Synthesis and structural characterization of $La_{.5}Sr_{.25}Ca_{.25}MnO_3$

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LA-SiGMA — Louisiana Alliance for Simulation-Guided Materials Applications

Solid State

Starting Materials

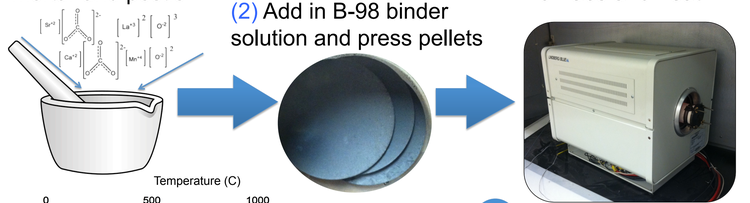
- Lanthanum Oxide
- Calcium Carbonate
- Strontium Carbonate
- Manganese (IV) Oxide

Method

(1) Mix powders in mortar and pestle

(2) Add in B-98 binder solution and press pellets

(3) Place pellets in furnace and heat

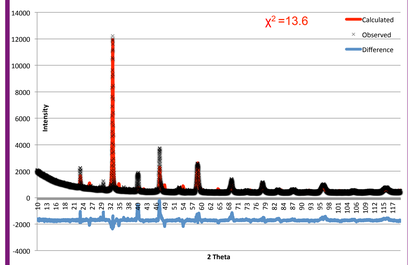


Red rings above demonstrate oxidization, with weight gain on the left and an endothermic spike that leads into exothermic on the right

TGA-DSC showed that the materials had reacted by 1000°C

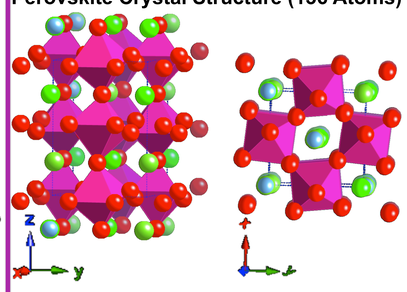
Structure and Composition

LSCMO fired up to 1500°C and GSAS structure refinement



XRD demonstrates phase purity and high symmetry of LSCMO, based on a small # of peaks and a pattern of peak to peak distance

Perovskite Crystal Structure (136 Atoms)

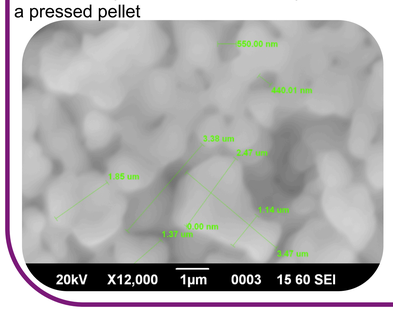


Space group: Pbnm (cubic)
La, Sr, Ca in the 4c, O1 + O2 in the 4c and 4d positions respectively, Mn in the 4b

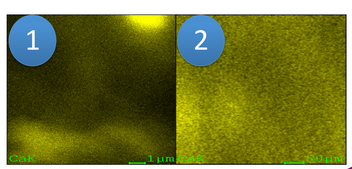
Note: Space group R -3 c also fit, further crystallography studies about the translation and matrix will be necessary to determine the relationship

- Red= O
- Magenta= Mn
- Green and Blue = La, Sr, and Ca

SEM shows particle size and void spaces within a pressed pellet



EDAX scan of calcium concentration compares two separate pelletized samples:
(1) Fired at 1000°C
(2) Fired at 1200°C
-Second sample shows better distribution of calcium



Introduction

- **Materials that exhibit Colossal Magnetoresistance (CMR) are of great interest to the electrical engineering application known as "spintronics."**
- **Perovskite ceramics like $La_{1-x}Sr_xMnO_{3+\delta}$, exhibit CMR**
- **Little research has been published on large doping (x>.33) or doping with multiple elements.**

- **Our goal was to synthesize $La_{.5}Sr_{.25}Ca_{.25}MnO_3$ (Lanthanum Strontium Calcium Manganate, or LSCMO) by way of two different methods:**

1. **A solid state method that produced large particles**
2. **A solution based method that produced nanoparticles.**

- **We then analyzed the structure and composition of the resulting compounds.**

Methods

The solid state method involved the mixing and pelletizing of powders. Powders were heated in a furnace to high temperatures in excess of 1300°C, and later heat treated to calcify. This method and structural characterization are shown in the left-hand column.

