

Short Communication

The Electrolyte for a Liquid Metal Battery: Liquidus Temperature, Conductivity, and Density

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Received: 13 November 2021 / Accepted: 3 January 2022 / Published: 2 February 2022

This study investigated the influence of the composition of the electrolyte used in Li-Pb,Sb liquid metal batteries on its liquidus temperature, conductivity, and density. On this basis, the appropriate electrolyte with a price advantage was selected. Moreover, the liquidus temperatures of electrolytes with different compositions used in Na-Pb,Sb liquid metal batteries were measured, and an electrolyte composition with a lower liquidus temperature was chosen. The density and conductivity results demonstrate that the selected electrolytes can meet the requirements of liquid metal batteries. The findings of this study provide data support for the selection of electrolytes for liquid metal batteries.

Keywords: Liquid metal battery, Li-Pb,Sb battery, Na-Pb,Sb battery, Liquidus temperature, Conductivity, Density

1. INTRODUCTION

Due to its clean and convenient features, electric energy has become an indispensable component of modern human production and daily life. In recent years, whether in response to the energy crisis or to ease the increasingly severe pressure on the environment, countries worldwide have set their sights on the further development and utilization of renewable energy for power generation. Therefore, the role of energy storage technology in modern power systems has become increasingly prominent. In the future, the demand for off-grid energy storage will increase [1,2], and energy storage batteries are an important development orientation for large-scale energy storage technology [3,4].

According to the working principle, engineering design, and existing research results, the new concept of the large-scale electrochemical energy storage method of liquid metal batteries put forward

by Sadoway [5] can better meet the technical requirements of the large-scale static energy storage of power grids; this has attracted widespread attention and reports from academia, industry, and government departments in the United States.

A liquid metal battery uses liquid metal as the positive and negative electrodes and molten salt as the electrolyte. In the molten state, the metal electrode and molten salt are automatically layered due to the differences in density. When the battery is discharging, the negative metal loses electrons and does work through an external circuit, and is then ionized and migrates to the positive electrode through the molten salt and forms an alloy with the positive electrode metal. When charging, the reverse process is performed [5].

Liquid metal batteries have a low cost and long service life, can be recycled, and avoid the formation of dendrites and the deformation of electrode materials [6], The liquid-liquid interface endows the battery with excellent dynamic performance, and the molten salt imparts the battery with high conductivity and low ohmic losses.

The properties of the electrolyte in a liquid metal battery have a great influence on battery performance, and the requirements of the electrolyte generally include the following: (1) a low liquidus temperature, as the operating temperature of the battery should be higher than the liquidus temperature of the electrolyte; moreover, the lower the operating temperature, the smaller the heat loss; (2) a very low ability to dissolve the positive and negative electrode metals; (3) a wide electrochemical window, as the electrolyte cannot be electrolyzed under the working voltage; generally, alkali chloride and fluoride can meet these requirements [5]; (4) a density between those of the positive and negative electrode metals to spontaneously form a three-layer structure; (5) high conductivity.

There have been many studies on the electrochemical behaviors of the anode and cathode processes of liquid metal batteries, including those in lithium-based systems [7-10], sodium-based systems [11,12], magnesium-based systems [4], calcium-based systems [13,14], and low-temperature molten salt systems [15,16]. However, there have been few investigations of the physical and chemical properties of electrolytes. In the present research, the liquidus temperature, conductivity, and density of electrolytes for use in Li-Pb,Sb and Na-Pb,Sb liquid metal batteries were measured in the temperature range of 340-530 °C. The research results provide a basis and data support for the selection and application of electrolytes in liquid metal batteries.

2. EXPERIMENTAL

2.1 Chemicals

The information on the chemicals used in this study is listed in Table 1, the compositions of the measured electrolytes are listed in Table 2 and Table 3. The purities of LiF, LiCl, KCl, LiBr, NaF, NaBr, and NaCl were 99.9%, and that of LiI was 99%; these chemicals were all purchased from Aladdin Reagent Co., Ltd. (Shanghai, China). Before the experiment, all reagents were vacuum-dried with desiccant at 150 °C for at least 4 h. Lithium halide is highly hygroscopic, so it cannot be exposed to the air when weighing. Moreover, lithium iodide will decompose when exposed to light. The operation

during weighing and mixing was as follows. The required chemicals, scales, and crucible were transferred to a glove box with $H_2O < 0.1\text{ppm}$ and $O_2 < 0.1\text{ ppm}$. The lighting switch was turned off when weighing lithium iodide. The chemicals were weighed, mixed, and then placed in the crucible in the glove box. The crucible was then wrapped with aluminum foil, after which it was quickly removed from the glove box and placed into a well furnace for measurement. The well furnace was preheated to $120\text{ }^\circ\text{C}$ in advance and maintained at this temperature, and Ar gas was introduced for protection.

