

University of Wollongong

Research Online

Faculty of Engineering and Information
Sciences - Papers: Part B

Faculty of Engineering and Information
Sciences

2019

Effect of austenisation temperature on bainite transformation below martensite starting temperature

Junyu Tian

Wuhan University of Science and Technology

Guang Xu

Wuhan University of Science and Technology

Zhengyi Jiang

University of Wollongong, jiang@uow.edu.au

Qing Yuan

Wuhan University of Science and Technology

Guanghai Chen

Wuhan University of Science and Technology

See next page for additional authors

Follow this and additional works at: <https://ro.uow.edu.au/eispapers1>



Part of the [Engineering Commons](#), and the [Science and Technology Studies Commons](#)

Recommended Citation

Tian, Junyu; Xu, Guang; Jiang, Zhengyi; Yuan, Qing; Chen, Guanghui; and Hu, Haijiang, "Effect of austenisation temperature on bainite transformation below martensite starting temperature" (2019).

Faculty of Engineering and Information Sciences - Papers: Part B. 3126.

<https://ro.uow.edu.au/eispapers1/3126>

Research Online is the open access institutional repository for the University of Wollongong. For further information contact the UOW Library: research-pubs@uow.edu.au

Effect of austenisation temperature on bainite transformation below martensite starting temperature

Abstract

Effects of austenisation temperature on martensite and bainite transformation behaviour, microstructure, and mechanical properties of a bainitic steel austempered below martensite starting temperature were investigated in this study. Results show that the amount of athermal martensite gradually increased with the increase of austenisation temperature, whereas the amounts of bainite and retained austenite initially increased and then decreased, resulting in the trend of the first increase and then decrease in the product of tensile strength and elongation. In addition, the transformation rate of isothermal bainite after athermal martensite formation revealed a trend of deceleration and then acceleration with austenisation temperature at the beginning period. Moreover, the size of bainite plates decreased first and then increased with austenisation temperature.

Disciplines

Engineering | Science and Technology Studies

Publication Details

Tian, J., Xu, G., Jiang, Z., Yuan, Q., Chen, G. & Hu, H. (2019). Effect of austenisation temperature on bainite transformation below martensite starting temperature. *Materials Science and Technology*, 35 (13), 1539-1550.

Authors

Junyu Tian, Guang Xu, Zhengyi Jiang, Qing Yuan, Guanghui Chen, and Haijiang Hu

1 **Effect of austenization temperature on transformation of bainitic steel**
2 **austempered below M_s**

3 **J.Y. Tian¹, G. Xu^{1,*}, Z.Y. Jiang², Q. Yuan¹, G.H. Chen¹, H.J. Hu¹**

4 ¹The State Key Laboratory of Refractories and Metallurgy, Wuhan University of
5 Science and Technology, Wuhan 430081, China

6 ²School of Mechanical, Materials, Mechatronic and Biomedical Engineering,
7 University of Wollongong, NSW 2522, Australia

8 * Corresponding author (Guang Xu)

9 Email: xuguang@wust.edu.cn

10 **Abstract**

11 Effects of prior austenite grain size (PAGS) on martensite and bainite transformation
12 behavior, microstructure, and mechanical properties of a bainitic steel austempered
13 below martensite starting temperature (M_s) were investigated in this study. **Results**
14 **show that the amount of athermal martensite (AM) gradually increased with the**
15 **increase of PAGS, whereas the amounts of bainite and retained austenite (RA) initially**
16 **increased and then decreased, resulting in the trend of the first increase and then**
17 **decrease in the product of tensile strength and elongation (PSE). In addition, the**
18 **transformation rate of isothermal bainite after AM formation revealed a trend of**
19 **deceleration and then acceleration with PAGS at the beginning period. Moreover, the**
20 **size of bainite plates decreased first and then increased with PAGS.**

21 **Keywords:** Prior austenite grain size; Martensite starting temperature; Transformation
22 kinetics; Microstructure; Mechanical properties

1 **1. Introduction**

2 Martensite and bainite transformations play very important roles in improving
3 mechanical properties of steels [1–3]. In some advanced high-strength steels (AHSSs),
4 such as the quenching and partitioning (Q&P) steels, bainitic steels, and transformation-
5 induced plasticity (TRIP) steels, their superior mechanical properties are directly
6 related to martensite and/or bainite transformations [4–7]. It is well known that the
7 kinetics of bainite and martensite transformations is generally affected by prior
8 austenite grain size (PAGS). Therefore, during the heat treatment of high-strength steels,
9 it is crucial to studying the effects of PAGS on martensite and bainite transformations
10 for achieving better mechanical properties of the steel.

11 In recent years, innumerable studies have been conducted to investigate the effect
12 of PAGS on martensite transformation [8-10]. For example, Sun et al. [8] investigated
13 the effect of PAGS on martensite transformation and mechanical properties of two high-
14 carbon steels and reported that grain refinement can induce a phase transformation of
15 high carbon martensite substructure from twin to dislocations. Similarly, Prawoto et al.
16 [9] studied the effect of PAGS on the morphology of the martensite microstructure in a
17 medium carbon steel . The block and packet sizes in the martensite increase with the
18 increase of PAGS. It is also well accepted that smaller PAGS causes a significant
19 decrease in martensite starting transformation temperature (M_s). Hanamura et al. [11]
20 investigated the effect of PAGS on martensite transformation in an air-cooled 0.1C–
21 5Mn martensitic steel and claimed that the value of M_s was decreased by 40 °C with a
22 decrease of PAGS from 254 μm to 30 μm , and similar findings were also reported by

1 Yang et al. [12] and Garcia-Junceda et al. [13]. In addition, Lee et al. [14] proposed a
2 new M_S equation and a new martensite transformation kinetics model based on the
3 effect of PAGES, and expounded that the value of M_S decreased gradually with the
4 decrease of PAGES. The decrease of M_S with smaller PAGES can be explained by the
5 theory of Hall-Petch strengthening effect. **Smaller PAGES increases resistance to the**
6 **invariant-strain deformation of martensite transformation** [15].

7 Furthermore, apart from martensite transformation, the effect of PAGES on bainite
8 transformation has also been widely explored [16–20]. Hu et al. [17] investigated the
9 effect of PAGES on isothermal bainitic transformation kinetics in a high-carbon bainitic
10 steel. They elucidated that a coarse PAGES accelerated the transformation of bainite and
11 increased the amount of bainite transformation. Similarly, Xu et al. [18] reported that
12 in a medium carbon bainitic steel, larger PAGES resulted in a higher bainitic
13 transformation rate and increased the volume fraction of transformed bainite.
14 Matsuzaki et al. [19] studied the effects of PAGES on bainite transformation kinetics in
15 a low-carbon bainitic steel and advocated that the increase of PAGES led to an
16 acceleration in transformation kinetics and caused a considerable increase of the
17 amount of transformed bainite. The aforesaid studies emphasize that although larger
18 PAGES yields fewer nucleation sites, and it is beneficial for the growth of bainite sheaves.

