PIE TECHNIQUES FOR THE STUDY OF IRRADIATION ASSISTED STRESS CORROSION CRACKING

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ABSTRACT

Irradiation assisted stress corrosion cracking (IASCC) is one of the critical concerns in materials for nuclear in-reactor components of light water reactors (LWRs). In general, IASCC, which is caused by a complicated synergistic effect of stress and chemical environment on materials that have experienced degradations by irradiation, can be reproduced on the materials irradiated over a certain threshold fluence level of fast neutrons by post-irradiation examinations (PIEs) at hot laboratories. We have carried out PIEs at the NFD Hot Laboratory on the materials obtained from LWR power plants. We describe the PIE techniques developed for our IASCC studies, especially the SCC tests, metallurgical tests using electron microscopes and sample preparation techniques.

1. Introduction

Nippon Nuclear Fuel Development Co., Ltd. (NFD), a joint venture of Hitachi Ltd. and Toshiba Corp., has been operating a hot laboratory facility since 1977 for extensive post-irradiation examinations (PIEs) of boiling water reactor (BWR) fuels and structural materials. The NFD Hot Laboratory is able to accommodate full sized commercial BWR fuel bundles and comprehensive PIE programs have been carried out on many BWR fuel bundles [1-3], as well as structural materials irradiated in BWRs.

Irradiation assisted stress corrosion cracking (IASCC) is one of the important concerns in materials for in-reactor components of light water reactors (LWRs) [4-7]. It takes the form of intergranular stress corrosion cracking (IGSCC), and the critical fluence level has been reported to be about $5x10^{24}n/m^2$ in Type 304 stainless steel (SS) [4-7]. IASCC failures have been attributed to radiation hardening and radiation induced segregation (RIS) of impurities and/or alloying elements at grain boundaries (GBs). Enrichment of impurities such as silicon and phosphorus and depletion of chromium at the GBs are considered to be the two most likely detrimental effects of RIS at the GBs. [7, 8]. However, it is not presently clear which parameter is dominant in IASCC.

To meet the demands for detailed mechanistic understanding of IASCC phenomena, more precise microscopic and specific PIE techniques are required and various examination techniques have been developed during the course of NFD's PIE programs to meet these demands. This paper presents testing facilities for PIE activities used for our recent IASCC studies at the NFD Hot Laboratory.

2. Facilities for stress corrosion cracking

2.1 Crack growth rate test facility

Crack growth rate tests of irradiated materials are carried out in order to support plant life assessment of reactor components. The test facility is schematically illustrated in Fig.1. Autoclaves with mechanical loading devices are located in the hot cell (shielding capacity: 740GBq ^{60}Co) while all other equipment is placed outside the hot cell. Fig.2 shows a photo of some of the equipment for the CGR tests. This test facility can perform the test in simulated BWR and PWR environments. Each autoclave can accommodate two 0.5T-CTs or one 1T-CT. The load is controlled using a load cell (load capacity: 50kN) installed inside the autoclave. DO concentration is measured continuously at the inlet and the outlet. Conductivity is measured continuously at the outlet and measured periodically at the inlet. The electrochemical corrosion potential (ECP) of the specimen is monitored using an internal Ag/AgCl reference electrode. Crack growth is monitored by the reversing dc potential drop (DCPD) method. After a CGR test, crack length is calibrated by the scanning electron microscope observation of fracture surface of the CT specimens.

