# Advanced Energy Materials - 2023 - Song - Li Ni Intermixing The Real Origin of Lattice Oxygen Stability in Co‐Free Ni‐Rich.pdf

Synthesis conditions:

Ni state: Ni is in its +2 oxidation state as it is present in the form of NiO\_{2}

Ni source: Not explicitly mentioned

Ni concentration: Not explicitly mentioned

Base: Not explicitly mentioned

Chelating agent: Not explicitly mentioned

pH: Not explicitly mentioned

Temp: Not explicitly mentioned

Atmosphere: Not explicitly mentioned

Pressure: Not explicitly mentioned

Ni flowrate: Not mentioned in the text

Chelating agent flowrate: Not mentioned in the text

OH flowrate: Not mentioned in the text

Mixing method: Not explicitly mentioned

Form: The synthesized material is in the form of LiNi\_{0.9}Mn\_{0.1}O\_{2} (NM90)

Calcination conditions:

Li source: Not explicitly mentioned

Ni to Li ratio: 9:1 in the composition of NM90

Final temp: Not explicitly mentioned

Ramp rate: Not mentioned in the text

Duration: Not mentioned in the text

Atmosphere: Not explicitly mentioned

Pressure: Not mentioned in the text

Intermediate annealing: Not mentioned in the text

Form: Not mentioned in the text (presumably the same as the synthesized form, NM90)

Observations:

Rock salt structure observed at: Not mentioned in the text

Layered LiNiO\_{2} observed at: Not mentioned in the text

Decomposition observed at: Not mentioned in the text

Intermediate phases: Not mentioned in the text

# Advanced Materials - 2024 - Tayal - In Situ Insights into Cathode Calcination for Predictive Synthesis Kinetic.pdf

Synthesis conditions:

Ni state: Ni(OH)2 (implicit from text)

Ni source: Not specified

Ni concentration: Not specified

Base: Not specified

Chelating agent: Not specified

pH: Not specified

Temp: Not specified during synthesis, but calcination temperature is mentioned later

Atmosphere: Not specified during synthesis, but calcination atmosphere is mentioned later

Pressure: Not specified during synthesis, but calcination pressure is not mentioned

Ni flowrate: Not applicable during synthesis as solid precursor used

Chelating agent flowrate: Not applicable during synthesis as no chelating agent mentioned

OH flowrate: Implicit in Ni(OH)2 precursor

Mixing method: Not specified

Form: Needle-like sub-10 nm-sized particles with a trigonal structure (P-3m1)

Calcination conditions:

Li source: Not specified

Ni to Li ratio: Not specified

Final temp: Varies during the calcination process, mentioned later

Ramp rate: Not specified

Duration: Not specified

Atmosphere: Not specified

Pressure: Not specified

Intermediate annealing: Not mentioned

Form: ∼100 nm-sized layered LiNiO2 cathodes with a hexagonal layered structure (R-3m)

Furnace type: Not specified

Observations:

Rock salt structure observed at: Not mentioned

Layered LiNiO2 observed at: During the calcination process, from needle-like sub-10 nm-sized particles to ∼100 nm-sized layered LiNiO2 cathodes

Decomposition observed at: Implicit in the transformation from Ni(OH)2 to NiO6 (during reconstruction of the basic building units)

Intermediate phases: Ni2+ in Ni(OH)6 octahedra (within hydroxides) to Ni3+ in NiO6 (within oxides)

The text does not provide enough specific information about the exact synthesis conditions for Ni(OH)2 precursor, intermediate phases during calcination, and any intermediates or byproducts formed during the process. The calcination process involves a transformation from needle-like sub-10 nm-sized particles with a trigonal structure to ∼100 nm-sized layered LiNiO2 cathodes, with intermediate phases being Ni2+ in Ni(OH)6 octahedra and Ni3+ in NiO6. The actual conditions for this process, such as temperature, pressure, atmosphere, and the exact duration of each stage, are not explicitly stated in the text.

# An in situ structural study on the synthesis and decomposition of LiNiO2.pdf

Synthesis conditions:

Ni state: Ni3+

Ni source: Ni(OH)2

Ni concentration: Not explicitly mentioned

Base: Not explicitly mentioned

Chelating agent: Not explicitly mentioned

pH: Not explicitly mentioned

Temp: 700 °C

Atmosphere: Oxygen gas

Pressure: Not explicitly mentioned

Ni flowrate: Not explicitly mentioned

Chelating agent flowrate: Not explicitly mentioned

OH flowrate: Not explicitly mentioned

Mixing method: Mixture of Ni(OH)2 and LiOH, heated in an alumina crucible

Form: Powder

Calcination conditions:

Li source: LiOH$H2O (BASF)

Ni to Li ratio: 1 : 1.01 (for the synthesis reactions investigated in this study)