Table 1. The chemical information.

Chemical	CAS Reg. No.	Suppliers	Mass fraction purity
LiF	7789-24-4	Aladdin Reagent	0.999
LiCl	7447-41-8	Aladdin Reagent	0.999
LiI	10377-51-2	Aladdin Reagent	0.99
LiBr	7550-35-8	Aladdin Reagent	0.999
KCl	7447-40-7	Aladdin Reagent	0.999
NaF	7681-49-4	Aladdin Reagent	0.999
NaBr	7647-15-6	Aladdin Reagent	0.999
NaCl	7681-52-9	Aladdin Reagent	0.999

Table 2. The composition of the measured electrolyte of Li-Pb,Sb liquid metal battery

Number	LiF (mol%)	LiCl (mol%)	LiBr (mol%)	LiI (mol%)
#1	15	30	55	0
#2	22	31	47	0
#3	14.7	35.5	20	29.8
#4	14.7	35.5	35	14.8
#5	14.7	20.5	50	14.8
#6	10	35	40	10

Table 3. The composition of the measured electrolyte of Na-Pb,Sb liquid metal batteries

Number	NaCl (wt%)	NaBr (wt%)	LiCl (wt%)	LiBr (wt%)	KCl (wt%)
#7	20	20	25	35	0
#8	8	42	20	30	0
#9	8	32	30	30	0
#10	10	20	20	50	0
#11	34.8	0	32.9	0	32.3
#12	20	0	35	0	45
#13	0	30	30	40	0
#14	0	30	40	30	0
#15	0	20	30	50	0
#16	0	15	35	50	0
#17	0	40	40	20	0
#18	0	30	50	20	0

2.2 Experimental method

Liquidus temperature. The liquidus temperature was tested via the cooling curve of the molten electrolyte. The schematic diagram of the experimental apparatus was described in detail in the authors' previous publication [17]. The procedure for the test was as follows. First, 250 g of electrolyte was evenly mixed and placed into a high-purity graphite crucible. A graphite stirrer was then inserted into the electrolyte, and the graphite crucible was then placed in a corundum furnace tube via two stainless steel rods connected to the wall of the graphite crucible. The upper open end of the corundum furnace tube was sealed by a brass assembly (consisting of a brass lid and brass O-ring Holder), between which was an O-ring. The brass assembly was cooled by circulating room-temperature water. The pipes and rods passing through the brass lid were made airtight with the lid, and Ar gas was introduced into the furnace tube during heating. After the electrolyte was molten, the agitator (Wiggens GmbH, Straubenhardt, Germany) was initiated at the rotating speed of 150 rpm. The temperature of the melt was measured by a calibrated Pt-Pt/Rh thermocouple with an accuracy of ± 0.2 K at 1073 K, which was protected with a SiC sheath (Hexoloy SE, SiC Materials, Saint-Gobain Ceramic Materials, Niagara Falls, NY, USA). When the temperature was about 50 °C higher than the estimated liquidus temperature, the furnace temperature was decreased at a rate of 1 °C/min, and the temperature-time curve was recorded by a computer. The uncertainty of the liquidus temperature results was ± 1 °C.

Conductivity. The continuously varying cell constant (CVCC) method [18] was used to test the conductivity. In this method, the conductivity of melts is calculated by Eq. (1).

$$k = \frac{1}{A\left(\frac{dR_m}{dl}\right)} = \frac{1}{A\left(\frac{dR}{dl}\right)} = \frac{1}{A\left(\frac{dZ}{dl}\right)} \quad (1)$$

When the frequency is fixed, the conductivity can be calculated by Eq. (1) by changing the length of the capillary conductivity cell and recording the corresponding total impedance. The advantage of the CVCC method is that it eliminates the influence of applied resistance and improves the accuracy of the experimental results.

