

**Sintering Studies of Zirconia-yttria Solid Electrolytes
by Scanning Electron Microcopy and Impedance
Spectroscopy Analysis**

R. Muccillo¹, D. Z. Florio², E. N. S. Muccillo¹

**Centro Multidisciplinar de Desenvolvimento de
Materiais Cerâmicos - CMDMC**

**¹Centro de Ciência e Tecnologia de Materiais - CCTM
Instituto de Pesquisas Energéticas e Nucleares - IPEN
C.P. 11049, Pinheiros, S. Paulo, SP, Brazil 05422-970**

**²LIEC - Instituto de Química
Universidade Estadual Paulista
R. Francisco Degni s/n, C.P. 455
Araraquara, SP, Brazil 14807455**

A set of cold-pressed $\text{ZrO}_2:8\text{mol}\% \text{Y}_2\text{O}_3$ (Yttria Fully-Stabilized Zirconia, Y-FSZ) specimens, using sub-micron sized powders, was pre-sintered to 70% of the full density, followed by sintering at different times at low temperatures (two-step sintering). The sintering temperature was chosen after dilatometric measurements of $\text{ZrO}_2:8\text{mol}\% \text{Y}_2\text{O}_3$ pellets in the range room temperature - 1500 °C. The two-step sintering procedure was expected to sinter the specimens to full density without grain growth [I.-Wei Chen, X.-H. Wang, Nature 404, 168 (March 9, 2000)]. All specimens were analyzed by impedance spectroscopy measurements in the frequency range 5 Hz - 13 MHz at a temperature low enough to inhibit grain growth, and by scanning electron microscopy after polishing and thermal etching, for morphology observation and for evaluating average grain sizes. Another set of YFSZ specimens was sintered for different times at a temperature corresponding to the third sintering stage, to promote grain growth without shrinkage [D. Z. de Florio, R. Muccillo, Solid State Ionics 123,301-305 (1999)]. The grain boundary electrical resistivity decreases for increasing sintering time in agreement with the elimination of interfaces during grain growth. The results on two-step sintering show that it is not effective in these ceramics, i.e., the second-step isothermal sintering always results in increase of the average grain size for increasing sintering times irrespective of the sintering stage. Moreover, the impedance spectroscopy technique showed to be a valuable tool for following grain size changes by monitoring the grain boundary electrical resistivity in these polycrystalline solid electrolytes.