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Damage Evaluation Techniques for FBR and LWR Structural Materials Based on Magnetic and Corrosion Properties along Grain Boundaries

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Japan Atomic Energy Research Institute and Japan Nuclear Cycle Development Institute have begun the cooperative research of novel evaluation techniques of structural material degradation in Fast Breeder Reactor (FBR) and Light Water Reactor (LWR), which based on magnetic and corrosion properties along grain boundaries. Magnetic method has been proposed as the one of the innovative non-destructive detection techniques on the early stage of creep-damage before crack initiation for aged structural materials of FBRs. On the other hand, corrosion properties and magneto-optical characteristics of ion-irradiated stainless steels in the vicinity of grain boundaries were estimated by AFM. These degradations were induced by changes in characteristics in the vicinity of grain boundaries, such as chromium depletion. It is found that the initial level of progressing process of damage can detect changes in magnetic and corrosion properties along grain boundaries of aged and degraded nuclear plants structural materials.

Key words: FBR, LWR, Irradiation Degradation, MFM, AFM

1. INTRODUCTION

Stainless steels are one of the indispensable materials to structural components in fission and fusion reactor environments. In Fast Breeder Reator (FBR) plants, these materials consist of structural components such as several kinds of primary and secondary coolant tubes and pipes inner and/or outer the pressurized reactor vessel (PRV). Specially, in these radiation environments, how to control and furthermore to suppress the aging and degradation phenomena of structural materials are the key issue governing the integrity of structural component over 60 years during operation from the view point of defect detection. On the other hand, the issue of irradiation assisted stress corrosion cracking (IASCC) is major concern on the irradiation degradation phenomena in Light Water Reactor (LWR) and major element effects on SCC susceptibility has already been discussed [1]. The occurrence of the degradation in both circumstances can be explained by introduction of changes in characteristics in the vicinity of grain boundaries, such as chromium depletion. In the collaboration research between JAERI and JNC, the detection of initial level of progressing process of damage by checking changes in magnetic and corrosion properties along grain boundaries of aged and degraded nuclear plants structural materials can be focused on the modulation of physical (magnetic properties) and chemical (corrosion properties) phenomena along grain boundaries to understand the mechanism of the irradiation degradation and also to detect the initiation of defects composing the damage. The purpose of this study is to clarify the mechanism the irradiation degradation and to understand the relationships between corrosion behavior and magnetic behavior of irradiated materials along grain boundaries and furthermore to introduce the outline of the collaboration research.

2. EXPERIMENTAL PROCEDURES

Fe-18Cr-12Ni alloys with high purity were used and solution treated at 1323K for 1.8 ks. Specimens were formed into sheets and the dimensions of specimens were 6 mm in length, 3 mm in width and 0.3 mm in thickness.

Some of specimens were irradiated by multiple beam irradiation facility at Takasaki Ion Accelerators for advanced Radiation Application (TIARA) of JAERI. The 12 MeV Ni^{3+} ion was irradiated up to maximum damage of 35 and 70 displacements per atom (dpa) at 473 and 673 K and He^{2+} ions were also implanted to some of specimens during irradiation.

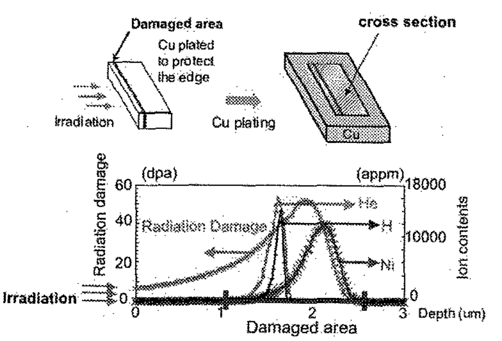

Figure 1 Sample preparation and radiation damage profile

Damage and ion implantation profiles were obtained by TRIM 85 calculation code as shown in Fig. 1[3] by Nemoto et al. Cu plating treatment was also performed on the edge of sample to protect the irradiated surface during etching and polishing as shown in Fig. 1.

The magnetic flux density on the specimens was measured by using a thin film flux gate sensor (FG sensor) developed by Shimadzu Corporation in JNC. The size of the FG sensor is about 3mm×3mm. The magnetic flux density resolution of this sensor is estimated to be about 5×10^{-4} Gauss which is much smaller than those of a hall sensor. The FG sensor needs no any cooling systems and so it is possible to measure in the vicinity of sample surface above 0.2mm. The FG sensor has major advantage of the higher spatial resolution rather than a superconducting quantum interference device (SQUID), although it is larger than that of a SQUID. In this system, not a sensor but a sample is moved to remove the geomagnetic effect depends on the sensor position. The lift-off distance between the sensor and the sample surface was kept to be 0.2mm. The sample was magnetized in the vertical direction before the measurement. A permanent magnet (~0.4T) was used to magnetize samples throughout this study.

Corrosion behavior was evaluated on the cross section of specimens after corrosion test by AFM (Thermo Microscope Co. Ltd, Explorer SPM system) in JAERI.

