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Molten salt synthesis of multi-faceted pure-phase spinel LiNi$_{0.5}$Mn$_{1.5}$O$_4$ platelets

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ABSTRACT: The spinel LiNi$_{0.5}$Mn$_{1.5}$O$_4$ is largely studied as a positive electrode material for lithium-ion batteries, but further optimization of its properties are required to enable its commercialization. The superior electrochemical performance of the disordered polymorph of LiNi$_{0.5}$Mn$_{1.5}$O$_4$ is limited by its traditional method of synthesis. This solid-state route generates impurities that reduce specific capacity and results in the formation of octahedral particles with exposed {111} facets, thus limiting exploration of the effects of surface orientation. In this work, we report for the first time the preparation of a disordered impurity-free LiNi$_{0.5}$Mn$_{1.5}$O$_4$ with platelet-like morphology via molten salt synthesis. We discovered that these platelets exhibit multiple surface orientations, including {111}, {112} and six other high-indexed facets, and deliver equivalent energy storage performance to their octahedral counterpart. Such ability to tune primary particle morphology and orientation will open the gate to investigate mechanisms at the individual particle level using spectroscopy and microscopy techniques.

KEYWORDS: high-voltage spinel, LiNi$_{0.5}$Mn$_{1.5}$O$_4$, molten salt synthesis, platelet morphology, surface orientation

INTRODUCTION

Spinel LiNi$_{x}$Mn$_{1-x}$O$_{4}$ (LNMO) is one of the most promising and cost-effective candidates for next-generation positive electrode materials for Li-ion batteries. This material offers a high capacity of 147 mAh.g$^{-1}$ due to the oxidation of Ni$^{2+}$ to Ni$^{4+}$ at an average potential of 4.7 V vs. Li/Li$^+$, which results in an appealing energy density of 650 Wh/kg.\textsuperscript{1,2} However, some issues still prevent its implementation in commercial devices. Limited stability of electrolytes at such high potentials leads to secondary reactions at the electrode/electrolyte interface involving Mn dissolution into the electrolyte and cathode electrolyte interphase growth.\textsuperscript{3} A deep comprehension and adjustment of its intrinsic properties are necessary to make LNMO-based batteries available.

In LNMO spinel structure, Ni and Mn can occupy either a single crystallographic site or two different ones within cubic symmetry unit cells. The first unit cell is described in the $Fd$3$m$ space group and associated with a disordered (statistical) distribution of transition metal ions, whereas the second one is described in the $P4_132$ space group and associated with a 1:3 ordering between the two transition metal ions. The disordered LNMO is generally obtained via high-temperature solid-state syntheses ($\geq$ 900°C), inevitably next to a rocksalt-type impurity such as NiO and LiNi$_{1-x}$O.\textsuperscript{4} The formation of this mixture of phases is driven by the oxygen loss from the stoichiometric phase LiNi$_{1-x}$Mn$_{x}$O$_4$ at such elevated temperatures. This phenomenon also causes the partial reduction of Mn$^{4+}$ into Mn$^{3+}$ to balance the decreasing Ni/Mn ratio within the spinel structure. Thus, the disordered LNMO is composed of LiNi$_{5}$Mn$_{4}$O$_{10}$.\textsuperscript{5} The material is commonly re-annealed at a lower temperature ($\sim$ 700°C) to homogenize it and re-oxidize Mn$^{3+}$ into Mn$^{4+}$ to obtain the stoichiometric and ordered LNMO LiNi$_{2+}$Mn$^{4+}$O$_4$.\textsuperscript{5} Regarding electrochemical performance, the disordered phase usually exhibits higher electronic conductivity due to manganese mixed valence, better structural stability and higher specific capacity, especially at high rates and for long-term cycling.\textsuperscript{6} As rock-salt impurity tends to be located at the surface of disordered phase primary particles,\textsuperscript{7-10} not only does it hinder access to the full electrochemical capacity of spinel, but it also leads to misleading investigations of surface reactivity.

