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Variation of martensite phase transformation mechanism in minor-stressed T91 ferritic steel

Baoqun Ning [a,b], Qingzhi Shi [c], Zesheng Yan [c], Jicheng Fu [c], Yongchang Liu [b,*], Lijian Bie [a]

[a] *School of Materials Science and Engineering, Tianjin University of Technology, Tianjin 300191, PR China*
[b] *School of Material Science and Engineering, Tianjin University, Tianjin 300072, PR China*
[c] *Tianjin Pipe (Group) Corporation, Tianjin 300301, PR China*

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ABSTRACT

The effects of compressive stress applied at different temperatures on martensite transformation process of the T91 steel were studied by high-resolution differential dilatometer. The stress applied above 850 °C exhibits no influence on the martensite formation. The stress applied below 850 °C not only facilitates the formation of martensite, but also enhances the onset temperature of the martensite transformation. There are two different transformation mechanisms occurring: when the compressive stress is applied at high temperature, the mechanism of strain-induced martensite transformation takes place, as a result, the microstructure tends to be refined with irregular grain boundary. When the compressive stress is applied at low temperature, the stress-induced martensite transformation occurs, and its morphology is similar to that of thermal-activated martensite. In addition, it is summarized that 200 MPa is the critical stress and 440 °C is critical temperature for the onset of the stress-induced martensite transformation for the investigated T91 ferritic steel.

1. Introduction

T91 steel (ASME SA-213 T91/P91) is a representative type of high Cr ferritic heat-resistant steel. It has been recognized as the preferable material and widely used in high-temperature structural components such as header and main steam pipe in advanced power plants in view of good high-temperature endurance, creep resistance properties, excellent heat conductivity, low thermal expansion coefficient and high performance-cost ratio. Additionally, it has also been widely used as a research benchmark for developing new ferritic heat-resistant steel with higher servicing temperature in Japan and Europe [1–5].

For that full martensitic structure can be obtained in the T91 steel as it is air-cooled down from hot rolling temperature, the final heat-treatment is provided as normalizing plus tempering and the service structure is tempered martensite. Applied stress will change the formation temperature and kinetic process of martensite transformation in the temperature range between the critical temperature (M_d) below which the subsequent martensitic transformation is sensitively influenced by the applied stress and the onset temperature of the martensitic transformation (M_s), and influence the final properties of the T91 steel [6,7]. Since the applied stress above M_d will not influence the kinetics of the martensite transformation, it is technologically important to determine the M_d temperature for controlling the final martensitic structure during the solid-state phase transformation process. Here, the effect of uniaxial compressive stress applied at different temperatures above the M_s point on the martensitic transformation in the T91 steel was systematically explored and desired to improve our theoretical understanding of transformation mechanism of the T91 steel.

2. Experimental details

The T91 steel was machined into cylindrical specimens with a length of 10 mm and a diameter of 5 mm. A Bähr DIL 805 A/D high-resolution differential dilatometer was used to record the length change of cylindrical specimens during heat-treatment. Fresh specimens were used for each dilatometric measurement cycle to ensure the same initial structure and thermal/mechanical history. All measurements were performed in the flowing protective argon atmosphere to avoid oxidation.

The cylindrical specimens were heated up to 1100 °C with a rate of 180 °C/min and held at 1100 °C for 10 min to achieve complete austenization. Then the specimens were cooled down to a certain temperature (880, 850, 760, 650, 550 and 440 °C) with a rate of 100 °C/min and deformed by 100, 150 and 200 MPa, respectively after holding 30 s. After releasing the applied load, the specimens were continuously cooled down to room temperature with a rate of 100 °C/min to get a full martensitic structure. Additionally, experiments without applied stress were also implemented in accordance with the same temperature program as mentioned above (see Fig. 1).

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[*] Corresponding author. Tel./fax: +86 22 87401873.
E-mail address: licmtju@163.com (Y. Liu).

