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The oxoacidic properties and deoxidization features of LiCl-based melts at 973 K

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The oxoacidic properties of LiCl melt were investigated on the basis potentiometric determination of magnesium oxide solubility at 973 K using the $Pt(O_2)|ZrO_2(Y_2O_3)$ indicator electrode. The solubility product of MgO $pK_{s,\ MgO,\ LiCl}$ was estimated as 4.57 in the molality scale ($pK_{sx,\ MgO,\ LiCl}$ = 7.33 in molar fractions), i.e. magnesium oxide is slightly soluble in the LiCl melt. The oxobasicity index of the LiCl melt is 4.29, i.e., it has stronger oxoacidic properties than the melt of the eutectic KCl-LiCl mixture (3.50), which is caused by the higher content of lithium cations in pure LiCl melt. The deoxidization of LiCl melt by carbochlorination at 973 K lasts 60 minutes and is limited by residual O^{2-} concentration of $1.9 \cdot 10^{-7}$ mol·kg^{-1}. The chemical reaction of second kinetic order becomes the limiting stage 30 minutes after the process starts and is defined by the rate constant of 3.3 (±0.2) $\cdot 10^5$ kg·mol^{-1}·min^{-1}. The strong oxoacidic properties of LiCl allow to quantitatively dissolve Eu_2O_3 which may prove useful from the viewpoint of scintillation materials science. At 973 K the solubility of Eu_2O_3 is equal to 0.1 mol·kg^{-1} (or approximately 3.4 % w/w). At the carbochlorination of LiCl melt containing 0.1 g of Eu_2O_3 the process of dissolving the Eu oxo-derivatives takes 100 min, and the total duration of the process increases to 4 h.

Keywords: molten lithium chloride, europium oxide, solubility, carbochlorination, potentiometry.

Кислотні властивості та особливості розкислення розтопів на основі LiCl при 973 K.
В.Л. Чергинець, Т.В. Пономаренко, О.Л. Ребров, Т.П. Реброва, О.І. Юрченко, В.В. Соловйов

Оксокислотні властивості розтопу LiCl досліджували потенціометричним визначенням розчинності оксиду магнію при 973 K з використанням індикаторного електрода $Pt(O_2)|ZrO_2(Y_2O_3)$. Добуток розчинності MgO $pK_{s,\ MgO,\ LiCl}$ оцінено, як 4,57 у шкалі моляльностей ($pK_{sx,\ MgO,\ LiCl}$ = 7,33 у молярних частках), тобто магній оксид є малорозчинним у розтопі LiCl. Індекс оксоосновності розтопу LiCl становить 4,29, він має сильніші кислотні властивості, ніж розтоп евтектичної суміші KCl-LiCl (3,50), що спричинено більшим вмістом катіонів літію. Розкислення розтопу LiCl за допомогою карбохлорування при 973 K триває 60 хв, а залишкова концентрація становить $1{,}9 \cdot 10^{-7}$ моль·кг$^{-1}$. Хімічна реакція другого кінетичного порядку стає лімітуючою стадією через 30 хвилин після початку процесу, а значення константи швидкості становить 3,3 (±0.2) $\cdot 10^5$ кг·моль$^{-1}$·хв$^{-1}$. Сильні оксокислотні властивості LiCl дозволяють кількісно розчиняти Eu_2O_3, що може бути корисним з точки зору матеріалознавства. При 973 K розчинність Eu_2O_3 становить 0,1 моль·кг$^{-1}$ (або приблизно 3,4 мас. %). При карбохлоруванні розтопу LiCl, що містить наважку 0,1 г Eu_2O_3, тривалість розчинення оксопохідних Eu складає 100 хв, а загальна тривалість процесу збільшується до 4 год.

