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# Synergistic effect of austenitizing temperature and hot plastic deformation strain on the precipitation behavior in novel HSLA steel



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#### ABSTRACT

Examination of thin foils of specimens with various austenitizing conditions by transmission electron microscopy revealed randomly homogeneous precipitation in the ferrite for each experimental condition. Though no interphase precipitation was found in the present study, two types of random precipitation morphologies were identified in the ferrite matrix. One was randomly and homogeneously precipitated carbides of smaller size (<10 nm), and the other was randomly precipitated carbides of larger size (10–30 nm). Transmission electron microscopy results provided evidence that both types of precipitation carbides could be associated with the supersaturation of microalloying elements in the ferrite and austenite, respectively. A higher austenitizing temperature treatment can lead to more microalloying elements dissolving in the austenite such that many tiny carbides precipitation at the low isothermal holding temperature, which is believed to effectively strengthen the ferrite.

Vickers hardness data revealed that, in specimens austenitized at  $1200\,^{\circ}\text{C}$  and deformed at  $900\,^{\circ}\text{C}$  with strains of 10% and 30%, the ranges of hardness distribution were  $250-360\,\text{HV}$  0.1 and  $310-400\,\text{HV}$  0.1, respectively. For specimens austenitized at  $1000\,^{\circ}\text{C}$  and deformed at  $900\,^{\circ}\text{C}$  with strains of 10% and 30%, the ranges of hardness distribution were  $220-250\,\text{HV}$  0.1 and  $220-260\,\text{HV}$  0.1, respectively. Therefore, the average Vickers hardness increased with the austenitizing temperature and deformation strain. However, a wider range of hardness distribution occurred in specimens that underwent treatment at higher austenitizing temperatures. The wider Vickers hardness distribution reflects non-uniform precipitation in each ferrite grain.

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#### 1. Introduction

There is currently a huge demand for higher strength and good formability steel for reducing vehicle weight to save fuel. Therefore, many advanced high strength steels (AHSS), such as transformation-induced plasticity (TRIP) steel, twinning-induced plasticity (TWIP) steel, and complex phase (CP) steel, have been developed since 2000 [1,2]. Although these steels can achieve excellent mechanical properties, the poor stretch flange formability due to mixed phases restricts their application in the automobile industry. It is pointed out that to achieve good stretch flange formability, a uniform microstructure is the most important requirement for a high value of hole expansion ratio,  $\lambda$ , to be

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achieved [3]. From the viewpoint of phase transformation in the steel, ferrite, which does not contain complex phases like bainite or martensite, is a good candidate for meeting this requirement. However, the low strength of ferrite suppresses its application in modern vehicles. Therefore, strengthening methods need to be applied to ferrite. Among the various hardening mechanisms, precipitation strengthening can maintain the original characteristics of ferrite, such as good elongation, while achieving a high strengthening effect. According to Gladman's report [4], precipitates of 3–5 nm in steel have the greatest power to resist dislocation cutting or bowing, so causing the formation of these tiny precipitates in ferrite would be beneficial for achieving a good quality of ferrite steel.

However, these tiny size precipitates do not commonly occur in traditional hot rolled steel because many microalloying elements will be exhausted in the high temperature manufacturing processes [5]. The properties of the steels can be modified through intrinsic or extrinsic routes, namely from steel chemistry and thermo-mechanical processing. In previous studies, much effort

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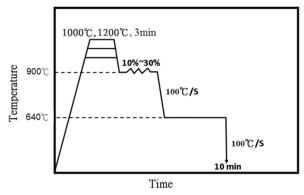
has been focused on the influence of the steel composition on the precipitation behavior in the novel HSLA steel. It has been reported that the titanium molybdenum complex carbide (TiMo)C has the lowest coarsening rate and can attain a tiny size (3–5 nm) under adequate isothermal aging treatment [6–8]. The accelerated decomposition of austenite under hot deformation can increase the amount of supersaturated precipitation in most ferrite grains [9–11]. In addition to isothermal aging temperature and hot deformation, other thermo-mechanical process parameters, such as austenitizing temperature, the amount of hot plastic deformation strain, and the cooling rate of coiling, also influence the precipitation strengthening effect greatly and are worthy of further study.

