

MECHANICAL PROPERTIES OF ZIRCONIA TOUGHENED ALUMINA COMPOSITES WITH TZP PARTIALLY NANOSTRUCTURED SINTERED BY LIQUID FASE.

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Abstract. A comparative study between alumina added niobia ceramics and two alumina zirconia composites from nanostructured TZP (7% and 14% weight) was made. On this composites the zirconia were yttria stabilized and the alumina were submicron structured. As sintering aid a mixture of magnesia, niobia and talc were used on all samples. The sintering was performed at 1450 °C during 60 minutes. The characteristic grain size and shape of an alumina and zirconia powders, aggregates and agglomerates were characterized. The sintering ceramics were evaluated through hardness, fracture toughness and 4 point bending test. Weibull statistic was applied on the flexural results. Although the fracture toughness result from ZTA were lower, and seems to be affected by the liquid fase, the hardness and Weibull modulus were higher than alumina niobia. The grains size and the homogeneity of its distributions on the microstructure of this ceramics was correlated to these higher values. The results from these alumina zirconia composites showed a potential to apply as a ballistic armor material.

Introduction

The ceramic materials have been demonstrating a great potential application in ballistic armour, because it allows to associate high hardness with low specific mass. However, for this kind of application, is necessary to the obtainment ceramics with high bending strenght and fracture toughness.

Alumina has been known as a study object for this kind of application due to some of their properties, mostly by the high value of your hardness. As your fracture toughness is low, the ceramic composites with alumina matrix reinforced with other ceramic materials have been much studied.

Zirconia is a material able to increase the fracture toughness of alumina, due to two reinforcement mechanisms: a) transformation induced by tension; b) microcracking^[1-7].

The sintering aid have a beneficial influence in sintering kinetics of alumina, because they are able to inhibit the grains growth and promoting a larger material densification.

Experimental Procedure

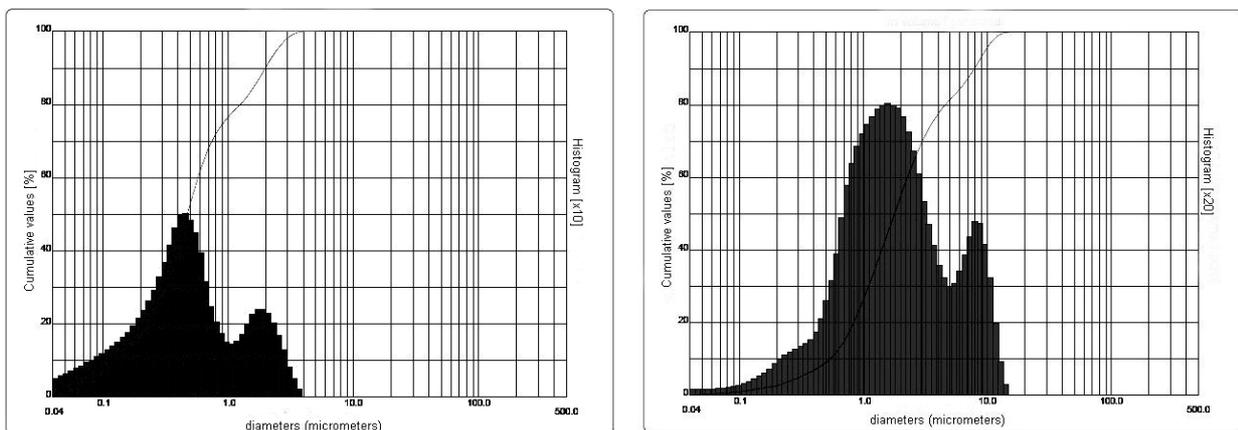
The used raw materials in this work were alumina A1000SG of Alcoa Chemicals, with average size of particles of 0,45 μm and the nanostructured tetragonal zirconia polycrystalline (TZP) stabilized with yttria, of Shandong Zhongshun Sci. & Tech. Dev. CO. Ltd, with average size of particles between 20 and 30 nm, characterized by an equipment of the mark CILAS. Sintering aid used were niobia (Nb_2O_5) of CBMM – Companhia Brasileira de Mineração e Metalurgia, the magnesium oxide (MgO) of Merck Chemical Products and the talc ($3\text{MgO}\cdot 4\text{SiO}_2\cdot \text{H}_2\text{O}$). Ceramics of alumina - niobia was produced adding to alumina 5,55 % in weight of the additives mixture and 0,15 % in weight of CMC. Alumina – zirconia composites (ZTA) were produced adding to alumina 7 or 14 % in weight of zirconia, 5,55 % in weight of the mixture of the sintering aid and 0,15 % in weight of CMC.

