

## Thermal batteries with ceramic felt separators – Part 2: Ionic conductivity, electrochemical and mechanical properties



Seung-Ho Kang<sup>a,d</sup>, Sang Hyeok Chae<sup>b</sup>, Hae-Won Cheong<sup>a</sup>, Kyung-Ho Kim<sup>c</sup>, Yoon Soo Han<sup>c</sup>,  
Sung-Min Lee<sup>c</sup>, Dang-Hyok Yoon<sup>b,\*</sup>, Junsin Yi<sup>d,\*</sup>

<sup>a</sup> Agency for Defense Development, Yuseong P.O. Box 35-41, Daejeon 34188, South Korea

<sup>b</sup> School of Materials Science and Engineering, Yeungnam University, Gyeongsan 38541, South Korea

<sup>c</sup> Engineering Ceramic Team, Korea Institute of Ceramic Engineering and Technology, Icheon 17303, South Korea

<sup>d</sup> College of Information and Communication Engineering, Sungkyunkwan University, Suwon 14619, South Korea

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### ABSTRACT

In this Part 2, the ionic conductivity of molten salt electrolytes, the electrochemical properties of single cells containing a ceramic separator infiltrated with an electrolyte, and the mechanical strength of the electrolyte layer are compared with those of the conventional pellet-pressed structure. The ionic conductivity for the molten electrolyte is higher than that of the previous report for both LiCl-KCl and LiF-LiCl-LiBr electrolytes, which is explained by the decrease in contact resistance using a graphite electrode instead of stainless steel. The electrochemical performance of the single cells containing a ceramic felt separator assembled with Li(Si)/FeS<sub>2</sub> electrodes shows longer operating time to a cut off voltage of 1.3 V compared to the conventional MgO-contained single cell. In addition, the flexural strength of the electrolyte layer with the ceramic felt separators is in the range of 2.80–6.29 kgf cm<sup>-2</sup>, which is incomparable to that (=0.01 kgf cm<sup>-2</sup>) of the pellet-pressed conventional separator. These findings suggest that the ceramic felt separator can be an alternative to mitigate the current problems of pellet-pressed structure in thermal batteries, enhancing the mechanical strength and electrochemical properties.

### 1. Introduction

Thermal batteries, which are used as non-rechargeable power sources for military applications, adopt an ionically non-conductive solid electrolyte at ambient temperatures. The solid electrolyte is activated instantly into an excellent ionic conductor to produce electric power upon pyrotechnic ignition. Thermal batteries should be able to operate under the extreme conditions experienced in various weapons, such as high spin (~16,000 rpm), shock (16,000×g), acceleration, vibration, and wide range of temperatures (typically -55 ~ +75°C), as well as have long storage life [1,2]. Owing to the high level of mechanical stresses occurring during operation at around 500 °C, retaining the molten salt is especially important to confer sufficient power.

The pellet-pressed eutectic salts of LiCl-KCl or LiF-LiCl-LiBr are used widely as an electrolyte after blending with a binder phase in conventional thermal batteries [3,4]. Many kinds of materials have been tested as a binder phase, including SiO<sub>2</sub>, TiO<sub>2</sub>, BN, and MgO. Of these, MgO has been chosen as the best candidate owing to its stability

and compatibility with the molten electrolyte [3,5–9]. On the other hand, the compaction of electrolyte powder to a pellet, 3–5 in. diameter with a thickness of < 0.020 in., is very difficult because of its inherent fragility. Approximately 40 vol% of MgO is needed to obtain the minimum strength for handling and to retain the molten salt during operation, which is a much greater amount than what is needed for actual battery operations [10]. At the same time, the high MgO content increases the internal resistance of the thermal batteries by decreasing the relative proportion of electrolyte, which also accompanies the decrease in energy density [11].

In this sense, the incorporation of a porous and tough ceramic felt into the electrolyte layer can be a solution for enhancing the electrochemical properties by eliminating the MgO binder in the electrolyte. Moreover, the ceramic separator is believed to decrease the short-circuit possibility by retaining the molten electrolyte and by conferring sufficient strength for the electrolyte compared to the pellet-pressed structure. Although similar studies have been conducted using a glass filter, MgO fiber or porous MgO tape as a separator [12–15], no practical applications have been found.

\* Corresponding authors.