Final temp: 700 °C

Ramp rate: Not explicitly mentioned

Duration: 6 h

Atmosphere: Oxygen gas

Pressure: Not explicitly mentioned

Intermediate annealing: Pre-annealing at 350 °C for 12 h before calcination

Form: Mixture of NiO and LiOH (for the reaction with pre-annealing)

Furnace type: Tubular furnace

Observations:

Rock salt structure observed at: Not explicitly mentioned (reported in TEM studies of LNO and related nickel-rich oxides)

Layered LiNiO2 observed at: During synthesis, with a compressed rhombohedral unit cell (c/a < 4.9) within the rock salt framework

Decomposition observed at: 700 °C and above, proceeds according to LiNiO2 → 1 - zLi1+zNi1+zO2 + zLi2O + 2(1 + z)θO2

Intermediate phases: Not explicitly mentioned in the text, but the pre-annealing process results in a mixture of NiO and LiOH

Decomposition of LNO is believed to mostly affect the surface region of the material.

# d4nr00473f.pdf

Synthesis conditions:

Ni state: Not mentioned, as the paper is about aluminosilicate nanoparticles

Ni source: Not mentioned

Ni concentration: Not mentioned

Base: Hydroxide ions (OH-) from base addition

Chelating agent: Not mentioned

pH: Not specified in the text, but it is implied that hydrolysis ratio and pH could influence the formation of aluminosilicate minerals

Temp: Not specified in the text, but temperature might have been controlled during synthesis as part of experimental conditions

Atmosphere: Not specified in the text

Pressure: Not specified in the text

Ni flowrate: Not applicable

Chelating agent flowrate: Not applicable

OH flowrate: Resulting from base addition

Mixing method: Not specified in the text, but it is implied that the samples were synthesized via sol-gel or hydrolysis reactions

Form: Nanoparticles (imogolite, proto-imogolite, pseudo-boehmite, amorphous silica)

Calcination conditions:

Li source: Not mentioned in the context of calcination

Ni to Li ratio: Not applicable, as there is no mention of Ni in this context

Final temp: Not specified in the text

Ramp rate: Not specified in the text

Duration: Not specified in the text

Atmosphere: Not specified in the text

Pressure: Not specified in the text

Intermediate annealing: Not mentioned in the context of calcination

Form: Not applicable, as there is no mention of Ni or its compounds being calcined

Observations:

Rock salt structure observed at: Not mentioned

Layered LiNiO2 observed at: Not mentioned

Decomposition observed at: Not mentioned

Intermediate phases: Al-(oxy)hydroxides and amorphous silica have been observed during the synthesis of aluminosilicate nanoparticles. Imogolite, proto-imogolite, pseudo-boehmite, and amorphous silica are some of the particles commonly researched.

# hua-et-al-2020-chemical-and-structural-evolution-during-the-synthesis-of-layered-li(ni-co-mn)o2-oxides.pdf

Synthesis conditions:

Ni state: Not explicitly mentioned

Ni source: NiSO4·6H2O (hydroxide coprecipitation method)

Ni concentration: Not explicitly mentioned

Base: Sodium hydroxide (NaOH)

Chelating agent: None explicitly mentioned, ammonia solution is used in the precipitation process

pH: 11.3 ± 0.3

Temp: 52 ± 2 °C during synthesis, 850 °C for heating

Atmosphere: Nitrogen during synthesis, air during heating

Pressure: Not explicitly mentioned

Ni flowrate: Not applicable (synthetic method)

Chelating agent flowrate: Not applicable (synthetic method)

OH flowrate: Amount of LiOH·H2O not specified but 7% excess is used for the synthesis of layered Li(Ni,Co,Mn)O2 oxides

Mixing method: Agitation speed of 500 rpm under a nitrogen atmosphere during synthesis

Form: Not explicitly mentioned in the text

Calcination conditions:

Li source: LiOH·H2O

Ni to Li ratio: Not explicitly mentioned, but a 7% excess of LiOH·H2O is used for the synthesis of layered Li(Ni,Co,Mn)O2 oxides

Final temp: 850 °C

Ramp rate: Not explicitly mentioned

Duration: 12 hours

Atmosphere: Air

Pressure: Not explicitly mentioned

Intermediate annealing: None mentioned in the text

Form: Layered Li(Ni,Co,Mn)O2 oxides

Furnace type: Not explicitly mentioned

Observations:

Rock salt structure observed at: Not applicable (not observed during the high-temperature lithiation reaction)

Layered LiNiO2 observed at: Not applicable (not specifically mentioned in the text)

Decomposition observed at: Not applicable (not specifically mentioned in the text)

Intermediate phases: The generation of layered LTMOs is a consequence of Li/O incorporation into the Li-free precursor obtained by the hydroxide precipitation reaction. However, specific intermediate phases are not explicitly mentioned.