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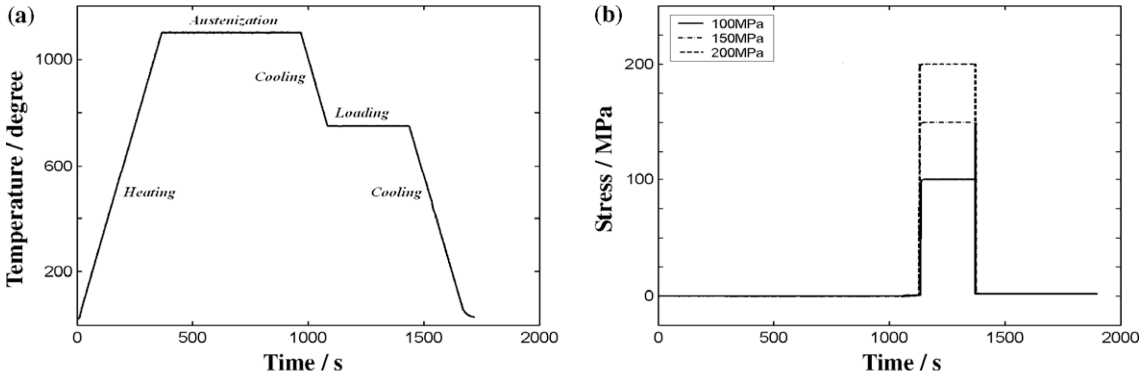

Fig. 1. Experimental program with applied compressive stress of the T91 steel, relationship between (a) temperature and time, (b) time and applied stress.

The heat-treated specimens were prepared by standard metallographic procedures, and $FeCl_3$ solution was used as an etchant. The microstructures of the specimens were observed by a C-35A OLYMPUS Optical Microscope.

3. Results and discussion

The formation of the martensite in the explored T91 steel during cooling was affected by the applied stress and the deformation temperature. Since the martensite transformation is associated with a volume expansion, which can be easily observed in the recorded linear dilation curves upon isochronal cooling. To make it clear, the volume fraction of martensite formed at different temperature was deduced from the measured dilatometric curves, and the onset point of martensitic transformation (M_s) was also determined (cf. segment JK in Fig. 2). Combined with microstructure observation, the effect of small applied stress on the martensite transformation in the T91 steel as well as the inherent mechanism would be clarified.

3.1. Phase transformation behavior under minor applied stress

3.1.1. Analysis of linear expansion behavior

The length changes of the T91 specimen deformed at 760 °C are shown in Fig. 2 as a function of time, in which segment AB corresponds with the normal thermal expansion of the specimen during continuous heating in the absence of a phase transformation; Part BC represents the formation of the austenite with a length contraction; CD represents normal expansion of the austenite. DE stands for holding process in the austenite region; EF indicates normal contraction of the austenite upon cooling. FG and HI parts represent the parts when compressive stress is applied (FG represents the part with applied stress starts to be applied, the length of sample decreases; GH section is the period when constant compressive stress is applying (see Fig. 1(b)); HI corresponds elastic recovery of the deformed specimen); IJ represents normal contraction of the austenite; JK indicates the expansion due to the occurrence of the martensite transformation; KL section represents normal contraction of the transformed martensite upon continuous cooling.

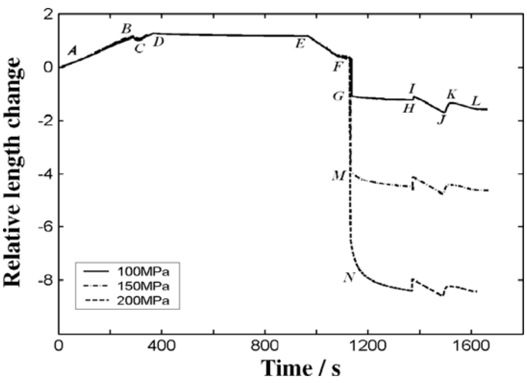

Fig. 2. Measured relative length changes of the T91 steel as a function of temperature during dilatometric experiment.

3.1.2. Determination of the M_s point

According to dilatometric measurements, the volume change during the martensitic transformation was analyzed according to lever rule [8] for the small-deformed T91 steel specimens. The fractions of martensite were obtained as function of temperature. Fig. 3(a)–(f) are the calculated fraction curves for the specimens deformed at 880, 850, 760, 650, 550 and 440 °C with different compressive stresses applied. Fig. 3(g) is the transformed fraction as a function of temperature without applied stress.

