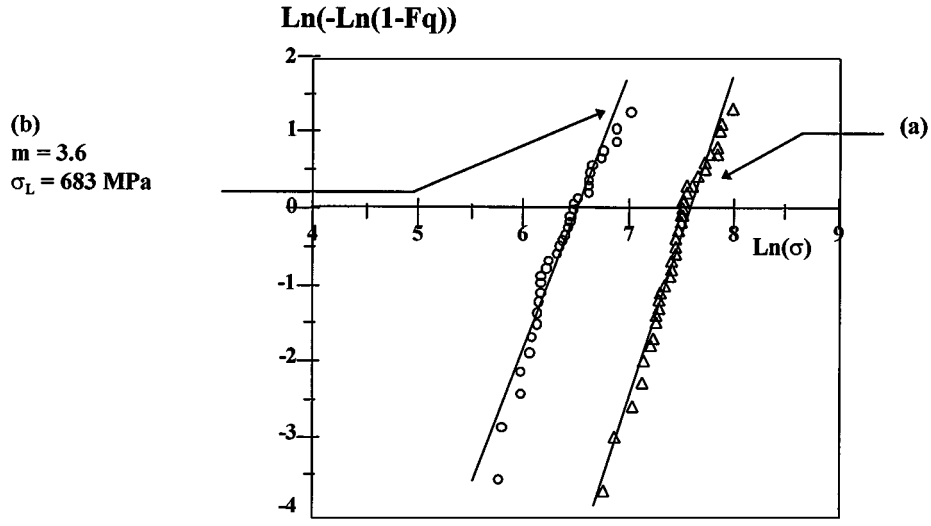

Figure 16 Weibull statistics of the Altex fibers after extraction from: (a) the pure aluminium matrix composite, (b) the 6013 matrix composite (treated two hours at 540 °C).

indicates that the chemical degradation of the fibers resulting from the reactions during the fabrication of the composite has a pronounced detrimental effect on the properties of the fibers. In this case, the decrease in strength of the fibers is of the order of 66%. In fact, 44% of this decrease can be attributed to the chemical degradation of the fibers, the rest being due to the processing technique.

6.1.5. Consequences of the degradation of the fibers on the longitudinal tensile strength of composites

The preceding results allow us to interpret the values of the longitudinal tensile strengths of the Al-Mg and Al-Mg-Si/Altex composites. In these materials, it was established that there were problems of reactivity between magnesium of the matrix and silica of the fibers. This reactivity is therefore likely to lead to a decrease in the strength of the fibers and subsequently in that of the composites. In the Al-Mg-Si/Altex composites, as will be discussed later, additional phenomena can lead to a further reduction of the longitudinal tensile strength of these materials. In contrast, in the Al-Mg/Altex composites, only chemical reactions can be responsible for the decrease in the longitudinal strength of the materials.

For example, in the Al-1wt%Mg/Altex fiber composite, characterized by a low magnesium content in the matrix, we noted that a reaction occurred during the processing by medium pressure infiltration, even if it was not very marked. Indeed, only a few Mg_2Si compounds were observed at the interfaces. This result corresponds well to the ultimate tensile strength obtained on this composite, which was not notably reduced ($\sigma_{UTS(Al\text{-}1wt\%Mg)}/\sigma_{UTS(Al)} = 80\%$).

After fifteen hours at 540 °C, the very large decrease in the longitudinal tensile strength (of the order of 50%, compared to the strength of the pure aluminium matrix composite) can be attributed to the deterioration of the fibers due to the extensive chemical reaction in the solid phase observed for such treatment times.

6.2. Influence of the precipitation phenomena on the longitudinal tensile strength of composites

6.2.1. Al-Si and Al-Mg-Si/Altex composites

In the case of the Al-Si/Altex fibers composites, no reactivity between silicon and the fibers is expected to take place. However, the presence of silicon in the matrix appears to be prejudicial to the strength of the composites.

