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Isothermal precipitation kinetics of carbides in undercooled austenite and ferrite of a titanium microalloyed steel

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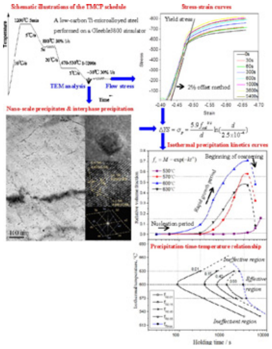
HIGHLIGHTS

- A new method for isothermal precipitation kinetics of carbides in ferrite is first proposed.
- Precipitation-time-temperature diagrams of carbides in ferrite are determined efficiently.
- A modified-form of Avrami formula is developed to describe the precipitation kinetic curves.
- Isothermal precipitation occurs preferentially at sub-boundaries and dislocations, then γ/α interphase and point defects.
- Strength increment is mainly attributed to interphase precipitation for a low-carbon titanium microalloyed steel.

GRAPHICAL ABSTRACT



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ABSTRACT

A new method for isothermal precipitation kinetics of carbides in undercooled austenite and ferrite has been established based on measuring the strength increments. Precipitation-time-temperature (PTT) diagrams of carbides were determined with this method performed on a Gleeble simulator, for a low-carbon titanium microalloyed steel in a temperature range from 530 °C to 670 °C. Fine precipitates were studied by using high resolution transmission electron microscope (HRTEM), and their volume fractions together with their attributions to strength increments were calculated and analyzed. Results show that the strength increments are mainly attributed to the γ/α interphase precipitation whose nano-scale TiC precipitates obey the Baker-Nutting orientation relationship with respect to the ferrite matrix: $(100)_{TiC} \parallel (100)_{\alpha\text{-Fe}}$ and $[011]_{TiC} \parallel [001]_{\alpha\text{-Fe}}$. The PTT curves obtained have a classic C-shape with their noses at about 600 °C, where the strength increment reaches 114 MPa after isothermally holding for 1 h. Additionally, the corresponding precipitation kinetics curves are presented in an S-shape, and a modified-form of Avrami formula is introduced to describe their growth periods. The presented mathematical expression fits the experimental data quite well. Consequently, the proposed new method is intuitive, effective and convenient, particularly suitable for detecting the precipitation kinetics in low temperature range.

1. Introduction

High-strength low-alloy (HSLA) steels are always alloyed with strong carbide-forming elements, such as Nb, V, Ti, and usually

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produced by thermo-mechanical controlled processing (TMCP) schedule, which intends to achieve a high-strength mainly by the combination of grain refinement and precipitation hardening [1–3]. Of particular note, only the α/γ interface precipitation and random homogeneous precipitation in the supersaturated ferrite matrix can bring about an excellent precipitation hardening effect [4,5]. It is therefore significant to investigate the precipitation characteristics and precipitation kinetics of the carbides in such steels. Nowadays, the common methods used to study the progress of precipitation include: (i) the chemical and electrolytic extraction [6], (ii) transmission electron microscopy (TEM) analysis [7–10], (iii) electrical resistivity measurements [11], (iv) hot compression testing [12–15], together with (v) stress relaxation method [16,17].

Chemical and electrolytic extraction can uniquely provide complete information, including particle size distribution and structure of precipitates. Unfortunately, it is difficult to examine the ultra-fine and unstable precipitates especially formed in the early stage of precipitation. On the contrary, TEM analysis is indispensable and useful for the direct observation of the fine precipitates, as well as determining their relationships with respect to the matrix, dislocations, and boundaries. However, the use of TEM analysis alone as a method for precipitation kinetics is restricted by the lengthy experimental times and limited observable area. Therefore, the two methods above are usually used as aided tools for precipitation kinetics study in steels.

Electrical resistivity measurements are sometimes used for the analysis of both dissolution and precipitation kinetics of precipitates. It was reported that this method was successfully applied to quantitatively analysis of isothermal precipitation kinetics of carbides over the temperature range from 850 to 1050 °C [18,19]. Interrupted hot compression tests are commonly applied to detect the isothermal precipitation at high temperature by measuring the flow curves deformed on the austenite. However, both of them are generally used for monitoring the progress of strain-induced precipitation, and conversely their applications to analysis of precipitation in ferrite are sparsely reported in literature.