It can be seen from Fig. 3 that the evolution of martensite fraction are similar when different applied stresses are applied at the same temperature. However, the M_s point changed as the deformation temperature and the applied stress varied: at 880 °C, the M_s points are 420 °C even different compressive stresses were applied. At 850 °C, the M_s points, for the applied compressive stresses of 100, 150 and 200 MPa, are 471, 460 and 430 °C, respectively. At 760 °C, the M_s points, for the applied compressive stresses of 100, 150 and 200 MPa, are 445, 435 and 424 °C, respectively. At 650 °C, the M_s points, for the applied compressive stresses of 100, 150 and 200 MPa are 443, 434 and 428 °C, respectively. At 550 °C, the M_s points, for the applied compressive stresses of 100, 150 and 200 MPa, are 430, 422 and 420 °C, respectively. Additionally, the M_s point of the reference sample without applied stress is 420 °C.

3.1.3. Relationship between the M_s point and the applied compressive stress

According to experimental results, the obtained M_s points of the investigated T91 steel are represented as a function of the applied compressive stress in Fig. 4 (thin dot line represents the M_s point of the reference sample without applied stress). The following observations could be reached: (i) at 880 °C, the applied stress has no effect on the M_s point, (ii) when different stresses were applied at 850 °C, the formation of martensite is promoted (the M_s point increased with the increase of the applied compressive stress). Hence, 850 °C is the M_d point for the explored T91 steel. Deformation above the M_d point would not prompt the formation of the martensite.

Applying compressive stress below 850 °C, most of the M_s points are higher than that of the reference sample (at 550 °C and 440 °C, the applied stress does not influence the M_s point), thus the applied compressive stress was benefit for the formation of the martensite