Particle morphology and surface orientations impact spinel LNMO energy storage performance\textsuperscript{11} by influencing the electrical conductivity, Li-ion diffusion kinetics, and surface reactivity at the electrode/electrolyte interface.\textsuperscript{12} Indeed, theoretical calculations have revealed that {111} surfaces show the lowest surface energy for spinel LNMO,\textsuperscript{13,14} favoring thus octahedral morphology. Additionally, these {111} surfaces promote superior electrochemical performance due to larger atomic density and reduced transition metal dissolution compared to other surfaces, such as {110} and {100} found on truncated octahedra.\textsuperscript{15-18} For all the particle morphologies that can be experimentally obtained (octahedra, truncated octahedra, sphere, nanorods, platelets), one or two crystallographic planes are usually exposed depending on the synthesis conditions. For instance, conventional solid-state synthesis does not allow the control of LNMO particle morphology or surface orientation and always leads to octahedra or truncated octahedra with exposed crystallographic planes of {111} or {100} or {110}, respectively. LNMO with platelet morphology reported in the literature revealed, until now, either {100} or {110} or {112} crystallographic oriented planes at their surface. Hai et al.\textsuperscript{21,24} have
shown that 112-exposed platelets also deliver a lower diffusion coefficient, poor rate capability, and significant self-discharge compared to 111-exposed octahedra. In all these experimental studies, establishing the relationship between morphology, stability, reactivity, and electrochemical performance is not straightforward due to the presence of rock-salt type impurity and a limited variety of exposed crystallographic planes. Therefore, it would be ideal to examine particles with similar morphologies, free of rock–salt type crystalline domains, and possessing various crystallographic planes on their surfaces to study interfacial reactivity and phase propagation during cycling. However, such impurity-free samples with multi-faceted oriented particles are lacking.

In this work, we report for the first time a disordered spinel-type sample, impurity-free and with micron-sized platelets showing multiple surface orientations. It could be obtained using molten salt synthesis in a LiCl medium. Compared to conventional solid-state syntheses, the molten salt approach permits high reaction rates at lower reaction temperatures, which allows impurity phase control. Furthermore, changing the characteristics of the salt and reaction parameters makes it possible to tune the particle size, shape, orientation growth, and homogeneity. According to transmission electron microscopy (TEM), we have shown that eight different platelet orientations, including [111], [112] and [110], as well as high index planes such as [126] and [334], could be obtained in platelet-like sample LNMO.

In this letter, we discuss the molten salt synthesis of platelet-shaped disordered LNMO with multiple crystallographic orientations, addressing the influence of annealing temperature and salt over transition metal ratio on the final material. The structural and electrochemical characteristics of a platelet-like sample are assessed in comparison to those of two LNMO samples with octahedral-shaped morphology.

**EXPERIMENTAL SECTION**

**Sample Preparation.** LiNi$_{0.5}$Mn$_{0.5}$O$_4$ (LNMO) samples were obtained by molten salt synthesis. All precursors were provided by Sigma-Aldrich (≥ 97.0 %) and directly used without any chemical/physical pretreatment. Stoichiometric amounts of Ni(NO$_3$)$_2$·6H$_2$O and Mn(NO$_3$)$_2$·4H$_2$O (1:3) were dissolved in 1 mL of deionized water. The LiCl salt was added to the mixture according to a defined molar ratio “R” (R = number of moles of LiCl / number of moles of transition metals) and mixed in the mortar. The obtained paste was dried in a muffle oven for 3 h at 180°C and blended using a SPEX Mixer/Mill for 20 min. The final mixture was annealed in an open alumina boat in a closed tubular furnace, under air for 4 h at 750°C or 900°C, depending on the targeted morphology.

