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Surface Tension and Density in the KF−NaF−AlF$_3$-Based Electrolyte

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ABSTRACT: The surface tension and density of molten KF−NaF−AlF$_3$-based electrolytes were determined as a function of the melting composition and temperature by the Archimedean method and maximum bubble-pressure method, respectively. The investigated temperatures range from (1013 to 1103) K, and the total cryolite molar ratio (CR_t = ([KF] + [NaF])/[AlF$_3$]) is 1.3 to 1.41; the mole fraction of NaF ranges from 0.15 to 0.4. The effects of temperature and the content of CaF$_2$ and Al$_2$O$_3$ on surface tension and density were also investigated. The results indicate that the density and the surface tension tend to increase with the NaF content and CR_t. The density and the surface tension fit the linear relationship with temperature.

INTRODUCTION

For more than 100 years, aluminum has been produced electrochemically by the Hall−Heroult process. This technology use carbon anodes and liquid aluminum cathodes to decompose alumina dissolved in molten NaF−AlF$_3$-based cryolite,[1] in which the cryolite ratio (the mole ratio of NaF to AlF$_3$) ranges from 2.0 to 2.7. However, this process is operated at temperatures of (1183 to 1233) K, which is a high energy consumption process. Lowering the operating temperature of the cell can efficiently reduce the energy consumption. Therefore, producing aluminum at lower temperatures (below 1123 K) is a hot spot technology carried out by many laboratories.[2−6] One of the problems is to find a solvent for satisfied alumina solubility with a lower liquidus temperature and with suitable physicochemical properties.

The NaF−AlF$_3$-based cryolite with a low cryolite ratio (CR = 1.2 to 1.5) has a low liquidus temperature (973 to 1013 K). But the alumina solubility in that system is poor.[3,5,6] So the NaF−AlF$_3$-based cryolite with a low cryolite ratio is hard to be industrialized for aluminum reduction.

The KF−AlF$_3$-based electrolyte has a much lower eutectic temperature[1] and a much wider range of low-temperature liquid composition than that of the NaF−AlF$_3$ system.[3,7] Furthermore, KF−AlF$_3$-based electrolyte can increase the solubility of alumina in electrolyte, so the KF−AlF$_3$-based electrolyte is considered to be a promising electrolyte used in aluminum reduction. But due to the accumulation of sodium (which is brought by alumina in the electrolysis process) in the electrolyte, the KF−AlF$_3$ system will change into the KF−NaF−AlF$_3$ system inevitably. As a whole, the KF−NaF−AlF$_3$ system is the best possible low-temperature electrolyte for aluminum reduction.

The published physical-chemical data of KF−NaF−AlF$_3$ system are deficient. Belayaev et al.,[8] Barton et al.,[9] and Danielik and Gabcova[10] reported a partial rough phase diagram of KF−NaF−AlF$_3$ system. Alexei et al.[11] measured the liquidus temperature and conductivity at a [KF]/([KF] + [NaF]) ratio ranging from 0 to 1 and at the fixed values of ([KF] + [NaF])/[AlF$_3$] ratio (CR_t) equal to 1.3, 1.5, and 1.7. Yang et al.[12] measured the alumina solubility in KF−AlF$_3$-based electrolytes. The liquidus temperature of Na$_3$AIF$_6$-K$_3$A1F$_6$ mixture melts was studied by Chin and Hollingshead[13] and Jiawei et al.[14] Youguo et al. report the conductivity of Na$_3$AlF$_6$-40% K$_3$A1F$_6$-AlF$_3$-A1$_2$O$_3$ melts.[15] In our previous work,[16] the liquidus temperature on the KF−NaF−AlF$_3$-based electrolyte has been measured at CR_t and is 1.3, 1.41, and 1.5 with the mole fraction of NaF ranging from 0 to 0.5.

Surface tension and density are important physical-chemical properties of the electrolyte. They are very valuable for the control and computer simulation of the electrolysis process. But unfortunately, as for surface tension and density in KF−NaF−AlF$_3$-based electrolytes, there are no published data because it is a relatively new electrolyte.

EXPERIMENTAL SECTION

Chemicals. The information of NaCl, KF, NaF, CaF$_2$, MgF$_2$, and Al$_2$O$_3$ are listed in Table 1. AlF$_3$ is prepared from AlF$_3$·3H$_2$O (analytical reagents, Aladdin Reagent) by the following steps: First, aluminum fluoride trihydrate is air-blast dried at (393 to 473) K for at least 8 h, then mixed with ammonium hydrogen fluoride (analytical reagents, Aladdin Reagent), and baked at (623 to 873) K for about 8 h in an airtight furnace. The exhaust gas was absorbed by a sodium hydrate solution and a hydrochloric acid solution. After baking, the final mass fraction purity of the prepared aluminum fluoride is higher than 0.995. All reagents are vacuum-dried with P$_2$O$_5$ at 423 K for at least 4 h before experiment.