The schematic diagram of the experimental apparatus is exhibited in **Fig. 1**. The experimental device consisted of a digital LCR bridge, an electric resistance furnace, a conductivity cell, and a precision riser. The electric resistance furnace was used to heat the corundum furnace pipe, which was also sealed by an O-ring and brass assembly, and cooling water was introduced into the brass lid. The pipes and rods passing through the brass lid had good airtightness with the brass lid. The conductivity cell was a pyrolytic boron nitride (PBN) pipe (inner diameter 5 mm, outer diameter 7 mm) fixed in the center of the BN crucible cover. The melt temperature was measured by a Pt-Pt/Rh thermocouple. The working electrode was a tungsten rod with a diameter of 3 mm and protected by a corundum sleeve, leaving two ends to conduct electricity. A precision riser with a 300-mm stroke was provided to move the tungsten rod. The digital LCR bridge (TH2819A) was used to read the electrolyte impedance; with the frequency set to 3-10 kHz, the impedance accuracy was ± 0.02 ohms.

The procedure for the measurement of conductivity was as follows.

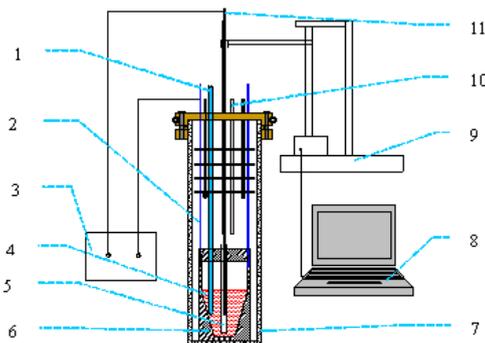


Figure 1. The experimental set-up for conductivity measurement: 1-thermocouple; 2-counter electrode; 3-digital LCR bridge; 4-melt; 5-PBN pipe; 6-high-purity graphite crucible; 7-corundum furnace tube; 8-computer; 9-precision riser; 10-corundum pipe; 11-working electrode (tungsten).

The graphite crucible filled with electrolyte was quickly transferred from the glove box to the corundum furnace tube, and the working electrode was inserted into the PBN tube. The working electrode sleeve was fixed at the riser, the corundum furnace tube was heated, and Ar gas was introduced via the corundum tube. After the electrolyte was melted and the temperature was stabilized, the working electrode was raised about 2.5 cm higher than the bottom of the PBN pipe. The tungsten electrode was moved downward in increments of 1 cm, and the corresponding total impedance was monitored by the digital LCR bridge. In most experiments, the electrode moved downward 6 to 8 times. The uncertainty of the conductivity results was $\pm 0.04 \text{ S cm}^{-1}$.

Density. The density was determined by the Archimedean method. The weight of a platinum sphere (18.4 mm in diameter) immersed in the melt was measured by a data-logged electronic balance (Satorius BSA-124S), and the sphere and balance were connected by a platinum wire (0.3 mm in diameter). The density of the melt was calculated by Eq. (2):

$$\rho = \frac{m - m_1}{V}, \quad (2)$$

where ρ is the density of the melt, m is the weight of the platinum sphere in air, m_1 is the weight of the platinum sphere in the melt, and V is the volume of the platinum sphere. The furnace used to heat the electrolyte was the same as that used to measure the conductivity, and a BN crucible (inner diameter 45 mm) with a lid was placed in the corundum furnace tube. There was a 20-mm hole in the center of the BN lid to allow the platinum sphere to pass through. The electrolyte temperature was measured by a Pt-Pt/Rh thermocouple.

The procedure for measuring density was as follows. In a glove box, 160 g of electrolyte was weighed, the electrolyte was placed into a BN crucible with a lid, and the BN crucible was then quickly placed into the corundum furnace tube. The corundum furnace tube was heated and Ar gas was introduced. After the electrolyte was molten, the platinum sphere was immersed in the melt through the hole in the center of the BN lid, and the measurement began after the reading of the balance was steady. The measurement was carried out during the process of cooling at a rate of $1 \text{ }^\circ\text{C}/\text{min}$. During the

measurement, the influence of electrolyte volatilization was neglected. The uncertainty of the density results was $\pm 0.02 \text{ g cm}^{-3}$.

