Cyclic stress induced phase transformation in super-bainitic microstructure*

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(Received 8 August 2016; revised manuscript received 22 November 2016; published online 18 January 2017)

The effects of cyclic stress loading on the microstructual evolution and tensile properties of a medium-carbon super-bainitic steel were investigated. Experimental results show that the cyclic stress can induce the carbon gathering in austenite and phase transformation from film-like retained austenite to twin martensite, which will obviously enhance the tensile strength and the product of tensile strength and ductility. The higher the bainitic transformation temperature, the lower the transformation rate of the retained austenite. The amount and thickness of the film-like retained austenite play an important role during the cyclic stress induced phase transformation.

Keywords: super-bainite, cyclic stress, retained austenite, phase transformation

PACS: 81.05.Bx, 62.20.–x, 81.30.–t **DOI:** 10.1088/1674-1056/26/3/038101

1. Introduction

Recently, the super-bainitic microstructure has been paid extensive attention because of its excellent combination of high strength and satisfactory toughness.[1–6] The high strength mainly depends on the ultra-fine carbide-free bainitic ferrite (BF) and many kinds of sub-structures within the BF, such as dislocation, grain boundary, phase boundary, etc.[7] Meanwhile, a certain amount of film-like retained austenite (RA) distributed between and within the BF laths makes the super-bainitic microstructure exhibit good ductility and toughness.[8,9] As a result, such sandwiched structure composed of BF and RA provides a space to further improve the mechanical properties of bainitic steels. However, from the thermo-dynamics analysis, the film-like RA in the super-bainitic microstructure is a metastable phase.[10–12] If a sufficient driving force of phase transformation is provided, the RA transformation may occur and a higher strength would be achieved. So in the present work, the super-bainite microstructures obtained from different heat treatments are subjected to the cyclic stress, and the impact of the cyclic stress loading on the microstructual evolution and tensile properties of 60Mn2SiCr medium-carbon super-bainitic steel is investigated. The conditions for the occurrence of stress induced phase transformation are also discussed.

2. Experiment

Chemical compositions of the 60Mn2SiCr bainitic steel are shown in Table 1. The high purity base materials of the steel were melted in a vacuum induction furnace and then hot rolled to square billets with a final rolling temperature of above 900 °C.

Table 1. Chemical compositions of the 60Mn2SiCr super-bainitic steel.

C	Si	Mn	Cr	S	P	Fe
0.58	1.54	1.87	0.87	0.015	0.004	Bal.

It is known that the identification of the martensite starting (Ms) temperature is useful for evaluating the heat treatment process, because the super-bainitic microstructure is obtained by the isothermal transformation at low temperatures that are slightly higher than the Ms temperature. In this work, the Ms temperature of the studied steel was determined by the Gleeble 1500D thermo-mechanical simulator with a cylindrical specimen of 4 mm diameter and 10 mm height. The specimen was heated at a rate of 10 °C/s to 900 °C and held for 8 min, and then cooled to the room temperature at a rate of 20 °C/s. According to the obtained thermal expansion curve, the Ms temperature was identified as 249 °C. On this base, two temperatures of 260 °C and 270 °C were selected for the isothermal transformation. Austenitization of the specimens was performed at 900 °C for 30 min. After that, they were immediately kept in a salt bath to preset the defined temperatures for 12 h, which was identified earlier as the sufficient time for

*Project supported by the National Natural Science Foundation of China (Grant Nos. 51171030 and 51604034), the Scientific and Technological Planning Project of Jilin Province, China (Grant No. 20150520030JH), and the Scientific and Technological Research Fund of Jilin Provincial Education Department during the Twelfth Five-year Plan Period, China (Grant No. 2015-95).

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the bainitic reaction, then followed by air cooling. The salt bath is composed of KNO_2 and $NaNO_3$ with 1:1 proportion.

Cyclic stress loading on the heat treated specimens at different temperatures was carried out on the electro-hydraulic servo static and dynamic fatigue testing machine (Type: EHF-UM100K2-040-0A) with the frequency of 1 Hz and the loading time of 72 h. The maximum cyclic stress is 600 MPa, about 50% of the yield strength of the steel. Figure 1 gives the dimension of the testing specimen.

