Stability of Lithium Aluminate in Reducing and Oxidizing Atmospheres at 700°C

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Abstract

The stability of lithium aluminate (LiAlO_2) powders has been studied under reducing ($4\% \text{ H}_2\text{-N}_2$) and oxidizing (Air) atmospheres at 700°C . While XRD results show the raw α - LiAlO_2 samples contain a minor fraction of $\text{LiAl}_2(\text{OH})_7 \cdot 2\text{H}_2\text{O}$ and $\text{LiAlO}_2 \cdot 0.25\text{H}_2\text{O}$ phases, high-temperature XRD study shows that $\text{LiAl}_2(\text{OH})_7 \cdot 2\text{H}_2\text{O}$ may have decomposed at 700°C to γ - LiAlO_2 and $\text{LiAlO}_2 \cdot 0.25\text{H}_2\text{O}$ to α - LiAlO_2 . Surface morphological studies show the samples consist of porous LiAlO_2 spherical agglomerates with interconnected nanoparticles. The XPS analyses indicate that the powder surface Li/Al ratio decreases after exposure to reducing atmosphere whereas much less change to the oxidizing atmosphere. The binding energy of Al 2p peak also changes in the high-temperature reducing and oxidizing environments, indicating the presence and co-existence of different oxidation states of Al species on the surface. The color of LiAlO_2 powder, examined visually, changed from white to gray after reduction and back to white after re-oxidation, indicating the likelihood of generation of non-stoichiometry under reducing atmosphere.

Keywords

Lithium aluminate; Color change; Crystal structure; Phase change; Surface morphology

1. Introduction

Lithium aluminate (LiAlO_2) has been extensively used for over three decades as a ceramic constituent material of the electrolyte matrix structure in molten carbonate fuel cell (MCFC) stacks for stationary power generation [1, 2]. MCFCs operate at intermediate temperatures in the range of 550-650°C and offer high electrical and combined heat and power (CHP) efficiency (>47% single-cycle electrical and >85% CHP). A wide variety of fuels derived from renewable resources (bio-fuels) and fossil fuels (coal and natural gas) have been successfully utilized in MCFCs. Although 40,000-50,000 hours of operation has been demonstrated at MW class MCFCs, further enhancements in operating life time and cell performance stability are considered important for accelerating large scale commercialization and global market penetration. The porous electrolyte matrix, which acts as an electrolyte retainer, has been considered as one of the most critical stack component impacting cell life and performance; continuous particle coarsening and structural changes could result in reduced capability of electrolyte retention, faster electrolyte loss and increased gas crossover [3]. LiAlO_2 exists in three different allotropic forms: the hexagonal α - LiAlO_2 , tetragonal γ - LiAlO_2 and monoclinic β - LiAlO_2 [4] of which the γ - LiAlO_2 is the stable phase at higher temperatures, while α - and β - LiAlO_2 phases show transformation to the γ - LiAlO_2 at elevated temperatures [5, 6]. For example, at very-high temperatures (e.g., 937 °C), α - LiAlO_2 can transform to γ - LiAlO_2 in air after 40 hours [7]. Particle coarsening and phase transformation of LiAlO_2 have been reported as one important cause for in-cell electrolyte depletion in MCFC [8]. Although γ - LiAlO_2 has initially been considered stable in MCFC, its instability has been revealed to result in the accelerated electrolyte loss and cell failure in long-term cell operation [9]. Since mid-1990s α - LiAlO_2 has been recognized as a more stable matrix support material for current commercial

MCFC deployment [10]. Nevertheless, fundamental understandings of its stability still need to be investigated for helping designing approaches to achieve durability for ≥ 10 -year service life.

LiAlO_2 has also found application as a solid tritium breeding material in nuclear fusion reactors due to its excellent thermochemical and mechanical stability at high temperature and favorable irradiation behavior [1, 2]. Apart from application in nuclear systems, LiAlO_2 has also found application as an excellent luminescent material [11]. $\gamma\text{-LiAlO}_2$ provides an attractive red phosphor for artificial illuminations [12].