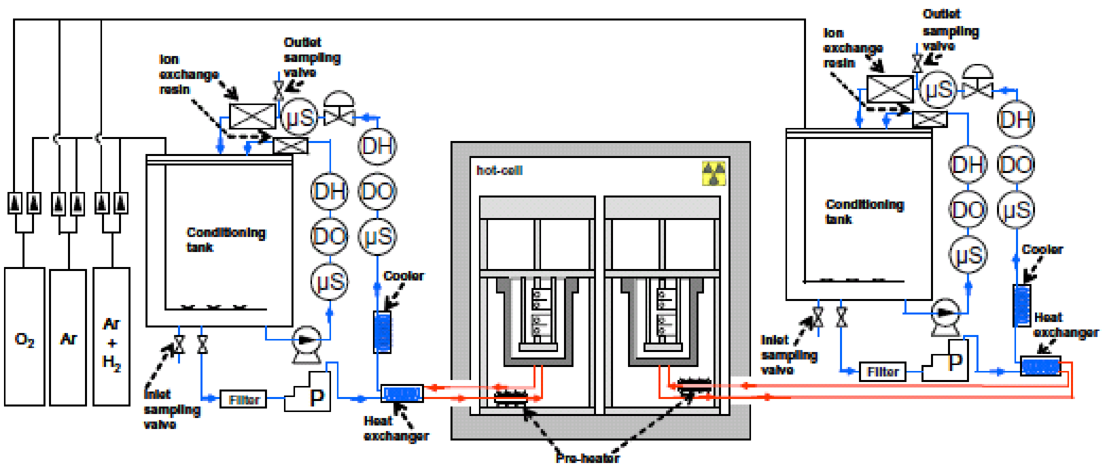

Fig.1 Schematic of CGR test facility [9]

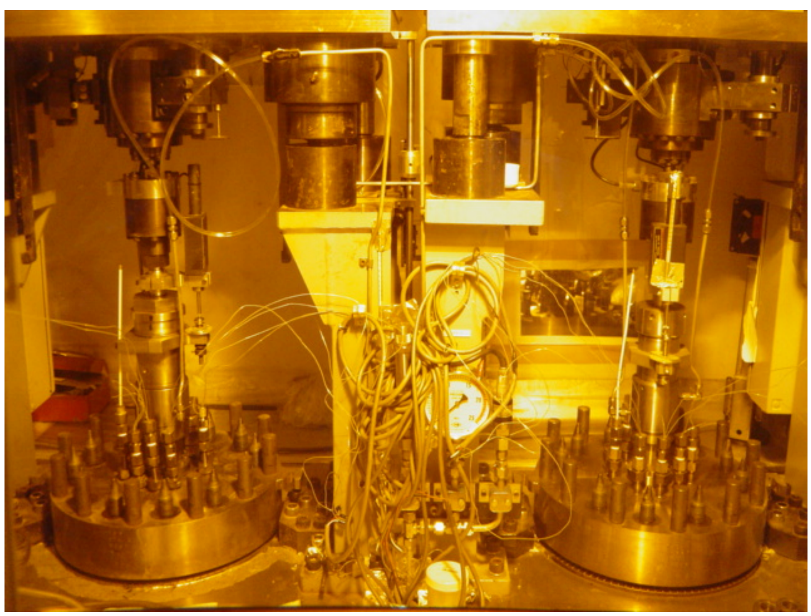

Fig.2 Photo of the equipment for the CGR test

Fig.3 shows the IASCC growth rate of in-core structural materials acquired under normal water Chemistry (NWC, 32ppm DO) and the disposition curves of JSME NA1-2002 standard for sensitized SUS304 and for L-grade SS at ECP>150mV$_{SHE}$. All of the data were below the upper limit, $9.2x10^{-10}$m/s. In addition, the CGRs certainly decreased under HWC (hydrogen water chemistry; 2ppb DO + 20-40ppb DH) although it did not definitely change under the deaerated condition (20ppb DO).

