MICROSTRUCTURE STUDY OF ALUMINA CERAMICS FROM POWDER MIXTURES OF NANO AND MICROSIZED PARTICLES

S. T. Fonseca[1]; M. C. A. Nono[1], and C. A. A. Cairo[2]

[1]Laboratório Associado de Sensores e Materiais - LAS, Instituto Nacional de Pesquisas Espaciais - INPE, São José dos Campos 12201-970, Brazil
[2]Divisão de Materiais – AMR, Instituto de Aeronáutica e Espaço – IAE, Centro Técnico Aeroespacial – CTA, São José dos Campos, SP, Brazil
solange@las.inpe.br

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Abstract: In this work, we propose the production of nanosized α-Al_2O_3 powder from precipitation reactions, with intention to reduce the sintering temperature of the alumina ceramics. For this we addition of nanosized powder in the commercial microsized powders. The nano and microsized powder particle mixtures were obtained by precipitation of nanosized aluminum hydroxide in microsized Al_2O_3 in liquid suspension. Several compositions of nano and microsized powders were carried out. These powder compositions were based on data of simulated particle packing. The nano and microsized powder mixtures was characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) and thermogravimetric and differential thermal analysis (TGA and DTG). The powder mixtures were compacted by uniaxial (75MPa) and isostatic (300MPa) pressing, producing cylindrical samples. The powder compacted samples were sintered at 1400°C. Sintered ceramics were characterized by XRD and SEM. The results showed that the higher density of sintered ceramic was obtained for 65% of microsized powder and 35% of nanosized powder mixture. This composition of size of particles of the powder was in agreement with the theoretical simulation.

Introduction

The study of the production and characterization of nanostructure materials has been one of the subjects most attractive of the basic and technological research in the last years, due to possibilities of improvement of the various properties that the nanostructure materials can have in comparison to the materials was obtain for the conventional processes

Alumina is one of the most important ceramics for structural applications, preferentially in the chemically most stable form of alpha (α-Al_2O_3). Decreasing grain sizes have been shown to improve the hardness and wear resistance of dense sintered products as well as their strength if advanced shaping approaches avoid defects as they are usually associated with the increasing tendency of fine-grained raw powders to agglomerate [1,2].

The fabrication of a nanostructured ceramic materials will undoubtedly involves preparing of a nanosized ceramic powder, which may then be consolidated to a high sintered density at a relatively lower sintering temperature. Many of desirable micro structural features may therefore be realized, including the improved sintered density, refined grain size, narrow grain size distribution and minimized degree of structural defects.

In this work are reported and discussed the powder characteristics and sintered microstructures of ceramics prepared from mixtures of nano and microsized powder particles. These powders were obtained from Aluminium hydroxide precipitated in a alumina aqueous suspensions composed by microsized particles.

Experimental

Nanocrystalline $Al(OH)_3$ powder was prepared by chemical precipitation from aluminum chloride using magnetic agitators under conditions controlled of temperature 50ºC and pH > 7.

Nanocrystalline $Al(OH)_3$ powder was washed with ethyl alcohol and dried using a rotative evaporator at vacuum. This process aided the total homogeneity and the break of the formed agglomerates, thus, minimizing the formation of kind of particles. The powder was calcined in an electric furnace for 1 hour at 300ºC and characterized by TGA/DTG, XRD and SEM.

Commercial α-Al_2O_3 powder was used to prepare mixture of 65% coarse powder. The nano and microsized powder particle mixtures were obtained by precipitation of nanosized aluminum hydroxide in microsized Al_2O_3 in liquid suspension, followed of the washing and drying.

The powder was compacted by a uniaxial (75 MPa) and isostatic (300 MPa) pressings, producing cylindrical test bodies.

Finally, they were synthesized and sintered at 1400ºC for 1 hours. Afterwards, the ceramic crystallographic phases were characterized by XRD. The fracture surfaces were observed by SEM.

Results and Discussion

Nanocrystalline $Al(OH)_3$ powder characterized by TGA/DTG was using to determine dehydration temperature and calcination temperature. Fig. 1, indicates that from 100 to 200ºC occurred weight loss proceeding of the evaporation not constitutional water, in the range from 280 to 330 occurs another great weight loss, proceeding of the constitutional water that is responsible for phase transformation of the $Al(OH)_3$ for η-Al_2O_3.

