INFLUENCE OF CONTENT AND PARTICLE SIZE ON PROPERTIES OF TiC REINFORCED 7075 ALUMINIUM MATRIX COMPOSITE

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Abstract

In this work mechanical alloying (MA) and sintering have been used to obtain aluminium matrix composites (AlMMCs) from AA7075 prealloyed powders and two types of TiC particles, nano- (n-TiCp) and micro-sized (m-TiCp).The aim has been to get a proper dispersion of the particles into the matrix, avoiding clustering and agglomeration, above all in n-TiC. Sintering of AlMMCs has been performed under argon atmosphere during 1h. Composite powders and compacts have been analysed by XRD, OM, SEM, TEM and microhardness. MA promotes a perfect TiC dispersion into the matrix after 16h of milling. The TiC particle size does not have significant influence on the final crystallite size and hardness of the AlMMCs but large quantities of n-TiC promotes larger crystallite size and lower hardness and density due to the lower ductility of the mechanical alloyed aluminium.

1 Introduction

Aluminium alloys are clearly the most used materials as matrices in metal matrix composites (MMCs), both in research and industrial applications, especially in transport. The reinforcement in MMCs, both the kind of material and the reinforcement size, is as important as the matrix material. Nowadays, ceramic discontinuous reinforcements such as SiC, Al_2O_3 and B_4C particles, are generally used due to their lower price and cost of manufacturing and their isotropic properties [1,2]. The most recent research related to aluminium metal matrix composites (AlMMCs) is focused on intermetallic and ceramic nanoparticles reinforcements [3-5]. Specifically, TiC has been recently investigated as a ceramic reinforcement due to its high melting temperature, low thermal coefficient of expansion, chemical inertness, extraordinary hardness and excellent wear and abrasion resistance [6,7]. The size of reinforcement particle is known to affect hardness, wear and corrosion resistance of the AlMMCs. Both tensile strength and ductility decrease with increasing particle size [8]. Therefore, decreasing the ceramic particulate size can lead to substantial improvements in mechanical performance of MMCs, especially in the case of using nanoparticles, which allow a greater improvement in properties with lower contents of reinforcement in comparison to micrometric ones [9-11].

AlMMCs can be processed by solid and liquid state techniques. Liquid state processing is generally cheaper and easier to handle, and the composites can be produced in variable shapes, using techniques already developed in the casting industry for monolithic metals. However there are some technical difficulties related to liquid state processing including reinforcement segregation and clustering, detrimental interfacial reactions, residual porosity and poor interfacial bonding which degrade the properties of the final materials. Meanwhile the lower manufacturing temperature used in powder metallurgy (P/M) processing can avoid strong interfacial reactions and to minimize the undesired reaction between the matrix and the reinforcement. The content and distribution of reinforcement, as well as the microstructure of the matrix can be controlled relatively easy. Hence the P/M products normally have superior properties than those of their cast counterparts. The extent of property improvement of MMCs at a given particle chemistry, size and volume fraction is related to the homogeneity of the particle reinforcement distribution. In special fatigue crack initiation is largely dependent on clustering of particles. However, homogeneous dispersion of ceramic nanoparticles in metals is difficult to achieve [12]. Mechanical alloying (MA) is a well-developed process for dispersing ex-situ nanoparticles more uniformly in metal matrix and therefore [13, 14] it has been successfully employed in this work to increase the quality of particle distribution in the aluminium. It should be an essential step within the powder metallurgy (PM) production route for manufacturing nano-AlMMCs.

In this study AlMMCs were produced with pre-alloyed AA7075 powders and TiC as reinforcement by mechanical alloying, pressing and sintering. The aim of this work is evaluate the effect of TiC particle size on the microstructure and mechanical properties of the AlMMC. On the other hand, the effect of the content of nanometric TiC was also studied.

