OXIDATION KINETICS AND MICROSTRUCTURE OF V-Cr-Ti ALLOYS EXPOSED TO OXYGEN-CONTAINING ENVIRONMENTS*

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OBJECTIVE

The objectives of this task are to (a) evaluate the oxygen uptake of several V-Cr-Ti alloys as a function of temperature and oxygen partial pressure in the exposure environment, (b) examine the microstructural characteristics of oxide scales and oxygen trapped at the grain boundaries in the substrate alloys, and (c) evaluate the influence of alloy composition on oxygen uptake and develop correlation(s) between alloy composition, exposure environment, and temperature.

SUMMARY

A systematic study is being conducted to determine the effects of time, temperature, and exposure environment on the oxidation behavior and microstructure of V-Cr-Ti alloys. All samples were from 1-mm-thick cold-rolled sheets, and each was annealed in vacuum at 1050°C for 1 h prior to high-temperature exposure. Different samples from each alloy were heated in air and low -oxygen environments at temperatures between 400 and 650°C for times up to a few hundred hours. Some exposures were conducted in a thermogravimetric analysis (TGA) apparatus, in which continuous measurements of weight change were recorded.

EXPERIMENTAL PROGRAM

The alloys selected for evaluation included vanadium, V-10 wt.%Cr, V-4 wt.%Cr-4 wt.%Ti, V-5 wt.%Cr-5 wt.%Ti, V-10 wt.%Cr-5 wt.%Ti, and V-15 wt.%Cr-5 wt.%Ti alloys. The alloys were obtained in 1-mm-thick sheets. Samples measuring about 1 x 10 x 20 mm were cut from each alloy. Before any further treatment or testing, all samples were annealed for 1 h at 1050°C under a pressure of $\approx 10^{-6}$ torr. The samples were wrapped in tantalum foil to protect them from contamination during this heat treatment process. Samples from each alloy were heated in a TGA apparatus in air at different temperatures to determine oxidation kinetics as a function of temperature. The TGA experiments were carried out at temperatures in a range of 320-650°C. Weight gain was recorded continuously on a strip chart throughout each experiment [1,2]. All samples were also weighed separately before and after any high-temperature exposure to determine the resulting total weight change. In addition, specimens of several of the alloys were also exposed to low-pO_2 environments in a vacuum system with a feed and bleed arrangement. Oxygen pressure in the sample exposure chamber was maintained at 0.1, 4×10^{-4}, or 1×10^{-6} torr oxygen. Samples were retrieved periodically, weighed, and re-exposed to obtain kinetic data.

The oxide scales on the samples were identified by X-ray diffraction (XRD) analysis on the surfaces of several samples, as well as on the oxides scraped from their surfaces. Metallographic examination of the longitudinal and transverse cross-sections of the cold-rolled and thermally treated samples used both an optical microscope and a scanning electron microscope (SEM). The metallographic specimens were chemically etched with a solution of lactic-nitric-hydrofluoric acids at a volume ratio of 30-15-5. The grain sizes of each sample exposed to high temperature (annealed or oxidized) are being determined by both lineal and areal analysis methods according to ASTM Standard E112, and the average of the two is reported as the grain size of each sample. Oxygen diffusion depth (or the depth of the hardened layer) of each oxidized sample is being estimated from the microhardness profile along its thickness, which is obtained with a Vickers microhardness tester and a load of 25-50 g.

*This work has been supported by the U.S. Department of Energy, Office of Fusion Energy Research, under Contract W-31-109-Eng-38.

RESULTS

Extensive studies were conducted on the oxidation kinetics of V-4Cr-4Ti and V-5Cr-5Ti alloys over a temperature range of 300-650°C; the results were reported earlier [1-3]. The current oxidation study on several of the V-Cr-Ti alloys supplements the data developed earlier on those two alloys. Figure 1 shows normalized weight changes (in mg/mm^2), obtained by TGA, of several V-Cr-Ti alloys in air at temperatures in a range 475-550°C. Additional experiments are in progress at other temperatures and will be used to develop correlations between oxidation kinetics, alloy composition, and exposure temperature.

During this period, experiments were also continued to evaluate the influence of pO_2 on the oxidation kinetics of V-Cr-Ti alloys. Figure 2 shows the weight change data obtained for the two alloys in a pure oxygen environment at pressures of 0.1, 5×10^{-4}, and 5×10^{-6} torr and temperatures of 500, 600, and 700°C. The preliminary indication is that for a given exposure time, oxygen enrichment of the alloys at lower oxygen pressures may be greater due to absence of and/or differences in oxide type and morphology that develop at different pressures. The exposed specimens are being analyzed for oxide type by XRD and oxygen diffusion in the alloy by hardness measurements.

REFERENCES

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[3] K. Natesan and M. Uz, "Oxidation Kinetics and Microstructure of V-(4-5) wt.%Cr-(4-5) wt.%Ti Alloys Exposed to Air at 300-650°C," Fusion Reactor Materials Semiannual Progress Report for Period Ending June 30, 1996, DOE/ER-0313/20, p. 105, Oct.1996.

