INVESTIGATION OF THE MECHANICAL PROPERTIES OF MAGNESIUM METAL MATRIX COMPOSITES WITH A FINE DISPERSION OF CeO_2 PARTICLES.

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ABSTRACT:

This paper aims to study magnesium composites with reinforcement of cerium oxide. For this, Mg-CeO_2 composites were produced by powder metallurgy, with variations of 1%, 2% and 4% by weight of reinforcement. The temperature used was 620°C for a time of 6h. These materials were metallographically analyzed by means of optical microscopy, SEM and EDS, and mechanically tested through the Vickers microhardness. Archimedes density analysis was applied to address the influence of the addition of reinforcements in the magnesium matrix quantitatively. The statistical method used consists in the analysis of variations and Tukey's test. The samples with 4% by weight of reinforcement obtained the best results of densification and higher microhardness values, as well as a homogeneous distribution of reinforcement and few defects. Conversely, experimental results suggest that recrystallization and grain growth were detrimental to the mechanical strength of the composites, therefore, shorter sintering times are preferred.

Keywords: Magnesium matrix composites, reinforcement of cerium oxide, metallography, microhardness.

1. INTRODUCTION

Magnesium and its alloys have drawn considerable attention on the last decades due to larger demands for lightweight materials, especially for the automotive and aircraft industry. The main reason for this interest comes from the fact that magnesium has a density of 1,74g/cm³, that is, 35% lighter than aluminum (2,71g/cm³) and almost 4 times lighter than steel (7,86g/cm³) (1). Moreover, it can be noticed from the Ashby diagrams that these alloys have equivalent tensile and compressive specific resistance to aluminum and steel. Furthermore, magnesium has also the highest specific resistance in the shape of beams and plates among all the conventional metallic alloys (2). Aside from these advantages, there are still some technological challenges that need to be overcame, such as low deformability, low resistance to creep and corrosion (1). As a result of its hexagonal closest packed (hcp) crystalline structure, the main mechanisms of deformation at low temperature are the slippage of the basal planes {0001} and twinning, making the production of plates and extruded products a problem. A second obstacle for the manufacturing of extruded magnesium profiles is the strength-differential effect (i. e., when the yield strength under compressive load is considerably lower than under tensile load), which can be addressed to the preferential deformation and lower energy activation by twinning (3)(4).

In this context, several attempts are being made to develop stronger materials and to find adequate production processes. This research describes the process of fabrication and characterization of magnesium matrix composites reinforced with fine dispersions of cerium oxide, produced trough powder metallurgy. Many tries have been made in the past, with special success to the composites made with addition of Al_2O_3 (5)(6), SiC (7)(8)(9), Y_2O_3 (10), and Carbon nanotubes (11). The metallic form of cerium is widely studied and used as alloying element on magnesium alloys, nevertheless, the use of its oxide, CeO2, as reinforcement for magnesium matrix composites is still incipient.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

Magnesium matrix composites were produced using sieved magnesium filings obtained from a pure casted ingot with particle sizes between 125 and 149 microns,

97% pure cerium oxide (CeO_2) powder with particle size under 44 microns was used as reinforcement. The morphology of both powders was examined on a Hitachi T3000 Scanning Electron Microscope (SEM) using magnification of 500x, which is presented on the following figure. One can also notice the difference on the particles sizes of the powders.

