THERMAL AGING OF PRIMARY COOLANT PIPE STEEL. CONF-850840--14

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The long term mechanical integrity of the pipes used to carry the primary cooling water in a pressurized water nuclear reactor is of the utmost importance for safe operation. A combined atom probe field-ion microscopy (APFIM) and transmission electron microscopy (TEM) study was performed to characterize the microstructure of this cast stainless steel and to determine the changes that occur during long-term low-temperature thermal aging.

The material used in this investigation was a commercial CF 8 type stainless steel with a bulk chemical composition as given in Table 1. The steel was examined in the as-cast, unaged condition and also after aging for 7500 h at 673K. This temperature is 100K higher than the normal service temperature and was chosen to accelerate the microstructural changes that may occur during service. As these pipes are external to the reactor they are not exposed to any significant radiation that may influence the aging behavior. The microstructure of the as-cast steel was found to be a duplex mixture of austenite with 19% ferrite. The composition of the austenite and ferrite of the as-cast unaged material as determined by X-ray microanalysis is given in table 2.

A TEM micrograph of the aged material is shown in figure 1. FIM micrographs of the austenite and ferrite phases of the aged steel are shown in figures 2 and 3 respectively. Two marked changes in the microstructure and microchemistry of the ferrite were found to accompany low temperature aging. Numerous small, roughly spherical precipitates, approximately 10 nm in diameter, were found distributed throughout the ferrite with a concentration of $10^{23} m^{-3}$. The precipitates exhibited bright contrast in the FIM micrographs. This type of precipitate was not observed in the as-cast unaged material nor in the austenite in the aged material. The bright spots in the FIM micrograph of austenite are probably due to the presence of Mo and Si and are typical for micrographs of austenite containing large amounts of solute. Atom probe selected area microchemical analysis and TEM analysis revealed that the precipitates were alloy silicides and were identified as G-phase [1].

Atom probe compositional analyses were made of the ferrite phase, avoiding the precipitates. The composition profiles revealed that the Cr-enriched ferrite matrix had separated into an extremely fine mixture of an Fe-rich phase and a Cr-enriched phase as shown in figure 4. This phase separation is not observable in the TEM because of the fine-scale and nature of the separation and the lack of any significant contrast mechanisms. The composition of the ferrite phase is in the range where a miscibility gap exists at low temperatures. Indeed the phase separation of the ferrite into a Cr-enriched and an Fe-rich phase is similar to that observed in Fe-Cr and Fe-Cr-X systems which undergo isotropic spinodal decomposition within a miscibility gap.

The presence of both the silicide precipitates and the fine scale spinodal decomposition of the ferrite could contribute to the changes observed in the mechanical properties. However, the increase in hardness that was observed is similar in magnitude to that previously found in a spinodally decomposed Fe-30% Cr alloy [2]. The results of this study have revealed the complexity and the ultra-fine scale of the decomposition processes that occur at low temperature in CF 8 type stainless steel. This study has shown that the near atomic resolution of the atom probe was able to detect and quantify the

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extremely fine-scale phase separation that occurred and has also demonstrated the benefits of using the combined techniques of TEM and APFIM [3].

REFERENCES

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2. S.S. BRENNER, M.K. MILLER and W.A. SOFFA, Scripta Met., 16 (1982) 831.
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TABLE 1. Composition of CF 8 type stainless steel.

Element	Cr	Ni	Mo	Mn	Si	C	P	S	N	Fe
(at %)	22.1	9.9	1.4	.79	1.6	.18	.027	.037	.165	balance

TABLE 2. X-ray microanalysis of the unaged material (at% ± 1 std. dev.).