Surface Tension. The surface tension was determined from the maximum pressure in a bubble formed on the tip of a platinum capillary; this method was described elsewhere.[17] The maximum bubble pressure is reached when the bubble formed a hemisphere with a radius equal to the radius of the capillary. The surface tension can be calculated according to eq 1:

$$\sigma = \frac{R}{2}(P_{\max} - \rho g h) \qquad (1)$$

where σ is the surface tension, R is the capillary radius, $P_{\max}$ is the maximum bubble pressure measured by a pressure gauge, ρ is the

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Table 1. Chemical Information

chemical name	source	initial mass fraction purity	purification method	final mass fraction purity	analysis method
NaCl	Aladdin Reagent	0.997	recrystallizations	0.999	ICP
NaF	Aladdin Reagent	0.997	none	0.997	ICP
KF	Aladdin Reagent	0.997	none	0.997	ICP
CaF_2	Aladdin Reagent	0.997	none	0.997	ICP
MgF_2	Aladdin Reagent	0.997	none	0.997	ICP
Al_2O_3	Aladdin Reagent	0.999	none	0.999	ICP

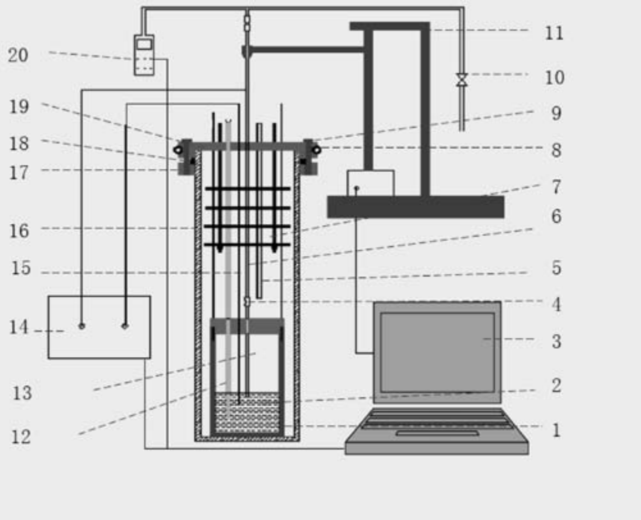

Figure 1. Schematic diagram of the surface tension measuring setup: 1, BN crucible; 2, melt; 3, computer; 4, fitting; 5, corundum pipe (Ar gas inlet); 6, stainless steel pipe; 7, thermal radiation-proof assembly; 8, copper cooling pipe; 9, fastening bolt; 10, needle valve; 11, positioner; 12, thermocouple; 13, platinum capillary; 14, LCR bridge; 15, stainless steel rod; 16, one-end-closed corundum furnace tube; 17, brass O-ring holder; 18, O-ring; 19, brass lid; 20, pressure gauge.

density of the melt, g is the gravitational content, and h is the depth of immersion of the capillary.

The schematic diagram of the measuring setup is shown in Figure 1. The measuring device is consisted of an electric resistance furnace, a digital pressure gauge, a platinum capillary and the gas pipe connected to it, a precise needle valve, a positioner, a computer, and a digital LCR bridge.

The electric resistance furnace is used to heat the melt. To seal the one-end-closed corundum furnace tube, a brass lid is tightened with a brass O-ring holder to press a fluoroelastomer rubber O ring between them by six fastening bolts. A thermal radiation-proof assembly is hung with the brass lid to ensure the homogeneity of temperature. The brass lid and the O-ring holder are kept cool by copper cooling pipe, in which flows 293 K cooling water from a chiller. All of the rods and pipes through the brass lid are fixed airtight. The temperature of melt is measured by a calibrated Pt—Pt/Rh thermocouple, whose accuracy is within ± 0.2 at 1073 K. The platinum capillary has an outer diameter of 1/8 in. and an inner diameter of 1.314 mm, which is the average diameter value in six directions determined by a metallographic microscope. The capillary is connected with a digital pressure gauge (Dwyer HM35) and a needle valve (Kofloc 2412MAT) by stainless steel tubes and fittings. The accuracy of the digital pressure gauge is ± 1.5 Pa. The precise needle valve can regulate the gas flow in the range of (0 to 3) $\text{mL}\cdot\text{min}^{-1}$. The digital LCR bridge (TH2819A) is used to monitor the exact contact point of the capillary with the liquid surface by measuring the resistance between the stainless steel tube (which is connected to platinum capillary), melt, and the stainless steel rod (which is immersed in bath). The positioner mainly consists of a controller, a servo motor, and a precise ballscrew; it can make the platinum capillary move up and down with an accuracy of ± 0.01 mm. The computer is used to control the pressure gauge, LCR bridge, and the positioner and, at the same time, collect the experimental data.

