

# Ultra-low corrosiveness molten LiCl-KCl-Li salt solution with dissolved metallic Li component

Jiehong Liang, Hongliang Bao and Junheng Ying Su

State Key Laboratory of Thorium Energy, Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai, China, and  
University of Chinese Academy of Sciences, Beijing, China

Ling Han

State Key Laboratory of Thorium Energy, Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai, China

Jingyuan Ma

Shanghai Synchrotron Radiation Facility, Shanghai Advanced Research Institute, Chinese Academy of Sciences, Shanghai, China

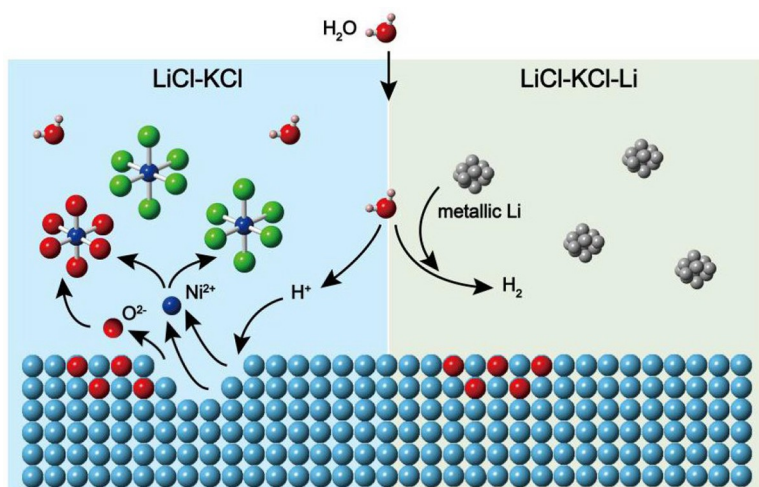
Min Ge and Hongtao Liu

State Key Laboratory of Thorium Energy, Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai, China, and

Jian Yao and Yuan Qian

State Key Laboratory of Thorium Energy, Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai, China, and  
University of Chinese Academy of Sciences, Beijing, China

## Graphical abstract



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## Abstract

**Purpose** – Chloride salts are highly hygroscopic, which can lead to severe corrosion of metallic materials at elevated temperatures, greatly limiting their practical applications. This paper aims to evaluate the impact of impurity moisture on the metal corrosiveness of molten salt and achieve complete removal of impurity moisture.

**Design/methodology/approach** – Here, the authors choose the dissolution corrosion of nickel metal in LiCl-KCl eutectic salt as a research model. The authors prepared LiCl-KCl eutectic salt and further synthesized molten LiCl-KCl-Li salt solution containing dissolved metallic Li component using it as raw material. The static corrosion test of nickel in molten LiCl-KCl-Li and LiCl-KCl was applied to evaluate their corrosiveness.

**Findings** – The Ni sheet in LiCl-KCl undergoes obvious dissolution corrosion, and the generated  $\text{Ni}^{2+}$  ions mainly exist in the form of  $\text{K}[\text{NiCl}_3]$  complex with a small amount of dissolved NiO. In contrast, LiCl-KCl-Li solution shows ultra-low corrosiveness toward metal Ni, and no significant Ni content was detected in the LiCl-KCl-Li sample that reacted with a Ni sheet at  $600^\circ\text{C}$  for 7 days.

**Originality/value** – Through near-ideal controlled experiments, the study deeply explored the impact of residual moisture in chlorine salts after heat treatment on metal corrosion. The research results reveal that the impurity moisture in LiCl-KCl plays a crucial role in the Ni corrosion, and can be completely removed by using an excess of metallic Li, resulting in a molten-salt solution with almost no corrosivity.

**Keywords** Molten salts, Molten-purification, Corrosion inhibition, Lithium metal, Metal corrosion

**Paper type** Research paper

## 1. Introduction

Molten chloride salts, such as LiCl-KCl eutectic salt, have important applications in various fields, including molten salt electrolysis and electroplating (Choi *et al.*, 2024; Han *et al.*, 2021; Xi *et al.*, 2024), heat transfer and thermal energy storage (Ding and Bauer, 2021; Rong *et al.*, 2024), battery electrolytes (Cui *et al.*, 2022; Zhou *et al.*, 2023), nuclear reactors (Cisneros *et al.*, 2024), spent fuel reprocessing (Liu *et al.*, 2023a, 2023b; Mullabaev *et al.*, 2018; Salyulev *et al.*, 2017), materials synthesis (Li *et al.*, 2022; Shen *et al.*, 2021; Yang *et al.*, 2024), etc. However, high-temperature molten salts are corrosive to metal materials. Especially, chloride salts are highly hygroscopic and can undergo hydrolysis to produce highly corrosive acids at high temperatures (Ding *et al.*, 2018; Guo *et al.*, 2018). The corrosiveness of molten salts is a problem that must be considered in their practical applications. On the one hand, the practical application of molten salts can be met to a certain extent by improving the corrosion resistance of metal materials or developing corrosion-resistant alloys (Wang *et al.*, 2022). Nickel metal or nickel-based alloys have better corrosion resistance than stainless steel and are an important type of structural material for high-temperature molten salts (Shankar *et al.*, 2013; Sun *et al.*, 2018, 2018b; Sun *et al.*, 2018a, 2018b). On the other hand, more and more researches have focused on the corrosiveness from the perspective of molten-salt and emphasized the importance of maintaining salt purity and redox potential (Ong *et al.*, 2020; Zhang *et al.*, 2018). Different purification methods have been used to reduce the corrosiveness of molten chloride salts, such as reducing the moisture content of chloride salts through heating, purifying LiCl-KCl by injecting  $\text{Cl}_2$ , and adding additional substances as corrosion inhibitors (Ding *et al.*, 2019a, 2019b; Huang *et al.*, 2011; Ravi Shankar and Kamachi Mudali, 2017). Heating is a simple and commonly used method to directly reduce the moisture content of chloride salts, but it is difficult to completely remove moisture (Huang *et al.*, 2011). The use of chlorine gas will bring additional corrosiveness (Ravi Shankar and Kamachi Mudali, 2017). Adding corrosion inhibitors to deeply purify molten salts is a flexible and efficient method, but it is necessary to consider the additional impact of corrosion inhibitors on the parent salts.