19 However, it is important to note that previous studies mainly focused on the effects
20 of PAGES on the single-phase transformation of martensite or bainite; hence, the
21 influences of PAGES on both bainite and martensite transformations during
22 austempering below M_S have been rarely reported. In recent years, the isothermal

1 transformation process austempered below M_s has been explored to obtain finer bainite
2 microstructures and faster bainitic transformation kinetics [20-22]. The formation of
3 athermal martensite (AM) before the holding process yields more nucleation sites for
4 isothermal bainitic transformation during the isothermal process, thus resulting in finer
5 bainite microstructures and faster bainitic transformation kinetics. In addition, lower
6 austempering temperature causes a larger driving force. It is beneficial for attaining
7 finer bainite microstructures and thus achieves the excellent performance [23].
8 Therefore, it is necessary to investigate the effects of PAGES on martensitic and bainitic
9 transformations in bainitic steels austempered below M_s . Moreover, according to the
10 results of above references [11–14], it is found that a reduction in PAGES lowers M_s and
11 also decreases the amount of AM before isothermal holding due to smaller undercooling
12 [11–14]. Consequently, **more residual austenite films and blocky particles are formed**
13 **for bainite transformation**, thus resulting in an increase of the amount of transformed
14 bainite. However, at the same time, the reduction of PAGES also leads to a decrease of
15 the amount of bainite itself [17–19]. Hence, two opposite effects occur on transformed
16 bainite due to different PAGESs when the austempering temperature is below M_s . The
17 effect of PAGES on bainite transformation during austempering below M_s has not been
18 clearly identified. Therefore, it is indispensable to investigate the effects of different
19 PAGES on martensite and bainite transformation behavior, microstructures, and
20 mechanical properties of a low-carbon bainite steels austempered below M_s , and the
21 obtained results could provide useful information to understand how PAGES affects both
22 martensitic and isothermal bainitic transformations during austempering below M_s .

1 2. Materials and experimental procedure

2 2.1 Materials

3 The experimental steel (chemical composition Fe-0.221C-1.802Si-2.013Mn -
4 0.227Mo-0.984Cr (wt%)) was first refined in a 50 kg laboratory-scale vacuum furnace,
5 then hot-rolled to 12 mm thick plates on a four-high mill, and finally, air-cooled to room
6 temperature. A considerable Si content was added to the steel in order to effectively
7 prevent the formation of brittle carbides [24]. Further, the addition of manganese (Mn)
8 and chromium (Cr) increased the stability and the hardenability of undercooled
9 austenite, and consequently, enlarged the range of bainitic transformation [25,26].
10 Moreover, molybdenum (Mo) was added to expedite the bainitic transformation and
11 prevent temper embrittlement [27].

12 2.2 Methods

13 Thermal simulation experiments were conducted on a Gleeble-3500 simulator.
14 Cylindrical samples of 6 mm diameter and 100 mm length were prepared. Dilatations
15 of the samples along the radial direction were measured during the entire experimental
16 process. The A_{c3} temperatures of the tested steels were calculated as 831 °C according
17 to the Andrews formula [28] (Eq. (1)). For obtaining the full austenized microstructure,
18 the range of austenitizing temperature of 1000–1250 °C was selected to obtain different
19 PAGS.

$$20 \quad A_{c3} \text{ (} ^\circ\text{C)} = 910 - 203\sqrt{x_c} - 15.2x_{Ni} + 44.7x_{Si} + 104x_V + 31.5x_{Mo} + 13.1$$
$$21 \quad x_W - 30x_{Mn} - 11x_{Cr} - 20x_{Cu} + 700x_P + 400x_{Al} + 20x_{As} + 400x_{Ti} \quad (1)$$

22 where x_i is the mass percentage of the element “ i ”. In addition, the bainite and
23 martensite starting temperatures (B_S and M_S) of the tested steel were 501 °C and 352 °C,

1 respectively, calculated by MUCG83 software developed by Bhadeshia at Cambridge
2 University [29]. In addition, the curves of transformation time temperature (TTT) for
3 the tested steel calculated by MUCG83 software is given in Fig. 1 in the supplemental
4 materials.

5 The samples were heated to different austenitizing temperatures of 1000 °C,
6 1100 °C, 1200 °C, and 1250 °C at heating rate 10 °C/s and held for 10 min to obtain
7 different PAGSs. After austenitization, all samples were cooled down to 320 °C, held
8 for 30 min to form martensite and austenite, and finally, air cooled to room temperature.
9 The proposed heat treatment procedure is presented in detail in Fig. 1.

10 **Fig. 1**

11 2.3 Characterization

12 The heat-treated samples were mechanically polished and etched with a 4% nital
13 to perform microstructural characterization. A Nova 400 Nano-field emission scanning
14 electron microscope (FE-SEM) coupled with an electron backscatter diffraction (EBSD)
15 measurements was used to observe microstructures and fracture morphologies, and the
16 crystallographic orientation relationship between different phases. In addition, a JEM–
17 2100F transmission electron microscope (TEM) was also used to observe the finer
18 microstructure. Tensile tests were carried out on a UTM–4503 electronic universal
19 tensile tester (with a cross-head speed of 1 mm/min) at room temperature, and an
20 average value of three tensile test data was considered for each sample in order to
21 maintain the accuracy. The volume fraction of RA was determined by X-ray diffraction
22 (XRD) experiments on an Empyrean diffractometer (equipped with an unfiltered Co-
23 K α radiation source) operating at 35 kV and 50 mA. The step size and the counting
24 time for XRD were set to 0.0263° and 77.265 s, respectively.

25 3. Results and Discussion

1 3.1.1 Dilatation

2 Fig. 2a displays the variations in the programmed and thermocouple temperatures
3 against time for the sample austenitized at 1000 °C (during the entire heat treatment
4 process). The insert image in Fig. 2a reveals that apart from the temperature **overshot**
5 from 320.0 to 317.6 °C, the temperature fluctuation was ± 1 °C, and it indicates that the
6 thermocouple mainly followed the programmed temperature. The small temperature
7 **overshot** disappeared in a very short time. Therefore, the effect of temperature
8 fluctuation was found to be negligible.