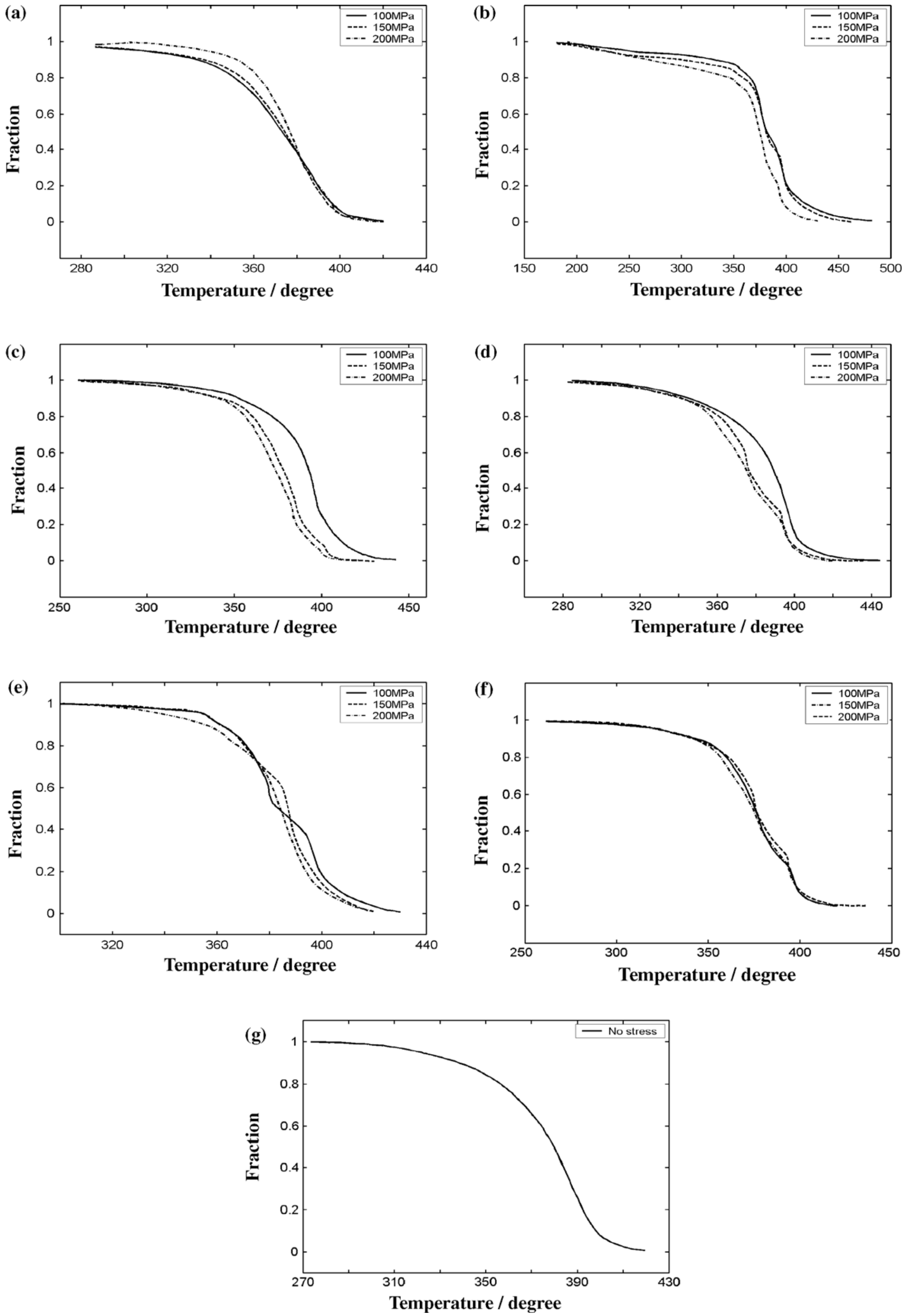

Fig. 3. The martensite fraction, *f*, as a function of temperature, *T*, during the martensite transformation of the T91 steel after deformation at (a) 880 °C, (b) 850 °C, (c) 760 °C, (d) 650 °C, (e) 550 °C, (f) 440 °C and (g) reference sample.

(corresponding to an increase of the M_s point). However, under different applied stresses, the variation of the M_s point is different.

In the temperature range of 850 °C and 550 °C, the M_s point decreased with the increase of the applied compressive stress.

