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Effects of Ni content on the microstructures, mechanical properties and thermal aging embrittlement behaviors of Fe–20Cr–*x*Ni alloys

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A B S T R A C T

The effects of Ni content on the microstructures, mechanical properties and thermal aging embrittlement behaviors of Fe–20Cr–*x*Ni alloys were investigated in the model alloys of Fe–20Cr, Fe–20Cr–5Ni and Fe–20Cr–10Ni, corresponding to ferritic stainless steel, duplex stainless steel and weld of austenitic stainless steel, respectively. Addition of Ni element will significantly change the solidification process and the microstructures of model alloys. Thermal aging at 400 °C for 3000 h leads to different degrees of hardening in ferrite of alloys, and a ductile–brittle transition in Fe–20Cr–5Ni alloy. It is well-known that Ni will accelerate the decomposition kinetics of ferrite. The thermodynamic calculation for Fe–Cr–Ni alloy indicates that addition of Ni will reduce the fraction of Cr-rich phase in the equilibrium phase diagram.

1. Introduction

Both Fe–Cr alloys and ferrite phases in Fe–Cr–Ni alloys or ferrite-containing stainless steels will suffer severe thermal aging embrittlement after long-term service at the 300–500 °C [1], and this phenomenon is well-known as "475 °C embrittlement" [2]. This embrittlement has been extensively investigated, and the generation of Cr-rich (α′ or α-Cr) and Cr-depleted (α or α-Fe) regions by spinodal decomposition or nucleation and growth in ferrite phases is considered as the primary mechanism [3–5]. The thermodynamics reason for this reaction is the miscibility gap in Fe–Cr system [6]. As the most important austenite-forming element, Ni is widely used in austenitic stainless steels and duplex stainless steels [7,8]. Nickel and chromium equivalents are widely used to predict the ratio of ferrite and austenite phases in welds and duplex stainless steels, such as Schaeffler diagram and Delong diagram [9,10]. Ni content will not only change the microstructures of Fe–Cr–Ni alloys, but also affects the thermal aging embrittlement behaviors in ferrite phases [11].

It has been pointed out that Ni element has an important influence on the spinodal decomposition thermodynamics and kinetics in both Fe–Cr alloys and ferrite phases. Hedström et al. [12] reported that addition of 2.9 wt% Ni to the binary Fe–20 wt% Cr significantly enhanced the kinetics of phase separation at 500 °C. Trindade and Vilar [13] investigated the embrittlement behaviors of Fe–Cr–Ni alloys containing 0.2 and 4 at% Ni using the Mössbauer effect and found that the addition of nickel to Fe–*x*Cr alloys ($x=25$ and 45 at%) increased the transformation rate notoriously in the first hours of aging. Miller and Russell [14] investigated the rate of decomposition in Fe–45 wt% Cr and Fe–45 wt% Cr–5 wt% Ni alloys during long-term aging at 400 °C. Their results showed that the addition of nickel to the binary Fe–45 wt% Cr alloy significantly increased the hardness of the alloy and also accelerated the kinetics of decomposition. Chung and Leax [15] studied a wide variety of laboratory aged CF3, CF8, and CF8M steels to investigate the effects of ferrite chemical composition on the kinetics of spinodal decomposition. They found that a higher Ni content in the ferrite matrix was expected to accelerate hardening by spinodal decomposition under otherwise identical conditions.

Three kinds of model alloys Fe–20 wt% Cr, Fe–20 wt% Cr–5 wt% Ni and Fe–20 wt% Cr–10 wt% Ni were investigated to study the effects of Ni content on the microstructures, mechanical properties and thermal aging embrittlement behaviors of Fe–Cr–*x*Ni alloys. Both of these model alloys contain a certain amount of ferrite phase, corresponding to ferritic stainless steel, duplex stainless steel and weld of austenitic stainless steel, respectively.

2. Experimental

Three model alloys were prepared by melting of pure Fe (99.9%), Cr (99.99%) and pure Ni (99.99%) with a vacuum induction

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Table 1
Chemical compositions of model alloys (wt%).

Alloys	Cr	Ni	Fe
Fe–20Cr	20.78 ± 0.36	–	79.21 ± 0.32
Fe–20Cr–5Ni	20.70 ± 0.17	4.60 ± 0.05	74.71 ± 0.18
Fe–20Cr–10Ni	20.87 ± 0.15	9.51 ± 0.13	69.62 ± 0.19

melting furnace (ZG-0.01). Alloys were remelted several times in order to obtain the homogenous composition. The ingots were homogenized at 1100 °C for 20 h and subsequently quenched in water. The chemical compositions of the model alloys were detected by an electron probe microanalyzer (EPMA, JEOL JXA 8100, Japan), and the results are listed in Table 1.

Microstructure of the model alloys was observed in a scanning electron microscope (SEM, Zeiss Supra 55, Germany). The compositions of ferrite and austenite phases were also detected by the previously mentioned EPMA. The aging treatments were performed at 400 °C for as long as 3000 h. Micro-mechanical properties in ferrite of both unaged and thermal aged alloys were studied by a nanoindenter (Nano Indenter DCM, MTS, USA) with an indentation depth of 500 nm using a Berkovich tip. An instrumented impact tester (NI 500, NCS, China) was used to evaluate the impact behaviors of aged and unaged model alloys. Full-sized V-notched Charpy specimens of dimensions $10 \times 10 \times 55$ mm^3, each with a 2 mm deep V-notch, were used in tests. Impact fracture characteristics were characterized by the previously mentioned SEM and electron back-scattered diffraction (EBSD). Thermodynamic calculation software, Java-based Material Properties (JmatPro, version 7.0), was used to estimate the influence of Ni content on the decomposition limit of Fe–Cr system.