1. Introduction

Molten lithium chloride plays important role in the processes of obtaining certain metals due to its possibility to dissolve appreciable amounts of the corresponding oxides because of relatively strong oxoacidic properties of Li^+ cation according to Lux – Flood definition [1]:

$$\underset{Base}{MeO_2}\downarrow + \underset{Acid}{2Li^+} = MeO^{2+} + Li_2O \quad . \tag{1}$$

LiCl melt is used as solvent for metallic lithium [2] or its oxide [3] in the processes of precipitation of such metals as Ti [3], Zr [4], actinides [5,6] and lanthanides from their compounds via the reduction by metallic lithium dissolved in the melt:

$$MeO^{2+} + 4Li = Me\downarrow + 2Li^+ + Li_2O\,, \tag{2}$$

or electrolysis:

$$MeO^{2+} + 4\bar{e} + 2Li^+ = Me\downarrow + Li_2O\,, \tag{3}$$

Therefore, it is quite naturally that molten LiCl is studied in various aspects [8-10]. From eqn. (2) and (3) it follows that the rate of the main process is dependent on the concentration of ionized compounds containing the precipitated metal (on their solubility) which, in turn, is determined by the acid-base equilibrium (1). However, there are no quantitative data on the oxoacidic properties of pure LiCl melt although the data on oxoacidity of widely used KCl-LiCl eutectic are known [11,12].

Another possible application of LiCl melt is obtaining of Eu^{2+}-activated scintillation materials. From this point it is interesting to study features of Eu_2O_3 dissolution in LiCl melt which is similar to the process of obtaining LiI:Eu^{2+} scintillator [13]. However, it is highly desirable to reduce oxide-ions concentration in the melt (to perform a deoxidization) as much as possible both to stimulate the process and to boost performance of the obtained scintillator [14].

Therefore, the purpose of the present work is to determine MgO solubility in LiCl melt in order to estimate its oxoacidic properties and Eu_2O_3 solubility from the viewpoint of scintillation materials applicability and also to study the features of 'pure' and Eu_2O_3-contaminated LiCl melt deoxidization in reactive gas atmosphere.

2. Experimental

Anhydrous LiCl was prepared by dissolution of 5 % excess amount of Li_2CO_3 (purum) in hydrochloric acid (reagent quality). The solution was preserved for 1 day and then was filtered to remove precipitates. Then NH_4Cl (pure) was added to the filtered solution in mole ratio of 0.1 NH_4Cl:1 LiCl and it was evaporated to dry powder state. Finally, the powder was heated in melted silica ampoule under vacuum of 10 Pa up to complete sublimation of NH_4Cl and melted. After cooling anhydrous LiCl was kept in glove box.

Anhydrous magnesium chloride was obtained according to the generally accepted routine by dissolution of MgO (reagent quality) in hydrochloric acid (reagent quality). Then NH_4Cl (pure) was added to the filtered solution in mole proportion of $MgCl_2$:NH_4Cl=1:1. After that the solution was evaporated up to dry powder state, placed in melted silica ampoule, heated under vacuum of 10 Pa up to complete sublimation of NH_4Cl and melted. The cooled $MgCl_2$ was placed in weighting bottle and kept in glove box.

Before the calibration the initial concentration of O^{2-} was decreased by addition of NH_4Cl:

$$2NH_4Cl + O^{2-} \rightarrow 2NH_3\uparrow + H_2O\uparrow + 2Cl^-\,, \tag{4}$$

KOH with initial mass fraction of 0.999 was melted in alundum crucible and kept for 1 h at 700 °C in argon atmosphere to remove the absorbed water. As it was determined in [15], the degree of KOH contamination with Al_2O_3 is negligible.

The high-purity Ar (99.99 % v/v) was used for the creation of inert and reactive gas atmosphere in the potentiometric cell with a provisional drying by P_2O_5.

The construction of potentiometric cell with the liquid junction for the determination of equilibrium O^{2-} molality is usual for such kind of measurements [7] and its electrochemical scheme is as follows:

$$\begin{gathered}(-)\left|Ag\right|Ag^+_{(l)}, LiCl \vdots\vdots LiCl + \\ + O^{2-}\left|YSZ\right|\left(O_{2,(g)}\right)Pt(+)\end{gathered}\,, \tag{5}$$

(*YSZ* is the solid electrolyte membrane of 0.9 ZrO_2+0.1 Y_2O_3 composition).

This cell was preliminary calibrated with known amounts of KOH as a strong base (oxide ion donor)

$$2KOH = 2K^+ + O^{2-} + H_2O\,, \tag{6}$$

to obtain the dependences of *emf vs.* the equilibrium oxide ion molality ($m_{O^{2-}}$). Describing the experimental results we further will use the oxide ion index $pO \equiv -logm_{O^{2-}}$. The calibration data also were used for the calculation of O^{2-} concentration from the potentiometric data during the course of the deoxidization process.

