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## Nanoscale cementite and microalloyed carbide strengthened Ti bearing low carbon steel plates in the context of newly developed ultrafast cooling



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#### ABSTRACT

We describe here the microstructural evolution, mechanical properties and comprehensive strengthening mechanism of Ti-bearing high strength steels with different finish cooling temperatures in the context of newly developed ultrafast cooling system. Pilot-scale studies demonstrated that high yield strength of ~650 MPa can be obtained with ultrafast cooling finish temperature of 580 °C after hot rolling. The underlying reason is that the microalloyed carbides and nanoscale cementites can precipitate simultaneously to improve the precipitation strengthening to a large extent. In order to estimate the precipitation strengthening, the volume fraction of the precipitates in different size ranges was obtained by chemical phase analysis, small-angle X-ray and neutron scattering. The results indicated that Fe<sub>3</sub>C, with a higher volume fraction, had a stronger precipitation strengthening effect than nanoscale TiC. The precipitation strengthening contribution of nanoscale precipitates can achieve 350 MPa. Together with solid solution strengthening and grain refinement strengthening, the theoretical calculated values match well with the experimental values.

#### 1. Introduction

Low carbon microalloyed steel plates are now widely used in buildings, bridges, ships, vessels, industrial equipment and storage tanks. Excellent mechanical properties with high strength and high ductility can be obtained by optimizing the new generation thermomechanical controlled process (NG-TMCP), which refers to the ultrafast cooling (UFC) combined with thermomechanical controlled processing [1–[4\]](#page--1-0). NG-TMCP has been currently applied in industrial production, aiming at reducing the consumption of alloying elements and making the steel manufacturing process economically viable. The superior mechanical properties are a consequence of grain refinement together with microstructural control and precipitation hardening. The strong carbide-forming elements titanium can not only facilitate grain refinement but also contribute to dispersion hardening through carbide precipitation in the matrix. In addition, UFC can greatly increase the precipitation hardening since it can suppress the amount of precipitates in austenite during the relatively longer rolling time and increase the supersaturation ratio in ferrite.

Precipitation strengthening has triggered extensive interests since the development of Mo-Ti-bearing steel with a strength of 780 MPa and more recently 1200 MPa through controlled precipitation strengthening. The level of the yield strength increment from precipitation strengthening was almost twice of that obtained by conventional HSLA steels [\[5,6\]](#page--1-1). However, steel industries continue to face the challenge of reducing the alloy costs. In order to meet the requirements of cost reduction and maintaining high strength, nanoscale cementite strengthening is regarded as a viable option to reduce the addition of microalloying elements because it is a common secondary phase in steels [7–[9\].](#page--1-2) Wang and his co-workers [\[7,10,11\]](#page--1-2) investigated the nanoscale cementites in different carbon content steels without alloy addition by controlling the cooling profile after hot rolling. The results indicate that a low UFC finish temperature (600 °C) and 0.17 wt% carbon content are most favorable for nanoscale cementite precipitation. The strengthening effect of nanoscale cementite precipitates can achieve  $\sim$ 158 MPa. Fu [\[12\]](#page--1-3) proposed that if we can combine micro-

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alloyed carbide and cementite strengthening together, then the strength will be improved to a large extent. They observed nanoscale cementite and microalloyed carbides precipitates simultaneously in 0.06C-0.10Si-0.08Ti-microalloyed high strength weathering steels during thin slab continuous casting and rolling. The nanoscale cementites and microalloyed carbides are both believed to play an important role in enhancing the strength of the steel. The yield strength calculation formula for low carbon steel was also proposed, where the yield strength of steel equals the sum of solid solution strengthening, grain refinement strengthening, and precipitation strengthening [\[12\]](#page--1-3). However, little work has been carried out in investigating UFC finish temperature to control nanoscale cementites and microalloyed carbides precipitated simultaneously in low-carbon steels plates during NG-TMCP process.

In this work, we investigated the effect of UFC finish temperature after hot rolling on the properties of a Ti-bearing low carbon steel plates. Chemical phase analysis, small-angle X-ray and neutron scattering (SAXS and SANS) and high-resolution transmission electron microscopy (HRTEM) were used simultaneously to conduct accurate quantitative characterization of the precipitates, in terms of precipitates size, morphology, chemical composition constituent and volume fraction. In addition, electron back-scattered diffraction (EBSD) and scanning electron microscopy (SEM) were also used to determine the microstructure evolution and fracture morphology, in order to clarify the relationship between microstructure and mechanical properties of the Ti-bearing steel processed by NG-TMCP.

