1. INTRODUCTION

In recent decades, new manufacturing processes have been developed to produce metal matrix composites (MMC) that give composites with higher properties, or even to maintain the same level of properties at lower cost [1]. The manufacturing process is of great importance, since with the same matrix reinforcement system, it is the manufacturing process that will determine the property/cost relation [2]. Powder metallurgy can produce metal matrix composites in the whole range of matrix reinforcement compositions without the segregation phenomena typical of the casting process, and mechanical alloying serves to optimise the particle mixing stage, enhancing the reinforcement distribution. This work investigates the use of mechanical alloying plus hot extrusion to obtain Al6061 matrix composites reinforced with Si₃N₄, AlN and ZrB₂, and compares the result with the same composite materials obtained by more conventional powder metallurgy techniques. The incorporation of the reinforcement does not suffice to produce a significant improvement of the mechanical properties of the conventional powder metallurgy composites. Mechanical alloying breaks the reinforcement particle clusters, eliminates most of the defects present in these particles, decreases their size and enhances their distribution, which together with the metallurgical phenomena that change the metallic matrix, such as work hardening and oxide and carbide dispersion, produces an increase of about 150% in the hardness of the powder, when compared with the hardness of the as-received, non-reinforced aluminium powder alloy; and of 100% in the hardness and ultimate tensile strength of the consolidated materials, when compared with material of same composition processed by conventional powder metallurgy.

Key words: Aluminium, Powder Metallurgy, Mechanical Alloying, Hot Extrusion

Powder metallurgy can produce metal matrix composites in the whole range of matrix reinforcement compositions [3, 4] without the segregation phenomena typical of the casting processes. However, the matrix and reinforcement mixing process is the critical step towards a homogeneous distribution throughout the consolidated composite material, although subsequent processes, such as powder extrusion, can help to eliminate the clustering of reinforcement particles and therefore achieve their more uniform distribution throughout the metal matrix [5-7].

High-energy ball milling has been used to improve particle distribution throughout the matrix [8-14]. This technique, first developed by John Benjamin [15] to produce nickel superalloys hardened by oxide...
dispersion, is now known as mechanical alloying or mechanical milling. The process in which mixtures of powders are milled together is denominated Mechanical Alloying; it involves material transfer to obtain a homogeneous alloy by repeated deformation/welding/fracture mechanisms. On the other hand, milling of powders of uniform composition, in which material transfer is not required for homogenisation, is termed Mechanical Milling [16].

The use of hot extrusion in powder metallurgy avoids the sintering process and results in a full density final product. Hot extrusion of powders allows a high shear strain rate, which promotes high strength bonding between particles and a microstructure very similar to that of a wrought product. In the case of aluminium and its alloys, hot extrusion breaks the typical oxide layer that coats the powder, and hence gives better bonding of the particles [17].

The purpose of this work is to investigate the effect of mechanical alloying on the characteristics of composite materials obtained by powder metallurgy. Composites obtained by hot extrusion of mechanically alloyed composite powders are compared with materials of the same composition but processed by conventional mixing of matrix and reinforcement powders.

2. EXPERIMENTAL PROCEDURE

Aluminium AA6061 powder alloy (Alpoco – The Aluminium Powder Co. Ltd., England) was used as the matrix. This powder was produced by gas atomisation, and displays an equiaxed or quasi-spherical morphology. Si₃N₄, AlN and ZrB₂ (ART – Advanced Refractory Technologies, Inc., USA) were used as reinforcement materials. The fraction of reinforcement was 5% by weight. Figure 1 shows the powders used as raw material in this work. Table 1 shows the chemical composition of the matrix powder, and Table 2 the average diameter size and the theoretical density of the reinforcement powders.

To mix the matrix and the reinforcement powders, two processes were used: a conventional low-energy mixing and high-energy milling, i.e. mechanical alloying. The conventional mixing does not alter the original characteristics of the raw materials, and merely homogenises the reinforcement throughout the matrix. A horizontal ball mill rotating at 150 rpm for 90 minutes was used. The mechanical alloying was per-

Fig. 1 Raw materials Al6061 aluminium alloy (a), Si₃N₄ (b), AlN (c) and ZrB₂ (d).
formed in an eccentric high-energy ball mill (Fritshc Gmbh, model “Pulverisette 6”) with the following parameters: charge ratio: 6 : 1 (wt); ball diameter: 20 mm; ball material: AISI 420 stainless steel; speed 700 rpm. 1% (wt) of microwax was added to control the process (PCA). The high-energy milling time was that required to complete the mechanical alloying process, from the point of view of the phenomenological aspects [18], and was determined for each composition: 10 hours for the composite reinforced with Si3N4 and 12.5 hours for the composites reinforced with AlN and ZrB2. The mixed powders (low-energy mixing process) and composite powders (high-energy mechanical alloying) were examined by Scanning Electron Microscopy. Microhardness was determined in the as-received aluminium alloy and in the composite powders.

