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Fracture analysis of aluminium matrix composite materials reinforced with $(Ni_3Al)p$

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This paper studies the influence of Ni_3Al intermetallic particles on the fracture behaviour of aluminium matrix (2014) composite materials. Intermetallics were obtained by mechanical alloying and by atomisation. The composite materials were manufactured by mixing, uniaxial compacting of a preform, and subsequent extrusion without canning or degassing. The study considered materials in extruded state and after T6 heat treatment. Assessments were made from the viewpoint of microstructure (by means of optical and scanning electron microscopy), and studying the reactions between the matrix and the reinforcement. These reactions produce a highly copper-enriched interphase. The influence of the reinforcement and state of the alloy on the fracture behaviour of the composite materials was studied through scanning electron microscopy.

1. Introduction

The development of metal matrix composite (MMC) materials in recent years has been one of the most important innovations in the field of materials. MMCs permit the obtainment of excellent combinations of stiffness and strength, thanks to the combination of physical and mechanical properties that complement metals and ceramics. Among the different types of MMCs, particulated composites are widely used due to the simplicity of their manufacturing, which implies low final costs. Aluminium matrix composites are among the most promising materials for wear-resistant and structural applications. The use of different possible reinforcement particles is proposed, and different aspects of service performance of these materials have been studied, specially wear [1, 2] and corrosion [3, 4] properties.

The two methods most widely used to obtain Al-based alloys with powder metallurgy (P/M) techniques are gas atomisation and mechanical alloying (MA) [5]. Aluminium alloys manufactured by MA generally present better mechanical properties due to the fine dispersion of precipitates in their microstructure [6]. This process can lead to the 'in situ' formation of intermetallics, and the particles, of irregular morphology, can be compacted better than gas atomised powders. Finally, the high density of dislocations, introduced in the powders by the high milling energy of the MA process, provides many paths for the diffusion of alloying elements in the base element [7].

Nowadays, the applications of MA include the manufacturing of intermetallics [8]. Of all the families of intermetallics (iron, nickel, aluminium, etc., based), Ni_3Al is probably one of the best known and characterised [9]. Its processing by P/M techniques provides structural materials with high strength and reliability [10]. Considering the different possible methods for obtaining intermetallics, MA is probably the system that permits the greatest variety of compositions within any metallic binary system [8]. Other processing techniques, such as atomisation, are also commonly used [8]. Recent research studied the feasibility of adding intermetallics to aluminium alloys in order to improve their wear behaviour [11, 12] and corrosion resistance [13].

This work considered an aluminium matrix composite material in which both the base alloy and the Ni_3Al reinforcement intermetallic are obtained by MA techniques. The same intermetallic obtained by atomisation is also used as reinforcement. One of the aspects of this work that differs from others dealing with similar materials is that the processing method followed (extrusion) is carried out without canning or degassing, but by direct extrusion of the powder.

2. Experimental procedure

The base aluminium alloy selected for the manufacturing of the composite material was aluminium alloy AA 2014. This alloy was manufactured by MA, using as base powders: aluminium (99.7% Al, size < 50 μm), copper (99.3% Cu, size < 50 μm), silicon (99.8% Si, size < 50 μm) and magnesium (97% Mg, size < 100 μm). The process control agent used in MA was Microwax C (Höechst). The AA 2014 base alloy powder, of composition (wt%) 4.5% Cu, 0.7% Si, 0.5% Mg, bal. Al, was optimised and characterised as has been described elsewhere [14]. MA conditions finally selected were 10 hours at 700 rpm in an argon atmosphere, and the balls/charge ratio (by wt.) used in the mill was 20 : 1, obtaining an appropriate grain size distribution for the extrusion process (75.2% < 45 μm, 90% < 53 μm) with guaranteed chemical composition and microstructural homogeneity [14, 15].

As has been noted, Ni_3Al was used as reinforcement for the composite material. Ni_3Al powders were obtained using two different techniques: gas atomisation and MA. The Ni_3Al intermetallic obtained by atomisation (from now Ni_3Al RST) was developed and characterised by Pérez *et al.* [9, 10] and the characterisation of Ni_3Al intermetallics manufactured by MA has been described elsewhere [15, 16]. Three different MA intermetallics were used in this study, namely:

- Ni_3Al mechanically alloyed for 5 hours (Ni_3Al 5MAST).
- Ni_3Al mechanically alloyed for 20 hours (Ni_3Al 20MAST).
- Ni_3Al mechanically alloyed for 20 hours and heat treated in vacuum at 1000°C (Ni_3Al 20MATT).

