Transformation Behavior of Bainite during Two-step Isothermal Process in an Ultrafine Bainite Steel

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Four different isothermal treatment processes were designed to investigate the effect of isothermal treatment parameters on the comprehensive property in an ultrafine bainite steel. Four different isothermal treatment processes, including two kinds of one-step bainite transformation procedures at temperature above MS and below M S, respectively, and two kinds of two-step isothermal processes, were employed in this study. The results indicate that compared with the one-step bainite transformation at temperature above Ms, the comprehensive property of ultrafine bainite steel was not improved by austempering at temperature below Ms. However, it was improved by the two-step isothermal process. In addition, at the condition that the total transformation time of multi-step isothermal process was the same, the product of tensile strength and elongation (PSE) of sample increased with the decrease of transformation time at the first stage. At the same time, the transformation kinetics of the second stage transformation was significantly accelerated with the decrease of transformation time at the first stage.

KEY WORDS: transformation kinetics; ultrafine bainite; multi-step process; Ms; mechanical properties.

1. Introduction

Since nanostructured bainitic microstructure with the excellent balance of strength and toughness is reported by Bhadeshia et al.,1–4) the approach of reducing the scale of the microstructure has been a hot topic. It is known that the size of bainite plates is determined by the chemical composition and heat treatment route.5–8)

In industrial production, in order to obtain the microstructure of ultrafine bainite plate and film-like retain austenite (RA), it may be necessary to handle the bainitic transformation at a very low temperature. In general, decreasing the bainite starting temperature (Bs) and martensite starting temperature (Ms) requires a high carbon content,9) but the high carbon content will significantly deteriorate the welding property and toughness of steels. Therefore, some researchers10–18) adjusted the heat treatment technology of bainite transformation to achieve the ultrafine bainitic microstructure and improve the mechanical properties. Among them, one effective method is the multi-step austempering. Hase et al.14) and Avishan et al.15) adopted a two-step heat treatment to obtain the bimodal size-distribution of bainite plates in an ultrafine bainitic steel. Coarse bainite plates were produced by austempering at a high temperature, followed by austempering at a lower temperature. It was helpful to decrease blocky RA, and achieve the microstructure of ultrafine bainite plates and film-like RA. The large blocky RA will be easily transformed into martensite when is loaded by an external strain, which is harmful to the toughness of steels. Thereby, the tensile properties and fracture toughness are significantly improved as a result of diminishing the blocky RA and increasing the amount of ultrafine bainite plates. Similarly, both Soliman et al.17) and Wang et al.18) demonstrated that there was a significant improvement in comprehensive properties of medium carbon (0.30 wt.%) and low carbon (0.26 wt.%) bainitic steels by inducing blocky RA to finer bainite through multi-step bainite transformation. So far, it has been proved that the second generation of bainite is formed at temperatures lower than the Ms of bulk alloy but higher than the Ms of partially untransformed austenite. The low temperature is the key point to obtain the ultrafine bainite microstructure.

To summarize, previous researchers have clarified that the mechanical properties of bainite steels can be improved using the multi-step austempering treatment by decreasing the size of bainite plates and controlling the morphology of RA from blocky to film-like. However, some of them14,15) utilized two-step process that the second step bainite transformation restarts immediately when the first step bainite transformation basically stops, whereas the others16–18) used such treatment scheme that the second bainite transformation restarts when the first step transformation completes only about 50% of the maximum volume fraction of bainite phase. They didn’t compare the difference of effect of the volume fraction of the bainite phase transformed in the first
2. Materials and Experimental Procedures

