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Surface treatment of iron by electrochemical oxidation and subsequent annealing for the improvement of anti-corrosive properties

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ABSTRACT

Corrosion resistance of iron oxides on iron foils prepared by anodization, annealing or a combination of both was characterized by electrochemical methods. Even though iron oxide film with a thickness of more than 2 μm could be prepared by single anodization, corrosion resistance deteriorated because the oxide film was in the amorphous phase and contained many defects. Corrosion resistance of iron oxides was also not enhanced by single annealing. Conversely, combination of anodization and subsequent annealing led to a positive shift of the corrosion potential in the Tafel plot, indicating that corrosion resistance was improved. Formation of thicker oxide during anodization was associated with a more positive shift in corrosion potential after annealing. Electrochemical impedance spectroscopy showed that the slowest charge transfer was observed in oxide films grown by a combination of anodization and annealing. We found that the optimum annealing temperature of anodic films in terms of the most positive shift of E_{corr} was 500 °C.

1. Introduction

In thermodynamics, corrosion occurs due to inherent metallic characteristic and tends to result in a negative change of Gibbs free-energy (ΔG). The electromotive force (EMF) series indicate the corrosive tendency of various metals, showing that a negative standard electrode potential (V_o) is associated with a greater tendency for oxidation of metal. The EMF of iron (−0.440 V) is more negative value than that of Ni (−0.250 V), Sn (−0.136 V), Cu (0.337 V), and Au (1.50 V). Since iron is a vital metal with diverse applications, anti-corrosion of iron has long been an important issue. To date, coating with protective materials, environment control, addition of inhibitors and electrochemical protection have been used as anti-corrosion methods [1–6].

Electrochemical oxidation, so called anodization, is a facile method to allow the production of passive oxide films with a controllable thickness on the surface of metal, which might inhibit the corrosion. For Al [7–9], Ti [10–13], Zr [14], Mg [15], Ni [16], and Zn [17], corrosion resistance can be dramatically enhanced by anodic formation of a dense barrier-type oxide film. Thus, anodization is an essential step for the surface treatment of some metals to increase corrosion resistance.

Conversely, anodization of iron generally produces porous or non-protective oxide films that are easily detached from the Fe substrate or dissolved by the electrolyte [18–21]. Thus, there have not been many successful attempts to produce anodic iron oxides on Fe for the purpose of corrosion inhibition.

According to Hollis's U.S. patent in 1899 [22], a protective iron oxide film was prepared by anodization in caustic soda (NaOH). Recently, Burleigh et al. have demonstrated that a nanoporous iron oxide film consisting of magnetite was prepared by anodization in hot sodium hydroxide solution. When applied with commercial inhibiting oil, the nanoporous iron oxide reduced the corrosion rate by 2 orders of magnitude [23,24].

In this study, we prepared non-porous iron oxide by a simple anodic process of iron in alkali solution at room temperature. Subsequent annealing allowed phase transformation from an amorphous to a crystalline structure as well as removal of defects as weak points against corrosion, resulting in reduced corrosion and positively shifted corrosion potential. These findings provide a new method of inhibiting the corrosion in Fe via a simple anodization and annealing process.

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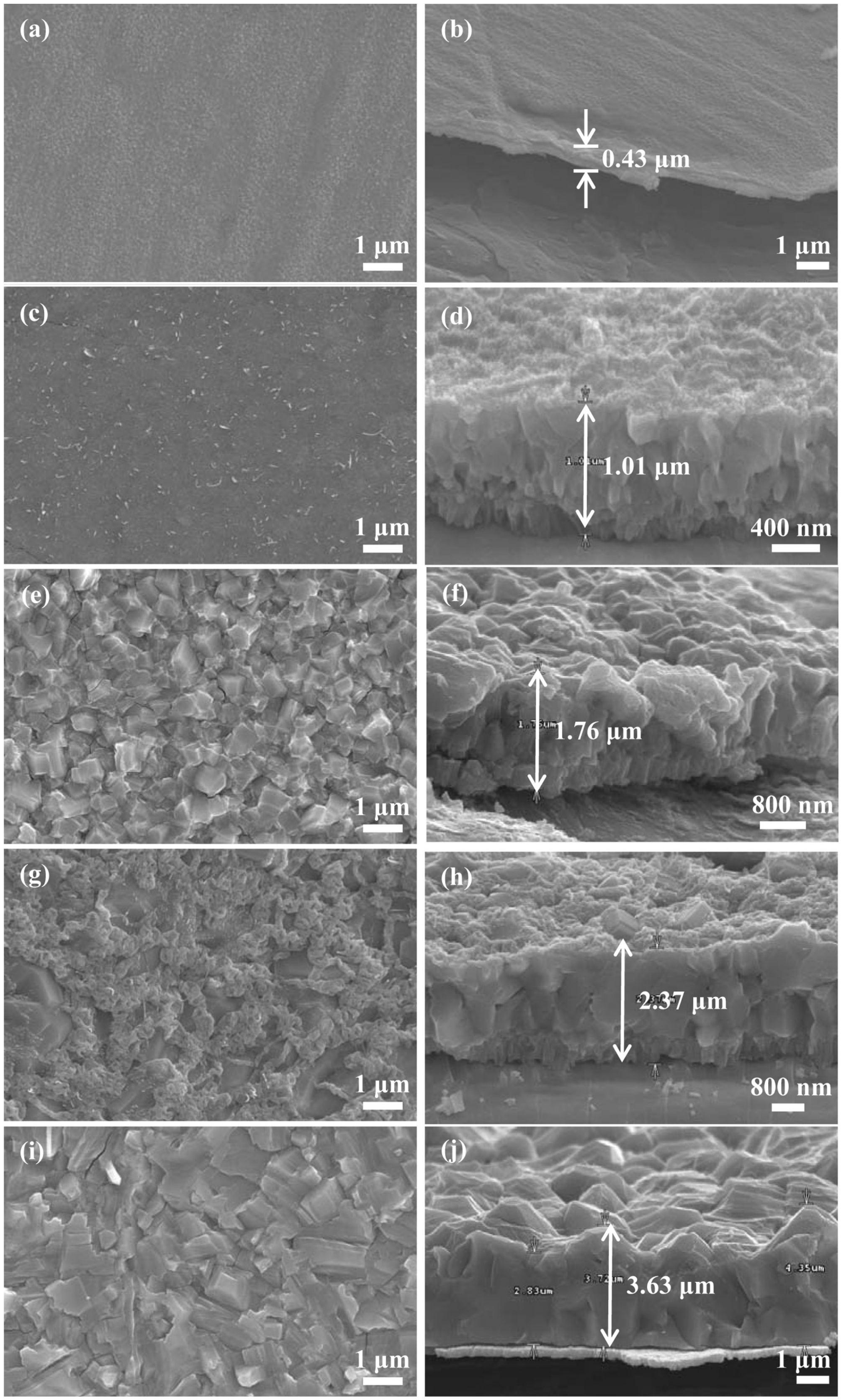

Fig. 1. FE-SEM images of iron oxides, which is formed by annealing of an iron foil at different temperatures: (a, b) 300 °C, (c, d) 400 °C, (e, f) 500 °C, (g, h) 600 °C, and (i, j) 700 °C.