

**MICROSTRUCTURAL ANALYSIS OF THORIA-YTTRIA CERAMIC POWDERS AND SINTERED PELLETS\***

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ThO<sub>2</sub>: 9 mol% Y<sub>2</sub>O<sub>3</sub> reactive ceramic powders have been prepared by a chemical route<sup>1</sup> to obtain dense ceramic pieces to be used as electrochemical transducers in gas sensing devices. Nb<sub>2</sub>O<sub>5</sub> addition has been tried to enhance densification of sintered pellets through the liquid phase sintering mechanism. The ceramic powders have been analyzed by X-ray diffraction for phase content and lattice parameters determination. Only the cubic fluorite phase has been detected. TEM analysis has been performed to study powder morphology and determine average particle size. The result is shown in Fig. 1 (top): the powders are agglomerated and have approximately 30 nm average particle size. ThO<sub>2</sub>: 9 mol% Y<sub>2</sub>O<sub>3</sub> pellets have been prepared by pressing at 147 MPa and sintering at 1550 °C during 2 h. Some specimens have also been prepared by adding 0.25 mol% Nb<sub>2</sub>O<sub>5</sub> to the thoria-yttria powder prior to sintering. Apparent densities have been determined by the water immersion method. X-ray diffraction analysis has been done to ascertain solid solution formation. SEM analysis has been carried out in samples polished down to 1 μm and thermally (1500 °C) and chemically etched. Lattice parameters have been determined and correlated to Vegard's law, an evidence of solid solution formation through Y<sup>3+</sup> substitution for Th<sup>4+</sup>. The micrographs in Figs. 1 (middle) and 1 (bottom) show surfaces of ThO<sub>2</sub>: 9 mol% Y<sub>2</sub>O<sub>3</sub> ceramic pellets with and without Nb<sub>2</sub>O<sub>5</sub> addition, respectively. Their microstructure is found to be composed of circular-shaped cross sections of grains with 0.2 μm average grain size. Moreover, Nb<sub>2</sub>O<sub>5</sub> addition prior to sintering leads to an increase in the porosity. TEM and SEM analysis have then been used as suitable tools to study ceramic powder and pellet morphologies to further understanding of the impedance spectroscopy electrical behavior of ThO<sub>2</sub>:9 mol% Y<sub>2</sub>O<sub>3</sub> solid electrolytes<sup>2,3</sup>.

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## References

1. M. Pechini, "Method of Preparing Lead and Alkaline Earth Titanates and Coating Method Using the Same to Form a Capacitor", U. S. Patent n° 3.330.697, 1967.
2. I. C. Cosentino, Dr. Thesis (in portuguese), IPEN - USP (1997).
3. I. C. Cosentino, R. Muccillo, *Mat. Letters* (1997) accepted for publication.

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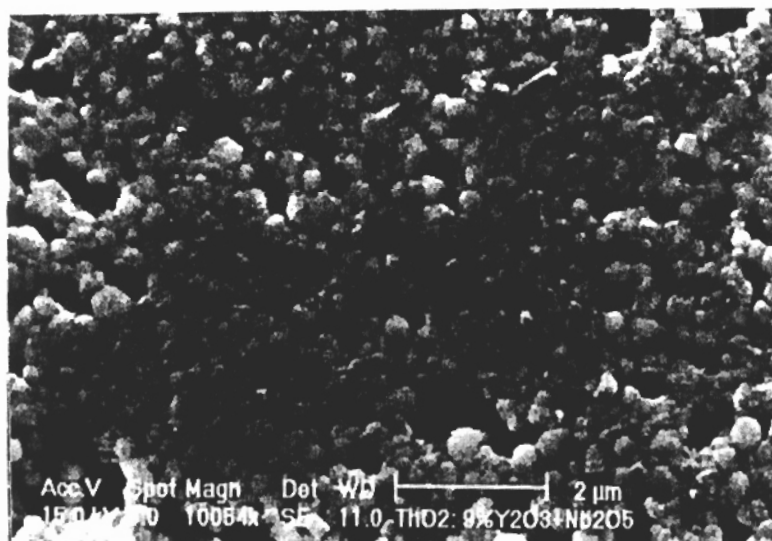
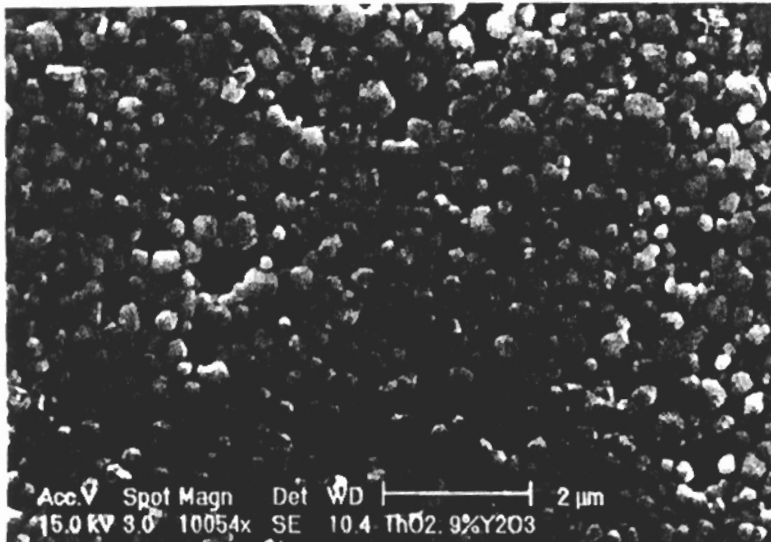
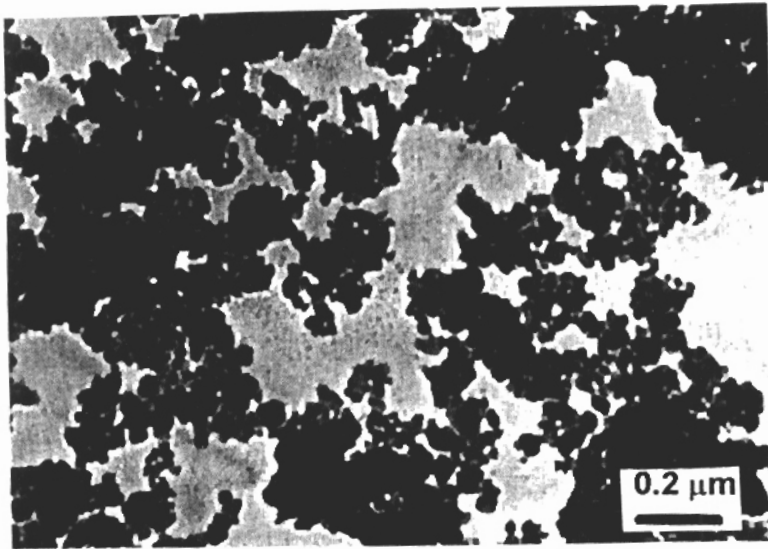


Fig. 1: TEM micrograph of  $\text{ThO}_2$ : 9 mol%  $\text{Y}_2\text{O}_3$  ceramic powder obtained by the citrate technique (top); SEM micrographs of polished and etched surfaces of sintered  $\text{ThO}_2$ :9 mol%  $\text{Y}_2\text{O}_3$  (middle) and  $\text{ThO}_2$ :9 mol%  $\text{Y}_2\text{O}_3$ + 0.25 mol%  $\text{Nb}_2\text{O}_5$  (bottom) pellets.