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Facile preparation of highly pure KF-ZrF_4 molten salt

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Abstract

The preparation of highly pure KF-ZrF_4 (FKZr) molten salt, a potential secondary coolant in molten salt reactors, was realized simply by heating a mixture of $(NH_4)_2ZrF_6$ and KF. X-ray diffraction analysis indicated that the FKZr molten salt was mainly composed of $KZrF_5$ and K_2ZrF_6. The melting point of the prepared FKZr molten salt was 420–422 °C under these conditions. The contents of all metal impurities were lower than 20 ppm, and the content of oxygen was lower than 400 ppm. This one-step protocol avoids the need for a tedious procedure to prepare ZrF_4 and for an additional purification process to remove oxide impurities, and is therefore a convenient, efficient and economic preparation method for high-purity FKZr molten salt.

Keywords KF-ZrF_4 · Molten salt · $(NH_4)_2ZrF_6$ · High purity · Preparation

1 Introduction

As one of the next generation of nuclear fission reactors, the molten salt reactor (MSR) can run at high temperatures with a high thermodynamic efficiency while maintaining a low vapor pressure. Owing to the potential advantages of MSRs, such as their inherently safe design, good neutron economy and the possibility for online processing, research on MSRs has received considerable attention again over the past decade and MSRs are anticipated to become operational by 2040 [1–6]. Because the heat produced from fission chain reactions in the reactor vessel is transferred through two separate coolant loops (primary and secondary), the selection of coolants is very important for MSRs. There have been determined efforts in the search for suitable coolants, and recent studies have indicated that KF-ZrF_4 (FKZr), a eutectic fluoride salt consisting of ZrF_4 (42 mol%) and KF (58 mol%), may act as a secondary coolant owing to its special properties, which include a relatively low melting point, low toxicity and low corrosivity [7–11]. Besides, these advantages allow FKZr to be used as an advanced heat transfer and storage material covering aspects such as solar power, fuel cell, metallurgical and chemical industry [12, 13]. Impurities in molten salts used as coolants result in serious corrosion and other issues, and therefore need to be strictly removed. This means that the development of efficient methods for the preparation of highly pure FKZr is of considerable importance.

Surprisingly, although the thermo-physical properties of FKZr have been adequately studied, the preparation of high-purity FKZr remains challenging. The only approach reported so far is to melt a mixture of KF and high-purity ZrF_4 [14, 15], but this approach suffers from a tedious procedure and the high cost of the purification of ZrF_4. Although ZrF_4 is commercially available, the commercial source is a hydrated salt. Because the presence of water would lead to a dramatic decrease in the quality of the final FKZr molten salt owing to hydration at high temperatures, the hydrated salt needs to be purified. This is usually done by sublimation under an inert atmosphere, but this is tedious and expensive. Although purification of FKZr obtained by melting a mixture of KF and commercial ZrF_4 may be realized by hydrofluorination using H_2 and HF as purification agents, special safety precautions are required because of the flammability and explosivity of H_2 and the high corrosivity of HF [16–19]. Therefore, a more practically useful, efficient and economical protocol for the preparation of pure FKZr is desired.

Previously, we have shown that heating a mixture of $(NH_4)_2BeF_4$ and LiF can afford the molten salt Li_2BeF_4 in high purity [20]. We therefore speculated that highly pure FKZr molten salt could be produced from a mixture of $(NH_4)_2ZrF_6$ and KF simply by heating at high temperatures.

