Densification and mechanical properties of sintered Al_2O_3-$Y_3Al_5O_{12}$ ceramic composite

Densificação e propriedades mecânicas do compósito cerâmico Al_2O_3-$Y_3Al_5O_{12}$ sinterizado

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Keywords

Al_2O_3-YAG composites

Pressing

Sintering

Hardness

Abstract

In this work, Al_2O_3-$Y_3Al_5O_{12}$ ceramic composites were developed with different proportions of Al_2O_3-Y_2O_3, which were mixed and compacted at different pressures of 40MPa to 100MPa, being consequently sintered at 1600°C-2h. The sintered samples were characterized by X-ray diffraction presenting $\alpha-Al_2O_3$ and $Y_3Al_5O_{12}$ as crystalline phases. Samples with relative densities ranging from 78 to 80% and 87 to 91% were obtained depending on the composition and the compaction pressure used. The hardness values obtained were of 1010 to 1080HV and 370-470HV, for mixes Al_2O_3-Y_2O_3 having the composition with levels of 20 and 36.5wt.%, respectively.

Resumo

Neste trabalho, compósitos cerâmicos (Al_2O_3-$Y_3Al_5O_{12}$) foram desenvolvidos com diferentes proporções de Al_2O_3-Y_2O_3. Foram misturados e compactados a diferentes pressões de 40MPa a 100 MPa, sendo, posteriormente, sinterizados a 1600 °C-2h. As amostras sinterizadas foram caracterizadas por difração de raios-X apresentando $\alpha-Al_2O_3$ and $Y_3Al_5O_{12}$ como fases cristalinas. As amostras com as densidades relativas que variam entre 78 a 80% e 87 a 91% foram obtidas em função da composição e da pressão de compactação utilizada. Os valores de dureza obtidos foram de 1010 a 1080HV e 370-470HV, para as misturas de Al_2O_3-Y_2O_3 tendo a composição de 20 e 36.5wt.%, respectivamente.

Palavras-chave

Compósitos Al2O3-YAG

Prensagem

Sinterização

Dureza

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1. Introduction

The biphasic composite Al_2O_3-YAG has several applications in the area of aerospace engineering, as being used as a tool for machining [1-3]. The microstructure resulting two-phase of the sintering composite Al_2O_3 and YAG ($Y_3Al_5O_{12}$) or Y_2O_3, presents the grains of the composite Al_2O_3 and YAG distributed homogeneously, which suffer after sintering and during cooling, compressive residual stresses because of the difference in coefficient thermal expansion between the two phases, increasing the toughness of the material by the difficulty of intergranular cracks propagation.

The advantages of using oxides ceramic in relation to non oxides ceramic, is its high resistance to oxidation and corrosion in aggressive environments and high temperatures. In the 90s some studies have demonstrated that the YAG ($Y_3Al_5O_{12}$-"Yttrium Aluminum Garnet") oxide has higher fluency resistance. The composite Al_2O_3-YAG when heated to the fusing temperature doesn't pass through phase transformations and forms a eutectic fusion allowing direct processing of the same. The mechanical integrity of the material at temperatures around 1.500°C was confirmed in several studies [3-6].

The objective of this study was to analyze the effect of parameters processing of the composite Al_2O_3-$Y_3Al_5O_{12}$ about the porosity of sintered product, and to correlate these values with the hardness of the material.

2. Materials and methods

Alumina, Al_2O_3 (TYPE A-1000 SG-Alcoa Aluminum SA) with purity of 99.83% and yttria, Y_2O_3 (TYPE REO, Alfa-Aesar), with purity of 99.90 % were used in this work. The powders were milled, in 63.65wt.% and 36.35wt.% (Al_2O_3 e Y_2O_3, respectively), denominated *mixture 1* and 80.00wt.% and 20.00wt.% (Al_2O_3 e Y_2O_3, respectively), denominated *mixture 2*. [7]. The mixtures were dried at 120°C-24h and sieved. The powders were compacted applying compression pressures of 40MPa, 60MPa, 80MPa or 100MPa, for 30s. The compacts were sintered at 1600°C-2h with heating and cooling rate of 5°C/min in oven MAITEC F1650.

3. Characterizations

The compacts were characterized by their green relative density, applying the geometric method. In this step, theoretical densities were used for each individual studied mixture, obtained by the rule of mixtures.

Relative density of sintered samples was determined by applying the principle of Archimedes. The sintered samples were characterized by X-ray diffraction, using Shimadzu-XRD6100 diffractometer with CuKα radiation, applying a rate of 5 seconds per point score, angular step of 0.05° and scan of 2θ between 20° e 80°. The crystalline phases found sintered materials, were identified by comparison with the JCPDS database **[8]**.

