

# Nanostructured CaSrTiFeO polycrystalline materials: synthesis, structural and microstructural characterization

L. G. Martinez, V. L. Mazzocchi, C. B. R. Parente, J. Mestnik-Filho, J. R. Carmo, R. Muccillo

*Energy and Nuclear Research Institute, S. Paulo, SP, Brazil*

$\text{Ca}_x\text{Sr}_{1-x}\text{Ti}_{1-y}\text{Fe}_y\text{O}_{3-d}$ ,  $x = 0, 0.5$  and  $1.0$ ,  $y = 0$  and  $0.35$ , polycrystalline ceramic powders were synthesized by a modified polymeric precursor technique starting with calcium and strontium carbonates, iron III oxide and titanium isopropoxide. The polymeric resins resulting from the synthesis were studied by thermogravimetry and differential thermal analyses under oxidizing conditions to determine the temperatures for organic compounds elimination and for crystallization. The ceramic powders were analyzed by X-ray and neutron diffraction for structural phase determination, and by scanning and transmission electron microscopy for evaluation of average particle size and observation of particle shape. X-ray and neutron diffraction experiments were carried out in powders of the  $x = 0, 0.5$  and  $1.0$ ,  $y = 0.35$  compositions calcined at  $1200\text{C}$ . Rietveld analyses of X-ray diffraction data give the following values for the crystal symmetries and lattice parameters: perovskite orthorhombic (SG: P c m n) with  $a = 5.413090$  (0.000038),  $b = 7.654964$  (0.000055) and  $c = 5.410911$  (0.000076) for  $\text{CaTi}_{0.65}\text{Fe}_{0.35}\text{O}_{3-d}$ ; perovskite orthorhombic (SG: P m -3 m) with  $a = 5.470773$  (0.000036),  $b = 5.471629$  (0.000045) and  $c = 7.739964$  (0.000056) for  $\text{Ca}_{0.5}\text{Sr}_{0.5}\text{Ti}_{0.65}\text{Fe}_{0.35}\text{O}_{3-d}$ ; perovskite cubic (SG: P b n m) with  $a = 3.901409$  (0.000005) for  $\text{SrTi}_{0.65}\text{Fe}_{0.35}\text{O}_{3-d}$ . TEM micrographs show that after resin calcination the powders are small agglomerates of nanosize particles. Polished and thermally etched surfaces of pellets sintered at  $1250\text{C}$  were observed in a scanning probe microscope for analysis of grain morphology, showing dense agglomerates of the nanostructured particles.

**Keywords:** strontium titanate, calcium titanate, nanoparticles, X-ray diffraction, neutron diffraction.

(CNEN, CNPq, CAPES, FAPESP)

[muccillo@usp.br](mailto:muccillo@usp.br), CCTM, IPEN, Travessa R 400, Cidade Universitaria, S. Paulo, SP, Brazil.