2 Materials and testing methods

Pre-alloyed 7075 powder aluminium alloy (90,53 Al; 5,14 Zn; 2,51 Mg; 1,46 Cu; 0,21 Cr; 0,071 Fe y 0,024 Si) with particle size nominally sub 90 µm and average size of 30 µm was used. TiC particles with purity of 99% and average particle size of 20 nm and sub 50 µm were used as reinforcement. The as-received AA7075 and TiC powders were supplied by the Aluminum Powder Company Limited (APC) and Iolitec (nano) and Kennametal INC (micro) respectively. Fig. 1. shows the size and morphology of powders used in this work.

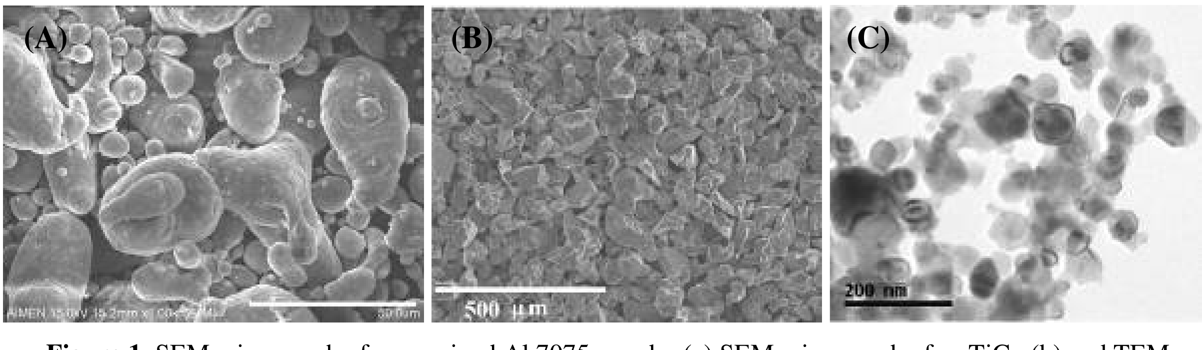

Figure 1. SEM micrograph of as-received Al 7075 powder (a) SEM micrograph of m-TiCp (b) and TEM micrograph of n- TiCp (c).

A cyclic process of MA (Fig. 2), typical in ductile materials, was carried out with a ZOZ Simoloyer CMO1 attritor mill, whose horizontal configuration avoids death zones resulting in a more homogeneous milling. Mill walls are made of AISI 304 stainless steel and the mill rotor of AISI 304/StelliteW/THM (Grade designation for the WC inserts used in the blades). Milling balls of 4.762mm are made of AISI 52100.

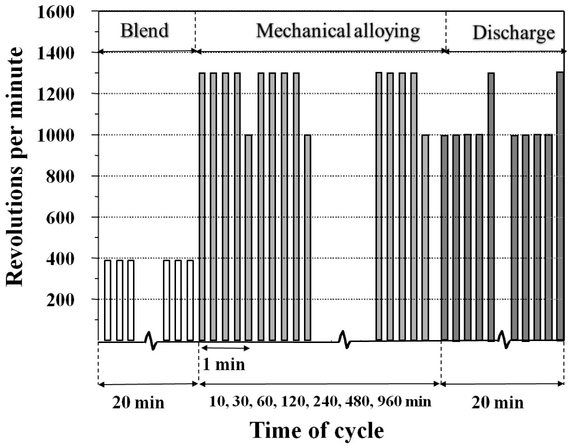

Figure 2. Operation and discharging cycle for the MA of the prealloyed AA7075 with n-TiCp and m-TiCp.