A number of synthesis processes including conventional solid-state reactions, sol-gel processing, co-precipitation and combustion synthesis have been extensively used for the preparation of LiAlO_2 powder. Solid-state synthesis in the range of 370°C to 1000°C has been reported to lead to the formation of a mixture of LiOH and a lithium dialuminate, depending on the calcination temperature [13, 14]. The process was found difficult for the synthesis of pure lithium aluminate with controlled size and morphology due to partial evaporation of lithium at higher temperatures and contamination from grinding operations [15]. $\alpha\text{-LiAlO}_2$ has been synthesized using oxide and salt precursors. A mixture of Li_2CO_3 and $\text{Al}(\text{OH})_3$ were dried at 60°C for 24 hours followed by heat-treated at 650°C for 24 hours in a pure CO_2 atmosphere. The final powders were produced by eliminating the residual carbonate by washing [16]. Formation of minor-phase lithium aluminum oxide hydrate, $\text{LiOH} \cdot 2\text{Al}(\text{OH})_3 \cdot 2\text{H}_2\text{O}$ (or $\text{LiAl}_2(\text{OH})_7 \cdot 2\text{H}_2\text{O}$) during powder synthesis was found to transform easily to $\gamma\text{-LiAlO}_2$ during exposure to nitrogen atmosphere at elevated temperatures [17].

Sol-gel technique has been used for preparing $\gamma\text{-LiAlO}_2$ utilizing the hydrolysis of lithium and aluminum alkoxide followed by calcination at temperature of 550°C [17]. The technique showed the limitation in terms of instability of precursors, precise control of reaction steps and

use of expensive reactants. Use of co-precipitation technique provided pure γ -LiAlO₂ in the presence of nonionic surfactant after calcination at 950°C [15]. The combustion synthesis technique using glycine nitrate–urea reactants was also found suitable for synthesizing γ -LiAlO₂ [18, 19].

As mentioned earlier, sufficient LiAlO₂ stability under MCFC operating environment is essential to assure long-term electrolyte retention for stable cell resistance and performance. LiAlO₂ in matrix experiences both reducing anode and oxidizing cathode atmospheres. However, detailed LiAlO₂ structure thermal stability under various gas atmospheres have been reported in the literature only to a limited extent. Discoloration of γ -LiAlO₂ powders have been reported due to the presence of residual amorphous carbon-containing compounds resulting from incomplete oxidation of the glycine-urea by the combustion synthesis technique [20]. Color change from white to dark blue in oxides such as Li₂TiO₃ exposed to reducing conditions has been attributed to oxygen non-stoichiometry and formation of vacancies and compound such as Li_{2-x}TiO_{3-y} [21].

In the present paper, we have investigated the structural and chemical stability of α -LiAlO₂ under reducing and oxidizing atmospheres at 700°C. We further develop fundamental understandings of the effect of crystalline structure, surface composition, and morphology on thermal stability.

2. Experimental

2.1. Materials and Methods

The LiAlO₂ powder samples were prepared by solid-state synthesis method reported by Choi, *et al.* [16]. The precursor materials, Al(OH)₃ (>99.9% pure) and Li₂CO₃ (>99.9% pure), were mixed with deionized water in the different molar ratio to control Li/Al ratio by ball milling. Uniformly dispersed mixtures were subsequently dried at 110°C for 24 hours. The dried powder

cake was calcined at 600-800°C for 12-24 hours in ambient stagnant air. The calcined powders are stored in desiccator. The powder samples A and B are essentially pure α -LiAlO₂ but contain small amounts of minor phases due to variation in the preparation conditions. The experimental set up used for conducting the long-term exposure tests is shown in Fig. 1. High-temperature exposure studies in controlled environments were conducted at 700°C for 100 hours in crucibles made from 99.5% pure alumina. Approximately 1.5 g LiAlO₂ powder, placed in alumina crucibles, was exposed under selected experimental conditions. The gaseous atmospheres used during the experiments are reducing (4% H₂-N₂ (balance)) and oxidizing (compressed air). After the completion of the experiments, powder samples were visually examined. Fig. 2 shows while the color of one batch of powder (sample A) changed from white to gray during the exposure to reducing atmosphere, the powder from another batch (sample B) showed little or no change in the color. The gray-color sample A subsequently changed to white after oxidation treatment in air at 700°C for 50 hours, similar to reported for Li₂TiO₃ [21].

Fig. 1 – Schematic setup for controlled reduction and oxidation of LiAlO₂

Fig. 2 – Color change of LiAlO_2 powders of as-prepared sample A and B (a), sample A after reduction (b), sample A after re-oxidation (c), and sample B after reduction (d).

