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Coarsening of carbides during different heat treatment conditions

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ABSTRACT

Coarsening of carbides in 1# Fe–5.96Cr–0.35C (wt.%) alloy and 2# Fe–0.5V–0.53C (wt.%) alloy during different heat treatment conditions was investigated by carbon replica, high-resolution transmission electron microscopy (HRTEM) , X-ray diffraction (XRD) and SEM techniques. The equilibrium phases at 850 °C constitute of austenitic matrix (γ) + M_7C_3 and austenite matrix (γ) + V_4C_3 for 1# and 2# alloy respectively. Morphology of M_7C_3 and V_4C_3 carbides was mainly determined by cooling mode due to the different nucleation sites and growth mechanisms. Under directly aging condition, most carbides nucleate in the grain boundaries and grow into rod-shaped or flake-shaped particles by discontinuous growth mechanism. These particles turn out to be excluded during coarsening simulation using Oswald ripening model to give a more reasonable result. In addition, interfacial energy between M_7C_3/γ and V_4C_3/γ for the coarsening of M_7C_3 and V_4C_3 during aging at 850 °C is evaluated by fitting experimental data using thermodynamic and kinetic calculations. The interfacial energy is determined to be 0.7 J/m^2 for the coarsening of M_7C_3 and V_4C_3 in austenitic matrix.

1. Introduction

The volume fraction, size, morphology and distribution of precipitates have strong effects on the mechanical properties of steel materials [1]. Studies have shown that precipitation strengthening effect of small carbides can significantly improve the mechanical properties of steel [2–4]. In recent years, coarsening of carbides are widely studied experimentally and through thermodynamic and kinetic calculations [5–9]. The precipitation behavior of carbides after different aging time was investigated by using carbon replica and TEM technique which greatly improved the accuracy of size measurement [10,11]. Many works [12–14] attempt to predict the coarsening of Vanadium and Chromium carbides after water quenching and aging by thermodynamic and kinetic calculations. However, little work focusing on the influences of morphology of carbides under different heat treatment conditions on the coarsening behavior has been reported.

In the present work, the coarsening behavior of M_7C_3 and V_4C_3 carbides was studied in Fe–Cr–C and Fe–V–C ternary alloys. Experiments were conducted using carbon replica, HRTEM and XRD techniques Thermodynamic and kinetic calculations were also conducted using Thermo-Calc and DICTRA software. With the combination of experimental study and calculations, the coarsening behavior of M_7C_3 and V_4C_3 carbides during different heat treatment conditions can be investigated.

2. Materials and experimental procedures

Raw materials of 1# and 2# alloy were electrolytic iron (99.99%), pure chromium (99.999%), pure vanadium (99.9%) and white cast iron (Fe–4.5%C). They were induction-melted into buttons and remelted several times under an argon atmosphere in order to increase homogeneity. The button was encapsulated in evacuated quartz encapsulation tubes and homogenized at 1100 °C for 5 days. The chemical composition of 1# and 2# alloy was listed in Table 1. Specimens with dimension of 8 × 8 × 8 mm were cut and encapsulated. Heat treatment processes illustrated in Fig. 1 were designed to study the coarsening behavior of M_7C_3 and V_4C_3 in austenite according to the phase diagrams calculated by Thermo-Calc with TCFE6 database, as shown in Fig. 2.

Carbide particles were chemically extracted in phosphoric acid (2:1) [15] at room temperature and filtered using a micro-porous membrane with 0.20 nm aperture and dried. A D/MAX-2500 X-ray diffractometer operating at 40 kV and 40 mA with scanning speed of 4°/min was used to determine the type of precipitates.

Size, morphology and distribution of precipitates were studied by carbon replica extraction and TEM technique. Heat-treated specimen was ground, polished and then etched in 2 vol. % nital. The carbon film was deposited on the etched surface and scored into 2.5 × 2.5 mm square grids before etching again in nital. Concentration of nital and etching time for this step was listed in Table 2. Finally, the specimen was slid into distilled water and the replicas were collected by a copper net and dried.

TEM observation was carried out using a JEM-2010F instrument and operating at 200 kV accelerating voltage. Precipitates were identified by a combination of electron diffraction patterns and energy dispersive spectrometer (EDS) analysis.

In addition, the precipitation position and morphology of carbides particles in the samples were also observed with SEM&EDS analysis.

3. Experimental results

X-ray diffraction patterns of carbides after aging in 1# and 2# alloy were shown in Fig. 3. The type of carbides in 1# and 2# alloy

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Table 1
Chemical composition of 1# and 2# alloy (wt.%).

Alloy	Cr	V	C
1#	5.96	–	0.35
2#	–	0.5	0.53

Table 2
Concentration of nital and etching time of 1# and 2# alloy.

Alloy	Concentration/%	Etching time/min
1#	8	8
2#	10	2

was determined to be M_7C_3 and V_4C_3 respectively after aging at 850 °C. No other types of precipitates were detected after different aging time.