The microstructural aspect of the sintered samples was evaluated by scanning electron microscopy (SEM/MEV), using SEM-Hitachi - TM3000.

On the microhardness tests, the samples were prepared by sanding and polishing using a polisher Buehler Automatic AUTOMET-250 following the sequence below:

- Use of sandpaper with a diameter of 220 µm and 320 µm, between 30 and 60 minutes, in the sanding process.
- Application of pressure of 120 N at 200 rpm, during polishing process, for 5 min in each polishing with diamond pastes of 9 µm to 1 µm.

4. Mechanical Properties

Vickers microhardness was calculated using the Vickers indentation method. The methodology used to determine the hardness of the samples followed the ASTM C 1327-99 [9]. The surfaces of the cross sections of the sintered samples were subjected to Vickers's measure hardness using microdurometer mod. DHV-1000Z-Time-corp China. Charges of 1000gF were applied during the indentation with measurement time of 15s.

5. Results and discussion

Figure 1 shows the results of relative green density of the composition studied in accordance with the compaction pressure applied to the samples.
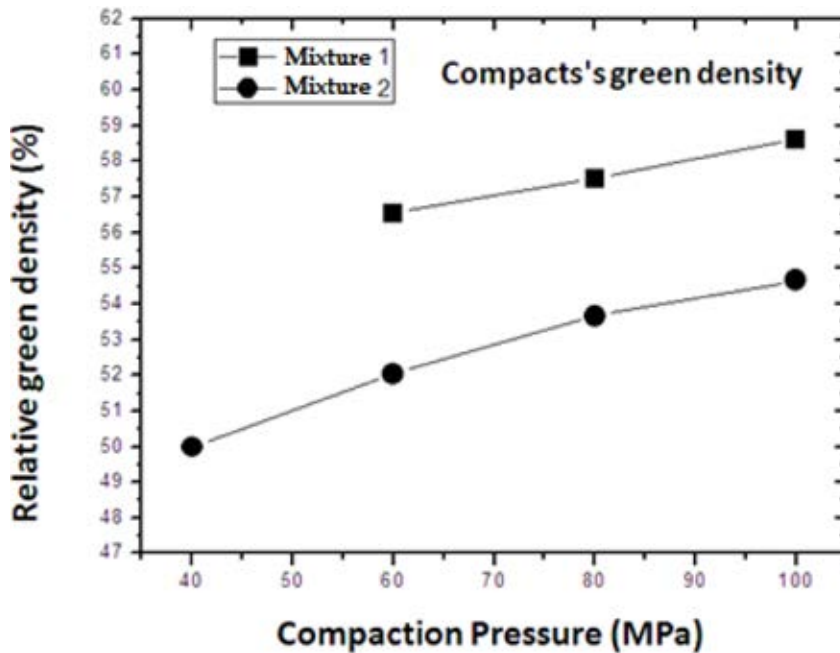
Figure 1 - Relative density of compacts as function of compaction pressure.

It is noted that mixture 1 having a greater amount of Y_2O_3 (36.35 wt.%), allowed the material to reach greater degree of compaction, possibly due to the variety of particle sizes from this material when compared with mixture 2, composed of 80% Al_2O_3 and 20% Y_2O_3.

The relative densities' values between 50 and 54% were obtained from "mix 2", while values between 56,5 and 58% were obtained from mixture 1.

Figure 2 shows X-ray diffraction of the sintered samples representing the different compositions studied in this work.
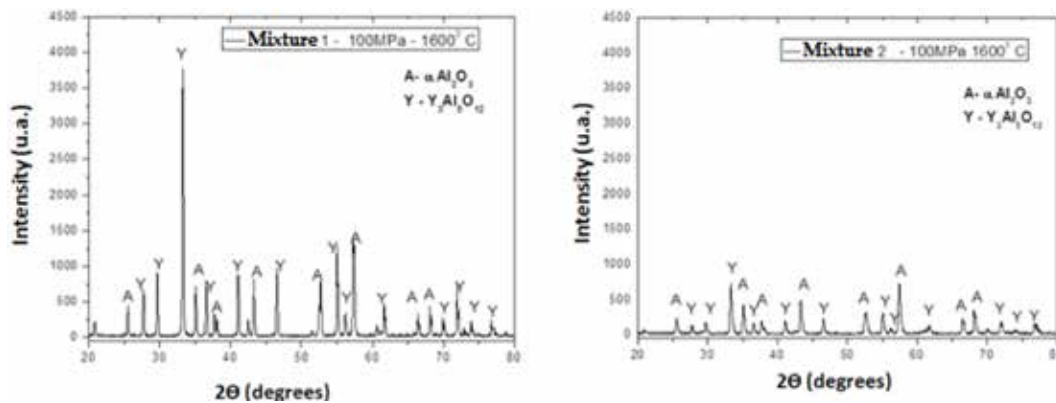

Figure 2 – X-ray diffractograms of the samples sintered at 1600°C-2h.