The measuring procedure of density is as follows: First, the electrolyte (the total mass is 160 g) is mixed in a glovebox under nitrogen atmosphere and then transferred to a BN crucible (45 mm in inner diameter) with a lid. Argon is used to maintain an inert atmosphere above the sample through a corundum pipe when the furnace is heated up. After the electrolyte is molten and the temperature is steady, the capillary will be lowered by the positioner. When the capillary is closed to the melt surface, the moving speed must be very slow. At the same time, the resistance is monitored by the LCR bridge; when there is a sudden change in resistance (usually from over 50 Ω to below 1.5 Ω), the positioner is stopped immediately, and this point is taken as the contact point of the capillary with the liquid surface. Usually the positioner is adjusted several times to ensure the contact point. Oxygen is selected to generate the bubble. The oxygen flow is adjusted by the needle valve to about 0.6 $\text{mL}\cdot\text{min}^{-1}$, and the bubble formation rate is about 1 bubble per 15 s. The immersing depth is controlled by the positioner. For every set temperature, the surface tension is measured at different immersion depths ((1, 2, and 3) mm), and the average value is taken as the final result.

Density. The density of the melts is measured by the Archimedean method. A platinum sphere, whose diameter is about 18.4 mm, suspended on a platinum wire 0.3 mm in diameter and attached to data-logged electronic balance (Satrious BSA-124S), is used as the measuring body. The furnace used to heat the electrolyte is the same as that used to measure the surface tension, and the BN crucible is also the same as the crucible mentioned above except the lid.

The volume of the sphere is calibrated by measuring the density of pure water at room temperature, by the following eq 2:

$$V_0 = \frac{m - m_0}{\rho_0} \tag{2}$$

where V_0 is the volume of the platinum sphere, m is the weight of the platinum sphere in air, m_0 is the weight in pure water, and ρ_0 is the density of pure water, which can be obtained from density table of pure water on the ITS-90.

The volume of the sphere at high temperature ((1013 to 1093) K) is calculated by eq 3:

$$V = V_0 + aV_0(T_2 - T_1) \tag{3}$$

Table 2. Surface Tension, σ, in Molten NaCl

T	$\sigma/\mu N \cdot m^{-1}$		deviation
K	works in ref 18	our works	%
1133	90.04	89.65	−0.43
1123	90.76	90.96	0.22
1113	91.48	92.04	0.61
1103	92.19	93.11	1.00
1093	92.91	94.06	1.24
1083	93.63	94.75	1.20

Table 3. Surface Tension, σ, in the KF−NaF−AlF_3-Based Electrolyte

	composition/wt %						T	σ
CR_t	NaF	KF	AlF_3	Al_2O_3	MgF_2	CaF_2	K	$\mu N \cdot m^{-1}$
1.3	19.6	23.8	56.6	0	0	0	1079	82.29
1.3	19.6	23.8	56.6	0	0	0	1069	83.85
1.3	19.6	23.8	56.6	0	0	0	1048	87.61
1.3	19.6	23.8	56.6	0	0	0	1033	89.52
1.3	19.6	23.8	56.6	0	0	0	1022	90.4
1.3	19.1	23.4	55.5	0	2	0	1099	77.08
1.3	19.1	23.4	55.5	0	2	0	1081	80.58
1.3	19.1	23.4	55.5	0	2	0	1058	84.9
1.3	19.1	23.4	55.5	0	2	0	1045	87.88
1.3	19.1	23.4	55.5	0	2	0	1024	90.89
1.41	19.7	25.8	54.5	0	0	0	1072	83.73
1.41	19.7	25.8	54.5	0	0	0	1066	84.62
1.41	19.7	25.8	54.5	0	0	0	1051	85.78
1.41	19.7	25.8	54.5	0	0	0	1039	87
1.41	19.7	25.8	54.5	0	0	0	1024	88.86
1.41	26.9	17.2	55.9	0	0	0	1076	93.35
1.41	26.9	17.2	55.9	0	0	0	1058	95.09
1.41	26.9	17.2	55.9	0	0	0	1045	96.7
1.41	26.9	17.2	55.9	0	0	0	1021	98.36
1.41	25.6	16.3	53.1	5	0	0	1078	101.08
1.41	25.6	16.3	53.1	5	0	0	1062	102.11
1.41	25.6	16.3	53.1	5	0	0	1046	103.08
1.41	25.6	16.3	53.1	5	0	0	1031	104.2
1.41	24.8	15.8	51.4	5	0	3	1078	107.14
1.41	24.8	15.8	51.4	5	0	3	1058	108.67
1.41	24.8	15.8	51.4	5	0	3	1049	109.5
1.41	24.8	15.8	51.4	5	0	3	1028	110.9