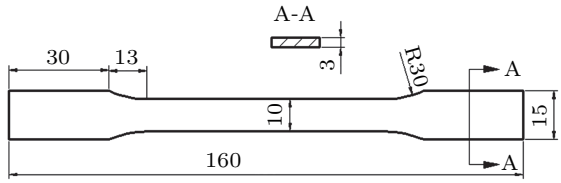


Fig. 1. Specimens used for cyclic stress loading.

The mechanical properties of the heat treated specimens before and after cyclic stress loading were measured by the electronic universal testing machine (Type: WDW-200). In order to reduce the experimental error, at least three tests were taken on each condition and the average value was reported. Besides, the standard deviations for the data in the same group were also calculated.

The optical (OM: Leica MDI3000 M) and scanning electron microscopes (SEM: SUPRA 40) were used for metallographic observations, where the specimens were mounted, ground, and polished according to the ASTM: E3-11, and then etched in the 4% nitric acid alcohol solution. A transmission electron microscope (TEM: JEM-2000EX) operated at 150 kV was used to examine the substructures and measure the thicknesses of the BF plates and filmy RA. TEM specimens were mechanically ground down to about 50 μm thick and then electro-polished at 40 V using a twin-jet unit. The electrolyte consisted of 5% perchloric acid and 95% alcohol solution.

To identify the phase constitutes in the steel, x-ray diffraction analysis (XRD: 2000/PC) was employed. The XRD specimens were scanned using Cu $K\alpha$ radiation in the range of 30°–100° at a rate of 2°/min. The amount of RA was quantitatively obtained by comparing the integrated intensities of the $(200)_\gamma$, $(220)_\gamma$, $(311)_\gamma$, and $(200)_\alpha$, $(211)_\alpha$ peaks.[13–15] The formula is[15]

$$f_\gamma = \frac{I_\gamma C_\alpha}{I_\gamma C_\alpha + I_\alpha C_\gamma}, \tag{1}$$

where I_γ and I_α are the intensities of the γ and α phases, respectively, and C_γ and C_α are the proportionality constants. The carbon concentrations in the retained austenite were calculated from the lattice parameters obtained from the corresponding diffraction peaks as follows:[14]

$$C_\gamma = \frac{a_\gamma - 3.578}{0.033}, \tag{2}$$

where a_γ is the lattice parameter of RA (10^{-10} m). It is noted that one sample and the same testing position marked previously were analyzed by XRD to avoid the fluctuation of the calculated results before and after cyclic stress loading caused by sample difference. Moreover, the XRD patterns of three samples for the same heat treatment were measured, which aims to ensure the accuracy and reliability of the result.

3. Results and discussion

Figure 2 shows the microstructures of the specimens isothermally treated at 260 °C and 270 °C, respectively. It can be seen that the typical super-bainitic microstructures consisting of BF laths and RA films are obtained in both transformation processes. Such retained austenite not only distributes between the BF laths but also within the BF laths, which is considered to be the main contributor to the ductility and toughness of the super-bainitic steel.[16–18] Furthermore, a small amount of blocky RA can be observed in the specimen isothermally treated at 270 °C (Fig. 2(d)). It indicates that the high temperature for the bainitic reaction is unfavorable for the refinement of the super-bainite microstructure. Figure 3 shows the corresponding TEM micrographs of the microstructures subjected to different heat treatments. Free carbides but high

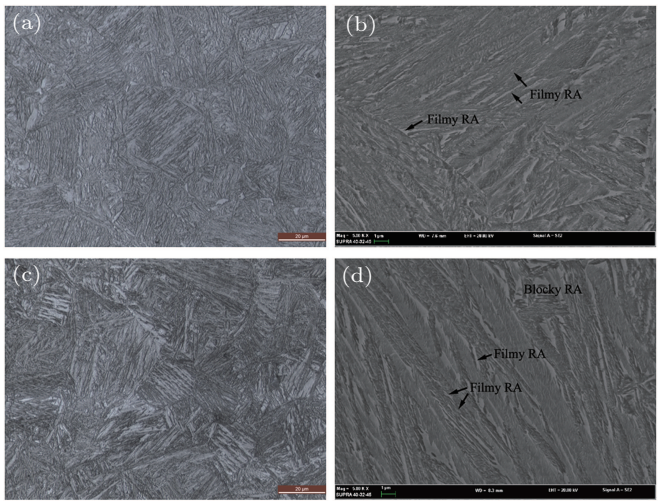


Fig. 2. (color online) Microstructures of the specimens isothermally transformed at (a), (b) 260 °C for 12 h, and (c), (d) 270 °C for 12 h.

