

The electrochemical property of the electrodeposited magnetite electrode with different pH values

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1. Introduction

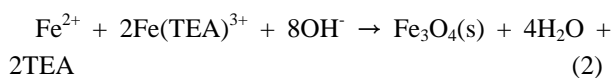
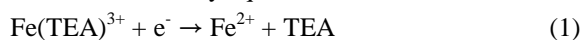
Flow accelerated corrosion (FAC) causes severe damage to secondary piping systems. An accident from FAC occurred at the Oyster Creek nuclear power plant (NPP) in 1978. Other NPPs such as Surry 2 and Mihama 3 have also experienced an FAC that induced damage to the carbon steel piping [1]. FAC is influenced by many factors such as the water chemistry (temperature, pH, dissolved oxygen (D.O.) in a solution, and etc.), chemical composition of carbon steel, and fluid dynamics [2]. Magnetite is formed at the inner surface of carbon steel, and protects the integrity of pipes from damage. The magnetite has a stable state at each equilibrium condition, so that it can be dissolved into the fluid under conditions that satisfy the equilibrium state. The iron solubility can be calculated by considering the reaction equilibrium constants for prediction of the change in the magnetite layer. On the other hand, it is necessary to measure the experimental solubility to compare the theoretical data and the experimental data. In addition, the solubility of magnetite can be predicted by measuring the electrochemical experiments. However, there are few studies related to the electrochemical property of magnetite owing to the difficulty of the electrode fabrication [3-5].

In the present work, a magnetite electrode was prepared using the electrochemical-assisted precipitation method, and the electrochemical property of the fabricated magnetite electrode was measured in an alkaline solution.

2. Method and Results

2.1 The fabrication of magnetite electrode

The solid magnetite electrode was fabricated by the electrochemical-assisted precipitation method according to the literatures [6-8]. The electrochemical-assisted precipitation reaction follows two step process, and it can be summarized by equation 1 and 2



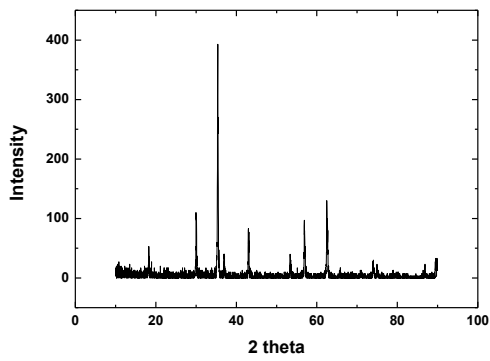
Where TEA is Triethanolamine. The concentrations of the electrodeposition bath were 80g/L NaOH, 20g/L TEA, 60g/L $\text{Fe}_2(\text{SO}_4)_3$. The temperature of the

electrolyte was maintained at 60°C. The current density was 5 mA/cm² and the electrodeposition time was 120s. The Pt wire anode was used, and the Ni or Pt wire used as the substrate. The fabricated magnetite electrode was characterized by using X-ray Diffraction (XRD). And it was shown in Fig. 1. The observed peaks in the spectrum were characteristic of magnetite, although there are some different peaks from other components, e.g. Ni substrate.

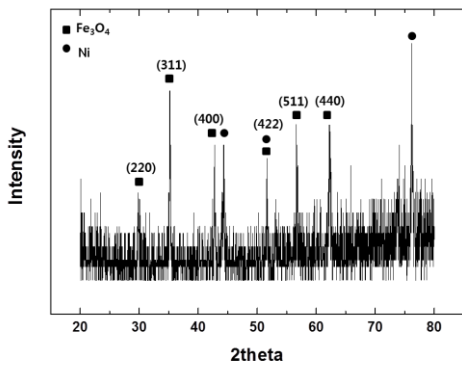
2.2 The electrochemical tests

The caustic solution was used to observe the effect of pH, and the different concentrations of NaCl were used to observe the effect of chloride ion concentration. And the bath temperature was maintained at room temperature. The solution was prepared with deaeration by the purging of N₂ gas, and was not agitated during the electrochemical tests. Each electrode was inserted into a 1 L five neck round flask. The electrodeposited wires were insulated with a Teflon heat shrink tubing.