The mechanical alloying cyclic operation mode was performed with a 48s high rotary speed of 1300 rpm and a 12s low rotary speed of 1000 rpm (1 min cycle) during 10, 30, 60, 120, 240, 480 and 960 min. This mode allows modification of the process kinetics to avoid sticking and agglomeration. The discharging cycle is the opposite of the operation cycle (12s at 1300 rpm and 48s at 1000 rpm). Powder oxidation was prevented using an argon atmosphere and 0.5% of process control agent (Licowax C from Clariant) which, furthermore, reduces the excessive cold welding between AA7075 particles at the beginning of the mechanical alloying. Sintering was performed at 600ºC for one hour in an argon atmosphere to prevent an excessive oxidation of the composite material. Particle size and morphology evolution were studied by high resolution scanning electron microscopy (SEM, Hitachi S4800 II FESEM and JEOL JSM 6700 F FESEM). The distribution of the TiC reinforcement particles in the AA7075 matrix was studied by FESEM. Transmission electron microscopy (TEM) (JEOL JEM 2010 F FETEM) was used for analyzing particle size and morphology of the n-TiCp, extracting lamellae from different powder particles by means of focused ion beam (FIB). Microstructures obtained after sintering were analyzed by optical microscopy (OM) (Olympus GX51 microscope) and SEM (Jeol JSM-5410) after etching with Keller's reagent. To study the hardening caused by mechanical alloying on the particles, 0.01 Kg Vickers hardness tests were performed in a Shimadzu and in a Durascan Emcotest durometer according to UNE-EN-ISO 6507-1:2006. Structural changes were examined by X-ray diffraction (Siemens D5000), with 2θ from 3º to 90º (step 0.030º and 5 s/step), and the crystallite size was determined taking into account the medium width of the diffraction peaks and using Scherrer's formula.

3 Results and Discussion

The main parameter to define in mechanical alloying processes is milling time. The optimum milling time, which leads to avoid the formation of non-desired phases and pollution (normally induced by excessive processing times) in the obtained material, is usually achieved when the equilibrium between the processes of fracture and cold welding of the matrix particles is reached.

Electronic microscopy analysis show that as milling time increases, both particle size evolution and reinforcement distribution have a similar behaviour in all studied cases, as it is usual in this kind of processing techniques [7].

With short milling times, ductile aluminium particles get deformed and laminar structures are generated (Fig. 3). The composition of these structures, referring to the amount of reinforcement, varies clearly from one sample to another. Furthermore, some non-deformed particles from the initial aluminum alloy powder can be detected.

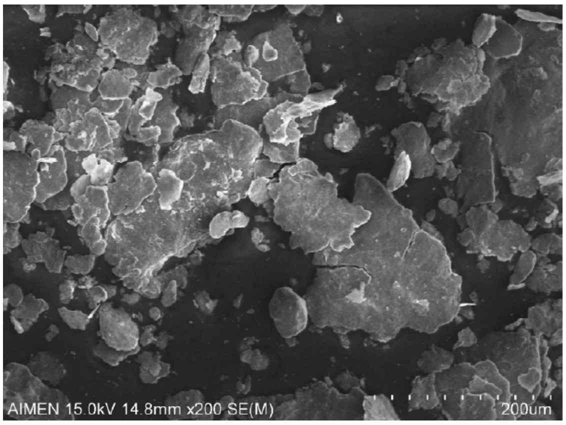

Figure 3. SEM. High resistance 2% TiC (20 nm) reinforced aluminium matrix composite particles after 60 minutes of mechanical alloying.

With longer processing times, cold-welding process predominates over fracture and previously described laminar particles get welded, increasing their size (Fig. 4-A).
After that, and due to the cold working, fracture process becomes stronger and so the laminar morphology gets lost. Later, cold welding continues and equiaxic morphologies are obtained.
With the longest processing times equilibrium between cold-welding and fracture processes is achieved (Fig. 4-B), and the obtained particles show such an homogeneity that their composition equals the percentage composition of the initial powder.