3. RESULTS AND DISCUSSION

3.1. Electrolyte for the Li-Pb,Sb liquid metal battery

The halide of the negative electrode metal is generally selected as the electrolyte for use in liquid metal batteries. For Li-Pb,Sb liquid metal batteries, lithium halide mixtures are generally used as electrolytes. Li has a low melting point, and its solubility in its pure molten halide salts is low [5]. Sb is a semi-metal with high electronegativity, and Li-Sb batteries have a high open-circuit voltage (OVC) [19]. Moreover, the alloying of Pb and Sb can significantly reduce the working temperature of the battery [20], and Pb is inexpensive ($0.59 \text{ \$/mol}^{-1}$).

In the present study, the liquidus temperatures of several lithium halide mixtures were tested, and the results are exhibited in Table 4.

Table 4. The liquidus temperature of lithium halide mixtures with different compositions

Number	LiF (mol%)	LiCl (mol%)	LiBr (mol%)	LiI (mol%)	Liquidus temperature (°C)
#1	15	30	55	0	476.5
#2	22	31	47	0	442.3
#3	14.7	35.5	20	29.8	320.4
#4	14.7	35.5	35	14.8	426.4
#5	14.7	20.5	50	14.8	421.6
#6	10	35	40	10	454.4

Among them, the composition of electrolyte #3 is the composition of the eutectic component of the LiF-LiCl-LiBr-LiI quaternary system, and this electrolyte has often been used as the electrolyte of liquid metal batteries in previous studies. However, electrolyte #3 contains nearly 30 mol% of expensive LiI (about 1234 $\text{\$/kg}$); thus, the liquidus temperature of the eutectic component of the LiF-LiCl-LiBr ternary system was tested. Furthermore, the composition of electrolyte #3 was adjusted to reduce the content of expensive LiI, and the liquidus temperatures of these electrolytes were measured.

The liquidus temperatures of these electrolytes are relatively low, which meets the requirements of battery use. According to the data reported in the existing literature, the liquidus temperature of electrolyte #2 is 1.7 °C lower than a previously reported electrolyte [21], thereby exhibiting little difference. The liquidus temperature of electrolyte #3 is 20.6 °C lower than that of LiF-LiCl-LiI [21,6], and its LiI content is reduced by 29.4 mol%. Moreover, the liquidus temperature of electrolyte #3 is lower than that of a previously reported LiCl-LiBr-LiI system [6].

The liquidus temperature of electrolyte #2 without LiI is 122 °C higher than that of electrolyte

#3. The liquidus temperatures of electrolytes #4-6 are not much different from that of electrolyte #2, but considering that they contain expensive LiI, these electrolytes are not suitable for use in Li-Pb,Sb liquid metal batteries from the economic perspective.

If a lower liquidus temperature is to be pursued, electrolyte #3 can be chosen as the electrolyte for Li-Pb,Sb liquid metal batteries. However, due to its higher content of LiI, and in consideration of the fact that large-scale liquid metal batteries use more electrolytes, the selection of electrolyte #3 will result in very high costs. If lower costs are to be pursued, electrolyte #2 can be chosen as the electrolyte for Li-Pb,Sb liquid metal batteries. Therefore, the conductivity and density of electrolytes #2 and #3 were subsequently measured, and the results are subsequently discussed.

