

Original Article

Solubility Study of Nickel Ferrite in Boric Acid Using a Flow-Through Autoclave System under High Temperature and High Pressure

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ABSTRACT

The solubility of nickel ferrite in an aqueous solution of boric acid was studied by varying the pH at the temperatures ranging from 25°C to 320°C. A flow-through autoclave system was specially designed and fabricated to measure the solubility of Fe in hydrothermal solutions under high temperature and pressure. The performance of this flow-through system was directly compared with the conventional static state technique using a batch-type autoclave system. The stability of fluid velocity for the flow-through autoclave system was verified prior to the solubility measurement. The influence of chemical additives, such as boric acid and H₂, on the solubility of nickel ferrite was also evaluated.

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1. Introduction

The stable operation of reactor coolant systems is strongly dependent on various processes, including the corrosion process in the cooling system, mass transport, deposition of corrosion products, as well as numerous physicochemical processes (e.g., propagation of radioactivity) [1]. These processes of the cooling system are mainly influenced by physicochemical factors such as temperature as well as chemical composition of the fluid mechanics or coolant, owing to the current trend of long-term period operation of the nuclear power plant [2,3]. For these reasons, water

chemistry control must be improved for the operational reliability and stability of nuclear power plants. In order to achieve optimal performance of the power plant cooling system, detailed experimental data such as mass transport and solubility of various corrosion products are necessary [4].

Moreover, the relationship between the effects of corrosion products, temperature, pH, and dissolved gases must be experimentally illustrated, for instance, as models that quantitatively evaluate the effect of water control on the nuclear power plant lifetime [5,6]. The solubility of the materials may vary according to temperature, amount of dissolved oxygen in the coolant, concentration of chemical additives, and the

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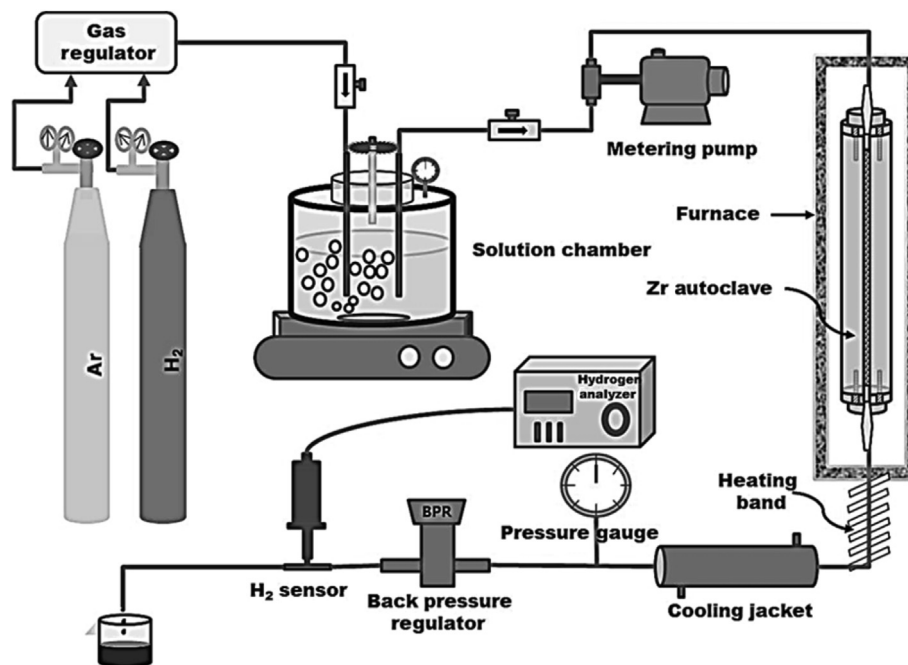


Fig. 1 – Schematic layout of the experimental setup for the flow-through autoclave system.

operating conditions of the nuclear reactor; however, there are many other factors that influence corrosion of the primary coolant system materials. The pH control of the coolant is known as a fundamental water control method that can be used to understand the corrosion behavior and transport route of the corrosion products, because it is closely related to the solubility of various corrosion products [7].

The primary coolant of the pressurized water reactor (PWR) is composed mainly of water with some chemical additives. H_3BO_3 , in particular, is added for the control of the nuclear reaction [8–11]. H_3BO_3 content at room temperature does not strongly influence the pH or conductivity of the coolant solutions, because it is a weak electrolyte and its conductivity decreases with the coolant temperature [12]. However, with the high operating temperatures of nuclear power plants (280–320°C), H_3BO_3 may influence the corrosion of manufactured materials because of the high pH, which drastically increases by the dissociation reaction of water. This corrosion phenomenon can cause significant problems for the safety of the operating system in nuclear power plants.

Corrosion products originate from the internal surface of piping and steam generator tubing. Nickel ferrite (NiFe_2O_4) is known as a main corrosion product in the primary coolant system, and its solubility becomes important to optimize water chemistry control for a long-term fuel cycle [13,14].

To determine the solubility under high pressure and high temperature conditions, conventional static state and fluid dynamic state methods can be used. The conventional static state method has advantages of simplicity and low cost. However, the conventional static state method is a slower measurement technique because a relatively long time is required to reach equilibrium. Experimentally, more than 10 days are required for equilibrium when measuring the solubility of magnetite (Fe_3O_4) at 423 K using this method [15]. By

contrast, equilibrium is achieved quicker when using the fluid dynamic state method because of the relatively high surface area of the sample.

In this study, a flow-through autoclave system for the measurement of NiFe_2O_4 solubility in an aqueous H_3BO_3 solution was specially designed and fabricated to simulate the flow circulation system of a nuclear power reactor. The time for Fe to reach chemical equilibrium was measured with the flow-through system, and the results were compared with those obtained using the conventional static system. Solubility measurements were carried out in aqueous solutions of primary coolant at temperatures of 25–320°C with pH values of 4.5–8.0. The solubility of NiFe_2O_4 in H_3BO_3 solutions under high pressure and high temperature is discussed considering parameters such as H_2 gas content and pH.

2. Materials and methods

Solubility measurements were performed in a flow-through autoclave system at temperatures and pressures up to 400°C and 1,500 bar, respectively. The experimental setup is illustrated in Fig. 1. The main structure of the flow-through autoclave system is composed of the solution chamber with a gas regulating system for Ar and H_2 , a high temperature and pressure autoclave system located inside a furnace, and a sampling unit with a cooling jacket, H_2 sensor, and back-pressure regulator.

A stainless steel solution chamber with a capacity of 4.5 L at 8 bar was used to contain the aqueous H_3BO_3 solution (various concentrations were used). Prior to the solubility measurement, the H_3BO_3 solution was carefully degassed by repeatedly pressurizing with Ar and decompressing, then flushing with H_2 gas at a constant pressure. This procedure

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