1.1 Stability of 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 prominent strengthening particles for advanced RAF steel development.

SUMMARY

The stability of VN, TaC, and TaN under thermal, stress, and radiation conditions is being investigated. Isothermal aging experiments at 600 and 700°C for 100, 1000, and 5000 h have been completed. VN and TaC showed similar resistance to thermal aging at 600 and 700°C. TaC may be slightly more resistant. Further study is in progress including TaN particles. The samples have been subjected to creep tests at 600°C and 170 MPa for TaC and TaN and 100 MPa for VN. Unlike VN and TaC, TaN was not resistant to the creep, which was broken into small pieces. Irradiation experiments, using Fe^{2+} ions at 500°C for 20 and 200 dpa and neutron at 300, 500, and 650°C for up to about 20 dpa, are in progress. Post-irradiation examination will be pursued.

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. They are 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 [1-3].

Experimental Procedure

Three model alloys, Fe-1WVN, Fe-1WTaC, and Fe-1WTaN, had been designed and procured. The alloys have been confirmed having VN, TaC, and TaN in nanometers respectively. Type SS-3 specimens were used in isothermal aging experiments and creep tests. The specimens were sealed in helium filled quartz tubes and isothermally aged at 600 and 700°C for 100, 1000, and 5000 h. Creep tests were conducted at 600°C using constant loads of ~70% of the yield stress of the alloys. Specimens were also prepared for Fe^{2+} ions and neutron irradiation experiments. The microstructural evolution of the nanoprecipitates was primarily characterized using transmission electron microscopy (TEM).

RESULTS

The isothermal aging experiments at 600 and 700°C h for up to 5000 h have been completed. Most of samples have been characterized except for a few aged samples of Fe-1WTaN. Using Fe-1WVN alloy as an example, Figure 1 shows bright-field and dark-field TEM images of the

samples in the control, 600°C for 5,000 h and 700°C for 1,000 h aged conditions. Statistical measurements indicate that the density of VN was slightly increased (~11%) with some decrease in particle size after 5,000 h aging at 600°C. In contrast, the particles were significantly refined with a noticeable increase in density after aging at 700°C for 1,000 h, which suggest the occurrence of dissolution and/or precipitation at this temperature. The sample aged at 700°C for 5,000 h is being prepared for TEM characterization. The results are expected to provide more insights on the particle stability. No significant changes were observed for TaC aged at 600°C for 5,000 h. However, the 700°C aging for 1,000 h also exhibited some refinement of TaC particles.

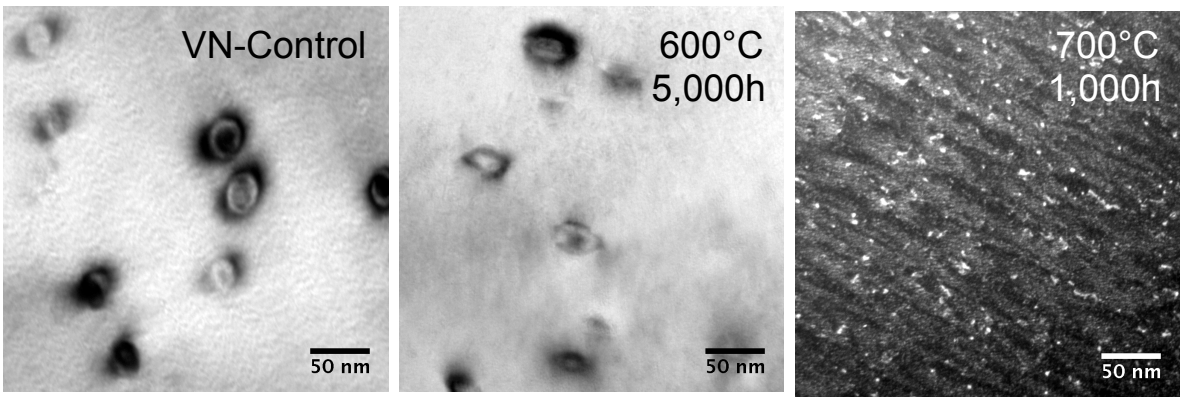

Figure 1. Bright-field (BF) and dark-field (DF) TEM images showing the evolution of VN nanoprecipitates under thermal aging at 600 and 700°C.