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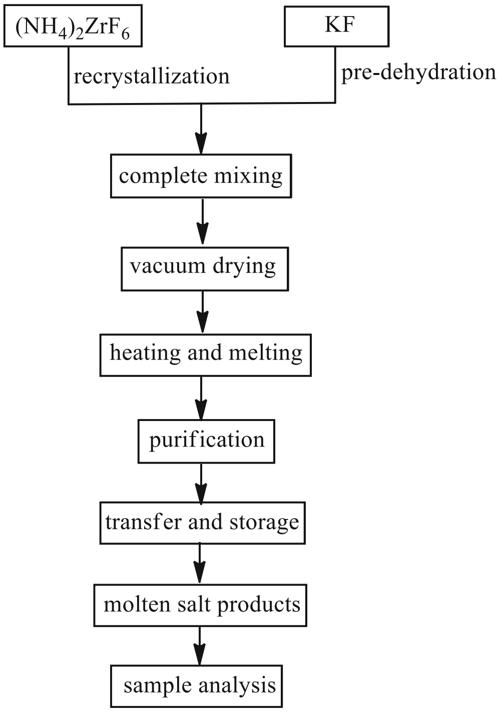

Fig. 1 Molten FKZr salt preparation flow chart

This protocol is convenient and economical because the pure product can be obtained in a one-step procedure without additional purification processes. Our preliminary results are described herein.

2 Experimental

2.1 Preparation of raw materials

All fluoride salts have a hygroscopic nature. This will trigger a hydrolysis reaction and produce oxides at high temperatures, which may introduce impurities into the FKZr molten salt. It is therefore necessary to remove the moisture from fluoride salts before using them. KF (99.99%) and $(NH_4)_2ZrF_6$ (99.9%) were purchased and treated separately before mixing. To remove the moisture from KF, it was heated at 400 °C for 4 h in a drying oven with simultaneous vacuum pumping [21]. Commercial $(NH_4)_2ZrF_6$ contains many types of impurities, so it was treated by recrystallization. The product was recrystallized several times as necessary, and then dried at 120 °C for 4 h before use. Treated KF and $(NH_4)_2ZrF_6$ were kept in an argon-filled glove box with O_2 and/or H_2O contents of less than 1 ppm. Dried $(NH_4)_2ZrF_6$ was characterized by inductively-coupled plasma (ICP) spectroscopy.

2.2 Preparation of FKZr molten salt

To prepare the FKZr molten salt, an initial molar ratio of $(NH_4)_2ZrF_6$:KF = 42:58 was used. $(NH_4)_2ZrF_6$ and KF were mixed and placed in a platinum crucible under argon. The crucible was taken out of the glove box and quickly transferred to a vacuum induction melting furnace. To ensure homogeneity, the FKZr salt was prepared and melted under an argon atmosphere in the electrical furnace. The furnace chamber pressure was kept under 10 Pa for 1 h to remove traces of water under high vacuum conditions. Then, the furnace chamber was recharged with high-purity argon to permit a minor positive pressure, and the mixture was melted according to a preset temperature program. After melting was complete, the salts were transferred to an airtight storage tank and cooled naturally to room temperature for sample analysis (Fig. 1).

A double-layer structure was used in the vacuum induction furnace. Internal circulating water was used to form a cooling wall and the cooling wall surface was treated using Teflon spray to give it anti-corrosion properties. Highly toxic HF and NH_3 were released and combined to give NH_4F, which condensed on the cooling wall. The vaporization products could then be removed easily from the FKZr molten salt system to avoid pollution of the molten salt.

The FKZr molten salt was ground to a fine powder in a glove box and then characterized by differential scanning calorimetry-thermogravimetric analysis (DSC-TGA), X-ray diffraction (XRD) and trace oxygen determination.

2.3 Characterization

Inductively-coupled plasma -optical emission spectrometry (ICP-OES) was carried out on a SPECTRO ARCOS spectrometer (Germany). Typically, a molten salt sample was dissolved in 2% HNO_3 solution. The operating conditions were: forward power 1.43 kW; argon gas flow rate 12.00 L/m (plasma), 0.9 L/min (auxiliary), 0.78 L/min (nebulizer) and spectral purity (99.99%) argon gas. DSC-TGA measurements were conducted using a TA SDT-Q600 thermogravimetric system (U.S.). Samples (~10 mg) were sealed hermetically in an