9 Fig. 2b exhibits the corresponding dilatation curves against temperature. **The slope**
10 **change at about 550 °C during heating in Fig. 2b may be caused by the dissolution of**
11 **previous carbides and the tempering of phase products.** The values of A_{C1} and A_{C3}
12 temperatures were measured as 792 °C and 875 °C, respectively, based on the tangent
13 method. Hence, it confirms that the temperature of 1000 °C was high enough to achieve
14 the full austenitization microstructure. Moreover, during the cooling process from
15 1000 °C to M_S (point A), the dilatation curve was found to be a straight line, thus
16 indicating that no high-temperature products were formed due to the high cooling rate
17 of 20 °C/s. **Fig. 2c shows that how to determine the temperature of M_S , and it indicates**
18 **that M_S is 354 ± 3 °C for the sample austenitized at 1000 °C. According to the result**
19 **proposed by Bhadeshia et al. [30], the conventional method to determine the M_S is**
20 **uncertain, so in the present study, the offset method is used and ensures that the**
21 **determination of M_S is more reproducible. The details of Bhadeshia's offset method for**
22 **determining M_S is given in the supplemental materials.** After point A (M_S), the
23 dilatation curve manifested an obvious increase in the **dilatation** from points A to B
24 (corresponding to the holding temperature of 320 °C). It signifies that some
25 transformation occurred before holding at a high cooling rate of 20 °C/s. It can be

1 inferred that the increase of dilatation from points A to B is caused by martensite
2 transformation (similar results have been also reported in our previous study [22]).
3 After isothermal treatment the slope change of the curve is caused by fresh martensite
4 (FM) transformation (Fig. 2b). Furthermore, by the same method, the M_S temperatures
5 of other three samples austenitized at 1100 °C, 1200 °C, and 1250 °C were calculated
6 to be 359 ± 2.2 °C, 370 ± 3.0 °C, and 376 ± 1.9 °C respectively. (The corresponding
7 dilatation curves of other three samples are given in Fig. 2 in the supplemental
8 materials.) Therefore, the value of M_S increased with the increase of austenitizing
9 temperature, and this result is consistent with the findings reported by Hanamura et al.
10 [11].

11 **Fig. 2**

12 Fig. 3 displays the dilatation curves of the samples austenitized at different
13 temperatures in the range of 1000–1250 °C. Fig. 3a illustrates the curves of total
14 dilatation (from point A (Fig. 2b) to the end of the holding process) as a function of
15 time (the time at point A was selected as the zero point of the abscissa and the ordinate
16 axes). According to the previous study [22], the total dilatation of all samples
17 austempered below M_S can be divided into two parts (based on the inflection points a,
18 b, c, and d): dilatation caused by athermal martensite transformation (D_{AM}) and
19 dilatation caused by bainite transformation (D_B). It is observable from Fig. 3a that
20 dilatation increases dramatically before the corresponding inflection point. However,
21 after corresponding inflection point, the increasing trend in dilatation tends to slow
22 down significantly. The rapid increase of dilatation is caused by the formation of
23 athermal martensite (AM), whereas the slow increase of dilatation is due to the
24 isothermal bainitic transformation [22,23,31]. Fig. 3b presents dilatations as a function
25 of holding time during isothermal transformation at 320 °C (the beginning of isothermal

1 bainite transformation was selected as the zero point of the abscissa and the ordinate
2 axes). The transformation temperature was constant and no extra force was applied to
3 the sample during isothermal holding, thus the dilatation in Fig. 3b represents the real
4 amount of bainite transformation. On the basis of dilatation results in Figs. 3a and 3b,
5 the variations in D_{AM} and D_B against austenitization temperature are presented in Fig.
6 3c. It is noticeable that D_{AM} increased with the increase of austenitization temperature,
7 whereas D_B increased first and then decreased. When the sample was austenitized at
8 1200 °C, D_B reached the maximum value. In addition, according to the result shown in
9 Fig. 3b, the curves of dilatation change rate indicating bainite transformation kinetics
10 are plotted in Fig. 3d. It shows that the initial transformation rate first decreases and
11 then increases with the increase of austenitization temperature. It is known that bainite
12 transformation contains nucleation and growth. According to the sub-unit theory
13 proposed by Bhadeshia [29], a sub-unit nucleates at austenite grain boundary and
14 lengthens until its growth is arrested by plastic deformation within the austenite. New
15 sub-units then nucleate at its tip, and the sheaf structure develops as this process
16 continues. Compared to nucleation, the growth of bainite sub-units is much faster.
17 Therefore, the nucleation rate has a crucial role on transformation rate at the initial stage
18 of bainite transformation. In the present study, the formation of AM expedites the
19 bainite transformation by providing the more nucleation sites due to the increase of γ/α
20 interface. However, the transformation rate is also affected by the PAGS. The larger
21 PAGS provides less grain boundary area, which is not beneficial to nucleation of bainite
22 transformation. Therefore, when the samples with different PAGS are austempered
23 below M_S , the bainite transformation rate is dependent on the competition between the
24 increase of nucleation sites brought by more AM formation and the decrease by the
25 larger PAGS. In the samples austenitized at range of 1000-1200 °C, although the

1 formation of AM compensates for the partial nucleation sites, the larger PAGS by
2 higher austenized temperature provides less nucleation sites to bainite transformation,
3 leading to the lower transformation rate with the increase of austenization temperature.
4 However, when the sample is austempered at 1250 °C, the role of more nucleation sites
5 brought by more AM formation is larger than that of the decrease of nucleation sites by
6 larger PAGS, so the transformation rate increases. Therefore, at the beginning period,
7 the transformation rate shows a trend of the first deceleration and then acceleration with
8 austenization temperature. The slowest transformation rate occurs in the sample
9 austenized at 1200 °C. However, as transformation progresses, the growth of bainite
10 sheaves is hindered by more austenite grain boundaries. The hindrance of bainite
11 growth less likely happens in larger PAGS, resulting in the different change trend of
12 transformation rate during the subsequent transformation period of 11~400 s. In this
13 period, the largest and slowest transformation rates occur in samples austenized at
14 1200 °C and 1250°C, respectively. Furthermore, the larger interior stress caused by the
15 more AM formation also promotes the bainite transformation. Therefore, the bainite
16 transformation amount increases with the increase of austenization temperature from
17 1000 to 1200 °C. It is noted that the amount of bainite transformation decreases when
18 the austenization temperature increases from 1200 to 1250 °C. It is mainly attributed to
19 the less residual austenite to be reserved due to the formation of large amount of AM.
20 As a result, the amount of bainite transformation shows the trend of the first increase
21 and then decrease with the increase of austenization temperature.

22 **Fig. 3**

23 3.1.2 Microstructure

24 Figs. 4(a–d) display the micrographs of prior austenite grains (PAGs) and prior
25 austenite grain boundaries (PAGBs) of different samples. The values of PAGS were

1 calculated by Image-Pro Plus software based on the diagonal method. Every nearly
2 complete grain of each micrograph is selected, and two diagonals are drawn randomly
3 in each grain. The average value of the two diagonals is calculated as the size of this
4 grain. Finally, the average value of the size of all grains is selected as the grain size of
5 the whole micrograph. In order to achieve the higher accuracy, at least the results of
6 three micrographs are reported for each sample. The average sizes of PAGSs for
7 different samples austenitized at 1000–1250 °C were measured as $29.7 \pm 4.3 \mu\text{m}$, 38.4
8 $\pm 5.7 \mu\text{m}$, $52.2 \pm 5.5 \mu\text{m}$ and $61.3 \pm 7.6 \mu\text{m}$, respectively. Therefore, it is observable that
9 larger PAGS values were obtained at higher austenitization temperatures.

