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DOCTORAL THESIS OUTLINE

Tedi Kurniawan

The title of the thesis is ñ**Hydrogen Permeability in the Scales of Iron Oxide at 973 K under Constant Oxygen Activity**ò. The thesis is divided into five chapters.

Chapter 1: “Introduction”.

Ferritic and Austenitic heat resistant steels are currently used as tubing materials of superheaters and reheaters in advanced power plants. The steam oxidation behavior of these steels remains a major issue. Protective chromium oxide (Cr_2O_3) that is expected to be formed on the surface of the tube was retarded by the enhanced growth of non-protected iron-oxides as the outer scale and Fe-Cr spinel as the inner scale. This oxidation behavior was not only observed in the steam side, but it also observed in the air side. It is strongly suggested that hydrogen permeation from the steam side to the air side of boiler tube played important role. Hydrogen permeability measurement is one of the effective ways to understand this mechanism. The objective of this thesis is to clarify the hydrogen permeation through iron-oxides under constant oxygen activity at 973 K, which is the target temperature of Advanced USC power plants. The information of hydrogen permeability constant of iron-oxide will provide valid information to determine the significance role of iron oxide layer in the permeation of hydrogen from the inner side to the outer side of the boiler tube.

Chapter 2: “Phase stability of iron oxides in the Pd-Fe-O system by electro-motive force measurement”.

The iron oxide for the hydrogen permeability measurement is required of a single phase with a known thickness. There are three phases of iron oxides, wüstite ($Fe_{1-x}O$), magnetite (Fe_3O_4) and hematite (Fe_2O_3). Accurate phase boundary condition between Fe oxide and Pd-Fe alloy is required to determine the alloy composition and the oxidation condition to obtain desired Fe oxide on the alloy. In this study, thermodynamic stability of iron oxides in Pd-Fe-O system was determined from Pdï (1ï 64)at%Fe alloys at the temperature 973 K to 1073 K. The equilibrium oxygen partial pressure of Pd-Fe alloy/iron-oxide was measured with oxygen concentration cell using Ca-stabilized ZrO_2 solid electrolyte. Based on the obtained result, the phase boundary condition between iron-oxides and Pd-Fe alloy was determined, and the thickness of iron-oxide was estimated as a function of equilibrium oxygen partial pressure and alloy compositions.

Chapter 3: "Formation of iron oxides on Pd-Fe alloy by high temperature oxidation".

Single-phase of iron oxide is grown directly from the Pd-Fe alloy by high temperature oxidation. The oxidation condition was determined from the phase boundary condition between Fe oxide and Pd-Fe alloy in the Chapter 2. Oxygen partial pressure was controlled by supplying gas mixture of Ar-H_2-H_2O with a certain H_2/H_2O ratio. Oxidation of Pd-64Fe alloy with about 200 μm thickness at 1273 K under the oxygen partial pressure of $(7.9\pm0.3)\times10^{-10}$ Pa for 259.2 ks has shown that single-phase FeO with about 30 μm thickness formed on the alloy. Furthermore, oxidation under the oxygen partial pressure of $(7.1\pm2.0)\times10^{-8}$ Pa has shown that single-phase Fe_3O_4 with about 70 μm thickness formed on the alloy. Both thickness were close to the estimated value. The composition of the alloy also shows that equilibrium condition has been reached at this temperature. The results clarified that the phase boundary between Fe oxide and Pd-Fe alloy which developed in the Chapter 2 is applicable to form single-phase iron oxide with the expected thickness.

Chapter 4: "Hydrogen permeability measurement of single-phase iron oxides at 973 K by dew point hygrometer".

Hydrogen permeation measurements through single-phase of Fe_3O_4 and FeO have been conducted at 973 K under constant oxygen activity. Pd-Fe alloy was sandwiched into two compartments of hydrogen provided side and detected side. The oxygen activity of both compartments was kept at the same value by supplying gas mixture of Ar-H_2-H_2O in the hydrogen provided side and Ar-CO-CO_2 in the detected side with certain H_2/H_2O and CO/CO_2 ratios. The hydrogen permeability of Fe_3O_4 and FeO were 8.1 x 10^{-11}molH m^{-1} s^{-1} $Pa^{-1/2}$ and 5.1 x 10^{-11}molH m^{-1} s^{-1} $Pa^{-1/2}$. Those values are about one order of magnitude lower than that reported hydrogen permeability constant of iron. This may indicate that permeation of hydrogen through boiler tube with the co-existence of Fe_3O_4 or FeO scale will be determined by the hydrogen permeation of the steel, since the thickness of boiler wall will be more than one order of magnitude thicker than the thickness of scale.

Chapter 5: "Conclusion".

The hydrogen permeability of single-phase FeO and Fe_3O_4 at 973 K in a steady-state condition has been determined. The result clarified that the hydrogen permeability of iron-oxide does not play important role in the growth of iron-oxide in the outer side of boiler tube.