**Material Characterization.** Powder X-ray diffraction (XRD) measurements were carried out with a PANalytical Empyrean Diffractometer with Cu K$_{α1,2}$ radiations in Bragg-Brentano configuration. Diffraction patterns were collected in a 28 range of 15-120° with a scan step of 0.008° for a total of 20 hours of acquisition. The lattice parameters were determined using the FullProf program via the full pattern matching refinement (Le Bail method). For microstructural analysis, the XRD data collected for highly crystalline Li$_x$B$_y$ were used to model the instrumental function. Crystallite size and microstrains were calculated using the Williamson-Hall method and a description of the peak profiles by the Thomson-Cox-Hastings Pseudo-Voigt function taking into account additional anisotropic Lorentzian size and strain broadening models. Size broadening can be written as the linear combination of cubic harmonics, and strain can be modeled using a quartic form in reciprocal space following Laue class $3m$.

The Raman spectra were acquired using a confocal LabRAM HR Evolution micro-spectrometer (Horiba) using a 633 nm Argon gas laser source and a 600 gr/mm grating. Each spectrum was collected from 100 to 800 cm$^{-1}$ using a 10.6 mm focal length lens, with acquisition time of 10 s and 40 accumulations.

In order to determine the composition in Li, Ni and Mn, elemental analyses by inductively coupled plasma-optical emission spectroscopy (ICP-OES) were performed using Agilent 5800 spectrometer after a complete dissolution of the sample in an equal mixture of hydrochloric (HCl) and nitric acid (HNO$_3$).

The surface chemical analysis was carried out by X-ray photoelectron spectroscopy (XPS) using a K-Alpha Thermo Fisher spectrometer equipped with a monochromatic Al-Ka (hv = 1486.6 eV) source and a 400 µm X-Ray spot size.

The scanning electron microscopy (SEM) images were taken using a TESCAN Vega microscope with a tungsten source at 15 kV accelerating voltage. The experiments were carried out using a microscope JEOL JEM-2100 (LaB$_6$) at 200kV, and electron diffraction patterns were collected using the selected area technique (SAED) with an Orius 200D (Gatan) camera. LNMO powder was dispersed in a small amount of ethanol in a mortar and deposited on a copper grid for sample preparation. A random particle selection was made on different zones of the grid, choosing isolated particles that were naturally laid on their platelet-like faces. The determination of the platelet orientation was done by collecting the corresponding electron diffraction pattern.

**Electrochemical Characterization.** The electrode slurry was prepared by mixing 80 wt.% of LNMO powder, 10 wt.% of carbon black conductive additive (Alfa Aesar, 99.9 %), and 10 wt.% of Poly(vinylidene fluoride) (PVDF, Sigma-Aldrich) binder in 1-methyl-2-pyrrolidone as solvent (NMP, Sigma Aldrich, 99.5 %) for 3 hours under stirring. The obtained slurry was cast on an aluminum foil used as the current collector, with a thickness of 150 µm, and dried in an oven at 80°C for at least 6 hours. 1.6 cm diameter disks of electrodes were cut and dried in a Buchi oven overnight at 80°C. CR2032-type coin cells were assembled in an Ar-filled glovebox using Li metal as negative electrode, Whatman® separator and 1M LiPF$_6$ EC/DMC electrolyte (1:1 w/w ethylene carbonate (EC) / dimethyl carbonate (DMC)). The electrode loading for all samples was around ~3 mg/cm² of LNMO active material.

**RESULTS AND DISCUSSION**

A series of three LiNi$_{0.5}$Mn$_{0.5}$O$_4$ (LNMO) samples was obtained by molten salt synthesis. Stoichiometric amounts of Ni(NO$_3$)$_2$·6H$_2$O and Mn(NO$_3$)$_2$·4H$_2$O (1:3) were mixed with the salt LiCl used at the same time as the molten medium and as Li precursor, according to a molar ratio “R” (R = number of moles of LiCl / number of moles of transition metals). The mixture was then annealed in an open alumina boat in a closed tubular furnace, under air for 4 h at 750°C or 900°C. LNMO polycrystalline powder was recovered embedded in excess molten LiCl, mainly removed by filtration using distilled water. The powder was then further washed in excess of ethanol under stirring for 2 days to eliminate the last salt residues remaining at the particles’ surface and in the grain boundaries, and it was finally recovered by centrifugation. The efficiency of the extended washing in ethanol was demonstrated by X-ray photoelectron spectroscopy, as discussed in the supporting information (Figure S1).