Adding reactive metals to molten salts is one effective method for removing moisture. For example, excess Be was

used to maintain reducing conditions in the FLiBe salt during the Molten Salt Reactor Experiment of Oak Ridge National Lab (Jiang *et al.*, 2022). Similarly, the addition of lithium as a reducing agent effectively controls the redox potential of molten LiF-NaF-KF, significantly reducing corrosion in Hastelloy N and 316H stainless steel (Sankar and Singh, 2021). Correspondingly, Ding *et al.* (Ding *et al.*, 2019a, 2019b) showed that the introduction of excess Mg resulted in prevention of corrosion of structural materials in  $\text{MgCl}_2$ -NaCl-KCl eutectic salt. However, the use of active metals for removing moisture contained in LiCl-KCl has rarely been reported. Recent researches have found that metallic lithium can dissolve in molten LiCl salt in the form of  $\text{Li}_n$  clusters to form LiCl-Li solution (Guo *et al.*, 2019; Merwin *et al.*, 2016). Our previous research has shown that other metals such as copper can also dissolve in LiCl-KCl in the form of  $\text{Cu}_n$  clusters (Liu *et al.*, 2023a, 2023b). These findings indicate that metal Li may also dissolve in molten LiCl-KCl salt in metallic form to form LiCl-KCl-Li solution. Due to the excellent moisture removal effect of metallic Li, such LiCl-KCl-Li solution is expected to exhibit extremely low corrosiveness.

In this paper, we first prepared LiCl-KCl eutectic salt, and then used it and lithium sheet as raw materials to further successfully synthesize molten LiCl-KCl-Li salt solution with dissolved metallic Li component. Their corrosiveness to metal was evaluated by the static corrosion test of nickel sheet in the LiCl-KCl and LiCl-KCl-Li. Because the chemical form of corrosion product ions is not only closely related to the corrosion mechanism, but may also have an impact on subsequent corrosion behavior, we also investigated the valence state and coordination structure of Ni in LiCl-KCl after the reaction by using X-ray absorption fine structure (XAFS) and ultraviolet-visible (UV-vis) spectra. Finally, we explored the corrosion mechanism of Ni in LiCl-KCl and the reasons for the ultra-low corrosiveness of LiCl-KCl-Li.

## 2. Experiments and methods

### 2.1 Sample preparation

LiCl (99.99%, Sigma Aldrich) and KCl (99.99%, Sigma Aldrich) were subjected to vacuum drying at  $50^\circ\text{C}$  for over 24 h to eliminate

the free water. Subsequently, a mixture of LiCl and KCl salt (44.5:55.5 Wt.%) was placed in a quartz crucible and transferred to an electric furnace located in a glove box ( $\text{H}_2\text{O} < 0.1 \text{ ppm}$ ,  $\text{O}_2 < 0.1 \text{ ppm}$ ) under an argon atmosphere. The mixture was then heated to  $150^\circ\text{C}$  at a rate of  $5^\circ\text{C}/\text{min}$  and kept at this temperature for over 24 h to remove water in the salt. The mixture was then further heated to  $600^\circ\text{C}$  and maintained at this temperature for at least 24 h to remove the most volatile impurities from the molten salt. The processed mixture was poured into a quartz crucible to cool to obtain solid LiCl-KCl eutectic salt.

Approximately 0.2 g of lithium metal (Li, 99.9%, Sinopharm Chemical Reagent Co., Ltd) was added to a BN crucible containing about 20 g of the prepared LiCl-KCl eutectic salt. The crucible was then heated to  $600^\circ\text{C}$  and held at this temperature for more than 24 h to obtain a mixture. After removing undissolved Li metal particles from the cooled mixture, a solid LiCl-KCl-Li<sub>600</sub> sample was obtained. The LiCl-KCl-Li<sub>600</sub> solution can be further obtained after heating and melting the solid sample. Similarly, LiCl-KCl-Li<sub>500</sub> sample was also prepared at  $500^\circ\text{C}$ .

