

Electrochemical Reduction of TiO_x at Room Temperature in Ionic Liquids

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Introduction

The reduction of the surface oxide and the oxide formed in the surface of the Ti metal on heat treatment has been a long standing research problem (1,2). In this paper we present data on our efforts to reduce the oxide on Ti using room temperature ionic conducting liquids.

Experimental

A titanium foil 10 cm long by 2mm wide by 0.25mm thick was oxidized in a furnace at 550°C in air for 140 hours. A simple test tube type cell was used and the experiments were carried out in a dry box. The non-aqueous ionic liquid used was aluminum chloride and 1-ethyl-3-methylimidazolium chloride (EMIC) in a mole ratio of 0.8:1.0 respectively, giving a mole fraction of AlCl₃ of 0.44 (basic melt). A sample of the oxidized Ti was placed in the cell so that ~1 cm was immersed in the electrolyte. A platinum wire was used as the counter electrode and an aluminum wire in 1.5:1.0 acidic melt was used as a reference electrode. Voltage was applied and controlled by a Princeton Applied Research 283 potentiostat through a computer controlled interface.

Results and Discussion

Figure 1 shows the voltammograms recorded at a sweep rate of 50mV/sec for the oxidized Ti strip in the EMIC/AlCl₃ electrolyte. The initial sweep toward more negative voltages exhibits two clearly-defined reduction waves past -0.5 V. After several cycles, the resistivity of the oxide film decreases as the titanium oxide film is reduced to the metal. This is evidenced by a decrease in the overall slope of the current - voltage curves. Further, the anodic peak observed in the solid curve at -0.5 V is indicative of metal dissolution, the metal having been formed in the original cathodic sweep. For more extensive reduction, the voltage was held at -1.6 V. This

value was chosen because that voltage lies beyond the two reduction waves observed in the initial cycle in Figure 1. The oxidized Ti strip was held at a voltage of -1.6V for 15 minutes, then the sweep was continued. The first full sweep after the 15 minute reaction is shown in Figure 1 with the filled dotted line. The area between the solid line and the top of the filled dotted line is the charge used to reduce the thermally grown oxide on Ti. Further, the anodic peak at -0.5 V is now considerably larger and better defined than in the initial sweep. This indicates that a substantial amount of fresh titanium metal was available for the oxidation occurring in this peak.

References

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2. T.H. Okabe, T.N. Deura, T. Oishi, K. Ono and D.R. Sadoway, J. Alloys Comp., 237,150 (1996)..