3. RESULTS AND DISCUSSION

3.1 Magnetic flux density measurement in JNC

Preliminary results were obtained on ion irradiated specimen as shown in Fig. 2. Irradiation damage is of 35 dpa at 473K. The heterogeneous distribution on flux density was observed in specimen region. At the present stage, obtained signal level was too low to evaluate the leakage of flux density along grain boundaries, since in trial specimen, damaged area was quite a smaller than total cross sectional area.

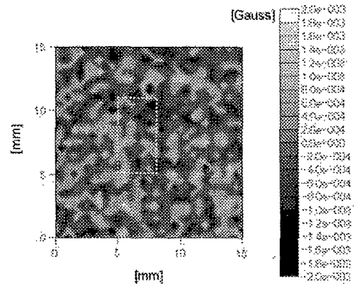

Figure 2 Magnetic flux density distribution on the suface of ion irradiated specimen

After these trial tests, the characteristics of ion irradiated specimens will be performed. It is supposed that large signal may be detected on another specimens with heavy damaged area and the leakage of flux density take place in the vicinity of grain boundaries. Ion irradiation effect has already been studied for stainless steel and formation of martensites due to existence of residual stress field, was reported in [4], [5]. In our study, deformation-induced martensites and/or stress-induced martensites with magnetization may be also stabilized during irradiation. And furthermore, latter case is closely associated with the existence of temporally applied stress field and reorientation of magnetic domain [6].

Magnetic Force Microscope (MFM) has been also carried out for reference specimen (Inconel 600 as shown in Fig. 3 and stainless steel type 304) which reveals high sensitivity to typical SCCs and clear signal was already obtained along grain boundaries as is described by Takaya et al. [2] which means the existence of magnetization and formation of martensites due to depletion of chromium. MFM and Kerr effect microscope observations are on going for ion-irradiated samples.

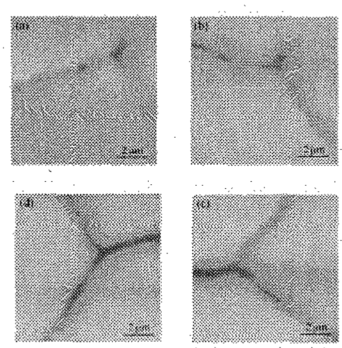

Figure 3 MFM Images of standard specimen (Inconel)

3.2 AFM measurement in JAERI

AFM observations results were shown in Fig. 3 at the cross section of irradiated specimens. Corrosion behavior was compared by cross sectional area of corroded region as is proposed by Nemoto et al [3]. Figure 4 shows the AFM topography after corrosion test. as described in [3].

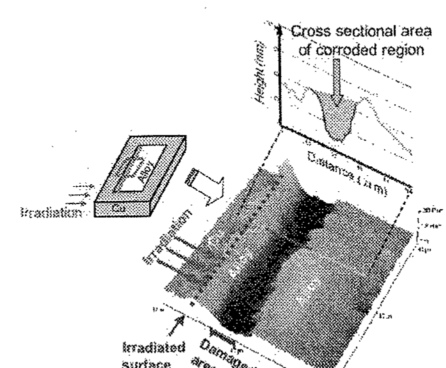

Figure 4 AFM topography of ion irradiated specimen

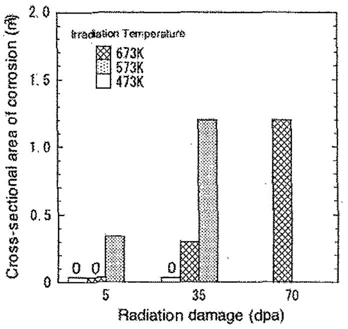

Figure 5 Irradiation damage and irradiation temperature dependence for corrosion behavior

Corrosion volume increased with increasing irradiation damage and irradiation temperature as shown in Fig. 5. This can be explained by the occurrence of irradiation induced phenomena such as segregation (chromium depletion) and precipitation.

This collaboration research includes the magnetic flux density measurement, MFM, Kerr effect microscope and AFM observations on ion and neutron irradiated specimens. Therefore, the remote-controlled AFM and remote-controlled flux density measurement apparatus will be newly developed and prepared in next year, to understand irradiation degradation phenomena of stainless steel, in Japan Materials Testing Reactor Hot Laboratory (JMHL) in JAERI and Materials Monitoring Facility (MMF) in JNC, respectively.

4. CONCLUSIONS

Japan Atomic Energy Research Institute and Japan Nuclear Cycle Development Institute have promoted the cooperative research of advanced evaluation techniques which based on magnetic and corrosion properties along grain boundaries. Magnetic method has been proposed as the one of the innovative non-destructive detection techniques on the early stage of damage before crack initiation for aged and damaged structural materials of FBR and LWR, and also Scanning Probe Microscope techniques including AFM and MFM has been already used as an innovative evaluation method to analyze corrosion behavior.

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