2.2. Characterization

Lattice structure and compound identification were conducted by X-ray diffraction (XRD) technique at room temperature and high temperature using a Bruker D8 Advance system with Cu K_α radiation ($\lambda = 1.5406 \text{ \AA}$) and an Anton Paar HTK 1200 oven chamber. In order to identify and understand changes in crystal structure, high-temperature XRD studies of LiAlO_2 samples were performed in the temperature range of 25-700°C in reducing atmosphere (4% H_2 - N_2) and oxidizing atmosphere (Air) respectively. LiAlO_2 powders were supported on an alumina sample holder disk for high temperature XRD. A heating and cooling rate of 5°C/min was used during the experiments with a dwell time of 50 hours at 700°C. The data were acquired over an angular range $10^\circ < 2\theta < 70^\circ$ with a scan step of 0.02° and a specific angular range to determine minor secondary phases with a scan step of 0.01° . Surface morphologies were obtained using an FEI Quanta 250 FEG HRSEM with a field emission source and imaged using an Everhart-Thornley SE (secondary electron) detector with an electron accelerating voltage of 10 kV and a sample height of 10 mm.

Nitrogen adsorption analysis was performed with Micromeritics ASAP 2020 at -196°C. Prior to nitrogen adsorption, the powder was degassed for 12 hours at a temperature of 250°C. The surface area of the powder was calculated using the Brunauer-Emmett-Teller (BET) equation [22]. The pore volume and pore diameter were determined from the adsorption data of isotherms using the Barrett-Joyner-Halanda (BJH) method. The X-ray Photoelectron Spectroscopy (XPS) measurement has been performed on a PHI 595 Multiprobe system. The Al K α radiation at 250 W with a pass energy of 100 eV was measured at a scan rate of 1 eV/step to study the surface compositions of the LiAlO₂ powder and with a pass energy of 50 eV at a scan rate of 0.1 eV/step for higher resolution scan. A take-off angle of 45° was used in all sample analyses. Accurate binding energies were determined by placing the C 1s peak at 284.6 eV.

3. Results and Discussion

3.1. Crystal Structure by XRD

The *in-situ* high-temperature XRD patterns of the powder samples are tabulated in Fig. 3. The major XRD peaks match with that of α -LiAlO₂, which is a rhombohedral structure (JCPDS 74-2232) with unit cell parameters of $a = 2.8034 \text{ \AA}$ and $c = 14.228 \text{ \AA}$ and a space group of R-3m. XRD patterns obtained at slow scan rate of 5° per minute (shown in inserts of Fig. 3) show the presence of minor phases of LiAl₂(OH)₇·2H₂O (JCPDS 40-0710) and LiAlO₂·0.25H₂O (JCPDS 21-0487) in the raw powder samples [23, 24]. Although the three minor peaks, observed at 11.7, 23.6 and 36 degree (inserts of Fig. 3), can be assigned to LiAl₂(OH)₇·2H₂O and Li₂Al₄(CO₃)₃·3H₂O (LACHH, JCPDS 37-0185) phases, relative intensities (372, 277 and 273) of the above peaks are found closer to that of LiAl₂(OH)₇·2H₂O.

In-situ high-temperature X-ray diffraction patterns for the above powder samples at 700°C after 50h exposure in 4% H₂-N₂ and air atmospheres showed that the XRD peaks shift to

lower angles indicating lattice expansion [25, 26]. It was also observed that $\text{LiAl}_2(\text{OH})_7 \cdot 2\text{H}_2\text{O}$ disappeared after the completion of the experiments under both reducing or oxidizing atmosphere at 700°C . Only a small amount of $\gamma\text{-LiAlO}_2$ (JCPDS 38-1464) was found to form in the sample A (containing $\text{LiAl}_2(\text{OH})_7 \cdot 2\text{H}_2\text{O}$) in the reducing atmosphere only [23].

Fig. 3 – X-ray diffraction (HT-XRD) patterns of sample A powder under 4% $\text{H}_2\text{-N}_2$ and air atmosphere; (a) room temperature, (b) 700°C for 50 hours under 4% $\text{H}_2\text{-N}_2$, (c) 700°C for 50 hours in air. The inserts show the details of the XRD patterns.