The samples are named in the following according to their annealing temperature and Salt / Transition Metal molar ratio (R). The sample of interest, with multi-faceted platelet morphology, was obtained at 750°C with R=35. A second LNMO sample was prepared with a reduced salt quantity, such as R=15 while maintaining the annealing temperature at 750°C in order to determine the influence of the amount of salt. For the third sample, a large excess of molten salt with R=35 was maintained, but the annealing synthesis temperature was increased to 900°C, as conventionally
used for synthesizing disordered spinel LNMO. The chemical composition was determined by inductively coupled plasma-optical emission spectroscopy, as given in Table S1, confirming the expected transition metal ratio (1:3 for Ni:Mn) for all samples.

Figure 1. Powder XRD patterns of LiNi0.5Mn1.5O4 samples synthesized at different temperatures and with different salt/transition metals molar ratios (R). On the right, enlargements demonstrate the absence of diffraction peaks corresponding to the Li3Ni2O4 rocksalt-type compound.

Figure 2. Evolution of apparent crystallite size for different planes calculated from XRD data by Williamson-Hall Plot (a); SEM images of the three samples prepared in different conditions: 750°C, R = 35 (b), 750°C, R = 15 (c), and 900°C, R = 35 (d).

750°C with R = 35, homogeneous crystal growth is observed in all directions with an average crystallite size of 63 (±5) nm. On the other hand, when for the same annealing temperature the salt over transition metal molar ratio was decreased to R = 15, the crystallites grew more and displayed an average size of 85 (±11) nm. They grew preferentially along [111] and [311] planes with crystallite sizes of 97 (±1) nm, versus only 75 (±3) nm along [400] and [331] planes. At 900°C, as expected due to higher synthesis temperature, the average crystallite size increased to 124 (±20) nm with a clear preferential growth along [111] planes (151 nm), the more thermodynamically favorable one. Interestingly, the crystallite size along the [400] planes is almost identical for all samples, whatever the synthesis conditions, with a maximum variation of 5 nm. This analysis demonstrates that molten salt synthesis allows tuning the crystal growth.

These observations are supported by Raman spectroscopy analysis, as shown in Figure S2. First, the spectrum of each sample reveals all the peaks expected for a disordered LNMO (Fd3m space group). In addition, a well-defined shoulder is observed at ~660 cm−1 for the sample prepared at 900°C, which reveals the presence of a significant amount of Mn4+. Considering the absence of rock-salt-type impurities (Figure 1) and the chemical composition in good agreement with that expected (Table S1), oxygen deficiency should occur to compensate for the partial reduction of Mn4+ into Mn3+ with thus defects within the spinel structure.

These results show that it is possible to both obtain pure-phase disordered LNMO at different temperatures and tailor Mn4+ content. Kim et al. discussed the importance of the oxygen quantity in contact with molten salt for obtaining a pure spinel phase, whereas they used a closed crucible for their synthesis. In our case, we used an open alumina boat but in a closed furnace. Nevertheless, the large excess of molten salt provides fast kinetics to prepare the lithiated spinel oxide, combined with a limited short calcination time of 4 h at an intermediate temperature of 750°C, the key to mitigate oxygen loss and the formation of rock-salt type impurity phase. More investigation should be done to determine the role of oxygen pressure in obtaining a pure-phase LNMO while using molten salt synthesis.