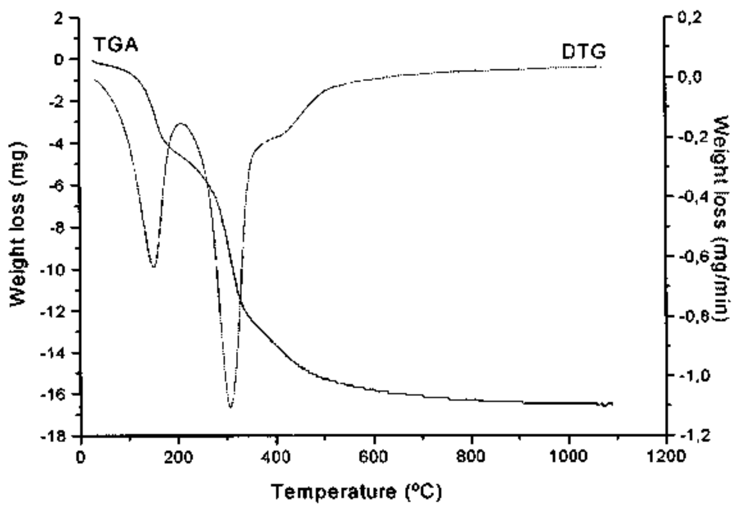

Figure 1 – TGA/DTG analysis for the $Al(OH)_3$ powder

The analysis of XRD shown in Fig. 2, confirms majority presence of $Al(OH)_3$ before of the calcination and after the powder calcination have the η-Al_2O_3 as the major phase content. The powder was calcined in the temperature of 300°C/1hora, in accordance with analysis TGA/DTG (Figure 1), also observed in these diffraction analyses low crystalline of the powder, which confirms the fine particle presence.

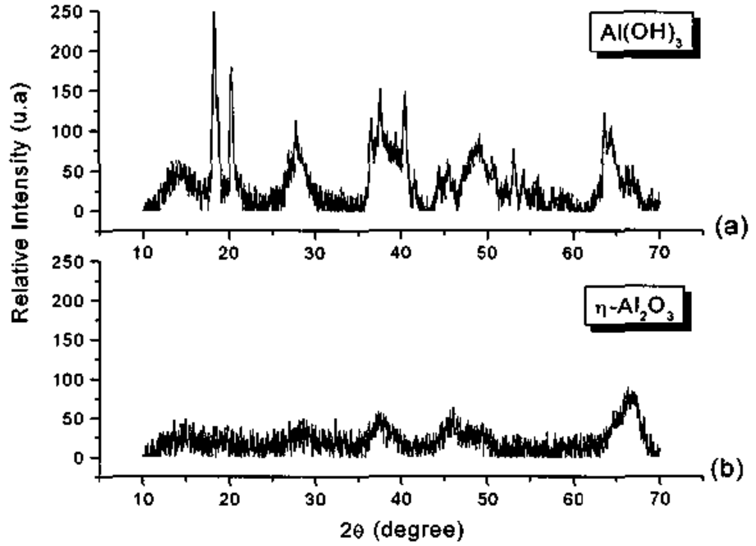

Figure 2 - X-rays diffraction patterns for the $Al(OH)_3$ powder: (a) without calcination and (b) with calcinations.

Fig. 3, show the analyses SEM of the Al(OH)3 powder without calcinations, observe that the gotten powder has a wide distribution of sizes of particles, where the particles biggest have characteristic appearance of porous agglomerates.