The electrochemical measurements were carried out on a Solartron 1260 instrument with a three electrode electrochemical cell. The reference electrode was SCE (Standard Calomel Electrode), and the counter electrode was a 1 mm Pt wire. The measured open circuit potential of the magnetite electrode was about -0.5V_{SCE} at 1 M NaOH solution. Cyclic Voltammograms of the magnetite electrode were measured with 100 mV/s scan rate, and the applied potential was from -1.5 V to +0.5 V. The cycle voltammetry result of the electrodeposited Fe₃O₄ electrode at the deaerated solution containing 1 M NaOH + 10,000 ppm NaCl, at 25°C was shown in Fig. 2. In this figure, the reaction peak of Fe₃O₄/Fe was shown at -1.15 V_{SCE}, and that of Fe₃O₄/Fe₂O₃ was shown at -0.74 V_{SCE}.



(a)



(b)

Fig. 1. X-ray diffraction patterns of (a) as-received magnetite powder (b) the electrodeposited Fe₃O₄ electrode.

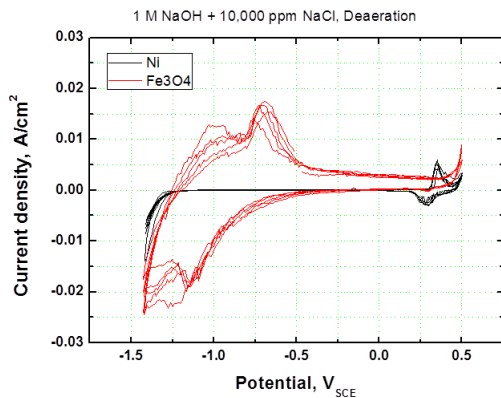


Fig. 2. Cycle voltammetry result of the electrodeposited Fe₃O₄ electrode at the deaerated solution containing 1 M NaOH + 10,000 ppm NaCl, at 25 °C.

3. Summary

The magnetite electrode was fabricated by using the electrochemical-assisted precipitation method for the measurement of the solubility of the magnetite. The prepared magnetite electrode showed the characteristic of the magnetite by an XRD spectrum. In addition, the electrochemical test, i.e. cycle voltammetry indicated

that the electrodeposited magnetite can be used as the measurement of the electrochemical properties.

REFERENCES

- [1] K. M. Hwang, Characteristics of flow-accelerated corrosion inside the carbon steel piping in nuclear power plants and wall thinning management life cycle of Korea, Corrosion and Protection Vol. 10 ~ 11, p. 13, 2012
- [2] K. Fujiwara, M. Domae, K. Yoneda, F. Inada, Model of physico-chemical effect on flow accelerated corrosion in power plant, Corrosion Science, Vol. 53, p. 3526, 2011.
- [3] K. -S. Jung, L. Pierrefeu, Electrochemical characterization of sintered magnetite electrode in LiOH solution, Corrosion Science, Vol. 52, p. 817, 2010.
- [4] J. W. Halley, A. Schofield, B. Berntson, Use of magnetite as anode for electrolysis of water, Journal of Applied Physics, Vol. 111, p. 124911, 2012
- [5] H. R. Zebardast, S. Rogak, E. Asselin, Use of EIS to measure the rate of H₂O₂ decomposition on a bulk magnetite electrode in alkaline solution, Journal of The Electrochemical Society, Vol. 159, P. B831, 2012
- [6] H. M. Kothari, E. A. Kulp, S. J. Limmer, P. Poizot, E. W. Bohannon, J. A. Switzer, Journal of Materials Research, Vol. 21, p. 293, 2006
- [7] P. L. Taberna, S. Mitra, P. Poizot, P. Simon, J. -M. Tarascon, High rate capabilities Fe₃O₄-based Cu nano-architected electrodes for lithium-ion battery applications, Nature Materials, Vol. 5, P. 567, 2006
- [8] S. Mitra, P. Poizot, A. Finke, J. -M. Tarascon, Advanced Functional Materials, Vol. 16, p. 2281, 2006