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EFFECT OF HIGH ENERGY MILLING ON THE MICROSTRUCTURE OF Al_2O_3-10%Fe METAL CERAMIC MATRIX COMPOSIT

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Abstract. *The Aluminum oxide's (Al_2O_3) ceramic phase with its high hardness and strength, allied with the iron's metallic phase, which it presents high ductility and high fracture toughness, make it a strong candidate to replace conventional materials such as WC-Co. The present work investigates the effect of high energy milling on the dispersion of Al2O3 and Fe phases. To achieve this goal, powders of Al_2O_3 and Fe in the proportion of 10% by mass of Fe were milled in the planetary mill Pulverisatte 7 of high energy. The powders were milled for 50 hours and they were collected at 5, 15, 30 and 50 hours. Throughout the work will be presented images of SEM, where greater homogenization between phases was observed, as well as the XRD to detect the present phases and to analyze the reduction of the sizes of the crystalline peaks.*

Keywords: *Al_2O_3, Fe, Powders, High Energy Milling.*

1. INTRODUCTION

Among the various ceramic materials used in the industry has the Aluminum oxide (Al_2O3), which in turn has low-density properties, low thermal conductivity, high modulus of elasticity, high resistance to thermal oxidation and abrasion. Allied to this has excellent properties of electrical insulation and the ability to withstand high energy without deforming. However, some properties such as low fracture toughness and high brittleness have considerably restricted their application. In order to improve tenacity properties, much has been studied on the insertion of reduced size Al_2O_3 particles in a metal phase (Fe, Co, Mo, We and Cu) and their results indicate to a new perspective on the improvement of the stress properties of fracture toughness and wear resistance of these composite materials. In the present work the iron, Fe has used as a metallic phase. (Xue et al., 2003; SING et al., 2013)

Powders can be obtained in a variety of forms; however, high energy milling deserves attention because it allows the production of ultra-fine gauge powders in the solid-state. This process consists of repeated cycles of deformation, cold welding, cold re-welding and fragmentation of the powder particles. (KOCH, 1991; SURYANARAYANA, 1998).

These deformation and fracture processes define the dispersion of the components, the homogenization and the final microstructure of the powder. High Energy milling has high speed and high impact frequency of the particles of grinding bodies, thus producing deformation of the crystalline structure, resulting in powders with small crystallites and large surface area. In addition, the evidence of solid formation can be observed and even the amorphization of the material happens. (TOMASI, 1998).

This study aims to investigate the effect of dispersing Alumina particles in ductile iron phase through the effect of high energy milling of powders composite Al_2O_3-10% Fe, from the application of scanning electron microscopy techniques (SEM) and X-ray diffraction (XRD).

2. MATERIALS AND METHODS

To the achievement of the Al_2O_3-10% Fe composite, the following steps were performed:

2.1. Initial powders

The Al_2O_3 and Fe powders used for the present study were obtained from the Laboratory of Ceramic Materials and Special Metals (LMCME) of UFRN. A total of 15 g of powder mass was used, of which 90% of Al_2O_3 and 10% of Fe. In addition, the characterizations of the initial powders were: the X-ray diffraction (XRD) and scanning electron microscopy (SEM).

2.2. High energy milling.

To the acquisition of the Al_2O_3-10%Fe composite, the high energy milling technique was used in a Pluverisette 7 planetary mill. The initial powders were initially placed in a crucible together with the ball, applying a ratio of 1: 4 (powder: ball) using ethyl alcohol. After that, the grinding process began. All powders were milled for 50 hours at a constant speed of 400 rpm. During the milling, samples were collected after 5, 15, 30 and 50 hours, to better understand the evolution of the technique.

2.2.1. Characterizations

The processes applied for the characterization of Al_2O_3-10% Fe composite powders were: scanning electron microscopy (SEM) and X-ray diffraction (XRD).

Scanning Electron Microscopy (SEM): Scanning Electron Microscopy (SEM) analysis was used to observe the shape of the initial powders and the dispersion of the Alumina phase in the iron phase of the ground powders. The equipment used to perform the SEM was the HITACHI TM 3000 of the Structural Materials Characterization Laboratory (LCEM) of the Department of Materials Engineering at UFRN.

X-ray Diffraction (XRD): X-ray diffraction (XRD) was used to detect the present phases and observe the effect of milling on the crystallinity of ground powders. The equipment used was the XDR-7000 SHIMADZU belonging to the Structural Materials Characterization Laboratory (LCEM) of the Department of Materials Engineering of UFRN.

