

Influence of substrate material and annealing process on phase and crack formation of LIB electrodes in the system Li-Mn-O

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In this work Li-Mn-O thin film cathodes have been deposited onto Silicon, oxidized Silicon and stainless steel substrates by non-reactive r.f. magnetron sputtering from a ceramic LiMn_2O_4 target. The pressure during sputtering was 7 Pa and r.f. power was 100 W. Film thickness was about 1.5 μm . The as-deposited films were annealed in vacuum in a temperature range from 300 °C to 700 °C for one hour to induce crystallization. The microstructure of the films was characterized by X-ray diffraction (XRD) at room temperature. Surface characterization of the films was carried out by scanning electron microscope (SEM).

LiMn_2O_4 - Target

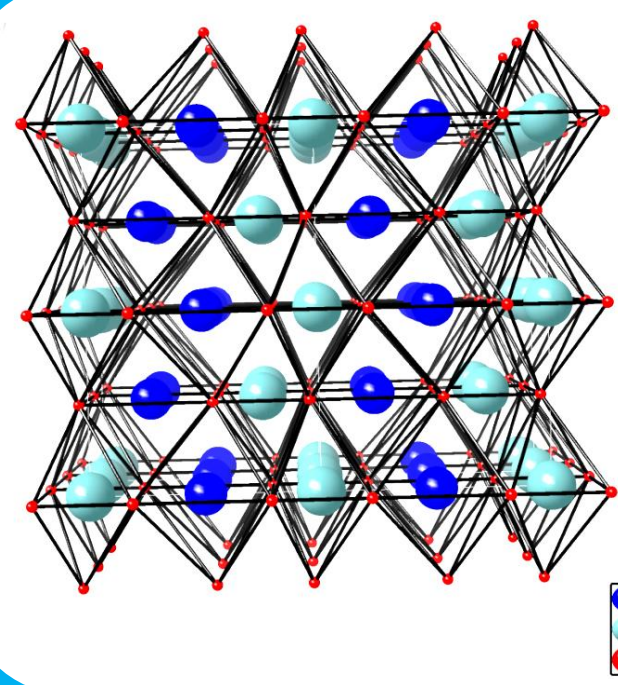
Selected Parameters:

Deposition: 7 Pa; 100 W r. f. power

Film thickness: 1.5 μm

Annealing: 300 – 700 °C (60 min): Air / Vacuum

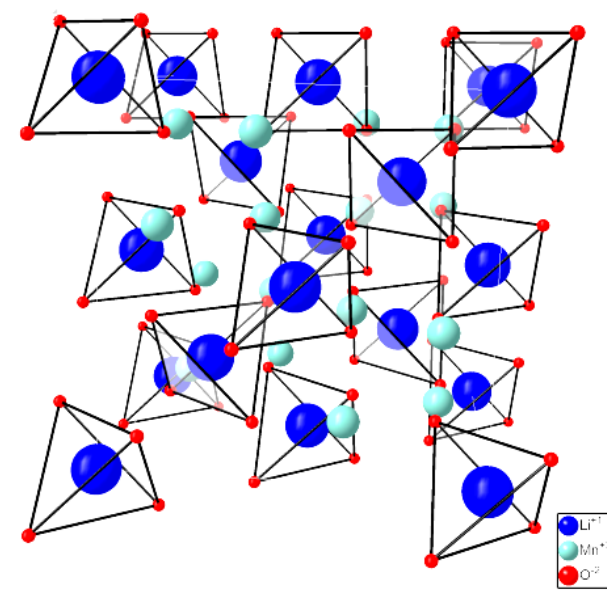
Substrates: Silicon / Stainless Steel (SS 30400)



Properties of $o\text{-LiMnO}_2$

Theoretical capacity: 285 mAh/g
Practical capacity: 120 - 180 mAh/g
Voltage vs. Li: 3.5 V - 4.5 V