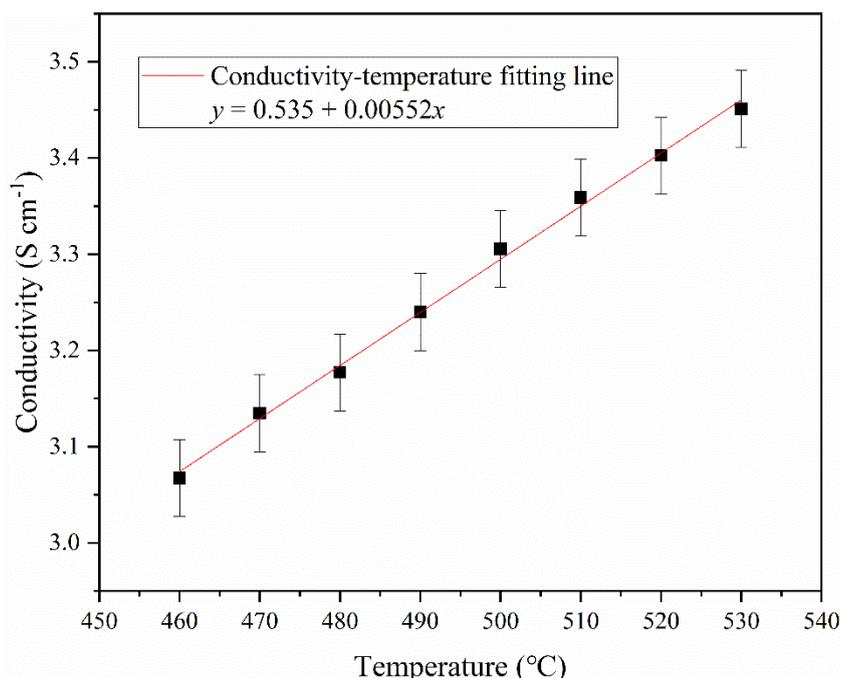


Figure 2. The conductivity of electrolyte #2 at different temperatures.

The conductivity of electrolyte #2 exceeds 3 S cm^{-1} at 460-530 °C, which indicates that it has good electrical conductivity, and when used as the electrolyte in a liquid metal battery, it can greatly increase ion mobility and maintain high energy efficiency during charging/discharging at a higher current density; thus, it meets the requirements of battery design. When the temperature is 460-530 °C, the temperature coefficient of electrolyte conductivity is $0.00552 \text{ S cm}^{-1}/\text{°C}$, and the conductivity increase by 0.0552 S cm^{-1} for every 10 °C increase in temperature.

The density of the electrolyte is very important for the battery, and can only be between the densities of the positive and negative electrode metals; under this condition, the liquid metal battery can spontaneously form a three-layer structure and work normally. The density of electrolyte #2 was therefore subsequently tested, and the results are as follows.

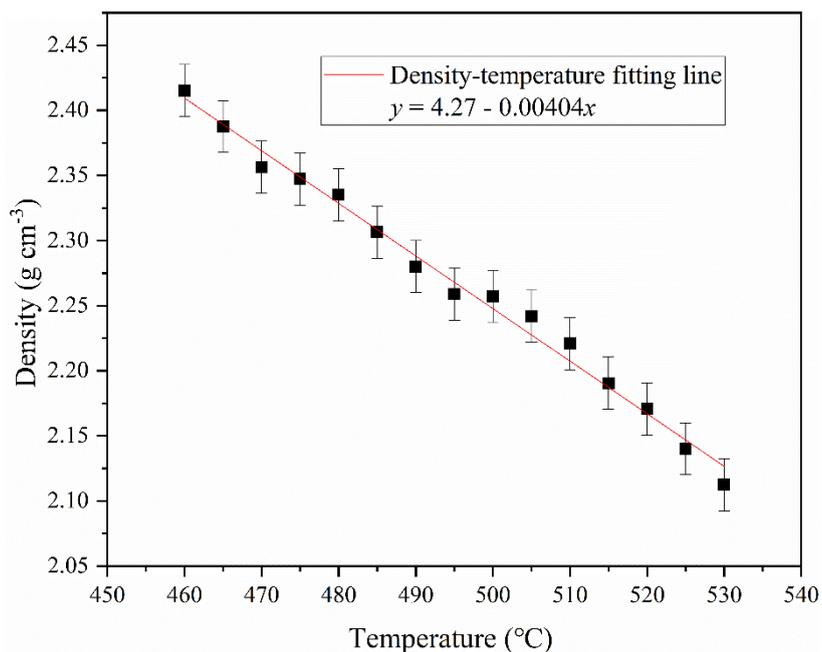


Figure 3. The density of electrolyte #2 at different temperatures.

At the temperatures of 460-530 °C, the density of electrolyte #2 is between 2.11 and 2.42 g cm⁻³. Moreover, the densities of the positive and negative electrode metals, namely Li and Pb-Sb alloy, were respectively calculated as 0.51 and 7.67 g cm⁻³; thus, the density of electrolyte #2 meets the requirements of electrolyte selection. The temperature coefficient of density is -0.00404 g cm⁻³/°C, and the density decreases by 0.0404 g cm⁻³ for every 10 °C increase in temperature.