Stress relaxation method has been successfully applied to determine the precipitation kinetics for low alloy steels at elevated temperatures. The precipitation kinetic of carbides for a 0.18 wt.% Ti steel over the temperature varied from 850 to 1050 °C was investigated by Liu and Jonas [20]; It was shown that stress relaxation method was so efficient that the starting and finishing times of precipitation could be detected in a single test. Nowadays, stress relaxation experiments are commonly carried out on Gleeble simulators. However, it is sometimes difficult to fix the start and finish times of precipitation on stress curves obtained by this method, mainly due to the involvement of stress fluctuation. Thus, there is a demand for better techniques to determine the PTT relationships, particularly at low temperatures below 800 °C.

In this work, a new approach to study the isothermal precipitation kinetics of carbides in undercooled austenite and ferrite is first proposed, which is based on measuring the compressive strength increment of samples held isothermally for various times at low temperatures below 800 °C. With this method and the aid of transmission electron microscopy (TEM), the influences of isothermal temperature and holding time on precipitation behaviors together with strength increment are investigated, and thereby the PTT relationships are easily obtained. Furthermore, the correlations between precipitation characteristics and softening behavior are elucidated in detail.

Table 1
Chemical composition of the tested steel used in this study (units: wt.%).

C	Si	Mn	S	P	Al	Ti	N	O	Fe
0.051	0.27	0.96	0.0039	0.0103	0.030	0.104	0.0026	0.0048	Balance

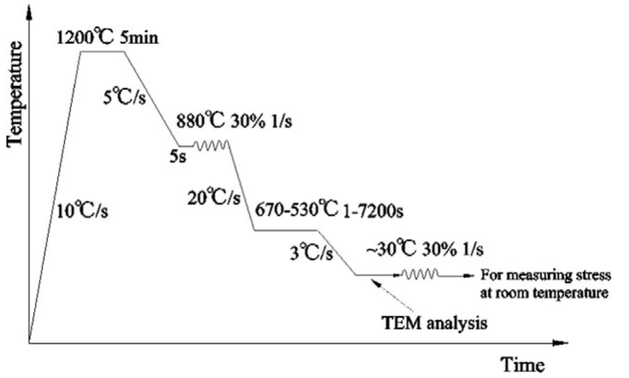

Fig. 1. Schematic illustrations describing the TMCP schedule carried out on a Gleeble simulator.

2. Material and test experiments

The tested low carbon steel was alloyed with 0.1 wt.% Ti for the investigation of the TiC precipitation in undercooled austenite and ferrite. Its detailed chemical composition is given in Table 1.

The tested steel was refined in a vacuum induction furnace, and cast into a square ingot of 200 × 150 × 150 mm^3. Subsequently, the ingot was heated at 1000 °C for 45 min, then forged into rod with 70 mm square cross section, and cooled in air to eliminate the cast texture in the as-cast ingot. After homogenizing, the material was sectioned and machined to small-scaled cylinders (ϕ10 mm × 15 mm) for thermal simulations. The simulated experiments were conducted on a Gleeble 3800® Thermo-Mechanical Simulator and the schematic illustrations of the TMCP schedule are shown in Fig. 1.

Based on the solubility product proposed by Taylor [21], all specimens were heated up to 1200 °C and austenitized for 5 min, in order to ensure the full dissolution of Ti elements in austenite and avoid the overgrowth of austenite grain size. After austenitization, specimens were slowly cooled down to 880 °C and soaked for 5 s, then deformed continuously to a strain of 0.42 at a strain rate of 1/s. After hot deformation, the specimens were rapidly cooled down to different test temperatures of 670, 630, 600, 570 and 530 °C, and held for different durations varied from 0 to 5400 s. After isothermal precipitation, specimens were rapidly cooled down to 530 °C at an average cooling rate of 20 °C/s, in order to avoid precipitating during cooling, then followed by air-cooling at ~3 °C/s to room temperature. The air-cooled specimens were deformed again to a true strain of 0.42 at a strain rate of 1/s at room temperature thus to obtain the stress-strain curves and determine the yield stress.

In order to evaluate the feasibility and accuracy of the new approach, TEM analysis was also used as contradistinctive approach to study the kinetics of precipitation. Thin foil samples were sectioned transversely from air-cooled specimens obtained from the thermal simulation tests. Precipitates as well as dislocation substructures were observed by using FEI Tecnai G2 F20 Transmission Electron Microscope.

3. Results and discussion

3.1. Change in yield stress vs. time and temperature

According to the data obtained from the Gleeble acquisition system, the true stress-strain curves during the second-stage deformation of specimens were plotted in Fig. 2. There are five groups of stress-strain curves corresponding to five different isothermal temperatures of 670, 630, 600, 570 and 530 °C for different holding time. It can be seen that the true stress-strain curves were in accordance with typical ones while their yield stresses and supreme stresses were different from