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Non-uniform phase separation in ferrite of a duplex stainless steel

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ABSTRACT

Phase separation in ferrite of a duplex stainless steel during aging at 400 °C for up to 20,000 h has been found to be non-uniform in nature by using atom probe tomography. Aging-induced segregation of Ni takes place strongly at the austenite/ferrite interfaces accompanied by the formation of Ni depletion zones (NDZs) in the ferrite in the vicinity of the interfaces. Consequently, the phase separation in the NDZs is microscopically and kinetically different from that in the inner ferrite. The nucleation and growth of G-phase in the NDZs are suppressed, and the growth of α'-phase is decelerated. In contrast, the G-phase at the austenite/ferrite interfaces exhibits a delayed heterogeneous nucleation and thereafter accelerated growth. The phase-separation difference between the NDZ and the inner ferrite results in a gradient microstructure (from outer to inner) developing in each ferrite grain during the prolonged aging. The strong segregation of Ni and the high decoration of G-phase at the ferrite/austenite interface are responsible for degradation in impact toughness of the steel.
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1. Introduction

Duplex stainless steels (DSS), with high strengths, excellent stress-corrosion resistance and good weldability, have a broad range of engineering applications, including in nuclear power, chemical processing, desalination and transportation industries [1]. The steels during long-term service at a temperature in the range from 280° to 320 °C suffer with thermal embrittlement due to phase separation of ferrite [2]. Consequently, their mechanical properties and performances including fracture toughness [3], stress corrosion resistance [4] and fatigue properties [5,6] deteriorate. A significant degradation of the DSSs during service will pose a great risk to the safe operation of nuclear power plants. As a result, fundamental understandings about the thermally–induced microstructural evolution of the steels have drawn huge research interests.

Thermally-induced microstructural changes of duplex stainless steels with high Cr contents mainly involve the formation of Cr-rich (α') phase and Fe-rich (α) phase [7–9], as well as G-phase in ferrite because of phase separation [1,10,11]. M23C6 precipitation on ferrite/austenite boundaries has been observed as well [12,13]. To understand the formation of Cr-rich (α') phase and Fe-rich (α) phase, binary Fe-Cr alloys have been extensively studied over the last 70 years [14–18]. The phase-separation behaviours of the Fe-Cr alloys are found to be highly dependent on the alloy's Cr content and aging temperature, with the α'-phase in the form of isolated particles or vein-like structures depending on the spinodal line of the Fe-Cr system [19–21].

Ni, as an important alloying element of DSSs, has complicated effects on the phase separation of ferrite. Firstly, Ni addition enhances phase separation kinetics of the Fe-Cr alloys. J.E. Brown [22] reported that the addition of Ni increased the rate of spinodal decomposition by studying a series of Fe-26% Cr-(0-3-5-8)% Ni alloys upon aging in the temperature range from 300 to 450 °C. A similar effect has been observed in DSSs [2]. Secondly, Ni is responsible for the G-phase formation in the ternary Fe-Cr-Ni system. G.T. Brown and R.T. Allsop [23], for the first time, observed G-phase particles in a Fe-12Cr-4Ni alloy thermally aged at 450 °C in 1960. Thereafter, the G-phase was widely reported in the DSSs. Si, Mn and Ni are reported to induce the G-phase precipitation in the DSSs [8,24,25]. The spinodal decomposition of ferrite has been found to enhance the G-phase precipitation, because the

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enrichment of the G-former elements at the α'/α inter-domains provides favourable conditions for the formation of the G-phase in a Mo-free DSS during aging at 350 °C [26]. Previous research has mostly focused on the microstructural change of the inner ferrite; this has greatly limited our understanding about phase separation in ferrite of the DSSs.

Ferrite/austenite interfaces in DSSs are known to be important for plastification of the neighbouring ferrite grains and crack initiation [27], and are of significance in determining the mechanical properties of the steels [28,29]. To date, little work has been done in addressing the phase separation near the ferrite/austenite phase boundaries of the DSSs. T. Hamaoka et al. [25,30] found that G-phase particles were small near the phase boundary in a DSS (designated as CF3M) aged at 400 °C for 5000 h and their number density increased with the distance from the phase boundary. There is a lack of information regarding the segregation of alloying elements and impurities as well as the precipitation at the ferrite/austenite interfaces of the DSSs during thermal aging. It is unclear if the phase separation across a ferrite grain is uniform, and how the ferrite/austenite interface affects the nucleation and growth of G-phase as well as the formation of Cr-rich (α′) phase and Fe-rich (α) phase near the interface. Comprehensive investigation of the ferrite/austenite interfaces is necessary for pinpointing the key microstructures or phases responsible for the mechanical properties and performance of the steels.