10 **Fig. 4**

11 Typical SEM micrographs of the samples treated by different austenitization
12 processes are presented in Fig. 5. The obtained microstructures mainly consisted of
13 bainite, athermal martensite (AM), film-like retained austenite (RA),
14 martensite/austenite islands (M/A), and fresh martensite (FM) [3,22,32]. AM was
15 formed during the cooling process before the holding process, whereas FM was
16 obtained in the final cooling process after the holding process. According to the method
17 provided in Ref. [32], M/A islands, FM, AM and B can be distinguished. Because M/A
18 and FM contained more supersaturated carbon, they were more difficult to be etched
19 than AM, and the strength of AM gradually decreased due to subsequent annealing
20 during the holding process [22], resulting in the convex blocky morphology of M/A and
21 FM and the concave polygonal morphology of AM. Moreover, carbides were also
22 observed in all samples because the tempering of AM during the isothermal holding
23 process led to the precipitation of carbides. The similar results are reported by Toji et

1 al. [33] and Tariq et al. [34]. In addition, a small amount of FM was observed in the
2 samples austenitized at 1000 °C (Fig. 5a) and 1250 °C (Fig. 5d). The phase of FM is
3 not observed in samples austenitized at 1100 and 1200 °C because a large amount of
4 bainite transformation leads to the increase of the stability of untransformed austenite,
5 which is retained at room temperature as M/A and RA.

6 **Fig. 5**

7 According to the identification method provided in Ref. [32], B and AM can be
8 distinguished, and then based on the SEM micrographs, the volume fractions of B and
9 AM were calculated using Image-Pro Plus software according to the method depicted
10 in the previous study [22], and the corresponding results are presented in Table 1. It is
11 evident that V_{AM} increased with the increase of austenitization temperature, whereas V_B
12 manifested different changing trends. With the increase of austenitization temperature,
13 V_B first increased and then decreased. The maximum value of V_B was obtained in the
14 sample austenitized at 1200 °C, whereas the sample austenitized at 1250 °C yielded the
15 maximum V_{AM} and the minimum V_B values, and these results are consistent with the
16 findings from the dilatation curves in Fig. 3. It is observable from Fig. 4 that the value
17 of PAGES increased from 29.7 μm to 61.3 μm with the increase of austenitization
18 temperature. The value of M_s generally increases with the increase of PAGES. Hence,
19 larger undercooling for martensite transformation occurs due to larger PAGES when
20 different samples are austempered at the same temperature [11–14]. It can be explained
21 by the Hall–Petch strengthening effect that smaller PAGES increases resistance to the
22 invariant-strain deformation of martensite transformation [15]. The Hall–Petch

1 strengthening effect can be explained by the relationship $\rho \propto 1/D$, where ρ is dislocation
2 density and D is austenite grain diameter. Therefore, smaller austenite grain diameter
3 results in larger dislocation density. An increase of dislocation density caused by
4 decrease of PAGES will result in a strengthening of the austenitic matrix by the Hall-
5 Petch effect, increasing the resistance of the austenite to plastic deformation locally as
6 well as macroscopically, which means a bigger impediment to martensite
7 transformation by increasing the non-chemical free energy [12,13]. Therefore, the
8 smaller PAGES leads to the decrease of M_s . In addition, Caballero et al. [35] referred a
9 probability equation (Eq. (2)) applied by Cohen and Olson.

$$10 \quad p = 1 - \exp(-\lambda v) \quad (2)$$

11 where p represents the fraction of crystals containing martensite, v is the grain volume,
12 and λ is the probability of nucleation of martensite per unit volume which depends on
13 the temperature. The aforesaid equation reveals that the probability of event taking
14 place decreases exponentially as the grain size decreases [35]. Therefore, the amount
15 of AM gradually increased with the increase of PAGES. Regarding to V_B , it is different
16 from the samples austempered above M_s , and it depends on not only the isothermal
17 temperature and time, but also the amount of AM. The amount of AM increases as
18 PAGES increases, which not only provides more nucleation sites for bainite
19 transformation, but also leads to the less amount of residual austenite for bainitic
20 transformation. That is to say, the AM formation has two opposite effects on the bainite
21 amount. Moreover, PAGES also affects the amount of bainite transformation. With the
22 increase in austenitization temperature from 1000 °C to 1200 °C, larger PAGES can be

1 obtained and generates more bainite phase. It happens because the diffusion coefficient
2 of grain boundary decreases with the increase of PAGES [36]. As a result, the carbon
3 content of residual austenite in coarse PAGES samples hardly reaches to the value
4 predicted by the T_0 diagram, thus more bainite can be transformed from residual
5 austenite [36]. In the present study, the amount of bainite was closely related to both
6 PAGES and V_{AM} . The effect of AM and PAGES and the reason for the change trend of V_B
7 are discussed in Section 3.1.1.

8 **Table 1**

9 Fig. 6 depicts the EBSD result of the sample austenitized at 1250 °C. Fig. 6a
10 displays the crystallographic orientation of the sample austenitized at 1250 °C. Fig. 6c
11 presents the corresponding diagram of frequency versus band contrast (in which higher
12 band contrast represents lower stresses in grains). Two peaks can be detected in Fig. 6c,
13 thus indicating the formation of two types of phase products – martensite and bainite.
14 According to the results of Gauss fitting, the boundary value of band contrast for these
15 two peaks was found as 80 (Fig. 6c). The band contrast smaller than 80 is colored by
16 red, and the remaining white area represents the band contrast higher than 80, thus the
17 red area represents the martensite phase and the white area denotes the bainite phase. It
18 is evident that the red martensite phase in Fig. 6b consisted of AM, FM, and M/As.
19 Moreover, film-like RA was not noticed in the image due to their very small grain size.
20 The volume fraction of white bainite area was calculated as 27.5% by Image-Pro Plus
21 software (Fig. 6b) [22]. Similarly, the volume fractions of bainite in the samples
22 austenitized at 1000 °C, 1100 °C, and 1200 °C were measured as 31.8%, 36.9%, and

1 44.1% respectively. These results are well consistent with the statistical results obtained
2 from SEM microstructures in Fig. 5.

