Chemical Reaction Complex Processes Research Section

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1. Introduction

In this research section, we study electrochemistry and materials science. We also apply them to the development of new metal production methods, new metal plating processes, and new rechargeable batteries.

In this fiscal year, we have researched a new terbium production method using molten salt electrolysis, a tungsten film plating process using molten salts, and potassium-ion batteries using ionic liquids.

2. Development of New Tb Production Method Using Molten Salt Electrolysis

Terbium (Tb) is added to neodymium magnets used in motors for electric vehicles (EVs) and hybrid electric vehicles (HEVs) to prevent deterioration of their magnetic properties at high temperatures. Tb metal added to neodymium magnets is currently produced by metallothermic reduction. As a novel method to produce Tb metal, we focused on the electrochemical reduction of Tb(III) ions using a liquid Zn electrode, followed by volatile separation and vacuum melting to obtain metallic Tb. In this fiscal year, we investigated the electrochemical formation of Tb–Zn alloys at the liquid Zn electrode in molten LiF–CaF_2–TbF_3 at 1123 K.

Cyclic voltammetry was performed in molten LiF–CaF_2–TbF_3 (0.30 mol%) at 1123 K. At the Mo flag electrode, a reduction current and the corresponding oxidation current peak were observed at approximately 0.2 V (vs. Li^+/Li), which were attributed to the deposition and dissolution of Tb metal, respectively. At the liquid Zn electrode, the reduction current increased at approximately 1.1 V. Since the potential was considerably more positive than the Tb metal deposition potential, the reduction current is likely due to the formation of a Tb–Zn alloy. To confirm the electrochemical formation of the Tb–Zn alloy, galvanostatic electrolysis was performed in molten LiF–CaF_2–TbF_3 (1.0 mol%) at 1123 K using a liquid Zn electrode at –100 mA cm^{-2} for 125 min. Fig. 1 shows the cross-sectional SEM image of the sample after electrolysis [1]. The compositions at points (1) and (2) were determined by EDX analysis and exhibited Tb:Zn ratios of 21:79 and 16:84 mass%, respectively. These results indicate that a liquid Tb–Zn alloy was successfully formed by galvanostatic electrolysis at –100 mA cm^{-2} using a liquid Zn electrode.

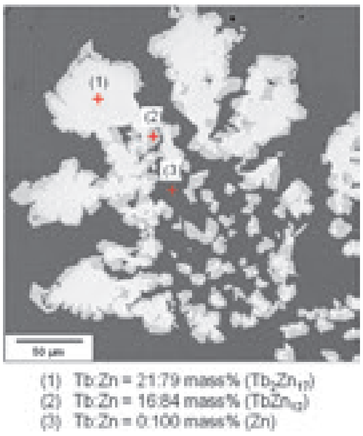

Fig. 1. Cross-sectional SEM image with the EDX analysis results of the sample after galvanostatic electrolysis in molten LiF–CaF_2–TbF_3 (1.0 mol%) at 1123 K [1].

3. Development of W film Plating Process Using Molten Salt Electrolyte: Effect of O^{2-} Ion Concentration on the Crystal Structure of W Films

Tungsten (W) is a metal with superior properties, such as heat resistance, high strength, and low thermal expansion. Therefore, there are many applications. In particular, W is expected to be used as divertor material in nuclear fusion reactors. However, its hardness and brittleness make it difficult to process into complex shapes and conventional tungsten processing methods are costly. Thus, the electrodeposition in molten salts was explored as an alternative processing method [2]. We have succeeded in electroplating extremely smooth tungsten films by electrodepositing β-W, which is a metastable phase [3]. We have also investigated the relationship between the crystal structure of electrodeposited W and the oxygen content in the film, and reported that 1.82–6.65 at% oxygen is present in electrodeposited β-W films [4]. We predicted that the existence of O^{2-} ion in molten salt has an influence on the crystal structure. To investigate the effect of O^{2-} ion, W electrodeposition was performed in molten CsF–CsCl–WCl_6 at 823 K before and after the addition of Li_2O.