Ni_3Al powder manufactured by atomisation presented a typical spherical morphology. Ni_3Al 5MAST powders presented an irregular morphology, and some free nickel was still present. Ni_3Al 20MAST and Ni_3Al 20MATT particles were totally dense and had equiaxial morphology [15, 16].

The composite materials were obtained by mixing the intermetallic powders (10% by wt.) with the base alloy powders (in a laboratory mixer) for 30 minutes. The mix was uniaxially compacted at 300 MPa to obtain a preform, which was protected by means of graphite in oil suspension. This suspension was applied at 150°C, producing a fine graphite layer on the surface. The specimens thus obtained were extruded from 500°C/30 minutes at 350 MPa with an extrusion ratio of 25 : 1. Extrusion was performed without prior canning or degassing, and oxidation was not seen on the specimens after the process.

TABLE I Tensile strength results for studied materials both as extruded and after T6 heat treatment

	As extruded	T6 state
AA2014	446 ± 23 MPa	492 ± 37 MPa
AA2014 + Ni_3Al RST	444 ± 27 MPa	455 ± 34 MPa
AA2014 + Ni_3Al 5MAST	450 ± 27 MPa	434 ± 32 MPa
AA2014 + Ni_3Al 20MAST	440 ± 26 MPa	361 ± 27 MPa
AA2014 + Ni_3Al 20MATT	384 ± 23 MPa	369 ± 28 MPa

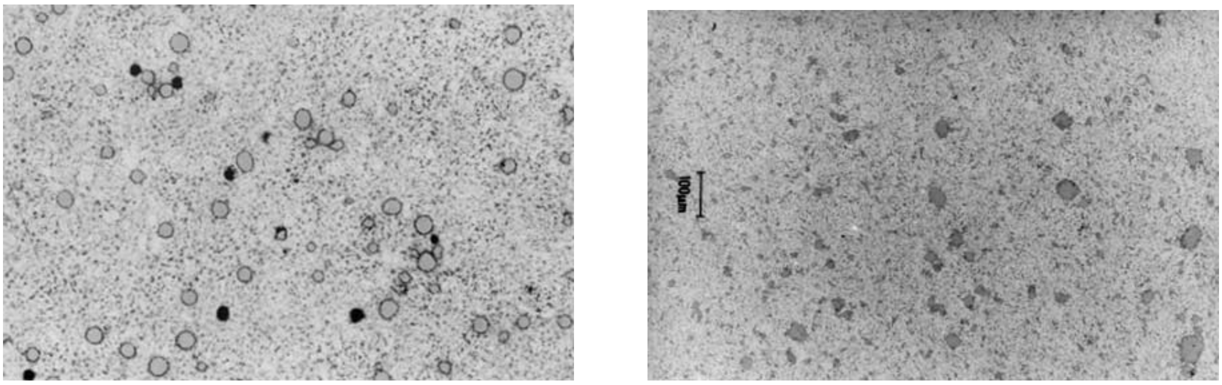
The properties of the different materials were determined in extruded state and after T6 treatment. The conditions used in this heat treatment were solution at 490°C (in a salt bath) and aging for 18 hours at 160°C. The solution temperature was optimised by means of differential scanning calorimetry (DSC) [14] and the aging temperature and time were selected according to ASTM standard B597.

A complete microstructural study was carried out on all the composite materials, in both extruded state and T6 state, by means of optical and scanning electron microscopy (SEM) combined with semiquantitative energy dispersive X-ray (EDX) analysis.

Tensile strength tests were carried out, with a load rate of 1.5 mm/min, making direct measurements on the bars obtained from the extrusion process, in both extruded and T6 state. Table I summarises UTS results. Finally, the fracture of all materials was studied by means of SEM.

3. Results and analysis

Fig. 1 shows the microstructural appearance of the composite materials obtained, whose microstructures are highly homogenous, both in the extruded material and even more after T6 heat treatment. This demonstrates the efficiency of mechanical alloying to obtain the aluminium matrix, and that mixing for 30 minutes is sufficient to distribute the reinforcement in the matrix.