3 **Fig. 6**

4 In order to more clearly observe the bainite morphology and measure the sizes of
5 bainite plates, the microstructure corresponding to Fig. 5 is presented in larger
6 magnification in Fig. 7. It is noticeable that carbides only existed in AM, whereas all
7 bainite plates were carbide-free, it can be attributed to the presence of considerable Si
8 content in the experimental bainite steel. Moreover, the thicknesses of bainite plates in
9 different samples were determined by the equation $n = 2L_t/\pi$ [5], where n is the
10 thickness of a bainite plate, L_t is the mean linear intercept in the direction normal to
11 plate length and was determined by Image-Pro Plus software. The average values of n
12 for the samples austenitized at 1000 °C, 1100 °C, 1200 °C, and 1250 °C were measured
13 as 376.5 ± 15.1 nm, 350.8 ± 21.3 nm, 311.4 ± 19.6 nm, and 352.6 ± 22.4 nm, respectively.
14 It indicates that the thickness of bainite plates continuously decreased from 1000 °C to
15 1200°C and then sharply increased from 1200 °C to 1250°C. **Bainite transformation**
16 **contains nucleation and growth. According to the sub-unit theory proposed by**
17 **Bhadeshia [29], a sub-unit nucleates at austenite grain boundary and lengthens until its**
18 **growth is arrested by plastic deformation within the austenite. Once lengthening stops,**
19 **thickening of laths can be a way to continue transformation in smaller prior austenite**
20 **grains. Thickening of laths by sidewise nucleation and growth of new sub-units has**
21 **been reported in other studies [37]. This thickening can continue as long as the chemical**
22 **driving force is available for bainitic transformation. Therefore, the thickness of bainite**

1 plates decreases with the increase of PAGS. However, when the austenized temperature
2 increases from 1200 to 1250 °C, the formation of a larger amount AM leads to the PAG
3 to be divided into several subgrains. The small subgrain limits the lengthening, so it has
4 to transform by the sidewise nucleation and growth. Therefore, the size of bainite plates
5 increases.

6 **Fig. 7**

7 Fig. 8 displays the diffraction patterns of the samples austenitized at 1000–1250 °C.
8 The integrated intensities and the angles of diffraction peaks were accurately
9 determined by HighScore Plus software based on the obtained diffraction patterns. The
10 volume fractions of RA (V_{RA}) of the four samples austenitized at different temperatures
11 are calculated according to the integrated intensities of (200) and (211) peaks of ferrite
12 and (200) and (220) peaks of austenite based on Eq. (3).

$$13 \quad V_i = \frac{1}{1+G(I_\alpha/I_\gamma)} \quad (3)$$

14 where V_i represents the volume fraction of austenite for each peak; G value is chosen
15 as follows, 2.5 for $I_\alpha(200)/I_\gamma(200)$, 1.38 for $I_\alpha(200)/I_\gamma(220)$, 1.19 for $I_\alpha(211)/I_\gamma(200)$,
16 0.65 for $I_\alpha(211)/I_\gamma(220)$; I_α and I_γ represent the corresponding integrated intensities of
17 ferrite and austenite [38], and the corresponding results are presented in Table 1. It is
18 observed that when the austenitization temperature increased from 1000 °C to 1100 °C,
19 the amount of RA increased significantly. It is well known that the process of bainite
20 transformation is normally accompanied by the rejection of carbon atoms. When a large
21 number of carbon atoms were ejected in adjacent untransformed austenite due to the
22 formation of bainite, the stability of untransformed austenite was significantly
23 increased. Therefore, the subsequent martensite transformation was retarded by stable

1 untransformed austenite during the cooling process, and consequently, more RA was
2 retained at room temperature. Moreover, the amount of RA generally depends on not
3 only the amounts of bainite, but also the residual austenite after AM formation. With
4 the increase in austenitization temperature from 1100 °C to 1200 °C, the formation of
5 a large amount of bainite and AM led to a small amount of untransformed austenite,
6 thus the amount of RA decreased. Similarly, when the austenitization temperature
7 further increased to 1250 °C, a sharp decrease in the amount of RA was observed. It is
8 also attributed to the obvious decrease of bainite transformation amount and the
9 increase of AM amount. It is well known that less amount of bainite transformation
10 means less rejection of carbon atoms into residual austenite, resulting in the unstable
11 residual austenite, which transformed to martensite during final cooling process.
12 Therefore, the amount of RA first increased and then decreased with the rise of
13 austenitization temperature.

14 **Fig. 8**

15 3.1.3 Tensile test

16 Tensile test results of the samples austenitized at different temperatures are
17 presented in Table 2, and the corresponding stress-strain curves are displayed in Fig. 9.
18 It is observable that the values of tensile strength (TS), yield strength (YS), and total
19 elongation (TE) of the samples first increased and then started to decrease. The highest
20 strength and the largest elongation were found in the sample austenitized at 1100 °C,
21 thus resulting in the largest product of tensile strength and elongation (PSE).

22 **Table 2**

1
2 In comparison to the sample austenitized at 1100 °C, the sample austenitized at
3 1000 °C contained a lower amount of lath-like bainite and RA as well as some hard FM.
4 Further, the presence of more nano-sized bainite causes higher strength in bainite steels
5 [5,6,39]. Moreover, the strength and the elongation of bainitic steels generally depend
6 on the amount of RA produced by the transformation induced plasticity (TRIP) effect.
7 In comparison to the sample austenitized at 1100 °C, less amount of RA was formed in
8 the sample austenitized at 1200 °C, thus resulting in a slight decrease in strength and
9 elongation. In addition, more AM also caused a decrease of strength. It is observable
10 from SEM microstructures in Fig. 7 that carbides were precipitated from AMs. A large
11 amount of AM resulted in more brittle carbides and degraded the mechanical properties
12 of the steel [22]. When the austenitization temperature reached 1250 °C, the strength
13 and the elongation of the steel sharply decreased, it can be attributed to the presence of
14 the smallest amounts of nano-sized bainite and RA and the largest amount of AM (Table
15 1). Figs. 10a and 10b, respectively, exhibit the TEM microstructure and the tensile
16 fracture morphology of the sample austenitized at 1250 °C. The presence of a large
17 amount of carbides significantly deteriorated the mechanical properties of the steel (Fig.
18 10a). It is evident from Fig. 10b that the fracture morphology was composed of a large
19 amount of cleavage facet and a small amount of dimple. It is well known that the brittle
20 fracture mode generates cleavage facets, thus resulting in an inferior elongation. On the
21 contrary, ductile fracture forms dimples and yields better elongation [40]. It is
22 noticeable that brittle fracture mainly occurred in the sample austenitized at 1250 °C,

1 thus resulting in the worst mechanical properties.

2 **Fig. 10**

3 **4. Conclusions**

4 In the present study, the effects of PAGES on martensitic and bainitic transformation
5 behavior, microstructures, and mechanical properties of a bainitic steel during
6 austempering below M_s were investigated. The main inferences are depicted below.

7 (1) With the increase of PAGES, the volume fraction of bainite first increased and
8 then decreased, which is significantly different from the results of samples austempered
9 above M_s . In addition, the volume fraction of AM increased with the increase of PAGES,
10 whereas the volume fraction of RA showed the same change trend with bainite volume
11 fraction.

12 (2) At the initial stage of transformation, the bainite transformation rate first
13 decelerated and then accelerated with the increase of PAGES, and the sample austenized
14 at 1200 °C had slowest transformation rate. However, in a very short time (about 11 s),
15 the transformation rate of sample austenized at 1200 °C became the largest because the
16 growth of bainite sheaves was less likely hindered by austenite grain boundaries,
17 resulting in the largest amount of bainite.

