

Contents lists available at ScienceDirect

Materials Science & Engineering A



journal homepage: www.elsevier.com/locate/msea

# Increase of martensite start temperature after small deformation of austenite



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#### ARTICLE INFO

Article history: Received 24 January 2014 Received in revised form 24 April 2014 Accepted 30 April 2014 Available online 9 May 2014

Keywords: Ms temperature Deformation GND Subgrains Martensitic transformation

#### ABSTRACT

It is generally considered that martensite start (Ms) temperature is decreased after plastic deformation of austenite if the previously applied load is retrieved. This can be explained by the dislocation stabilization mechanism. The present work performed systematic deformation and dilatometer experiments to investigate the effect of plastic deformation on Ms temperature. It is found that Ms temperature first increases at small strain and then decreases at large strain. Since the dislocation stabilization mechanism can only predict the decrease of Ms temperature after plastic deformation, a new mechanism is thus proposed to describe this new interesting finding. That is, the increase of Ms temperature is due to the pile-up of geometrically necessary dislocations at austenite grain boundaries, while the decrease of Ms temperature is caused by the formation of subgrains in austenite grain interior.

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#### 1. Introduction

An important feature of martensitic transformation (MT) in steels is that it has a certain transformation starting temperature, namely, the martensite start (Ms) temperature. Ms temperature is also an important parameter for the thermal/mechanical treatment of steels. For example, the fabrication of carbide-free bainite (CFB) steel and quenching and partitioning (Q&P) steel, which are new advanced high strength steels (AHSS) for automotive applications [1,2], requires the knowledge of Ms temperature in advance [3,4]. Ms temperature depends on the chemical composition [5], grain size [6], cooling rate [7], applied elastic stress [8] and defects [9]. It is reported that certain kinds of austenite grain boundaries may provide the potent nucleation site [9,10]. On the other hand, the role of dislocation on MT is more controversial. The growth of pre-existing martensite embryos requires the nucleation of new dislocation loops at the embryo/austenite interface and the glide of these dislocation loops can extend to the interface [11]. However, the large amount of dislocations generated by a large plastic deformation of austenite prior to the MT may stabilize the glissile embryo-austenite interface, leading to a decrease of Ms temperature. This is known as the dislocation stabilization mechanism and has been studied intensively in literature [12,13]. Nevertheless, the effect of small deformation on Ms temperature is much less

\* Corresponding author. Tel.: +852 28597906; fax: +852 28585415. *E-mail address:* mxhuang@hku.hk (M.X. Huang). investigated and not yet well understood. A recent report showed that Ms temperature continuously decreased with the increase of amount of deformation [14]. But the experiment suffered from decomposition of austenite into bainite during the deformation and quenching process so that the carbon content of retained austenite could be changed due to carbon partitioning or carbide formation, which would affect Ms temperature. Careful experiments should be designed to avoid austenite decomposition during the deformation and quenching processes so that the effect of deformation on Ms temperature can be accurately measured. Thus, the present work aims to carry out well-designed systematic deformation-dilatometer experiments to investigate the effect of both small and large deformations on Ms temperature, excluding the effect of austenite decomposition.

#### 2. Experimental procedures

Steel with a chemical composition of Fe–0.2C–1.5Mn–2Cr (in wt%) was employed as a model material for the present work. The material was cast by levitation casting and hot rolled to a final thickness of 6 mm. Cylindrical dilatometry samples with a length of 10 mm and a diameter of 5 mm were prepared from the hot-rolled sheets along the rolling direction. Dilatometry tests were performed in a Bähr, 805A/D deformation dilatometer. The  $A_{c1}$  and  $A_{c3}$  temperatures were found to be 762 and 795 °C, respectively, with a heating rate of 10 °C s<sup>-1</sup>. The samples were homogenized at 900 °C for 300 s. The heating and homogenization were carried

out under a vacuum condition. The critical cooling rate for obtaining a fully martensitic microstructure was verified from the continuous cooling experiments to be 10 °C s<sup>-1</sup>. The isothermal holding experiments were performed to search for the best deformation temperature to avoid the decomposition of austenite during deformation. The time-temperature-transformation (TTT) diagram was measured and is shown in Fig. 1(a) where the typical C curves for the ferrite/pearlite and bainite domains are observed. The incubation time is based on the 5% transformed volume fraction. Fig. 1(a) shows that the decomposition of austenite at 550 °C has the longest incubation time, indicating that 550 °C is the best temperature to carry out the plastic deformation of austenite. Such low deformation temperature of austenite can minimize the dynamic recovery of dislocations, leading to a more noticeable effect of deformation on Ms temperature.