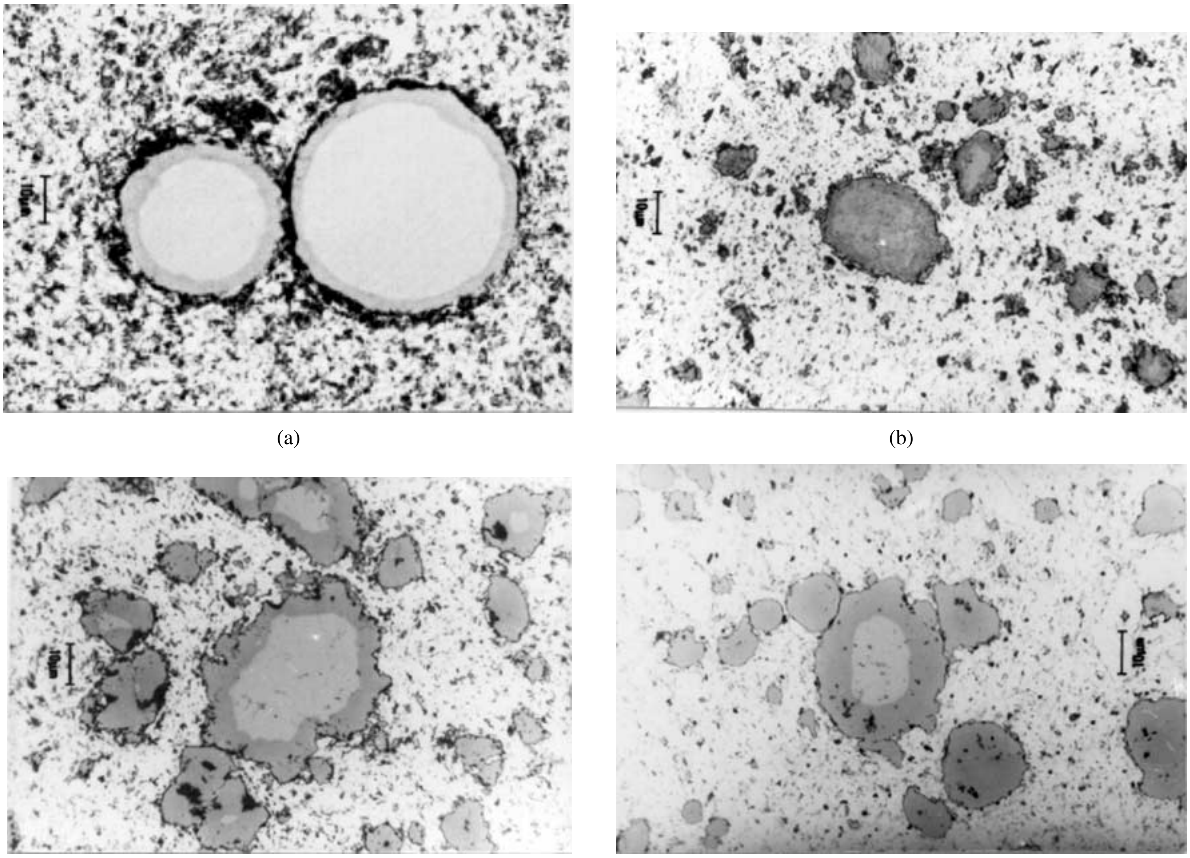


(a) (b)

Figure 1 Main microstructures of the extruded composite materials. MA24 reinforced with: (a) atomized Ni_3Al and (b) Ni3Al MATT.

In the base alloy, the precipitates are perfectly dispersed in the material, as a consequence of the good distribution of the alloying elements in the aluminium matrix. The extrusion process improves the distribution of the reinforcement in the matrix because of the high extrusion ratio employed. Thanks to that, intermetallics are well linked to the aluminium alloy matrix.

