The essential differences in polymerization of C_{60_hcp} are a preservation of hcp-structure up to 200^0 C and the enormous broadening of diffraction peaks of the polymerized phases. Diffractograms of the polymeric phases of C_{60_hcp} contain halos. C_{60_hcp} polymers are less stable than fcc polymers. The destruction point of 2D polymerized structure C_{60_hcp} is 600^0C, the total destruction of fullerene molecules is observed at 800^0 C. The fullerene molecules of 2D polymeric structures of C_{60_fcc} are destroying at 950^0 C. XPS measurement has shown that the shape of molecules polymerized changes.

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Rietveld refinement of the x-ray diffraction pattern of $lini_{0.65}Co_{0.25}Mn_{0.10}O_2$ layered oxide M. Dahbi[a], I. Saadoune[a], K. Edström[b] and T. Gustafsson[b] [a] Equipe Chimie des Matériaux et de l'Environnement, LP2E2M, FST Marrakech, Université Cadi Ayyad, Morocco [b] Ångström Laboratory, Department of Materials Chemistry, Uppsala University, Sweden

Keywords: Rietveld Refinment, Functionally graded Materials, Energy storage

The AMO_2 compounds such as $LiCoO_2$ and $LiNiO_2$ are important prototypes for modern high energy density battery materials awaiting commercial application as a large scale [1-2]. Many years ago, we have investigated the $LiNi_{1-y}Co_yO_2$ solid solution and shown that the $LiNi_{0.7}Co_{0.3}O_2$ composition presents the best electrochemical cycling properties. Nevertheless, this phase shows a low electronic conductivity which contributes to the decrease of the electrochemical properties upon cycling [3]. The substitution of Mn^{4+} for Ni^{3+} and Co^{3+} in this phase leads to the presence of both Ni^{2+} and Ni^{3+} which makes easier the electronic hopping and enhances the electronic conductivity. The $LiNi_{0.65}Co_{0.25}Mn_{0.10}O_2$ oxide was prepared at three different temperatures: S_1 (900°C/1h), S_2 (900°C/12h) and S_3 (1000°C/12h) with the aim to show the effect of the thermal treatment on the cationic structural distribution. Indeed, $LiNi_{0.65}Co_{0.25}Mn_{0.10}O_2$ compound crystallizes in the trigonal system (space group R-3m) that belongs to the α-NaFeO$_2$ structure type. Some transition metal is invariably found in the lithium layer (3a site) displacing that lithium to the transition metal layer (3b site). A full Rietveld refinement of the X-ray diffraction pattern of $LiNi_{0.65}Co_{0.25}Mn_{0.10}O_2$ compound was performed to determine the degree of cation mixing. The splitting of the (018)/(110) doublet and the intensity ratio o the (003)/(104) diffraction lines superior to unity suggests a good 2D character of the structure. The resulting data shows that only 0.03 Ni^{2+} are presents in the lithium site for S1 sample, whereas for S2 and S3 samples, the degree of cation mixing is 0.06 and 0.12 respectively. This results is confirmed by the continuous increase of the MO_2 (M : $Ni_{0.65}Co_{0.25}Mn_{0.10}$) slab thickness, given by $2(1/3-z_{oxygen})c_{hex.}$, as a results of the increase of the Li^+/Ni^{2+} mixing leading to the decrease of the covalency within the slab. Structural features deduced from this study were correlated to the electrochemical behaviour of the three studied samples. Presence of Ni^{2+} ions in lithium plane clearly reduce the electrochemical capacity and the rate capability as a results of Li^+-Ni^{2+} electrostatic repulsion during the intercalation process.

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The Li_2FeSiO_4 structure under battery cycling Anton Nytén, Torbjörn Gustafsson & Josh Thomas, Ångström *Advanced Battery Centre, Department of Materials Chemistry, Uppsala University, Sweden.* E-mail: anton.nyten@mkem.uu.se

Keywords: Li-ion battery; cathode material; in situ XRD

Li_2FeSiO_4 has recently emerged as a promising new cathode material for Li-ion battery applications [1,2]. The lithium-ion extraction/re-insertion mechanism in the material has sub-sequently been shown to be somewhat complex, involving some type of structural rearrangement – mainly taking places during the first cycle [3]. This is the prime focus of the work reported here.

Li_2FeSiO_4 powder was prepared by the solid-state reaction of $FeC_2O_4 \cdot 2H_2O$ and Li_2SiO_3; the powders were thoroughly mixed with a carbon precursor (10 wt.%) and heated at 700°C for 20 h under a flow of CO/CO_2. The electrodes studied here by *in situ* powder XRD comprised Li_2FeSiO_4:CB:EPDM in a 80:10:10 ratio. Electrochemical cycling was performed in galvanostatic mode at 60 °C. "Coffee-bag" type cells were prepared inside an Ar-filled glove-box with lithium metal as anode and 1M LiTFSI in EC:DEC 2:1 as electrolyte. Cells were tested in the voltage range 2.0 - 3.7 V at a C/25 rate on a Digatron BTS-600 battery tester. The *in situ* XRD profiles were measured on a STOE PSD powder diffractometer.

All samples contain a small amount of impurities, mainly FeO (~5wt.%). The crystal system of the as-prepared Li_2FeSiO_4 has been confirmed to be orthorhombic, space-group: Pmn2_1; Z=2, a=6.2667(5)Å, b=5.3296(5)Å, c=5.0147(4)Å, V = 167.5(3) Å^3, with the Li, Fe and Si atoms all tetrahedrally coordinated to four oxygen; see Fig. 1. However, a few weak reflections in the diffractogram indicate the presence of a superstructure ordering dependant upon the thermal history of the sample. A fully refined structure of this compound has yet to be reported.
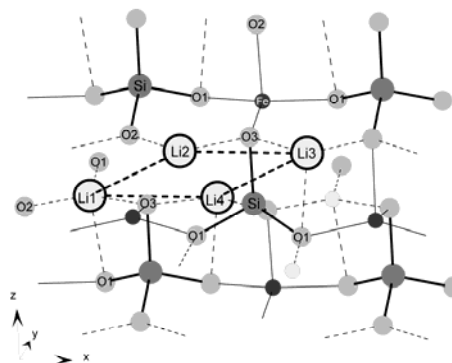

Fig.1. The crystal structure of Li_2FeSiO_4

During the first electrochemical cycle, a shift in the potential plateau from 3.1 to 2.8 V indicates an interesting rearrangement of the structure, presumably involving a phase transition to a more stable structure.