The powders were uniaxially cold-pressed at 300 MPa, and cylindrical samples of 25 mm diameter and around 16 mm were obtained. The cold-pressed samples were then hot extruded at 500°C, at a strain rate of 5 mm/s, without canning or degassing. The extrusion die was maintained inside a resistive furnace, which ensured good temperature control of the test. The sequence of the extrusion tests was randomly organized, in order to avoid systematic interference on the results. Figure 2 shows the various stages of the production of the composite materials.

The extruded materials were examined by optical microscopy and by density measurements; ultimate tensile strength (UTS) (1 mm/min) and hardness (Vickers, 10 KN) were determined.

### Table 1

| Chemical composition of the as-received PM 6061. |
|-----------------------------------------------|
| Mg | Si | Cr | Cu | Fe | Others | Al |
| 0.96 | 0.69 | 0.24 | 0.19 | 0.06 | <0.3 | Bal. |

### Table 2

| Material | Average particle size (µm) | Theoretical density (g/cm³) |
|----------|---------------------------|-----------------------------|
| Si3N4    | 8.6                       | 3.44                        |
| AlN      | 8.0                       | 3.26                        |

Figure 2 Routes used to obtain the composite materials.

#### 3. RESULTS AND DISCUSSION

3.1. Obtaining composite powders by mechanical alloying

Figure 3 compares the PM6061 with 5% AlN mixed powders with the composite particle obtained from the same composition after mechanical alloying. The AlN particles are bigger in the low-energy mixed powders (Fig. 3a) than in the mechanical-alloyed composite particle (Fig. 3b), and also show defects, such as cracks. The high-energy milling process reduces the reinforcement size, and tends to eliminate reinforcement clustering as well as reinforcement defects and sharp edges, producing a rounder reinforcement morphology, which will result in better composite properties. After mechanical alloying (Fig. 3b), each particle is a composite, displaying a uniform distribution of the reinforcement phase in the whole particle.

The degree of influence of the distinct reinforcement materials used in this work on the milling mechanisms can be attributed to their individual characteristics. Each kind of reinforcement material shows a different tendency to fragmentation when submitted to milling: the Si3N4 showing the highest tendency to fragmentation and the ZrB2 the lowest. This difference is attributed to their different hardness and morphology.

As shown in Fig. 1, Si3N4 particles have an irregular morphology, while AlN and ZrB2 particles have a more regular, polygonal morphology. These morphologies can in part explain the difference in the tendencies to fragmentation during milling: a more regular, polygo-
nal morphology has a better inherent resistance to impact, while an irregular one has a larger surface area and sites that concentrate the stress and propagate cracks. From the point of view of the particle hardness, technical data in the ceramic literature [19] state known that Si$_3$N$_4$ is harder than AlN, and the results obtained in this work confirm the greater brittleness of the harder particle.

Figure 4 shows the composite powders reinforced with (a) Si$_3$N$_4$ and (b) ZrB$_2$ after mechanical alloying. The higher tendency to fragmentation during milling of Si$_3$N$_4$ particles produces a finer distribution of the reinforcement particles into the aluminium matrix.

Mechanical alloying promotes structural refinement, high dislocation density, and oxide and carbide dispersion (the aluminium carbide is derived from the PCA contamination), which should result in a great increase of the matrix hardness. In addition to this, the effect of reinforcement of the harder ceramic particles also contributes to raising material hardness. Table 3 shows the microhardness of the as-received aluminium powder alloy and the composite powder produced by mechanical alloying. The great increase of hardness, around 150%, confirms the effectiveness of the process.

As mentioned above, Si$_3$N$_4$ is the reinforcement with the highest tendency to fragmentation during milling, and the composite powder reinforced with this material is the one that presents the finest distribution of the reinforcement throughout the matrix. ZrB$_2$ shows the least tendency to fragmentation during milling, and the composite powder reinforced with ZrB$_2$ is the one that presents the coarsest distribution of the reinforcement throughout the matrix. So the composite reinforced with Si$_3$N$_4$ displays the highest hardness among the composites analysed in this work, and the composite reinforced with ZrB$_2$, the lowest one. A more extended description of the pro-

Fig. 4 Composite powders reinforced with (a) Si$_3$N$_4$ and (b) ZrB$_2$ after mechanical alloying.
duction of composite powders reinforced with Si$_3$N$_4$ and AlN by mechanical alloying is given elsewhere [18].

### 3.2. Powder consolidation by cold pressing and hot extrusion

Table 4 shows the pressures necessary to extrude the mixed and composite powders reinforced with Si$_3$N$_4$ and AlN; the reported values are the mean of eight tests. As the extrusion of powders consists in deforming and bonding the powder particles [20, 21], the higher the hardness of the powder to be extruded, the higher the pressure required to extrude. As a consequence, the pressure required to extrude the mixed powders is lower than those required to extrude the composite powders. A more extended description of the extrusion of the mechanical-alloyed powders is given elsewhere [21].