# Monitoring the Formation of Nickel-Poor and Nickel-Rich Oxide Cathode Materials for Lithium-Ion Batteries with Synchrotron Radiation.pdf

Synthesis conditions:

Ni state: Ni1/3Co1/3Mn1/3, Ni0.8Co0.1Mn0.1

Ni source: Nickel sulfate hexahydrate (NiSO4·6H2O), cobalt sulfate heptahydrate (CoSO4·7H2O), manganese sulfate monohydrate (MnSO4·H2O)

Ni concentration: Not specified

Base: Sodium hydroxide solution (4.875 M)

Chelating agent: Not mentioned

pH: Controlled between 10.9 and 11.1

Temp: 60 °C during reaction, not specified during calcination

Atmosphere: Nitrogen (N2) during reaction, oxygen (O2) for NCM811 during calcination, air for NCM111 during calcination

Pressure: Not mentioned

Ni flowrate: Not mentioned

Chelating agent flowrate: Not mentioned

OH flowrate: 12 wt % ammonia solution (0.5 mL/min), 4.875 M sodium hydroxide solution (0.25 mL/min)

Mixing method: Stirring at 1000 rpm

Form: Precipitate

Calcination conditions:

Li source: Lithium hydroxide monohydrate (LiOH·H2O)

Ni to Li ratio: 1:1.03 for NCM811, not specified for NCM111

Final temp: 800 °C

Ramp rate: Not mentioned

Duration: 10 hours for Hold-, 2 hours preannealing at 500 °C followed by 10 hours at 800 °C for Pre-, and a combination of both for the third calcination program

Atmosphere: Oxygen (O2) for NCM811, air for NCM111

Pressure: Not mentioned

Intermediate annealing: Discussed in the Supporting Information (Figure S5)

Form: Not specified

Furnace type: Not mentioned

Observations:

Rock salt structure observed at: Not mentioned

Layered LiNiO2 observed at: Throughout the synthesis for NCM111, not mentioned for NCM811

Decomposition observed at: Not mentioned

Intermediate phases: Not mentioned in the provided text, more information can be found in the Supporting Information

Decomposition observed at: Not mentioned in the provided text

# On the Sensitivity of the Ni-rich Layered Cathode Materials for Li-ion Batteries to the Different Calcination Conditions.pdf

Synthesis conditions:

Ni state: Ni-rich (LiNi0.6Mn0.2Co0.2O2 and LiNiO2)

Ni source: Not specified in the text

Ni concentration: Not specified in the text

Base: Not mentioned in the text

Chelating agent: Not mentioned in the text

pH: Not mentioned in the text

Temp: 750–950 ◦C for NMC622, 650–850 ◦C for LNO

Atmosphere: Not specified in the text

Pressure: Not mentioned in the text

Ni flowrate: Not mentioned in the text

Chelating agent flowrate: Not mentioned in the text

OH flowrate: Not mentioned in the text

Mixing method: Not mentioned in the text

Form: Not mentioned in the text

Calcination conditions:

Li source: Not specified in the text

Ni to Li ratio: Varies depending on the material (0.6 for NMC622, 1 for LNO)

Final temp: 900 ◦C for NMC-900, 700 ◦C for LNO-700 (optimal calcination temperatures mentioned in the text)

Ramp rate: Not mentioned in the text

Duration: Not mentioned in the text

Atmosphere: Not specified in the text

Pressure: Not mentioned in the text

Intermediate annealing: Not mentioned in the text

Form: Powder

Furnace type: Not mentioned in the text

Observations:

Rock salt structure observed at: Not mentioned in the text

Layered LiNiO2 observed at: Not mentioned in the text

Decomposition observed at: Not mentioned in the text

Intermediate phases: Not mentioned in the text

# Phase Transformation Behavior and Stability of LiNiO2 Cathode Material for Li-Ion Batteries from In Situ Pressure and Gas Analysis and Operando X-Ray Diffraction.pdf

Synthesis conditions:

Ni state: Ni rich, not specified concentration

Ni source: Not specified

Ni concentration: Not specified

Base: Not mentioned in the text

Chelating agent: Not mentioned in the text

pH: Not mentioned in the text

Temp: Not mentioned in the text

Atmosphere: Not mentioned in the text

Pressure: Not mentioned in the text

Ni flowrate: Not mentioned in the text

Chelating agent flowrate: Not mentioned in the text

OH flowrate: Not mentioned in the text

Mixing method: Not mentioned in the text

Form: Powder, spherical shape, micrometer-sized particles with a hierarchical structure of secondary particles made of densely packed primary particles of 100-250 nm diameter.