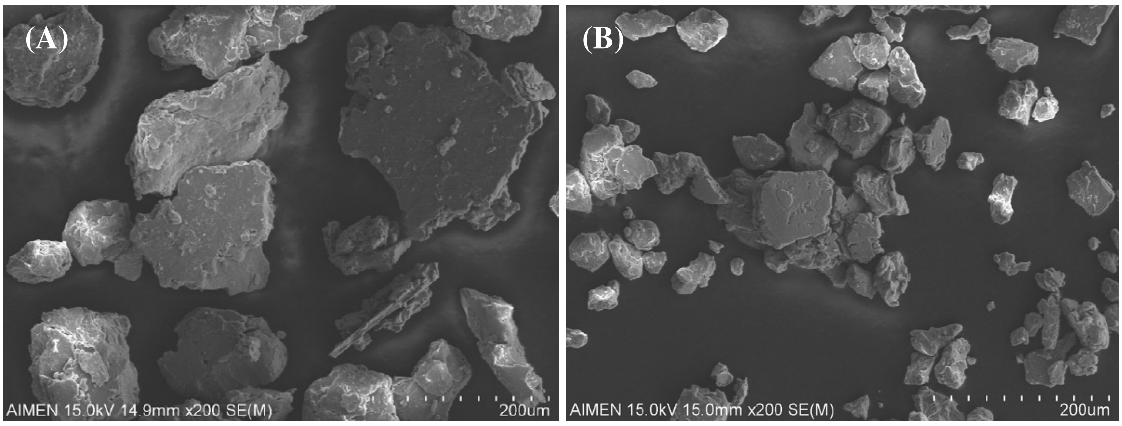

Figure 4. SEM. High resistance 2% TiC (20 nm) reinforced aluminium matrix composite particles after 240 minutes of mechanical alloying (A) and high resistance 2% TiC (20 nm) reinforced aluminium matrix composite particles after 960 minutes of mechanical alloying (B).

In the same way, as milling time increases, aluminium powder microstructure and aluminium crystallite size change. Initially, non-processed aluminium particles show a fully dendritic microstructure (Fig.5-A), which gets lost after only 60 minutes of mechanical alloying (Fig. 5-B).
In samples which were mechanically alloyed for longer times (960 minutes) aluminium grains are strongly deformed. It is easy to detect perfectly embedded and randomly dispersed particles in the aluminium matrix. Their size and their round-shape morphology are similar to those of the initial TiC particles. Agglomerations were not detected. Compositional mappings obtained by means of electronic microscopy techniques (SEM and TEM) allowed to determine that those ones are titanium-rich particles [15]

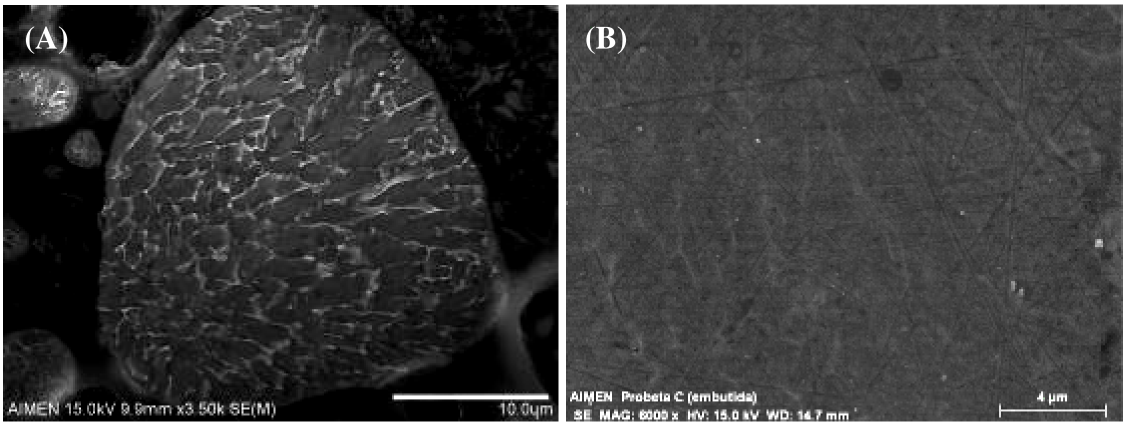

Figure 5. SEM: Dendritic microstructure of the original 7075 aluminium alloy (A) and loss of dendritic microstructure after 60 min of mechanical alloying (B).