Table 1 Analysis of impurities (ppm) in $(NH_4)_2ZrF_6$ using inductively coupled plasma optical emission spectrometry

	B	Si	Ca	Fe	S
Raw material	12.9	69.2	11.5	12.8	27.6
Recrystallization-1st (cooling rate10 °C/h)	8.6	29.7	7.2	11.1	18.2
Recrystallization-1st (cooling rate 5 °C/h)	5.1	16.1	5.3	5.5	11.0
Recrystallization-2nd (cooling rate 5 °C/h)	4.6	4.3	3.3	2.1	6.3

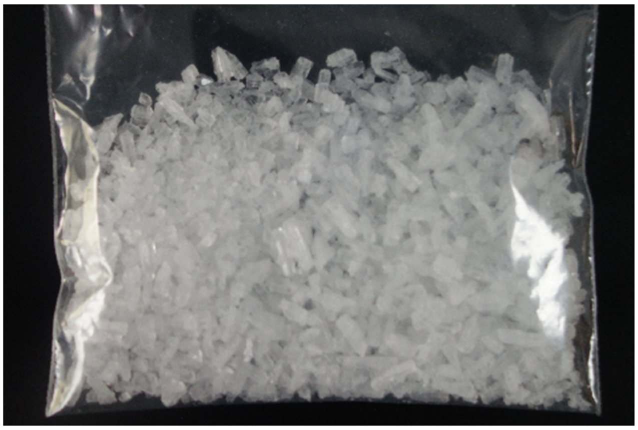

Fig. 2 $(NH_4)_2ZrF_6$ crystals after the second crystallization process

aluminum pan, heated to 800 °C at 20 °C/min, held for 3 min at this temperature and then cooled to room temperature at the same rate. Phase constitutions were identified by powder XRD using an X'Pert Pro diffractometer with CuKα radiation operated at 40 kV and 40 mA. Oxygen content analysis was carried out using a Leco O836 meter. Because fluoride is corrosive and will volatilize easily at high temperatures and thus damage the detection system, a halogen trap assembly was installed after the cracking furnace to eliminate pollution by fluoride vapor [22].

3 Results and discussion

3.1 Purification of $(NH_4)_2ZrF_6$ by recrystallization

Because commercial $(NH_4)_2ZrF_6$ (99.9%) is contaminated with impurities, purification is necessary. $(NH_4)_2ZrF_6$ is soluble in water, but its solubility depends greatly on temperature, with a solubility in water (100 g) of 83 g at 80 °C and 22 g at 10 °C. This variation in the solubility suggests that $(NH_4)_2ZrF_6$ could easily be purified by recrystallization from water.

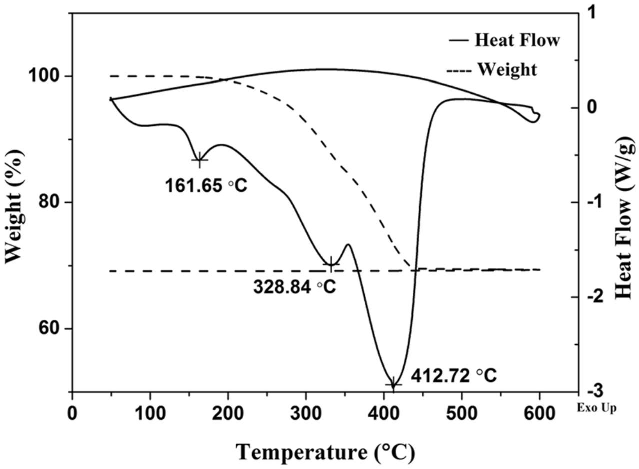

Fig. 3 Differential scanning calorimetry-thermogravimetric analysis of $(NH_4)_2ZrF_6$