3. RESULTS AND DISCUSSIONS

3.1. Initial Powders

First, the scanning electron microscopes and the X-ray diffractograms of the initial powders will be presented.

Figure 1 (a-b) shows the initial micrographs of the Al_2O_3 and Fe powders, respectively, obtained by SEM, which shows the typical shapes and initial agglomeration states of the materials.

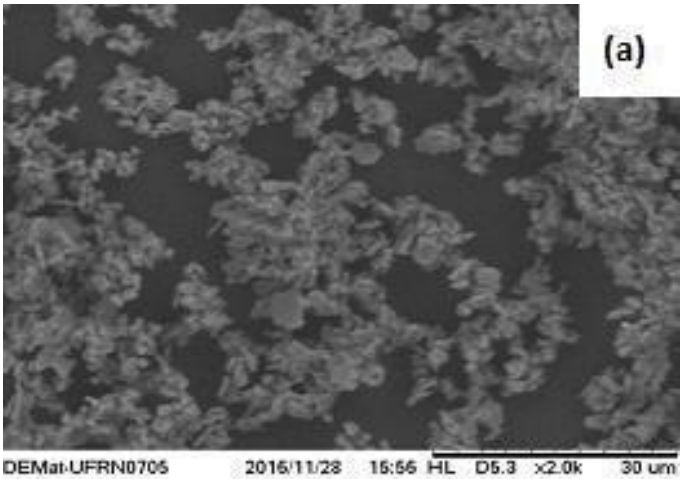

Figure 1a. Micrograph of Al_2O_3 powder

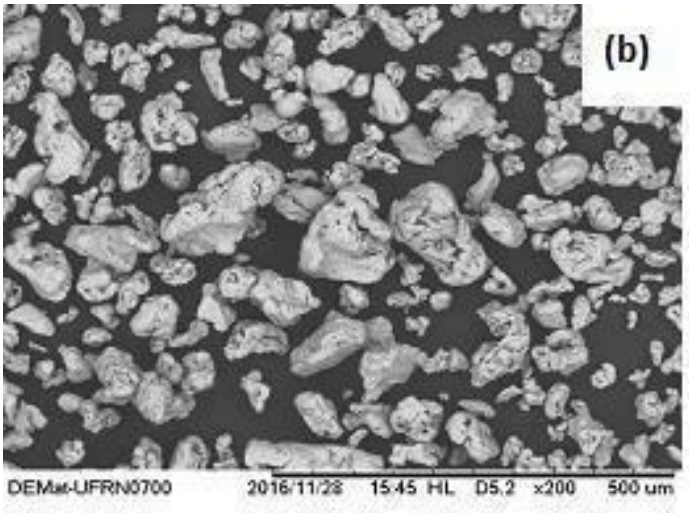

Figure 1b. Micrograph of Fe powder.

Figure 2 (a-b) shows the XRDs of the initial Al_2O_3 and iron powders, where it is possible to reveal the initial situation of the characteristic peaks of each.