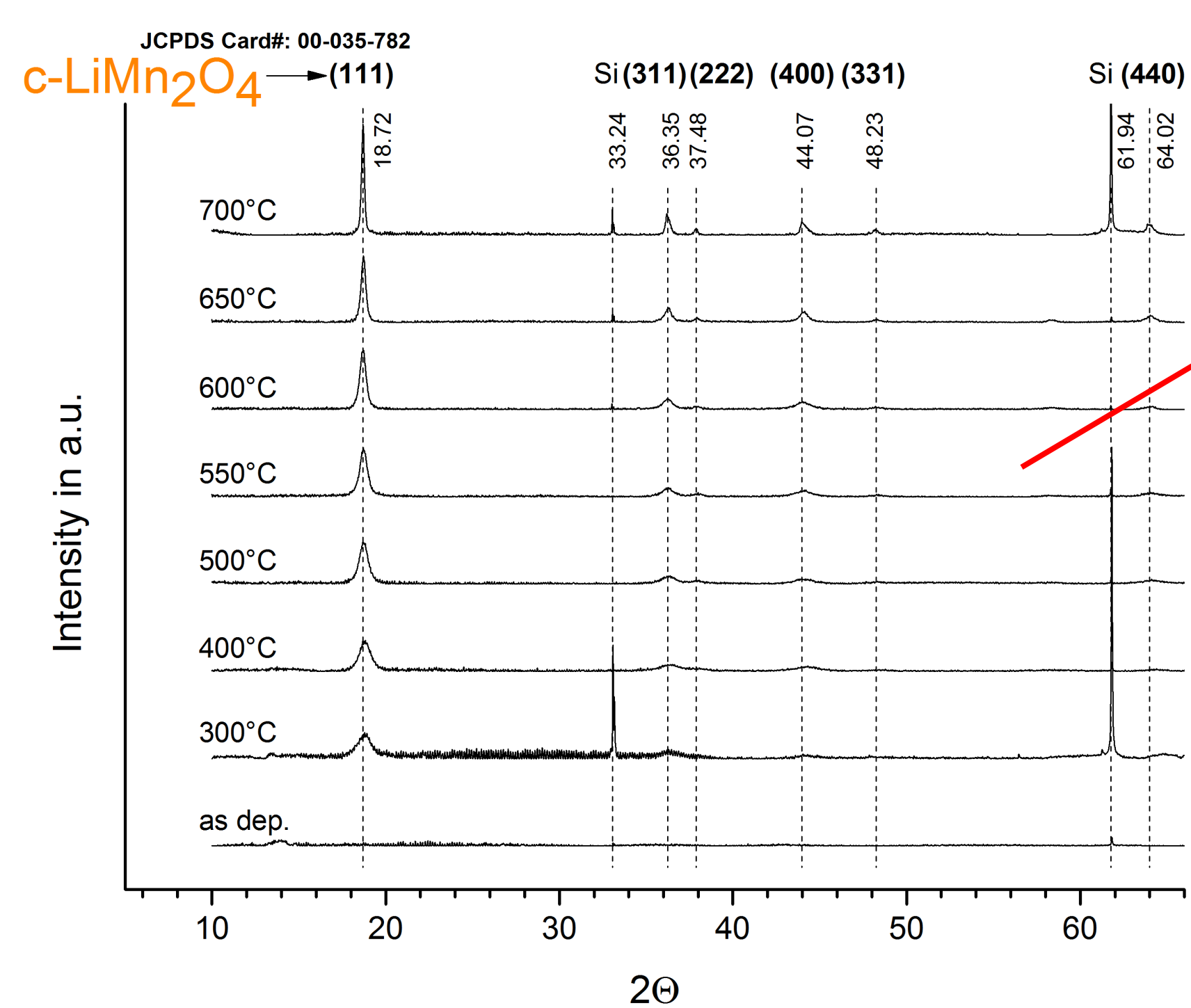
Space group: Pmmn
 LiO_6 and MnO_6 octahedral are arranged in corrugated layers



