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Development of a scratch electrode system in high temperature high pressure water

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ABSTRACT

The scratch electrode system in high temperature high pressure (HTHP) water has been built. The design temperature and design pressure of this system are 350 °C and 20 MPa respectively, and the maximum scratch speed is 3.3 m/s. By using this scratch electrode system, the repassivation kinetics of Alloy 690 in high temperature pressurized water with different dissolved hydrogen (DH) at 300 °C is studied. The research results justify that the scratch electrode system is valid and the scratch technique in HTHP water is a new effective and reliable method to study the SCC susceptibility of nuclear materials.

1. Introduction

The film repassivation kinetics as a key parameter used in SCC models has been broadly studied [1–7]. Among many methods used for measuring the film repassivation kinetics, including scratched electrode [7–17], drop-weight [18–20] and abrading electrode [21–25], the rapid scratching method is the most often used. However, the rapid scratching method requires sophisticated instrumentation to record the high current density within milli- or micro-seconds [2,26], and it is difficult to achieve rapid scratch and measure the electrochemical signals during repassivation in high temperature high pressure (HTHP) water. Therefore, there are quite few papers related to the study of repassivation kinetics of nuclear materials using scratch electrode technique in HTHP water.

Angeliu et al. [19] studied the repassivation behavior of Alloy 600 in sodium hydroxide solution with boric acid under different dissolved hydrogen conditions at 288 °C by combining drop-weight straining method with cathodic reduction/potential pulse techniques. Bosch et al. [27] built an in-situ scratch electrode system in the static autoclave and performed a slow scratch experiment on AISI 304 alloy at 300 °C. However, the finite scratching speed resulted in mixed activation/repassivation, i.e., repassivation started before the scratching was finished, although they put forward a related mathematic model to solve this problem. Kwon et al. [10,28–30] investigated the repassivation behavior of stainless steels and Alloy 690 using rapid scratch technique at temperature below 100 °C. With the introduction of parameter *cBV* denoting the repassivation rate, they predicted the SCC susceptibility of ferritic stainless steels (SS), 304 SS and Alloy 690 in different environments.

The purpose of the paper is to describe our self-built rapid scratch electrode system using in HTHP water and testify the validity of this system by studying the repassivation kinetics of Alloy 690 in HTHP water with different concentration of dissolved hydrogen (DH).

2. Design of the rapid scratch system with re-circulated water loop

The rapid scratch electrode system in this experiment is designed and built by ourselves. This system includes a re-circulated water loop, a potentiostat, three-electrode system and an autoclave with a built-in scratch unit as shown in Fig. 1. The water chemistry parameters of the re-circulation loop are monitored online, including dissolved oxygen (DO), DH, pH values and electrical conductivity. The DH in the loop is measured by a dissolved hydrogen meter made by DKK-TOA Japan. The DO and DH values are automatically controlled and adjusted using software LabVIEW 8.5. The control error is lower than 1%. A Gamry Reference 600 potentiostat and corresponding software is used for all electrochemical measurements.

The design temperature and the design pressure of the autoclave are 350 °C and 20 MPa respectively. The cover and body of the autoclave are made of 316 stainless steel. The plan view of built-in scratch unit is shown in Fig. 2 and it mainly includes the ceramic pin, pull rod, sample holder, guide frame, compression bolt and compression spring. The scratch sample is installed in a