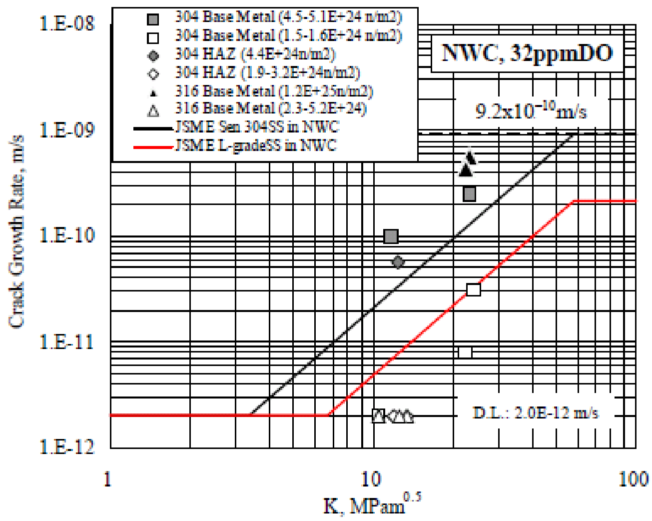

Fig.3 Dependence of CGR on neutron fluence [9]

2.2 Facility for slow strain rate and uniaxial constant load tests

SSRTs and uniaxial constant load (UCL) tests are carried out in order to evaluate the neutron fluence, applied stress and water chemistry dependency of IASCC susceptibility. These tests performed in a once-through autoclave system installed in a hot cell (Shielding capacity: 3.7TBq ^{60}Co). A schematic of SSRT/UCL test facility is shown in Fig. 4. For UCL tests, load is controlled using a load cell (load capacity: 10kN). DO concentration is measured continuously at the inlet and measured periodically at the outlet using a bypass line. Conductivity is measured continuously at outlet and measured periodically at inlet. ECP is measured using the Ag/AgCl external reference electrode.

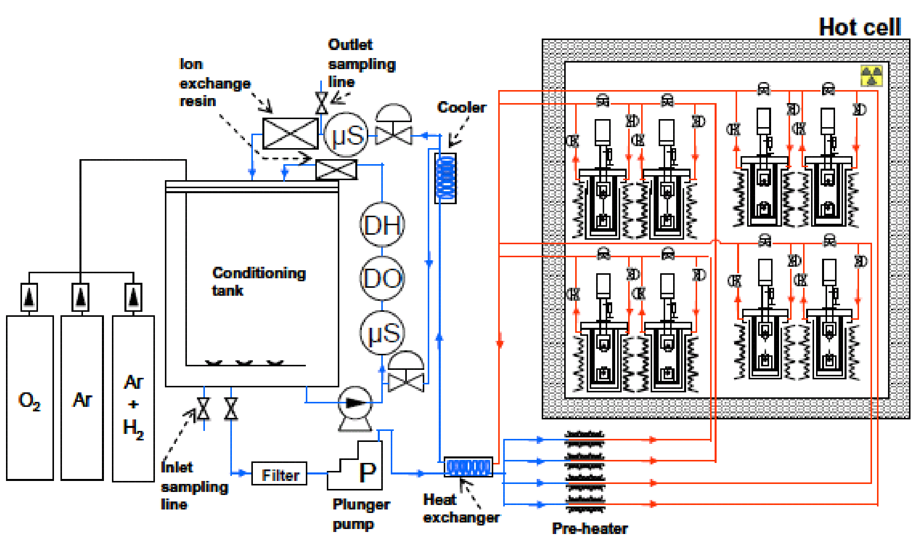

Fig.4 Schematic of SSRT/UCL test facility [10]

3. Highly irradiated specimen preparation technique

In order to carry out the CGR and SSRT/UCL tests of in-reactor components, it is necessary to machine CT and tensile specimens from the LWR in-core structure materials. They are machined using a numeric control milling machine and electric discharge machine which are installed in a hot cell. We have machined a maximum of 1.5 T-CT specimens (Fig.5) from the block of approximately 650w x 140h x38.1t mm until now. Although remote handling was difficult for this block, it could be done and machining was carried out using various devices.

