§22. Precipitation Process of Ti(OCN) in Vanadium Alloys in Thermal Ageing and Creep Deformation

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A vanadium alloy is expected as a candidate material of fusion reactor material by superior characteristics under neutron irradiation. However, remarkable irradiation hardening and irradiation embrittlement by heavy neutron irradiation is one of key issues for development of vanadium alloys in fusion reactor application. There are unsolved question in precipitation process during ageing process and role for hardening factor of interstitial gas atom in Ti(OCN) precipitate in order to estimate the increase of the irradiation and precipitation hardening. In this study, we developed a replica extraction method for TEM sampling in order to investigate the precipitation behavior in vanadium alloys.

A plate of 15x15x1mm of V-4Cr-4Ti alloys was cut out and electro-polished. The carbon replica technique yields measurements of precipitate chemical compositions without matrix contribution. This technique consists of a single-stage carbon extraction. A carbon layer is sputtered on the specimen surface and then, an electro-etching procedure is performed in order to collect the replica. The carbon replica technique allows the determination of particle chemical composition as small as a few ten nanometers. The determination of SPP stoichiometries was performed using a scanning energy dispersive device (STEM/X-EDS). The electrolyte composition is methanol: 80vol.%, 2-butoxyethanol: 10 vol.% and perchloric acid: 10 vol.%. The operating voltage intensity is 5V at room temperature. Before the surface polishing for extracting the carbon replica, it was necessary to exam a surface treatment using chemical-etching. The composition of chemical etching solution is hydrofluoric acid: 15ml, nitric acid: 35ml and water: 75ml.

It was confirmed that it was possible for sample manufacture for precipitation observation from a vanadium alloy due to an extraction replica method after the chemical etching. A base material was dissipated by electro-polishing, and precipitates were cropped out on the surface.

The precipitates were fixed by vapor deposition carbon film, and the vapor deposition carbon layer that fixed precipitates was exfoliated by original surface of vanadium alloy by the electro-polishing. It allowed us to have the extraction of the precipitate in vanadium alloys. From this study, for the sake of extraction replica method for s vanadium alloy, it was found that prior-chemical treatment before the carbon vapor deposition is important to hold the precipitate on the surface.

A STEM/X-EDS and TEM observation was examined with JEM -2100 at the University of Fukui.

Figure 1 shows a TEM image and diffraction pattern of precipitates in V-4Cr-4Ti annealed at 800C for 10 hours. The size distribution of precipitate taken from the replica sample showed a good agreement with one taken from the sample fabricated by the usual electro-polishing. It was confirmed that the replica extraction method in this study was succeeded. Figure 2 shows an EDS spectrum plot from the EDS analysis of a precipitate in Fig.1. From the analysis of fig.1 and fig.2, it was found that the precipitate was Ti(OCN) because it showed the NaCl type of crystal structure and metallic composition was occupied by titanium.

Finally, the extraction replica method of the vanadium alloy was established in this study.

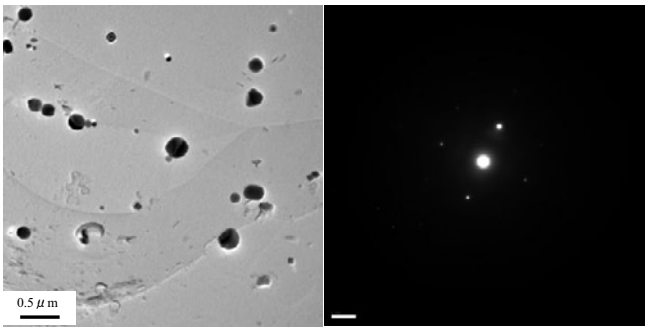

Fig.1. A TEM image and a diffraction peteern from the precipitate in the replica sample of V-4Cr-4Ti alloy

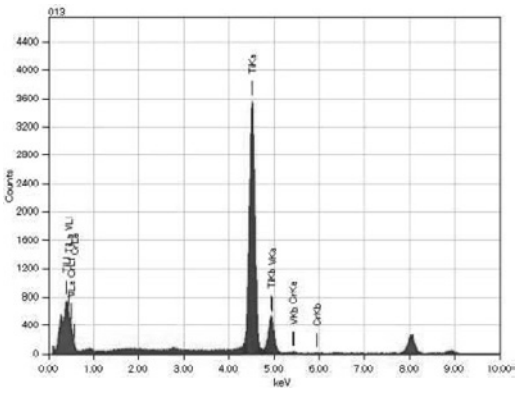

Fig.2. A EDX spectrum obtained from the precipitate showing the diffraction pattern in fig.1