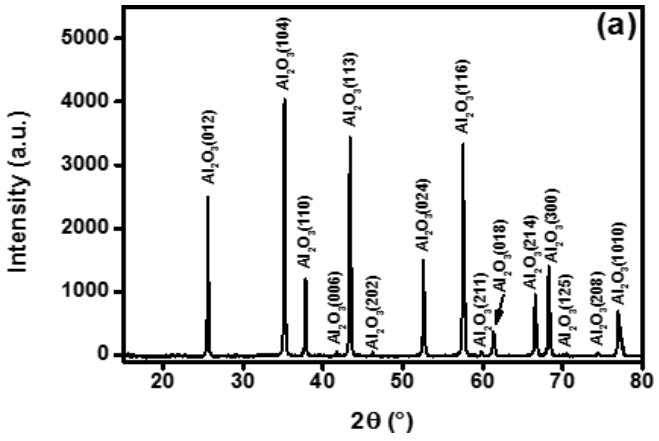

Figure 2a. X-ray diffraction of Al_2O_3 powder

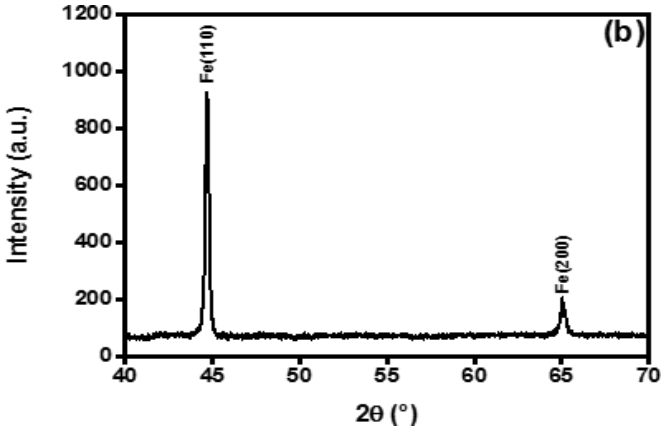

Figure 2b. X-Ray diffraction of Fe powder

3.2. High Energy milling

The second stage of the work will show the results of the characterization of the milled powders, analyzing the effect of the milling time on the microstructure and the crystalline structure of the Al_2O_3-10% Fe composite powders.

3.2.1. Effect of the time of the High Energy milling on the morphology of the milled powders.

Scanning electron microscopy analysis showed the influence of milling time on the morphology of the milled powders. The objective was to identify the two phases of the composite (Al_2O_3 - 10% Fe), to analyze the effect of the high energy milling in the refinement of the phases and the dispersion and homogenization of the powders.

Figure. 3 (a-d) presents through the micrographics of powders of Al2O3-10% Fe milled for 5, 15, 30 and 50 hours, in order to show the evolution of the characteristics of the particles during the high energy milling process. The particles with lighter staining are iron, while the darker particles are Al_2O_3.

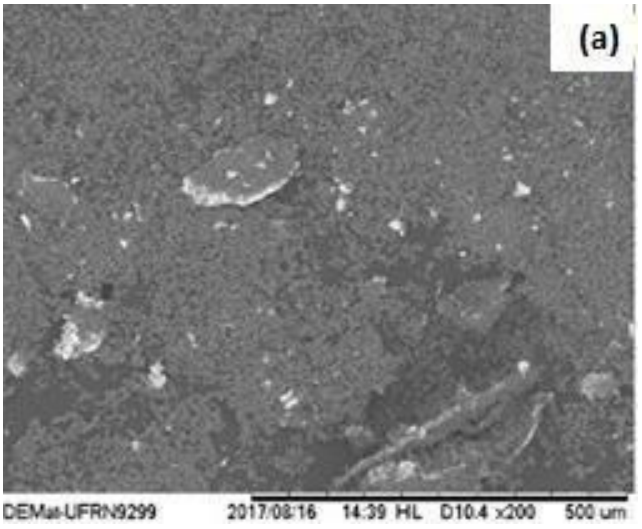

Figure 3a. Micrograph of Al_2O_3-10% Fe powder composite milled for 5 hours.