In the past, many reports have focused on the regular array of precipitation carbides within the ferrite matrix (i.e., interphase precipitation). Smith and Dunne even distinguished the morphologies of interphase precipitation carbides in different microalloyed steels and summarized them into three types: (1) planar interphase precipitation with regular sheet spacing, (2) curved interphase precipitation with regular sheet spacing, and (3) curved interphase precipitation with irregular sheet spacing [12]. Focusing on the interphase precipitation mechanism, Funakawa et al. successfully developed a new kind of steel named Nanohiten steel [13]. On the other hand, compared to those examining interphase precipitations, more and more studies have proved another precipitation morphology (i.e., random or general precipitation) and conducted research on their hardening effects. For example, based on a mathematical modeling and computer simulation approach, Chen et al. suggested that random array carbides can achieve a better strengthening effect if distributed as finely as in interphase precipitation [14]. Misra et al. reported that in Ti-Nb-Mo steel, interphase precipitation occurs primarily during austenite/ferrite phase transformation, and random precipitation, during isothermal holding [15]. Despite the superior hardening effect of these nano-sized particles, the large discrepancy in microhardness distribution between each ferrite grain is worthy of study. Without providing any solution approach, Chen et al. only pointed out that the different precipitation behaviors in each ferrite could be the main cause of such non-uniformity of mechanical properties [16]. Therefore, understanding the influence of austenitizing conditions on the mechanical properties of each ferrite grain can provide insight to improve the mechanical properties of precipitation strengthened

In order to simplify the experiment variables, isothermal aging at a fixed temperature ( $640\,^{\circ}\text{C}$ ) for various austenitizing temperatures and different amounts of deformation can shed light on the precipitation behavior more clearly. It can do so because of the opposite trends of austenitizing temperature and deformation described above. In short, the objective of the present study is to provide insight into the synergistic effect of austenitizing temperature and the amount of hot plastic deformation strain on the precipitation behavior of the novel HSLA steel containing Ti and Mo. The mechanism of precipitation strengthening after different amounts of hot deformation strain is examined at various austenitizing temperatures. The precipitates are examined in terms of their size, morphology, chemistry, and crystallography to determine the strengthening mode in the HSLA steel.

#### 2. Experimental procedure

The chemical composition of the steel studied was Fe–0.06 C–1.51Mn–0.11Si–0.21Ti–0.19Mo–0.004N–0.002S (wt%). The asreceived material was HSLA steel plate (with a thickness of about 45 mm) produced through high temperature soaking, hot rolling, and accelerated cooling.



**Fig. 1.** Schematic diagram of microalloyed steel heat treatment process; austenitizing at  $1000\,^{\circ}$ C,  $1200\,^{\circ}$ C, 10-30% plastic deformation strains at  $900\,^{\circ}$ C, isothermal holding at  $640\,^{\circ}$ C for 10 min.

In the present work, all isothermal holding treatments were carried out on a Dilatromic III RDP deformation dilatometer produced by Theta Industries, Inc. Before preparation of the dilatometry specimens, the pieces of steel were homogenized at 1250 °C for 2 days while sealed in a quartz tube containing argon and subsequently quenched in water. After the decarburization layer was removed, the specimens were then machined into cylinders 3 mm in diameter and 6 mm in length. The dilatometer was interfaced with a computer workstation (PDP 11/55 central processor) for analysis of the resulting data. A software package (provided by Theta Industries, Inc.) allowed flexible and complete control in the execution of the isothermal aging experiments. The length, time, and temperature information was recorded in microsecond intervals, and the level of vacuum was maintained at  $10^{-5}$  Torr to protect the specimens from oxidation. A schematic diagram of the isothermal heat treatment experiments performed in the dilatometer is presented in Fig. 1. After being austenitized at 1000 °C or 1200 °C, respectively for 3 min, the specimens were directly cooled to 900 °C for 10%, 20%, and 30% deformation. After that, the specimens were cooled to the isothermal aging temperature (640 °C) at a cooling rate of about 100 °C/s to prevent carbide precipitation prior to isothermal aging and then were isothermally held at that temperature for 10 min before being quenched to room temperature.

The samples were mainly characterized by optical microscopy (OM), scanning electron microscopy (SEM, JEOL JSM-6360LV and FEI Quanta 600 at 20 kV), transmission electron microscopy (TEM, JEM-2000), and field emission gun transmission electron microscopy (FEG-TEM Tecnai F30) equipped with a nanometer probe energy dispersive spectrometer (EDS). The size of the ferrite grains was estimated in terms of mean intercept length. A microhardness measurement of the specimens to be examined optically was taken using a Vickers hardness tester with a load of 0.1 kg. In order to determine the precipitation status in the ferrite, for each steel and processing condition, measurements from 100 ferrite grains were used to plot the final microhardness distribution. To prevent the influence of the strain field of each indentation test and interference with other grains, the position and the distance of each indentation mark was carefully chosen.

Thin foil specimens were prepared for transmission electron microscopy from 0.25 mm thick disks slit from the specimens used in the dilatometry experiments. The disks were thinned to 0.5 mm by abrasion on silicon carbide paper and then electropolished in a twin jet electropolisher using a solution of 5 vol% perchloric acid+25 vol% glycerol+70 vol% ethanol at  $-2\,^{\circ}\text{C}$  and 30 V potential.

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