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MASTER

THERMAL STABILITY OF FERRITIC ALLOYS
FOR FOSSIL-FUEL PROCESSING SYSTEMS

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The experimental work within the recent reporting period has concentrated on the application of refined electrical resistance measurements and analytical techniques to quantify the thermal instability of ternary Fe-Cr-Al alloys as a function of alloy composition and temperature. The aging reactions are being examined over the temperature range where 475°C embrittlement is of concern in commercial ferritic stainless steels of similar Cr contents.

TERNARY Fe-Cr-Al ALLOYS

The variation in electrical resistance from the solution-treated and quenched value is shown in Figure 1 for Fe-Cr-Al alloys as a function of the isothermal aging time at 400 and 500°C; similar data for aging at 475°C is shown in Figure 2. These curves point to several interesting features concerning the aging behavior of Fe-Cr-Al alloys:

1. Within the first ten hours of aging, the magnitude of the resistance drop increases with increasing aging temperature. This behavior is tentatively attributed to the precipitation of carbides and nitrides from solid solution with the rate of precipitation increasing with

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temperature. Confirmation of this hypothesis awaits careful examination of appropriately aged thin foils by means of transmission electron microscopy techniques.

2. Decomposition due to aging appears to be a single-stage process at 450°C, while at 475°C a two-stage process occurs in these alloys, with the exception of the Fe-15Cr-6Al alloy. The resistance behavior at 500°C shows that only the 18 Cr alloys undergo a two-stage aging process, while the aging of the 15 Cr alloys is confined to a single stage.
3. Some tentative conclusions can be drawn regarding the effect of composition on the instability of these alloys. The magnitude of the resistance drop for the two 18 Cr alloys is larger than that for the 15 Cr alloys at all three aging temperatures, indicating that raising the Cr content increases the amount of microstructural decomposition. This is as expected if the precipitation of the embrittling Cr-rich alpha prime phase is the principal cause of the resistance decrease.

The effect of Al content on the metallurgical stability of Fe-Cr-Al alloys has not yet been clearly resolved. Higher Al content decreases the magnitude of the resistance drop due to aging in the 15 Cr alloys at 450 and 475°C; however, higher Al content in the 18 Cr alloys reduces the resistance drop at both 450°C and 500°C, but increases the drop at 475°C. For all but the Fe-18Cr-5Al alloy aged at 475°C and the Fe-15Cr-6Al aged at 500°C, the lower resistance decrease with increasing Al content suggests that Al is beneficial in reducing the rate and amount of alpha prime precipitation. Additional resistance experiments and TEM observations are necessary to clarify and confirm the above trends as a basis for developing firm conclusions regarding the effect of varying amounts of aluminum on the thermal stability of Fe-Cr-Al alloys.

Resistance measurements at two temperatures, 0°C and -196°C, have been performed on the ternary alloys aged at 475°C. By plotting the resistance at 0°C versus that at -196°C the presence or absence of any changes in specimen geometry or dimensions (including internal cracking and void growth) can be detected. For all of the ternary alloys the resistance data for isothermal aging times fall on a straight line, thereby indicating that the resistance decrease is due solely to decreases in specimen resistivity and not the result of any change in specimen geometry. The slope of the line is unique for each alloy composition and decreases sharply with increasing Al content. The variation of the slope of these resistivity plots with aluminum content and aging temperature may provide a basis for comparing the kinetics of the decomposition reactions in these alloys; this approach is to receive further consideration.

Recent work in the present program has shown that the resistance ratio $\frac{R_{0^\circ\text{C}}}{R_{-196^\circ\text{C}}}$ increases with aging time while exhibiting similar apparent reaction stages to those revealed by plotting the electrical resistance ($R_{-196^\circ\text{C}}$) versus aging time at 475°C. The resistance ratio $\frac{R_{0^\circ\text{C}}}{R_{-196^\circ\text{C}}}$ was previously determined to be strongly dependent on the composition parameter, At.% (Fe+Cr) Al.

BINARY Fe-Cr ALLOYS

Preliminary aging studies have been conducted on two binary Fe-Cr alloys with Cr contents corresponding to those of the ternary alloys. The resistance data taken at -196°C on Fe-15Cr aged at 475°C is consistent with the trends of the data for the ternary alloys except that a larger percent decrease occurs in the electrical resistance in comparison to that of the ternary alloys. This is interpreted as further evidence that Al is beneficial in reducing the

severity of microstructural instability at 475°C. The data also indicate the occurrence of a single-stage decomposition process, in contrast to that of the ternary alloys.

Two Fe-18Cr specimens have shown anomalous resistance behavior due to aging at 475°C. Resistance measurements at -196°C have shown that an initial sharp drop in resistance within the first hour of aging is followed by a continuous and steady increase over aging periods up to 900 hours. The results of the dual temperature resistance plotting method described earlier indicate erratic behavior, suggesting changes in the specimen geometry such as internal cracking. Additional Fe-18Cr binary samples are being carefully prepared in order to check the validity of these recent results.

X-ray diffractometer experiments on the Fe-18Cr-5Al alloy aged various lengths of time at 475°C have proven to be of little value in characterizing the instability reaction. This is probably due to the expected small volume fraction of precipitate particles for these low Cr alloys. Precipitate extraction techniques may give better results, and it is expected that this method, together with the observations of thin foils with a Philips EM-300 transmission electron microscope (currently being installed) will make it possible to identify the structural mechanisms accompanying the reaction stages. This information is needed to develop meaningful quantitative descriptions of the reaction kinetics from resistance data. Experimental work within the next reporting period will concentrate on TEM observations and further resistance measurements on the binary and ternary alloys in order to understand and quantify the reactions responsible for 475°C embrittlement. An important objective of the research is the formulation of kinetic equations describing the effect of temperature and composition on the thermal instability of these ferritic stainless steels.

Figure 1: Electrical Resistance Decrease versus Aging Time - Ternary FeCrAl Alloys at 450 and 500°C.

Figure 2: Electrical Resistance Decrease versus Aging Time - Ternary Fe-Cr-Al alloys at 475°C.