3. Results and discussion

3.1. Microstructural characterization

Fig. 1 shows the SEM observation of the back-scattered electron images (BEI) for the Fe–20Cr, Fe–20Cr–5Ni and Fe–20Cr–10Ni alloys in the conditions of as-cast, homogenization treated and subsequently quenched. Fe–20Cr alloy shows a polycrystalline α-Fe phase, with coarse columnar grains (Fig. 1a). Addition of Ni significantly changes the solidification process and the microstructures of model alloys. For the Fe–20Cr–5Ni alloy, allotriomorphic austenite formes at the ferrite grain boundaries, and Widmanstätten austenite needles extends inside the ferrite grains, as shown in Fig. 1b. Fe–20Cr–10Ni alloy has the microstructure of austenite matrix with discontinuous vermicular ferrite.

A metallographic method, calculating the area fraction of each phase by Image-Pro Plus (version 6.0), was used to quantitatively measure the ferrite content, as shown in Table 2. Ferrite content declines significantly with increasing Ni content, from 55.3% of Fe–20Cr–5Ni alloy to 2.4% of Fe–20Cr–10Ni alloy. Fig. 2 shows the variation of phase fraction with Ni content for Fe–20Cr–*x*Ni ($0 \leq x \leq 10$) alloy, calculated using JMatPro (version 7.0) software. When $x < 2.72$ wt%, Fe–20Cr–*x*Ni alloy has the single phase of ferrite, and the single phase of austenite forms when $x > 9.65$ wt%. The ferrite contents of the three model alloys are also listed in Fig. 2, and the experimental results are consistent with the thermodynamic calculation.

The compositions of ferrite and austenite in Fe–20Cr–5Ni and Fe–20Cr–10Ni alloys were measured by EPMA, and the results are also shown in Table 2. Ferrit is enriched in Cr element while austenite has a higher Ni content. With increasing Ni content in the alloys, Cr and Ni contents in both of ferrite and austenite increase. The element distribution in ferrite certainly will influence

Table 2
Ferrite content and phase compositions of model alloys.

Alloys	Ferrite content (%)	Ferrite		Austenite	
		Cr	Ni	Cr	Ni
Fe–20Cr	100.0	20.78 ± 0.36	–	–	–
Fe–20Cr–5Ni	55.3	22.78 ± 0.17	3.53 ± 0.05	18.16 ± 0.17	5.93 ± 0.05
Fe–20Cr–10Ni	2.4	27.90 ± 0.17	5.16 ± 0.15	20.70 ± 0.15	9.62 ± 0.13

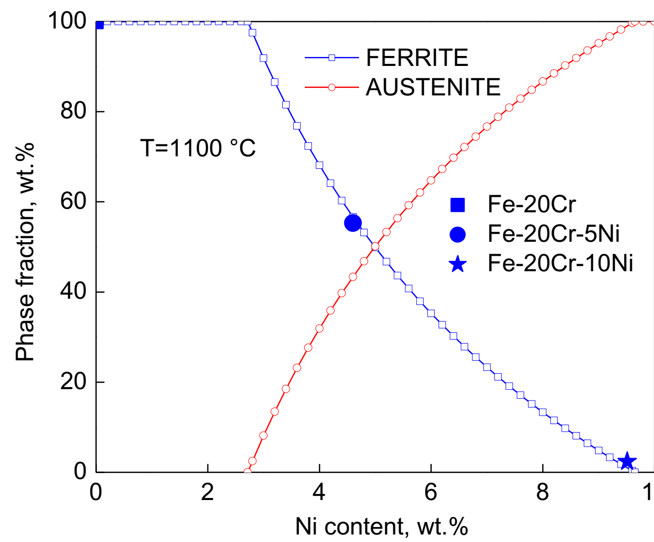

Fig. 2. Variation of phase fraction with Ni content for Fe–20Cr–*x*Ni alloy, calculated using JMatPro (version 7.0) software.

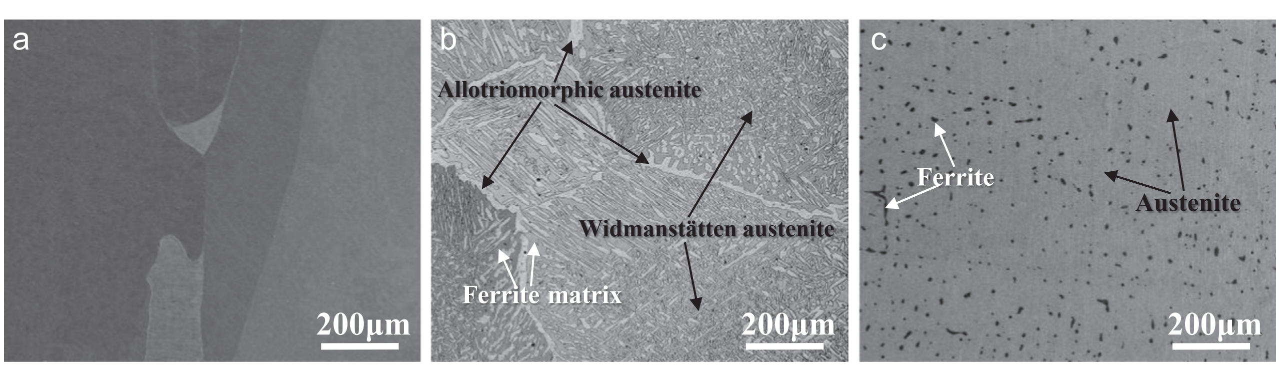

Fig. 1. Microstructure (SEM-BEI) of the model alloys (a) Fe–20Cr, (b) Fe–20Cr–5Ni and (c) Fe–20Cr–10Ni.