In this study, we systematically investigate the thermal decomposition of a duplex steel with an emphasis on addressing the segregation and precipitation at ferrite/austenite phase boundaries, and the non-uniformity of phase separation in a ferrite grain. To achieve this, a focused ion beam (FIB) has been employed to prepare atom-probe tip samples containing δ/γ interfaces, to overcome the technical difficulties in making such samples by electro-polishing. Atom probe tomography (APT) offers a high spatial resolution and a high chemical sensitivity, it is employed to quantitatively characterise the distribution, partition and segregation of alloying elements and impurities in the steel. This research aims to comprehensively address the phase-separation mechanisms of the ferrite grain in the steel. By systematically measuring the evolutions of size, number density and chemical compositions of precipitates and phases formed in the different regions of the ferrite grain, this research will reveal the phase-separation kinetics in the different regions of the ferrite grain, and identify key microstructural features important for thermal embrittlement of the steel.

2. Experiments

A duplex stainless steel (designated Z3CN20-09M) with a nominal chemical composition listed in Table 1 was used in this work. The composition of the steel is similar to that used in the primary circuit pipeline of a nuclear power plant. The typical microstructure of the cast steel quenched in water after a homogenization treatment at 1100 °C for 6.5 h is shown in Fig. 1, which consists of ferrite in a dark contrast and austenite in a light grey contrast. The area fraction of the ferrite was measured to be less than 20% using Ferrite Scope (Fischer Feritscope MP30). Thermal aging of the steel was carried out at 400 °C for up to 20,000 h.

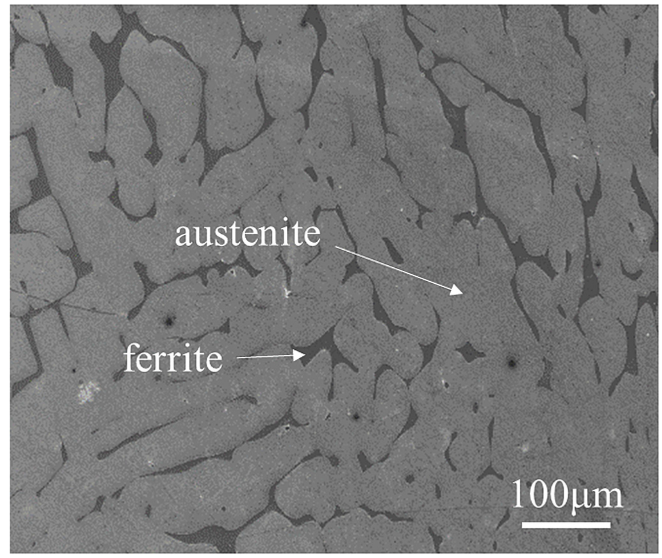

Fig. 1. A SEM micrograph of a duplex steel in as-cast condition.

Charpy V-notch impact tests at room temperature were conducted on the as-cast and thermally aged samples. Charpy specimens with a size of 10 mm × 10 mm x 55 mm with a 45° V-notch at the centre were machined according to the ISO standard 14556:2000 [31]. Since no thermal decomposition occurred in the austenite of the duplex stainless steel, nano-indentation tests were performed only on the large ferrite phases with an objective to avoid the influence of the underlying soft austenite and edge effects of grain/phase boundaries. Before nano-indentation, all finely-polished specimens were slightly etched to clearly reveal ferrite and austenite regions in the microstructures. The nano-indentation measurements were performed by using an MTS Nano-Indenter DCM tester with a 20 mN load. Each hardness value reported in this work was taken from the mean of at least five nano-hardness measurements from the ferrite of each specimen.

For atom probe tomography analysis, sharp APT tip samples containing ferrite/austenite interfaces were fabricated by FIB milling in a Zeiss Auriga FIB/SEM, with sample preparation procedures as summarized in Fig. 2. The region of interest containing a ferrite/austenite interface was firstly deposited with Pt to protect it from ion-bean damage during subsequent milling. Tilt and ion milling were secondly performed to cut a wedge bar out from specimen, as shown in Fig. 2a. The lift-out wedge bar specimen was thirdly cut into individual sections after each was mounted on a pre-sharpened tip or a micro-post of a Si micro-tip array coupon, as shown in Fig. 2b. Annular milling, as shown in Fig. 2c, was finally performed to produce a sharp tip with an apex radius of <50 nm, suitable for APT analysis. With this site-specific sample preparation method, tips containing an interface inclined to their shank axes were consistently made. APT experiments were performed by using a CAMECA local electrode atom probe LEAP™ 4000X Si at a specimen temperature of 20 K, under an ultrahigh vacuum of 2.5×10^{-9} Pa, a pulsing UV laser with a wavelength of 355 nm and a laser energy of 40 pJ, at a pulse repetition rate of 200 kHz, and an

Table 1
Nominal composition of the steel.

Alloy	Cr	Ni	Si	Mn	Cu	Mo	Co	C	N	S	P
wt. %	20.12	9.73	1.04	0.96	<0.10	0.14	0.098	0.033	0.044	0.0009	0.014
at. %	21.13	9.05	2.02	0.95	<0.09	0.08	0.091	0.15	0.17	0.0015	0.025

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