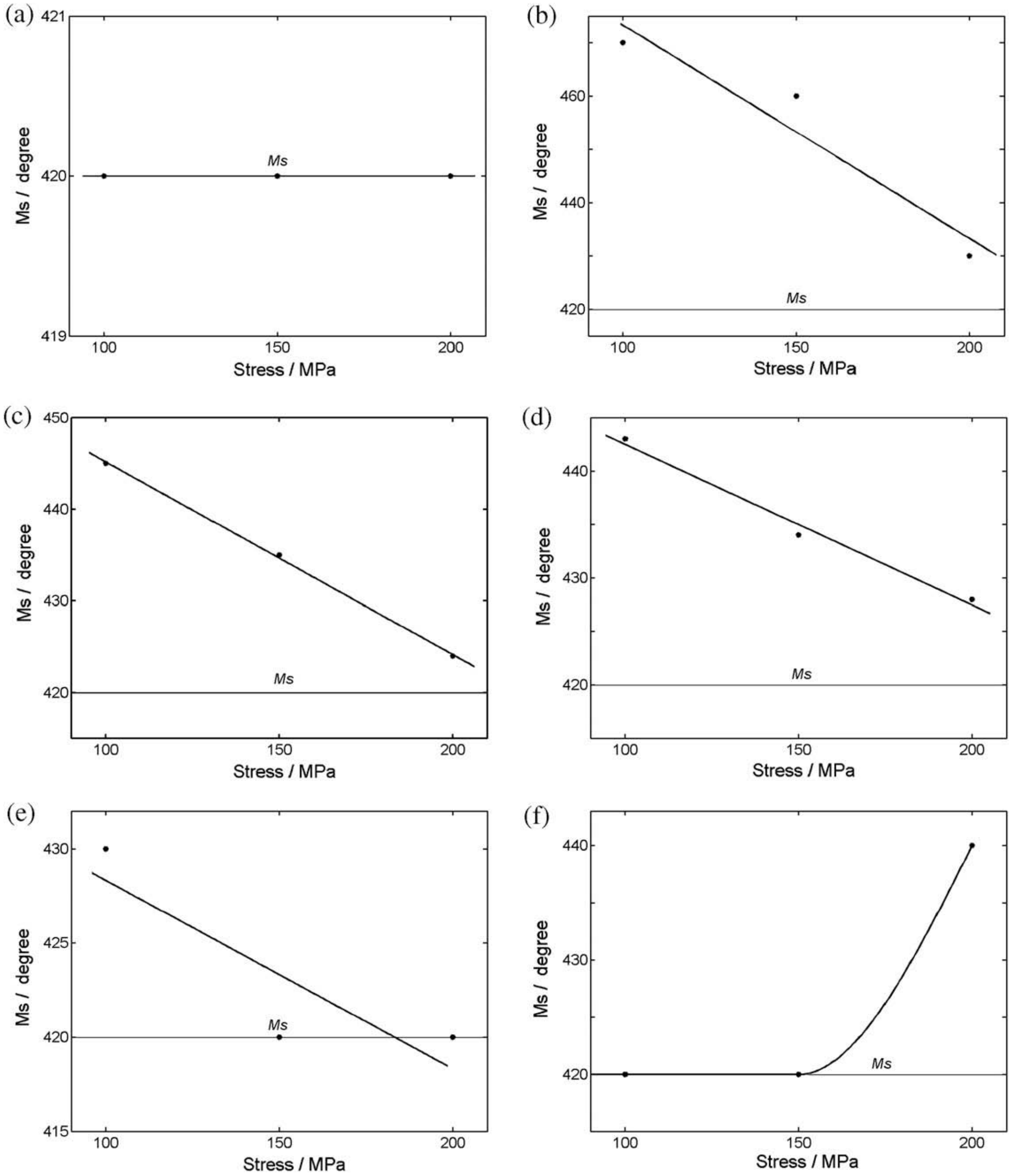

Fig. 4. The determined M_s point of the T91 steel as a function of applied compressive stress at, (a) 880 °C, (b) 850 °C, (c) 760 °C, (d) 650 °C, (e) 550 °C and (f) 440 °C.

However, the M_s point of the T91 steel not vary with the applied stress of 200 MPa at 550 °C. At 440 °C, the M_s point did not change until the applied compressive stress approached to 200 MPa.

3.2. Martensite transformation mechanism

At 850, 760, 650 and 550 °C, the applied stress of 100 MPa increased the M_s point of the T91 steel obviously. However, the M_s point decreased as the applied stress increased at the same temperature. At 440 °C, the applied stresses of 100 and 150 MPa show no effect on the M_s point. When the applied stress increased to 200 MPa, the martensite transformation occurred instantly. Though the M_s points were all increased when the applied stress at 850, 760, 650, 550 and 440 °C, two different martensitic transformation mechanisms exist: strain-induced and stress-induced.

3.2.1. Strain-induced martensitic transformation

Full martensitic transformation occurred because the T91 steel has high hardenability and its yield strength decreases with the increase of temperature. It is noted that even when the applied stress is possibly lower than the yielding stress, the transformation strain is large enough to induce local plastic deformation in the untransformed austenite [9]. Thus, at relative high temperatures (850, 760, 650 and 550 °C), the austenite has low yield strength and thus the enhanced stress would induce large local plastic deformation of the austenite (see Fig. 2, the length contraction increased with the increase of the applied compressive stress).

Deformation would increase the free energy of the austenite, decrease its stability and introduce defects in the austenite. However, the dynamic and static recovery would take place slowly at the temperature (for example, 760 °C) lower than recrystallization temperature, and therefore the defects (such as grain boundary, slipping band and dislocation) would be introduced in the austenite. The grain boundary energies and defects caused by deformation provide more nucleation sites which would prompt the martensitic transformation [10,11].

Additionally, the austenite would be strengthened and the corresponding yield strength ($\sigma_{0.2}$) of the T91 steel would be increased as the austenite deformed in the non-recrystallization region. Moreover, this tendency would become stronger and stronger with the decrease of the temperature. Finally, it would induce an increase of the yield strength of the martensite (σ_{M_s}).