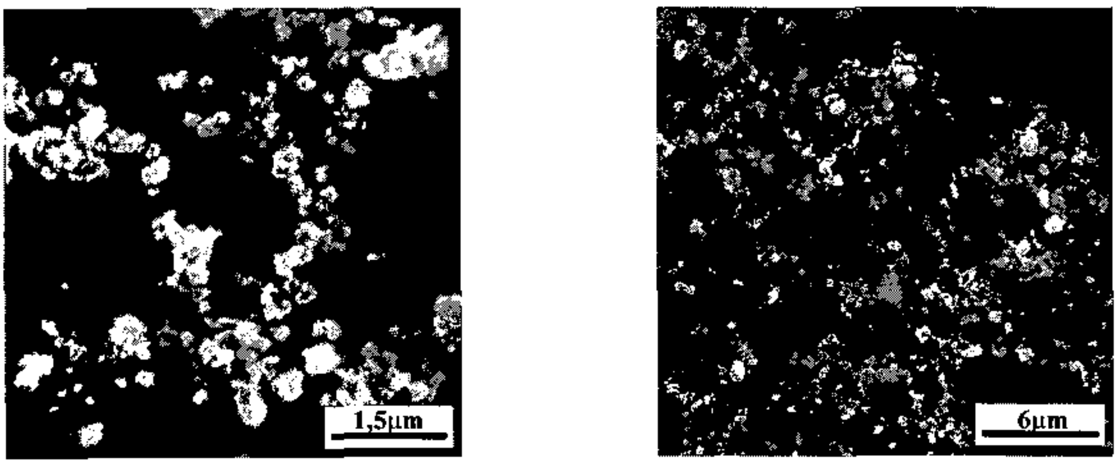

Figure 3 - SEM micrographs of $Al(OH)_3$ without calcinations.

The X-rays diffraction analyses showed that investigated ceramics have the α-Al2O3 as the major phase content (Fig. 4).

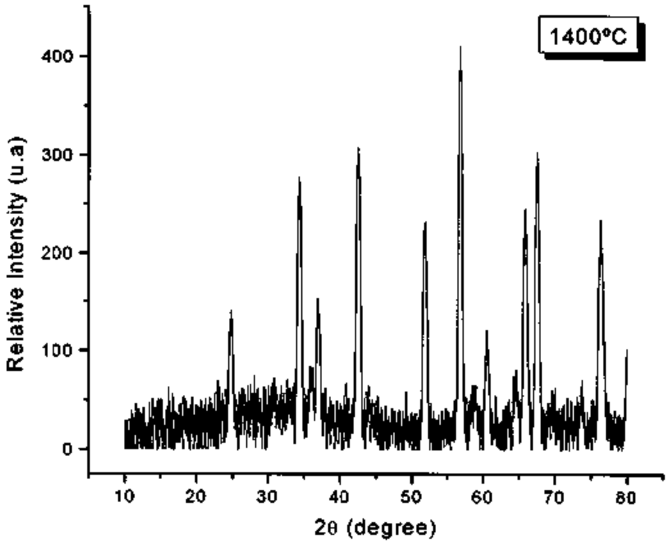

Figure 4 - X-rays diffraction patterns for the α-Al_2O_3 ceramic.

The SEM analyses of the ceramic fracture surfaces showed that ceramic treated in 1400°C presented homogeneous microstructure for the size of particles and few pores, providing a bigger mechanics resistance (Fig. 5).

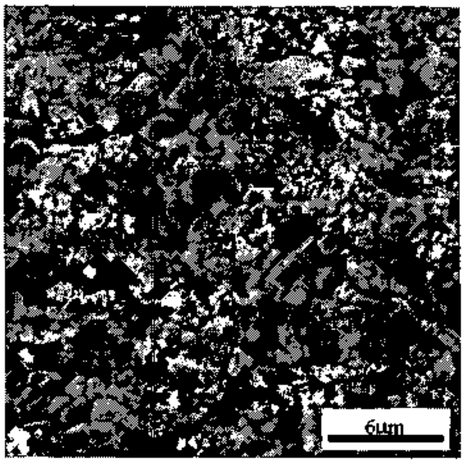

Figure 5 - SEM micrographs for the α-Al_2O_3 ceramics

Conclusion

The production of nanocrystalline powder was gotten by a process fast e low cost, having been necessary the control some parameters essential to guarantee the reproducibly.

The stage of drying of the material was one of the essential parameters of control of the size and density of the agglomerates. This form, the dryings had been carried through in dynamic conditions and assisted by bomb of vacuum for efficient extration of the liquid phase (faster and in lesser temperature).

References

[1] A. Krell, Mater. Sci. Eng. A. 209 (1996), p.156.
[2] A. Krell and P. Blank, J. Eur. Ceram. Soc. 16 (1996), p.763