

Resolution Powder Diffraction beamline of the European Synchrotron Radiation Facility (ESRF, Grenoble, France). The study was performed at two different temperatures (750°C and 850°C), during 60 hours under 200 mbar of oxygen. Data analysis was done using Topas program (Bruker AXS).

A complex oxide structure was identified, with five different oxides forming in three steps during the high-temperature oxidation treatment. The phase growing during the first oxidation stages was identified and the time when detrimental oxides appear was measured at both temperatures. In parallel, the quantitative analysis of the in-situ powder diffraction patterns brought information about the reaction kinetics.

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**Keywords: low-chromium steel, oxidation, in-situ synchrotron diffraction**

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### Structural properties of AFeO<sub>3-δ</sub> perovskites. Effect of A-site parameters

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The structural properties for the AFeO<sub>3-δ</sub> perovskite materials can be changed using the size and charge of the A sites cations (A = Ln<sub>1-x</sub>M<sub>x</sub>) [1]. The A-size can be evaluated through the mean A-cation radius,  $\langle r_A \rangle$ , and the size variance or size disorder,  $\sigma^2(r_A)$ , which describes the mismatch in ionic radii of the cations at the A-site [2].

The AFeO<sub>3</sub> system has been chosen due to the potential properties and stability of these materials as cathodes for Solid Oxide Fuel Cells [3].

This work summarises the independent studies on the separate effects of  $x \langle r_A \rangle$  [4] and  $\sigma^2(r_A)$  [5] in the structural properties of AFeO<sub>3</sub> perovskites. In order to separate their contribution to the changes in the structural properties, perovskites with general formula Ln<sub>1-x</sub>M<sub>x</sub>FeO<sub>3-δ</sub> (Ln = La, Pr and/or Nd; M = Sr, Ca and/or Ba) with  $0.2 \leq x \leq 0.8$ ;  $1.21 \leq \langle r_A \rangle \leq 1.25 \text{ \AA}$  and  $0.0021 \leq \sigma^2(r_A) \leq 0.0155 \text{ \AA}^2$  have been synthesised by conventional ceramic solid state reaction under identical synthetic conditions. The samples have been characterised by X-ray powder and neutron powder diffraction and Rietveld analysis. For each series, one parameter has been varied independently keeping the other two constant.

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**Keywords: perovskites, synthesis, characterization, structure**

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### Investigations of LiMn<sub>2</sub>O<sub>4</sub> nanocrystalline electrode materials

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Lithium manganese oxide (LiMn<sub>2</sub>O<sub>4</sub>) is a potential candidate for cathode material in Li-ion batteries (LIBs) due to its low cost, low toxicity and high safety. These make LiMn<sub>2</sub>O<sub>4</sub> a material promising for use in large scale batteries for powering electric vehicles (EV) or hybrid electric vehicles (HEV) [1]. The high energy density lithium ion batteries need cathode materials with both large volumetric capacity and high density. Nanocrystalline lithium manganese oxide (LiMn<sub>2</sub>O<sub>4</sub>) material of spinel structure was synthesized by modified sol-gel method using citric acid as a first chelating agent. As the second agent was used glycol ethylene, glycolic acid or acetic acid [2]. The calcination temperature was 450-700°C for a few hours in air. The obtained samples were characterized by the following methods: scanning electron microscopy (SEM/EDX), X-ray photoelectron spectroscopy (XPS), SQUID magnetometry and EPR. Chemical composition, oxidation state of manganese and magnetic properties were examined.

The SEM images show different size of the grains depending on the synthesis process (the example image is shown below). Some contaminations with sodium, potassium or fluor were coming from the starting materials or technological processes. The XPS the complex Mn3p lines were deconvoluted to find the intensity of the Li 1s line and its content in the examined compounds was determined. The ratio of Mn<sup>3+</sup>/Mn<sup>4+</sup> depended on the synthesis process. The C1s lines of the measured samples were intense and complex due to not well-chosen calcination time and temperature. The results were referred to the magnetic measurements results and ESR showing a presence of some extra magnetic phases.

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### Probing 'breathing' mechanisms of ZIFs with high pressure

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Porous materials have attracted a wide scope of interest within the scientific community for a range of possible uses based primarily upon their large surface areas. Metal-organic frameworks (MOFs) are forming an increasingly attractive sub-section of porous materials due