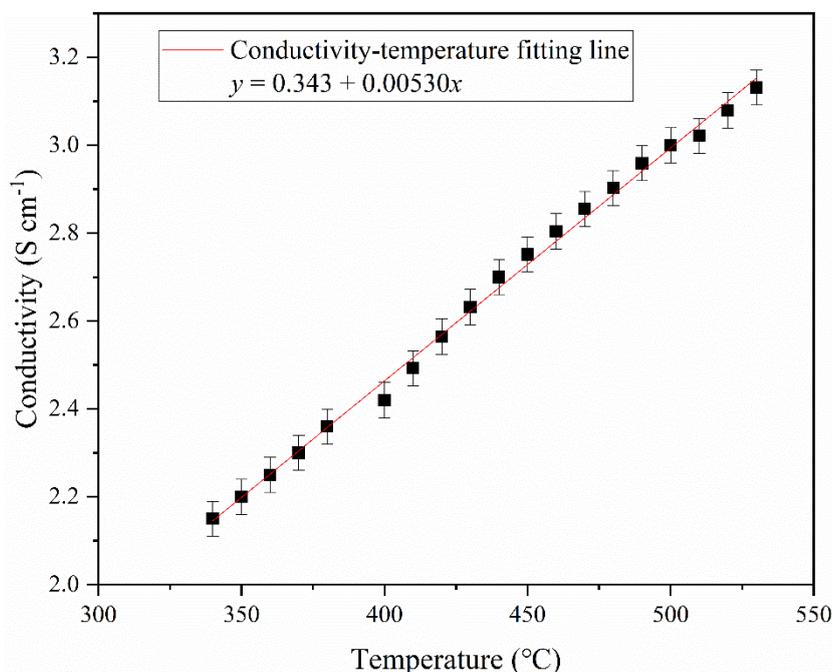


Figure 4. The conductivity of electrolyte #3 at different temperatures.

In the range of 460-530 °C, at the same temperature, the conductivity of electrolyte #3 is lower than that of electrolyte #2, which demonstrates that electrolyte #2 has better conductivity.

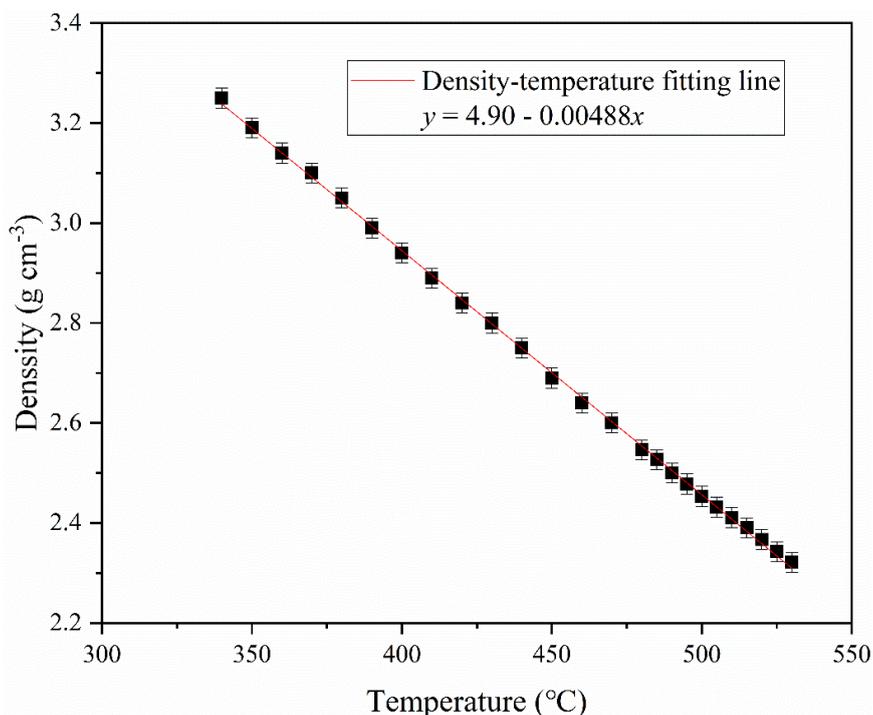


Figure 5. The density of electrolyte #3 at different temperatures.

The density of electrolyte #3 is between 2.32 and 3.25 g cm⁻³. As noted previously, the densities of the positive and negative electrolyte metals, namely Li and Pb-Sb alloys, were respectively calculated as 0.51 and 7.67 g cm⁻³; thus, the density of electrolyte #3 is in line with the requirements of the electrolyte system.

According to the test results, electrolytes #2 and #3 can both be used as electrolytes for Li-Pb,Sb liquid metal batteries, but electrolyte #2 is more economical. At the same weight, the price of electrolyte #3 is about 6 times that of electrolyte #2. From the economic perspective, electrolyte #2 is preferred.

