§5. High Temperature In-Situ Observation of Progressed Reactor Materials

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In material development for fusion reactor, the determination of basic characteristics of materials under the operating condition is significantly important. In this study hardness and structure analyses were carried out under in–situ conditions. Additionally, surface analyses using TOF-SIMS and XPS were also carried out after the in-situ mechanical tests.

i) Hardness measurements using high-temperature hardness tester

For the low emission ferrite steel material JLF-1 as progressed reactor material, the determination of the mechanical characterization is quite important. Therefore, mechanical in-situ characterization at higher temperature was carried out using high temperature hardness tester. <Method> Using high temperature micro hardness tester (Nikon QM-2) mechanical characteristics and its time dependence were observed. <Results> The samples of JLF-1 indicated lower hardness at higher temperature. The hardness Hv was decreased to 70% at 773K and to 55% at 873K from the hardness value at 373K. This results show that the JLF-1 indicates no significant strength change up to 773K, however, the strength may be decreased at over 873K and after long term usage.

ii) Structure investigation using high temperature X-ray diffraction analyses

Material structures of ferrite steel of JLF-1 at higher temperature, such as Carbide precipitation and initiation temperature of phase transformation were observed using high temperature X-ray diffraction analyses. <Method> High temperature X-ray diffraction analyses (RIGAKU : RINT2200) were carried out for the sample of JLF-1 heated from RT upto 1573K under N_2 atmosphere. At each pre-setting temperature the sample temperature was kept constant and in-situ diffraction analyses were carried out. <Results> At over the temperature of 873K(600℃) precipitations of W- and Cr-carbides were confirmed from the diffraction patterns. By further heating, phase transformation from fcc to bcc was observed just over the temperature of 1273K(1000℃).

iii) Surface analyses using TOF-SIMS and XPS for impurities and chemical bonding states

Time of flight secondary ion mass spectroscopy (TOF-SIMS) and X-ray photo emission spectroscopy (XPS) were used for analyzing surface characteristics of the materials for low emission ferrite steel of JLF-1. <Method> Reflector type TOF-SIMS (ION-TOF, TOF-SIMS IV) was used for spots and 40x40um^2 areas analyses using 25kV Ga^+ primary focused pulse ion beam. Positive and negative secondary ions were observed. XPS apparatus equipped with Al monochromatic X-ray gun (PHI-QUANTUM2000) was used for surface chemical analyses. In both cases, the samples for the surface analyses were taken after the high temperature hardness testing and carried out at RT. <Results> Hydrogen distribution on the surface and depth profiling (3kV Ag^+) indicated that the highest concentration of H was observed in a few to several atomic layers in the surface region and decreasing into the depth. The top of the surface was covered by hydrocarbons, which decreased drastically into the depth direction. From the results of peak-form change of carbon obtained by XPS, carbide formation could be occurred during the higher temperature treatment. The higher the temperature the more the dissolving adsorbed molecules and the reaction with the surface materials may occur more frequently. This is the most possible reaction mechanism for the carbide formation if the surface carbon concentration is considered. In case of espousing the material to plasma, a similar reaction mechanism can be considered, which results in the formation of compound/carbide forming in the surface region of the sample.

Publication

Two oral presentations were published at the spring meeting in 2002 of The Japan Institute of Metal. Additionally, related paper concerning to the mechanical and structural investigations was presented at the Seventh Japan International Conference of the Society for the Advancement of Material and Process Engineering(SAMPE), Nov. 13-16, 2001, TOKYO, Japan. The paper was included in the symposium proceeding, pp. 685-688.

Acknowledgment

We are grateful to Dr. A. Kohyama and Dr.Y.Katoh, Institute of Advanced Energy, Kyoto University and Dr.A. Nishimura, NIFS, Japan for the sample supply.