The uniaxial pressing was accomplished with pressure of 40 MPa and the isostatic pressing with pressure of 300 MPa. The sintering was accomplished in 1450 $^\circ\text{C}$ for one hour, using heating rate of 5 $^\circ\text{C}/\text{min}$.

For the characterization of alumina - niobia and the alumina – zirconia composites, they were accomplished analyses for X-ray diffraction, scanning electron microscopy, specific mass, hardness, fracture toughness for Vickers penetration and 4 point bending test, where was applied Weibull's Statistics.

Results and Discussion

The Fig. 1 shows the results of the particles size distribution analysis of alumina and nanostructured TZP stabilized with yttria. It can note that the zirconia dust introduced high values of average diameters, what it indicates the presence of aggregate or agglomerates, which it could be proved with the powder's analysis of ZTA for scanning electron microscopy, shown in Fig. 2. The presence of aggregate complicates the packing of the particles of the dust during compaction stage and consequently in densification during the sintering.



a b
Fig. 1 – Particle size distribution of the: a) alumina; b) TZP

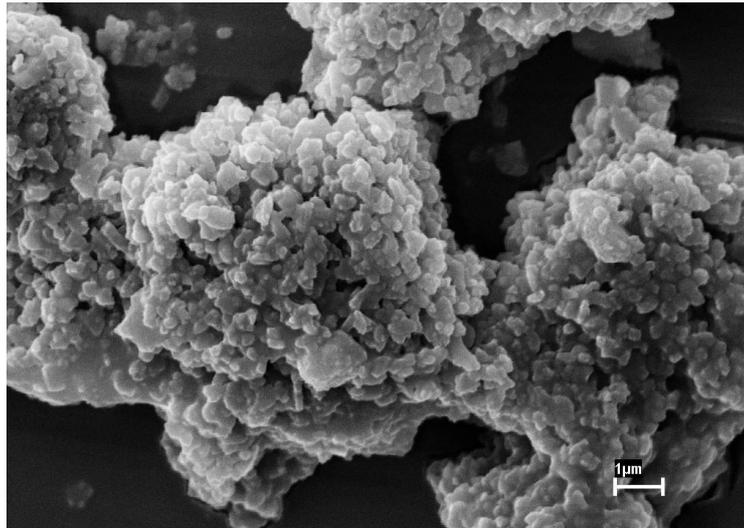


Fig. 2 –Scanning electron microscopy of the powder of ZTA (7 % in weight), shown aggregates and agglomerates.

It could observe, by the X-ray diffraction analyses, which ZTA composites introduced the formation of some composed, among them MgAl_2O_4 , what it indicates the decrease of the solubility limit of MgO in the net of alumina^[8] and the reaction of these two composed are thermodynamically favorable in high temperatures because of the high free energy of formation of this composite^[9]. Also the formation of other zirconium phases was observed, besides tetragonal, like for monoclinic, and that it could have occurred the formation of cubic and orthorhombic phase, besides a chemical composite with for yttria ($\text{Zr}_{0,82}\text{Y}_{0,18}\text{O}_{1,91}$). The orthorhombic phase, according to the literature^[10], only occurs when the system is in high pressure conditions, and it occurs by the fast transformation, of martensitic characteristic, of the monoclinic phase^[11]. In the case of the cubic phase, if after formation for MgAl_2O_4 still occurs a MgO's excess who does not react with for alumina, this excess can if react in the crystalline net of the zirconia and will stabilize in the cubic form^[9].

In the Table 1, it can observe the values of relative specific mass, linear retraction and porosity and of the mechanical properties of alumina – niobia ceramics and of the composites of ZTA. Alumina – niobia introduced a high value of relative specific mass, low porosity and high degree of linear retraction. Already ZTA's composites introduced a smaller linear retraction than alumina – niobia. Some factors as the solubility decrease of MgO in the net of alumina and as well, the presence of small quantities of niobia up to 5 % in weight in zirconia's ceramics, contribute for a continuous decrease in the specific mass of the material^[12].

It can observe an increase of about 6,5 % in the hardness of the composite with addition of 14 % in weight of zirconia regarding alumina – niobia, showing the influence of the smaller porosity and larger densification of this composite in relation to the others ceramics. However there was no increase of the fracture toughness, due to formation of aggregate that result in regions with different densifications from the matrix during the sintering, generating cracks that degrade the mechanical resistance of the material and eliminate the effect of the toughness, emphasizing the need to a homogeneous distribution of the second phase (zirconia) in the matrix (alumina)^[13].