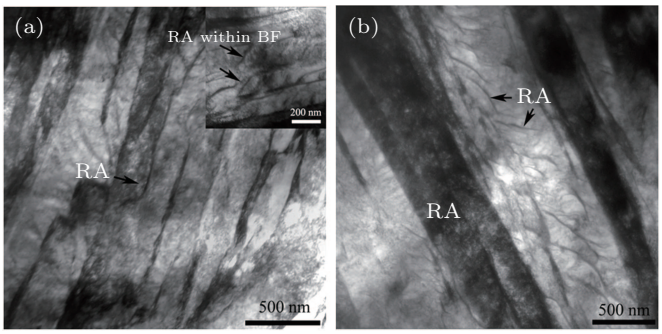


Fig. 3. (color online) TEM micrographs of the microstructures after being isothermally heat treated at (a) 260 °C for 12 h and (b) 270 °C for 12 h.

densities of dislocations can be observed in both the BF and the surrounding RA, which implies the mechanism of shear transformation. From Fig. 3(a), at the transformation temperature of 260 °C the substructures of the steel exhibit fine sheaves of super-bainite composed of BF and RA, which is supported by the thickness measurements in Figs. 4(a) and 4(b). The weighted average thicknesses of the BF lath and RA film, 171.80 nm and 81.17 nm, respectively, are obtained at 260 °C for 12 h. It is also found that the RA formed within the BF laths has much finer scales (about 40 nm in thickness) than that of the RA formed between the BF laths and accounts for a large percentage. Generally, the nano-films of RA within the BF laths are more beneficial to improve the toughness and delayed fracture resistance. However, with increasing isothermal temperature, such as 270 °C, the sheaves of the super-bainite are coarsened, as shown in Fig. 3(b). The weighted average thicknesses of the BF lath and RA film increase to 193.03 nm and 93. 51 nm, respectively, as shown in Figs. 4(c) and 4(d).

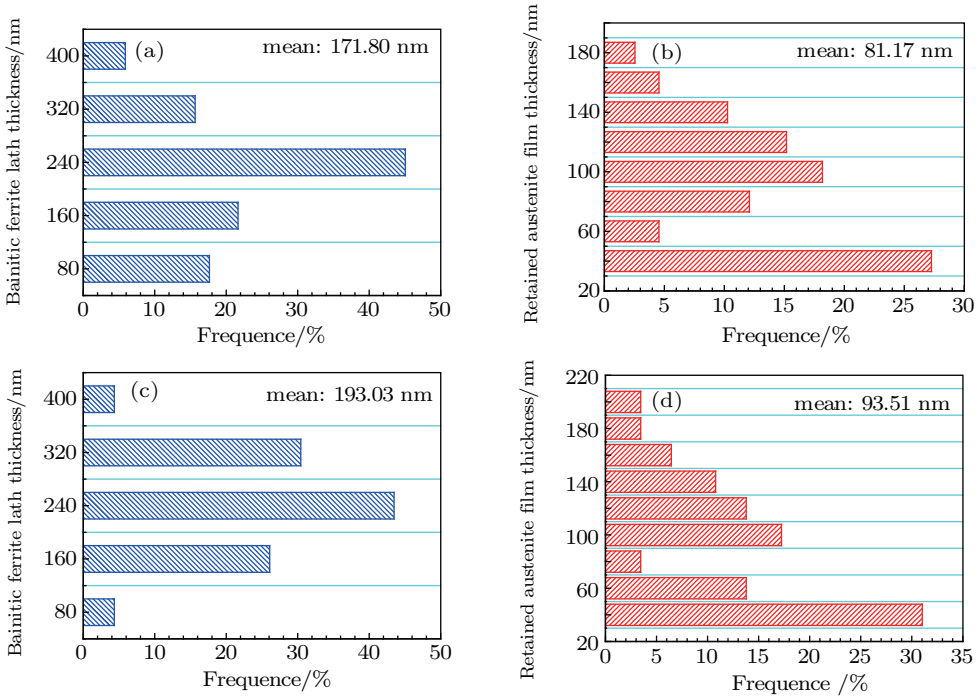


Fig. 4. (color online) BF lath and RA film thickness distributions in the microstructures obtained at (a), (b) 260 °C for 12 h, and (c), (d) 270 °C for 12 h.