Potentiometric titration was performed by sequential additions of KOH (its initial molality was designed as $m_{O^{2-}}$) to the solution of Mg^{2+} ($m_{Mg^{2+}}$) in LiCl melt.

Eu_2O_3 solubility in LiCl was determined by sequential additions method (SAM), i.e. addition of small amounts of the oxide to the melt with the fixation of equilibrium *emf* (pO) after each addition. The stabilization of *emf* permits to consider the solution as saturated with the respect to europium oxide derivatives.

The reactive gas atmosphere during the deoxidization process was created by passing Ar through liquid CCl_4 (reagent quality) and carrying its vapor into the melt at 200 ml·min^{-1} rate. The changes of *pO* were fixed each 30 sec during first 10 min, then the interval between the consecutive measurements was increased to 1, 2 and, finally, 5 min up to the *emf* stabilization.

3. Results and discussion

1. Solubilities of magnesium and europium oxides in molten LiCl at 973 K

MgO solubility in LiCl melt was studied by a potentiometric titration method. The titration process occurs according to the following equation:

$$Mg^{2+} + O^{2-} = MgO(\downarrow). \qquad (7)$$

This equilibrium is described by the solubility product, $pK_{s,MgO}$:

$$pK_{s,MgO} = m_{Mg^{2+}} \cdot m_{O^{2-}}, \qquad (8)$$

where $m_{Mg^{2+}}$ and $m_{O^{2-}}$ are the equilibrium molalities of Mg^{2+} and O^{2-}, respectively. In order to compare the obtained data with similar results obtained in other melts $pK_{s,MgO}$ value was recalculated in mole fraction scale, $pK_{s,x,MgO,LiCl}$.

The potentiometric curve of Mg^{2+} titration with O^{2-} additions in molten LiCl is presented in Fig.1 and the calculated data are collected in Table 1.

pO range at Mg^{2+} excess (1.5–3.5) gives possibility to refer MgO as merely soluble in LiCl oxide similarly to BaO in 2CsBr-KBr melt [16]. The ligand number $\bar{n}$ $\bar{n} = (m^{m_0}_{O^{2-}} - m_{O^{2-}}) / m^0_{Mg^{2+}})$ increases with

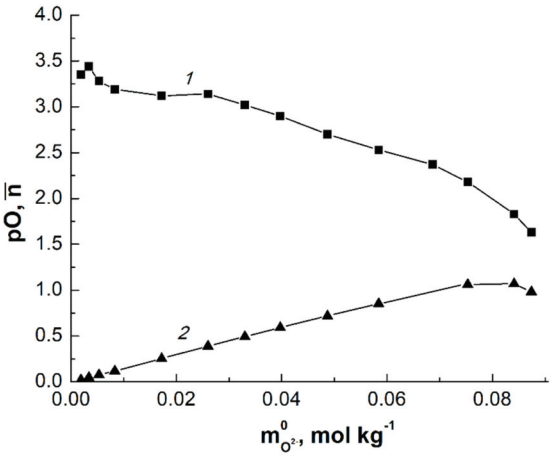

Fig. 1. The potentiometric curve obtained at the titration of $MgCl_2$ (Mg^{2+}) solution in LiCl melt with KOH (0.5 O^{2-}) at 973 K: the dependences of pO (1) and ligand number $\bar{n}$ (2) vs. initial O^{2-} addition.

the increase of O^{2-} addition up to 1 due to fixation of Mg^{2+} cations in MgO. At the equivalence point the increase stops that means that MgO does not add additional O^{2-} and is a base oxide in LiCl melt. The averaged value of MgO solubility product index is estimated as $pK_{s,MgO}$=4.57±0.05 (value 3.53 is not taken into account as close to the equivalence point) that corresponds to $pK_{s,x,MgO,LiCl}$=7.33 in mole fraction scale. The latter value gives us possibility to estimate the relative oxoacidic properties of LiCl melt at 973 K. The estimation performed according to [17] yields $pI_{L,LiCl}$=4.29, i.e. LiCl melt possesses relatively strong oxoacidic properties.

