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M&M 2012
**MICROSCOPY &
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Synthesis and Microstructural Analysis of $\text{La}_{0.7}\text{Sr}_{0.3}\text{Cr}_{0.4}\text{Mn}_{0.6}\text{O}_{3-\delta}$ Perovskite

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Nowadays, there has been much interest in new cermet materials development to enhance hydrocarbon fuels electrooxidation and reduce the catalyzing of C-C formation during SOFC operation [1]. An interesting point is that in the transition metals family, copper metal inhibits C-C bond formation when the C-H bond of a hydrocarbon fuel is broken; besides, it's stable under reduction atmospheres. The main propose is to use new or improved electro-catalyst for solid oxide fuel cells application and temperature ranges from 600-800°C. In order to contribute to this point, we have conducted our research capabilities in LSCM perovskite anodes combined with $\text{XCu}_{0.75}\text{Ni}_{0.25}$ ($x=25, 35, 45, 55, 65, 75\%$) intermetallic solid solutions for SOFC applications. The main objective of this research was to study structural changes by HRTEM, SEM and their relation with crystal structure when there are composition variations.

Nanocrystalline LSCM- $\text{XCu}_{0.75}\text{Ni}_{0.25}$ powders were synthesized by sol-gel method using metal nitrates as raw materials. All reagents were dissolved at 60°C in distilled water, citric acid (chelating agent) and ethylene glycol as an esterification agent and annealed at 1050°C for 6h. LSCM were impregnated with cooper and nickel nanoparticles using the same method and treated thermally at 600°C during 1h. Crystal phases, crystallite size distribution and annealing characteristics of the nanoparticles were investigated and compared by TEM and Rietveld analysis. LSCM- $\text{XCu}_{0.75}\text{Ni}_{0.25}$ powders morphological characterization confirmed that sol-gel method privileged the nanostructures formation, which are formed for an LSCM $\text{Cu}_{0.75}\text{Ni}_{0.25}$ agglomerates from 200 to 500nm; also revealed homogeneous dispersion of metallic cooper and nickel nanoparticles. As an example, STEM-BF image for the 25%wt $\text{Cu}_{0.75}\text{Ni}_{0.25}$ is shown in the figure 1a, where CuO and NiO on the LSCM surface were detected.

Crystallite size distribution obtained from TEM images has been fitted by a log-normal distribution function (Fig. 1b). Geometric mean crystallite size for CuO, NiO and LSCM perovskite was 17.1(7), 21.2(3) and 70.2(4) nm respectively. These data are consistent with XRD refinement results. Crystallite sizes; weren't detected changes in the compositions significantly, only between LSCM annealing and impregnation stages. This can be explained by the agglomeration and growth grains at higher temperatures where atoms mobility is higher. At low impregnation temperatures the atomic mobility is lower; therefore, the grain growth is restricted. Crystallite size variations can be obtained from 200nm to >1 μm (after pressed and sintering process). LSCM crystal structure by Rietveld method showed a R-3c symmetry (Fig. 1c). Rhombohedral structure is highly stable because the Pm-3m symmetry was not detected during all process (before and after reduction process). HAADF-HRETEM image (Fig.1d) taken along [100] direction, can be more easily interpreted because the column intensities of the incoherent image obtained in STEM mode only depends of overall atomic mass (Z). Therefore, the Fourier spectrum shows a good coincidence with the SAD pattern intensity which has been indexed as rhombohedral structure. Our results concluded that annealing temperature

was sufficient for obtain total LSCM perovskite formation without secondary phases. Microstructural defects weren't detected in all compositions when the composition of $\text{Cu}_{0.75}\text{Ni}_{0.25}$ was varied. LSCM perovskite high stability is due to stoichiometric change, which was increased for the Sr^{2+} ion from 0.25 to 0.3 and from 0.5 to 0.6 for the Mn^{3+} ion.

References

- [1] Jiang-Tao Zhang et al., *J. Power Sources*. 200 (2012) 29.
- [2] This research was supported by project CONACYT No. 105837.