3.2. Electrolyte for the Na-Pb,Sb liquid metal battery

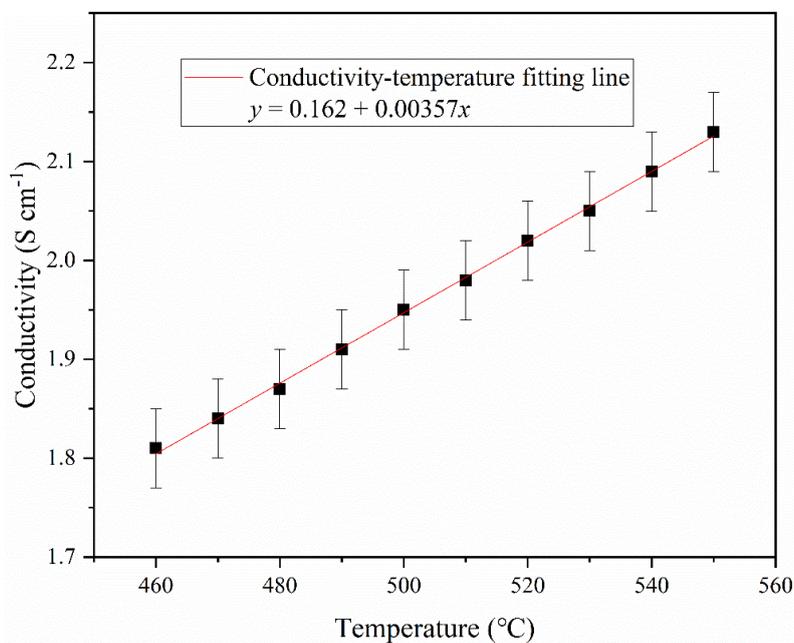
Sodium-based batteries are attractive due to their abundant reserves, low melting point, low price, and high alloying voltage [22,23].

The electrolytes for Na-Pb,Sb liquid metal batteries are similar to those for Li-Pb,Sb liquid metal batteries, and are composed of several substances including NaF, NaBr, NaCl, KCl, LiCl, and LiBr. The liquidus temperatures of some electrolytes were tested. Because these substances are highly hygroscopic, they were weighed in a glove box with H₂O < 0.1 ppm and O₂ < 0.1 ppm. The measurement process was the same as that described in Section 2.1. The liquidus temperature was measured by the cooling curve, and the test results are as follows.

Table 5. The liquidus temperatures of electrolytes with different components.

Number	NaCl (wt%)	NaBr (wt%)	LiCl (wt%)	LiBr (wt%)	KCl (wt%)	Liquidus temperature (°C)
#7	20	20	25	35	0	514
#8	8	42	20	30	0	522
#9	8	32	30	30	0	492.2
#10	10	20	20	50	0	482.5
#11	34.8	0	32.9	0	32.3	541.2
#12	20	0	35	0	45	444
#13	0	30	30	40	0	488.5
#14	0	30	40	30	0	498.4
#15	0	20	30	50	0	496
#16	0	15	35	50	0	507
#17	0	40	40	20	0	491.8
#18	0	30	50	20	0	514.3

The liquidus temperatures of the Na-Pb,Sb electrolytes for use in liquid metal batteries are generally higher than those of the Li-Pb,Sb electrolytes, and only electrolyte #12 has a lower liquidus temperature. The liquidus temperature of electrolyte #12 is 94 °C higher than that of NaCl-LiCl-KCl reported in the existing literature [12]. When the NaCl content is less than 10 mol%, the NaCl-(LiCl-KCl) system is a pseudo-binary system [12], and the liquidus temperature of this system is approximately 350 °C. However, due to the low NaCl content, the mass transfer of Na⁺ will slow down when this system is used as the electrolyte of a Na-Pb,Sb battery.

**Figure 6.** The conductivity of electrolyte #12 at different temperatures.

Based on the comprehensive consideration of the results, electrolyte #12 was initially used as the electrolyte of a Na-Pb,Sb liquid metal battery, and its density and conductivity were investigated.