Fig. 4 shows minor peaks of $\text{LiAlO}_2 \cdot 0.25\text{H}_2\text{O}$ in the sample B that also disappeared after 50 hours under the reducing atmosphere at 700°C . The sample showed only the presence of $\alpha\text{-LiAlO}_2$ and unlike the sample A that showed the formation of $\gamma\text{-LiAlO}_2$. The presence of hydrated compounds $\text{LiAlO}_2 \cdot 0.25\text{H}_2\text{O}$ in $\alpha\text{-LiAlO}_2$ has been reported by Pavel, *et al.* [24] who reported that a sample with a Li/Al ratio of 1.0 mainly consist of $\alpha\text{-LiAlO}_2$ with a minor $\text{LiAlO}_2 \cdot 0.25\text{H}_2\text{O}$ phase whereas a sample with lower Li/Al ratio of about 0.4 contains $\alpha\text{-LiAlO}_2$, $\text{LiAl}_2(\text{OH})_7 \cdot 2\text{H}_2\text{O}$ and $\text{LiAlO}_2 \cdot 0.25\text{H}_2\text{O}$. The stability of the powder samples were analyzed by

using the integrated intensity ratio $I_{(003)}/I_{(104)}$ of (003) and (104) peaks indicative of cationic mixing in the lattice and a measure of lattice stability [27, 28]. If $I_{(003)}/I_{(104)}$ is lower than 1.1, the lattice structure is less stable due to the more cationic disorder, which indicates that many Al^{3+} are mixed with Li^+ in 3a sites [29]. The integrated intensity ratio $I_{(003)}/I_{(104)}$ of powder sample A decreased from 0.84 to 0.74 during the reduction treatment, indicating an increase of disorder between Li^+ and Al^{3+} , meaning a high concentration of Al^{3+} ions in the Li interlayer sites. However, after heating under the oxidizing atmosphere, the ratio of $I_{(003)}/I_{(104)}$ increased to 0.98, due to an increase in crystallinity and cationic ordering. The integrated intensity ratio $I_{(003)}/I_{(104)}$ of powder sample B remains essentially at 1.0 regardless of the reduction treatment. This indicates the sample B has a higher cationic ordering and is more resistant to increase in disordering even under the reduction treatment.

Above processes are schematically presented in Fig. 5. The formation of hydrated oxide along with absorbed water on the $\alpha\text{-LiAlO}_2$ and their subsequent dissociation and conversion to $\gamma\text{-LiAlO}_2$ is presented at elevated temperature.

Fig. 4 – High temperature X-ray diffraction (HT-XRD) pattern of sample B powder under 4% $\text{H}_2\text{-N}_2$ atmosphere; room temperature, after 20 hours at 700°C , after 50 hours at 700°C and after cool down to 30°C . The inserts show the details of the XRD patterns.

Fig. 5 – A schematic depicting surface reaction processes on LiAlO_2 powder during treatment in oxidizing and reducing atmospheres.

3.2. Powder Surface Composition by XPS

The surface chemistry of the powder samples (depth 0-10 nm) were determined by XPS (Table 1). The Li/Al ratio on the surface of the sample A is 0.83, indicating Li^+ deficiency on the LiAlO_2 particle surface. The Li deficiency may be attributed to the presence of $\text{LiAl}_2(\text{OH})_7\cdot 2\text{H}_2\text{O}$ (low Li/Al ratio), as confirmed by XRD (Fig. 3). The Li/Al ratio of the surface decreases to 0.66 during the exposure to the reducing atmosphere, suggesting that a large quantity of Li^+ ions located on the surface is removed. The powder also showed grey discoloration. The exposure of powder samples to the oxidizing atmosphere at 700°C , on the other hand, led to an increase in the surface Li/Al ratio to 0.87 without visual color change. In comparison, $\text{LiAlO}_2\cdot 0.25 \text{H}_2\text{O}$ (Li/Al=1/1) has been observed in the sample B (Fig. 4) and may

explain the higher surface Li/Al ratio (0.96), as shown in Table 1. The sample B also showed less decrease of the surface Li/Al ratio during the reduction treatment compared to the sample A. The power remained white. In conclusion, a higher Li/Al ratio may indicate less Li-deficient surface minor phases and/or more stable stoichiometry that have prevented the color change of the power sample B [30].

Table 1. The Li/Al ratio of LiAlO₂ samples of as-prepared sample A, Sample A exposed to 4% H₂-N₂ atmosphere at 700°C, Sample A exposed to air atmosphere at 700°C, as-prepared sample B and sample B exposed to 4% H₂-N₂ atmosphere at 700°C.