According to the theory of martensitic transformation, the difference in Gibbs energy between the austenite and the martensite ($\Delta G^{\gamma\to M}$) can be calculated as [7]:

$$\Delta G_{M_s}^{\gamma\to M} = \Delta G_{ch}^{\gamma\to\alpha} + 2.1\sigma_{M_s} + 900 - 0.2\sigma \quad (1)$$

where σ_{M_s} is yield strength of austenite at the M_s point, σ is the applied stress.

As seen from Eq. (1), the applied stress would induce two contrary effects on the subsequent martensitic phase transformation: one is the increase of the M_s point (due to the negative energy contribution from the item of 0.2σ), the other one is the effect of austenite strengthening (due to the positive energy contribution from the item of σ_{M_s}), which leads to a mechanical stabilization of the T91 Steel. For the reason that the yield strength of the austenite (σ_{M_s}) might increase (due to the stress-hardening) with the applied compressive stress, this would lead to more transformation strain for the martensitic transformation, which retards the phase transformation. Hence, more chemical driving force is expected to overcome the transformation strain induced by the stress-hardening of the austenite, which would be directly linked to a decrease of the M_s point.

Hereby, the effects of the applied stress on the M_s point can be ascribed to two reasons: (i) the new defects, induced by the local plastic deformation [10], which are benefit for the nucleation of the product phase; (ii) deformation strengthens the parent phase and inhibits the nucleation of the product phase. When the contribution of the defects dominates, the M_s point of the deformed T91 steel increases. Instead, the corresponding M_s point decreases when the strengthening effect of the austenite dominates. In the temperature range of 550–850 °C, the applied compressive stress would induce local plastic deformation in the austenite [9] and make the effect of the formation of the defects dominate, thus the M_s point of these samples would be higher than that without applied stress. However, as the stress increased at the same temperature, the yield strength of parent phase increased, which led to a decrease of the M_s point (the strain-induced martensite transformation). The relationship between the M_s point and the applied compressive stress can be approximately expressed as:

$$850\,^\circ\mathrm{C} \quad \Delta M_s/\Delta\sigma \approx -0.41\,^\circ\mathrm{C/MPa}, \quad (2)$$

$$760\,^\circ\mathrm{C} \quad \Delta M_s/\Delta\sigma \approx -0.21\,^\circ\mathrm{C/MPa}. \quad (3)$$

When the stress was applied at a relative low temperature (for example, 440 °C), the deformation of the austenite is unconspicuous for the high yield strength of the austenite. However, the martensitic transformation occurred immediately as the applied compressive stress increased to 200 MPa. The transformation mechanism is different form the previous one.

Fig. 5 shows variation of the Gibbs energy of the austenite (G^γ) and the martensite (G^M) as a function of the temperature. In it, T_0 is the temperature at which G^γ equals to G^M. At other temperatures, the difference in Gibbs energy ($\Delta G^{\gamma\to M}$) can be expressed as:

$$\Delta G^{\gamma\to M} = G^M - G^\gamma \quad (4)$$

When $\Delta G^{\gamma\to M}$ is positive, the energy of martensite is higher than that of austenite, the transformation from austenite to martensite would not occur. On the contrary, when $\Delta G^{\gamma\to M}$ is negative, the austenite would transform into the martensite. When $\Delta G^{\gamma\to M}$ is equal to the critical driving force for transformation ($\Delta G^{\gamma\to M}$), the transformation from austenite into martensite would occur.When the compressive stress was applied at the temperature between T_0 and M_s, such as T_1 in Fig. 5, an mechanical driving force (U) was provided based on chemical driving force ($\Delta G^{\gamma\to M}$) . As U plus $\Delta G_{T_1}^{\gamma\to M}$ equaled to $\Delta G_{M_s}^{\gamma\to M}$, the martensitic transformation occurred at the temperature of T_1. The phenomenon of martensite formation promoted by the mechanical driving force (U) was called stress-induced martensite transformation. The nucleation of martensite belongs to synergic nucleation mode and U is critical driving force for the formation of the stress-induced martensite:

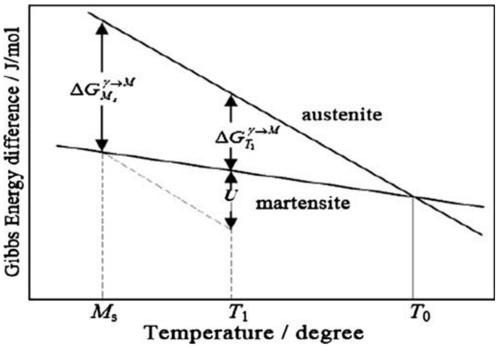

Fig. 5. Schematical diagram of the stress-induced martensite transformation [6].

$$U = \Delta G_{M_s}^{\gamma\to M} - \Delta G_{T_1}^{\gamma\to M} \quad (5)$$

In 1970, Richman and Rolling have found the same phenomena in other martensite steels and measured the critical temperature. As for the T91 steel studied here, the applied compressive stresses of 100 and 150 MPa at 440 °C can not provide enough mechanical driving force for the formation of the stress-induced martensite. Whereas the applied compressive stress of 200 MPa can provide enough mechanical driving force for the formation of the stress-induced martensite, which would lead an increase of the M_s point (440 °C).

The results obtained above can be testified by the formation rate of the martensit martensite as calculated from the fraction curves. Fig. 6 shows the martensite formation rates as a function of fraction under three different applied compressive stresses (100, 150 and 200 MPa). Formation rates under applied compressive stresses of 100 and 150 MPa are the same, while transformation with applied compressive stress of 200 MPa has different formation rate. As the onset transformation temperature is higher, the maximum formation rate of the martensite in the T91 sample

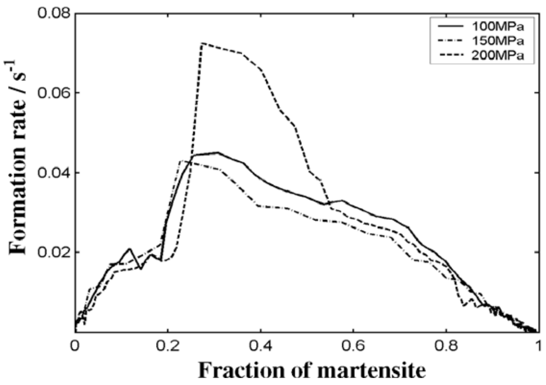

Fig. 6. The martensite formation rate as a function of martensite fraction of the T91 steel deformed with different applied compressive stresses at 440 °C.