where V is the platinum volume at high temperature, V_0 is the calibrated volume of the platinum sphere, a is the volume expansion coefficient ($a = 0.000027$), T_2 is the melt temperature, and T_1 is the calibrated temperature. The density of the melt is calculated by the formula 4:

$$\rho = \frac{m - m_1}{V} \tag{4}$$

where ρ is the density of the melt, m is the weight of the platinum sphere in air, m_1 is the weight of it in the melt, and V is the platinum volume at high temperature.

Table 4. Coefficients A and B and Correlation Coefficient R^2 for Equation 5

	composition/wt %						empirical coefficient		
CR_t	NaF	KF	AlF_3	Al_2O_3	MgF_2	CaF_2	A	B	R^2
1.3	19.6	23.8	56.6	0	0	0	241.32	−0.142	0.99
1.3	19.1	23.4	55.5	0	2	0	283.16	−0.187	1
1.41	19.7	25.8	54.5	0	0	0	193.69	−0.103	0.99
1.41	26.9	17.2	55.9	0	0	0	192.72	−0.0922	0.99
1.41	25.6	16.3	53.1	5	0	0	171.94	−0.0658	1
1.41	24.8	15.8	51.4	5	0	3	188.78	−0.0757	1

Table 5. Density, ρ, in Molten NaCl

T	$\rho/ g \cdot cm^{-3}$		deviation
K	works in ref 19	our works	%
1143	1.517	1.521	0.28
1133	1.522	1.525	0.24
1123	1.527	1.531	0.26
1113	1.531	1.535	0.25
1103	1.536	1.540	0.24
1093	1.541	1.544	0.18
1083	1.546	1.547	0.12

The measuring procedure of density is as follows: The electrolyte (the total mass is about 160 g) is mixed in a glovebox under nitrogen atmosphere and transferred to a BN crucible (45 mm in inner diameter) with a lid. There is a 20 mm hole in the center of the BN lid to allow the platinum sphere to go through. The BN crucible is placed in the one-end-closed corundum furnace, and the setup is heated up with argon purging the furnace tube. The temperature of the bath is measured by a calibrated Pt−Pt/Rh thermocouple, whose accuracy is within ± 0.2 at 1073 K. When the electrolyte is melted completely, the platinum sphere was placed into the melt. When the reading of the balance is steady, the measurement started. The measurement is carried out in cooling process with a cooling rate of 1 $K \cdot min^{-1}$. After the experiment, the total weight change of the electrolyte is less than 1 %, so the influence of bath volatilization on composition is neglected.

■ RESULTS AND DISCUSSION

Surface Tension. The surface tension of sodium chloride was measured at (1083 to 1143) K to validate the accuracy of the testing. The obtained results are listed in Table 2. The results are in good agreement with that reported by Janz et al.,[18] and the deviation is less than 1.5 %.

The surface tension of the KF−NaF−AlF_3-based electrolyte was investigated with CR_t = 1.3 and 1.41. The mole fraction of NaF was 0.3 and 0.4. The measurement was carried out at temperatures ranging from (1023 to 1103) K. The influence of CaF_2, MgF_2, and Al_2O_3 content on the surface tension was investigated. The obtained data are listed in Table 3.

