Electrochemical Processing of Regolith Simulant

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Introduction

ISRU is the most important subject after ISS projects. Electrochemical processing at high temperature may play an important role in ISRU, because regolith stimulant must be treated to extract oxygen, light structural metals and cement. It is also deeply relating to the global sustainability and low carbon society research.

Development of a new oxygen evolution electrode in molten fluorides is required, since molten fluorides are expected to be an electrolyte for electrochemical reduction processes such as metal extraction, metal refining and nuclear fuel reprocessing. An inert electrode available for oxygen evolution in molten fluorides at elevating temperatures has thus been investigated by many researchers. Considering sustainability of the processes, the anode composing of nontoxic and abundant materials is preferential. In this study, $FeAl_2O_4$, hercynite has been selected and attempted to be prepared by a conventional ceramic method. Structure, morphology and resistivity of the obtained sample at room temperature were studied. Electrochemical behavior of oxide ions was also investigated in a LiF-KF-NaF melt to test the possibility of a new anode.

Experimental

Electrochemical measurement in fluoride melts at temperature ranging from 723 K to 1023 K was carried out by a three electrode system under argon atmosphere. Lithium oxide (Li_2O) and lunar regolith were used as the oxide source, respectively. The prepared hercynite electrode was tested for the new anode material and Mo plate was used as a cathode. A glassy carbon rod was used as a counter electrode for all experiments. The reference electrode was an Ag^+/Ag electrode and the potential of this reference electrode was calibrated with reference to deposition potential of potassium.

Results and Discussion

Electrochemical behavior of metal oxides in molten fluoride systems

The regent grade raw materials of γ-Al_2O_3 and α-Fe_2O_3 in molar ratio of 2.0 were mixed with alcohol and calcined at 1123 K under a N_2–10vol% H_2 flow for 2 h to form $FeAl_2O_4$ powder. The obtained sample was compacted by CIP of 200 MPa to obtain a square-pole shape. The compact was then sintered at 1723 K for 8h in a N_2 - 0.1 vol% H_2 gas flow. The samples were characterized by an x-ray diffraction. As shown in fig. 1, all diffract peak positions of the sample were coincided

with a spinel-type structure, which indicated the obtained sample composed of single-phase hercynite $FeAl_2O_4$. From the SEM image of the fracture surface of the sample, intergranular pores of about 3 μm was observed, which implied that the rate of bulk formation from powder was too fast. We have used hercynite as anode material and studied the electrochemical behavior of oxide ions on the anode in a LiF-NaF-KF melt by means of electrochemical measurements.

From the polarization curve of the diamond electrode in Fig. 2, anodic current increases from 2.0 V to 4.0 V after addition of 4 mol% Li_2O. The observed current corresponds to the oxidation of electrochemical active species in the melt. On the other hand, the current before adding oxide may be attributed to the fluoride film on the anode surface. The increase current after adding Li_2O is considered to correspond to oxygen gas evolution.

Fig.3 shows the relationship between the concentration of oxide ion and the rest potential of the hercynite electrode under argon pressure of 1 atm. The potentials vary linearly with the additive amount of oxide ions. The slope corresponded to the theoretical value of a one-step two electrons reaction between $O_{(in\ hercynite)}$ and $O^{2-}_{(in\ the\ melt)}$;

O(in hercynite) + 2e$^-$ = O^{2-}.
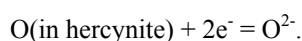
This result also indicates that the hercynite electrode is inert for oxygen gas evolution in LiF-NaF-KF melt.

Metal extraction from regolith

In order to investigate the electrochemical behavior of lunar regolith, cyclic voltammetry was conducted in a molten fluoride at 1023 K. On the cathodic sweep, cathodic wave was observed at more negative potentials than -1.24 V, which correspond to the electrodeposition of metallic cations dissolving from the regolith. Based on the results of cyclic voltammetry, the sample was prepared by potentiostatic electrolysis using Mo electrode at -1.24 V for 1 h in the fluoride melt containing lunar regolith at 1023 K. After the 1 h electrolysis, the whole surface of the Mo electrode changed from silver to gray color and it is confirmed that the Mo electrode was covered with deposit.

EDS was therefore conducted in order to investigate the surface condition of the sample. X-ray fluorescence corresponding to magnesium atoms was observed from the sample. This results supports that the regolith in the melt is dissolved and the dissolved elements can electrochemically deposited.

Conclusions

A hercynite electrode ($FeAl_2O_4$) was prepared as a potential inert anode for oxygen gas evolution in molten fluorides containing oxides by a conventional ceramic method. In a molten LiF-NaF-KF with the use of hercynite at 1023 K, the rest potentials logarithmically decreased with increasing the additive amount of lithium oxides, which indicated that the potentials corresponded to the following reaction; O(in hercynite) + 2e$^-$ = O^{2-}.

Metal extraction from regolith was confirmed by conducting electrodeposition in the fluoride melts containing lunar regolith.

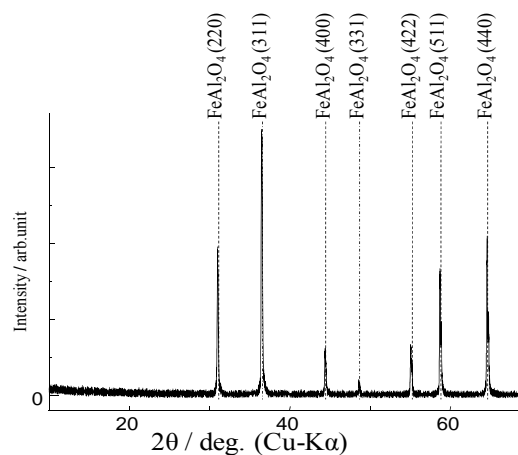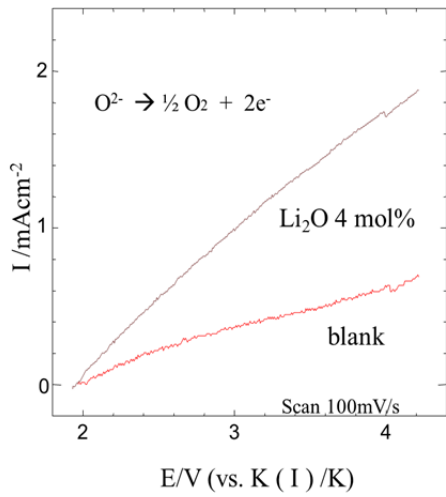Fig. 1 XRD pattern of the sample.

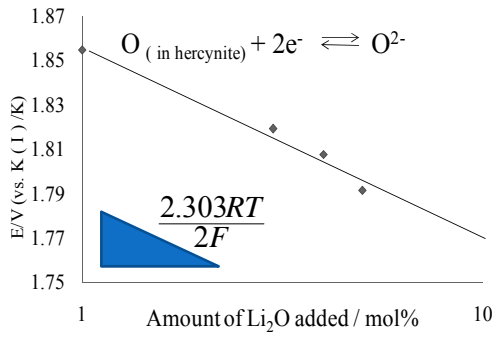Fig.2 Polarization curve of hercynite in
LiF-KF-NaF-Li$_2$O 4mol%.

Fig.3   Nernstian plots of EMF between O$_{(in\ hercynite)}$ and
O$^{2-}_{(in\ the\ melt)}$•