Operando SEM observation of corrosion process of Al foil

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Aluminum (Al) foils start to corrode by making pits through a protective oxide film when immersing them in sodium chloride solutions. This process has been investigated by electrochemical methods such as cyclic voltammogram, which provides average information.[1] However, due to the lack of local structural information, it is still unclear which sites are preferred for pitting corrosion formation. Transmission electron microscope (TEM) has been used as a tool to obtain local structure information even in a solution, since it has a high spatial resolution. However, it has disadvantages that the sample preparation is not easy, the electron beam irradiation effect is severe, and so on. On the other hand, scanning electron microscopy (SEM) seems to be better than TEM in the operando observation of corrosion process in a solution.

In this study, our aim is to develop an electrochemical cell for a scanning electron microscope (SEM) in order to achieve operando SEM observation of corrosion process of Al foil. Before starting operando SEM observation, ex-situ observation was first performed. Fig. 1(a) shows the optical microscope images of the initial Al foil and of pitting corrosion formed on the Al foil in a 4.3 wt% NaCl solution. Fig. 1(b) is the cyclic voltammetry curve with the Al foil as a working electrode. The voltage was varied from –1.5 V to 0.5 V at a scan speed of 0.05 V/s. From the CV curve, the current was observed to start increasing sharply around –800 mV. Simultaneously, the pitting formation was also observed to start. Therefore, the pitting potential was estimated to be about –800 mV in this condition.

The next step would be operando SEM observation. The schematics of the electrochemical cell used is shown in Fig. 1(c), which has two gold electrodes as counter and working electrodes. A tiny Al foil is attached to the working electrode. After filling the cell with a NaCl solution, the cell is sealed by the custom-made Si chip with an observation window. The pitting will be observed in conjunction with the pitting potential of the CV curve.

[1] Jadranka Malina, Jagoda Radošević, Zaštita materijala **56** (2015) 47-51.

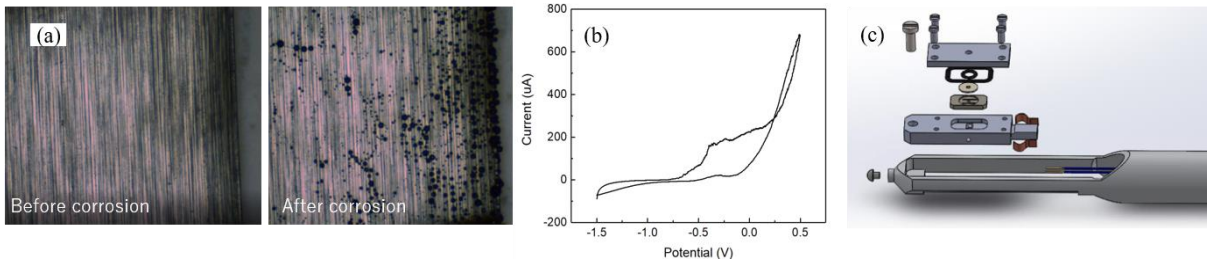

Fig. 1 (a) Optical microscope images of the initial Al foil and of that with pitting corrosion after immersion in a 4.3 wt% NaCl solution. (b) CV curve for the Al foil as a working electrode, and (c) electrochemical cell for the operando SEM, schematically.