The linear dependence of surface tension on temperature was fitted as eq 5:

$$\sigma = A + BT \tag{5}$$

Table 6. Density, ρ, in the $KF-NaF-AlF_3$-Based Electrolyte

	composition/wt %				ρ at different temperatures/ $g \cdot cm^{-3}$							
CR_t	NaF	KF	AlF_3	Al_2O_3	1103 K	1093 K	1083 K	1073 K	1063 K	1053 K	1043 K	1033 K
1.3	9.41	36.00	54.59	0	1.786	1.793	1.802	1.811	1.819	1.829	1.841	1.851
1.3	16.08	28.00	55.92	0	1.846	1.850	1.861	1.867	1.873	1.878	1.884	1.891
1.3	23.08	19.59	57.33	0	1.855	1.860	1.868	1.873	1.882	1.886	1.893	1.899
1.3	15.55	27.07	54.07	3.31	1.835	1.841	1.847	1.856	1.862	1.868	1.875	1.882
1.41	9.49	38.01	52.50	0	1.800	1.807	1.820	1.824	1.832	1.843	1.854	1.871
1.41	16.21	30.00	53.80	0	1.873	1.878	1.883	1.889	1.894	1.899	1.904	1.910
1.41	23.26	21.58	55.16	0	1.890	1.894	1.898	1.905	1.910	1.913	1.920	1.924

Table 7. Coefficients a and b and Correlation Coefficient R^2 for Equation 6

	composition/wt %				empirical coefficient		
CR_t	NaF	KF	AlF_3	Al_2O_3	a	$b \cdot 10^4$	R^2
1.3	9.41	36.00	54.59	0	2.813	−9.33	0.99
1.3	16.08	28.00	55.92	0	2.558	−6.45	0.99
1.3	23.08	19.59	57.33	0	2.559	−6.38	1.00
1.3	15.55	27.07	54.07	3.31	2.580	−6.76	1.00
1.41	9.49	38.01	52.50	0	2.860	−9.63	0.98
1.41	16.21	30.00	53.80	0	2.453	−5.26	1.00
1.41	23.26	21.58	55.16	0	2.438	−4.98	0.99

where σ is the surface tension of the electrolyte, A and B are the empirical coefficients, and T is the temperature, in K. The coefficients A and B and the correlation coefficient R^2 are given in Table 4.

The data in Table 3 indicate that the surface tensions tend to increase with the NaF content and CR_t. The addition of alumina and calcium fluoride can increase the surface tension of the electrolyte, while the addition of magnesium fluoride has little influence on the surface tension. The surface tension decreases (0.7 to 1.8) $\mu N \cdot m^{-1}$ with the temperature increase of 10 K.

The surface tension relates to the separation effectiveness of gas from the melt. The higher the surface tension is, the more easily the gas separates from the melt. Therefore, improving the surface tension of the melt is beneficial to aluminum reduction, because it can make the separation of anode gas from the melt easier, which will lead to improvements on the current efficiency. Compared with conventional industrial electrolytes, whose surface tension is about 130 $\mu N \cdot m^{-1}$ at 1223 K, the $KF-NaF-AlF_3$-based electrolyte has lower surface tension. It is not favorable for aluminum reduction.

Density. To testify the accuracy of density measurement, the density of sodium chloride was measured at (1083 to 1123) K, and the obtained results are listed in Table 5. The results are in good agreement with the widely recognized data reported by Kirshenbaum et al.,[19] and the deviation is less than 0.5 %.

The $KF-NaF-AlF_3$-based electrolyte was investigated with $CR_t = 1.3$ and 1.41. The mole fraction of NaF ranged from 0.15 to 0.35. The measurement was carried out at temperatures ranging from (1033 to 1103) K with a interval of 10 K. The obtained data are shown in Table 6.

The density results fit the linear eq 6:

$$\rho = a + bT \tag{6}$$

where ρ is the density of the electrolyte, a and b are the empirical coefficients, and T is the temperature, in K. The coefficients a and b and the correlation coefficient R^2 are given in Table 7.

The data in Table 6 show that the density tends to increase with the NaF content and CR_t, and a reasonable explanation needs to be studied further. But when alumina is added, the density decreases, which is most likely because the formatted Al-O-F complex ion could enlarged the volume of melt electrolyte. The density decreases (0.05 to 0.1) $g \cdot cm^{-3}$ with the temperature increase of 10 K. Generally speaking, the measured density is lower than that of conventional industrial electrolytes (whose density is about 2.0 $g \cdot cm^{-3}$) for aluminum reduction, which is helpful for the separation of molten electrolyte and aluminum.

CONCLUSION

The surface tension and density were measured by the Archimedean method and maximum-bubble-pressure method, respectively, at temperatures ranging from (1013 to 1103) K. The electrolyte was studied with the cryolite ratio (CR_t) from 1.3 to 1.41 and the mole fraction of NaF from 0.15 to 0.4. The surface tension tends to increase with the NaF content and CR_t. The addition of alumina and calcium fluoride can increase the surface tension of the electrolyte, while the addition of magnesium fluoride has little influence on the surface tension. The density tends to increase with the NaF content and CR_t. The addition of alumina can reduce the density. The density and the surface tension fit the linear relationship with temperature.

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