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The Kinetic diagram of sigma phase and its precipitation hardening effect on 15Cr-2Ni duplex stainless steel

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ABSTRACT

The kinetics of sigma phase precipitation at the temperature range of 750–950 °C in a rapidly solidified 15Cr-2Ni-2Al-11Mn resource-saving duplex stainless steel was investigated. After fitting the experimental results with the Avrami equation, the TTT diagram of sigma phase was established. It is found that the precipitation rate of sigma phase maximizes at about 850 °C and that the precipitation hardening effect sharply peaks at about 1.5 vol% sigma phase content, which is obtained by aging at 850 °C for 180 min. With the further increase of sigma phase content from 1.5 vol%, the strength reduces and the ductility increases again.

1. Introduction

Duplex stainless steel (DSS) is composed of both ferrite (δ) and austenite (γ) phases, and exhibits an excellent combination of mechanical property and corrosion resistance [1,2], which is thus increasingly used in stringent environment [3–7]. DSS can be precipitation hardened by sigma (σ) phase at the expense of plasticity and toughness [8]. Several precipitation reactions can occur during the aging temperature of 600–1000 °C, leading to the formation of intermetallic compounds such as σ, χ, $M_{23}C_6$, Cr_2N, etc. [9], among which σ phase deteriorates most on the toughness as well as the corrosion resistance. The hardness of σ phase is high to about 17 GPa at the peak load of 500 μN by using nanoindentation [10], and it is a non-magnetic intermetallic phase consisted mainly with iron and chromium. The diffusion velocity of the σ phase forming elements in δ phase is faster than that in γ phase, and the precipitation rate of the σ phase in δ phase is nearly about 100 times as that of γ phase [11]. Growing proper size and amount of σ phase through aging is mostly used to develop precipitation hardening in DSS. It relies on the temperature-dependent solid solubility to produce fine particles of intermetallic compound, which impedes the movement of dislocations or other defects in the crystal lattice. The σ phase is formed in DSS during annealing at 550–950 °C, so the application of DSS is usually limited to temperature not exceeding 500 °C. The hardening effect of σ phase precipitate on the mechanical property of stainless steel depends on the factors such as the size, morphology, volume fraction and distribution of σ phase in the matrix [10].

The kinetics of σ phase precipitation were subjected to extensive investigations. Kim et al. [12] studied the precipitation behavior of σ phase in cast DSS CD3MN and CD3MWCuN, and established the time-temperature-transformation (TTT) diagram of σ phase. The effects of plastic deformation [13–16] and alloying elements [17–20] were investigated to reveal the basic mechanism of σ phase precipitation in stainless steel. It was found that both the decreased grain size [21] and high crystallographic misorientation between the γ and δ phases [22] promote σ phase precipitation. The effect of σ phase precipitation on the mechanical performance of stainless steel were also investigated and well in progress. Some early reports showed that creep strength is reduced by the presence of σ phase [23–25], and the dendrite of σ phase leads to embrittlement of stainless steel [26]. However, recent investigations indicated that σ phase can also be beneficial for mechanical property. Plastic deformation can be used to refine the σ phase particles into stable globular morphology. The fine and homogeneous dispersion of σ phase can play a role of precipitation strengthening in DSS alloys [27,28], and it improves the creep strength [29] and plasticity [30] of DSS alloys. Shek et al. [31,32] found that the strength and ductility of 25Cr-8Ni DSS can be enhanced when the distribution and morphology of σ phase are properly controlled through appropriate heat treatment and that the hot tensile strength of aged samples is higher than those of unaged ones since the ($\sigma + \gamma_2$) structure behaves like a reinforcing phase. Pohl et al. [33] reported that σ phase precipitation of 1 vol% leads to two thirds reduction of impact toughness and that the strengthening effect of σ phase peaks at about 18 vol% without considering the variation of γ phase content.

Understanding the impact of σ phase precipitation on the mechanical performance is crucial to the development of high performance DSS. This work establishes the TTT diagram of σ phase after the non-equilibrium kinetics diagram of γ phase was established in previous

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Table 1
The specific chemical compositions of 15Cr-2Ni DSS (wt%).

Element	Fe	Cr	Al	Ni	Mn	C
wt%	Balance	15.27	1.96	2.04	11.05	0.02

work [34], and then the quantitative relation between σ phase and the mechanical property of DSS is sought.

2. Experimental

The raw materials were melted in an electric arc furnace and then fast solidified into a copper mould at room temperature, which resulted in about 70% δ phase in the as-cast alloy. The specific chemical composition of the casting alloy was obtained through spectroscopic analysis and shown in Table 1. Cold-rolled (70% thickness reduction) samples were then annealed at 750 ~ 950 °C for different time followed by water quenching, respectively. The centre region of the DSS samples was examined in order to avoid any surface effect. Samples were electrolytically etched in 15 wt% KOH solution, which makes the γ bright, δ gray and σ reddish brown under an optical microscope. The optical images from ten randomly selected areas of the etched surface were taken using a 500x objective lens in order to analyze the volume fraction of σ phase using the metallography image analysis software. Selected sample was subjected to investigation using transmission electron microscope (TEM, JEOL 2000FX working at 200 kV) to identify σ phase. The sample was thinned into 30 μm by sand paper, punched into wafers of 3 mm diameter and ion milled to get the TEM foils. For tensile test, all samples were wire-cut into dog-bone shape with the gauge dimension of 5 × 30 × 1 mm, to which a 25-mm extensometer can be attached for an accurate measurement of the elongation. The fracture surface of samples after tensile test were observed using a Jeol 6490 scanning electron microscope (SEM).