The density of the extruded materials is always between 98 and 100% of the theoretical density, whichever the process used, which confirms the effectiveness of the cold pressing plus hot extrusion to obtain full density products from powder material.

### Table 4  Pressures necessary to extrude the mixed and the composite powders.

| Reinforcement | Powder       | Average pressure (MPa) | Standard deviation (MPa) |
|---------------|--------------|------------------------|--------------------------|
| Si$_3$N$_4$   | Mixed powder | 441                    | 23                       |
|               | Composite powder | 510                  | 23                       |
| AlN           | Mixed powder | 503                    | 32                       |
|               | Composite powder | 579                  | 30                       |

### 3.3. Characterization of the extruded materials

Figure 5 shows the microstructures of PM6061 reinforced with Si$_3$N$_4$ (Fig. 5a and b), AlN (Fig. 5c and d), and ZrB$_2$ (Fig. 5e and f), extruded from the mixed powders (Fig. 5a, c and e) and from the composite powders (Fig. 5b, d and f). These microstructures are observed in the transverse section of the extruded bars.

The materials extruded from the composite powders display a more homogeneous distribution of the reinforcement particles in the aluminium matrix. Also, the reinforcement particle is smaller in the mechanical-alloyed materials. As the Si$_3$N$_4$ is the reinforcement with the highest tendency to fragmentation, the composite reinforced with this material presents the finest dispersion of the reinforcement particles, as already observed in the powder characterization.

Table 5 shows the ultimate tensile strength (mean of eight tests) and the hardness (mean of twelve tests) of the composite materials obtained in this work. The UTS and the hardness of the mechanical-alloyed composites increases by around 100% above that of composite materials obtained by conventional powder metallurgy.

The increases observed are due to the extremely refined microstructure, oxide and carbide dispersion, reduction of the reinforcement particle size, elimination of the particle defects such as cracks and sharp edges, which reduce the tension concentration at critical points and the appearance of cracks during pressing or extrusion. All these effects are promoted by mechanical alloying.

As shown in Table 3, the hardness of the mechanical-alloyed composite powder is some 150% higher than that of the as-received aluminium powder alloy. However, the increase in the hardness of the extruded material due to the use of mechanical alloying is not so high: the hardness of the material extruded from the composite powders is around 100% higher than that extruded from the mixed powder, as shown in Table 5.

It is well known that extrusion enhances the material strength by work hardening, but the composite powders obtained by mechanical alloying have already an extremely deformed structure, and the effect of the temperature of extrusion on this deformed structure promotes recuperation. The effect of oxide and carbide dispersion throughout the matrix on the material hardness is not influenced by the temperature of extrusion.

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**Table 3**  Microhardness of the as-received aluminium powder and the mechanically alloyed composite powders.

| Material                  | Microhardness (HV) | Standard deviation |
|---------------------------|--------------------|--------------------|
| As-received PM6061        | 65.25              | 15.22              |
| PM6061/5% AlN after 12.5 hours of milling | 191.7              | 14.6               |
| PM6061/5% ZrB$_2$ after 12.5 hours of milling | 186.9              | 19.6               |
| PM6061/5% Si$_3$N$_4$ after 10 hours of milling | 208.9              | 13.4               |
Fig. 5  Microstructures of PM6061 reinforced with Si₃N₄ (a and b), AlN (c and d), and ZrB₂ (e and f), extruded from the mixed powders (a, c and e) and from the composite powders (b, d and f).

Table 5  Ultimate tensile strength and hardness of the composite materials.

| Reinforcement | Mixing process | UTS (MPa) | Standard deviation | Hardness (HV) | Standard deviation |
|---------------|----------------|-----------|--------------------|---------------|--------------------|
| AlN           | Conventional   | 206       | 2                  | 63            | 2                  |
|               | Mechanical alloying | 402     | 22                 | 126           | 6                  |
| Si₃N₄         | Conventional   | 214       | 3                  | 63            | 1                  |
|               | Mechanical alloying | 422     | 6                  | 119           | 4                  |
| ZrB₂          | Conventional   | 192       | 1                  | 59            | 2                  |
|               | Mechanical alloying | 320     | 20                 | 110           | 4                  |
4. CONCLUSIONS

The results obtained in this work confirm the efficiency of mechanical alloying and hot-extrusion to produce composite materials with highly refined structures, strengthened by oxide and carbide dispersion, with homogeneous distribution of the reinforcement particles, fully densified and showing better mechanical properties.

The use of mechanical alloying increases the powder hardness by about 150% when compared with the as-received non-reinforced aluminum powder alloy, and the UTS and hardness of the extruded materials by about 100%, when compared with the same materials, processed by conventional powder metallurgy.

Among the reinforced materials used in this work, the silicon nitride has the highest tendency to fracture during milling and promotes the highest increase in the composite strength.

5. ACKNOWLEDGEMENTS

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