2.1. Materials

The tested steel was refined using a 50 kg laboratory-scale vacuum furnace followed by hot-rolled to 12 mm plates and then air-cooled to room temperature. The chemical composition of the tested steel is presented in Table 1. Manganese (Mn) and chromium (Cr) are added to enhance the stability and hardenability of austenite.\textsuperscript{8,19} Molybdenum (Mo) can improve temper-embrittlement phenomena caused by inevitable impurities.\textsuperscript{20,21} High silicon (Si) and aluminum (Al) are added to hinder the formation of carbide.\textsuperscript{22} Moreover, Al can also accelerate the kinetics of bainite transformation.\textsuperscript{8} The $B_\text{s}$ and $M_s$ for the tested steel were calculated to be 520°C and 371°C, respectively, by the software of MUCG83.\textsuperscript{23} The software of MUCG83 is a powerful suite of software for modelling of the thermodynamics and kinetics of solid-state transformations in steels. It can be used to calculate not only the starting temperatures of bainite and martensite based on the thermodynamic calculation, but also the driving force for diffusionless transformation, T-zero carbon concentration and the time-transformation-temperature (TTT) diagram.\textsuperscript{24} It is developed by Mathew Peet and H.K.D.H. Bhadeshia, Department of Materials Science and Metallurgy, University of Cambridge, and it is available from the Materials Algorithm Project website (MAP).\textsuperscript{25}

2.2. Thermal Simulation Experiments

The cylindrical samples with a diameter of 6 mm and a length of 100 mm were prepared for the dilatometric experiments. Thermal simulation experiments were performed on a Gleeble-3500 simulator. The dilatations along the diametrical direction were recorded by the dilatometer equipped on simulator. The diameter of 6 mm was used to minimize the temperature gradient along diameter direction. The copper has an excellent thermal conductivity and copper clamp was used to minimize the temperature gradient in length direction.

Figure 1 shows the heat treatment procedures of samples. The samples were heated to 1 000°C at a rate of 5°C/s and kept at the temperature for 15 min to obtain the full austenitic structure. After austenitization, samples 1 and 2 were treated by one-step bainite transformation route and respectively cooled down to 430°C (above $M_s$) and 330°C (below $M_s$) with a cooling rate of 30°C/s, and then held at the corresponding temperature for 30 min for bainite precipitation as shown in red line in Fig. 1. Samples 3 and 4 were treated by two-step route. Sample 3 was austempering at 430°C (above $M_s$) for 2 min, and then was immediately cooled down to 330°C (lower than the $M_s$ of bulk alloy but higher than the $M_s$ of partially untransformed austenite) to restart the bainite transformation where it was kept for 28 min. For sample 4 the second step transformation restarts after the first step transformation proceeded for 4 min, and then was immediately cooled down to 330°C where it was kept for 26 min. It should be noted that when the sample was isothermally transformed at 430°C, only bainite transformation happened because the temperature of 430°C was higher than $M_s$. Moreover, during the holding process, the temperature was held at 430±1°C, the dilatation data during the holding process represented the amount of bainite transformation. Bainite transformation is an incomplete transformation.\textsuperscript{26–28} The volume fraction of bainite transformation can be determined by the microscopic observation using the method proposed in Ref.\textsuperscript{29} or the dilatation method. In the present study, the relative volume fraction of bainite was defined as the proportion of instantaneous volume fraction to the maximum volume fraction ($f(t)/f_{\text{max}}$), which equaled to the proportion of instantaneous dilatation to the maximum dilatation in Fig. 2(b). Figure 2(b) shows the maximum volume fraction (amount) of bainite transformation at 430°C. It can be seen that the relative volume fraction of the bainite transformation in the first process was about 50% ($f(t)/f_{\text{max}}$), when sample 3 was transformed for 2 min at first process. On the other hand, when sample 4 was transformed for 4 min at first process, the relative volume fraction of the bainite transformation in the first process was about 95%; so that bainite transformation in first step basically stopped.

![Fig. 1. The heat treatment procedure.](image_url)
in 4 min.

