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# A novel phase transition behavior during dynamic partitioning and analysis of retained austenite in quenched and partitioned steels



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#### ABSTRACT

We have studied here the carbon partitioning during continuous cooling at different cooling rates using dilatometer. An interesting phase transition was observed involving multiple stages of transformation during dynamic partitioning and a mechanism was proposed to explain the observations. Two morphologies of nano-sized retained austenite were observed, film-like and block-type. It was proposed that the amount of retained austenite was stable in a certain range of cooling rate. When the cooling rate was between 0.05 °C/s and 1 °C/s, the carbon partitioning was adequate and  $\sim$  11 to 14% retained austenite was obtained. With increase in cooling rate from 5 and 10 °C/s, carbon partitioning was not adequate, which resulted in decrease in film-like austenite. The austenite adjacent to ferrite had two different transformation products, M/A (martensite-austenite island) and twin martensite, based on the degree of carbon transfer from ferrite. However, macro-hardness test showed that cooling rate had little effect on hardness and the hardness was between 398 HV and 407 HV at cooling rate of  $\sim$  0.05 to 10 °C/s. Additionally, samples cooled at low cooling rate indicated continuous TRIP effect and excellent combination of high strength 1030 MPa and high elongation of  $\sim$  25% was obtained in the plate cooled at 0.1 °C/s. On the basis of results, a strategy was proposed to obtain high performance hot rolled Q & P steels. The study confirmed the viability of implementing quenching and partitioning process in the hot rolling production line.

#### 1. Introduction

Quenching and partitioning (Q & P) process [1-6] is an approach to produce advanced high strength steels (AHSS) with high strength-high ductility combination. The microstructure consists of predominantly martensite and retained austenite. The key point of the process is carbon partitioning, which occurs on isothermal holding of steel at the quench temperature (QT) or at an evaluated temperature. During isothermal partitioning, carbon diffuses from supersaturated martensite to untransformed austenite, and thus the chemical stability of austenite is increased and is partly retained at room temperature (RT). The isothermal partitioning procedure has been studied, where competing actions including carbon partitioning, martensite nucleation, austenite decomposition and bainite transformation [7-10] occur. Generally, the isothermal partitioning has two important parameters, partitioning temperature (PT) and partitioning time (Pt) that need optimization. However, on combining the thermo mechanical control processing (TMCP) and Q & P concept as illustrated in Fig. 1, the conventional

Q & P process is simplified and the new process has many advantages. First, deformation during rolling can refine the prior austenite, thereby improving the austenite stability via grain size effect [11-14]. Second, the microstructure can be tuned to obtain the required mechanical properties, such as low yield radio, different strength levels and high elongation [15-17]. However, the approach is not widely accepted because QT, PT and coiling temperature (CT) all depend on single temperature and dynamic carbon partitioning is difficult to control. Compared to isothermal partitioning, dynamic partitioning has the following characteristics: (a) quenching temperature determines the initial fraction of martensite and untransformed austenite and the driving force for carbon partitioning. (b) The phase transformation and carbon partitioning are competed during the continuous coil cooling process. (c) The coil cooling rate determines the time for different reactions and generally varies because of different heat transfer conditions in the hot-rolled coil.

The dynamic partitioning is not well understood and there are no direct observations on carbon partitioning process. Moreover, the coil

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**Fig. 1.** Schematic thermal profile of combination of Q & P concept and TMCP technology: The process combines TMCP and Q & P concept to obtain the required phases. Carbon partitioning occurs during the cooling of steel from the quenching temperature to room temperature. HR is hot rolling. DQ is direct quenching. QT is quenching temperature. CT is coil cooling temperature. PT is initial partitioning temperature. V is coil cooling rate.

cooling rate is an important kinetic parameter and has not been studied.

The objective of this study is to elucidate the mechanism of carbon partitioning during continuous cooling process and propose a relationship between coil cooling rate and retained austenite, which provides guidance to hot rolled Q & P steels.

#### 2. Materials and methods

A low carbon steel 0.19C-1.5Si-1.6Mn (wt%) was subjected to direct quenching and partitioning process using dilatometer equipment (DIL 805), which can precisely control temperature to the extent of 0.1 °C. The critical temperature of Ae3 was calculated by Thermo-Calc 5.0 (Fig. 2). The Ms of ~ 400 °C was calculated using an equation in reference [18]. It can be seen from Fig. 1 that an experimental plan was designed to study the effect of coil cooling rate. The steel was first heated to 1200 °C and held for 2 h. After 40% compression deformation two times at 1080 °C and 920 °C at deformation rate of 1 mm/s, the steel was slow cooled to T<sub>1</sub> (in our case, 760 °C) at cooling rate of 2 °C/s to obtain ferrite. Next, the steel was quenched to a predetermined quench temperature of 320 °C at 40 °C/s and cooled to room temperature at different cooling rate of 0.05, 0.1, 0.5, 1, 5 and 10 °C/s. The temperature and dilatation were measured by the dilatometer.