Because the cooling rate in the recrystallization process has a considerable influence on the crystal size and purity, it is necessary to select an appropriate cooling rate for purification. Two different rates (5 °C/h and 10 °C/h) were investigated for the recrystallization of $(NH_4)_2ZrF_6$ (83 g) from water (100 g). Analysis of the impurities in $(NH_4)_2ZrF_6$ determined by ICP indicated that the lower cooling rate was preferred for elimination of the impurities (Table 1). Furthermore, compared with the cooling rate of 10 °C/h, cooling at 5 °C/h gave bigger and more regular crystals. The optimal conditions for recrystallization were thus identified as follows: a solution of $(NH_4)_2ZrF_6$ (83 g) in water (100 g) at 80 °C was cooled slowly to 10 °C at a rate of 5 °C/h. A second recrystallization process gave higher purity $(NH_4)_2ZrF_6$. As shown in Fig. 2, regular white crystals were obtained after the second crystallization. Recrystallization of $(NH_4)_2ZrF_6$ was thus successful and is a very convenient and efficient method of purification.

3.2 Preparation of highly pure FKZr

3.2.1 Effect of the decomposition rate of $(NH_4)_2ZrF_6$

Thermal gravimetric analysis showed that decomposition of $(NH_4)_2ZrF_6$ began at around 150 °C, and full decomposition was observed at 450 °C (Fig. 3). The residue after decomposition is expected to be ZrF_4. The mass percentage of the residue with respect to the mass of $(NH_4)_2ZrF_6$ was 69.3% as determined by TGA analysis, which is consistent with the theoretical percentage of ZrF_4 in $(NH_4)_2ZrF_6$.

Because 150 and 450 °C correspond to the starting and full decomposition temperatures, respectively, the mixture of $(NH_4)_2ZrF_6$ and KF was heated sequentially to three temperatures, 150, 450 and 600 °C, to prepare pure FKZr (Fig. 4). The mixture was first heated to 150 °C under vacuum to remove free water. This procedure is necessary because the presence of water in the mixture is unavoidable owing to the high hygroscopicity of KF. However, water is not completely removed in this process. It is well known that at high temperatures, the presence of water or hydroxide leads to the formation of oxide impurities. These oxide impurities then need to be removed to obtain the pure molten salts. Fortunately, the decomposition of $(NH_4)_2ZrF_6$ gives not only ZrF_4, but also NH_3 and HF. HF acts as an efficient purification agent and

$$25°C \xrightarrow{1h} 150°C \xrightarrow{1h} 150°C \xrightarrow{3h} 450°C \xrightarrow{1h} 600°C \xrightarrow{3h} 600°C$$

Fig. 4 Temperature gradient for the preparation of $KF\text{-}ZrF_4$ (FKZr) molten salt

Table 2 Effects of heating rate on increasing the temperature from 150 to 450 °C on the purity of the molten salt

Entry	n (h)	Heating rate (°C /min)	Oxygen content (ppm)[a]
1	0.5	10	1104
2	1.0	5	786
3	2.0	2.5	578
4	3.0	1.67	382
5	4.0	1.25	369

[a] Oxygen content analysis was determined using a Leco O836 meter

converts the oxide impurities back into fluoride salts. Therefore, elevation of the temperature from 150 °C to 450 °C to allow the decomposition of $(NH_4)_2ZrF_6$ is very important for obtaining a molten salt with high purity. This process cannot proceed in vacuum because the presence of HF is necessary, and must therefore be carried out under an argon or nitrogen atmosphere. The last process, in which the temperature is raised from 450 to 600 °C, melts the mixture and yields the pure molten salt.

Owing to the importance of elevating the temperature from 150 to 450 °C, the effect of heating rate on the purity of the final molten salt was investigated (Table 2). Indeed, a high heating rate resulted in a low purity (Table 2, entries 1–2). When the heating rate was 10 °C/min, the oxygen content in the molten salt preparation was as high as 1104 ppm. This is probably because the generated HF did not fully react with the oxide impurities owing to the rapid decomposition of $(NH_4)_2ZrF_6$ during this period. To our delight, a low heating rate gave the FKZr molten salt in high purity (Table 2, entries 4–5). Heating rates of 1.67 °C/min (Table 2, entry 4) and 1.25 °C/min (Table 2, entry 5) gave comparable purities. To improve the efficiency of the process, a heating rate of 1.67 °C/min was selected as the optimal rate.