Analyzing the diffractograms presented in Figure 2, we can observe the presence of phases $\alpha-Al_2O_3$ and $Y_3Al_5O_{12}$ in both compositions. The fact that the phase Y_2O_3, present in the initial powder mixture, cannot be observed in the diffractograms of the sintered samples, indicates that this material is converted during the sintering phase YAG, $Y_3Al_5O_{12}$, reacting stoichiometrically with Al_2O_3 solid part available mixture composition. The observation of the peaks of higher intensity in the phase YAG of the mixture 1 is consistent with the fact that there is this composition, a greater amount of Y_2O_3 in the range of 36.35% by weight

Figure 3 shows the results of the relative density of the sintered samples as function of composition studied and compacting pressure applied during compacting. The samples were sintered at 1600°C-2h.
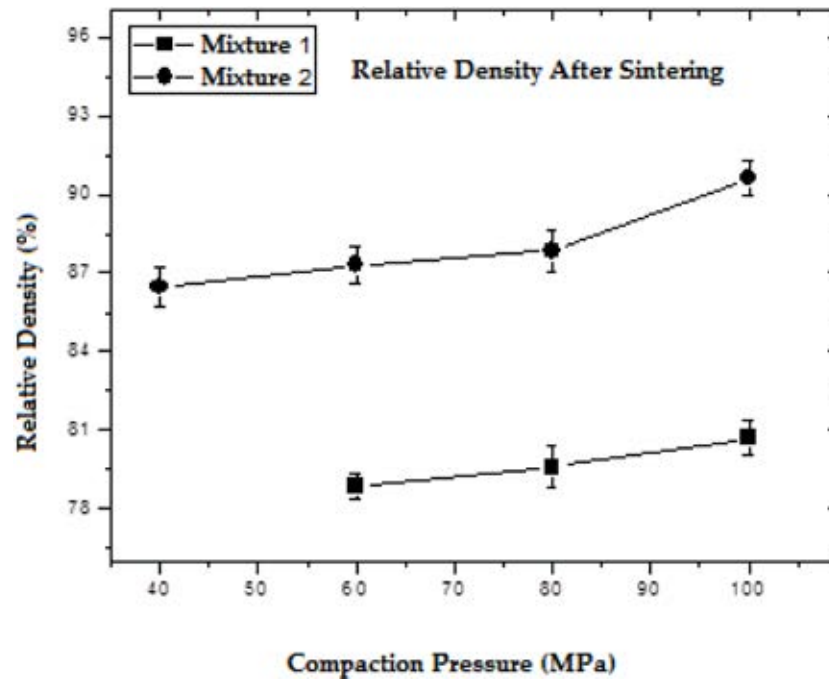
Figure 3 – Density of the sintered samples according to the composition studied.

We can observe that the sintered samples based on mixture 2, present relative density considerably higher than the samples sintered from mixture 1. Taking into consideration the results presented on figure 1, which shows higher green density from mixture 1 to mixture2, an easier sintering was expected from this mixture. However, the observed results were the opposite. Moreover, in spite of having a higher density in mixture 1, favoring the densification during the sintering, it was harder for it to densify (eliminate pores). This has, possibly, happened due to the high level of Y_2O_3 present in the mixture. Therefore, the increase in the amount of Y_2O_3 requires from the mixture more time and temperature needed for the reaction $Y_2O_3 + Al_2O_3 \rightarrow Y_3Al_5O_{12}$ to

happen in solid estate. Thus, the densification phenomenon is harmed, since more time is required for the phase transformation to happen completely, competing with the role of densification mechanisms in the sintered material.

