A Study for an Electrolytic Reduction of Tantalum Oxide in a LiCl-Li_2O Molten Salt

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1. Introduction

Korea Atomic Energy Research Institute (KAERI) has developed the Advanced Spent Fuel Conditioning Process (ACP) to be an innovative technology for handling the PWR spent fuel. As part of ACP, the electrolytic reduction process (ER process) is the electrochemical reduction process of uranium oxide to uranium metal in a molten salt. The ER process has advantages in a technical stability, an economic potential and a good proliferation resistance. KAERI has reported on the good experimental results of an electrochemical reduction of the uranium oxide in a 20 kg HM/batch lab-scale [1]. The ER process can be applicable to the reduction of other metal oxides. Metal tantalum powder has attracted attention for a variety of applications. A tantalum capacitor made from superfine and pliable tantalum powders is very small in size and it has a higher-capacitance part, therefore it is useful for microelectronic devices [2]. By the ER process the metal tantalum can be obtained from tantalum pentoxide. In this work, a 40 g Ta_2O_5/batch electrochemical reactor was used for the synthesis of the metal tantalum. From the results of the cyclic voltammograms for the Ta_2O_5-LiCl-Li_2O system, the mechanism of the tantalum reduction in a molten LiCl-Li_2O salt system was investigated. Tantalum pentoxide is chemically reduced to tantalum metal by the lithium metal which is electrochemically deposited into an integrated cathode assembly in the LiCl-Li_2O molten salt. The experiments for the tantalum reduction were performed with a chronopotentiometry in the reactor cell, the reduced products were analyzed from an analysis of the X-ray diffraction (XRD), scanning electron microscope and energy dispersive X-ray (SEM-EDX). From the results, the electrolytic reduction process is applicable to the synthesis of metal tantalum.

2. Experimental

The electrolytic reduction experiments were carried out in a 40 g Ta_2O_5/batch scale electrochemical reactor, which was composed of the resitance furnace, K-type thermocouple, alumina and magnesia crucible, anode, cathode, reference electrode and an Ar gas feeder etc. Electrolytic reactor cell was a stainless steel crucible fitted with an alumina crucible liner. The cathode was an assembly of a stainless steel conductor and a porous magnesia crucible loaded with tantalum pentoxide. The porosity of the magnesia membrane was 22~25 %. The anode of 8 mm diameter was a platinum tube of 1 mm thick. Three platinum anodes were used. A platinum rod of 3 mm diameter was used as a quasi-reference electrode for measuring the cathode and anode potentials. The WMPG 1000 Multichannel Potentiostat/Galvanostat from WonA-Tech Co. was used for the electrochemical experiments. The WMPG 1000 Ver.3.00 software was also used for electrochemical control and data acquisitions. The electrolytes used were anhydrous 20-mesh 99.6 % purity LiCl and 99.5 % purity Li_2O powder from Alfa AESAR Co. Tantalum pentoxide used was > 8 μm powder.

3. Results and Discussions

The cyclic voltammetry was applied to the electrolytic reduction cell to measure the reduction potentials of the Li_2O and Ta_2O_5. The Li_2O and Ta_2O_5 were reduced to metals at the cathode, and the oxygen ions were reduced to oxygen gas at the anode. Fig.1 shows the cyclic voltammogram of the LiCl-3wt% Li_2O molten salt system at 650°C. The reduction potential of Li_2O was -1.80 V at the cathode (peak A), where the Li ion started to be reduced to lithium metal. And the lithium metal was oxidized at -1.52 V (peak B). Fig.2 shows the cyclic voltammogram of the Ta_2O_5-LiCl-3wt% Li_2O system. As shown in Fig.2, the reduction potentials of the Ta_2O_5 and Li_2O were -1.17 V and -2.13 V (peak A and B), respectively. The reduction potential The reduction potential of Li_2O is similar to the results of the LiCl-3wt% Li_2O system in Fig.1. The tantalum and lithium metal was oxidized at -0.60 V and -1.66 V, respectively (peak A′ and