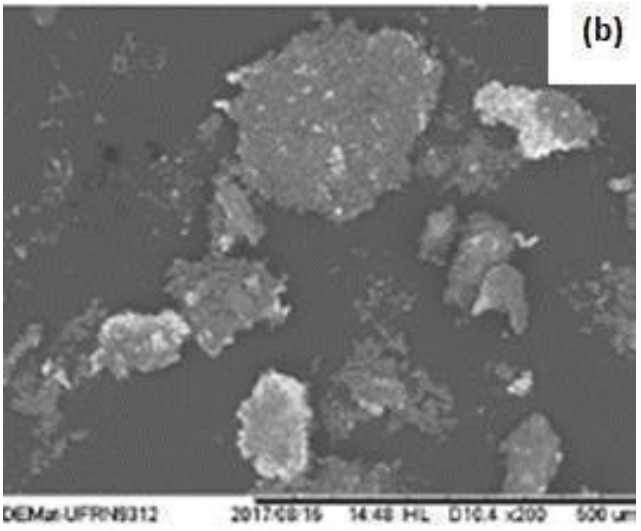

Figure 3b. Micrograph of Al_2O_3-10% Fe powder composite milled for 15hours

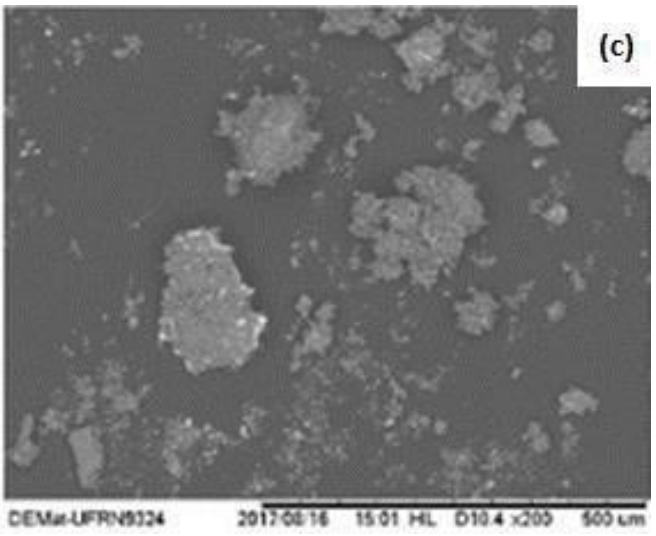

Figure 3c. Micrograph of Al_2O_3-10% Fe powder composite milled for 30hours

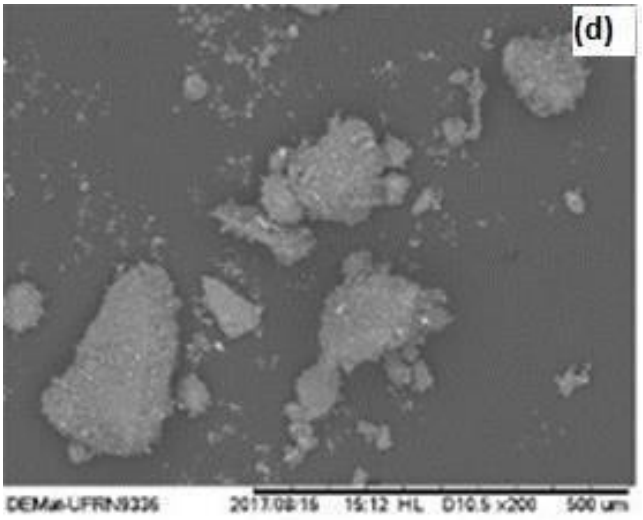

Figure 3d. Micrograph of Al_2O_3-10% Fe powder composite milled for 50 hours.

During the milling time is observed a greater dispersion, homogenizing and impregnating the Al_2O_3 particles in the iron phase, and smaller composite particles. It can also be observed that the agglomerates formed by Al_2O_3 particles.

In fig. 3a is shown micrograph of milling powders for 5 consecutive hours. Through these micrographs, it can be noted that High Energy Miling was able to disperse the Al_2O_3 particles in the iron matrix. The onset of Alumina phase clusters in the iron phase is also noticeable.

In fig. 3b shows the micrograph of milling powers for 15 hours. In this phase, more refined aluminum oxide particles are observed. Also a greater dispersion and impregnation of the Al_2O_3 phase in the iron phase which presents even more deformed particles and the emergence of the fragmented iron phase represented by some bright spots.

In fig. 3c shows the milling powers for 30 hours. Greater dispersion, homogenization, and impregnation of alumina particles in the iron matrix, as well as smaller composite particles, are observed. It can also be seen that the agglomerates are now formed by Al_2O_3 particles and a larger amount of metal phase fragments.

The micrograph of the milling powers for 50 hours are shown in fig. 3d. In this phase, a greater dispersion and more effective homogenization can be observed.

3.2.2. Effect of high energy milling time on the crystalline structure of milled powders.

In Fig. 4, shows the X-ray diffractograms performed on samples collected from Al_2O_3-Fe of 5, 15, 30 and 50hours of the milled powders.