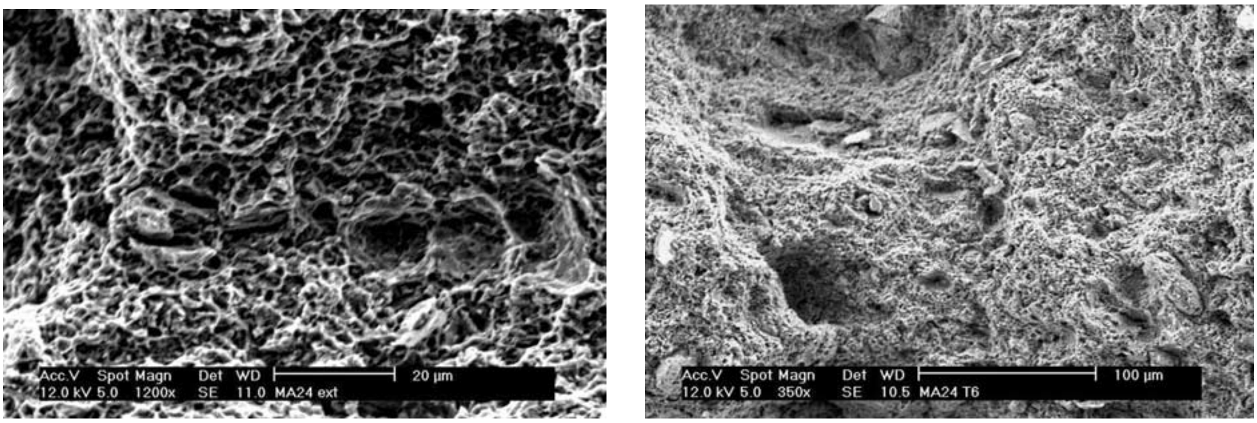
This link is even improved in heat treated materials, due to the fact that the interaction between matrix and reinforcement is greater in this case because of the diffusion caused at the boundary of the intermetallics, which leads to their perfect integration in the matrix. After T6 heat treatment, an interphase is formed between the matrix and intermetallic reinforcement, where copper-rich compounds are formed, giving rise to the diffusion of this element towards the interior of the intermetallic. Fig. 2 shows the reaction zone in the composite materials. This phenomenon occurs in all the particle-reinforced composite materials obtained by MA or by atomisation. This interaction is greater in particles manufactured by MA than in those manufactured by atomisation. The reason for this is that the MA process produces a high density of dislocations which facilitates the diffusion of alloying elements (in this case copper) within the particles [7, 16, 17]. Equiaxial morphology means a greater specific surface area, and consequently greater reactivity of the powders. The powders manufactured by atomisation



(a) (b)

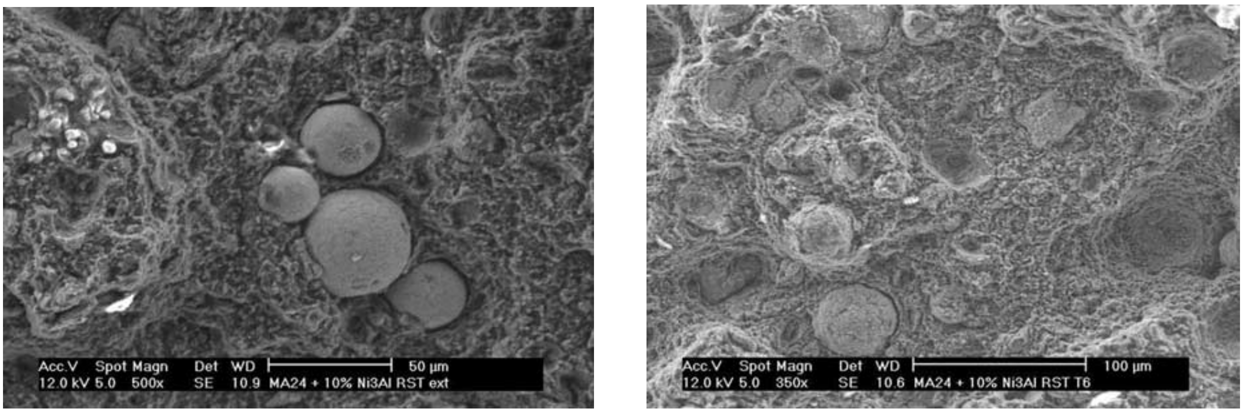
(c) (d)

Figure 2 Microstructure of the composite materials after T6 heat treatment, showing the reaction between matrix and reinforcement. (a) atomized Ni_3Al, (b) Ni_3Al 5MAST, (c) Ni_3Al 20MAST, and (d) Ni_3Al 20MATT.