## 2.2 Static corrosion test

A polished nickel sheet (Ni, 99.5%,  $2 \text{ cm} \times 1 \text{ cm} \times 0.5 \text{ cm}$ , Qinghe Jinou metal material Co., LTD) added to a BN crucible containing about 20 g of LiCl-KCl-Li<sub>600</sub>, and the crucible was heated to  $600^\circ\text{C}$  and kept at that temperature for 7 days. During the reaction process, we collected quenched salt samples by using BN threaded tubes on the first and seventh days, respectively. The two quenched salt samples were both named LiCl-KCl-Li-Ni<sub>600</sub>. Similarly, we also collected the quenched salt samples for Ni reacting with LiCl-KCl-Li<sub>500</sub> at  $500^\circ\text{C}$  and named both samples LiCl-KCl-Li-Ni<sub>500</sub>. As a comparison, the corresponding quenched salts for Ni and LiCl-KCl after reaction at  $500^\circ\text{C}$  and  $600^\circ\text{C}$  were also collected and named LiCl-KCl-Ni<sub>500</sub> and LiCl-KCl-Ni<sub>600</sub>, respectively.

## 2.3 Characterization methods

**UV-vis:** Liquid LiCl-KCl-Ni<sub>500</sub>, LiCl-KCl-Ni<sub>600</sub>, LiCl-KCl-Li-Ni<sub>500</sub> and LiCl-KCl-Li-Ni<sub>600</sub> samples were analyzed by high-temperature UV-vis spectroscopy which was designed and verified by our group (Liu et al., 2020). Its apparatus is shown in Fig. S1 (Supplementary Information). The light beam was from a deuterium tungsten lamp (ideaoptics, iDH2000), which was guided through the middle of the sample cell by a set of optical lens, and the transmitted light was focused into a spectrometer (from Ocean Optics Co., QE Pro 65) to obtain the absorption spectra. About 2.0 g of sample was transferred into a quartz sample cell (light path of 10 mm) in an electric furnace that was placed in a glove box, and heated to corresponding reaction temperature and maintained at this temperature.

**Gas Chromatography (GC):** Approximately 1 g of sample was added into a 20 ml glass bottle with a rubber stopper in a glove box. Approximately 5 mL of deionized water was injected into the bottle by using a syringe. We shook the bottle to dissolve the sample. Next, we collected the upper layer of gas inside the bottle using a syringe and inject it into a GC for testing.

**Inductively coupled plasma atomic emission spectrometer (ICP-OES):** Nickel concentration of samples is measured by ICP-OES (Spectro Arcos, Germany ametek Co., LTD). Approximately 0.2 g of sample was added in 50 ml of 2 wt% dilute nitric acid. The nitric acid solution was placed in a water bath simultaneously

and kept the temperature at  $60^\circ\text{C}$  for 24 h to ensure complete dissolution of the sample. Each sample was measured twice and the average value was taken as the testing result.

**Scanning electron microscope (SEM):** The reacted nickel sheet was removed from the crucible. The surface of the nickel sheet was then cleaned with deionized water, followed by drying. A small sample of the nickel sheet was subsequently sectioned using a electric saw for SEM characterization. The morphology of the sample was analyzed using a Zeiss Merlin Compact LEO 1530 VP electron scanning microscope (Carl Zeiss, Inc., Germany). The elemental composition of the samples was characterized using the energy dispersive spectroscopy (EDS, Aztec X-Max 50). It should be noted that the samples used for cross-sectional analysis were abraded and polished.

**XRD:** Solid salts were grinded and loaded into sample stages in the glove box. The samples were sealed with Kapton film to prevent contact with air. X-ray diffraction data was measured on Bruker D8 ADVANCE with Cu-K $\alpha$  (1.5406 Å) radiation (40 kV, 40 mA). All samples were mounted on the same sample holder with the scanning range from  $20^\circ$  to  $90^\circ$  at the sweep speed of  $5^\circ \text{ min}^{-1}$ .

**XAFS:** XAFS spectra at the Ni K-edge were recorded on beamline 20U1 at the Shanghai Synchrotron Radiation Facility (SSRF). The electron beam energy was 3.5 GeV with a stored current of approximately 200 mA in top-up operation. A fixed-exit double crystal Si <111> monochromator was used for the incident energy selection. The XAFS data including the X-ray absorption near-edge structure (XANES) and extended X-ray absorption fine structure (EXAFS) were collected in fluorescence mode by applying a 4-element silicon drift detector. Ni foil, NiO and NiCl<sub>2</sub> powder were also measured under similar condition for comparison.

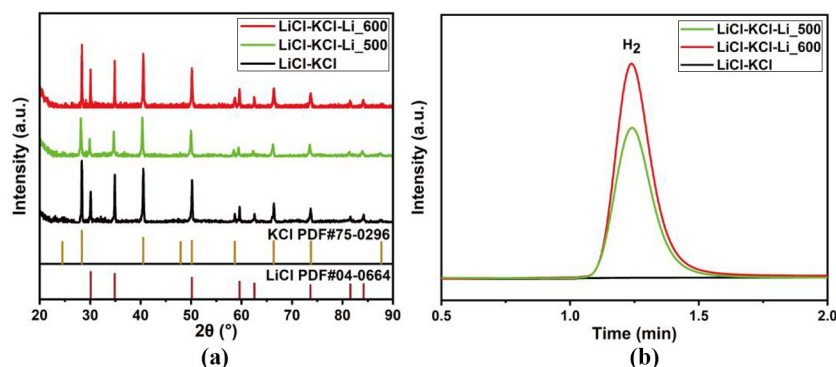
Standard procedures were followed to analyze the XAFS data using the software package Demeter (Ravel and Newville, 2005). The backscattering amplitude and phase shift were calculated with the program FEFF9 (Rehr et al., 2010). The Fourier transformation of the  $k^3$ -weighted EXAFS oscillations from  $k$ -space to  $r$ -space was performed over a range of  $2.8\text{--}11.5 \text{ \AA}^{-1}$ . The value of amplitude reduction factor  $S_0^2$  was fixed at 0.9. Other structural parameters, such as coordination numbers ( $N$ ), bond distance ( $R$ ), Debye-Waller factor ( $\sigma^2$ ) and inner potential shift ( $\Delta E_0$ ), were obtained from the fitting.

