§5. Study on Radiation Induced Precipitates in Vanadium Alloys

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Introduction

Vanadium alloy is one of the excellent candidate materials for fusion reactors. Because of its good resistance to neutron irradiation, V-4Cr-4Ti, in particular, has been studied extensively in the past decades [1]. However, it has been reported that significant radiation hardening and embrittlement occurred in V-4Cr-4Ti irradiated at relatively low temperatures by neutrons. According to the previously reported TEM studies [2], radiation-induced titanium precipitates are the major cause of irradiation hardening, while the microstructural information is still far from comprehensive. Especially, the nature of black-dot clusters which were observed under irradiation at low temperatures is still not clear while the large size of Ti-precipitates with platelet shapes are relatively well characterized. The objective of this study is to investigate both the nature of black dotted cluster formed under low temperature irradiation and the chemical composition of titanium rich precipitates in vanadium alloy. For the analysis of fine precipitates, 3-dimensional atom probe microanalysis (3DAP) is a very powerful tool with its atomic scaled spatial resolution. In the present paper, experimental results using 3DAP combined with TEM observation on fine Ti-precipitates in vanadium alloys are presented.

Experimental procedures

V-4Cr-(0.1,1,3wt.%)Ti were prepared. Neutron irradiation was conducted in Japan Material Testing Reactor (JMTR) up to 0.2 dpa at 350°C. The damage rate was 9.4 × 10-8 dpa/sec. After electro polishing, TEM observation was performed using JOEL-2010 equipped with an energy dispersive X-ray spectrometry (EDX) detector. To prepare needle-like specimens for the 3 dimensional atom probe (3DAP) analysis, the irradiated tensile specimens were cut into small square rods of approximately 0.25 × 0.25 × 7 mm, which were subsequently electropolished to blunt needles. Finally a sharp needle tip were fabricated by the focused ion beam (FIB) processing with an annular gallium ion beam of 30 keV. 3DAP measurements were carried out using energy compensated position-sensitive atom probe facilities which consists of a reflectron energy compensator, a position sensitive detector and a high-resolution flight time detector. Measurements were conducted at a sample temperature of 60 K with the pulse fraction of 0.2 under an ultra-high vacuum condition.

Results and discussions

In TEM observations, platelet precipitates on {100} planes were observed in V-4Cr-4Ti-0.1Si, while a high density of small defect clusters was observed as black dotted contrast in V-4Cr-1Ti. Enrichment of titanium as well as oxygen and titanium oxide in the matrix were observed by 3DAP microanalysis in V-4Cr-3Ti, V-4Cr-1Ti, V-4Cr-0.1Ti (Fig.1), which may corresponds to the precipitates and black dotted defect clusters observed by TEM. The size of precipitates increased with the titanium concentration. Small cluster with a sphere shape were observed in V-4Cr-0.1Ti, while the platelet shape in other alloys. It could be deduced that the spherical cluster was on the nucleation stage of precipitates. In other words, the nucleation of titanium-rich precipitates might be started with the segregation of titanium and oxygen to dislocation loop. Nitrogen and carbon were also contained in the large precipitates of platelet shape in V-4Cr-3Ti, which will be explained by the difference in the extent of strain field depending on the coherency of precipitates.

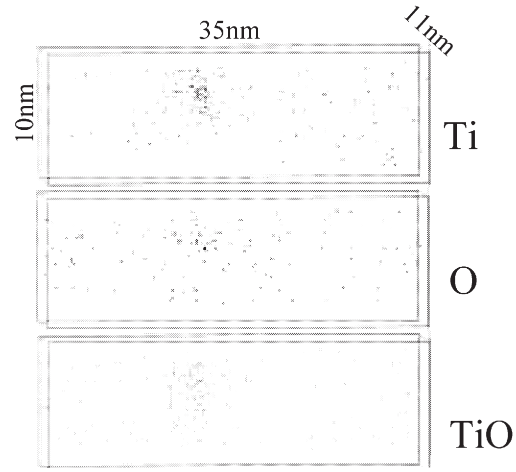

Fig.1 Atom maps of V-4Cr-0.1Ti

Conclusions

Neutron irradiated vanadium alloys were examined by TEM and 3DAP. A high density of small defect clusters was observed as black dotted contrast in V-4Cr-1Ti. Although it was difficult to determine the nature of these fine defect clusters by a conventional TEM with an EDX technique, enrichment of titanium as well as oxygen and titanium oxide in the matrix were observed by 3DAP microanalysis. It could be deduced that the spherical cluster was on the nucleation stage of precipitates. In other words, the nucleation of titanium-rich precipitates might be started with the segregation of titanium and oxygen to dislocation loop. It could be said that the precipitates grow with changing their morphology from sphere to platelet and the ratio of titanium to oxygen. The spherical cluster during the nucleation stage is TiO and the planar cluster during the growth stage is Ti_3O_2.

References

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