

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

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### 1. Introduction

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 try to exam the replica extraction method for TEM sampling in order to investigate the precipitation behavior in vanadium alloys.

### 2. Experimental procedure

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 determination of SPP stoichiometries was performed using a scanning energy dispersive device (STEM/X-EDS). The carbon replica technique allows the determination of particle chemical composition as small as a few ten nanometers. The electrolyte composition is methanol: 90vol.%, athetil-aceton: 9 vol.% and tetramethylammonium: 1 vol.%. The operating voltage intensity is 5V at room temperature.

### 3. Results

Because separation between precipitate and the parent phase were not succeeded at the time of electrolytic polishing in the sample preparation for extraction replica method, it was not possible for

getting the compositional information of precipitate using the extract of the precipitate replica. It is considered that precipitates could not be remained on the carbon film of the extract replica because titanium oxide/nitride is semi-conductors and reaction decomposition by the electric retroaction advances occurred. On this account the settlement of techniques such as the TEM sampling with the sample work only for the precipitate replica region will be testified with WDX measurement and FIB-fabrication at the next stage.

Extraction replica method is applied to Fe-9Cr alloying, and heat treatment condition and the study about the component composition change of the precipitate by the irradiation is introduced. When the replica extraction method for vanadium is established, the following experimental result will be available for research and development of vanadium alloys application.

For investigation of stoichiometric components in precipitate in Fe-9Cr modified steels after thermal ageing, the extraction replica method was applied for specimen preparation and a JEM-2100TM of TEM was used in Univ. of Fukui.

TEM evaluation and EDS were mapped for the extract replica sample from a Fe-9Crmod. steel. An EDS analysis for each precipitates for 50 seconds was performed and the information about ratio of metal abundance components in precipitate was obtained. A figure shows the ratio of metallic compound for total precipitate from 150 to 200 pieces. The precipitate comprised half of Cr-enriched carbides and the rest of V-X and Nb-X precipitates. The compositional information of precipitate is expected to be obtained in vanadium alloy after the establishment of technical settlement for extraction replica method in vanadium alloys soon.

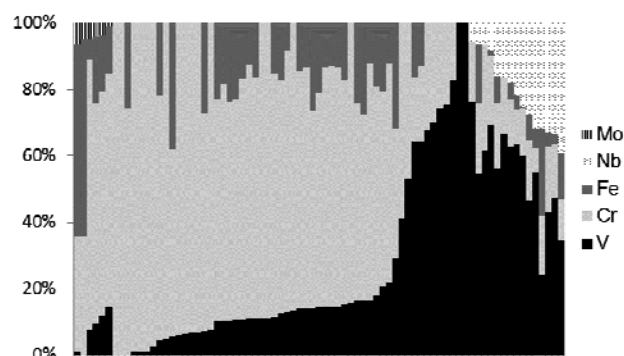


Fig. A result of chemical composition of metal element in precipitate in Fe-9Cr.mod steels