	Cr	Ni	Mo	Mn	Si	Fe
Austenite	22.3 ±.43	10.8 ±.46	1.8 ±.16	1.1 ±.06	1.7 ±.09	62.3 ±.74
Ferrite	28.5 ±1.06	6.1 ±.25	2.8 ±.19	0.8 ±.18	1.9 ±.10	60.6 ±.96

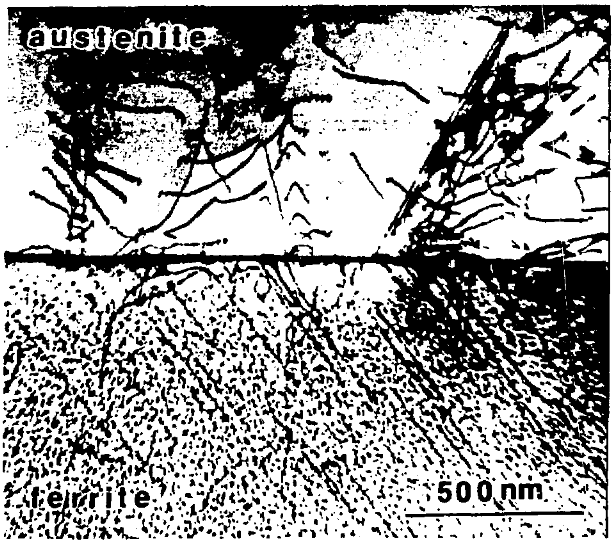

FIG. 1. TEM micrograph of austenite and ferrite + G-phase in aged CF 8 Steel.

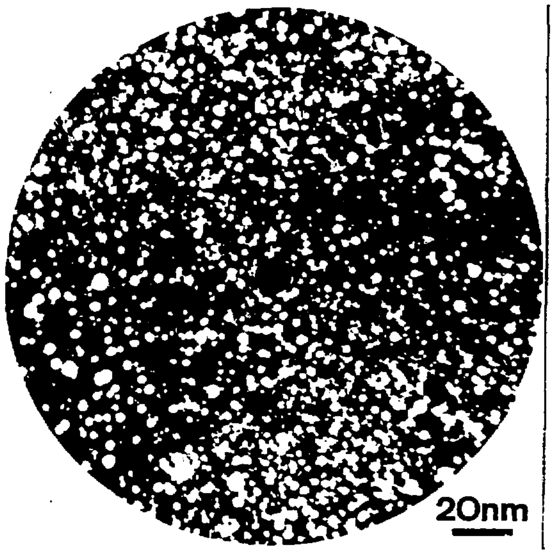

FIG 2. FIM micrograph of precipitate free austenite in aged CF 8 steel.

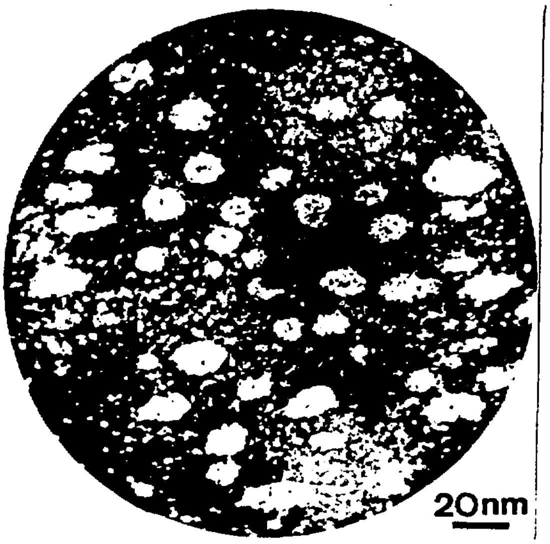

FIG 3. FIM micrograph of ferrite containing G-phase precipitates.

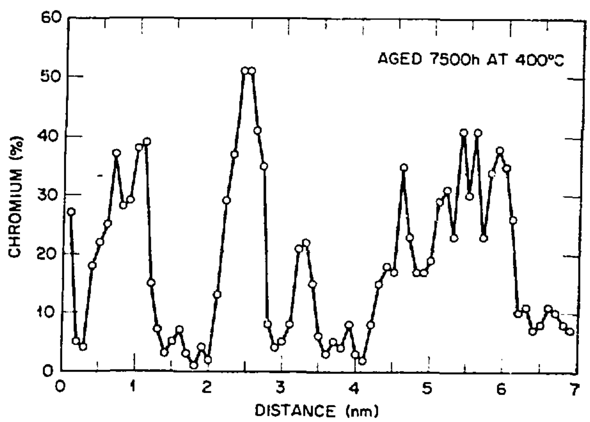

FIG 4. Atom Probe composition profile through ferrite showing separation into Fe-rich and Cr-enriched phases.