Table 3 Samples crystal data information from the XRD patterns of $KZrF_5$, K_2ZrF_6, $ZrF.H_2O$ and ZrF_4

	$KZrF_5$	K_2ZrF_6	$ZrF_4 \cdot H_2O$	ZrF_4
Crystal system	Anorthic	Orthorhombic	Tetragonal	Monoclinic
Space group	P $\bar{1}$	Cmcm	I $\bar{4}$ 2d	C2/c
a (Å)	7.3700	6.5800	7.7080	9.5700
b (Å)	8.4610	11.4000	7.7080	9.9300
c (Å)	10.7110	6.9400	11.6640	7.7300
α(°)	75.0400	90.0000	90.0000	90.0000
β(°)	71.5700	90.0000	90.0000	94.0000
γ(°)	66.1300	90.0000	90.0000	90.0000

3.2.2 Effect of the melting temperature and time

The next step after the decomposition of $(NH_4)_2ZrF_6$ at about 450 °C is the melting process. The mixtures were further heated to different temperatures (500, 550, 600 and 650 °C) to maintain the melted state, and the preservation time was set at 2–4 h.

The experimental results indicate that the melting temperature and melting time have important effects on the final quality of the FKZr molten salt. Figure 5 shows the XRD pattern of the FKZr molten salt prepared under different conditions. The XRD patterns show that the FKZr molten salt is mainly composed of $KZrF_5$ and K_2ZrF_6 (Tables 3 and 4). The height and width of the diffraction peaks are associated with the crystallite size and the degree of crystallinity; small, broad peaks represent small grain sizes, and sharp, narrow peaks indicate high crystallinity and large grain size. When the salt was melted at 500 °C, there was still some loose powder in the product. This means that the mixture was not completely melted or fully mixed at this low temperature. With a melting temperature of 550 °C or 600 °C and a melting time of 2 h, the

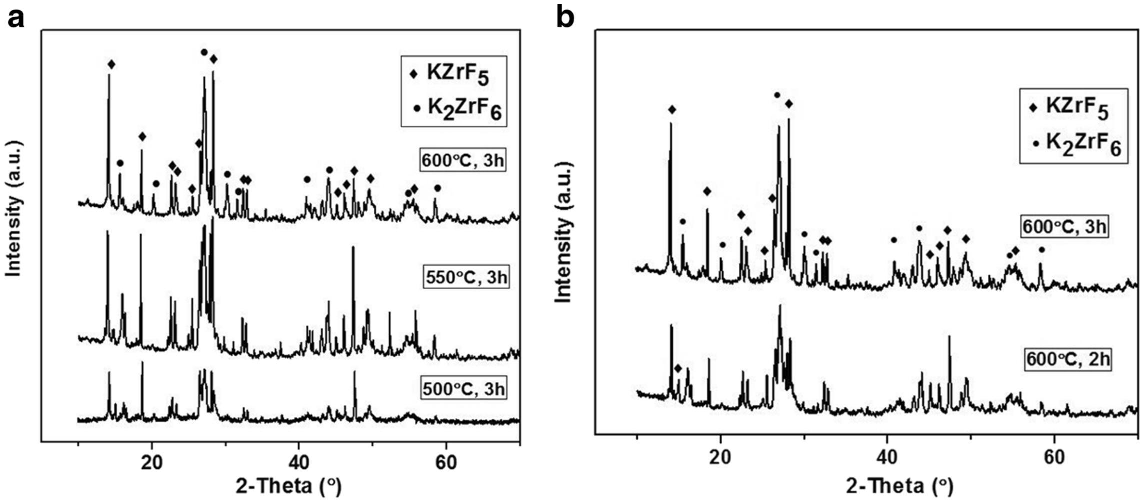

Fig. 5 X-ray diffraction patterns of KF-ZrF_4 (FKZr) molten salts prepared with **a** different melting temperatures and **b** different times