Samples	Li/Al ratio by XPS	Intensity ratio
		$I_{(003)} / I_{(104)}$
As-received sample A	0.83 ± 0.02	0.84 ± 0.02
Sample A after reduction	0.66 ± 0.02	0.74 ± 0.01
Sample A after oxidation	0.87 ± 0.03	0.98 ± 0.02
As-received sample B	0.96 ± 0.03	1.02 ± 0.02
Sample B after reduction	0.85 ± 0.03	1.01 ± 0.02

3.3. High resolution XPS Al 2p spectra

Binding energy (BE) shift of the Al 2p peaks for the sample A after the reduction and oxidation treatments is shown Fig. 6. For the as-prepared sample A, the spectrum was deconvoluted into two peaks at 74.3 and 72.9 eV. The higher peak at 74.3 eV was assigned to the formation of Al-O bond [31]. The energy value of lower peak was 72.9, which is 0.5 eV higher than that of metallic Al. The binding energy of a small metal cluster was reported to shift to higher value than the bulk metal [32, 33, 34]. After the reduction treatment at 700°C, a shift of

the binding energy of Al 2p to 73.9 [35] and 72.8 eV [36] was observed. The intensity of Al-O decreased and that of metallic Al considerably increased after the exposure to the reducing atmosphere at 700°C. Similar observation has been reported for Rh/Al₂O₃ catalyst treated in a reducing condition by M. Zimowska *et al.* [37]. After the oxidation treatment, an additional asymmetric component appeared at 75.5 and 76.9 eV while the peaks of metallic Al decreased. This high BE peaks at 75.5 and 76.9 eV are assigned to fully-coordinated and under-coordinated Al³⁺ in Al₂O₃ [38, 39]. The oxidation of the metallic aluminum is confirmed by the decrease in Al-Al bond around 73 eV [40]. This indicates that metallic aluminum shifted toward higher binding energy due to the oxidation of the surface enriched metallic aluminum atoms while exposure to the oxidizing air atmosphere [41].

Fig. 6 – High resolution XPS Al 2p spectra of samples A as-prepared (a), after reduction (b), and after oxidation (c).

3.4. Morphologies by SEM

A typical low-magnification surface morphology of the LiAlO_2 (sample A) powders obtained by FESEM is shown in Fig. 7 (a). The powder morphology shows the presence of dispersed microspheres of $\sim 20 \mu\text{m}$ diameter with very porous surface while powder sample B displays similar micro but relatively less porous spherical morphology as shown in Fig. 7 (b). Fig.

8 shows the high-magnification surface morphology of the samples before and after the reduction/oxidation treatments at 700°C for 100 hours. For the sample A, it is observed that microspheres consist of agglomerated nanoparticles of spherical and plate-like loosely interconnected particles generating nano- and micron-size pores. The above observation is consistent with the findings of H. Xiao *et al.* [42] who reported that porous LaFeO_3 microspheres exhibit much higher response to reducing gas than denser LaFeO_3 particles. The sample B powder in Fig. 8 (d) shows that the microsphere is also assembled from a large amount of interconnected uniform nanoparticles with only micron-sized pores.

Fig. 7 – Low-magnification FESEM images of the LiAlO_2 Sample A (a) and Sample B (b)

Fig. 8 – High-magnification FESEM images of the LiAlO_2 samples. (a) as-prepared sample A, (b) sample A after reduction, (c) sample A after oxidation, (d) as-prepared sample B and (e) sample B after reduction.

3.5. Surface Area and Pore Size Distribution

Nitrogen adsorption-desorption measurements were carried out in order to study the surface area and pore-size distribution of the powder samples exposed to both reducing and oxidizing atmospheres. N_2 adsorption isotherms of the powder sample A and B as prepared and after reduction are shown in Fig. 9 (a-d). All samples exhibited a typical type II isotherm, characteristic of micro- and mesoporous materials [43]. The adsorbed amount of N_2 decreased from $95.9 \text{ cm}^3/\text{g}$ on the sample A to $68.9 \text{ cm}^3/\text{g}$ (a decrease of 28.2%) after the reduction treatment. The sample B (before and after reduction) shows that the adsorbed amount of N_2 decreased from $87.2 \text{ cm}^3/\text{g}$ to $77.6 \text{ cm}^3/\text{g}$ (a decrease of 11.0%), indicating that adsorption is easier on the as-prepared powders. Adsorption and surface coverage is also favored on sample A which shows a more porous structure, when compared with the sample B that contains more compact interconnected structure.