**Infrared (IR):** The IR spectra of samples were measured by the Fourier Transform Infrared Spectroscopy (Perkin Elmer spectrometer). Each sample and KBr were thoroughly ground and pressed into a plate of 13 mm diameter. The plate then placed into a slide holder under argon atmosphere. IR spectra were recorded in the transmission mode with a  $2 \text{ cm}^{-1}$  resolution between 4000 and  $1000 \text{ cm}^{-1}$ . Each spectrum was averaged by 32 scans.

## 3. Results and discussion

### 3.1 Preparation and characterization of LiCl-KCl-Li

As shown in Figure 1, we synthesized liquid LiCl-KCl eutectic salt. The colorless and transparent LiCl-KCl was cooled to obtain a solid sample. Powder XRD profile of the solid LiCl-KCl sample is shown in Figure 2(a). All diffraction peaks of the LiCl-KCl can be assigned to the signals of LiCl and KCl. No other components were detected in the XRD pattern, which is consistent with the fact that Li<sup>+</sup> and K<sup>+</sup> ions do not tend to form

**Figure 1** Preparation flow chart for LiCl-KCl and LiCl-KCl-Li samples**Figure 2** (a) XRD patterns of solid LiCl-KCl, LiCl-KCl-Li\_500 and LiCl-KCl-Li\_600 samples; (b) GC data for the gas from the reaction between solid LiCl-KCl, LiCl-KCl-Li\_500 and LiCl-KCl-Li\_600 with deionized water, respectively

complexes. The experimental results indicate that we have successfully prepared high-purity LiCl-KCl molten salt.

As shown in Figure 1, the solid LiCl-KCl-Li samples were obtained by removing undissolved Li metal particles from the cooled mixture. As shown in Figure 2(a), the XRD patterns of solid LiCl-KCl-Li\_500 and LiCl-KCl-Li\_600 have no signals of metal Li and lithium compounds, except for diffraction peaks similar to the LiCl-KCl. Similar to molten LiCl-KCl salt, liquid LiCl-KCl-Li\_500 and LiCl-KCl-Li\_600 are both transparent. Their UV-vis spectra have no absorption in the range of 300 nm to 850 nm (Fig. S2, Supplementary Information). These results indicate that metal Li has dissolved in molten LiCl-KCl salt to form a LiCl-KCl-Li solution. We used a GC to detect the gas components generated by the reaction between solid LiCl-KCl-Li and deionized water. The gas chromatography data of LiCl-KCl-Li\_500 and LiCl-KCl-Li\_600 are shown in Figure 2(b). Compared with LiCl-KCl, both LiCl-KCl-Li\_500 and LiCl-KCl-Li\_600 exhibit a strong peak around 1.25 min corresponding to the position of H<sub>2</sub> gas. These results suggest that a portion of Li metal can dissolve into LiCl-KCl molten salt as a metallic Li component to form a LiCl-KCl-Li solution.

As it is well-known, simply heating not only makes it difficult to completely remove moisture from chloride salts, but also causes hydrolysis of chloride salts containing moisture at high temperatures. For LiCl-KCl containing moisture impurities, hydrolysis of LiCl produces Li<sub>2</sub>O and HCl (Gao et al., 2025; Wang et al., 2025). HCl has strong corrosiveness to metals at high temperatures, and can dissolve metal Ni and its surface oxide NiO to form NiCl<sub>2</sub> (Horvath and Simpson, 2018). Therefore, metal Ni can be used as a metal model for evaluating the corrosiveness of molten salts, and the corrosiveness can be reflected by detecting the Ni content of the reacted molten salts.

The obtained LiCl-KCl-Li samples in this article, including LiCl-KCl-Li\_500 and LiCl-KCl-Li\_600, have dissolved metallic Li components which may exist in the form of Li<sub>n</sub> clusters (Fan et al., 2013). The occurrence of metallic Li component reveals that impurities such as H<sub>2</sub>O and HCl in LiCl-KCl molten salt have completely reacted with Li. Therefore, liquid LiCl-KCl-Li can be regarded as a deeply purified molten LiCl-KCl salt solution, and is expected to have low corrosiveness. Moreover, the dissolved metallic Li in LiCl-KCl-Li does not react with nickel ions or metal Ni, so it does not interfere with the content of

Ni element in the molten salt. Therefore, the Ni content of the molten salt after reacting with metallic nickel can be used to evaluate the corrosiveness of the molten salt.

