Anodic Behavior of Mg Alloy (AZ31) in Ionic Liquid

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The magnesium anode is used as a consumptive electrode for the electroorganic synthesis. However, the research on the anode reaction and the surface morphology in organic solution is seldom found. As a result of examining the oxidation of pure magnesium in the mixed electrolytic solution of THF and DMF, it was found that the porous oxide layer is deeply formed on the surface in the solution with the slight water (>0.25 %)¹¹. It was clarified that trace amount of water is difficult to affect Mg oxidation in the mixed electrolytic solution of the 1,3-dimethyl -2- imidazolidinone (DMI) and THF²⁾. When magnesium alloy (AZ31) was anodized in the mixed electrolytic solution of DMI and THF, the surface was considerably rough³⁾.

Recently, the ionic liquid with the properties of chemical stability, no steam pressure, incombustibility, high polarity and ionic conductivity is noticed.

In this work, the electrochemical behavior and surface state of Mg-alloy (AZ31) anode were investigated in ionic liquid.

Disk-shaped magnesium alloy (AZ31) containing 3 % aluminum and 1 % zinc was used as a working electrode. The electrode (surface area 2.01 cm²) was used for the experiment immediately, after the surface was polished on #2000 polishing paper and with alumina powder (40nm particle size), and in addition, it performed ultrasonic cleaning in the IPA. The Teflonmade cell, consisting of anode and cathode 54 mm apart, was used. Ag/AgCl electrode was used through liquid junction as a reference electrode.

N-butyl-pyridinium bis(trifluoromethanesulfone) imide being ionic liquid was used as an electrolytic solution. Using the commercial product, the reagent was not refined.

The electrolysis with the constant current density was carried out at the room temperature, and the cell voltage and anode potential etc. was measured. After the electrolysis, the weight loss of the magnesium alloy anode was measured, and the surface was observed with FE-SEM (JEOL, JSM-6500F) and DFM (SII).

Time dependence of cell voltages at various current densities was examined. At 1 mAcm⁻², a dramatic rise in cell voltage was not observed. The cell voltage rises with electrolysis time at >2.5 mA cm⁻² in the ionic liquid. The rise of the cell voltage quickens with the increase in the current density.

The anode potentials were measured after electrolysis for 30 seconds at various current densities. At 2 mAcm⁻², the anode potential remarkably rose. Over 3 mAcm⁻², it became almost constant value. At < 1.5 mAcm⁻², the anode potential remarkably lowered, when the current density was lowered from 5 mAcm⁻². On second cycle, the anode potential remarkably rose at > 0.75 mAcm⁻². It was found that the rise of the cell voltage is caused by the rise in the anode potential. In the electrolysis, it seems to cover the anode surface for the remarkably low electroconductive product.

The relationship between weight loss of Mg alloy (AZ31) and electric quantity during the electrolyte was examined at various current densities (1, 2, 2.5 and 5 mAcm⁻²). In spite of the high overvoltage, Mg-alloy anode was oxidized to Mg^{2+} ions in the ionic liquid.

The surface of magnesium alloy after the anodic oxidation at 5 mAcm⁻² for 5 minutes was observed with FE-SEM. It was found that large number of minute concaves exist in the anode surface after the electrolysis at 5 mA cm⁻². It was found that the concave is crater state of the about 250nm diameter and 50nm depth using DFM observation.

Reference

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