2.3. Examinations

After thermal simulation experiments, the specimens were mechanically polished and etched with a 4% nitral solution for microstructure examination. The microstructure morphology was observed by a Nova 400 Nano field emission scanning electron microscope (FE-SEM). X-ray diffraction (XRD) measurements were carried out to determine the volume fraction of retained austenite (RA) on an Empyrean diffractometer, using unfiltered Co Kα radiation and operating at 35 kV and 50 mA. The step size and counting time were 0.0263° and 77.265 s, respectively. In addition, tensile tests at room temperature were conducted on a UTM-4503 electronic universal tensile tester with a cross-head speed of 1 mm/min. It should be pointed out that the sub-size samples were used in tensile tests because of the dimension limits of the thermal simulation samples. The tensile tests were performed without extensometer, which may result in the possible overestimate of elongation. Even so, the elongations between different samples are comparable due to the same specimen dimension. Four tensile tests were repeated for each heat treatment route and the average values were given.

3. Results and Discussion

3.1. Dilatation

3.1.1. One-step Bainite Transformation

The purpose of the one-step heat treatment at temperature above Ms is to compare the transformation behavior and mechanical property with the one-step heat treatment at temperature below Ms and the two-step bainite transformation route. Figure 2(a) presents the curve of dilatation versus temperature during the whole heat treatment for sample 1 austempered at 430°C for bainite transformation. It is a straight line during cooling process from 1000°C to 430°C at a rate of 30°C/s, indicating that no any high temperature form before the isothermal temperature of 430°C. Moreover, a deviation in linearity of the dilatation curve initiating at the point M (221°C) in Fig. 2(a) can be observed during the final cooling to ambient temperature, which represents the formation of fresh martensite (FM). It also indicates that the Ms of the retained undeformed austenite is about 221°C, which is lower than 330°C. For ensuring that the second bainite transformation restarts at temperature higher than the Ms of the untransformed austenite, the second isothermal transformation temperature was designed as 330°C.

The dilatation versus holding time of sample 1 isothermally transformed at 430°C is presented in Fig. 2(b). The obvious increase in dilatation during holding time is caused by the bainite transformation. As mentioned above, bainite transformation shows incomplete transformation phenomenon due to T0 limit.26-28 Therefore, although bainite transformation basically stops after about 4 min at 430°C, it doesn’t mean that all austenite are transformed into bainite.

In addition, Fig. 3 shows the dilatation and temperature curves of sample 2 austempered at 330°C. In Fig. 3(a), the dilatation begins to increase during the cooling process before the temperature reached the isothermal transformation temperature of 330°C. When the temperature is cooled to the isothermal temperature, the dilatation curves show an obvious inflexion point (point A in Figs. 3(a) and 3(c)). The point A is corresponding to the beginning of isothermal holding at 330°C. Figure 3(b) gives the measured and programmed temperature versus time of sample 2 during the whole process. It is noted that there is a temperature rebound from 330°C to 370°C due to the temperature gradient and the heat transmission from interior to surface in the sample caused by the high cooling rate of 30°C/s. Moreover, Fig. 3(c) shows the curves of dilatation versus holding time. It can be observed that the slope of dilatation curve before point A in Fig. 3(c) is much larger than that during the isothermal period (after point A). According to the previous result reported in Ref.30 it is reasonable to infer that the rapid increase in dilatation before austempering is caused by the formation of preformed athermal martensite (AM). Therefore, the zero point (point Z) in Fig. 3(c) is corresponding to the starting point of AM transformation. It can be seen from Fig. 3(c) that the dilatation of AM is 0.0123 mm, whereas the dilatation caused by the bainite transformation is 0.0114 mm (from point A to the maximum value).

3.1.2. Two-step Bainite Transformation

Figure 4 shows the recorded dilatation versus holding time curves of samples 3 and 4 during the two-step bainite transformation. It can be observed that the bainite amount at the first step transformation in sample 3 (point P in Fig.
4(a)) reaches about 50% of the maximum volume fraction of the bainite phase transformed in the first process (sample 1 in Fig. 2(b)), followed by the restart of the second step transformation. However, for sample 4 the second step bainite transformation at 330°C restarts after the dilatation of the first step reaches about 95% of the maximum volume fraction of the bainite phase transformed in the first process (sample 1 in Fig. 2(b)) as shown by point Q in Fig. 4(b), that is, the second step transformation of sample 4 begins after the first step transformation stops basically. The total

Fig. 3. The dilation of sample 2 austempered at 330°C: (a) the dilatations versus temperature curve; (b) the temperature versus time during the whole process; and (c) the dilatations versus holding time.