The microstructure was characterized using a Zeiss Ultra-55 field emission scanning electron microscope (SEM) equipped with an electron backscattered diffraction (EBSD) system, which was used to distinguish phases. In order to study the effect of cooling rate on elemental diffusion, specimens were studied by a JXA-8530F electron probe microanalyzer (EPMA) equipped with energy dispersive X-ray spectrum (EDS) system at an operating voltage of 20 kV, current of  $2 \times 10^{-8}$ A and a step size of 40 nm which based on the size of martensite lath and RA.



Fig. 2. The equilibrium phase diagram calculated by Thermo-calc 5.0 software for the experimental steel.

Further observation of fine structure in samples was carried out using a TECNAI G220 microscope at an operating voltage of 200 kV. For SEM and EPMA observations, the specimens were metallographically polished via standard procedure and etched by 4% nital for 10–15 s. The specimens for EBSD analyses at a step size of 40 nm were prepared by first grinding, followed by electro-polishing using an electrolyte containing alcohol, perchloric acid and water in the proportion of 13:2:1 at room temperature. The current was  $\sim 1.0$  A and the time was 25 s for the electro-polishing process. The TEM specimens were first ground to a thickness of 45  $\mu$ m and then electro-polished at - 20 °C using a twin-jet polisher.

Retained austenite was measured by a D/max2400X-ray diffractometer (operated at 56 kV, 182 mA) with Cu K $\alpha$  radiation at room temperature. The samples were scanned over a 2 $\theta$  range from 40° to 110° at a step of 0.04°/s. The integrated intensity of ferrite peaks of (200), (211) and austenite peaks of (200), (220) and (311) were used to calculate the volume fraction of austenite using Jade version 6.5. The amount of retained austenite was obtained using the standard equation in reference [19]. The (200) austenite peak was selected to calculate the average carbon concentration of retained austenite according to the equation in reference [20].

The tensile samples of dimensions 12.5 mm in width, 4 mm in thickness and 50 mm in gauge length were prepared along the rolling direction and tested using a CMT5105-SANS machine at room temperature with a tensile speed of 2 mm/min. The ultimate tensile strength (UTS), 0.2% yield strength (YS) and elongation were obtained based on the average of three tests for each plate.

#### 3. Results and discussion

#### 3.1. The novel phase transformation behavior during dynamic partitioning

The dilatometer can measure temperature and dilatation precisely, so the phase transformation process can be outlined in the dilatation curves. As Fig. 3(a) shows, ferrite transformation occurs at 880 °C, which is higher than Ae3(855 °C). The twice compressive deformation promoted ferrite formation by providing a number of defects [21]. It is interesting that when the temperature was decreased to 780 °C, the dilatation curve increased suddenly caused by high rate of ferrite transformation. Below the critical temperature of  $\sim$  780 °C, the amount of ferrite is sensitive to temperature and is difficult to control. Thus, by introducing ferrite into the Q & P process, it is important to control the amount of ferrite based on the critical temperature. The dilatation during quenching process was observed as shown in Fig. 3(b). The dilatation curve was not straight between 503 °C and 625 °C, where bainite transformation occurred. Bainite transformation was due to low C and Mn concentration and martensite transformation occurred at 355 °C, and was different from the calculated value of  $\sim$  400 °C.

The dilatation curves of dynamic partitioning are shown in Fig. 4. It is interesting to note that the dilatation curves are different from the conventional continuous cooling process in terms of multiple phase transformation points. It can be seen from Fig. 4(a) that on cooling the steel from 320 °C to 150 °C at 0.05 °C/s, the dilatation curves indicated four distinct discontinuities (rise) that were caused by volume expansion induced by the transformation (red broken lines). The sudden rise was caused by martensite transformation and according to the mechanism of martensitic transformation, martensitic transformation should occur once on undercooling. Thus, continuous martensite transformation was expected during the continuous cooling process rather than multiple-stages of phase transformation. This result indicates that a new mechanism must be operative. The reason is that carbon partitioning occurred and changed the Ms of untransformed austenite. A few studies suggested that coil cooling process had adequate time for carbon partitioning [22,23]. So, the new phenomenon is a combined effect of transformation and carbon partitioning.

As coil cooling process progressed, the untransformed austenite was

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