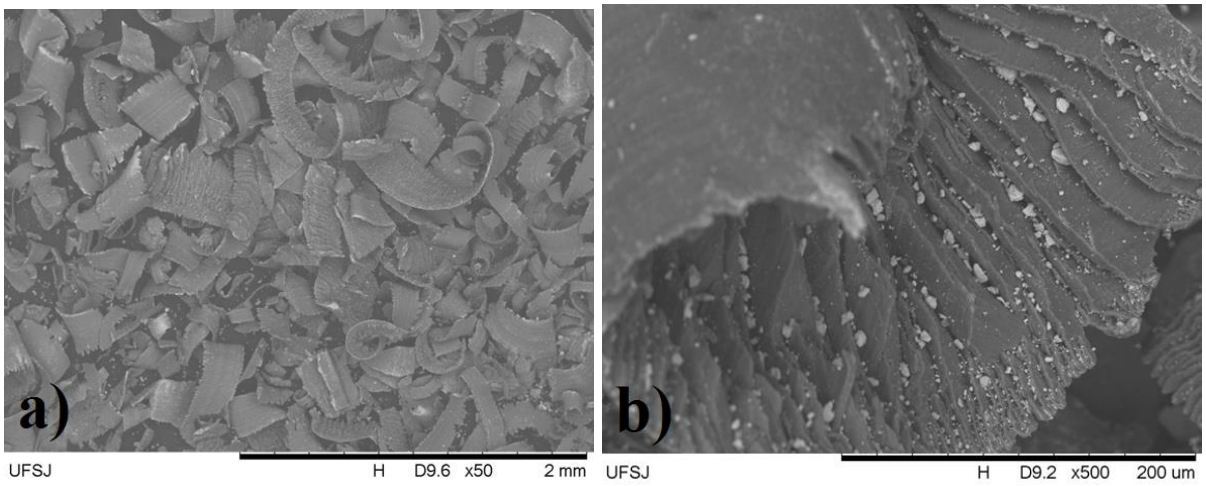

Figure 1. SEM image of a mixed Mg powder containing 4 wt.% CeO_2 displaying (a) the morphology of the particles that constitute the matrix, (b) the homogeneous distribution of reinforcements (white) along the magnesium filings (grey). The cavities on the surface of the matrix give support for the placement of the oxide particles.

2.2. Fabrication of Mg/CeO2 composites

Since there are apparently no results published on the literature for this specific composite material, additions of 1, 2 and 4 wt.% were specified on a general basis for magnesium matrix composites with ceramic micro particles as reinforcements (5-11). The powders used were mixed on an Across International PQ-N2 planetary ball mill for 1 hour at 580 rpm, with three containers per ladle, one for each sample. Thereupon, these were compacted on a uniaxial hydraulic press using a tool steel cylindrical die with internal diameter of 10mm. The compressive stress to be used was determined based on the relative density curve shown at the figure 2. Since the densification reaches approximately 85% at 250MPa, one can already create an interesting pre-sintering specimen at lower loads. In addition, previous results (12, 13) demonstrate excellent results for compacting pressures between 200 and 300 MPa, however, these were obtained for industrial magnesium powders. The density measurements were performed through the Archimedes method, on a Marte AL500C

analytical balance, using absolute ethyl alcohol (99.5% pure), at a refrigerated room maintained at 22°C. The ASTM standard B311-13 was used as guideline for the experimental procedures.

The samples were sintered on an induction-heated furnace with inert atmosphere created purging pure argon at flow rate of 10 L/min. In accordance with the results reported by Hal et al. (13) on a similar study, the finest mechanical properties are obtained for sintering temperatures between 610 and 630°C, hence the temperature adopted was 620°C for a time of 6 hours.

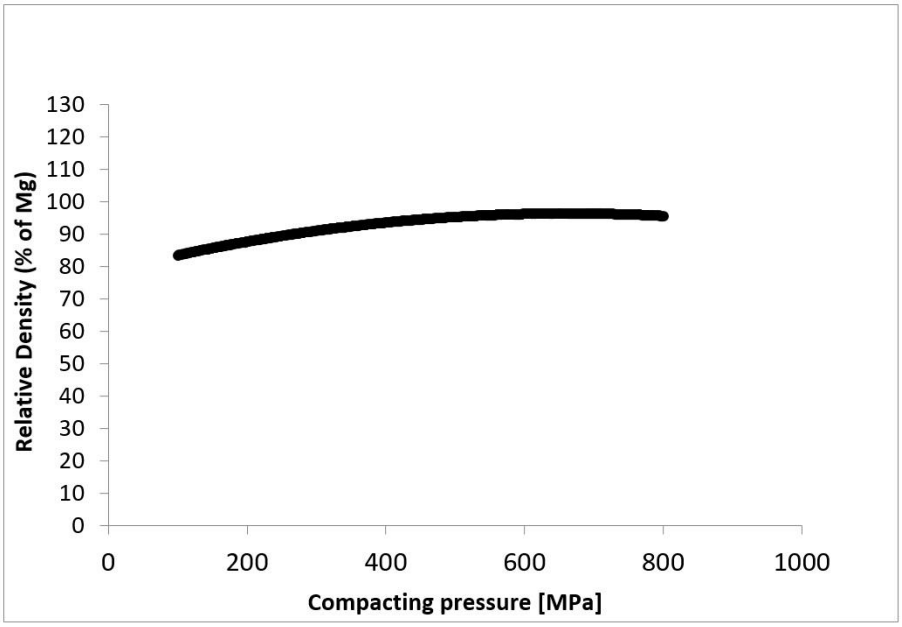

Figure 2. Relative density curve obtained from green density measurements of magnesium filings compacted from 100 to 800 MPa in intervals of 25 MPa.