The X-Ray diffraction analysis shows that there is not a clear relation between the size of the reinforcement particles and the size of the aluminium crystallite (Fig. 6).

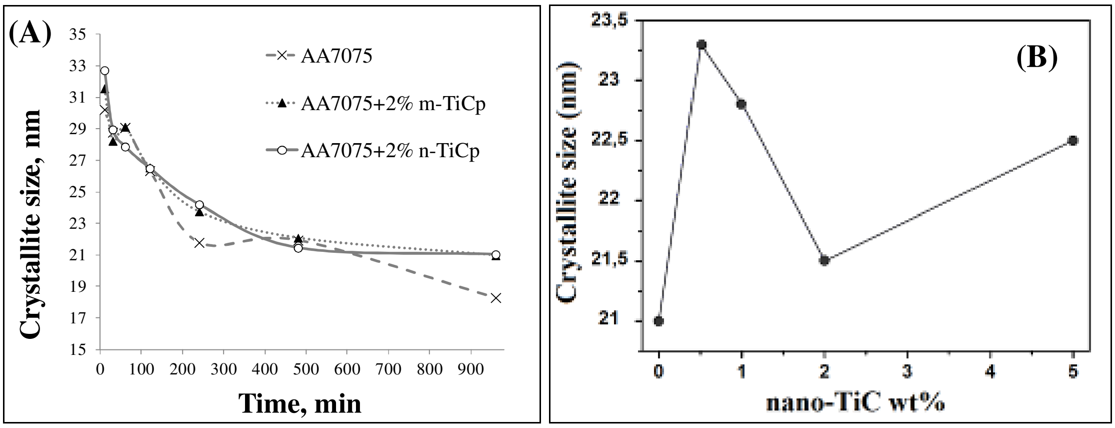

Figure 6. Aluminium crystallite size as a function of the reinforcement size and the milling time (A) and as a function of the reinforcement percentage after 960 minutes of mechanical alloying.

However, aluminium ductility decreases if it is reinforced, allowing lower deformations and generating greater crystallite sizes. In the non-reinforced Al alloy, more pronounced deformations are achieved and, if compared with reinforced alloys, a smaller crystallite size is obtained (Fig. 6-A). Referring to the reinforcement percentage, after 960 minutes of mechanical alloying, in samples with nano-TiC amounts from 0,5% to 2%, the crystallite size tends to decrease when TiC content increases (Fig. 6-B). Nevertheless, in samples with 5% of TiC, crystallite size increases again, decreasing the ductility of the pre-alloyed aluminium.
As mechanical alloying time growths, also does powder microhardness just in the same way as crystallite size decreases. Hardness increases from about 100 HV0.01 in the original aluminium up to 276 HV0.01 at the end of the process when prealloyed aluminium is mechanically alloyed without reinforcement. When 2% of n-TiCp is added, microhardness is increased up to 293 and up to 320 when 2% of m-TiCp is used. These values cannot be analyzed only regarding crystallite size.

After sintering in argon atmosphere it is possible to find in all samples very small zones with low reinforcement content (white pools in Fig. 7(A)). These are formed by an excessive cold welding of the AA7075 particles during the first moments of mechanical alloying. This process could be avoided using a higher content of process control agent which, at the same time, would be a contamination source. Segregation of alloying elements as Mg and Zn was found. Zn segregates to the outermost zones of the sintered samples (Fig. 7(B)), while Mg segregates to the outermost surfaces of the composite particles [15], maybe due to the low atomic weight of Mg and to its reactivity.

