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Strengthening by intermetallic nanoprecipitation in Fe–Cr–Al–Ti alloy



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ABSTRACT

The strengthening mechanism observed during ageing at temperatures of 435 and 475 °C in the oxide dispersion strengthened (ODS) Fe–Cr–Al–Ti system has been investigated. Atom probe tomography (APT) and high-resolution transmission electron microscopy (HRTEM) analyses determined that the alloy undergoes simultaneous precipitation of Cr-rich (α' phase) and nanoscale precipitation of TiAl-rich intermetallic particles (β' phase). APT indicated that the composition of the intermetallic β' phase is $\text{Fe}_2\text{AlTi}_{0.6}\text{Cr}_{0.4}$, and the evolving composition of α' phase with ageing time was also determined. The results obtained from HRTEM analyses allow us to confirm that the β' precipitates exhibit a cubic structure and hence their crystallography is related to the Heusler-type Fe_2AlTi ($L2_1$) structure. The strengthening could be explained on the basis of two hardening effects that occur simultaneously: the first is due to the α - α' phase separation through the modulus effect, and the second mechanism is due to the interaction of nanoscale β' particles with dislocations.

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1. Introduction

High-Cr ferritic steels are widely used for heat resistant structural applications in nuclear and thermal power plants and fast breeder reactors due to the favorable combination of properties, such as good swelling resistance, low coefficient of thermal expansion, high thermal conductivity, good oxidation and creep resistance, and high tensile/compressive strength at ambient and elevated temperature. However, the use of ferritic steel is limited to temperatures below 550 °C due to an inadequate creep strength above that limit. In order to overcome this limitation, oxide dispersion strengthened (ODS) ferritic steels have drawn much attention due to their excellent high-temperature tensile strength and creep resistance [1–4].

Besides the homogeneous dispersion of nano-sized oxides in the ferritic matrix, ODS ferritic steels usually exhibit high-strength and creep resistance properties as a consequence of being far from the equilibrium state [5,6]. Many features, such as a large proportion of

interfaces and triple junctions, possible irregular distributions of alloying elements and admixtures, the occurrence of non-equilibrium phases and supersaturated solutions, residual stresses, and excess concentrations of lattice defects, increase the Gibbs free energy. All these features are closely connected with the non-equilibrium conditions of the ODS fabrication methods, such as powder technology [7,8]. Such conditions are prone to the formation of nanoscale precipitates that will significantly increase both the strength and creep resistance of the ODS ferritic steel.

In this paper, we study the simultaneous nanoscale precipitation processes that occur in an ODS ferritic steel during ageing at temperatures of 435 and 475 °C, i.e., the α - α' phase separation and β' intermetallic precipitation. The goal is not only to explore the nature and mechanism of precipitation of TiAl-rich (β' -phase) particles, but to describe the interaction with the Cr-rich α' -particles resulting from the α - α' phase separation, and the strengthening mechanisms induced by these nanoscale precipitation processes.

2. Materials and experimental techniques

PM 2000 TM is a yttria dispersion strengthened, mechanically

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alloyed, ferritic steel containing ~20 Cr and 5 Al (wt.%) for oxidation and corrosion resistance. After mechanical alloying, the alloyed powder is canned, hot-extruded and hot-rolled into bar form [9–11]. The PM 2000™ alloy used in this study was provided by PLANSEE GmbH in the form of as-rolled bars. The chemical composition of the commercial purity PM 2000™ used in this study as determined by X-ray Fluorescence is given in Table 1.

The inspection of the microstructure was done using a combination of atom probe tomography (APT) and high-resolution transmission electron microscopy (HRTEM) imaging. TEM thin foils were prepared from cylindrical samples of 3 mm diameter which were cut along the rolling direction and they were mechanically thinned down to 0.05 mm. Later, these discs were electropolished in a double jet Struers TenuPol-5, using a 5% perchloric acid, 15% glycerol and 80% ethanol mixture at –6 °C and 20 V. HRTEM examination was performed with a JEOL model JEM 3000F microscope, operating at 300 keV and with a point-to-point resolution of 0.17 nm.

In this study, two local-electrode atom probes (LEAP®) (CAMECA instruments Inc. LEAP 2017 and 4000× HR) were used to quantify the size and composition parameters of the α - α' phase separation and β' precipitation. APT specimens were cut from bulk material and electropolished with the standard double layer and micro-polishing methods [12]. For some specimens, focused ion beam (FIB) technique was used in the final stage of specimen preparation. The LEAP® was operated in voltage pulse mode with a specimen temperature of 50 K, a pulse repetition rate of 200 kHz, and a pulse fraction of 20%. Only data sets with more than 2.5 million atoms were used to ensure that the reconstructed volume of material is sufficient to characterize the phase separation.