Fig. 17 represents the evolution of the ultimate tensile strength of the composites investigated in this work (as a percentage of the value corresponding to an Al/Altex fibers composite) as a function of the silicon content of the matrix. In addition, we reported the results given by Nolte *et al.* [19] on the same graph, on which it is possible to distinguish two domains.

The first domain corresponds to silicon contents lower than 2 wt%. As the solubility limit of silicon in aluminium at a temperature of 540 °C is slightly superior to 1%, a large fraction of silicon is in solid solution for alloys of this low magnesium content domain. In this case, we observe a limited decrease (32% for an Al-2wt%Si matrix) in the longitudinal strength of the composites when the silicon content is increased. This may be due to the fact that: (i) silicon in solid

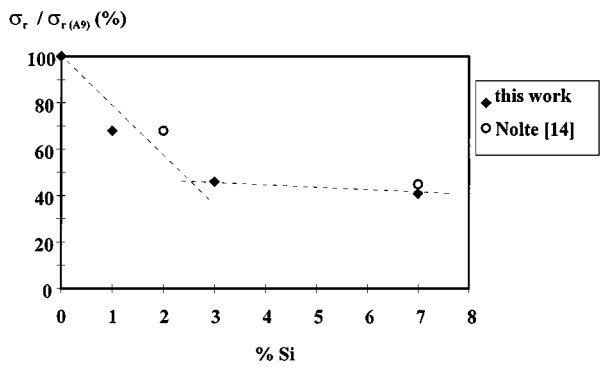

Figure 17 Evolution of the ultimate tensile strength of the composites (in percentage of the value corresponding to the pure aluminium matrix composite) as a function of the silicon content of the matrix.

solution tends to increase the work-hardening rate of the metal matrix and (ii) in unidirectional composites tested in longitudinal direction, the rupture is mainly controlled by the failure of the weakest fibers. After the failure of these fibers, the surrounding fibers are overstressed, the stresses applied to these fibers being all the greater as the work-hardening rate of the matrix is high. Thus, the rupture of the composites is likely to be promoted when the work-hardening rate is increased. However, as the work hardening variations are rather low, a second mechanism, suggested by Nolte *et al* [19], may be responsible for the reduction of strength of the Al-Si/Altex fibers composites: for silicon contents higher than the solubility limit of this element in aluminium, a fine precipitation of silicon at the interfaces may take place and be detrimental to the mechanical properties.

The second domain corresponds to industrial Al-Mg-Si casting alloys, with a high silicon and a low magnesium content. Chemical reactions are thus reduced, due to the very low magnesium content of these alloys. In contrast, the silicon content is such that the amount of eutectic silicon in the matrix is high, as can be seen in the micrographs of Fig. 13. The very important decrease in the longitudinal strength of composites is thus due to the presence of this brittle phase.

6.2.2. 6013/Altex fibers composites

The case of the 6013/Altex fibers composites is particular, in so far as the 6013 alloy contains not only magnesium and silicon but also other alloying elements, the effect of which has to be taken into account.

Referring to the results given in Table IVa, it appears that the mechanical properties of these composites are lower than those of the Al-1wt%Mg/Altex fibers composites (although the two materials have almost the same magnesium content), but also lower than those of the Al-1wt%Si-1wt%Mg/Altex fibers composites (which have almost the same magnesium and silicon content). This seems to be due to the fact that the 6013 matrix contains many alloying elements, which lead not only to chemical interactions between magnesium and the fibers but also to segregation phenomena of the alloying elements in the interfacial region between the matrix and the fibers during processing. These alloying elements tend to form different kinds of intermetallic compounds (Mg_2Si precipitates or more complex compounds of Al-Fe-Si-Mn type). These compounds are regularly distributed along the interfaces or in the matrix and can promote a premature failure of the composites.

6.3. Transverse tensile strength

In pure aluminium matrix composites, the transverse strength is essentially controlled by the mechanical properties of the matrix, as rupture takes place in this matrix and not at the fiber-matrix interface or in the fibers.