Fig. 4. The dilatations versus holding time during the two-step bainite transformation: (a) sample 3 austempered at 430°C for 2 min followed by 330°C for 28 min; (b) sample 4 austempered 430°C for 4 min followed by 330°C for 26 min; and (c) the dilatation in the second step of samples 3 and 4.
dilatation of sample 3 in the first step and the second step is 0.0262 mm, and it is 0.0251 mm in sample 4. Figure 4(c) shows the dilatation of samples 3 and 4 in the second step bainite transformation. The dilatations during the second step isothermal transformation in samples 3 and 4 are 0.0135 mm and 0.0042 mm, respectively. Although the total amounts of bainite transformation of samples 3 and 4 have no significant difference, the dilatation (0.0135 mm) of sample 3 in the second step transformation is far more than that (0.0042 mm) of sample 4. The bainite plates formed at lower temperature are finer. It means that the more finer bainite plates are obtained in sample 3. In addition, compared with the dilatation amount caused by bainite transformation in sample 2 austempered at 330°C (0.0114 mm, Fig. (3)), the dilatation amounts in samples 3 (0.0262 mm) and 4 (0.0251 mm) are obviously larger, indicating that the bainite transformation is greatly promoted with the two-step isothermal treatment.

The dilatation change rates (representing the transformation rate) versus time of samples 3 and 4 during the second step bainite transformation are plotted in Fig. 5. During the second step bainite transformation, the maximum of transformation rate of sample 3 is 0.22 μm/s, while it is about 0.02 μm/s for sample 4. The former is more than ten times as that of the latter, indicating that the transformation kinetics of the second step transformation in sample 3 is obviously accelerated. This is because the bainite transformation process is accompanied by carbon ejection. There is more bainite amount at the first step in sample 4 than that in sample 3, which leads to the higher carbon content in untransformed austenite. So the nucleation and growth of bainite are inhibited because of the higher chemical stability. Therefore, compared with sample 4, the transformation kinetics of the second step transformation in sample 3 is obviously accelerated.

### 3.2. Microstructure and XRD Results

The SEM micrographs treated by one-step process are presented in Fig. 6. For sample 1, the microstructure mainly consists of fresh martensite (FM), bainite and the blocky RA as shown by arrows in Fig. 6(a), while few FM and the blocky RA appears in sample 2, but AM, bainite and film-like RA appear in Fig. 6(b). The presence of AM in Fig. 6(b) confirms that it is AM that leads to the rapid increase in the initial stage of dilatation in Fig. 3(c). Figures 6(c) and 6(d) give the higher magnification SEM micrographs of samples 1 and 2. According to the microstructures in Figs. 6(c) and 6(d), the size of bainite is measured by the
software of Image-Pro Plus. Three high magnification SEM micrographs of each sample are used. The size of bainite in sample 1 is calculated to be 757±23.9 nm, whereas it is 468±26.5 nm in sample 2. Therefore, the bainite plates become finer when the sample is treated below Ms. Moreover, the amount of bainite transformation decreases due to the formation of large amount of AM (Fig. 3(b)).