In the sol-gel method, the materials were formed into a gel and only heated up to 900°C. This method and structural characterization are shown in the right-hand column.

For the analysis:
a JEOL JSM-6340 SEM identified distribution (via EDAX) and particle size, an SDT performed TGA and DSC analysis, while a Bruker copper anode tube XRD, at 40kV and 40 mA went from 10°<2Θ<120° to identify the phases and crystal structure, and crystal analysis was performed on GSAS and Topaz

The Next Step

Now that we have the desired materials, we need to make the measurements of the material's magnetoresistivity at very low temperatures and compare it with previous findings, as well as perform other characterization tests and refine the synthesis process even further.

Questions? Comments? Concerns?

Acknowledgements

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Sol-Gel

Starting Materials

- Lanthanum Hydroxide
- Strontium Hydroxide
- Calcium Carbonate
- Oxalic Acid
- 70% Nitric Acid
- Manganese (II) Nitrate

Method

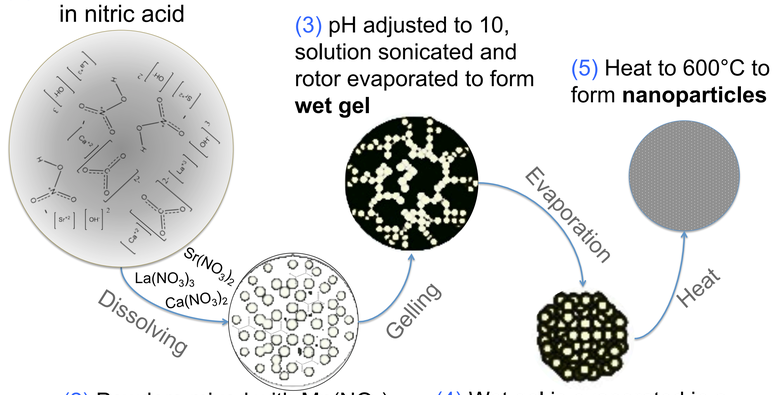
(1) Powders dissolved in nitric acid

(2) Powders mixed with $Mn(NO_3)_2$ and Oxalic acid to form **solution**

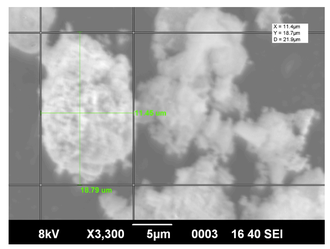
(3) pH adjusted to 10, solution sonicated and rotor evaporated to form **wet gel**

(4) Wet gel is evaporated in a vacuum oven to form **gel/ powder**

(5) Heat to 600°C to form **nanoparticles**



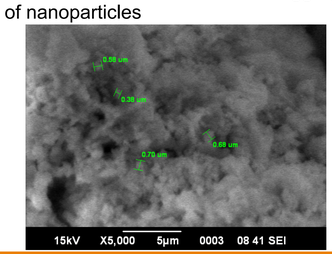
Structure and Composition

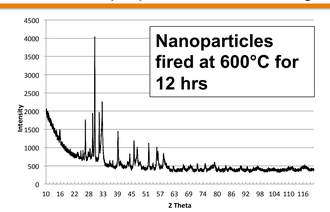


- Particle sizes are shown on a 5 micrometer scale, and the sample on the right was done using a solution preparation on a TEM grid

SEM photos of nanoparticles

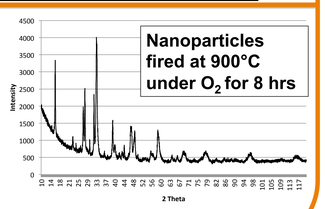
- The flowering of these microparticles demonstrates the material is an aggregate of nanoparticles





XRD Analysis

Large peak width is a sign of nanoparticles (compare with solid state, which has thin peaks)



XRD asymmetry showed reaction hadn't gone forward 100%

Peaks show increasing similarity to solid state XRD, but more treatment needs to be done