1.1 Stability of MX-Type Nanoprecipitates in Fe-Base Model Alloys — L. Tan, Y. Katoh, L. L. Snead (Oak Ridge National Laboratory)

OBJECTIVE

Study the stability of MX-type nanoprecipitates in reduced-activation ferritic (RAF) steels using Fe-base model alloys under thermal, stress, and radiation conditions to help understanding the failure mechanisms of RAF steels and identify promising strengthening particles for advanced RAF steel development.

SUMMARY

The stability of MX-type nanoprecipitates TaC, TaN, and VN has been evaluated using thermal aging at 600 and 700°C for up to 5000 h, creep testing at 600°C, and Fe^{2+} ion irradiation at 500°C and up to ~49 dpa. Transmission electron microscopy (TEM) has been primarily used to characterize the evolution of the nanoprecipitates. The results indicate different levels of stability of the nanoprecipitates under the three types of experimental conditions. Selected particle degradation modes, e.g., dissolution, growth, re-precipitation, and fragmentation, were observed on the nanoprecipitates. The results are being reported and compared to literature data in our recent papers. A parallel experiment using neutron irradiation at 300, 500, and 650°C for up to about 20 dpa has been completed recently. Post-irradiation examination (PIE) of the neutron-irradiated samples will be conducted to compare them with the Fe^{2+} ion irradiated results.

PROGRESS AND STATUS

Introduction

MX-type precipitates have shown excellent coarsening resistance compared to Laves phase and chromium-rich $M_{23}C_6$ in 9-12% Cr ferritic-martensitic steels as well as RAF steels. The MX-type precipitates are believed to be critical for high temperature strength of this class of steels. Recently, however, limited data have shown the instability of MX, e.g., VN, TaC, and TaN, at elevated temperatures and irradiation conditions [1-4]. Therefore, three model alloys, respectively favoring the formation of TaC, TaN, and VN nanoprecipitates, had been designed and prepared to investigate the stability of these nanoprecipitates under thermal aging, creep testing, and irradiation conditions.

Experimental Procedure

Samples of the TaC, TaN, and VN bearing model alloys were subjected to thermal aging at 600 and 700°C for up to 5000 h, creep testing at 600°C, and Fe^{2+} ion irradiation at 500°C for up to ~49 dpa. The microstructures of the samples were characterized using transmission electron microscopy (TEM) in both conventional and scanning modes (STEM) on a FEI CM200 field-emission-gun TEM/STEM equipped with an EDAX energy dispersive X-ray spectroscopy (EDS) detector. TEM specimens, perpendicular to the surfaces, were lifted out and thinned to electron-transparent using focused ion beam (FIB) on a Hitachi NB5000. Specimen thickness

of the characterized regions was estimated using convergent beam electron diffraction (CBED) technique.

Results

The thermal aging experiment exhibited different effects on the stability of the nanoprecipitates. The lower temperature aging at 600°C basically led to coarsening in different degrees, accompanied with corresponding reduction in number density. In contrast, the higher temperature aging at 700°C resulted in a bimodal phenomenon to the nanoprecipitates. Figure 1 shows an example of TaC nanoprecipitates after aging at 600 and 700°C for 5000 h. Compared to the slightly coarsened TaC particles at 600°C in the bright-field (BF) image, 700°C-aged sample in the dark-field (DF) image is predominant with ultrafine TaC particles, together with a few large particles that have size similar to the 600°C-aged sample. The high-resolution image shows lattice fringes of two large particles and one ultrafine particle pointed with a white arrow. The ultrafine particle having a different orientation from the two large particles suggests that the ultrafine particle may have been re-precipitated during the 700°C aging.

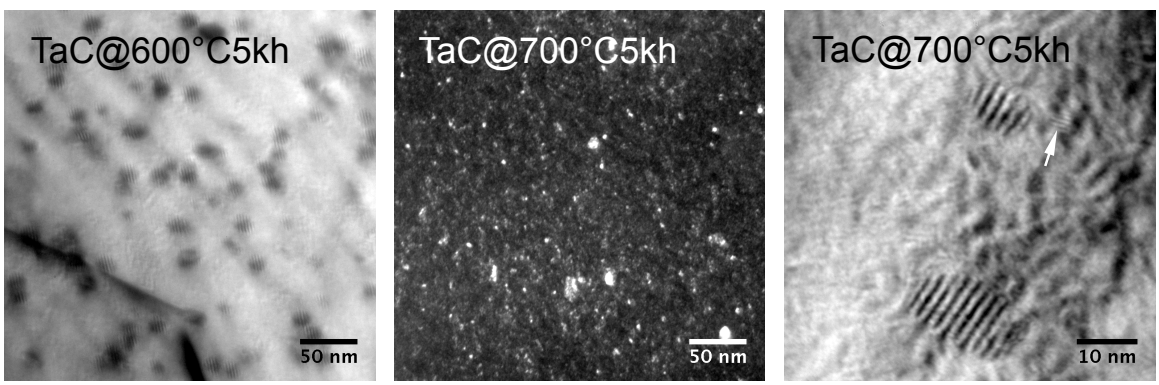

Figure 1. Effect of thermal aging at 600 and 700°C for 5000 h on the stability of TaC nanoprecipitates.