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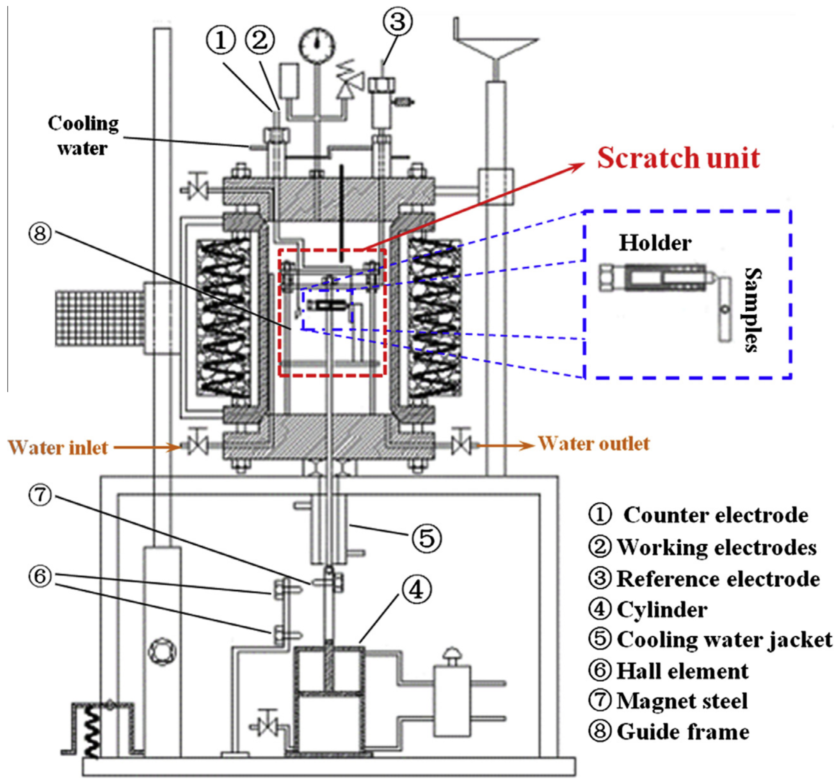

Fig. 1. Schematic diagrams of the rapid scratch electrode system.

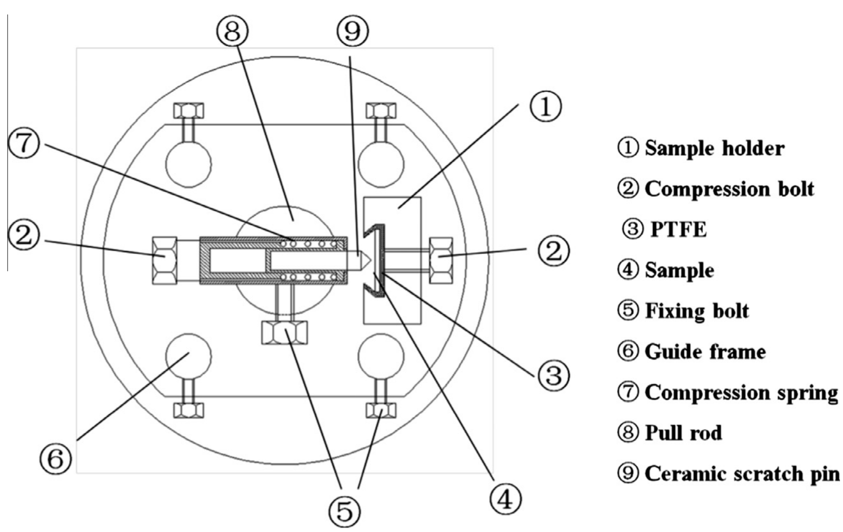

Fig. 2. Plan view of the scratch unit shown in Fig. 1.

sample holder as shown in Fig. 2. The sample is insulated from the holder using polytetrafluoroethylene (PTFE). The ceramic pin is made of high-purity Al_2O_3 (99.999%) and can be tightly pressed on the surface of the samples by a compression spring when scratching the sample surface. The size of the pin is shown in Fig. 3a. For each scratch experiment, the compression bolt is turned to the same position so that the normal pressure from the pin on the sample surface remains the same. The pin is connected with the pull rod which can be rapidly pulled out when the external air cylinder is vented. The scratch speed is controlled by adjusting the air cylinder pressure, and the scratch time is measured by the Hall elements as shown in Fig. 1. Fig. 4 shows the changes of scratch time and scratch speed under different cylinder pressures in autoclave at 300 °C. It can be seen that the top scratch speed in HTHP water at 300 °C can reach 3.3 m/s. The ceramic pin is replaced with a new one for each experiment and the width (about 50 μm) and depth of the scratches remain the same under the same pressure of cylinder.

Four electrode leads are connected to the outside of the autoclave, including one counter electrode Pt ($20 \times 20\ mm^2$) and three working electrodes (one for the scratch experiment and two for measurement of the potentiodynamic polarization curves). An external Ag/AgCl/0.1 M KCl reference electrode is inserted at the top of the autoclave.