Pore size distributions obtained by the BJH method as shown in Fig. 10 indicate the presence of pores in the size range of $25 \text{ \AA}$ in both sample A and B. The pore volume increases and widens after the high temperature heat treatment. While the pore volume below $25 \text{ \AA}$ of pore diameter slightly increases in sample A, such pores and pore volume remain unchanged in sample B before and after reduction.

Specific surface area of the sample A and B were calculated according to nitrogen adsorption measurements. Table 2 summarizes the nitrogen sorption data of the sample A and B.

The sample A has a sum of meso- and micro- pores' volume equal to 0.006 cm³/g. After the reduction treatment, the pore volume increases to 0.007 cm³/g due to the removal of LiAl₂(OH)₇·2H₂O and formation of γ -LiAlO₂, as shown in Fig.3. The surface area increases by 6.7%, which is also a result of increase in the meso- and micro-pore volume. The sample B showed only slight change in the surface area (0.6%) and the pore volume (both 0.005 cm³/g) after the reduction treatment. Stable behavior of the powder is considered to be related to the presence of denser surface and higher Li/Al integrated ratio. The presence of the hydrated oxides, crystalline water, absorbed water, and Li/Al ratio all influences the mesopores, specific surface area pore volume as well as phase stability at elevated temperatures.

Fig. 9 – Adsorption isotherm patterns of the LiAlO₂ powders: (a) as-prepared sample A powder, (b) sample A after reduction, (c) as-prepared sample B, and (d) B1 powder, and (d) sample B after reduction.

Fig. 10 – BJH pore size distribution of the LiAlO_2 powders: (a) as-prepared sample A powder, (b) sample A after reduction, (c) as-prepared sample B, and (d) sample B after reduction.

Table 2. BET surface area and micro- and meso-pore volume of LiAlO_2 samples of as-prepared sample A, Sample A exposed to 4% $\text{H}_2\text{-N}_2$ atmosphere at 700°C , as-prepared sample B and sample B exposed to 4% $\text{H}_2\text{-N}_2$ atmosphere at 700°C .

Samples	BET Surface Area	V micro + V meso
	Change (%)	(cm^3/g)
As-prepared sample A*	100%	0.0056 ± 0.0001
Sample A after reduction	+ 6.7 %	0.0066 ± 0.0001
As-prepared sample B*	100 %	0.0048 ± 0.0001
Sample B after reduction	-0.6 %	0.0050 ± 0.0001

Note: * BET surface area is $15\text{-}25 \text{ m}^2/\text{g}$

4. Conclusions

The XRD analysis show that the as-prepared $\alpha\text{-LiAlO}_2$ samples contain small amounts of $\text{LiAl}_2(\text{OH})_7 \cdot 2\text{H}_2\text{O}$ phase in the sample A and $\text{LiAlO}_2 \cdot 0.25\text{H}_2\text{O}$ phase in the sample B. The

XPS surface analysis of the α -LiAlO₂ powder shows Li-deficiency exists on the surface and becomes more deficient upon treatment in the reducing atmosphere. After exposure to the reducing atmosphere, LiAl₂(OH)₇·2H₂O peak disappeared followed by the formation of γ -LiAlO₂ phase. A decrease in the integrated intensity ratio $I_{(003)}/I_{(104)}$ of (003) and (104) peaks from 0.84 to 0.74 for the sample A after exposure to the reducing atmosphere indicates a higher degree of the cationic disorder and relatively poor structural stability under reducing environment. On the other hand, the sample B showed relatively less $I_{(003)}/I_{(104)}$ of (003) and (104) peaks and Li/Al ratio changes, indicating less cationic disorder and hence improved structural stability. Apparently a reducing environment may promote cationic disorder and surface non-stoichiometry that reduce powder structural thermal stability. The above inference may be supported by the powder color change under a reducing atmosphere. The presence of hydrated oxide phases LiAl₂(OH)₇·2H₂O and LiAlO₂·0.25H₂O, crystalline water, and Li/Al ratio influences the thermal stability during exposure to elevated temperatures. To fully understand the LiAlO₂ under MCFC environments, further investigation in the presence of electrolyte needs to be conducted.

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