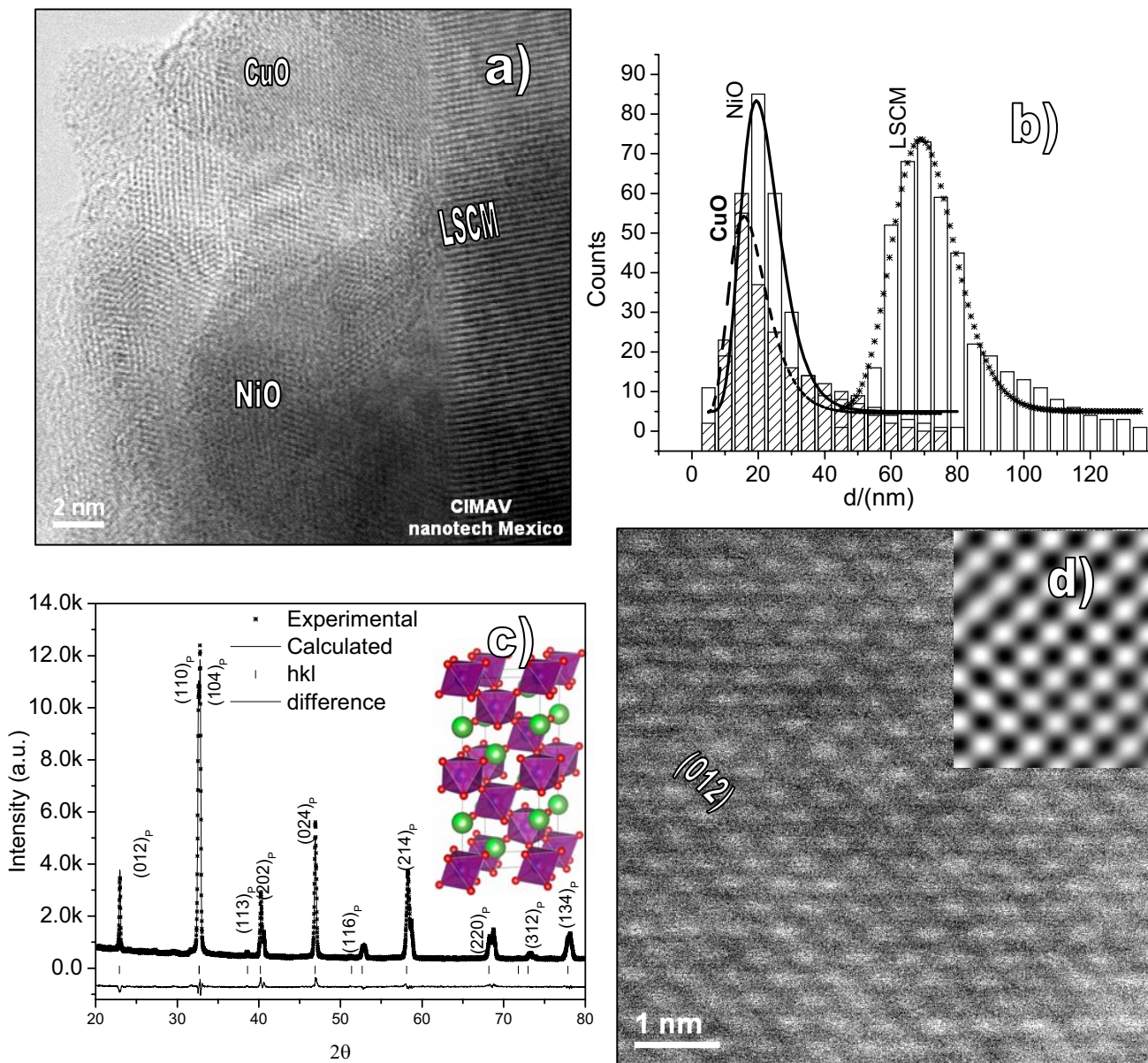


FIG. 1. TEM image of the Nanocrystalline $\text{La}_{0.7}\text{Sr}_{0.3}\text{Cr}_{0.4}\text{Mn}_{0.6}\text{O}_{3-\delta} - \text{XCu}_{0.75}\text{Ni}_{0.25}$ ($x=25, 35, 45, 55, 65, 75\%$) powder at 1050°C during 6h, a) STEM-BF of LSCM particle surrounded by CuO and NiO nanoparticles, b) Grain size distribution obtained from TEM images of LSCM- $\text{XCu}_{0.75}\text{Ni}_{0.25}$ powder synthesized by sol-gel method, c) LSCM perovskite powders Rietveld refinement as R-3c symmetry and d) Z-contrast image of LSCM perovskite closer to $[100]$ zone axis.