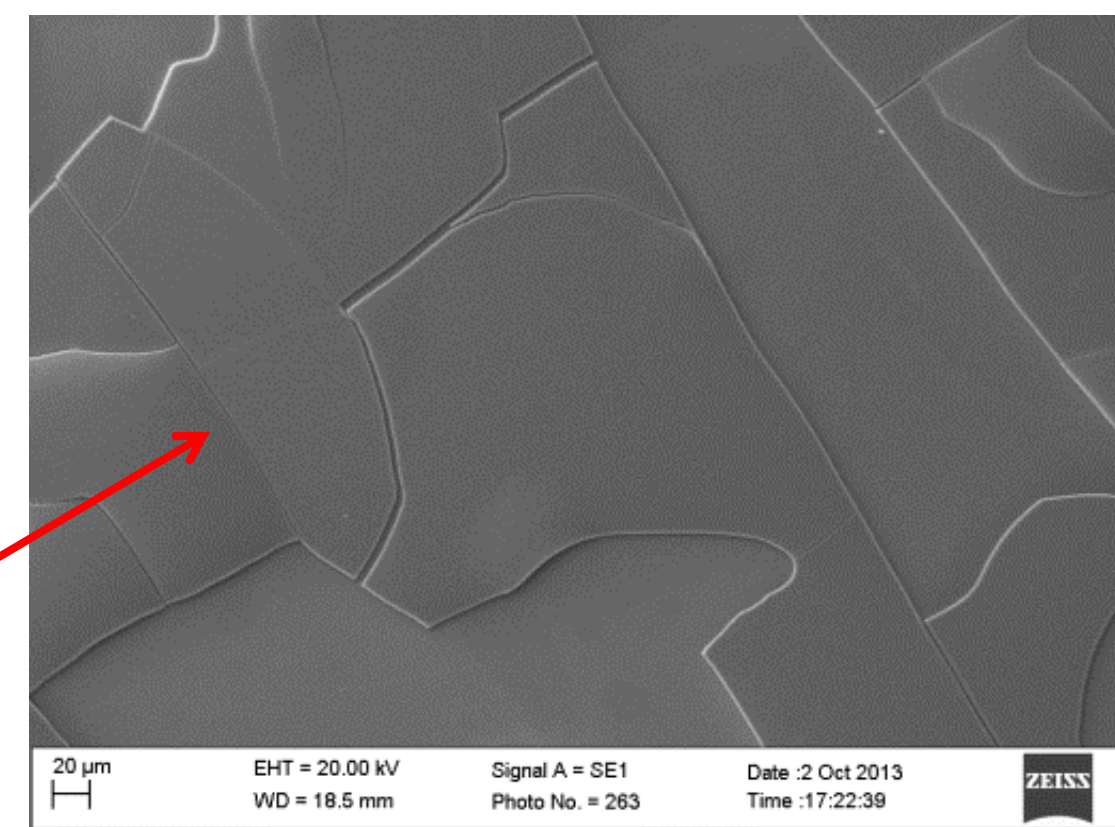
Properties of $c\text{-LiMn}_2\text{O}_4$

Theoretical capacity: 148 mAh/g
Practical capacity: 80 - 135 mAh/g
Voltage vs. Li: 3.0 V - 4.3 V