The calculated value exceeds the corresponding parameter for KCl-LiCl eutectic ($pI_{L,KCl\text{-}LiCl}$=3.50 [14] apparently due to higher content of Li^+ in the compared melts (mole fractions of Li^+ are 0.73 in KCl-LiCl eutectic and 1 in pure LiCl, respectively).

Now let us consider the data referred to Eu_2O_3 solubility in molten LiCl. The dependence of pO *vs.* amount of Eu_2O_3 added to the melt is presented in Fig. 2.

As is seen the dependence consists of 2 sections: inclined and practically horizontal. The former section corresponds to dissolution of Eu_2O_3 and the latter – to the formation of the saturated solution that prevents further Eu_2O_3 dissolution in the melt. From the obtained data the total solubility of Eu_2O_3 in LiCl melt both in form of EuOCl and in form Eu^{3+} (Eu^{2+}) can be estimated as *ca.* 3.4 mas.% (10^{-1} mol·kg^{-1}).

Table 1. The experimental and calculated data obtained at the potentiometric titration of Mg^{2+} solution with O^{2-} in molten LiCl at 973 K

m_{KOH}	pO	$m_{O^{2-}}$	$\bar{n}$	$K_{s,\ MgO}$	$pK_{s,MgO}$
0.0019	3.45	3.59^{-4}	0.02	2.27^{-5}	4.64
0.0053	3.37	4.24^{-4}	0.08	2.55^{-5}	4.59
0.0083	3.28	5.23^{-4}	0.12	2.99^{-5}	4.52
0.0172	3.21	6.18^{-4}	0.26	2.99^{-5}	4.52
0.0260	3.23	5.93^{-4}	0.39	2.34^{-5}	4.63
0.0330	3.11	7.78^{-4}	0.50	2.55^{-5}	4.59
0.0397	2.99	1.02^{-3}	0.60	2.68^{-5}	4.57
0.0487	2.79	1.62^{-3}	0.73	2.89^{-5}	4.54
0.0584	2.53	2.97^{-3}	0.85	2.85^{-5}	4.54
0.0688	2.37	4.27^{-3}	0.86	2.95^{-4}	3.53
0.0753	2.18	6.60^{-3}	1.06		
0.0841	1.83	1.47^{-2}	1.07		
0.0874	1.63	2.37^{-2}	0.98		

2. Deoxidization of LiCl and LiCl+Eu_2O_3 in reactive gas atmosphere (carbochlorination).

The action of reactive gas atmosphere is one of the most convenient ways of deoxidization of halide and especially chloride melts. The application of CCl_4 (carbochlorination) allows to remove oxide ions in a form that does not interact with the melt (unlike H_2O) and to supply melt only with its constituent anions:

$$CCl_4 \uparrow +2O^{2-} = CO_2 \uparrow +4Cl^-, \qquad (9)$$

The dependences of pO *vs.* time (curve 1) and inverse molality of O^{2-} *vs.* time (curve 2) are presented in Fig.3. They resemble similar curves obtained for KCl-LiCl melt (plateau-like section in 12–25 min time range) [18] and demonstrate the complexity of carbochlorination process from the viewpoint of formal kinetics.

Usually, for the melts with low oxoacidic properties (KCl-NaCl) the dependence of $1/m_{O^{2-}}$ *vs.* time contain a small non-linear section that corresponds to gradual saturation of the melt with CCl_4 vapor followed by a sharp linear section that corresponds to chemical reaction (9). However, Li^+ cations possess extremely strong affinity to oxide-ions and the processes of deoxidization of Li-containing melts are significantly slowed down due to titration of oxide ions by the chlorinating gas mixture (up to 17 min of the treatment). Similar behavior of molten systems has been observed, for example, in [19,20].

The plateau-like section (17–25 min of the carbohalogenation) is similar to the one observed in [17] where it was explained by ending the above-described titration of oxide ions by the chlorinating gas mixture.