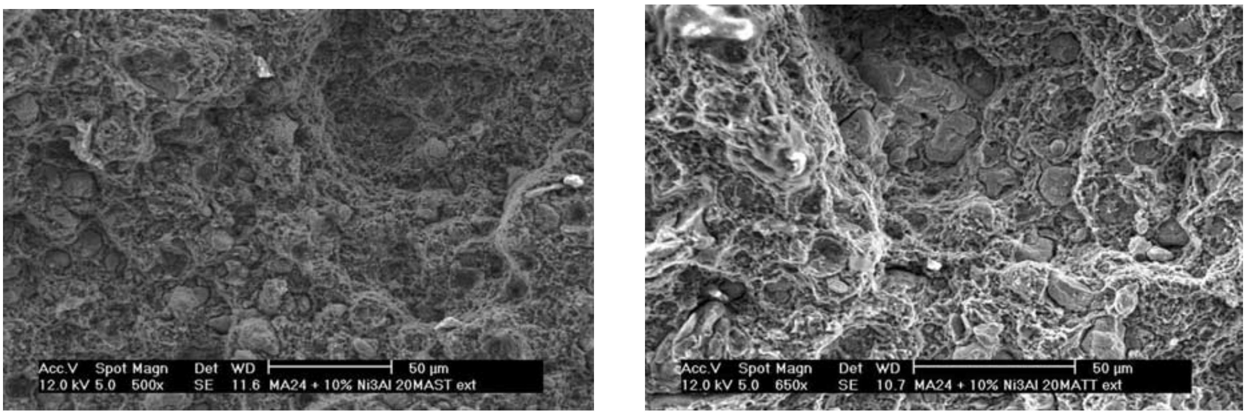
(a) (b)

Figure 3 Fracture surface of base alloy (a) in as extruded state and (b) after T6 heat treatment.



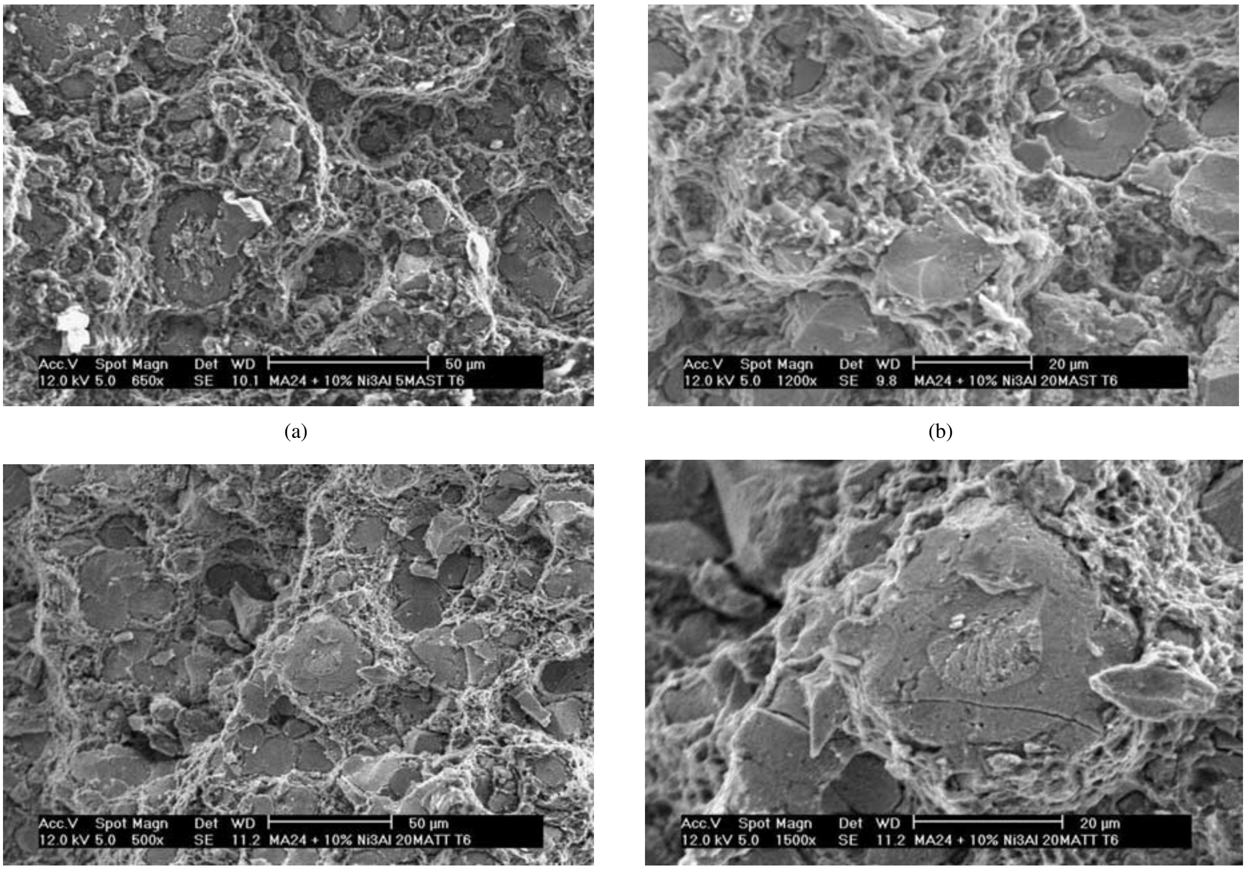
(a) (b)