18 (3) Differing from the commonly accepted viewpoint that the increase in PAGES
19 led to the finer bainite plates in bainitic steels austempered above M_s , the bainite plates
20 became finer firstly and then coarse with PAGES when samples are austempered below
21 M_s .

22 (4) When the tested low-carbon bainitic steel was austempered below M_s , the

1 mechanical properties increased initially, followed by a decrease with the increase in
2 PAGES. The best mechanical properties were achieved in the sample austenitized at
3 1100 °C, whereas the worst properties were found at the highest austenitizing
4 temperature.

5 **Acknowledgements**

6 The authors gratefully acknowledge the financial supports from the National
7 Nature Science Foundation of China (Nos.51704217, 51874216), the Major Projects of
8 Technological Innovation in Hubei (No.2017AAA116), Hebei Joint Research Fund for
9 Iron and Steel (E2018318013) and the State Scholarship Fund of China Scholarship
10 Council.

11 **References**

- 12 1. Bag A, Ray KK, Dwarakadasa ES. Influence of martensite content and morphology
13 on tensile and impact properties of high-martensite dual-phase steels. Metall Mater
14 Trans A. 1999; 30: 1193-1202.
- 15 2. Kumar A, Singh SB, Ray KK. Influence of bainite/martensite-content on the tensile
16 properties of low carbon dual-phase steels, Mater Sci Eng A. 2008; 474: 270-282.
- 17 3. Tian JY, Xu G, Jiang ZY, Hu HJ, et al. Effect of Ni addition on bainite
18 transformation and properties in a 2000 MPa grade ultrahigh strength bainitic steel.
19 Met Mater Int. 2018; 24:1202–1212.
- 20 4. Sun SH, Zhao AM, Ding R, et al. Effect of heat treatment on microstructure and
21 mechanical properties of quenching and partitioning steel, Acta Metall Sin (Eng
22 Lett). 2018; 31: 216–224.

- 1 5. Caballero FG, Bhadeshia HKDH, Mawella KJA, et al. Very strong low temperature
2 bainite. *Mater Sci Technol.* 2002; 18: 279-284.
- 3 6. García-Mateo C, Caballero FG, Bhadeshia HKDH. Acceleration of low-
4 temperature bainite. *ISIJ Int.* 2003; 43: 285-288.
- 5 7. Zhao JW, Jiang ZY. Thermomechanical processing of advanced high strength steels.
6 *Prog Mater Sci.* 2018; 94: 174–242.
- 7 8. Sun J, Jiang T, Wang Y, et al. Effect of grain refinement on high-carbon martensite
8 transformation and its mechanical properties. *Mater Sci Eng A* 2018; 726: 342-349.
- 9 9. Prawoto Y, Jasmawati N , Sumeru K. Effect of prior austenite grain size on the
10 morphology and mechanical properties of martensite in medium carbon steel. *J.*
11 *Mater Sci Technol.* 2012, 28: 461-466.
- 12 10. Dong J, Liu CX, Liu YC, et al. Influence of austenite grain size on martensite start
13 temperature of Nb-V-Ti microalloyed ultra-high strength steel. *Mater. Sci Forum.*
14 2016; 848: 624-632.
- 15 11. Hanamura T, Torizuka S, Tamura S, Enokida S, et al. Effect of austenite grain size
16 on the mechanical properties in air-cooled 0.1C-5Mn martensitic steel. *Mater Sci*
17 *Forum.* 2014; 783-786: 1027-1032.
- 18 12. Yang HS, Bhadeshia HKDH. Austenite grain size and the martensite-start
19 temperature. *Scripta Mater.* 2009; 60: 493-495.
- 20 13. Garcia-Junceda A, Capdevila C, Caballero FG, et al. Dependence of martensite
21 start temperature on fine austenite grain size, *Scripta Mater.* 2008; 58: 134–137.
- 22 14. Lee SJ, Tyne CJV. A kinetics model for martensite transformation in plain carbon

- 1 and low-alloyed steels. *Metall Mater Trans A*. 2012; 43: 422-427.
- 2 15. Brofman PJ, Ansell GS. On the effect of fine grain size on the M_s temperature in
3 Fe-27Ni-0.025C alloys. *Metall Mater Trans A*. 1983; 14: 1929-1931.
- 4 16. Lan LY, Qiu CL, Zhao DW, et al. Effect of austenite grain size on isothermal bainite
5 transformation in low carbon microalloyed steel. *Mater Sci Technol*. 2011; 27:
6 1657-1663.
- 7 17. Hu F, Hodgson PD, Wu KM. Acceleration of the super bainite transformation
8 through a coarse austenite grain size. *Mater Lett*. 2014; 122: 240-243.
- 9 18. Xu G, Liu F, Wang L, et al. A new approach to quantitative analysis of bainitic
10 transformation in a superbainite steel. *Scripta Mater*. 2013; 68: 833-836.
- 11 19. Matsuzaki A, Bhadeshia HKDH. Effect of austenite grain size and bainite
12 morphology on overall kinetics of bainite transformation in steels. *Met Sci J*. 1999;
13 15: 518-522.
- 14 20. Yakubtsov IA, Purdy GR, Analyses of transformation kinetics of carbide-free
15 bainite above and below the athermal martensite-start temperature, *Metall Mater*
16 *Trans A*. 2012; 43: 437-446.
- 17 21. Samanta S, Biswas P, Giri S, et al. Formation of bainite below the M_s temperature:
18 kinetics and crystallography. *Acta Mater*. 2016; 105: 390-403.
- 19 22. Tian JY, Xu G, Zhou MX, et al. Refined bainite microstructure and mechanical
20 properties of a high-strength low-carbon bainitic steel treated by austempering
21 below and above M_s , *Steel Res Int*. 2018; DOI: 10.1002/srin.201700469.
- 22 23. Zhao L, Qian L, Meng J, et al. Below- M_s austempering to obtain refined bainitic

- 1 structure and enhanced mechanical properties in low-C high-Si/Al steels, Scripta
2 Mater. 2016; 112: 96-100.
- 3 24. Tian JY, Xu G, Jiang ZY, et al. Transformation behavior and properties of carbide-
4 free bainite steels with different Si contents, 2018; DOI: 10.1002/srin.201800474.
- 5 25. Long XY, Zhang FC, Kang J, et al. Low-temperature bainite in low-carbon steel.
6 Mater Sci Eng A. 2014; 594: 344-351.
- 7 26. Tian JY, Xu G, Zhou MX, et al. The Effects of Cr and Al addition on transformation
8 and properties in low-carbon bainitic steels, Metals. 2017; 7: 40-50.
- 9 27. Caballero FG, Bhadeshia HKDH. Very strong bainite. Curr Opin Solid State Mater
10 Sci. 2004; 8: 251-257.
- 11 28. Andrews KW, Empirical formulae for the calculation of some transformation
12 temperatures. ISIJ Int. 1965; 203: 721–727.
- 13 29. Bhadeshia HKDH. Bainite in Steels. Institute of Materials, London, 1992, 1–450.
- 14 30. Yang HS, Bhadeshia HKDH. Uncertainties in dilatometric determination of
15 martensite start temperature. Mater Sci Technol. 2007; 23: 556-560.
- 16 31. Da Silva EP, De Knijf D, Xu W, et al. Isothermal transformations in advanced high
17 strength steels below martensite start temperature. Mater Sci Technol. 2015; 31:
18 808-816.
- 19 32. Navarro-López A, Hidalgo J, Sietsma J, et al. Characterization of
20 bainitic/martensitic structures formed in isothermal treatments below the M_s
21 temperature. Mater Charact. 2017; 128: 248-256.
- 22 33. Toji Y, Miyamoto G, Raabe D. Carbon partitioning during quenching and