The influence of molten salt synthesis parameters on apparent crystallite size evolution was investigated using the four most intense peaks from XRD data (Figure 2a). For the sample prepared at

![Figure 1](image1.png)

![Figure 2](image2.png)
In order to investigate the crystallographic planes exposed at the surface of the obtained platelets, SAED patterns were collected on more than 40 particles of sample 750°C, R = 35. Whereas conventional synthesis usually leads to a uniform preferential growth for all the particles, eight different orientations could be observed in the same batch for those platelets. It was found that 40% of the particles were exposed with [111] planes, 24% with [112] planes, and 11% with [123] and [110] planes (Figure 3). Additionally, platelets oriented along high-indexed facets such as [233], [334], [130], and [126] could also be found (Figure 4 and S4).

It is known that the [111] direction of the spinel cubic structure is equivalent to the [001] direction of the layered oxide hexagonal lattice, both having the same triangular oxygen network (Figure S3). As a consequence, the projections along [112] of the first structure and along [110] of the second one are comparable. The presence of platelet-like primary particles with [112] planes could thus be reminiscent of an intermediate state in molten salt synthesis before growth towards thermodynamically favored [111] surfaces. Furthermore, in these synthesis conditions, the steric effect of an excessive amount of LiCl salt is expected to have a greater impact than the thermodynamic growth of particles. The role of alkali ions in the molten medium was for instance reported in the literature for NiO crystal synthesis to stabilize high-index facets. Susman et al. and Cheula et al. suggested that alkali chlorides play a growth inhibitor role on metal oxide surfaces, making high index faceting possible. However, to the best of our knowledge, the presence of platelets with multiple orientations in the same batch has never been observed before, i.e. with as expected those oriented along the most stable surface (111), but also along kinetically favorable surfaces such as (112) and other “uncommon” planes.

The electrochemical behavior of multi-faceted platelet-type LNMO samples was evaluated and compared to those obtained for two octahedral-type LNMO samples (Figure 5). The materials were cycled vs. Li metal at a cycling rate of C/10 (theoretical exchange of one Li+ in 10 hours) in a potential window of 3.5 - 4.9 V vs. Li+/Li. The second charge-discharge curves and the associated dQ/dV plots are presented in Figures 5 (a-b). All samples show an electrochemical behavior typically observed for disordered LiNi0.5Mn1.5O4, with reversible capacities close to the theoretical value of 147 mAh.g⁻¹ and a phase diagram in agreement with that already reported in literature. A first solid-solution reaction is involved in the voltage range [3.9 - 4.2 V] with the activation of the redox couple Mn⁴⁺/Mn³⁺. At higher voltages, a second solid-solution reaction is observed in the voltage range [4.6 - 4.73 V] with the activation of the Ni⁴⁺/Ni³⁺ redox couple and lastly a biphasic reaction, associated to a voltage plateau at ~ 4.75 V and related to the Ni³⁺/Ni⁴⁺ redox couple. From dQ/dV plots, it is found that the platelet-like sample 750°C, R = 35 and the octahedral-like samples 750°C, R = 15 are characterized by the same amount of Mn⁴⁺ within the structure (i.e. 7%); for the other octahedral-like sample 900°C, R = 35, this content rises to 10%, supporting again that the Mn⁴⁺ content is dependent on the synthesis temperature but not on the salt quantity used.

Figure 3. TEM images (a-b) and associated SAED patterns (c-d) showing the crystal plane orientation: [111] and [112] respectively. (e) Polyhedral representations of (111) and (112) surfaces of LNMO.

Figure 4. Distribution of different platelet crystallographic orientations, quantified over 40 particles of the same LNMO sample (750°C, R = 35). In inset are given in yellow the representation of the 3D Li⁺ ions pathways within the LNMO structure, and in black their cross section with the (111) plane in blue. Transition metal octahedra are represented in purple and lithium ions in green.
It is worth noting that when using an excess of salt during synthesis, the impact of surface purity on electrochemical performance should not be disregarded. The consequence of a small amount of residual chlorine on the material’s performance in Li-ion batteries is shown in Figure S6. Extended ethanol-washing step allows removing any salt residue at the active material surface, allowing access to the overall bulk capacity and fair comparison of our results.

