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Strain-induced Precipitation in Ti Micro-alloyed Interstitial-free Steel

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Abstract: Stress relaxation method was carried out on a Ti micro-alloyed interstitial-free (IF) steel at the temperature ranging from 800 to 1000 ℃. The results show that the softening kinetics curves of deformed austenite can be divided into three stages. At the first stage, the stress has a sharp drop due to the onset of recrystallization. At the second stage, a plateau appears on the relaxation curves indicating the start and finish of strain-induced precipitation. At the third stage, the stress curves begin to descend again because of coarsening of precipitates. Precipitation-time-temperature (PTT) diagram exhibited a "C" shape, and the nose point of the PTT diagram is located at 900 ℃ and the start precipitation time of 10 s. The theoretical calculation shows that the strain-induced precipitates were confirmed as almost pure TiC particles. The TiC precipitates were heterogeneously distributed in either a chain-like or cell-like manner observed by transmission electron microscopy (TEM), which indicates the precipitates nucleated on dislocations or dislocation substructures. In addition, a thermodynamic analytical model was presented to describe the precipitation in Ti micro-alloyed IF steel, which shows a good agreement between the experimental observation and the predictions of the model.

Key words: Ti micro-alloyed IF steel; stress relaxation; strain-induced precipitation; analytical model

The development of interstitial-free (IF) steels has been a milestone for achieving high drawability of sheet steels and it is being increasingly considered as material for the automotive industry[1]. It is well known that the reduction of the carbon content results in higher levels of deep drawability[2-4]. Recent advances in the steel making technologies, such as improved vacuum degassing, have helped to create a new generation of steels with exceptionally low levels of solute carbon and nitrogen contents (less than 0.003 mass%)[5,6]. Titanium and/or niobium are added in these steels to bind the remaining solute carbon or nitrogen in the form of carbides, nitrides or carbonitrides rendering them virtually free from solute atoms. Hence, these steels are also known as interstitial-free steels[1,7]. As known, the good deep drawing properties in interstitial-free steels are attributed to the presence of strong γ-fibre ({111} or (ND∥<111>)) recrystallization texture, while {001}<*uvw*> and α-fibre (RD∥<110>) are considered harmful for deep drawability[7-15]. During the last two decades, most of the investigations for the interstitial-free steels have been laid on the manufacturing route and seeking correlation between structures (related to grain size, texture and anisotropy ratio, etc.) and properties (related to strength, formability and drawability, etc.) for varied material chemistry and thermo-mechanical treatment[16-18]. The recent researches showed that the uniform and fine grains in hot-rolled sheet steel, coarsening and sparse second phase particles and low levels of solute carbon and nitrogen contents were beneficial to the development of γ-fibre ({111} or (ND∥<111>)) recrystallization texture, which was beneficial to the deep drawability of the annealed board. The grain size, morphology and distribution of second phase and low level contents of interstitial elements are closely associated with the precipitation of second phase particles during hot rolling, which are hardly changed in the cold rolling process. It is well established that the precipitation of carbides or nitrides or carbonitrides in micro-alloyed steels containing one or more of the transition metals, such as Nb, V and Ti, plays a critical role in determining the properties of high-strength low-alloy (HSLA) steels[19-22]. And most of investigations on the precipitation behavior have been concerned about the HSLA steels, while the precipitation behavior of IF steels was rarely repor-

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ted. Thus, the further study on the precipitation behavior of second phase in IF steel during hot rolling has important theoretical and practical significance.

In order to understand the precipitation behavior of IF steel clearly, a mechanical method was carried out in this study, which is based on the analysis of stress relaxation data. The hot rolling process for steel production, generally, is conducted under a quite severe deformation condition. So the thermal simulation compression test in a Gleeble simulator is applied ideally to simulate the hot rolling process. Stress relaxation measurements have been extensively used for studying dislocation movement in metal and alloys[22]; the start and finish time of TiCN precipitation were obtained by analyzing the stress relaxation curve, which usually exhibits a stress plateau when the precipitation occurs, and thus the precipitation-time-temperature (PTT) diagram for TiCN precipitation kinetics could be drawn from the stress *vs*. time curves at different temperatures[19,20].

1 Experimental Procedure

The chemical composition of the Ti micro-alloyed IF steel examined in this study is given in Table 1. The specimen was prepared by vacuum induction melting, continuously cast to slab, and homogenized at 1 250 ℃ for 2 h, and then hot rolled to 34 mm thick intermediate slab. And the cylindrical specimens (ϕ10 mm×12 mm) were then machined from the intermediate slab with the compression axis parallel to the rolling direction.

Table 1 Chemical composition of Ti micro-alloyed IF steel mass%

C	Si	Mn	P	S	Al_t	Ti	N
0.002	0.004	0.11	0.011	0.008	0.03	0.06−0.07	0.000 02−0.000 03

The mechanical stress relaxation tests were carried out in a computer controlled Gleeble 3500 thermo-mechanical simulator. Prior to the deformation, all the samples were reheated to 1 250 ℃ and soaked for 3 min to dissolve the carbonitride particles, eliminate the rolling texture and produce approximately the same austenite grain size in all the samples. The specimens were cooled down to the deformation temperatures at 10 ℃/s and held for 5 s at that temperature before applying the compressive deformation. The deformation temperatures were chosen as 800, 850, 900, 950 and 1 000 ℃. When the temperature became stabilized, a 20% pre-strain was applied at a true strain rate of 1 s^{-1}. Then different time (1, 10, 60, 360, 600, 1 200 and 1 800 s) of stress relaxation was preceded immediately following the pre-strain. After the stress relaxation, the samples were quenched to room temperature. The schematic diagram of the experiment is shown in Fig. 1.

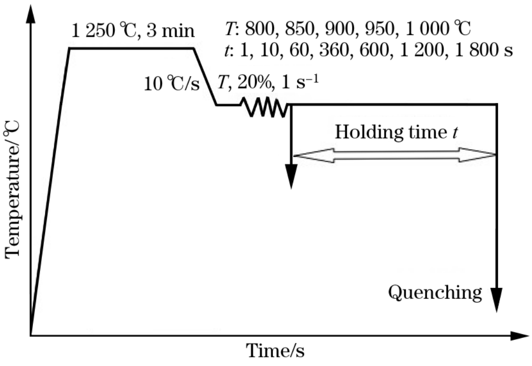

Fig. 1 Schematic diagram of the experiment

The size, type, morphology and distribution of the second phase precipitation particles were determined with the aid of extraction replicas. The quenched samples were sectioned parallel to the deformation axis, polished, and lightly etched in 4% nital for about 60 s. A carbon layer of 20 nm in thickness was deposited on the surface of the etched samples using an evaporator (Polaron E6500) operated at a high vacuum. The carbon coated surfaces of the samples were then scribed to about 2 mm×2 mm squares in size. The replicas were released in the etched solution, and then observed in a transmission electron microscope (TEM-JEM 2010F). The chemical analysis for the individual precipitates was conducted using an energy dispersive X-ray spectrometer (EDX).

2 Results

2.1 A_{r3} temperature of the Ti micro-alloyed steel

In order to ensure that the tests were carried out in austenite phase or ferrite-austenite phase region, A_{r3} (the start point of austenite to ferrite phase transformation) temperatures were determined at a cooling rate of 1 ℃/s after the sample was reheated to 1 200 ℃ and soaked for 3 min. The dilatometer *vs*. temperature curves for the sample is presented in Fig. 2. As known, the volume changed when the phase