Microstructures at both the tab and gauge sections of the 600°C creep-tested samples were characterized using TEM. The tab section is assumed to be stress-free other than the stress-intensified regime at gauge section. This assumption is reasonable as the characterized results at the tab section are generally consistent with the thermal aging results at 600°C. The stress at gauge section significantly altered the geometry of TaN particles, as shown in DF images of Figure 2. The aligned plate-shaped particles were getting thinner and fragmented under the stress, which significantly reduced the size and increased the number density of the particles. Unlike TaN particles, the stress did not alter the geometry but slightly or noticeably increased the size of the TaC and VN particles.

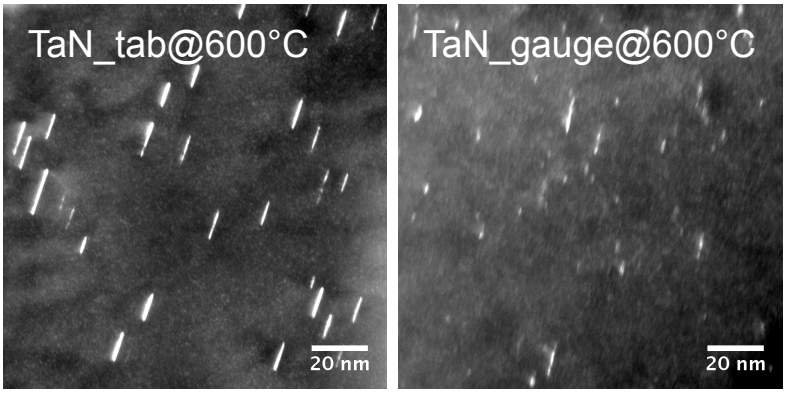

Figure 2. DF TEM images of the tab and gauges sections of the TaN sample after creep testing at 600°C and 170 MPa.

The Fe^{2+} ion irradiated samples were characterized using FIB-ed TEM specimens. Figure 3 shows an example of the VN-bearing sample from the irradiated surface on left to the matrix on right of the DF image. The irradiation damage (dpa) depth profile in the model alloy, aligned beneath the DF image, was simulated using the stopping and range of ions in matter software SRIM-2013 [5] with parameters recommended by Stoller *et al.* [6]. The length, thickness, and number density of the particles were statistically analyzed as a function of the irradiation depth as well as irradiation dose (dpa) according to the SRIM-simulated result. Similar analyses were conducted for the TaC and TaN particles.

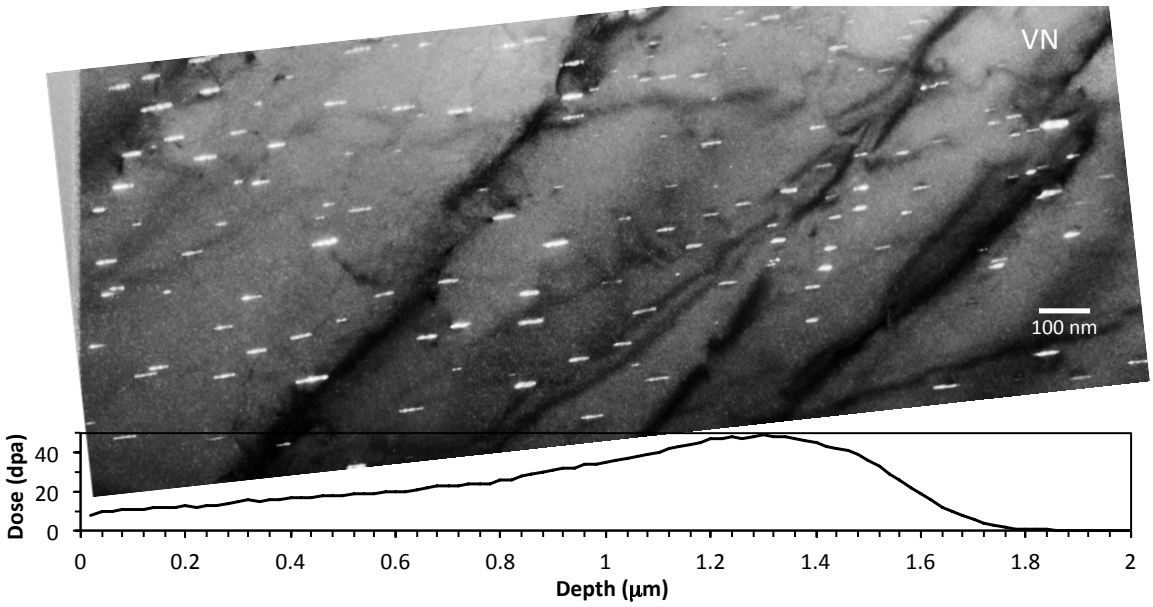

Figure 3. DF TEM image of the VN sample after Fe^{2+} irradiation at 500°C, accompanied with the simulated depth profile of irradiation dose in dpa using SRIM-2013.

Compared to the nitrides of TaN and VN, generally, TaC nanoprecipitates exhibited relatively greater stability in terms of thermal aging, creep testing, and Fe^{2+} irradiation resistance. Both consistency and discrepancies with literature reported results are discussed in our recent papers [7,8]. Neutron irradiation experiment of the model alloy samples has been completed recently. The PIE of the samples will be conducted to compare them with the Fe^{2+} ion irradiated results.

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