- 1 partitioning heat treatment accompanied by carbide precipitation. *Acta Mater.* 2015;
 2 86: 137-147.
- 3 34. Tariq F, Baloch RA. One-step quenching and partitioning heat treatment of medium
 4 carbon low alloy steel. *J Mater Eng Perform.* 2014; 23: 1726-1739.
- 5 35. García-Junceda A, Capdevila C, Caballero FG, et al. Dependence of martensite
 6 start temperature on fine austenite grain size. *Scripta Mater.* 2008; 58: 134-137.
- 7 36. Jiang T, Liu HJ, Sun JJ, et al. Effect of austenite grain size on transformation of
 8 nanobainite and its mechanical properties. *Mater Sci Eng A.* 2016; 666: 207-213.
- 9 37. Singh K, Kumar A, Singh A. Effect of prior austenite grain size on the morphology
 10 of nano-bainitic steels. *Metall Mater Trans A.* 2018; 49: 1348-1354.
- 11 38. Zhou MX, Xu G, Wang L, et al. Effects of austenitization temperature and
 12 compressive stress during bainitic transformation on the stability of retained
 13 austenite. *T Indian I Metals.* 2017; 70: 1447-1453.
- 14 39. Hu H, Xu G, Wang L, et al. The effects of Nb and Mo addition on transformation
 15 and properties in low carbon bainitic steels. *Mater Des.* 2015; 84: 95-99.
- 16 40. Du WS, Cao R, Yan YJ, et al. Fracture behavior of 9% nickel high-strength steel at
 17 various temperatures: Part 1. Tensile tests. *Mater Sci Eng A.* 2008; 486: 611-625.

18

19 **Tables**

20 **Table 1.** The calculated volume fractions of B, AM and RA of different samples

T (°C)	V _{AM} (%)	V _B (%)	V _{RA} (%)
1000	28.8±0.7	31.3±0.5	6.6±0.4
1100	34.3±0.8	37.1±0.4	14.5±0.7
1200	40.9±0.5	43.8±0.5	12.8±0.8

1250 52.3±0.4 26.7±0.6 6.5±0.5

1

Table 2. The tensile test results of different samples

T (°C)	TS (MPa)	YS (MPa)	TE (%)	PSE (GPa%)
1000 °C	1394±31	998±19	11.72±0.45	16.337±0.244
1100 °C	1444±28	1092±21	13.51±0.57	19.508±0.435
1200 °C	1408±34	1004±28	13.08±0.64	18.416±0.574
1250 °C	1286±25	874±18	10.92±0.46	14.043±0.693

2

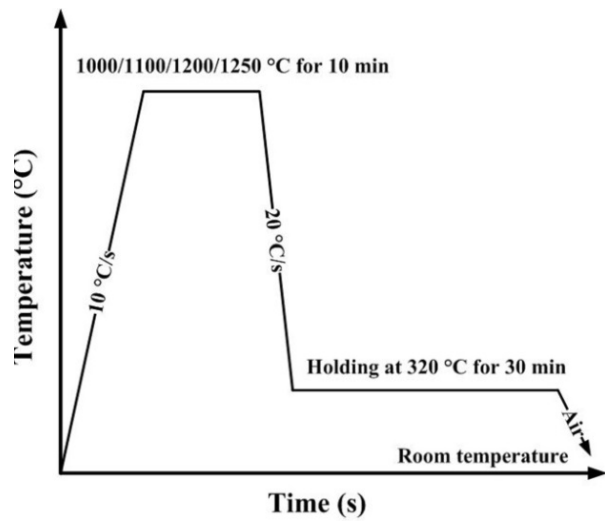
TS—tensile strength; YS—yield strength; TE—total elongation