2.3. Characterization

The samples had their densities measured before and after the sintering procedure through the Archimedes method, on a Marte AL500C analytical balance, using absolute ethyl alcohol (99.5% pure), at a refrigerated room maintained at 22°C. The standard ASTM B311-13 was used as guideline for the experimental procedures. The data displayed on the figure 2 has also been acquired with this procedure. Subsequently, the samples were cold mounted and prepared for metallographic analysis on SEM, the grinding and polishing process followed the methods described on the standard ASTM E3-11. Thereafter, the samples were etched by swabbing for approximately 120s with a solution containing 1ml of HNO3, 75 ml of ethylene glycol

and 25 ml of water, as recommended by Leng, Y. (16). Thereafter these were also with an Olympus BX51 light microscope. The microhardness of the specimens was measured in conformity with the standard ASTM E384-16 using a Mitutoyo MVK 54 G1 microhardness tester equipped with a pyramidal indenter angled at 136°. The load applied was 200gf for a time of 15s.

2.4. Statistical treatment of the results

The only variable of proportion on this study was the amount of reinforcement (1, 2 or 4 wt. %). The experimental level of the investigated factors conceives a planning corresponding to 3 conditions, each one with 3 replications. Two factors were investigated using the tool ANOVA, the density and the Vickers micro hardness, both after sintering.

3. RESULTS AND DISCUSSION

3.1. Production procedures

The characterization of the powder on the form of fillings presented heterogeneous distribution of particles with appearance of arc chips, with negligible thickness and width of approximately 200 µm. The following SEM image, figure 3 (a), also shows that there is almost no oxidation on the matrix.

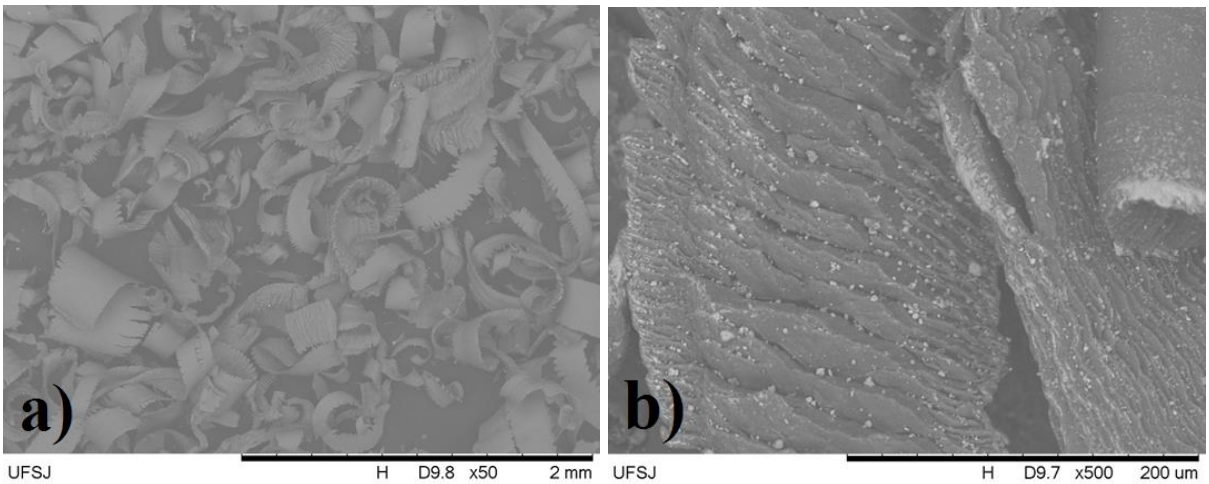

Figure 3. SEM images presenting (a) the morphology of the matrix particles and the apparent absence of oxidation, (b) the homogeneous distribution of reinforcements along the matrix.

The figure 3 (b) confirms the efficiency of the mixing process, as one can easily notice that the ultra-fine oxide particles (white) were deposited homogeneously on the cavities and irregularities of the matrix particles.