3. Results and discussion

The relative increase in hardness, as compared with the as-hot rolled condition, during ageing treatments at 435 and 475 °C is shown in Fig. 1. An abrupt increase of ~75% was observed in the aged condition of 435 °C for times longer than 100 h. However, the increase experienced at 475 °C is not as pronounced as the one observed in samples aged at 435 °C. The goal of this paper is to clarify the reason for this increment in the relative hardness.

3.1. Nanoscale α - α' phase separation

It has been reported previously [13–15] that the α - α' phase separation process is the main hardening mechanism during ageing for temperature ranging between 400 and 515 °C. The misfit effect between α and α' phases and the incremental change of Young's modulus (modulus effect), as a consequence of the corresponding decrease in the lattice parameter during phase separation, are the main contributors to the hardening of PM 2000™ during aging [16].

Fig. 2 displays the isoconcentration surfaces obtained from APT analysis for the resulting microstructure after ageing at 435 °C and 475 °C. The size and morphology of the Cr enriched α' can be observed from these isosurfaces: For the long ageing time, the roughly spherical α' particles are isolated and do not form a percolated microstructure. The morphology of these α' particles is similar to the one previously observed in Fe-19 at% Cr binary alloys

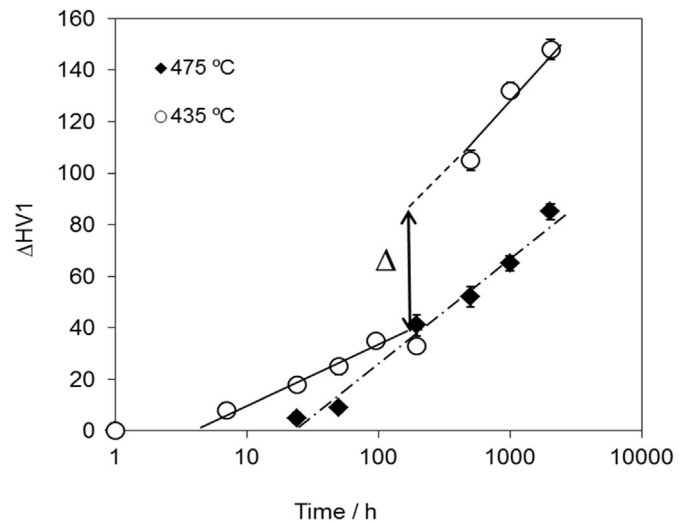


Fig. 1. Incremental hardness evolution (ΔHV) during ageing time at 435 and 475 °C in PM 2000.

and distinctly different to the interconnected network structure observed in higher chromium content (24–45 at%) binary alloys [17]. At the low ageing temperatures of 435 °C, the morphology of the Cr-enriched α' phase is of a finer scale and keeps an interconnected network after much longer ageing times than that observed at 475 °C, as seen in Fig. 2.

The composition of the α and α' phases was evaluated with the use of proximity histograms [18]. These are profiles of local atomic concentrations vs. proximity to an isoconcentration surface whose composition is selected to represent an interphase. In this work 30 at% Cr isoconcentration surfaces were selected to systematically study the composition of the α and α' phases. Table 2 summarizes the compositions of the α and α' phases for samples aged at 435 °C and 475 °C during different times.

The chromium level in the center of α' regions progressively increases with ageing time for both temperatures. The observation that the concentration of α' phase is not constant indicates that phase separation cannot be described simply by classical nucleation and growth processes. Besides, previous work [15] reported a value of activation energy for α - α' phase separation in PM 2000™ of 264 kJ mol⁻¹ determined by means of thermoelectric power measurements. This value is close that of 248 kJ mol⁻¹ for self-diffusion of Cr in α -Fe [19], which is consistent with the assumption that α - α' phase separation is driven by Cr-diffusion and the activation energy for nucleation, if any, should be small.

It could be argued, at least to a first approximation, that the variations in the volume fraction, morphology, and size of α' particles between the two ageing conditions, might be responsible for the increase in hardness shown in Fig. 1. Therefore, each of these parameters was examined.

A two-fold strategy was used to determine the volume fraction of the α' phase. First, the volume fraction was calculated from the miscibility gap in the phase diagram. Secondly, an experimental determination was made from the lever rule of the APT data.

The positions of the miscibility gap and spinodal region in the Fe–Cr–Al–Ti system were established from the Computer Coupling of Phase Diagrams and Thermochemistry (CALPHAD) formulation with the commercial software MT-DATA thermodynamic database [20]. Following the method described elsewhere [19], the free energy of mixing (ΔG_{mix}) was determined. The variation of ΔG_{mix} with Cr content was evaluated for temperatures ranging between 300 and 700 °C. The Fe–Cr miscibility gap, as

Table 1
Chemical composition of PM 2000™ as determined by X-ray Fluorescence.

	Cr	Al	Ti	C	O	N	Y	Fe
wt.-%	18.60	5.20	0.54	0.04	0.09	0.006	0.391	balance
at.-%	18.50	10.10	0.58	0.17	0.28	0.022	0.228	balance

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