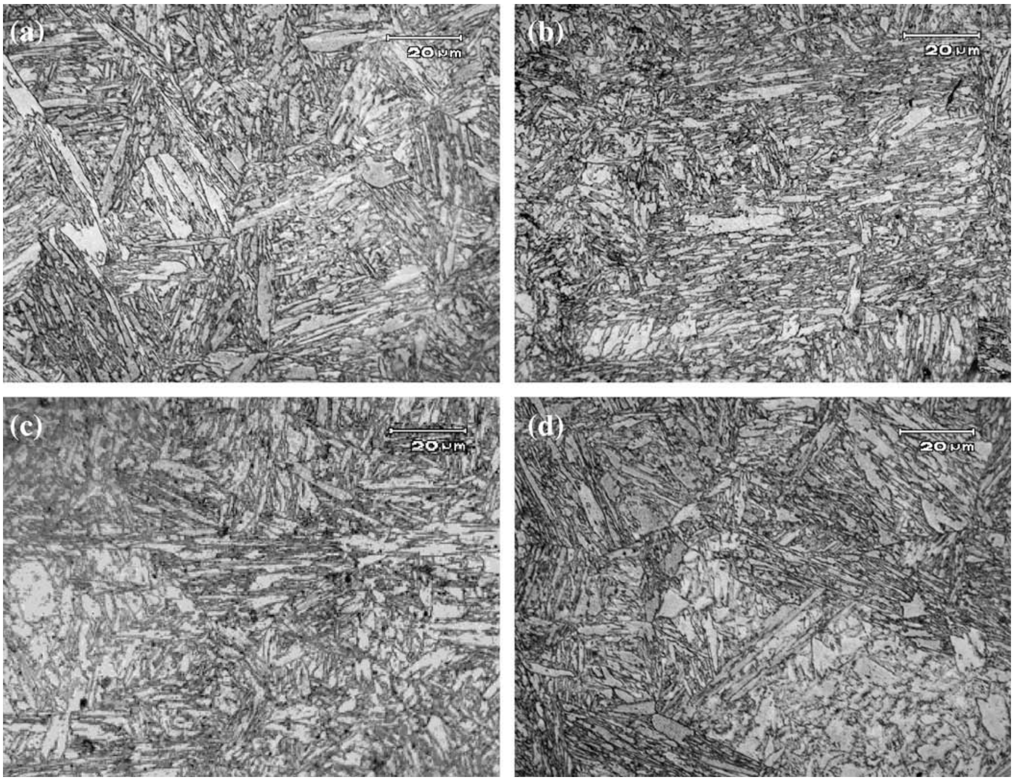

Fig. 7. Microstructures of the T91 steel subjected to different uniaxial compressive loads, (a) no stress, (b) 760 °C, 200 MPa, (c) 650 °C, 200 MPa and (d) 440 °C, 200 MPa.

with 200 MPa is higher than that of other two T91 samples. Therefore, 440 °C is the critical temperature for the stress-induced martensite transformation of the T91 steel, and the critical stress for the occurrence of the stress-induced martensite transformation at 440 °C is 200 MPa.

3.3. Typical microstructures

Different transformation mechanism leads to various microstructure. Fig. 7 shows the microstructures of the T91 steel deformed with different uniaxial compressive stresses. It can be seen from Fig. 7(a) that the austenite boundaries are still retained in the specimen without applied compressive stress, and most martensite nucleated at the boundaries and grew up into lath-shaped grains. The martensite laths are arrayed in bundles, which present the typical feature of the thermal-induced martensite. Fig. 7(b) and (c) show the microstructures of the T91 specimens with applied stress at 760 and 650 °C. The austenite boundaries are dimmer and martensite lathes become shorter and thinner, moreover, the martensite bundles exhibit no apparent phase relationship. Martensite lathes do not only form at the austenite boundaries but also within the grains. Fig. 7(d) shows the microstructure of the T91 specimen with applied compressive stress at 440 °C and the morphologies of the martensite are similar to the specimen without applied stress (see Fig. 7(a)).

Compared with microstructures of specimen without applied stress (see Fig. 7(d)), martensite lathes of in the specimens, which are deformed with 200 MPa at 760 and 650 °C (see Figs. 7(a)–(c)), have unconspicuous phase relationship and become refined. The formation of the refined martensite microstructure and abnormal morphologies may be caused by the following reasons: (i) austenite boundaries were destroyed after deformation and more lattice defects and distortions formed at the boundaries, hence the nucleation of martensite was promoted; (ii) the deformed austenite grains will inhibit the growth of the martensite grains. Thereby, when specimens are deformed in the temperature range of $M_S^\sigma \sim M_d$ (850, 760, 650 and 550 °C), the following phase transformation process at low temperature is controlled by the yielding strength of the austenite, which belongs to a strain-induced martensite transformation.

When the T91 specimen is deformed at 440 °C, its final microstructure turns into the stress-induced martensite for the formation process lies on the critical driving force. Its formation process has no difference with the thermal-induced martensite and hence they exhibit similar morphologies.

4. Conclusions

The effect of uniaxial compressive stress on martensite transformation of the T91 steel has been studied by high-resolution dilatometric measurement, it is found that:

(i) The compressive stress prompts the martensite transformation of the T91 steel when it is applied below 850 °C. Two different mechanisms could be recognized when it was minor-deformed in the temperature range of M_s and M_d: applying compressive stress at 850, 760, 650 and 550 °C, M_s increased with increasing of the applied stress. However, as the applied stress increased, M_s tends to decrease at the same temperature, which corresponds to a strain-induced martensite transformation mechanism.

(ii) 440 °C is the onset temperature (M_s^σ) and 200 MPa is the critical stress for the occurrence of the stress-induced martensite transformation. When applying stress in the range of M_s^σ and M_d, the tendency of the M_s point changed with the stress was slowed down as the deformation temperature decreases. The formed martensite lathes tends to be refined with irregular boundaries. However, when applying compressive stress in the range of M_s and M_s^σ, the morphologies of the transformed martensite are similar to thermally-activated one.

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