Sintering of Mechanically Alloyed YSZ Nanocrystalline Powders

M. A. López de la Torre[1, a], O. J. Durá[1, b], M. Hernández[1], M. García-Cordobés[1], G. Herranz[2, c], C. Sánchez-Bautista[2, d] and G. P. Rodríguez[2, e]

[1]Departamento de Física Aplicada, Escuela Técnica Superior de Ingenieros Industriales, Universidad de Castilla-La Mancha, 13071-Ciudad Real, Spain
[2]Área de Ciencia de Materiales, Escuela Técnica Superior de Ingenieros Industriales, Universidad de Castilla-La Mancha, 13071-Ciudad Real, Spain
[a]Marcoantonio.lopez@uclm.es, [b]oscar.juan@uclm.es, [c]gemma.herranz@uclm.es, [d]Carlos.SBautista@uclm.es, [e]Gloria.Rodriguez@uclm.es.

Abstract

We report on the mechanical and structural properties of nanocrystalline 8% and 10% mol yttria stabilized zirconia (YSZ) obtained using mechanical alloying (MA). The as-milled powders show a body-centered cubic structure with grain sizes in the nanometer scale. After uniaxial pressing and sintering the compacts exhibit good mechanical properties. We discuss the correlation of these enhanced properties with the microstructural changes induced by heat treatment.

Keywords : Nanostructured materials, Mechanical alloying, Sintering, YSZ

1. Introduction

The mechanical properties of nanocrystalline materials have been the subject of a big deal of research effort. The possibility of obtaining considerable amounts of nanocrystalline powders makes mechanical alloying and milling a candidate method for technological uses [1]. However, the compaction of nanocrystalline powders is usually a complex issue. These materials are reported to be difficult to process using the desirable conditions to preserve their microstructure [2, 3].

The application of these materials in different fields (fuel cells, gas sensors), makes nanocrystalline ceramics based on YSZ an interest research subject.

2. Experimental and Results

Nanocrystalline powders of YSZ were obtained using mechanical alloying of ZrO_2 (99.7 % purity, particle size ≤ 40 μm) and Y_2O_3 (99.9 % purity, particle size ≤ 1 μm) from Alfa Aesar. The process was carried out in Retsch PM400 planetary ball mill, equipped with ZrO_2 pots and balls to avoid contamination of sample. A ball/powder ratio was 7:1. Small amounts of powder were extracted at milling times of 2, 4, 8, 16, 32, and 48 hours, with the aim of monitoring the alloyin process using x-ray diffraction experiments. Powder obtained after 48 h were preset into pellets (10 mm diameter, 1 mm thickness) with an uniaxial pressure of 1 GPa. The compacts were sintered in air at temperatures between 750º C and 1220º C. We used the Archimedes method to obtain the density of pellets. Finally, microhardness was measured on polished surfaces with a Future Tech FM-7 microdurometer using a 0.5 kg load.

For milling times of 32 and 48 hours, x-ray diffraction patterns indicate that a stationary milling state has been reached. For those milling times, only five peaks are detected, from which we calculated the lattice parameter and grain size. The results are show in Table I. The grain size was calculated using two methods, Scherrer (using the average results of three principal peaks (111), (220), and (311)), and Integral Breadth method. Although the Integral Breadth method doesn't include the axial divergence at small angles, we can use it because our patterns only include data above 2θ=30º. A Si sample was used for calculation of instrumental broadening. Both methods yield grain sizes in order of 10 nm

Density measurements for 8% and 10% YSZ compacts after sintering at temperatures near to 1220º C, are higher than 93 % of the theoretical density ($\rho_{th} = 6.09\ g/cm^3$ and $6.10\ g/cm^3$ for 10% and 8% YSZ respectively).

Table 1. Lattice parameter (a), grain size (d) and microhardness (ε)

Sample	a [nm]	d [nm] (Scherrer)	d[nm] (I.B.)	ε (I.B.)
YSZ (8%)	0.512	8	15	0.0088
YSZ(10%)	0.515	8	13	0.0107

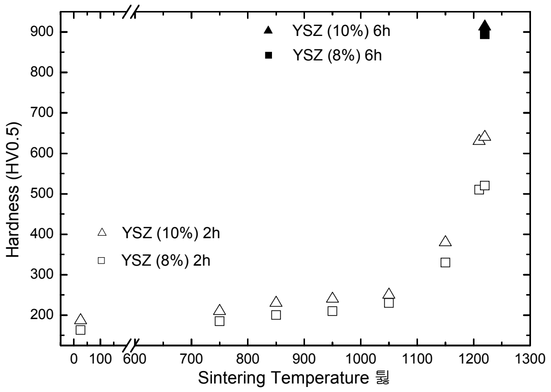

Fig. 1. Vickers hardness as a function of sintering temperature, for 2 h (open symbols) and 6 h (closed symbols) sinterin times.

Finally, in Fig. 1 we present the results of Vickers hardness measurements (HV0.5), as a function of sintering temperature.

These results are in good agreement with those of density measurements and indicate a significant dependence of both properties with the sintering time.

3. Summary

Mechanical Alloying is an efficient method to perform a complete alloying of YSZ in concentrations upper 8%. For a milling time of 32 h, we have obtained single phase powders of 8% and 10% mol YSZ with lattice parameters in agreement with those reported for sintered polycrystalline samples.

The grain size of the as-milled powder has been determined to be around 10 nm. Cold compaction at 1 GPa followed by sintering at 1220º C allow us to obtain high density compacts ($\rho \geq 93\%$ of the theoretical density) with good mechanical properties (913 HV0.5 for 6 h sintering).

4. References

1. C. Suryanarayana, Progress in Material Sciences 46, 1 (2001).
2. Ya-Li li, Yong Liang, fen Zheng, Xian-Feng Ma and Suo-Jing Cui, Journal of Materials Research 15, 988 (2000).
3. Z. Livne, A. Munitz, J.C. Rawers and R.J. Fields, Nanostructured Materials 10, 503 (1998).