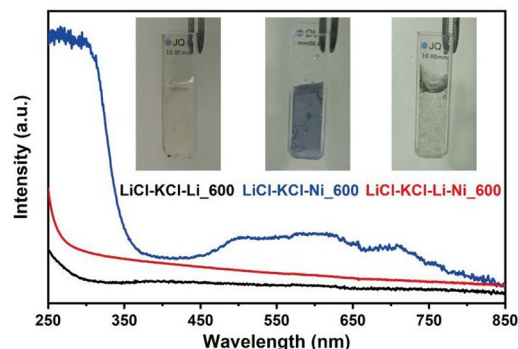
### 3.2 Low corrosiveness of LiCl-KCl-Li

We chose the static corrosion test of nickel in LiCl-KCl-Li samples as the molten-salt evaluation model, and measured the Ni content in the samples after the reaction to evaluate their corrosiveness. A nickel metal sheet polished with sandpaper was added to a BN crucible containing about 20 g of LiCl-KCl-Li<sub>600</sub>. After heating the crucible to 600°C and holding it for 1 day and 7 days, we collected corresponding quenched salt samples using BN threaded tubes and named both samples LiCl-KCl-Li-Ni<sub>600</sub>. Similarly, we also collected the quenched salt samples for Ni reacting with LiCl-KCl-Li<sub>500</sub> at 500°C and named both samples LiCl-KCl-Li-Ni<sub>500</sub>. As a comparison, the corresponding quenched salts for Ni and LiCl-KCl after reaction at 500°C and 600°C were also collected and named LiCl-KCl-Ni<sub>500</sub> and LiCl-KCl-Ni<sub>600</sub>, respectively.

The LiCl-KCl-Li-Ni<sub>500</sub> and LiCl-KCl-Li-Ni<sub>600</sub> samples are white, and the representative LiCl-KCl-Li-Ni<sub>600</sub> sample collected on the seventh day is shown in Figure 3(a). The LiCl-KCl-Ni<sub>500</sub> and LiCl-KCl-Ni<sub>600</sub> samples appears light blue, and the sample LiCl-KCl-Ni<sub>600</sub> collected on the seventh day, as a representative, is shown in Figure 3(b). The high temperature UV-vis spectrum of the LiCl-KCl-Ni<sub>600</sub> collected on the seventh day is shown in Figure 4. The liquid LiCl-KCl-Ni<sub>600</sub> is a blue purple solution and exhibits absorption in both visible and near ultraviolet regions. There is a strong absorption band centered at 270 nm in the ultraviolet region. In the visible light region, the LiCl-KCl-Ni<sub>600</sub> displays a weak broad absorption with three distinguishable bands centered at 510 nm, 600 nm and 710 nm. These absorption bands are typical characteristics of Ni<sup>2+</sup> in molten LiCl-KCl eutectic salt and are in good agreement with other literature report (Harrington and Sundheim, 2006). These findings suggest that the nickel sheet have undergone dissolution corrosion in LiCl-KCl molten salt. In contrast, LiCl-KCl-Li-Ni<sub>600</sub>, similar to LiCl-KCl-Li<sub>600</sub>, is transparent and does not exhibit absorption in both visible and near ultraviolet regions. The phenomenon indicates that the LiCl-KCl-Li samples have low corrosiveness and the nickel sheet did not undergo visible dissolution corrosion.

To further evaluate the difference in corrosiveness between LiCl-KCl-Li and LiCl-KCl, we detected the Ni content of the corresponding representative samples by using ICP-OES. The Ni content in LiCl-KCl-Ni<sub>600</sub> and LiCl-KCl-Li-Ni<sub>600</sub> samples are shown in Table S1 (Supplementary Information). The Ni

**Figure 4** UV-vis spectra of liquid LiCl-KCl-Li<sub>600</sub> (black), LiCl-KCl-Li-Ni<sub>600</sub> (red) and LiCl-KCl-Ni<sub>600</sub> (blue) at 600°C with corresponding physical photos



content in the LiCl-KCl-Ni<sub>600</sub> samples collected on the first and seventh day was determined to be approximately 1 ppm and approximately 151 ppm, respectively. In contrast, no Ni content above the ICP-OES detection limit was detected in the LiCl-KCl-Li-Ni<sub>600</sub> samples (Table S1) collected on the first and seventh day. These results reveal that the LiCl-KCl has obvious corrosiveness toward Ni, while the LiCl-KCl-Li shows ultra-low corrosiveness to Ni.

### 3.3 Origin of low corrosiveness of LiCl-KCl-Li

To explore the reason for the ultra-low corrosiveness of LiCl-KCl-Li, we studied the corrosion mechanism of Ni in LiCl-KCl molten salt. To better understand the corrosion mechanism of nickel in LiCl-KCl, we investigated the chemical form of Ni in LiCl-KCl-Ni<sub>600</sub>. XRD data of the LiCl-KCl-Li-Ni<sub>600</sub> and LiCl-KCl-Ni<sub>600</sub> samples collected on the seventh day is shown in Figure 5(a). Similar to LiCl-KCl, no other components were observed in their patterns except for the signals of LiCl and KCl. To explore the local structure of Ni in solid LiCl-KCl-Ni<sub>600</sub>, we implemented XAFS measurements for the LiCl-KCl-Ni<sub>600</sub> sample collected on the seventh day. The Ni K-edge XANES data for Ni foil, NiCl<sub>2</sub>, NiO and LiCl-KCl-Ni<sub>600</sub> are displayed in Figure 5(b). Ni foil has absorption edge at approximately 8332 eV and exhibits distinct absorption peaks at approximately 8334 eV, approximately 8348 eV and approximately 8357 eV, respectively. These features are attributed to the electric dipole 1s→4p transitions (Nakai et al., 2001). The absorption edge positions of both NiO and NiCl<sub>2</sub> shift to high energy direction compared with Ni foil. There is a strong main edge crest corresponding to 1s→4p absorption in the XANES spectra of both NiCl<sub>2</sub> and NiO. In addition, they show a