**Figure 7** presents the SEM micrographs of samples 3 and 4 treated by the two-step transformation process. The microstructures of samples 3 and 4 mainly contain bainite and film-like RA. It is obvious that compared the microstructure of sample 1, the volume fraction of the blocky martensite drastically decreases, and the overall microstructure is obviously refined after two-step isothermal process. In Figs. 7(a) and 7(b), bainite can be classified to two types according to the different bainite morphology, which is marked B1 and B2. Similarly, according to the SEM microstructures, the size of B1 can be calculated to be 783±29.6 nm. It is very close to the size of bainite plates (757 nm) in sample 1 austempered at 430°C with one-step process. Therefore, it is reasonable to infer that B1 is formed in the first step of the two-step isothermal process. In addition, it can be seen from Figs. 7(a) and 7(b) that the size of B2 is obviously smaller than that of B1. It is known that the lower the bainite transformation temperature is, the finer bainite plates are. Bhadesia *et al.* reported that one of the main factors on plate thickness is the strength of the austenite. The thickness of bainite plates can be refined by austenite with higher strength. In the present case, B2 is finer than B1, so B2 should be formed in the second step of the two-step isothermal process from the austenite with higher carbon content at lower transformation temperature. Therefore B1 and B2 could be identified to be formed in the first step and the second step process, respectively. In addition, it can be observed from Figs. 7(a) and 7(b) that there is more amount of fine bainite in sample 3 than in sample 4. It is consistent with the result of dilatation in Fig. 4(c). The more untransformed austenite after the first stage bainite transformation in sample 3 results in more ultrafine bainite plates and film-like RA at lower temperature.

Figure 7(c) shows the higher magnification SEM micrographs of B2 in sample 3, while Fig. 7(d) shows the higher magnification SEM micrographs of B2 in sample 4. The high magnification SEM micrographs are used to measure the size of B2. It is clear that there is no carbide beside bainite plates due to the adequate addition of Si, so this kind of bainite is also named as the carbide-free bainite. The bainite plate thickness “n” was determined by measuring the mean linear intercepts “L,” in the direction normal to the plate length. The thickness n is related to L, by the relationship \( n = 2L/\pi \). The value of n of B2 in sample 3 is calculated to be 386±32.7 nm, whereas it is 242±18.3 nm in sample 4. More bainite formed in sample 4 than that in sample 3 during the first stage transformation, leading to the less untransformed austenite but with higher carbon content and higher strength in sample 4. According to the results reported by Bhadesia *et al.*, the thickness of bainite plates mainly depends on the strength of austenite. The higher carbon content results in the higher strength of austenite, so the thickness of bainite plates can be refined in the carbon enriched austenite. Therefore, the bainite plate of B2 in sample 4 is thinner than that in sample 3.

**Figure 8** shows an example of diffraction pattern of sample 1 depicted by XRD experiments. The integrated intensities and the angles of diffraction peaks are accurately determined by the software of HighScore Plus based on the
the carbon contents in RA (C_{RA}) were calculated based on the integrated intensities of (200) \( \alpha \), (211) \( \alpha \), (200) \( \gamma \), and (220) \( \gamma \) diffraction peaks, and the angles of (200) and (220) austenite peaks, respectively, according to the method proposed in Ref.\textsuperscript{32,33}. The calculated results are given in Table 2. The V_{RA} of samples 3 and 4 treated by two-step isothermal route have the higher volume fraction of RA (V_{RA}) and carbon content. Therefore, less residual austenite transforms into martensite during the cooling process.

Table 2. The volume fraction of RA and the carbon content.

| Samples | V_{RA} (%) | C_{RA} (wt.%) |
|---------|------------|---------------|
| 1       | 7.86       | 0.76          |
| 2       | 7.51       | 0.82          |
| 3       | 12.70      | 0.90          |
| 4       | 11.36      | 0.96          |