Space group: Fd3m
Li and Mn atoms occupy octahedral and tetrahedral sites

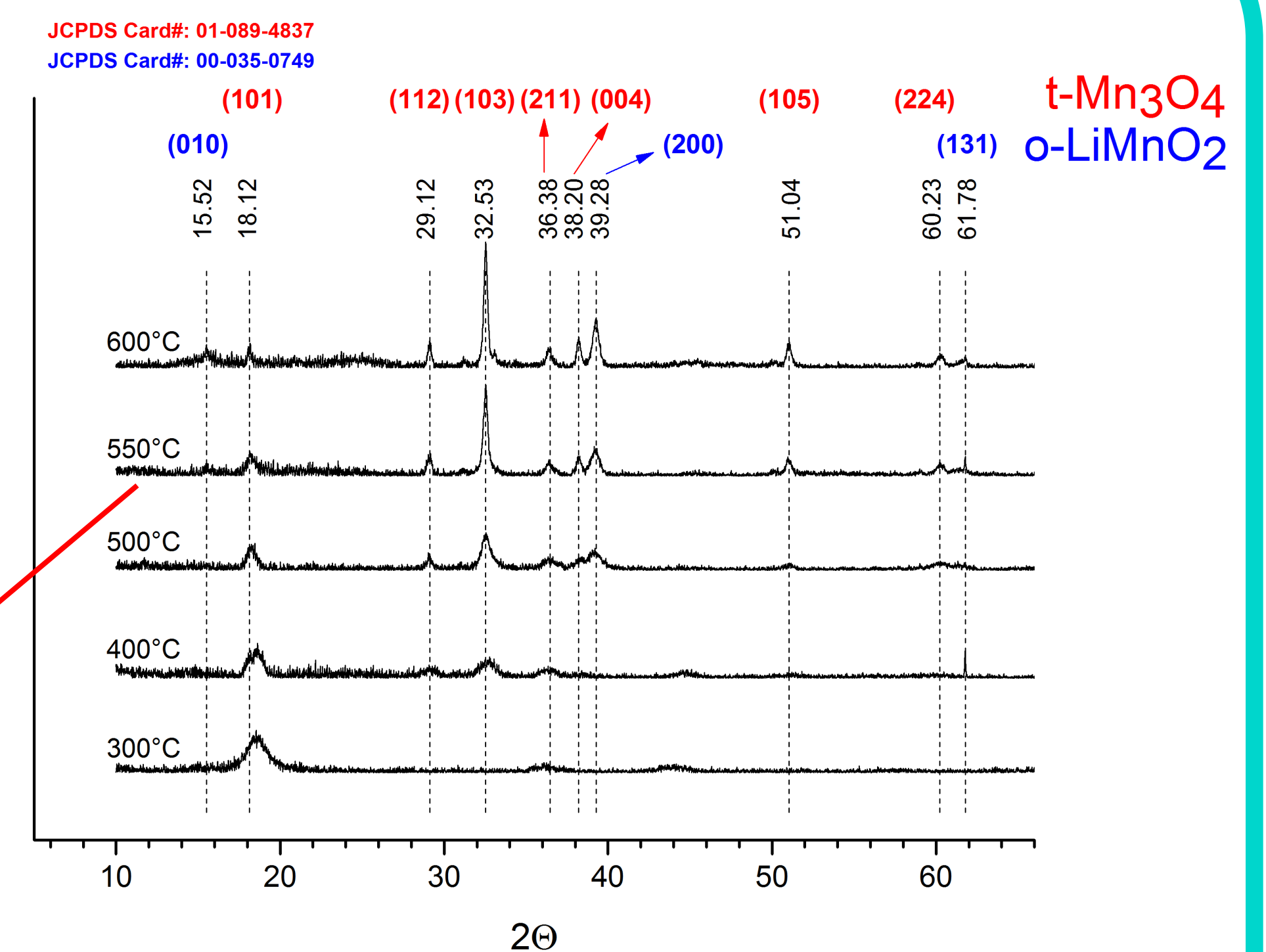
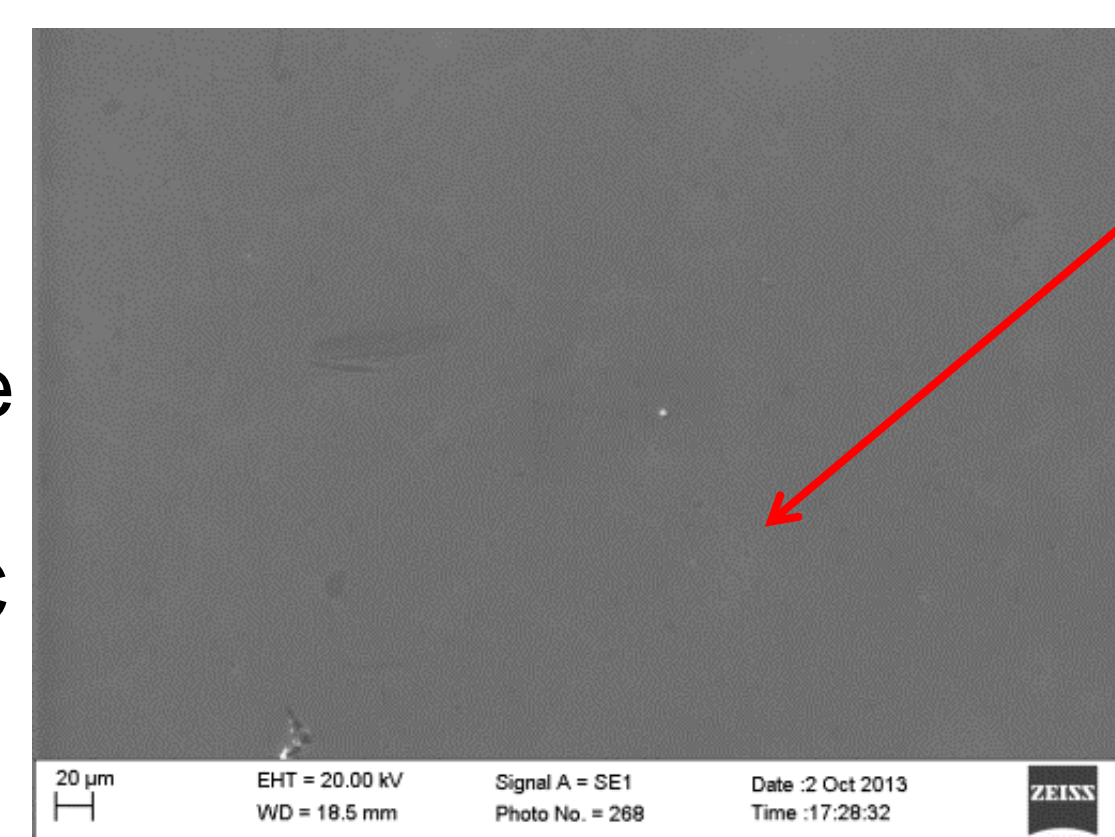