According to the test results, although the conductivity of electrolyte #12 is slightly lower than those of electrolytes #2 and #3 used in the Li-Pb,Sb battery, it exhibits high conductivity ($>1 \text{ S cm}^{-1}$) [12] and can be used as an electrolyte in liquid metal batteries. The temperature coefficient of conductivity is $0.00357 \text{ S cm}^{-1}/^\circ\text{C}$, and the conductivity increases by 0.0357 S cm^{-1} for every $10 \text{ }^\circ\text{C}$ increase in temperature.

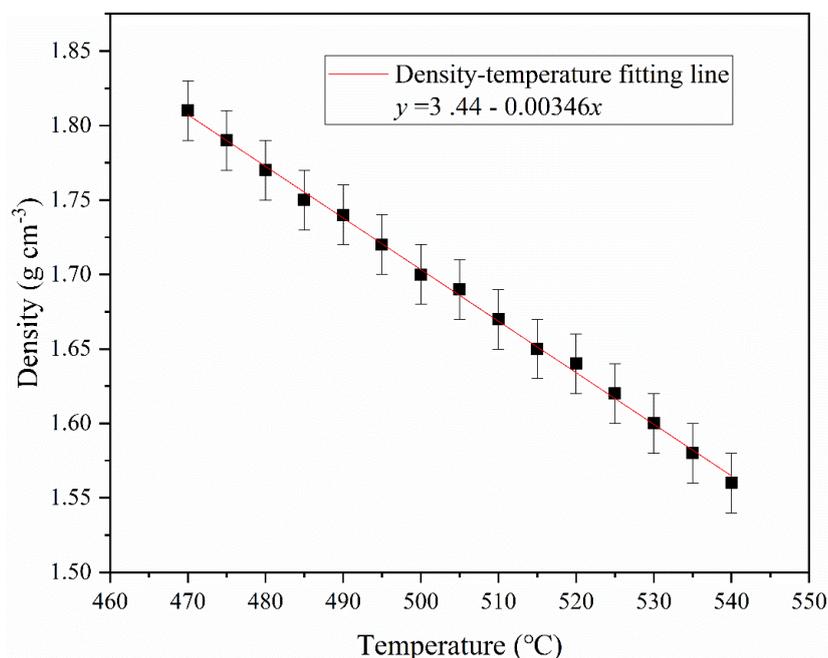


Figure 7. The density of electrolyte #12 at different temperatures.

When the temperature is $470\text{-}540 \text{ }^\circ\text{C}$, the density of electrolyte #12 is about $1.6\text{-}1.8 \text{ g cm}^{-3}$, which is between the densities of the negative electrode metal Na (0.9 g cm^{-3}) and the positive electrode alloy Pb-Sb (7.67 g cm^{-3}); thus, the selection of electrolyte #12 would be conducive to the spontaneous formation of the three-layer structure, and meets the requirements of the battery.

According to the test results, electrolyte #12 can be selected as the electrolyte for use in Na-Pb,Sb liquid metal batteries.

4. CONCLUSIONS

The liquidus temperature of the electrolytes used in Li-Pb,Sb liquid metal batteries is generally relatively low. In this study, the liquidus temperature of electrolyte #3 was found to be the lowest, namely $320.4 \text{ }^\circ\text{C}$. In consideration of the expensive LiI, electrolyte #2 was selected as a low-cost electrolyte for use in Li-Pb-Sb liquid metal batteries. Both electrolytes have high conductivity, and their densities meet

the requirements of Li-Pb,Sb liquid metal batteries. While both electrolytes can be selected as electrolytes for use in Li-Pb,Sb liquid metal batteries, the former has a lower liquidus temperature and the latter has a better economic advantage.

Moreover, the liquidus temperature of the electrolyte used in Na-Pb,Sb liquid metal batteries is generally higher than that used in Li-Pb,Sb liquid metal batteries. Electrolyte #12 has a lower liquidus temperature. The conductivity of this electrolyte is relatively low, and its density is also between those of the positive and negative metal electrodes. Therefore, electrolytes #2 and #3 can be selected as electrolytes for use in Li-Pb,Sb liquid metal batteries, from the economic perspective, electrolyte #2 is preferred. The electrolyte #12 can be selected as the electrolyte for use in Na-Pb,Sb liquid metal batteries.

ACKNOWLEDGEMENT

The financial support from NSFC 52064030 and NSFC 22068021, National key R & D plan 2019YFC1904205, and the Program for Innovative Research team in university of Yunnan province (No.201808) are acknowledged.

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