#### 2. Materials and methods

#### 2.1. Materials and thermo-mechanical Processing

The nominal composition of the experimental steel in weight percentage (wt%) was 0.15 C, 0.98Mn, 0.28Si, 0.02Al, 0.0027 N, 0.0048 O and balance Fe. A small amount of Ti (0.08 wt%) was added based on the composition Q345 for the maximum precipitation strengthening and grain refinement strengthening. The experimental steels were vacuum-melted in an induction furnace and forged into ingots. After austenitizing at 1250 °C for 2 h, hot rolling was carried out using Φ450 mm experimental hot mill with the finish rolling temperature of 880 °C. Then the steels were ultrafast cooled to different temperatures (620, 580 and 540 °C) and held for 20 min in asbestos. Finally, the steels were air cooled to room temperature. The detailed processing parameters are described in [Table 1.](#page-1-0)

#### 2.2. Microstructural characterization

The specimens were cut from the hot rolled plates with their surfaces along thickness direction and rolling direction. The microstructure was observed using a combination of optical microscope (LEICA DMIRM), scanning electron microscope (Quanta 600), and transmission electron microscope (Tecnai  $G^2$  F20). For EBSD detections, the specimens were electrochemically polished at room temperature in

<span id="page-1-0"></span>Thermal mechanical processing parameters of the experimental steels.

a solution consisting of 650 ml ethyl alcohol, 100 ml perchloric acid, and 50 ml distilled water. Thin foils for transmission electron microscopy (TEM) observations were mechanically thinned to  $\sim$  0.06 mm and then electrochemically jet polished at −30 °C in a solution containing 9 vol% perchloric acid in ethanol.

#### 2.3. SANS, chemical phase analysis and SAXS analysis

SANS was employed to analyze volume fraction of tiny precipitates. Basics of the SANS technique can be found in literature [13–[16\]](#page--1-4). The scattering intensity  $I(q)$  is measured as a function of scattering vector q =  $4\pi \sin\theta/\lambda$ , where λ is the wavelength of the incident neutrons, and θ is half of the scattering angle. A monochromatic neutron beam with a mean wavelength of  $0.53 \text{ nm}$  ( $\Delta \lambda / \lambda = 10\%$ ) was produced using a multiblade mechanical velocity selector. The samples were measured under a magnetic field of 1.5 T in order to separate the magnetic and nuclear scattering signals. By changing the sample-detector distances, ranging from 0.08 to 2.0 nm<sup>-1</sup> has been covered.

In this work, chemical phase analysis and SAXS methods were used simultaneously with TEM and SANS to investigate the chemical composition constituents, volume fraction of nanoscale precipitates with different sizes. The precipitates were electrochemically extracted from the steels, according to the test standard ISO/TS 13762. The detailed procedures for chemical phase analysis were as follows:

- (1) Electrolytic dissolution of the steel sample to obtain electrolyzed powders containing iron carbides, microalloyed carbides, sulfides and nitrides.
- (2) Elimination of iron carbides, sulfides, and AlN to obtain microalloyed carbides and oxides.
- (3) Removal of microalloyed carbides to get stable oxides.

After the above separation procedure, the electrolyzed powders were dissolved and the content of Fe, Mn, N, and C was analyzed. Then the mass fraction of cementite based on formula  $(Fe_aMn_b)_{3}(C_xN_y)$  was calculated. According to GB/T 1322191 (ISO/TS 13762 2001) the size distribution of precipitate powders was analyzed by SAXS with a Kratky small-angle X-ray scattering (Rigaku Corporation, Tokyo, Japan). Using this approach, the analyzed error was less than 10 pct.

#### 2.4. Tensile and impact toughness tests

Standard cylindrical tensile test samples with a gage length of 25 mm and diameter of 5 mm were prepared from the steel plates perpendicular to the rolling direction. Tensile tests were conducted at room temperature to measure the yield strength, tensile strength and elongation with a crosshead speed of 1 mm/min, using a SANS-5000 tensile tester. Standard Charpy v-notch impact samples of dimensions  $10\times10\times55$  mm<sup>3</sup> were prepared along the rolling direction to determine impact toughness at  $-20$  and  $-40$  °C using an Instron 9250 impact tester.



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