W was electrodeposited in molten CsF–CsCl–WCl_6(1.0 mol%) at 1.2 V vs. Cs^+/Cs for 2 min and in molten CsF–CsCl–WCl_6(1.0 mol%)–Li_2O(1.0 mol%) at

−20 mA cm^{-2} for 75 min. Fig. 2 shows the appearance and SEM images of the electrodeposited sample. The sample obtained before the addition of Li_2O (Fig. 2(a)) was grey in color, and granular deposits were observed on the surface. On the other hand, the sample obtained after the addition of Li_2O (Fig. 2(b)) had a black metallic luster, and no granular deposits were observed on the surface. XRD analysis confirmed that the sample obtained before the addition of Li_2O was α-W, and that the sample obtained after the addition of Li_2O was β-W. From the above, it is suggested that the surface structure and crystal structure of the electrodeposited W film are affected by the existence of oxide ions.

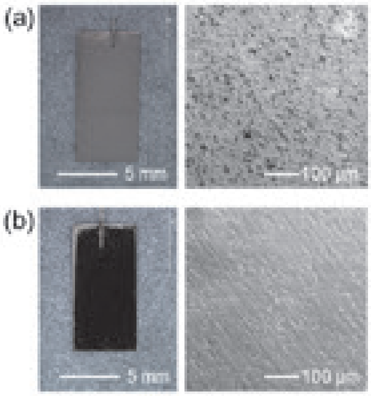

Fig. 2 Optical and SEM images of the samples obtained by (a) potentiostatic electrolysis at 1.2 V for 2 min in molten CsF–CsCl–WCl_6 (1.0 mol%) and (b) galvanostatic electrolysis at −20 mA cm^{-2} for 75 min in molten CsF–CsCl–WCl_6 (1.0 mol%)–Li_2O (1.0 mol%) at 823 K.

4. Development of Potassium-ion Batteries Using Ionic Liquid Electrolytes

Towards the further spread of renewable energy resources such as solar and wind power, it is necessary to install electric load leveling systems combined with large-scale batteries. Although lithium-ion batteries are being used in electric vehicles, there are remaining risks of supply instability of scarce resources such as lithium, cobalt, and nickel. Moreover, flammable and volatile organic solvents used in electrolytes may induce serious accidents of large-scale batteries. Thus, we have developed potassium-ion batteries with ionic liquid (IL) electrolytes because potassium resources are abundant in the Earth's crust and ILs possess high safety of nonflammability and negligible volatility [5]. Instead of cobalt and nickel used in lithium-ion batteries, we have focused on iron- and manganese-based positive electrode materials [6,7].

Fig. 3 shows charge–discharge performance of $K_{0.46}MnO_2$ positive electrode using K[FSA]–[C_3C_1pyrr][FSA] IL electrolyte (FSA = bis(fluorosulfonyl)amide, C_3C_1pyrr = *N*-methyl-*N*-propylpyrrolidinium) at 298 K. The $K_{0.46}MnO_2$ electrode exhibits stable discharge capacities of 55–60 mAh g^{-1} in the initial 50 cycles. Although the capacity gradually decreases to 50.3 and 39.4 mAh g^{-1} after 100 and 400 cycles, respectively, no significant increase of voltage polarization is confirmed in discharge curves, indicating the feasibility of $K_{0.46}MnO_2$ positive electrode in the IL electrolyte.

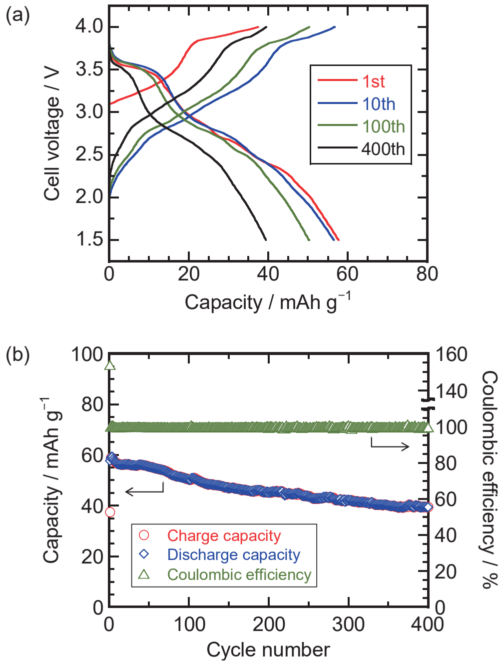

Fig. 3. (a) Charge–discharge curves and (b) cycling properties of $K_{0.46}MnO_2$ positive electrode at 100 mA g^{-1} [7]. Electrolyte: K[FSA]–[C_3C_1pyrr][FSA] (molar fraction: x(K[FSA]) = 0.20). Temperature: 298 K.

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