In order to determine the optimal compacting pressure, the relative green density curve previously presented at the figure 2 was established. This result revealed an excellent compacting behavior of the powder, which was able to achieve densifications up to 80% under loads as low as 100Mpa. This outcome can be attributed to the arc shape of the fillings that differently from the conventional powder with spheroidal particles, can easily fill the interstitial vacancies and therefore, can be better accommodated within the matrix after pressing.

Considering the necessity of breaking eventual nanometric oxide layers that may be involving the matrix particles and thus, could be significantly detrimental to the diffusion mechanism that drive the sintering process (18, 19), a compacting pressure of 250MPa was adopted. The coming figure exhibits a green body (Mg + 4%CeO_2) compacted with 250MPa. One can clearly examine the delineation of the powder formed by the cerium oxide particles (white), in addition to a compacting defect that also outlines the distribution of the cerium oxide along the magnesium filings that constitute the matrix.

3.2 Microhardness

The sintered specimens presented a severe drop on the microhardness in comparison to the green samples. From the prospect that a high concentration of oxides would decrease the diffusion rate and slow down the sintering process, longer sintering times should be used to achieve better sintering. Likewise, it was expected that the fine cerium oxide particles could block the grains growth during the sintering process, preventing any loss on the mechanical resistance due to microstructural coarsening. Many authors investigated the process of recrystallization and grain growth on pure magnesium and its alloys. Fromageau describes a slight grain growth starting at 400K (20), similarly, Miao investigated the grain growth on a AZ31 alloy and noticed an extremely quick advance of this process after 723K (21). Therefrom, it can be concluded that the hardness losses are related to the considerable grain growth of the matrix, in accordance to the Hall Petch effect.

Table 1. Average Vickers microhardness results for the samples with pure magnesium, 1 %, 2% and 4% in weight of reinforcement, and for a die casted pure magnesium ingot.

Material	Microhardness (HV)
Mg – Die casted	43.0
Mg - Green	42.7
Mg + 1%CeO_2 - Green	43.9
Mg + 2%CeO_2 - Green	46.0
Mg + 4%CeO_2 - Green	46.4
Mg - Sintered	28.3
Mg + 1%CeO_2	29.3
Mg + 2%CeO_2	32.4
Mg + 4%CeO_2	33.4

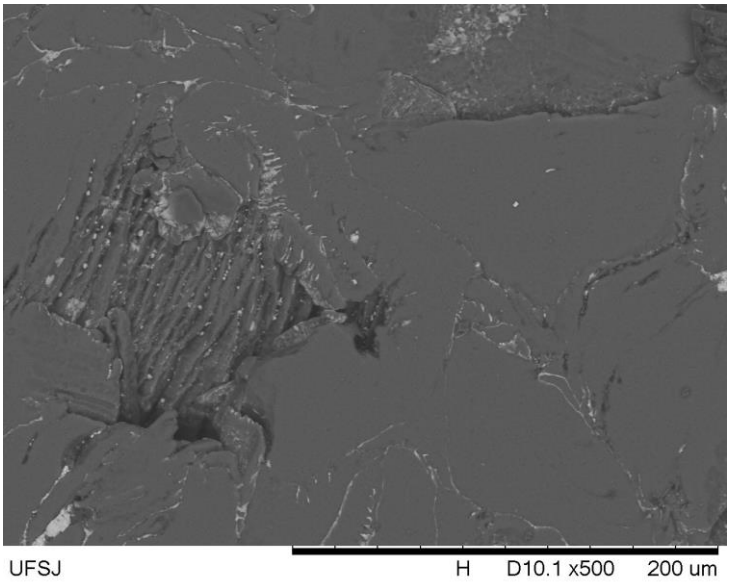

Figure 4. Green body (Mg + 4%CeO_2) compacted with 250MPa.

4. CONCLUSIONS

Sintering can be efficiently used to produce final products of magnesium; however, as the process parameters have a direct influence on the quality of the final product, these should be carefully studied. The magnesium filings attained good mixing and excellent compatibility, what coupled with the possibility of being produced from industrial wastes, at reduced cost, makes this a preeminent raw material for powder metallurgy. In spite of the losses of mechanical strength caused by the grain growth, it is noticeable that the presence of CeO_2 increases the strength

of the composite, which archived a difference up to 18% in hardness between pure magnesium and the specimens with 4 wt. % of reinforcement.

5. ACKNOWLEDGEMENTS

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