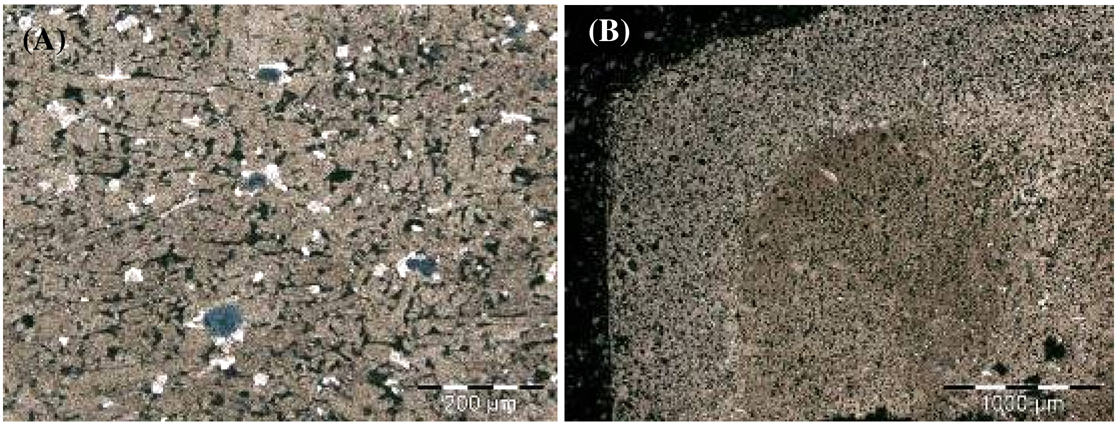

Figure 7. Aluminium-2%n-TiCp composite after 960 min of mechanical alloying and posterior argon sintering. Optical micrograph showing low-reinforced zones (white pools) and suitably reinforced areas (grey zones) (A) and Zn segregation to the outermost zones of the sintered samples (B).

On the other hand, n-TiC reinforcement particulates, identified as the light grey round particles of Fig. 8, are suitably dispersed over the matrix (as in the case of powders [15]).

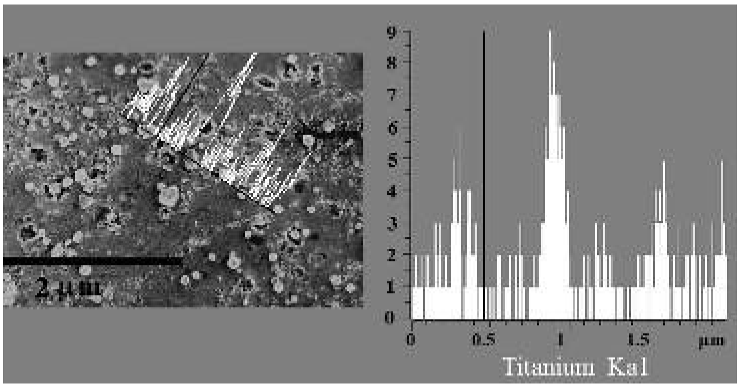

Figure 8. SEM micrograph and corresponding line scan EDS spectra showing distribution of Ti in Al-2%n-TiCp composite after 960 min of mechanical alloying and sintered 60 min in argon.

The presence of porosity is expected from calculation of relative density values, which is confirmed with experimental results (See Fig.7). All samples show porosity and this is increased with passing optimum levels (2% n-TiCp), the more the reinforcing weight percent, the more the porosities and the less the relative density. On the other hand nano-TiC seems to be more effective on sintering than micro-TiC when the same weight percentage of reinforcement is used, although the difference in density is not so significant.
Microhardness after argon sintering falls pronouncedly in all samples as a consequence of the thermal treatment. This drop in the hardness is more relevant in the 2% n-TiCp (104 HV0.01) compared to the counterpart 2% m-TiCp (166.01 HV0.01). This result is unexpected because of, nano-particles dispersed gradually in the Al matrix, must impede the motion of

dislocations, increasing hardness of the n-TiCp reinforced AlMMC. Moreover, grain/cell boundaries, arrested by dispersed n-TiCp,must prevent the increase of grain/cell size during the sintering. A reason for that could, be the presence of some original aluminium particles with lower quantity of reinforcement but more studies should be performed.