Calcination conditions:

Li source: Not mentioned in the text

Ni to Li ratio: Not specified

Final temp: Not mentioned in the text

Ramp rate: Not mentioned in the text

Duration: Not mentioned in the text

Atmosphere: Not mentioned in the text

Pressure: Not mentioned in the text

Intermediate annealing: Not mentioned in the text

Form: Not mentioned in the text

Furnace type: Not mentioned in the text

Observations:

Rock salt structure observed at: Not mentioned in the text

Layered LiNiO2 observed at: The paper focuses on the study of as-prepared LNO powder, not on calcined samples.

Decomposition observed at: LNO decomposes exothermally at temperatures of 150-200 °C and side reactions leading to formation of spinel (LiNi2O4) and rock-salt-type phases are responsible for this instability.

Intermediate phases: At least four different Li1−xNiO2 phases have been described in the literature, namely the hexagonal H1, monoclinic M, hexagonal H2, and hexagonal H3 phases.

The text does not provide enough information to fill out the template for calcination conditions and observations related to specific intermediate phases or structures observed during cycling operation.

# Process design for calcination of nickel-based cathode materials by in situ characterization and multiscale modeling.pdf

Synthesis conditions:

Ni state: Not explicitly mentioned in the text, but likely Ni(OH)2 or a nickel-containing hydroxide

Ni source: Not explicitly mentioned in the text

Ni concentration: Not explicitly mentioned in the text

Base: Not explicitly mentioned in the text

Chelating agent: Not explicitly mentioned in the text

pH: Not explicitly mentioned in the text

Temp: High temperatures, but no specific value provided

Atmosphere: Controlled atmosphere during calcination, but no specific details provided

Pressure: Not explicitly mentioned in the text

Ni flowrate: Not explicitly mentioned in the text

Chelating agent flowrate: Not explicitly mentioned in the text

OH flowrate: Implied as part of the hydroxide precursors

Mixing method: Not explicitly mentioned in the text

Form: Hydroxides, likely powders or slurries

Calcination conditions:

Li source: Not explicitly mentioned in the text

Ni to Li ratio: Not explicitly mentioned in the text

Final temp: High temperatures, but no specific value provided

Ramp rate: Not explicitly mentioned in the text

Duration: Not explicitly mentioned in the text

Atmosphere: Controlled atmosphere during calcination, but no specific details provided

Pressure: Not explicitly mentioned in the text

Intermediate annealing: Yes, with phase transformation and crystallization of intermediates

Form: Layered oxides

Furnace type: Not explicitly mentioned in the text

Observations:

Rock salt structure observed at: Not explicitly mentioned in the text

Layered LiNiO2 observed at: During calcination process, but no specific time or observation noted

Decomposition observed at: During calcination process, but no specific details provided

Intermediate phases: Yes, transition metal (TM) hydroxides and oxides with layered structures

Intermediate phases are reconstructed during calcination to form layered LiNiO2.

# Tuning Molten-Salt-Mediated Calcination in Promoting Single-Crystal Synthesis of Ni-Rich LiNixMnyCozO2 Cathode Materials.pdf

Synthesis conditions:

Ni state: Ni-rich (x ≥0.8)

Ni source: Not specified in the text

Ni concentration: Not specified in the text

Base: Not mentioned

Chelating agent: Not mentioned

pH: Not mentioned

Temp: Not specified in the text

Atmosphere: Oxygen-rich

Pressure: Not mentioned

Ni flowrate: Not mentioned

Chelating agent flowrate: Not mentioned

OH flowrate: Present in Ni0.85Mn0.05Co0.15(OH)2 precursor

Mixing method: Mixed with a lithium source and a low-melting-point salt (KCl)

Form: Particles (not specified whether they are powders, nanoparticles, etc.)

Calcination conditions:

Li source: Not specified in the text (assumed to be used along with KCl)

Ni to Li ratio: 0.85:1 (from Ni0.85Mn0.05Co0.15(OH)2 precursor)

Final temp: Not specified in the text

Ramp rate: Not mentioned

Duration: Not mentioned

Atmosphere: Oxygen-rich

Pressure: Not mentioned

Intermediate annealing: Not mentioned

Form: Single crystals

Furnace type: Not specified

Observations:

Rock salt structure observed at: Not mentioned

Layered LiNiO2 observed at: Not mentioned (assumed to be the final product of the calcination process)

Decomposition observed at: Not mentioned

Intermediate phases: Ni0.85Mn0.05Co0.15(OH)2 precursor

The text does not provide enough information for a detailed analysis of the synthesis and calcination conditions, intermediate phases, or observations. It focuses on discussing the benefits and potentials of molten-salt-assisted calcination in obtaining well-grown single crystals, but it does not explicitly report specific synthesis or calcination parameters.