In the case of the alloyed matrix composites, the fracture behaviour described in Section 3.2.3 and the increase in transverse tensile strength in comparison with the pure aluminium matrix composite can be explained in two different ways, the relative importance of which cannot be easily evaluated:

(i) The strength of the interface is increased because of a chemical bond between the matrix and the fibers, so that the rupture strength of the fibers is reached before that of the matrix or that of the interface.

(ii) The fibers deteriorate because of the reactivity phenomena occurring during the processing or the thermal treatment of the composites, and are thus characterized by a transverse strength lower than those of the matrix and of the interface.

7. Conclusions

The study of the microstructure of the matrix and of the interfacial region of Al-Mg, Al-Si or Al-Mg-Si alloys reinforced with Altex fibers allowed us to establish the influence of alloying elements, such as magnesium and silicon, on the tensile properties of the composites tested in the longitudinal direction. Using binary Al-Mg and Al-Si matrices, it was shown that:

- Magnesium is responsible for reactions in both the liquid state (during processing) and the solid state (during solutionizing treatment). They lead simultaneously to an embrittlement of the fibers by the consumption of the silica binder and to a precipitation of brittle phases at fibre-matrix interfaces. In fact, these phenomena are not very noticeable during processing, if the magnesium content is not too high (of the order of 1 wt %). In contrast, they can become very extensive during a long term exposure at a high temperature. However, as classical treatment times are of the order of 2 h, the solid state reactivity is not very pronounced for such a treatment.
- Silicon has two main effects depending on its concentration. At a low content, this element is in solid solution and leads to a work-hardening of the matrix which seems to promote a brittle fracture of the composite. At a higher content, silicon forms acicular phases, the premature failure of which is responsible for the catastrophic failure of the composite.

In the case of the Al-Mg-Si alloy matrix composites, we confirmed the effects of magnesium and silicon and showed that the simultaneous presence of these two elements leads to their segregation at the interfaces in the form of large Mg_2Si precipitates during processing. Furthermore, the presence of other intermetallic compounds at the interfaces was found to be detrimental to the tensile strength of the composite.

Fig. 18 gives the classification of the detrimental effects linked to the alloying elements. As the maximum strength ($\sigma_{UTS(A9)}$) is obtained for composites with a pure aluminium matrix, data for other materials are given with the ratio $\sigma_{UTS}/\sigma_{UTS(A9)}$. It appears that the presence of brittle phases, whatever their origin, has the

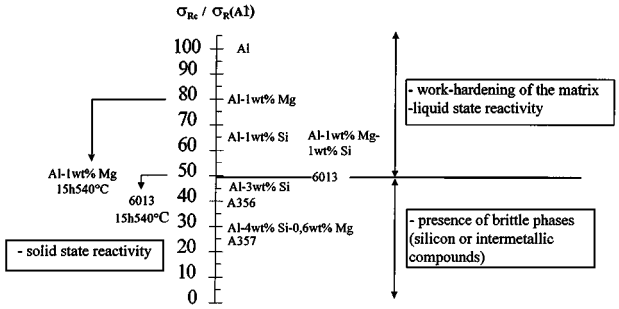

Figure 18 Effect of the alloying elements. Mechanism responsible for the decrease in longitudinal tensile strengths.

most detrimental effect on the tensile properties of the composite. σ_{UTS} is then less than 50% of $\sigma_{UTS(A9)}$. This is the case for the alloys with a high silicon content. Long treatments at a high temperature in the solid state (i.e., 15 h at 540 °C) lead also to the same decrease in σ_{UTS}.

In contrast, the reactivity between the matrix and the fibers in the liquid state, under our processing conditions, is much less prejudicial, as can be seen in the case of composites with a low content of alloying elements (Al-1wt%Mg, Al-1wt%Si-1wt%Mg) for which $\sigma_{UTS}/\sigma_{UTS(A9)}$ is about 0,65 to 0,8.

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