Finally, even after 960 min of MA equilibrium between cold welding and fracture, and after suitable distribution of reinforcement particles are achieved, those times of MA are quite large from the powder contamination point of view. Therefore, a few areas rich in Fe zones, probably detached because of wear of rotor, mill walls and milling balls, were found (blue round particles on Fig. 7) and elongated particle on Fig. 8).

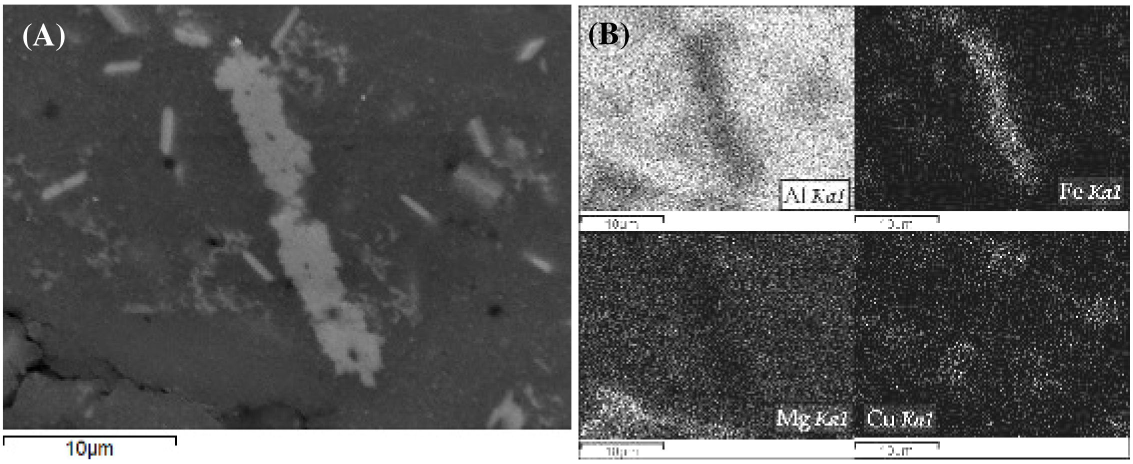

Figure 9. SEM micrograph showing elongated iron-rich particles (A) and energy-dispersive X-Ray spectroscopy (EDS) mapping of A (B).

4 Conclusions

- The aluminium particles initially have a dendritic microstructure which is lost only after 60 minutes of mechanical alloying. Increasing the time of mechanical alloying causes a decrease of crystallite size from 80nm at the beginning of the mechanical alloying to 21nm after 480 minutes; size stays the same with 960 min of milling time (both 2%TiC nano and micro reinforcement). At the same time the hardness of the TiC nano and micro reinforced aluminium 7075 matrix increases from about 100 HV0.01 at the beginning of the MA to almost 320 HV0.01 after 960 min. Therefore, the reinforcement particle size does not have significant influence on the final crystallite size of the aluminium.
- The deformation, fracture and welding of the aluminium particles during the mechanical alloying promotes the TiC deagglomeration and dispersion into the matrix after 16h of milling.
- The TiC percentage affects to the final crystallite size of the aluminium. In samples with TiC contents of 0.5 and 2% in weight, the crystallite size diminishes with the content of TiC. However, samples with more quantity of TiC (eg. 5%) present a larger crystallite size due to the lower ductility of the mechanical alloyed aluminium.
- After sintering, it is possible to detect the presence of some small zones, caused by cold welding between AA7075 particles in the first stages of mechanical alloying, with low reinforcement content. Segregation of some alloying elements was found, magnesium segregates to the outermost zones of the reinforced particles while zinc segregates to the outermost zones of the sintered samples. Hardness falls as a consequence of the thermal treatment, more markedly in the 2% n-tTiCp compared with the micro counterpart.

- Contamination by Fe particles, detached from mill walls, from mill rotor and from milling balls during mechanical alloying can be found in all samples.

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