Figure 4 shows micrographs obtained through MEV, from the fracture surface of the sintered samples. Results show a homogenous dispersion of YAG in the Al_2O_3 matrix. Consisting with the results shown in figure 3, the samples with higher level of Y_2O_3, have shown higher levels of YAG and a more pronounced porosity. Comparatively, the mixture with 20% of Y_2O_3 have shown YAG grains more scattered and the microstructure, generally speaking, has a smaller and well distributed porosity.
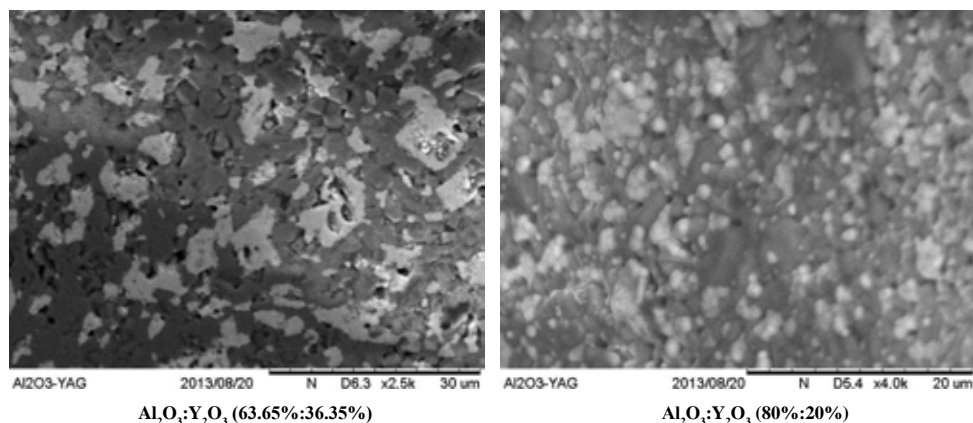

$Al_2O_3:Y_2O_3$ (63.65%:36.35%)

$Al_2O_3:Y_2O_3$ (80%:20%)

Figure 4 - Micrographs obtained through MEV, from samples Al_2O_3-YAG sintered at1600ºC-120min.

6. Mechanical Properties

Table 1 and Figure 5 shows the Vickers hardness results regarding the pressure compaction and composition studied.

Table 1 – Hardness results of sintered materials

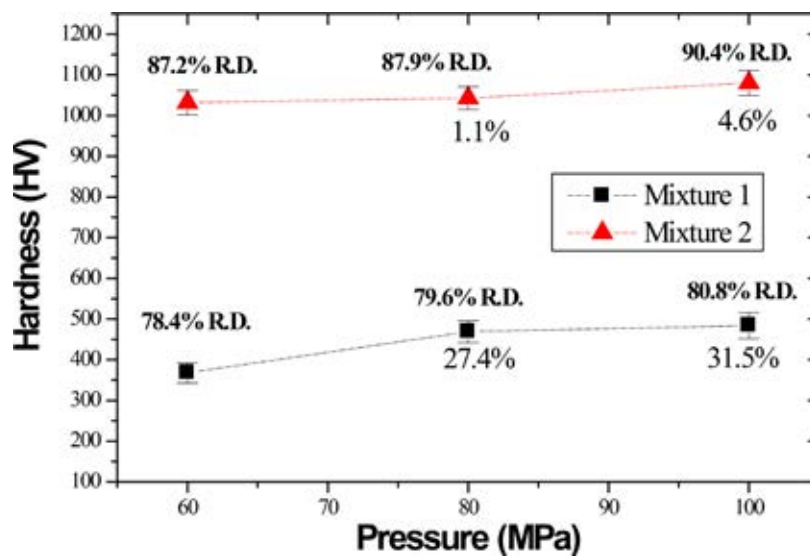| Mixture	Compacting Pressure (MPa)	Vickers Hardness (HV)
Mixture 1	60MPa	368 ± 25
	80MPa	469 ± 27
	100MPa	484 ± 32
Mixture 2	60MPa	1032 ± 30
	80MPa	1043 ± 28
	100MPa	1080 ± 31

Figure 5 – Influence of pressure compaction in hardness of sintered samples.

The hardness obtained by the Vickers indention technique was directly influenced by the composition and respectively pressure (consequently the porosity) of the materials. Generally speaking, mixture 1 has shown hardness which varies from 370 to 470HV for mixture sintered at 1600°C-120min. Hardness in mixture 2 varies from 1010 to 1080HV. The results indicates that the increasing of compaction pressure of 60 to 100MPa, leads to increasing of 31.5% and 4.6% on hardness of the sintered samples for mixture 1 and mixture 2, respectively.

7. Conclusions

It is possible to identify that the increase in Y_2O_3 level in mixtures Al_2O_3-Y_2O_3 makes densification hard due to difficulty in transformation in the solid state required to obtain $Y_3Al_5O_{12}$. Therefore, mixtures of different compositions, with 20 e 36,5% Y_2O_3 weight levels, sintered to 1600°C-120min, allow samples with relative density of 87-91% and 78 to 80%, respectively. In such conditions, the hardness numbers obtained were 1010 to 1080HV and 370 to 470HV, respectively.

8. Acknowledgment

9.   Referências Bibliográficas

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