Table 4 The diffraction angles (2θ) with indices (hkl) for $KZrF_5$ and K_2ZrF_6 crystals from the XRD data

$KZrF_5$				K_2ZrF_6	
2θ/°	h k l	2θ/°	h k l	2θ/°	h k l
11.559	0 1 0	32.290	1 −2 1	15.533	0 2 0
14.012	1 1 1	32.806	0 2 3	20.165	1 1 1
18.512	1 1 2	45.085	0 0 5	26.998	1 3 0
22.569	0–1 2	46.078	0 −2 4	29.982	1 3 1
23.145	−1 1 1	47.307	−2 2 2	31.328	0 4 0
25.477	1 1 3	49.472	4 2 2	40.759	0 4 2
26.503	−1 −2 1	55.466	−1 2 5	43.827	1 5 1
28.240	2 2 2			58.195	1 7 0

Table 5 Effect of the melting conditions on the melting point of the molten salt

Sample	Melting temperature/°C	Melting time/ h	Melting point/°C
1	500	3	–
2	550	2	427
3	550	3	420
4	600	2	425
5	600	3	421
6	650	3	–

intensity of the XRD peaks was weak and the melting point was increased (Table 5). This shows that shorter melting times were not suitable for FKZr preparation. When the melting time was extended to 3 h at the same melting temperature, the intensity of the diffraction peaks was clearly enhanced. Under these conditions, the melting point of the product was about 420 °C, which is consistent with the value reported in the literature. However, the gloss appearance of FKZr at a melting temperature of 550 °C was generally darker than that at 600 °C. That is, the preparation of the molten salt also has a certain temperature requirement. However, higher temperatures require harsh conditions, and an important factor to consider is that ZrF_4 begins to sublime at above 600 °C. When the melting temperature was set at 650 °C, the inner wall of the melting crucible was covered with volatile ZrF_4 powder. The molar ratio of the initial mixture was thus changed, and the melting point of the final molten salt may deviate from the accurate value. We therefore suggest that an appropriate melting temperature is 600 °C with a reaction time of 3 h. The melting point of the prepared FKZr molten salt was 420–422 °C under these conditions (Fig. 6). The contents of all metal impurities were lower than 20 ppm, and the content of oxygen was lower than 400 ppm (Table 6).

Table 6 Impurities (ppm) in the final KF-ZrF_4 (FKZr) molten salt

Entry	O	Cr	Fe	Ni	B	Si	Ca	S
1	370	<5	11	< 5	< 5	17	<5	<5
2	363	<5	14	< 5	< 5	19	<5	5.8
3	396	<5	13	< 5	<5	15	<5	<5
4	356	< 5	10	<5	<5	17	<5	<5
5	378	<5	9	< 5	<5	11	<5	5.6

3.2.3 Effect of the preparation process on the quality of FKZr

To investigate the effect of the generated HF on the quality of the prepared FKZr, a two-step preparation method was carried out and compared with the one-step method. First, a known amount of $(NH_4)_2ZrF_6$ was placed in a platinum crucible, heated to 450 °C and maintained at this temperature for 3 h under a nitrogen atmosphere to prepare high-purity ZrF_4. Then, the obtained ZrF_4 was removed from the melting crucible and mixed with KF in the appropriate ratio, and the FKZr molten salt was prepared according to the temperature gradient shown in Fig. 4.