The temperature-deformation program employed in the present study is schematically illustrated in Fig. 1(b). As it shows, the sample was heated with a heating rate of 10 °C s<sup>-1</sup>, followed by an isothermal holding at 900 °C for 300 s and then quenched to 550 °C with a cooling rate of 30 °C s<sup>-1</sup>. This cooling rate is high enough to avoid the decomposition of austenite. After an isothermal holding of 5 s at 550 °C the sample was immediately quenched to room temperature with a cooling rate of 100 °C s<sup>-1</sup> to capture the Ms temperature. The samples were reheated and quenched three times to avoid any effect of initial microstructure and to achieve a better sample accommodation and a precise dilatation measurement. The variation of Ms temperature among these three thermal cycles was about  $\pm 1$  °C. The varied amount of deformation (compression) was applied at 550 °C with a strain rate of 1 s<sup>-1</sup> during the fourth circle and then immediately



**Fig. 1.** (a) The TTT diagram of the present steel. The ideal temperature for deformation of austenite without decomposition is 550 °C. (b) A schematic illustration of temperature program for the present steel ( $\times$ 3 means that such treatment repeats 3 times). The red line represents the applied deformation at 550 °C during the fourth cycle. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

quenched to room temperature. The resultant Ms temperature after deformation was compared with the Ms temperature from the third quenching of the same sample to avoid sample inhomogeneity. The only difference between thermal cycles and the deformation cycle was the applied deformation. For microstructure analysis, the deformed samples were cut into two parts along the radial direction and were observed at the center of the cross section using a scanning electron microscope (SEM) at 5 kV (LEO 1530) and electron backscattered diffraction (EBSD) at 20 kV (HKL Channel 5). The samples for SEM observation were prepared by mechanical polishing down to 1 µm and then slightly etched with 2% nital solution for 15 s. The samples for EBSD measurement were vibratory polished with colloidal silica after the mechanical finish of 1 µm. A step size of 0.15 µm was used for EBSD measurement. The mechanical properties of martensite transformed from the deformed austenite were characterized by measuring the Vicker's hardness with an applied load of 10 kg at room temperature.

#### 3. Results and discussions

Fig. 2(a) shows the dilatation curves for the cycles with and without a deformation of 4.7%. The dilatometer curves were arranged vertically to give a proper comparison. The definition of Ms temperature is based on the tangent method [15]. Fig. 2 (a) shows that the Ms temperature during the third quenching was 391 °C, which was increased to 402 °C after a deformation of 4.7%. The increase of Ms temperature (  $\sim$  11 °C) is much larger than the variation of Ms temperature among different thermal cycles, showing an obvious increase of Ms temperature after a small deformation of 4.7%. The net change of Ms temperature ( $\Delta$ Ms) is adopted here because the Ms temperature was only compared between the cycles with and without deformation. The positive value of  $\Delta Ms$  indicates an increase of Ms temperature after deformation, while the negative one shows a decrease of Ms temperature after deformation. The  $\Delta Ms$  with respect to the different applied strains is summarized in Fig. 2(b). Each data point represents an average of two independent tests. The error bar is a standard deviation. As Fig. 2(b) shows,  $\Delta Ms$  increases with strain initially up to a peak value of  $\Delta$ Ms at a strain of 9.4%, with the maximal  $\Delta Ms$  of about 20 °C. Then,  $\Delta Ms$  decreases with an increase of strain. It is noted that the zero point of  $\Delta Ms$  was around the deformation of 22%. With further straining, the  $\Delta Ms$ became negative and decreased with the increase of deformation, indicating that Ms temperature was suppressed by large deformation, which is consistent with reports in literature [12]. The corresponding engineering stress-strain curves for different amounts of deformation are shown in Fig. 2(c). The austenite was continuously work hardened with the increase of applied strains. Fig. 2(d) shows that the average Vicker's hardness of the product martensite increases with the increase of applied strains. This may be due to the continuous refinement of martensite microstructure after an increase of applied plastic deformation on austenite.

The microstructure of product martensite transformed from the parent austenite with a prior deformation of 4.7% is shown in Fig. 3(a). Several large martensite blocks were observed. Interestingly, the one marked with red arrow had a straight block boundary, across the austenite grain and halted at the prior austenite grain boundary (PAGB). The morphology of this martensite block may indicate that its propagation was not hindered by the small deformation of 4.7%. Therefore, from the observation in Fig. 3(a), it may be concluded that a small deformation of 4.7% did not affect the growth of martensite. With the increase of applied strains, the martensite blocks which penetrated the whole austenite grain are

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