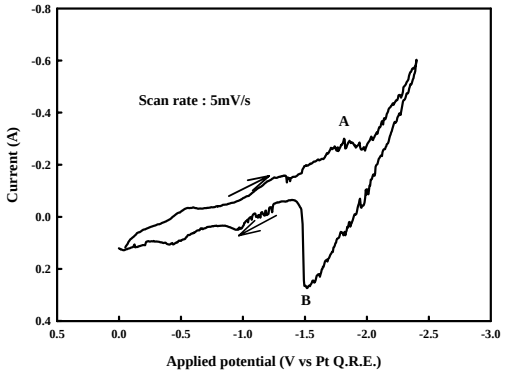

Fig. 1. Cyclic voltammogram of the LiCl-3wt% Li_2O molten salt system at 650°C.

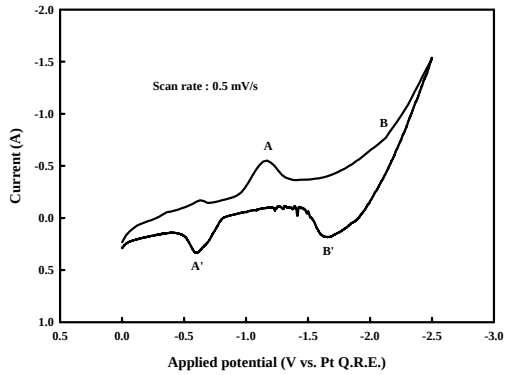

Fig. 2. Cyclic voltammogram of the LiCl-3wt% Li_2O-Ta_2O_5 system at 650°C.

B′). From the results of the cyclic voltammetry of the Ta_2O_5-LiCl-3wt%Li_2O system, Ta_2O_5 is chemically reduced by the electrolytic reduced lithium metal in less than a -2.13 V cathode potential. Fig.3 shows the chronopotentiogram of the reduction of tantalum pentoxide in LiCl-3wt% Li_2O at 650 °C. The potential of the anode increased rapidly at the end of the reaction. At that time, the reaction of the reduction was thought to be terminated. The reduced product was analyzed with SEM-EDX. Fig.4 shows the results of the SEM method. The result of the EDX analysis at the surface of the reduced product was pure tantalum metal. From the results, tantalum metal was successfully obtained from the tantalum oxide in LiCl-3wt% Li_2O molten salt by the ER process.

REFERENCES

[1] S.Y. Park et al., Development of Advanced Spent Fuel Management Process, KAERI/RR-2427/2003, 2003.
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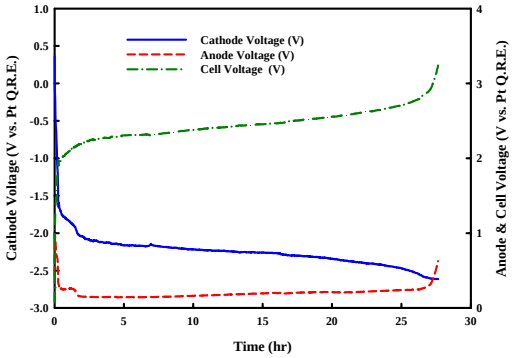

Fig. 3. Chronopotentiogram of the electrolytic reduction of Ta_2O_5 in LiCl-3wt% Li_2O molten salt at 650°C. The applied current is 1.0 A.

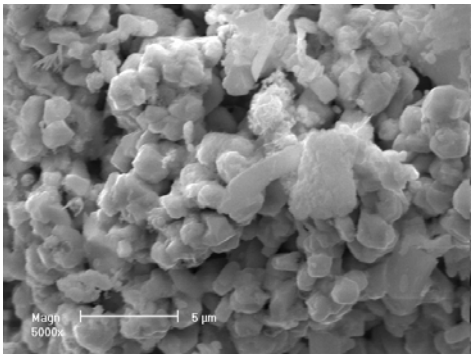

Fig. 4. Surface SEM image of the reduced product of the electrolytic reduction of Ta_2O_5 in LiCl-3wt% Li_2O molten salt at 650°C.