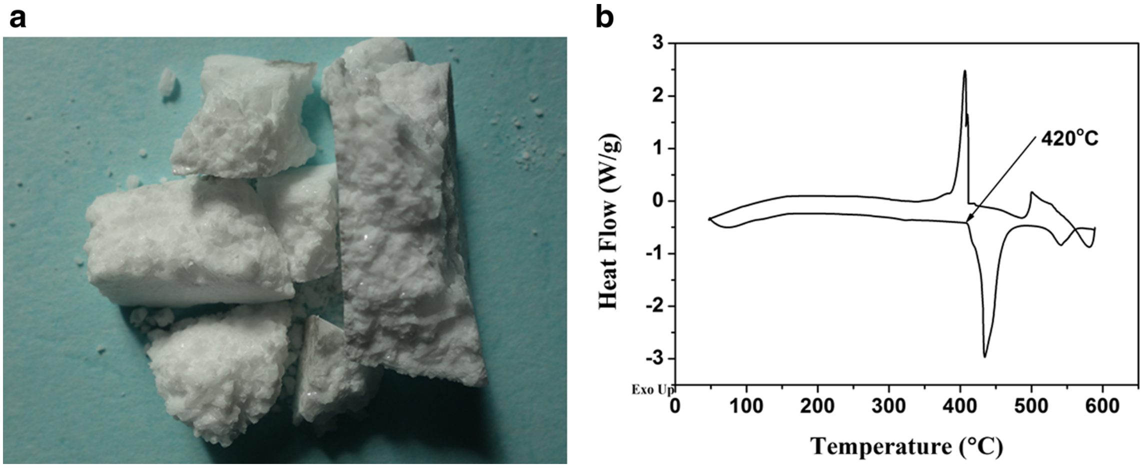

Fig. 6 **a** KF-ZrF_4 sample and **b** differential scanning calorimetry curve of the molten salt

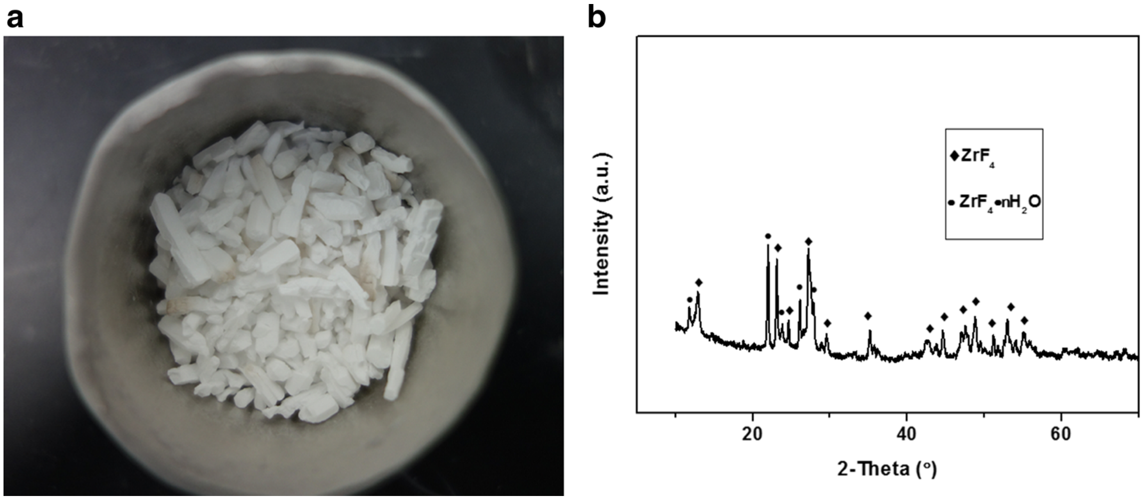

Fig. 7 **a** ZrF_4 sample prepared by decomposition of $(NH_4)_2ZrF_6$ and **b** its X-ray diffraction pattern

The prepared ZrF_4 sample and its XRD pattern are shown in Fig. 7. The typical peaks of the ZrF_4 phases were detected and most of the diffraction peaks are in accordance with the standard spectrum of ZrF_4. However, there are some impurity peaks at 11.74°, 22.00°, 23.80°, 26.09° and 27.89°, which correspond to the typical peaks of the crystalline hydrate of ZrF_4 (Tables 3 and 7). It is possible that the prepared ZrF_4 is the crystalline hydrate because of its hygroscopic nature. These salts must be protected from atmospheric contamination by handling and storage in sealed containers.