Figures 5 (c-d) show rate capability tests and long-term cycling at C/2. Overall, from slow to fast cycling rates, the reversible capacities obtained for the three LNMO samples showing different morphologies are similar. The platelet-like sample 750°C, R=35 delivers a reversible capacity close to 100 mAh.g\(^{-1}\) at 4C and a capacity retention of 80% after 175 cycles at C/2, whereas for the octahedral-like samples 110 mAh.g\(^{-1}\) and 90% are obtained respectively. The slight difference in discharge capacities at fast cycling rate of 4C can be attributed to the enhanced electronic conductivity of the sample prepared at 900°C with increased Mn\(^{3+}\) content and thus mixed valence Mn\(^{3+}/Mn^{4+}\) on one side, and to the octahedral morphology showing [111] oriented facets and thus faster Li\(^+\) diffusion kinetics on the other side.

In order to determine possible effect of self-discharge on capacity loss, after 3 formation cycles the cells were charged up to 4.9 V at C/10 and maintained in open-circuit during 168 h (1 week) before a discharge down to 3.5 V (Figure S7), this protocol was repeated five times. Despite the high cut-off voltage, all samples delivered a specific capacity ranging between 110 and 120 mAh.g\(^{-1}\) (85 to 90% of capacity retention). It is important to keep in mind that all electrochemical experiments were carried out with a conventional electrolyte, without any additives (LP30), and that the formulation of the electrodes was not optimized. Despite this, the platelet-like sample not only performed equally to its octahedral-like counterparts but also, all of them showed lower capacity loss during self-discharge tests than those usually obtained for LNMO.\(^{37–39}\)

From results already reported in literature, it appears that LNMO platelet-like samples should deliver in average poor electrochemical performance compared to octahedral-like samples.\(^{19,22}\) The superior electrochemical properties of the latter, especially at high rates and for long-range cycling, and their greater stability when in contact with electrolytes were attributed to the [111] orientation stabilized for all the facets of the octahedral-like particles.\(^{11,15,17,40}\) Our LNMO platelet samples have multiple surface orientations, nevertheless with a larger proportion (~40%) of [111] orientation, it could thus explain the electrochemical performance similar to their octahedral-shaped counterparts. Furthermore, the overall primary particle morphology of the platelets promotes fast diffusion for Li\(^+\) ions due to short distances along their nanometric thicknesses.\(^{41}\)

**CONCLUSIONS**

In conclusion, to the best of our knowledge, this is the first time that stoichiometric and disordered LiNi\(_{0.5}\)Mn\(_{0.5}\)O\(_4\) micrometric platelets, free from rock-salt impurities, have been obtained using molten salt synthesis. TEM imaging and electron diffraction studies revealed that synthesis with a large excess of LiCl salt and a moderate temperature leads to platelet-shaped LiNi\(_{0.5}\)Mn\(_{0.5}\)O\(_4\) that have at least eight different orientations, including [111], [112], [110] and high index planes such as [332]. Despite different orientations, these platelets were found to perform just as well as their octahedral-shaped counterparts. The ability to prepare pure
LiNi₀.₅Mn₁.₅O₄ platelets with nanometric thickness and various facets opens up opportunities for research using spectroscopy and microscopy to investigate the progress of intercalation and deintercalation reactions at the individual particle.

ASSOCIATED CONTENT

Supporting Information

Description of samples preparation, material, and electrochemical characterization methods; XPS analysis of sample 750°C, R=35 before and after extended washing in EtOH; ICP-OES analyses of all prepared samples; Raman spectra and their de-convolution of prepared LNMO samples; Representation of the equivalence between layered hexagonal and cubic spinel structures; Additional SAED patterns showing platelet orientations; Representations of Li⁺ ions diffusion pathways (BVEL) for eight different surface orientations observed on platelets of sample 750°C, R=35; Galvanostatic cycling curves of sample 750°C, R=35 before and after extended washing in EtOH; Self-discharge study on all prepared LNMO samples.

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