Corrosion behavior of the aged DSS samples was investigated based on potentiodynamic polarization curves. Polarization test was conducted using potentiodynamic polarization electrochemical methods and evaluated in the solution of 3.5 wt% NaCl at 25 °C. The experiments were carried out in a 200 ml conventional three-electrode cell comprising the sample as the working electrode, a Pt foil as the auxiliary electrode and a saturated calomel electrode as the reference one. Prior to testing, samples were mechanically polished from 240 to 1200 grit abrasive paper, washed with distilled water, degreased with acetone and dried in air. The interface between sample and resin was coated with a polyacrylate quick-setting resin to avoid the possibility of crevice corrosion during measurement. Polarization test of each sample was performed at least three times using a Potentiostat/Galvanostat (EG&G Princeton Applied Research, Model 273A).

3. Results

3.1. The microstructure of σ phase

Fig. 1(a) (c) (d) show the DSS samples aging at 750 °C for 360 min and 900 °C for 45 min and 90 min followed by water quenching, respectively. The matrix of samples shows an obvious duplex structure of δ and γ phase, and σ phase was found in γ phase. No σ phase was found in the sample aging at 950 °C. Fig. 1(b) shows the SEM image of the DSS sample aging at 750 °C for 1050 min followed by water quenching and the chemical composition of σ, δ and γ phases obtained using the energy dispersive X-ray spectroscopy (EDS) is listed in Table 2. It is noted that

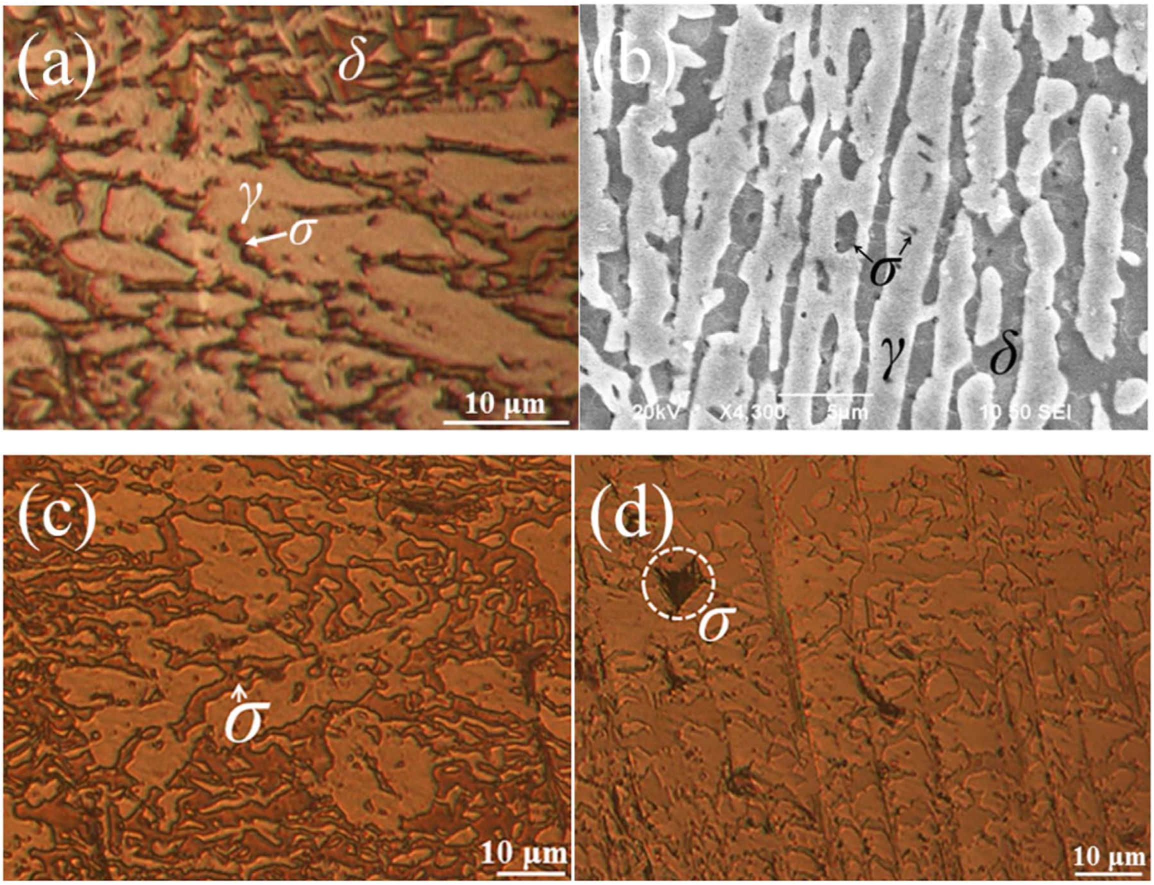

Fig. 1. DSS samples aging at 750 °C for (a) 360 min and 900 °C for (c) 45 min (d) 90 min followed by water quenching, respectively. (b) Scanning electron micrograph on the DSS sample aging at 750 °C for 1050 min followed by water quenching.