X-Ray diffraction of samples deposited on **Silicon** and annealed in **air**

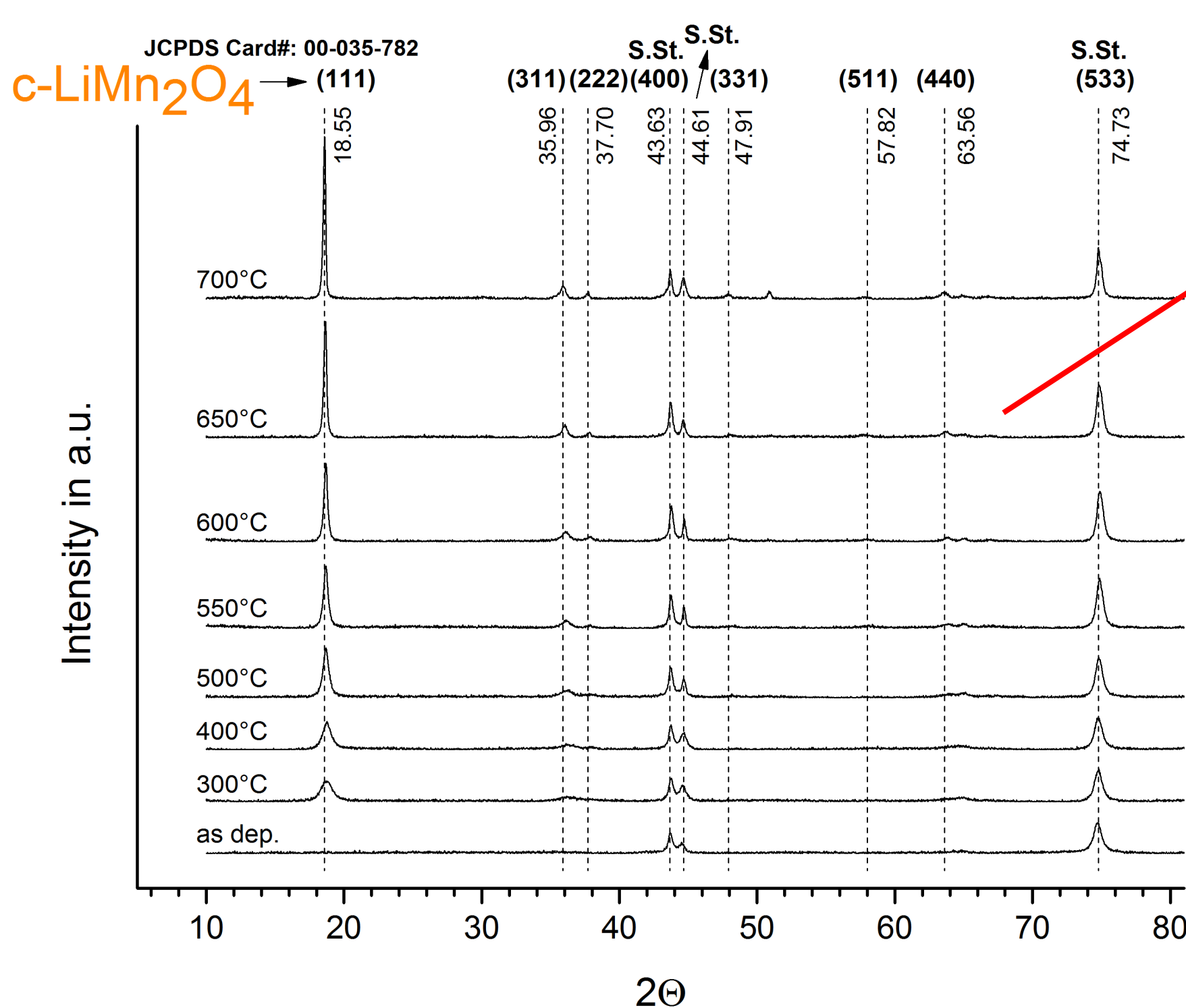


SEM picture the sample deposited on **Silicon** and annealed