Creep tests have been completed at 600°C with a load of ~70% yield stress for the alloys. Figure 2(a) shows the creep life of the tested samples compared to the literature date of F82H [4]. Despite the low alloy of Fe-1W base metal, the Fe-1WTaC exhibited creep life (~371 h) closing to F82H. The Fe-1WVN showed the least creep resistance in the three alloys. The order of creep resistance of the alloys is consistent with the corresponding particle volume fraction in the alloys as shown in Figure 2(b). The larger volume fraction of TaC benefited the higher strength and greater creep life of Fe-1WTaC compared to the TaN and VN bearing alloys.

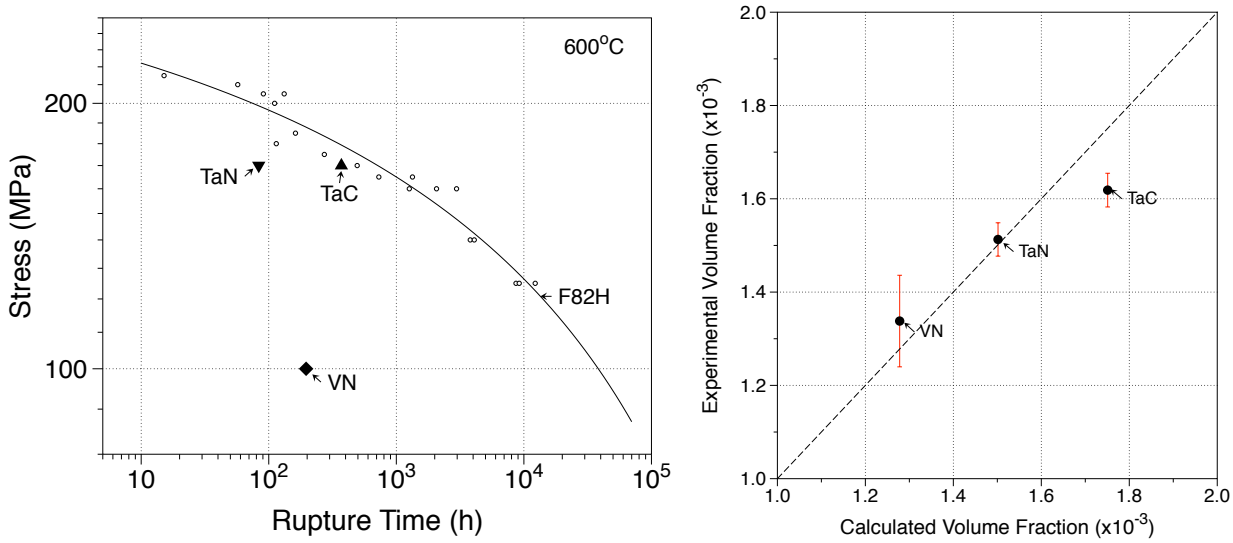

Figure 2. (a – left) Creep rupture time of alloys Fe-1WTaC, Fe-1WTaN, and Fe-1WVN compared to literature data of F82H; (b – right) Calculated and experimental volume fraction of TaC, TaN, and VN precipitates in alloys Fe-1WTaC, Fe-1WTaN, and Fe-1WVN, respectively.

Microstructural characterization indicated that the stress in the gauge section of the creep samples did not result in significant changes to the size/shape of VN and TaC particles. In contrast, the TaN particles were broken into small pieces under the stress of 170 MPa. Additionally, a few Fe_2W-type Laves phase particles with a size of ~100 nm were observed at grain boundaries in the gauge section of the creep ruptured Fe-1WTaC sample, which were not observed in the "unstressed" tab section of the creep sample. In comparison, Laves phase in a size of ~30 nm was found at grain boundaries in the Fe-1WTaC sample aged at 600°C for 1,000 h. The results suggest that the formation of the Laves phase was greatly accelerated by the stress during creep.

Fe^{2+} ion irradiation of the alloy samples has completed 20 dpa at 500°C at University of Michigan. The ion-irradiated samples have been received at ORNL and are ready for TEM specimen preparation using focused-ion-beam (FIB). The 200 dpa irradiation at 500°C is in progress. Additionally, a set of type SS-J2 specimens of the alloys has being neutron-irradiated, using HFIR at ORNL, at 300, 500, and 650°C to damage levels ranging from ~0.1 dpa to ~20 dpa. Post-irradiation examination including tensile tests at room temperature and selected TEM characterization will be pursued.

REFERENCES

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