**Figure 3** (a) Physical photo of the LiCl-KCl-Li-Ni<sub>600</sub> sample collected on the seventh day; (b) Physical photo of the LiCl-KCl-Ni<sub>600</sub> sample collected on the seventh day; Inset is the corresponding nickel metal sheet that has been cleaned after the reaction

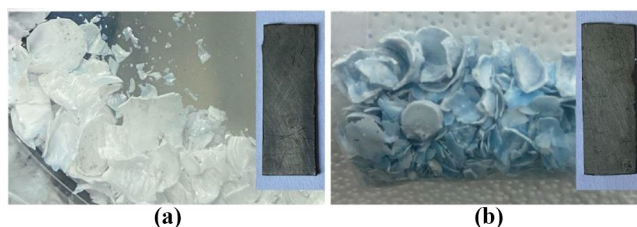
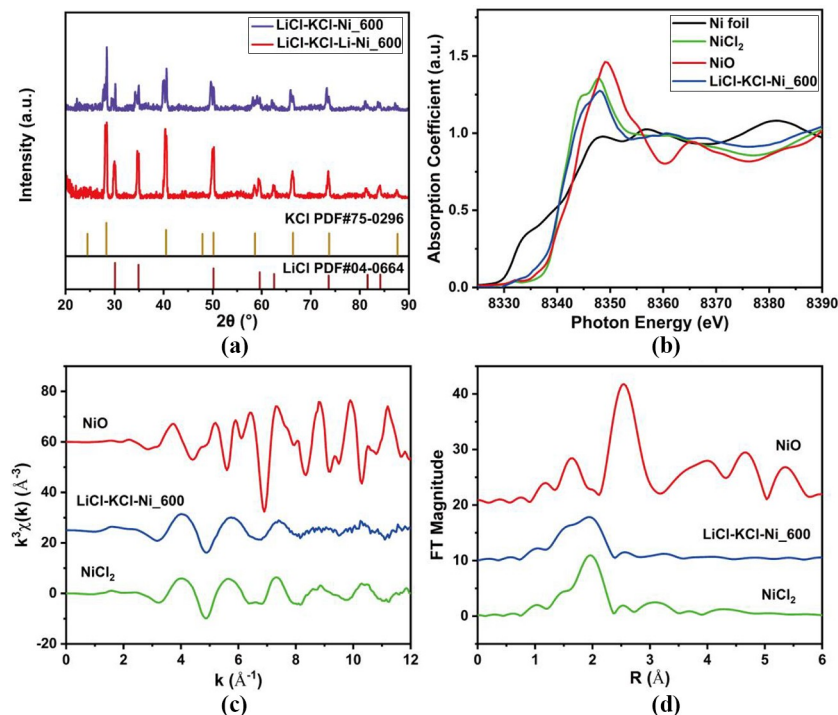


**Figure 5** (a) XRD patterns of LiCl-KCl-Li-Ni\_600 and LiCl-KCl-Ni\_600; (b) The Ni *K*-edge XANES spectra of Ni foil, NiCl<sub>2</sub>, NiO and LiCl-KCl-Ni\_600; (c) The Ni *K*-edge *k*<sup>3</sup>-weighted EXAFS oscillations for NiCl<sub>2</sub>, NiO and LiCl-KCl-Ni\_600; (d) Fourier transform of *k*<sup>3</sup>-weighted EXAFS data for NiCl<sub>2</sub>, NiO and LiCl-KCl-Ni\_600



weak pre-edge peak at approximately 8332 eV, which is due to the electric quadrupole  $1s \rightarrow 3d$  transition (Yamamoto, 2008). The LiCl-KCl-Ni\_600 also has a pre-edge peak at approximately 8332 eV and has the same absorption edge position located at about 8340 eV as NiCl<sub>2</sub>. The finding reveals that the oxidation state of Ni in LiCl-KCl-Ni\_600 is 2+, which is consistent with the above UV-vis result.

The coordination structure of Ni<sup>2+</sup> in LiCl-KCl-Ni\_600 was further determined by analyzing the Ni *K*-edge EXAFS data. The *k*<sup>3</sup>-weighted Ni *K*-edge EXAFS oscillation for NiCl<sub>2</sub>, NiO and LiCl-KCl-Ni\_600 are shown in Figure 5(c). The LiCl-KCl-Ni\_600 displays a different EXAFS spectrum compared to NiCl<sub>2</sub> and NiO. Fourier transform (FT) of the EXAFS spectra without phase shift correction are shown in Figure 5(d). The NiO displays two symmetrical main peaks below 3 Å. The first peak at approximately 1.6 Å arises from Ni-O coordination and the second peak at approximately 2.5 Å results from Ni-Ni scattering contribution. The NiCl<sub>2</sub> has two main peaks. The strong peak at approximately 2.0 Å is ascribed to Ni-Cl coordination and the weak peak at approximately 3.1 Å is attributed to Ni-Ni contribution. Significantly, by comparing with NiO and NiCl<sub>2</sub>, it can be distinguished that LiCl-KCl-Ni\_600 has only overlapping Ni-O and Ni-Cl peaks. The LiCl-KCl-Ni\_600 can be well fitted by using both Ni-O and Ni-Cl shells. (Figure S3, Supplementary Information) The structural parameters derived from the EXAFS fitting are compiled in Table 1. The average coordination numbers of Ni-O and Ni-Cl in LiCl-KCl-Ni\_600 were determined to be  $2.5 \pm 0.4$  and  $5.0 \pm 0.5$ , respectively. The average bond lengths of Ni-O and Ni-Cl in LiCl-KCl-Ni\_600 were determined to be approximately 2.04 Å and approximately 2.35 Å, respectively. The