Table 1 – Properties of the alumina – niobia and the ZTA composites

	ρ_R	P (%)	RL [%]	H _v [MPa]	D _{Hv} [MPa]	K _{1C} [MPa.m ^{1/2}]	σ [MPa]	D _{σ} [MPa]	$\sigma_{50\%}$ [MPa]	m
Al ₂ O ₃ –Nb ₂ O ₅	95,57	0,24	18,14	13,7	± 0,25	4,34	197	± 33,62	197	8
Al ₂ O ₃ + 7 % TZP	91,26	0,33	16,92	13,5	± 0,62	2,29	185	± 28,91	188	9
Al ₂ O ₃ + 14 % TZP	92,55	0,23	16,89	14,6	± 0,52	2,50	186	± 20,11	188	13

(ρ_R) relative specific mass, (P) porosity, (RL) linear retraction, (H_v) Vickers microhardness, (K_{1C}) fracture toughness, (σ) rupture tension, (D) standard deviations, ($\sigma_{50\%}$) rupture 50% e (m) Weibull's modulus.

The transformations of crystalline phases (for the cubic and orthorhombic structures) occurred in the zirconia during the sintering and the cooling, could have influenced the fracture toughness values of this ceramics, because they do not allow the performance of the increase same mechanisms of the fracture toughness related to tetragonal phase^[14]. It could have occurred due to one not stabilization of the crystalline structure tetragonal of the zirconia in this ceramics. Some researches^[12,15] have been demonstrated that for niobia has been playing an unstabilizer role of the Y-TZP tetragonal structure, eliminating the oxygen vacancies that are important for the zirconia stability in this structure. The rupture tension obtained by ZTA is comparable to the rupture tension obtained by alumina - niobia, considering the standard deviations and an increase of the Weibull's modulus, demonstrating that they are more reliable material for structural applications.

In the Fig. 3 can observe the different grains size and porosities in ZTA composites. A decrease is observed in the grain size in the structure with the increase of the quantity of Y-TZP added. These results show that the zirconia acted as an inhibitor of the grains growth in the ceramic composite, but that did not result in a considerable increase in the mechanical properties, mostly when compared to alumina – niobia.

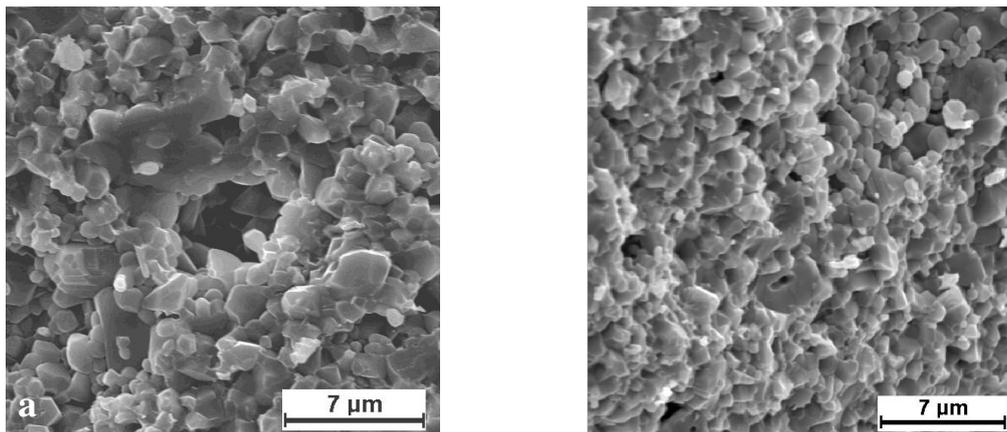


Fig. 3 – Scanning electron microscopy of the fracture surfaces of the ZTA samples sintering in 1450°C, showing the different grains size and porosities; a) 7 % in weight and b) 14 % in weight.

Conclusions

The ceramic composite of alumina – zirconia with 14 % in weight, introduced hardness values and Weibull's modulus higher than the values of alumina-niobia, with the same flexural strength level, authorizing this material to the ballistic rehearsal, even with the low fracture toughness found by microhardness technique. However there is the need to optimization of the processing of the powders, to avoid the formation of aggregate, mostly when are used nanometric powders. The sintering aid used as formers of vitreous phase in this work (niobia, magnesia and talc), were not effective in the densification promotion of the bodies, because the zirconia addition as second phase limited the solubility of these composed in alumina and formed chemical composite with the zirconia, reducing the quantity of vitreous phase for the sintering with consequent decrease in the specific mass; besides niobia act in alumina composite - doped zirconia with yttria (Y-TZP) like unstabilizer of the tetragonal zirconia's crystalline structure, transforming part of this in monoclinic in the cooling. For this striking influence of the vitreous phase and of niobia in the present zirconia in these ceramic composites, it was not possible to relate the effect of the grains size to the mechanical properties of this material.

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