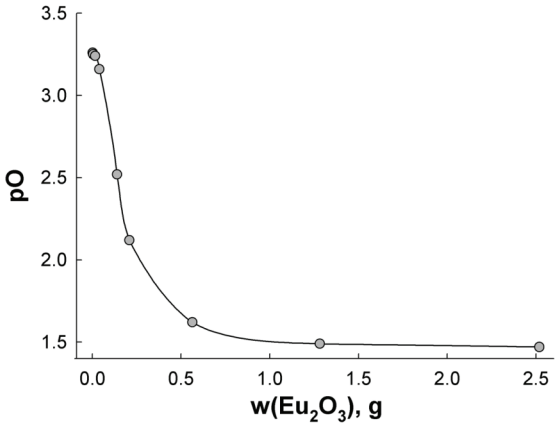

Fig. 2. The dependence of pO vs. Eu_2O_3 amounts added to the molten LiCl (37.29 g) at 973 K

The linear section which indicates the running of chemical reaction of 2nd kinetic order is situated in 44–60 min range (Fig.3b). It is described by equation $1/m_{O^{2-}} = 3.13 \cdot 10^5\, t - 1.1 \cdot 10^7$ (R_{xy}=0.99, n=5) and the slope $3.3 \cdot 10^5$ kg·mol^{-1}·min^{-1} corresponds to the reaction (9) rate constant which value is in satisfactory agreement with the cases for other melts with similar acidity.

The curve 1 achieves a plateau after 70 min which indicates that chemical reaction (9) becomes equalized by the contamination of the melt (from atmosphere or construction materials). The final pO value is 6.72 that corresponds to the purification limit of $m_{O^{2-}} = 1.9 \cdot 10^{-7}$ mol·kg^{-1}.

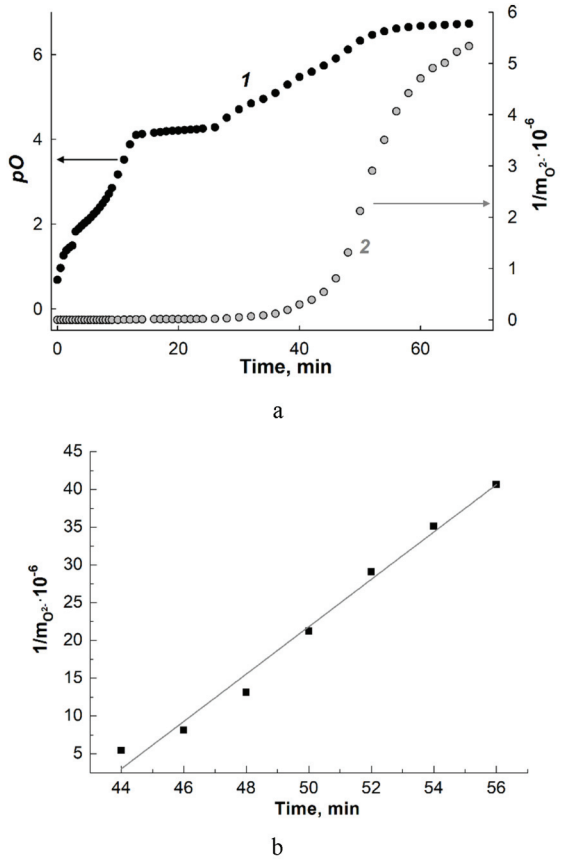

Fig.3. The dependences for carbochlorination of molten LiCl at 973 K: a) pO *vs.* time (curve 1) and $1/m_{O^{2-}}$ *vs.* time (curve 2); b) $1/m_{O^{2-}}$ *vs.* time in 44–60 min time range.

We performed the investigation of the carbochlorination process of LiCl melt (37.29 g) containing 0.1 g of Eu_2O_3, the corresponding potentiometric curve is presented in Fig. 4a.

This curve in Fig.4a can be conditionally divided in 4 sections. Section 1 corresponds the running of process (9) at relatively high oxide ion concentrations (titration of O^{2-} with CCl_4). Here the supply of Ar+CCl_4 gas mixture is the rate-determining process. The decrease of O^{2-} concentration results in the dissociation of Eu-O complexes and this process becomes the rate-determining in section 2 (30–100 min time range). These complexes are destructed within 110 min of the carbochlorination and the stage 3 (110–190 min time range) corresponding to the chemical reaction (9) which becomes the limiting stage and undergoes according to the 2nd order kinetics. Since the acidic LiCl melt additionally contains Eu^{3+} cations, i.e., is more acidic than pure LiCl, the rate constant is lower than that for the 'pure' LiCl melt, from the presented in Fig.4b linear dependence (R_{xy}=0.987, n=13) it can be estimated as