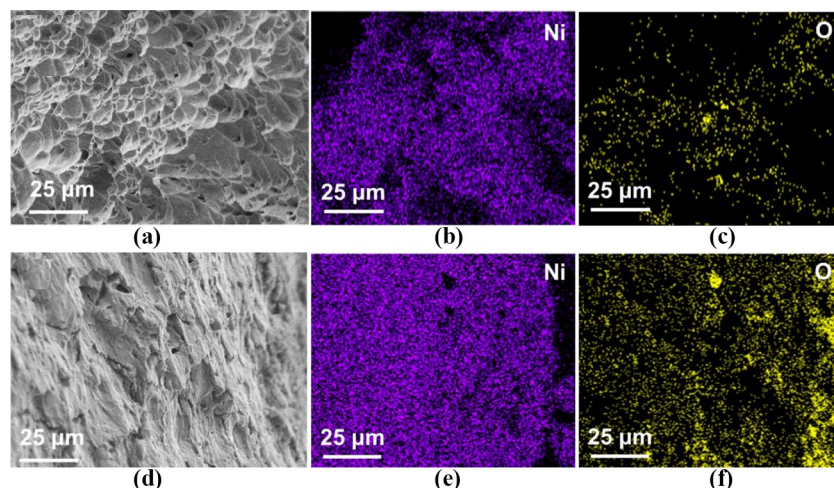
**Table 1** Structural parameters derived from the EXAFS curve-fitting for NiCl<sub>2</sub>, NiO and LiCl-KCl-Ni\_600

| Sample            | Path  | N             | R               | $\sigma^2$ ( $10^{-3} \text{ \AA}^2$ ) | $\Delta E_0$ (eV) |
|-------------------|-------|---------------|-----------------|----------------------------------------|-------------------|
| NiO               | Ni-O  | 6.0           | $2.09 \pm 0.01$ | $7.5 \pm 0.9$                          | -4.0              |
|                   | Ni-Ni | 12.0          | $2.95 \pm 0.02$ | $6.9 \pm 0.3$                          | -7.0              |
| NiCl <sub>2</sub> | Ni-Cl | $6.0 \pm 0.5$ | $2.39 \pm 0.01$ | $10.0 \pm 0.7$                         | 1.5               |
|                   | Ni-Ni | $5.7 \pm 1.0$ | $3.45 \pm 0.02$ | $14.5 \pm 1.0$                         | -1.0              |
| LiCl-KCl-Ni_600   | Ni-O  | $2.5 \pm 0.4$ | $2.04 \pm 0.01$ | $5.1 \pm 1.6$                          | 6.0               |
|                   | Ni-Cl | $5.0 \pm 0.5$ | $2.35 \pm 0.02$ | $9.4 \pm 1.0$                          | -8.5              |

results indicate that Ni<sup>2+</sup> in LiCl-KCl-Ni-600 mainly exists in the form of KNiCl<sub>3</sub> complex with a small amount of dissolved NiO.

The SEM image of the nickel sheet after reacting with LiCl-KCl at 600°C for 7 days is shown in Figure 6(a). The Ni sheet surface displays uneven fish scale-like corrosion pits, which further confirms that nickel metal undergoes obvious dissolution corrosion in LiCl-KCl. EDS elemental mappings of the Ni sheet are shown in Figure 6(b). There are some obvious Ni depletion zones in the Ni element distribution map. The distribution of O element [Figure 6(c)] is more uneven and the distribution area is noticeable smaller than that of Ni. In addition, the EDS analysis (Figure S4a, Supplementary Information) shows that the content of Ni and O elements is 99.2 Wt% and 0.5 Wt%, respectively. In contrast, as shown in Figure 6(b), the surface of the Ni sheet after reacting with LiCl-KCl-Li has no obvious pitting and is flatter. This phenomenon further confirms the ultra-low corrosiveness of LiCl-KCl-Li. The surface of the Ni sheet exhibits a more uniform distribution of Ni and O elements

**Figure 6** Surface morphologies and EDS elemental mappings of the Ni sheet after reacting with LiCl-KCl (a,b,c) and LiCl-KCl-Li (d,e,f) at 600°C for 7 days



