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The influence of nanoparticle agglomerates and aggregates on mechanical properties of Ce-TZP sintered ceramics

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Abstract – Ceramic powder compaction by pressing requires weak-agglomerated powders to obtain high dense compacts and consequently dense sintered ceramic bodies. In this work powders with different agglomerated and/or aggregated size distributions were compacted by uniaxial and isostatic pressing, and sintered. The results showed that all aggregated and weak agglomerated powders were composed of nanoparticles. This study indicated that the nanoparticle agglomerates were destroyed during the pressing stage leading to an increase of the packing degree of the powder particles andthe mechanical parameters of sintered ceramic.

In the majority of techniques developed for the production of ceramic nanoparticles the focus has been to reduce the primary particle size to below 30 nm. However, the problems related to agglomeration and aggregation to obtain dense sintered ceramics have not been adequately addressed [1-3]. Chemical methods are frequently used to obtain nanoparticulated and homogeneous powders. Ceramic nanoparticles prepared by chemical methods have advantages over physical methods in some respects such as lower cost, higher productivity level and easier to store. One of the most critical steps in these methods is the extraction of the liquid phase (generally water) from the solid precipitated, or drying of the precipitate [3]. The way adopted to dry the precipitates influences the amount of agglomerates and/or aggregates in the powder[1-2].

In this investigation, compaction of several powder mixtures dried by different methods and consequently with different amount of aggregates and/or agglomerates was studied. The selection of the powder mixtures of zirconia-ceria for this study was based on the availability of several powders with different morphological characteristics. Different agglomerated powders were obtained from zirconium and cerium hydroxides coprecipitated and: i) conventional dried at 110^{o}C (ZCH-S), ii) freeze dried (ZCH-L), and iii) by extraction of water with isopropyl alcohol (ZCAP). After the dry step the powders were calcinated at 400 ^{o}C in order to obtain the oxide mixtures. These powders were characterized by X-rays diffraction, Hg and N_2 porosimetry and SEM. Green bodies were uniaxial (at 40 MPa) and isostatic (at 300 MPa) pressed. In order to investigate the compaction behavior of the different powders Hg and N_2 porosimetry were used. The green compact bodies sintered at 1400 ^{o}C and characterized by SEM. It was adopted the bending strength tests and fracture toughness technique to the determination of the mechanical parameters.

ZCH-S and ZCAP powders showed a high reduction on their pore volume after compaction step (Fig 1). However, in both samples there were small volumes of pore sizes between 0.2 to 4 μm which was related to packing flaws. Calcinated powder mixture from water substitution using isopropyl alcohol (ZCAP) presented the best compaction degree. In this case the majory of pores were found between 3 to 11 nm and the smallest volume of pore. The relative density obtained for the sintered ceramics showed the best densification for ZCAP ceramics (98.7 %) (Table 1). The results showed that the agglomerates and aggregates in the powders studied were composed of nanoparticles (average particle size < 50nm). Nanoparticle agglomerates were destroyed during the pressing stage leading to an increase of the packing degree of the powder particles. This study indicated that the combination of the porosimetry techniques and SEM supplies important information about the nanoparticle aggregation/agglomeration state and the accommodation capacity of these particles during the compaction step (powder compaction ability). These results were coherent with the mechanical parameters of these ceramics (Table 1).

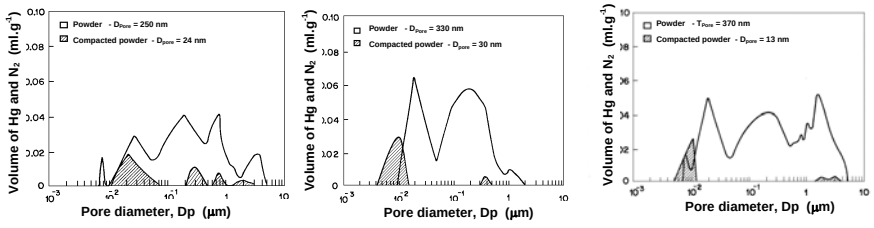

Figure 1: Pore size distribution for the calcinated powder mixtures.

CERAMIC	ZCH-S	ZCH-S	ZCAP
Relative density (%)	93,3	96.5	98.7
Bending strength (MPa)	430±36	584±18	728±11
Fracture toughness (MPa.m$^{1/2}$)	5.3	12.6	19.3

Table 1: Mechanical parameters for sintered 14Ce-TZP ceramics.

Reference

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