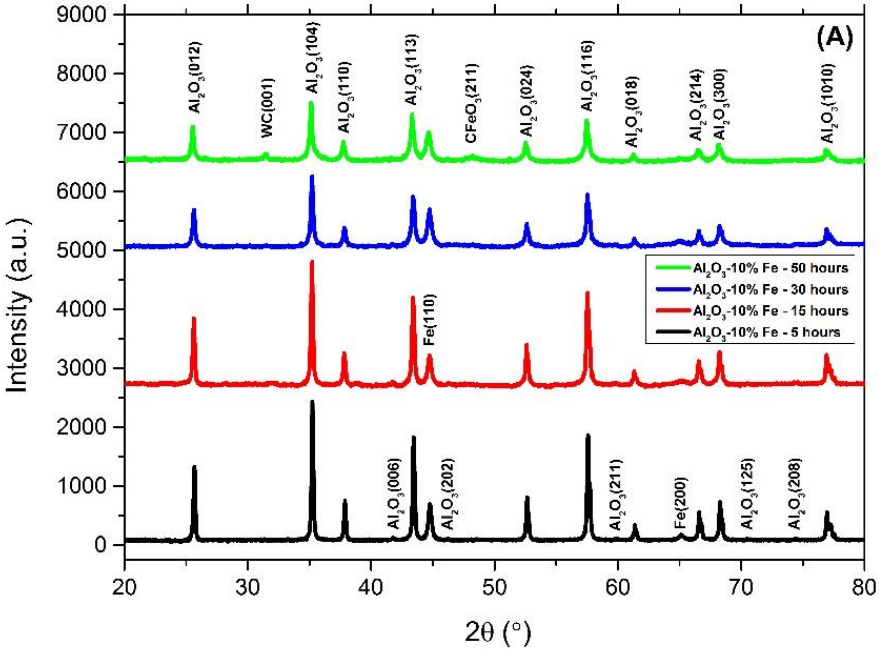

Figure 4. XRD of Al_2O_3-10% Fe powder composite milled for 5, 15, 30 and 50 hours

Through Fig. 4 it was possible to identify the decrease in the intensity, as well as the enlargements of the peaks of alumina and iron over the milling time.

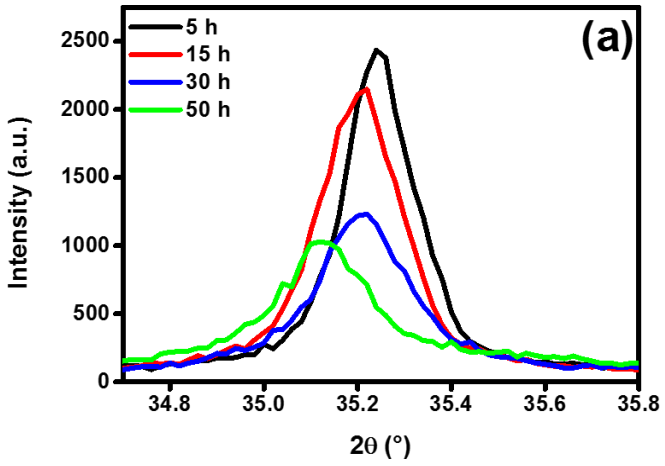

Figura 5a.Ampliação pico principal Al_2O_3

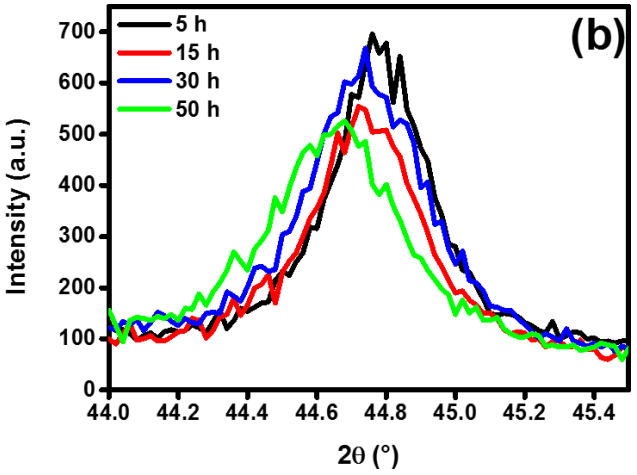

Figura 5b.Ampliação pico principal Fe

Finally, in Fig. 5 (a-b) are shown the two main peaks, Al_2O_3 and Fe, respectively, in order to confirm the above, that in both figures it is possible to notice a reduction in the peak size, and consequently its enlargement. This phenomenon is intrinsically associated with the method used to obtain the Powder: High Energy Milling since the technique has as its strong characteristic the amorphization of the microstructure of the material. This phenomenon was more present in Al_2O_3, due to being more exposed to the effects of milling.

4. CONCLUSION

From the results obtained and discussed in this work, we can make some conclusions:

- It is possible to obtain Al_2O_3-Fe composite particles using High Energy Milling;
- Through high energy milling is possible to obtain a material with a more refined microstructure can be obtained;
- High energy milling can provide a greater dispersion and homogenization of the particles of the composite powders;
- Reduction and enlargements of the peak, furthermore the amorphization of the microstructure of the material.

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