The above isothermally treated specimens were subjected to cyclic low stress loading for 72 h, after which the tensile properties of the loaded specimens were tested. For a comparative study, the unloaded specimens (only heat treatment) were also tested. Figure 5 shows the typical tensile engineering stress–strain curves of the heat treated steels before and after cyclic stress loading. It is seen that, without stress loading, the isothermally treated specimen at 260 °C has obviously lower yield strength and higher elongation. More importantly, the steel exhibits a long homogeneous deformation stage. It indicates that the bainitic transformation at lower temperature can improve the plasticity because of the fine sheaves of the super-bainite. Table 2 shows the corresponding performance indicators. It is obvious that the high tensile strength and product of tensile strength and ductility are acquired in specimens treated at 260 °C and 270 °C after cyclic stress loading. For the specimen obtained at 270 °C, the ultimate strength increases from 1814.88±21.25 MPa to 1899.80±37.69 MPa, and the elongation increases from 7.72±0.31% to 8.97±0.36%. Although the elongation is slightly decreased after cyclic stress loading in the specimen isothermally treated at 260 °C, the product of tensile strength and ductility, a combination index of strength and toughness, is enhanced remarkably. As a result, it implies that the cyclic stress loading after heat treatment will further increase the strength and plasticity of the super-bainitic steel.

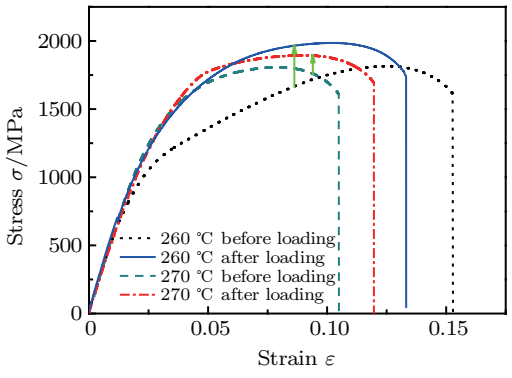


Fig. 5. (color online) Tensile stress–strain curves of the heat treated specimen before and after stress loading.

Table 2. Mechanical properties of the heat treated specimen before and after cyclic stress loading.

Condition		$\sigma_{0.2}$/MPa	σ_b/MPa	δ/%	U/MPa·%
260 °C/12 h	unloading	1125.67±35.51	1816.80±30.17	11.06±0.41	20093.81±661.70
	loading	1497.59±29.94	1995.90±25.34	10.32±0.37	20597.69±809.37
270 °C/12 h	unloading	1540.37±45.57	1814.88±21.25	7.72±0.31	14010.87±505.52
	loading	1693.10±41.10	1899.80±37.69	8.97±0.36	17041.20±457.23

In fact, the increase of tensile property through applying cyclic stress prior to deformation is dominated by the microstructural change. Figure 6 shows the XRD patterns of the heat treated specimens before and after cyclic stress loading. It is seen that, whether subjecting to the cyclic stress loading or not, the microstructures are composed of bcc and fcc structured phases, and no carbides are detected. The TEM micrographs of the heat treated specimens after cyclic stress loading are shown in Fig. 7, where reveals the substructure of the film-like RA and BF lath morphology. Note that, the twin crystallite martensite is observed in both heat treatment conditions. Meanwhile, after cyclic stress loading, the amounts of RA in the specimens with 260 °C and 270 °C isothermal transformation decrease to 5.6% and 6.7%, respectively, as shown in Table 3. This indicates that the phase transformation of RA→M has taken place during cyclic stress loading.

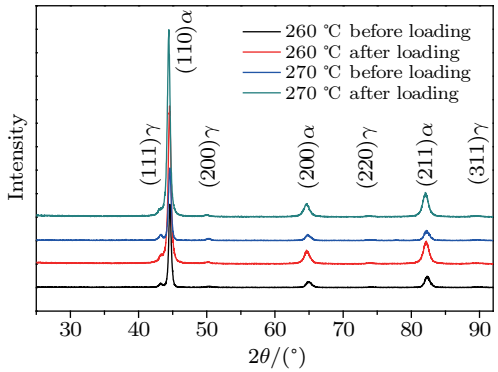


Fig. 6. (color online) XRD patterns of the heat treated specimens before and after cyclic stress loading.