Figure 4 Fracture surface of Ni_3Al RST composite material (a) in as extruded state and (b) after T6 heat treatment.



(a) (b)

Figure 5 Fracture surface of Ni_3Al MA as extruded composite materials: (a) Ni_3Al 20MAST and (b) Ni_3Al 20MATT.



(a) (b)

(c) (d)

Figure 6 Fracture surface of Ni_3Al MA T6 composite materials: (a) Ni_3Al 5MAST, (b) Ni_3Al 20MAST, (c) Ni_3Al 20MATT, and (d) Ni_3Al 20MATT (high magnification).

present a spherical shape, which provides greater stability, a lower specific energy and, consequently, lower reactivity. The good matrix-reinforcement interaction promotes the good mechanical properties exhibited by these materials [15, 16] compared to other aluminium alloys obtained by powder metallurgy [18, 19, 20].

Fig. 3 shows the fracture surface of base alloy, both in as extruded state and after T6 heat treatment. As expected, the base alloy presents high ductility, and dimples are present in the entire surface. It is evident that the process of powder manufacturing produces powder hardening that increases properties of extruded material, but the fracture analysis reveals the ductility of the matrix. After heat treatment, some ductility is lost, and dimples are not clearly observed as in extruded alloy.

The addition of intermetallics does not modify this behaviour (Figs 4–6), and numerous dimples can be appreciated in the fracture surface of composite materials. In Ni_3Al RST composite materials (Fig. 4), the reinforcement is clearly observed in the fracture surface, as a result of the weaker link between matrix and reinforcement. This link is enough to keep reinforcements bonded to the matrix, and they are clearly seen in the surface fracture. Stronger bonding is found between matrix and reinforcement after heat treatment as can be seen in Fig. 4b. Aluminium matrix keeps its ductility.

MA intermetallics promote different behaviour (Fig. 5). The bonding between matrix and reinforcement is better in this case than with atomised reinforcements. So the crack advances through the intermetallic particles, and they are fractured when the critical stresses reach them, especially for Ni_3Al 20MATT (Fig. 5b). The matrix is still ductile, and dimples can be observed in its fracture surface. After T6 heat treatment, the reactions between MA intermetallics and aluminium matrix are strong, as appears in the fracture behaviour of these composite materials (Fig. 6). Intermetallics are crushed in two parts, and the copper-enriched reaction zone is perfectly seen. Now there exists a real matrix-reinforcement link (a chemical one), better than the mechanical link obtained during extrusion process.

4. Conclusions

- The processing method followed for the obtainment of these composite materials (mechanical alloying, uniaxial compacting, hot extrusion without canning) is shown to be efficient for obtaining this type of materials, which reach their theoretical densities and consequently provide high performance.
- The addition of intermetallics does not modify the fracture behaviour of the matrix.
- The link of MA intermetallics to the aluminium matrix, better than atomised ones, can be appreciated in the analysis of fracture surface. The high specific surface characteristic of MA intermetallics promotes an improved mechanical bonding to the matrix, as well as chemical bonding after heat treatment.

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