at 550 °C in **air** show cracks

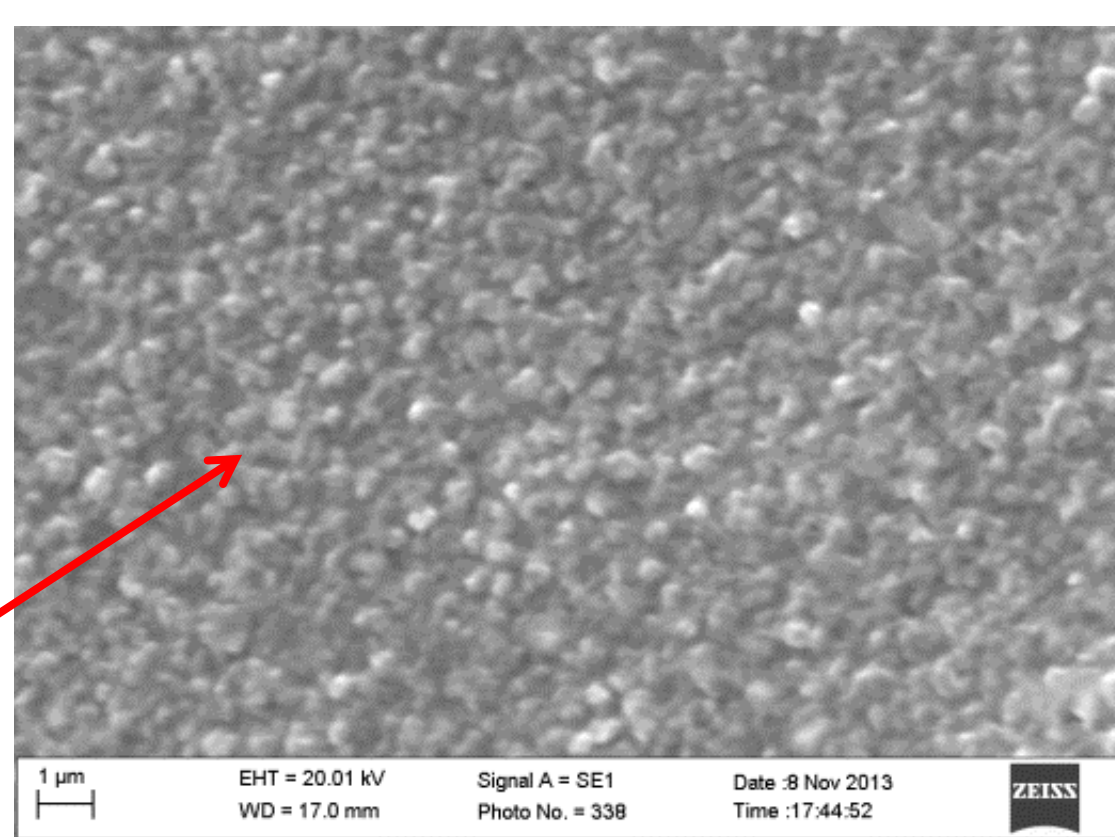
SEM picture the sample deposited on **Silicon** and annealed at 550 °C in **vacuum** show cracks