3.3. Mechanical Property

The tensile results of specimens treated by different routes are given in Table 3. For one-step isothermal process, the tensile strength (TS), yield strength (YS) and elongation (TE) of sample 1 austempered at 430°C are respectively 1123 \pm 24 MPa, 865 \pm 41 MPa, and 13.9 \pm 0.80%, while they are 933 \pm 31 MPa, 748 \pm 47 MPa, and 15.0 \pm 1.12% for sample 2 austempered at 330°C, respectively. When the samples are austempered below MS \( \sim 658 \) MPa and 15.1% for sample 2 austempered at 330°C, the tensile strength (TS), yield strength (YS) and elongation (TE) of sample 1 austempered at 430°C are respectively 1199 MPa, 715 MPa and 13.9%, while they are 927 MPa, 715 MPa and 13.9%, while they are 1123 \pm 24 MPa, 865 \pm 41 MPa, and 13.9 \pm 0.80%, respectively, according to the method proposed in Ref.\textsuperscript{32,33}. The calculated results are given in Table 2. The V_{RA} of samples 3 and 4 treated by two-step isothermal process have the higher volume fraction of RA (V_{RA}) and carbon content. Therefore, less residual austenite transforms into martensite during the cooling process.

Table 3. The tensile results of samples treated by different heat treatments.

| Samples | TS (MPa) | YS (MPa) | TE (%) | PSE (GPa%) |
|---------|----------|----------|--------|------------|
| 1       | 1123 \pm 24 | 865 \pm 41 | 13.9 \pm 0.80 | 15.61 \pm 1.23 |
| 2       | 933 \pm 31  | 748 \pm 47  | 15.0 \pm 1.12 | 13.99 \pm 1.80 |
| 3       | 1086 \pm 17 | 806 \pm 27  | 18.2 \pm 0.34 | 19.76 \pm 0.96 |
| 4       | 1031 \pm 22 | 786 \pm 29  | 17.5 \pm 0.52 | 18.04 \pm 0.85 |

Compared with one-step isothermal process, samples 3 and 4 treated by two-step isothermal route have the higher PSE. The higher PSE means the better comprehensive properties. It is mainly attributed to the finer structure and more amount of film-like RA. It has been reported by some researchers\textsuperscript{34,35} that the comprehensive property can be improved by refining bainite microstructure. Furthermore, more amount of film-like RA can improve the mechanical property in low carbon bainite steel by the effect of transformation induced plasticity (TRIP).\textsuperscript{36,37}

In addition, for two-step isothermal process, it can be observed from Table 3 that the PSE of sample 3 is higher than that of sample 4. Although the thickness of B2 in sample 4 (242 nm) is smaller than that in sample 3 (386 nm), the strength and elongation of sample 4 is lower than sample 3. It is mainly because of more finer bainite and film-like RA in sample 3 than that in sample 4. Bainite plates with the size smaller than 1 \( \mu m \) is termed as ultrafine bainite in the present study. It is observed from Fig. 4(c) that the increment in dilatation caused by the refined B2 during the second step isothermal transformation in sample 3 are 0.0135 mm, while it is 0.0042 mm in sample 4. As is aforementioned, the total amounts of bainite transformation of samples 3 and 4 have no significant difference, but the dilatation of sample 3 (0.0135 mm) in the second step transformation is far more than that of sample 4 (0.0042 mm). It means that the more finer bainite plates are obtained in sample 3. The more finer bainite plates results in the better comprehensive property. In addition, the volume fraction of RA in sample 3 is higher than that in sample 4 (Table 2).
The film-like RA can significantly improve the mechanical property by the TRIP effect. Therefore, the better comprehensive property is obtained in sample 3.

4. Conclusions

Four different isothermal treatment routes, i.e., two kinds of one-step bainite transformations treatments at the temperature of above $M_S$ and below $M_S$, respectively, and two kinds of two-step bainite transformation treatments were designed to investigate the effect of two-step treatment for bainite transformation on the microstructure and property in an ultrafine bainite steel. The following main conclusions can be drawn:

1. At the condition that the total transformation time of two-step isothermal process is the same, the comprehensive property of ultrafine bainite steels is improved with the decrease of the first step transformation time.

2. For two-step isothermal process, the transformation kinetic of the second step is obviously accelerated with the decrease of transformation time in the first step.

3. Compared with the conventional one-step bainite transformation above $M_S$, the comprehensive property of ultrafine bainite steel is not improved by austempering below $M_S$.

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