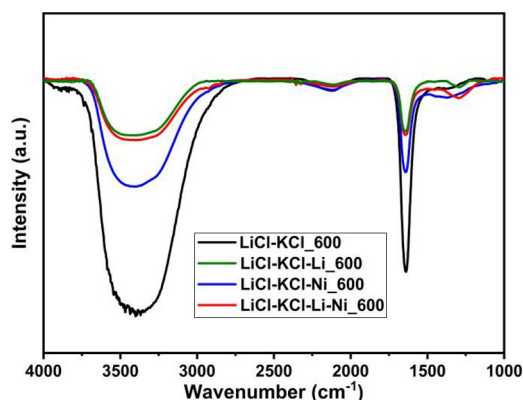
[Figure 6(e) and (f), ] with higher oxygen content (Figure S4b Supplementary Information). These findings reveal obvious dissolution of surface NiO and Ni on the Ni sheet in LiCl-KCl. Cross-sectional SEM images of the post-corroded Ni sheets in LiCl-KCl and LiCl-KCl-Li at 600°C for 7 days are shown in Figure S5 (Supplementary Information). The Ni sheet after reacting with LiCl-KCl exhibits some irregular corrosion pits with a maximum depth of approximately 3 μm (Figure S5a). No corrosion pits were observed in the Ni sheet after reacting with LiCl-KCl-Li (Figure S5b).

IR spectra of the LiCl-KCl-Ni\_600 and LiCl-KCl-Li-Ni\_600 samples collected on the seventh day are shown in Figure 7. LiCl-KCl exhibits strong characteristic absorption peaks of H<sub>2</sub>O at approximately 3410 cm<sup>-1</sup> and approximately 1645 cm<sup>-1</sup>. The broad peak centered at approximately 3410 cm<sup>-1</sup> is referred as the O-H stretching mode and the intense peak at approximately 1645 cm<sup>-1</sup> is assigned to the bending vibration of O-H (Sun *et al.*, 2020; Yu *et al.*, 2020). This result further confirms that the synthesized LiCl-KCl contains a certain degree of moisture, indicating that it is difficult to completely remove moisture from LiCl-KCl solely

through heating. It should be noted that the area of the H<sub>2</sub>O absorption peaks of LiCl-KCl-Ni\_600 is significantly smaller than that of LiCl-KCl\_600. This phenomenon implies that impurity moisture in LiCl-KCl is involved in the dissolution corrosion of metal Ni. Due to the moisture absorption during the testing process, the LiCl-KCl-Li\_600 and LiCl-KCl-Li-Ni\_600 samples also exhibited characteristic H<sub>2</sub>O peaks whose intensities are obvious lower than those of LiCl-KCl-Ni\_600.

Based on the above analysis, it can be concluded that the impurity moisture in LiCl-KCl plays a key role in the dissolution corrosion of Ni. The highly corrosive HCl generated by the hydrolysis of chloride salts at high temperatures will undergo redox reaction with Ni atoms on the surface of nickel sheet. The Ni<sup>2+</sup> ions generated by the reaction dissolve into LiCl-KCl and mainly exist in the form of KNiCl<sub>3</sub> complex. In addition, the highly corrosive HCl will also dissolve the unavoidable oxidation component NiO on the surface of nickel sheet into LiCl-KCl molten salt. When metallic lithium dissolves into LiCl-KCl to form a LiCl-KCl-Li solution, the impurity moisture of LiCl-KCl will react with Li and be completely consumed. Therefore, LiCl-KCl-Li samples with dissolved metallic Li component neither exist nor produce highly corrosive HCl at high temperatures, ultimately exhibiting ultra-low corrosiveness to metal nickel.

**Figure 7** IR spectra of LiCl-KCl, LiCl-KCl-Li\_600, LiCl-KCl-Ni\_600 and LiCl-KCl-Li-Ni\_600



#### 4. Conclusions

The LiCl-KCl-Li samples with dissolved metallic Li component were synthesized by heating a mixture of LiCl-KCl eutectic salt and metal lithium. The static corrosion test of nickel in molten LiCl-KCl-Li and LiCl-KCl was applied to evaluate their corrosiveness. It is proved that the Ni sheet in LiCl-KCl undergoes obvious dissolution corrosion, and the generated Ni<sup>2+</sup> ions mainly exist in the form of KNiCl<sub>3</sub> complex with a small amount of dissolved NiO. In contrast, the molten LiCl-KCl-Li salt solution shows ultra-low corrosiveness toward metal Ni. No Ni content above the ICP-OES detection limit was detected in the LiCl-KCl-Li sample that reacted with a Ni sheet at 600°C for 7 days. It was demonstrated that the impurity moisture in LiCl-

KCl plays a key role in the dissolution corrosion of Ni and is difficult to be completely removed by heating. Fortunately, the dissolved metallic Li in LiCl-KCl-Li ensures the complete removal of impurity moisture, thereby cutting off the pathway for Ni to react with highly corrosive HCl. This work provides a scientific basis for inhibiting metal corrosion in chloride salts from the perspective of molten-salt regulation.

## Data availability

All data included in this research are available upon request by contact with the corresponding author.

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## Supplementary material

The supplementary material for this article can be found online.

## Corresponding authors

Hongliang Bao can be contacted at: [baohongliang@sinap.ac.cn](mailto:baohongliang@sinap.ac.cn); Jian Yao can be contacted at: [yaojian@sinap.ac.cn](mailto:yaojian@sinap.ac.cn); Yuan Qian can be contacted at: [qianyuan@sinap.ac.cn](mailto:qianyuan@sinap.ac.cn)

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