X-Ray diffraction of samples deposited on **Silicon** and annealed in **vacuum**

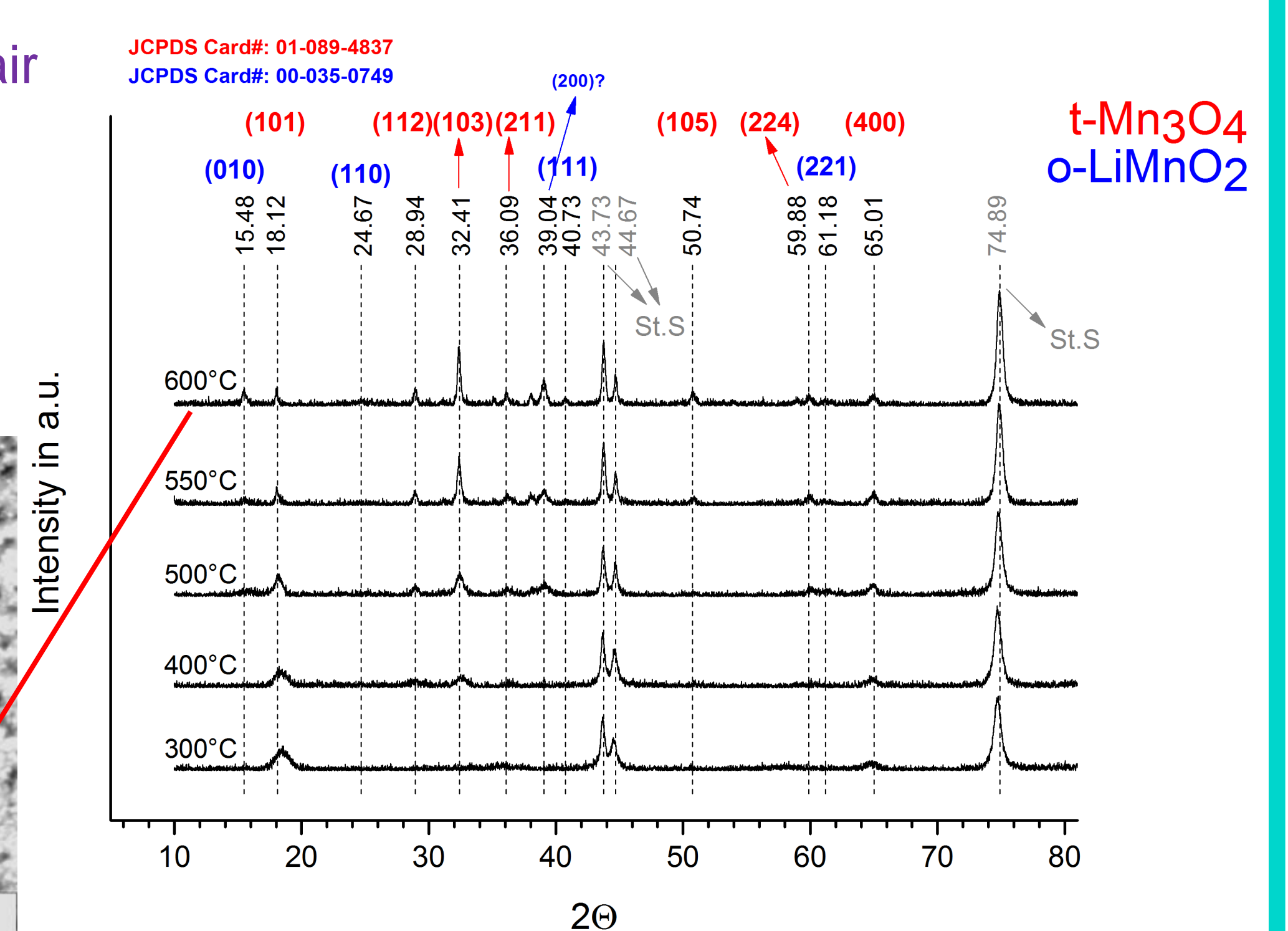
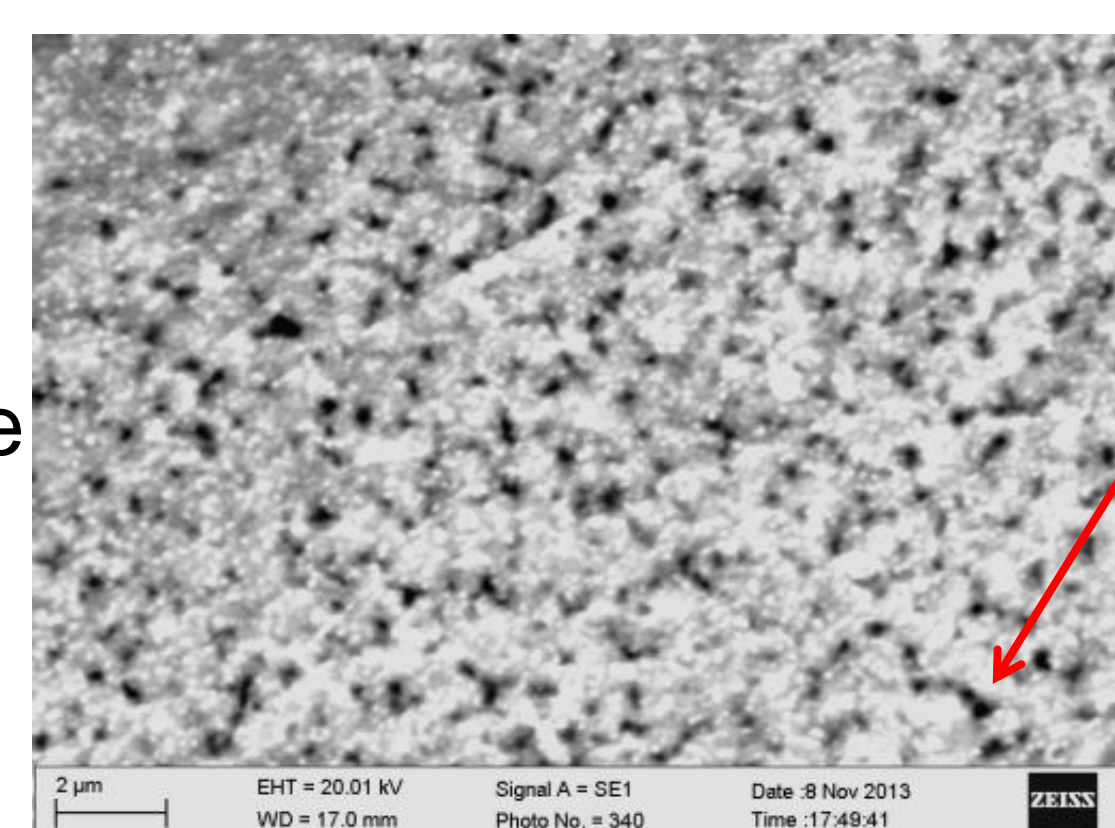


X-Ray diffraction of samples deposited on **SS** and annealed in **air**



SEM picture the sample deposited on **SS** and annealed at 550 °C in **air** without cracks

SEM picture the sample deposited on **SS** and annealed at 550 °C in **vacuum** without cracks



X-Ray diffraction of samples deposited on **SS** and annealed in **vacuum**

We investigated the synthesis of Li-Mn-O thin film cathode materials by magnetron sputtering, as well as possible effects of heat treatment. It has been shown that the thin films were successfully deposited onto different substrates. During annealing in **air** implemented the formation of the **cubic spinel phase**. Different microstructures containing orthorhombic LiMnO_2 and tetragonal Mn_3O_4 phases were obtained by **vacuum** annealing. The second major finding was that the crack formation in the Li-Mn-O films was observed first at 400 °C for Silicon and can be avoided on stainless steel.