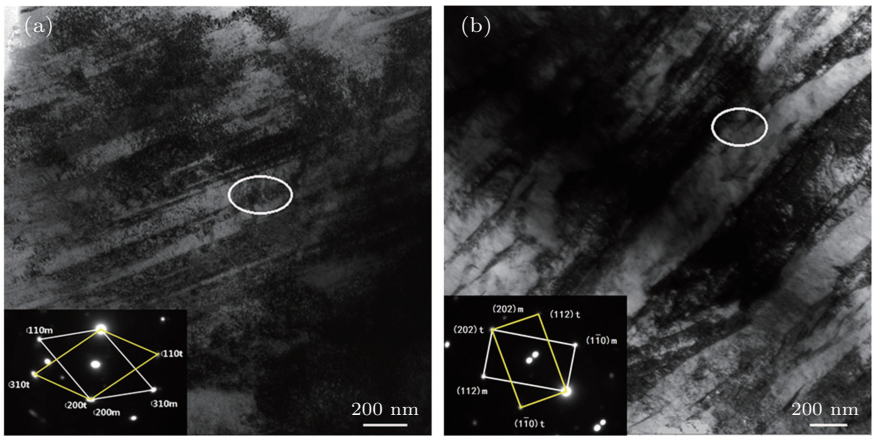


Fig. 7. (color online) TEM micrographs of the heat treated specimens after cyclic stress loading: (a) and (c) 260 °C/12 h; (b) and (d) 270 °C/12 h.

It is well known that the film-like RA with nano-scale can improve the plasticity of the super-bainitic steel because it can prevent crack propagation through martensitic transformation during tensile deformation.[19] However, in this study, the stress induced martensitic transformation occurs before the plastic deformation, which means that the starting microstructure for tensile involves fine BF plates and film-like RA and a small amount of twin crystallite martensite. With the formation of the twin crystallite martensite, the original excess carbon in RA will be easily rejected into the nearby austenite region due to its much greater solubility comparing to the BF.[20] Therefore, the RA after cyclic stress loading becomes more stabilized because of the enrichment of carbon. The amount of unstable austenite begins to decrease. From the XRD results in Table 3, the carbon concentration in RA is indeed increased after stress loading. For example, after the cyclic stress loading, the carbon concentration in RA is increased to 1.425% for 260 °C and 1.342% for 270 °C, respectively. These concentrations are much higher than the average carbon content of the steel, which leads to the RA being more stable and retained to room temperature. Generally, the stable RA can increase the strengthening effect caused by phase transformation. It is noted that, the amount of RA increases with the increment of the isothermal transformation temperature. This is because the lower the transformation temperature, the greater the phase transformation driving force and, hence, the lower the RA content is obtained.[8,21] However, the transformation rate of RA is obviously reduced from 34.89% to 27.96% as the

transformation temperature increases from 260 °C to 270 °C. This illustrates that the blocky RA and coarsened film-like RA do not easily lead to stress induced martensite transformation. Accordingly, the cyclic stress loading prior to plastic deformation provides the opportunity for the improvement of the integrated mechanical properties due to the formation of twin crystallite martensite and more stable RA film with high carbon concentration.

Table 3. RA and C_γ in the heat treated specimens before and after cyclic stress loading.

Condition		RA/%	C_γ/%	Transformation rate/%
260 °C/12 h	unloading	8.6	1.344	34.89
	loading	5.6	1.425	
270 °C/12 h	unloading	9.3	1.276	27.96
	loading	6.7	1.342	

4. Conclusion

The effects of cyclic stress loading on the microstructural changes and tensile properties of 60Mn2SiCr super-bainitic steel were studied in this work. The cyclic stress prior to plastic deformation can induce the phase transformation from film-like RA to twin martensite, which leads to the microstructure changes into a mixture of fine BF plates, more stable film-like RA, and a small amount of twin martensite. The high transformation rate of RA after cyclic stress loading is found in the super-bainitic microstructure obtained at low isothermal temperature because of its low amount of blocky RA and much finer RA films. The new microstructure caused by the cyclic stress loading can obviously improve the integrated mechanical properties, especially the ultimate strength and the product of tensile strength and ductility. The strength increment is increased as the transformation rate of RA increases.

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