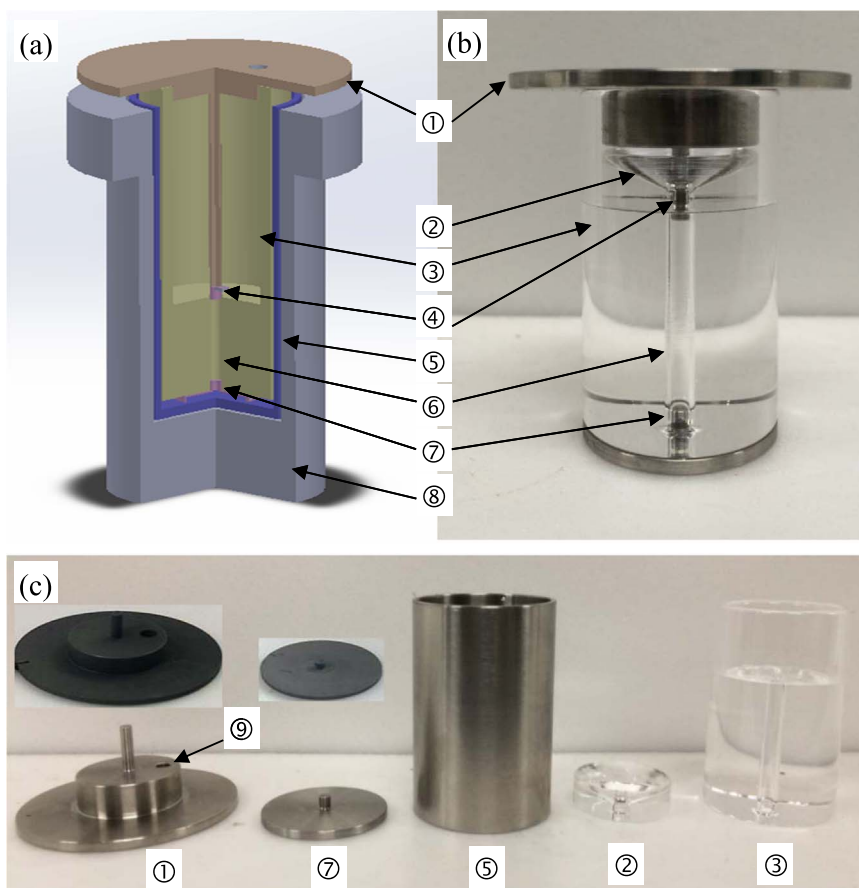
E-mail addresses: [dhyoon@ynu.ac.kr](mailto:dhyoon@ynu.ac.kr) (D.-H. Yoon), [junsin@skku.edu](mailto:junsin@skku.edu) (J. Yi).

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**Fig. 1.** Images of the (a) schematic, (b) assembled and (c) unfold parts used for ionic conductivity measurement: ① upper electrode, ② funnel disc, ③ quartz cell with capillary, ④ upper electrode tip, ⑤ lower electrode, ⑥ molten salt capillary ( $\phi=3$  mm,  $l=30$  mm), ⑦ lower electrode tip, ⑧ graphite crucible, and ⑨ thermocouple hole. For convenience, both stainless steel and graphite electrodes are shown in (c).

With the objectives of enhancing the reliability and electrochemical properties of the conventional MgO-containing pellet structure, thermal batteries with a ceramic felt separator infiltrated with the electrolyte are examined. Using 3 types of ceramic felt separators, the wetting, infiltration, and loading behavior of these felts for the LiCl-KCl and LiF-LiCl-LiBr electrolytes along with the chemical stability at a molten electrolyte environment are explained in Part 1 [16]. In Part 2, the ionic conductivity of the electrolytes, electrochemical properties of single cells containing the ceramic felt separator, and the mechanical properties of the electrolyte layer are explained in comparison with the conventional pellet-pressed structure.

## 2. Experimental procedure

### 2.1. Experimental setup and measurement of ionic conductivity

A special cell is designed for the ionic conductivity measurement of the molten LiCl-KCl and LiF-LiCl-LiBr electrolytes, which is modified from the design reported by Masset et al. [17]. Fig. 1 presents a schematic diagram and actual images of the cell used in this study. The cell assembly includes the upper and lower electrodes, which made electrical contact with the molten electrolyte stored in a quartz cell capillary. The capillary has a diameter and length of 3 and 30 mm, respectively. The cell assembly containing the electrolyte powder is placed into a graphite crucible, as shown in Fig. 1(a), while the graphite crucible is located in a small furnace for the measurements. A K-type thermocouple is placed on top of the molten electrolyte through the upper electrode.

After inserting the electrolyte powder in the capillary, the cell is kept for 30 min at 80 °C higher than the melting point of each

electrolyte to achieve thermal equilibrium. Subsequently, ionic conductivity measurements were taken at every 20 °C step upon cooling. An LCR meter (E4980A, Agilent) is used after calibration for the impedance measurement from 20 Hz to 2 MHz using a LabVIEW interface connected to a laptop. The dimensional changes due to thermal expansion of the capillary in the quartz cell are not considered when calculating the ionic conductivity because it is reported to be negligible [17]. All measurements are taken in a glove box in triplicate for each electrolyte, while maintaining a relative humidity less than 2% by flowing dry Ar gas.

The main differences in this measurement system from that used by Masset et al. [17] are the use of graphite electrode tips instead of stainless steel, a decrease in the capillary diameter to 3 mm from 6 mm, and the use of a funnel disc. These are determined based on a range of preliminary tests, which are performed to decrease the contact resistance between the electrode and molten electrolyte by enhancing the electrical contact or minimizing the chemical reaction between them. To check the reactivity of stainless steel with the electrolyte, an AISI 316 stainless steel sample is exposed to the molten electrolyte for 30 min followed by characterization by scanning electron microscopy (SEM, S-4800, Hitachi, Japan) and energy dispersive X-ray spectroscopy (EDS) attached to the SEM.

### 2.2. Preparation of the samples for the electrochemical measurements

The LiCl-KCl or LiF-LiCl-LiBr electrolyte are infiltrated into 3 types of commercial ceramics felts, high porosity Al<sub>2</sub>O<sub>3</sub> (ALF-50, 97% porosity,  $t=1.27$  mm), low porosity Al<sub>2</sub>O<sub>3</sub> (APA-03, 79% porosity,  $t=0.30$  mm), and fused ZrO<sub>2</sub> (ZYP-50, 96% porosity,  $t=1.27$  mm), as described in Part 1, and are used as an electrolyte layer. At the same

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