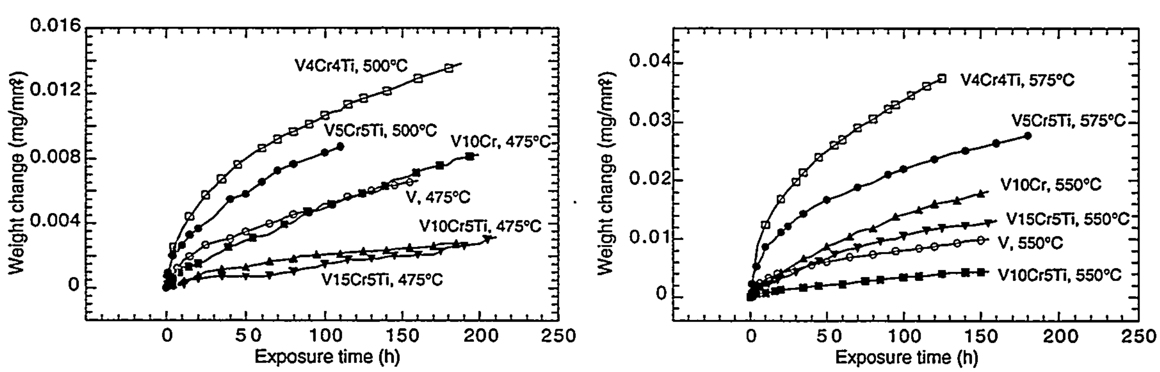

Figure 1. Thermogravimetric weight change data for several V-Cr-Ti alloys exposed to air at 475-550°C.

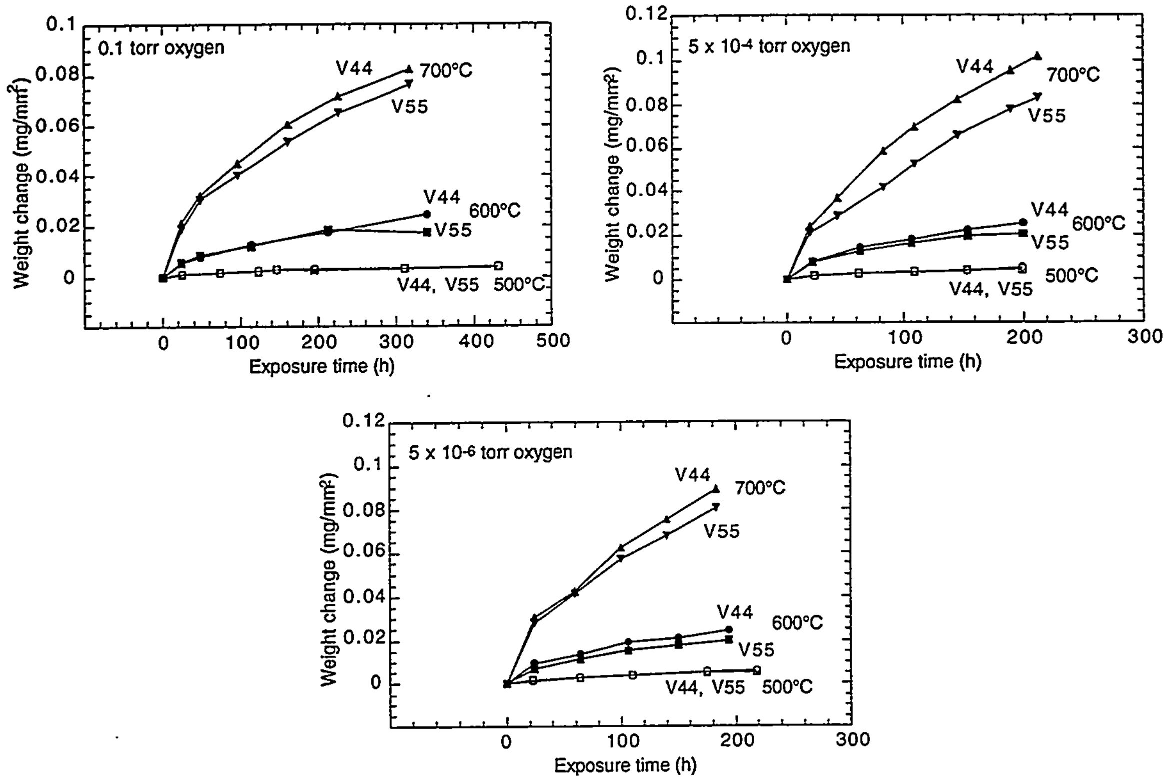

Figure 2. Weight change vs. time data for V-4Cr-4Ti and V-5Cr-5Ti alloys in oxygen environment at pressures of (top) 0.1 torr, (middle) 5×10^{-4} torr, and (bottom) 5×10^{-6} torr