Figs. 4 and 5 showed the TEM micrographs with electron diffraction pattern and EDS analysis of carbides in 1# alloy obtained under different heat treatment conditions. The carbides regardless of their shape of rods or spheres were identified to be M_7C_3 precipitates.

Figs. 6 and 7 showed the TEM micrographs with electron diffraction pattern and EDS analysis of carbides in 2# alloy obtained under different heat treatment conditions. The carbides regardless of their shape of rods or spheres were identified to be V_4C_3 precipitates.

As shown in Fig. 8, morphology of carbides precipitate in grain boundaries were rod-shaped or flake-shaped and which in grain are mostly spherical or nearly spherical.

4. Discussion and simulation

4.1. Determination of particle size distribution

In the present work, Qin's [16] method is adopted by drawing the circumference of each precipitate by hand and automatically determining the corresponding diameter of a circle of equal area with Image Analysis System (Image J) . For each aging time

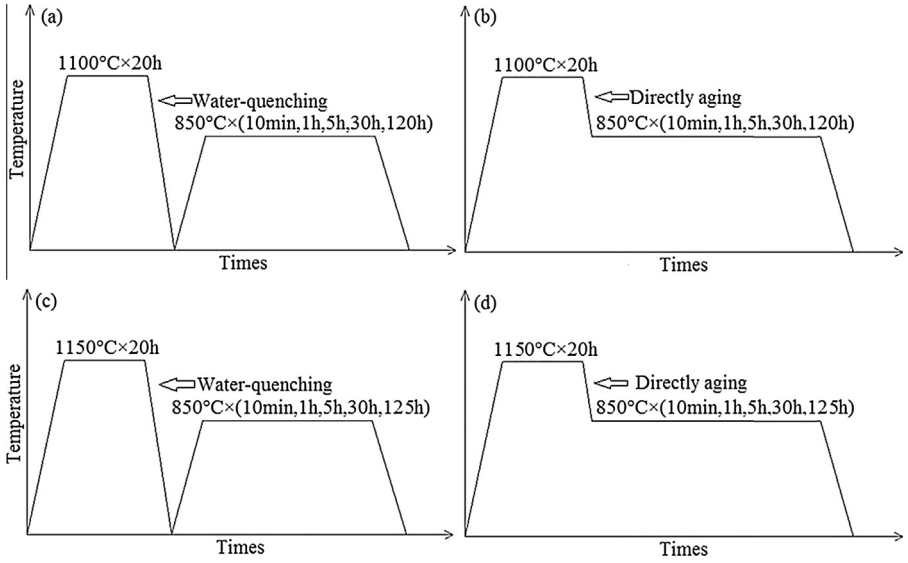

Fig. 1. Schematic diagram of heat treatment processes of 1# and 2# alloy.

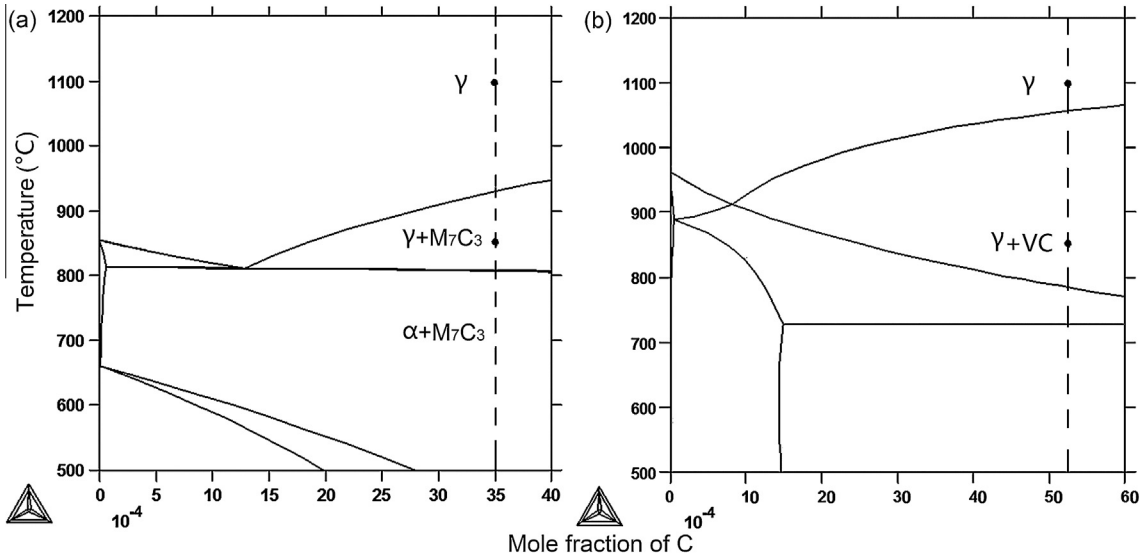

Fig. 2. Calculated phase diagram of (a) Fe–5.96Cr–0.35C (wt.%) and (b) Fe–0.5V–0.53C (wt.%) with C content and heat treatment temperature marked by filled square.