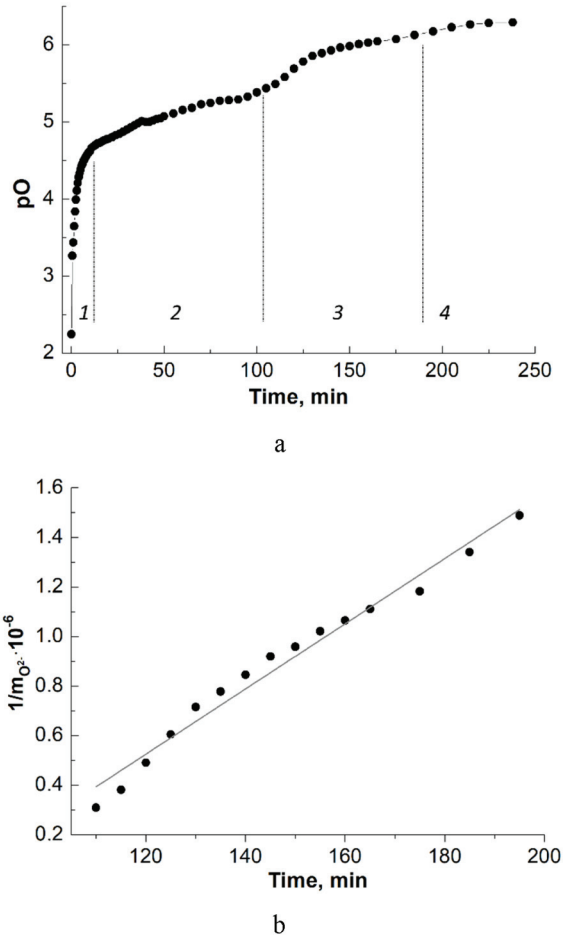

Fig.4. The experimental data of carbohalogenation of LiCl+Eu_2O_3 melt at 973 °C: a – 'pO – time' dependence, the sections: 1 – 'titration' of oxide ions with CCl_4, 2 – the dissociations of Eu-O complexes is the rate-determining reaction, 3 – the reaction (9) is the rate-determining one, 4 – the 'plateau' section; b – '$1/m_{O^{2-}}$ - time' dependence.

ca. 1.3(±0.06)·10^4 kg·mol^{-1}·min^{-1}. Finally, after the quantitative removal of oxide ions the curve reaches a plateau (section 4) at pO=6.29 (section 4, 200–240 min time range).

So, due to very long duration (4 h), the process of LiCl+Eu_2O_3 mixture carbochlorination cannot be recommended for the obtaining LiCl+Eu^{2+}(Eu^{3+}) melt for the crystal growth.

4. Conclusions

The oxoacidic (Lux-Flud acidity) properties of the LiCl melt were investigated (the oxo-basicity index, pI_L, of the lithium chloride melt was estimated to be 3.74), the solubilities of MgO ($pK_{s,MgO}$=4.57±0.05) and Eu_2O_3 (0.1 mol·kg^{-1}, 3.4 mas.%) oxides in it were determined, and the features of the carbochlorination process at 973 K were established.

LiCl melt has stronger oxo-acidic properties than the melt of the eutectic KCl-LiCl mixture ($pI_{L,\ KCl\text{-}LiCl}$ = 3.50), which is caused by the higher content of lithium cations.

The process of carbochlorination of LiCl melt containing oxide ions lasts 60 minutes, the course of the chemical stage corresponds to 2nd kinetic order and becomes limiting after 30 min of treatment, the rate constant is $3.13 \cdot 10^5$ kg·mol^{-1}·min^{-1}, and the purification limit is $1.9 \cdot 10^{-7}$ mol·kg^{-1} of O^{2-}.

During the carbochlorination of a melt containing a sample of 0.1 g of Eu_2O_3 the decomposition process of Eu oxocompounds takes 100 min, and the total duration of the process increases to 4 h. This is explained by the fact that after the dissolution of europium, the main oxo-compound in the melt is the EuO^+ complex, which has a significantly higher affinity to oxide ions.

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