Figure 8 shows the XRD pattern of the FKZr molten salt prepared by the two-step method. The degree of crystallization and the intensity of the peaks was decreased compared with those observed using the one-step method. Furthermore, the oxygen content in the molten salt was above 2000 ppm. The dehydration of hydrated ZrF_4 is extremely difficult during the preparation of the molten salt. Some water of crystallization was removed at around 120 °C, but the residual compound began to decompose and produce zirconium oxides such as $ZrOF_2$, rather than undergoing further dehydration on increasing the temperature. Therefore, the addition of a purification agent, such as NH_4HF_2, is usually required during the preparation of fluoride molten salts. Notably, the HF generated by decomposition of $(NH_4)_2ZrF_6$ in the one-step method was used as the purification agent in place of the NH_4HF_2 salts mentioned above. This not only allowed the preparation of pure products without the introduction of other impurities, but also simplified the preparation process. As a result, our development of a molten salt preparation method using a facile one-step procedure without the need for additional purification agents should be of great significance.

Table 7 The diffraction angles (2θ) with indices (hkl) for ZrF_4 and $ZrF_4{\cdot}H_2O$ crystals from the XRD data

ZrF_4				$ZrF_4{\cdot}H_2O$	
2θ/°	h k l	2θ/°	h k l	2θ/°	h k l
13.013	1 0 1	44.833	−3 3 2	11.743	1 0 1
22.962	−2 1 1	47.593	−3 2 3	22.001	1 1 2
24.435	2 1 1	48.652	5 1 0	23.809	2 0 0
27.250	1 1 2	50.978	−2 5 1	26.093	2 1 1
29.555	−3 0 1	52.975	−5 1 2	27.89	2 0 2
36.041	0 1 3	55.660	2 2 4		
42.783	3 2 1				

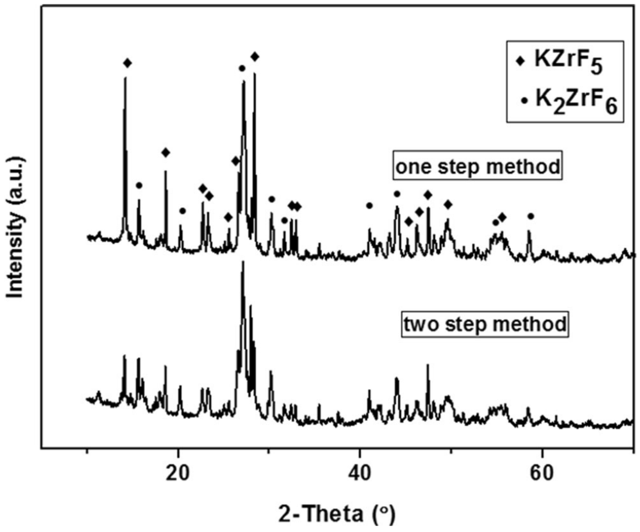

Fig. 8 X-ray diffraction patterns of KF-ZrF_4 (FKZr) molten salt using different preparation methods

4 Conclusion

A new one-step synthesis method was developed for the preparation of high-purity FKZr molten salt by melting a mixture of KF and purified $(NH_4)_2ZrF_6$ under high-temperature conditions. $(NH_4)_2ZrF_6$ functions both as a reactant and as a fluorinating agent, and decomposes at high temperatures to form ZrF_4 and release HF. ZrF_4 is the raw material required to prepare the molten salt, and HF is used as a fluorinating agent to remove oxygen and thus oxide impurities from the molten salt. This method is efficient, simple to operate, convenient, safe and will be of benefit in industrial production processes.

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