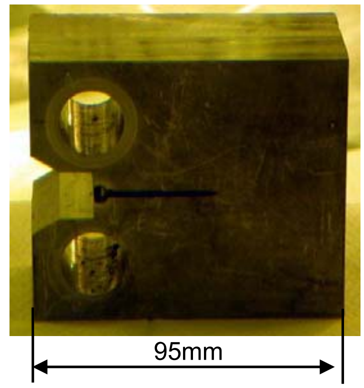

Fig.5 1.5T-CT specimen (maximum size)

4. Field-emission type transmission electron microscope and focused ion beam processing technique

In order to elucidate the IASCC mechanism, it is important to study the chemical and physical properties at and/or near the GBs, because IASCC propagates along GBs. To study the chemical compositional change at and/or near the GBs, a field-emission type transmission electron microscope (FE-TEM) with energy dispersive X-ray spectrometer (EDS) was installed in a hot laboratory. The analysis samples are punched into 1 mm diameter disks for a reduced volume to minimize radiation flooding of the X-ray detector. High-angle, random GBs identified with diffraction patterns are selected for the EDS analysis. The EDS analysis is done using a fine electron probe of less than 2 nm in diameter.
Example compositional profiles across a GB are reproduced in Fig. 5 for Type 316 SS. The depletion of chromium and about 10% enrichment of nickel were detected at the GB.

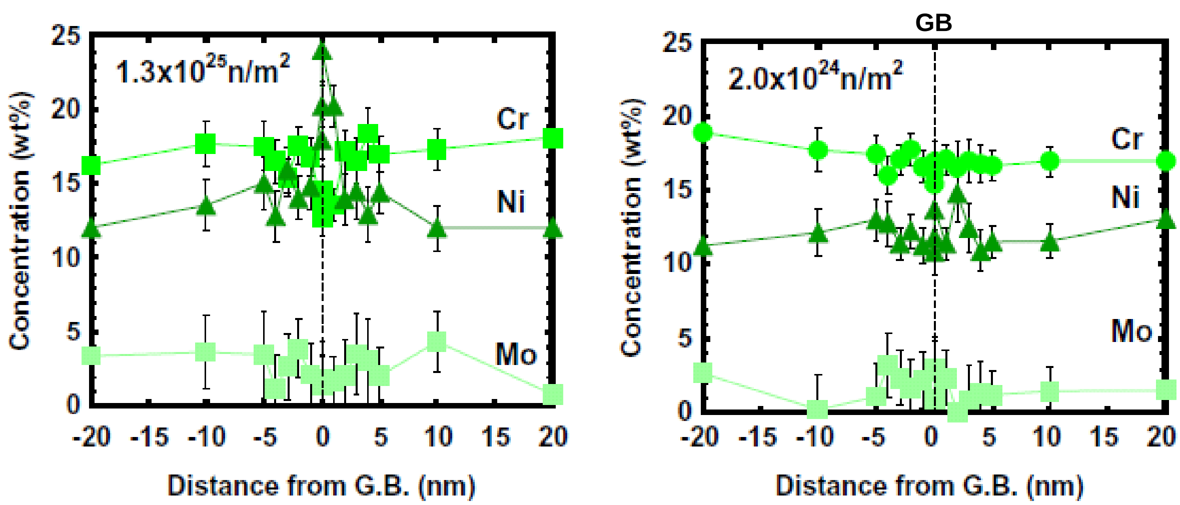

Fig.6 Example compositional profiles across a GB in irradiated Type 316 SS [10]

The focused ion beam (FIB) micro-sampling technique can be also applied to a TEM specimen preparation from any places such as IASCC crack tip. Micro-sampling technique is enable us to observe and identify the oxides in cracks and the dislocation structure near cracks and also to estimate the deformation behaviour near cracks.

5. Summary

In this paper, we described several test techniques that had been adopted in our recent IASCC studies. They were PIE techniques for CGR, SSRT, and UCL tests, metallurgical examinations using FE-TEM/EDS, and the FIB micro-sampling technique for microscopic evaluation of chemical and physical properties at and or near GBs of in-core structural materials. There are still many unknown points about IASCC phenomena. For mechanistic understanding and development of countermeasures against IASCC of in-core structural materials, we have to make the further improvements and upgrades in present PIE techniques.

References

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