3

4

1

Figures

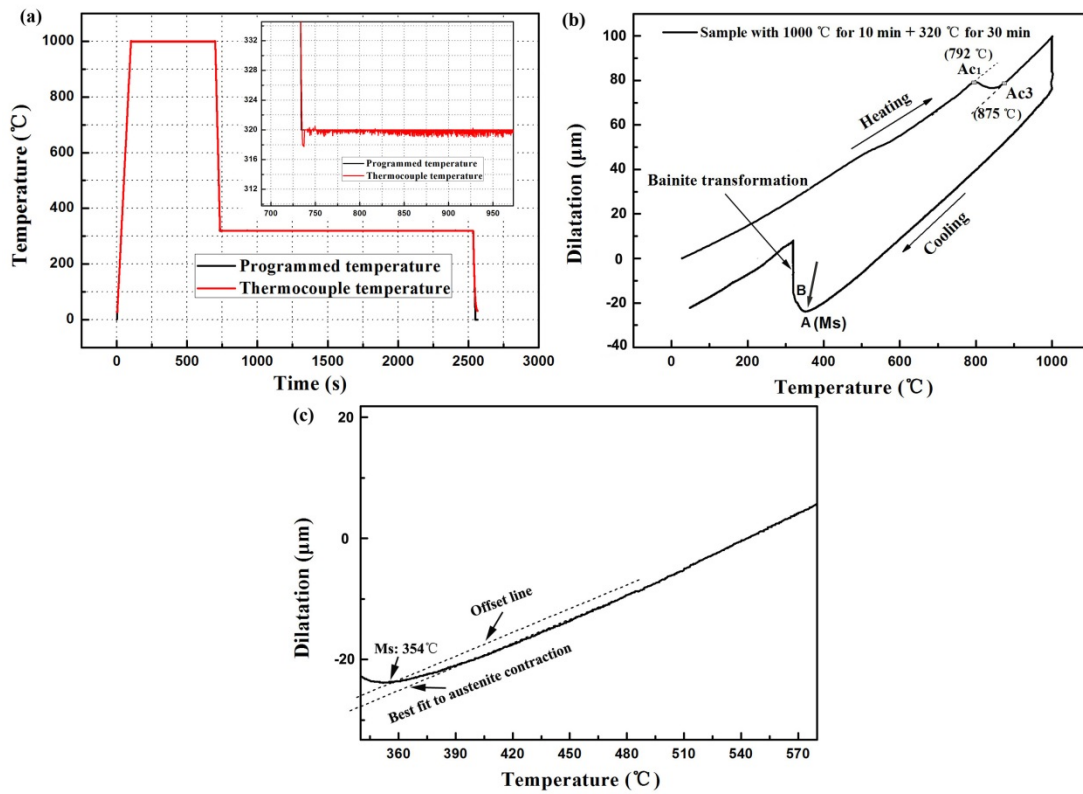


2

3

Figure. 1. The experimental procedures

4

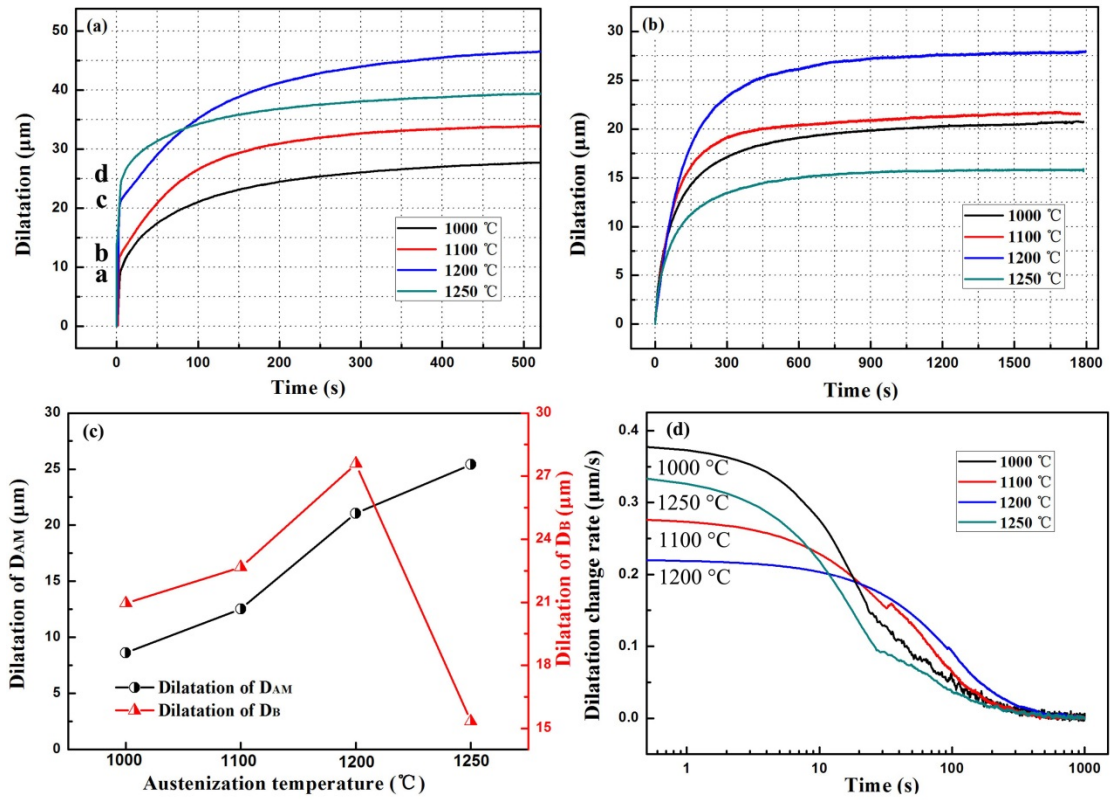


1

2 **Figure. 2.** Examples: (a) the programmed and thermocouple temperature versus

3 time; and (b) dilatation versus temperature during the whole transformation process

4



1

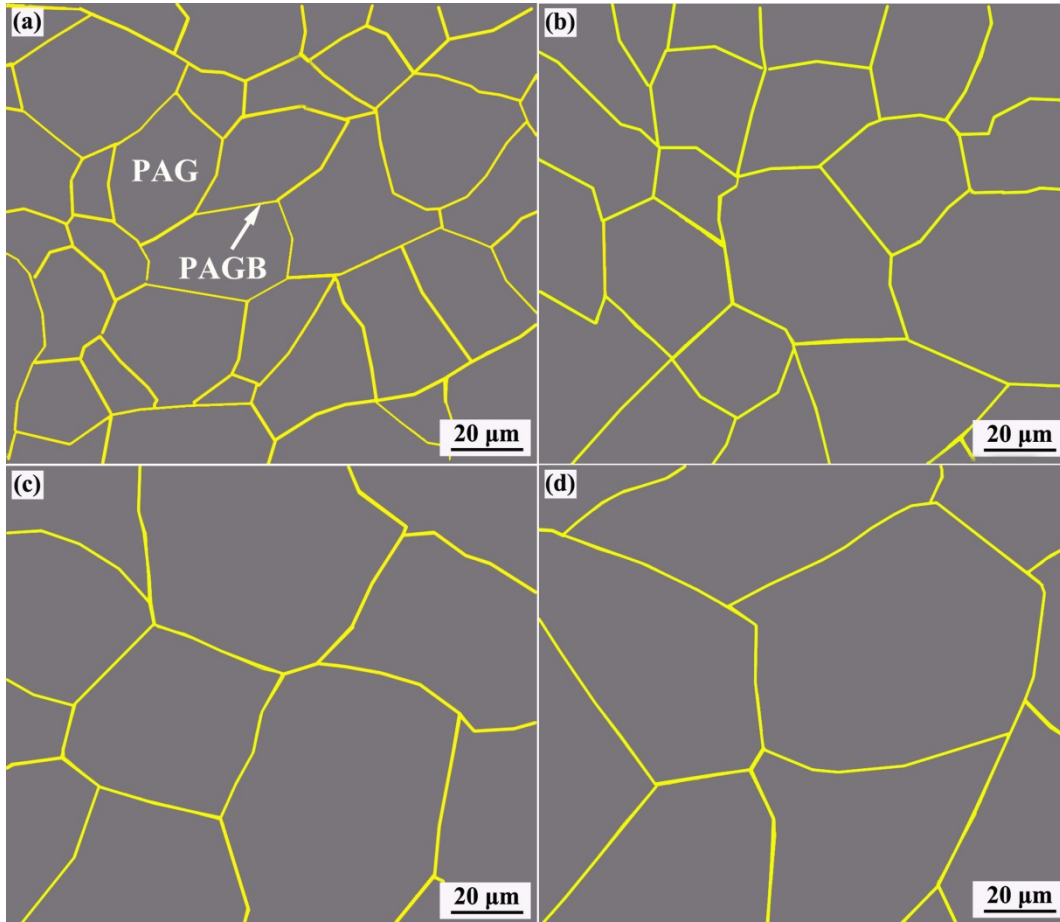
2

3

4

5

Figure. 3. (a) The curves of the total dilatation versus time; (b) the curves of D_B versus time; (c) the change trends of D_{AM} and D_B versus austenization temperature; and (d) the curves of dilatation change rate versus time indicating the bainite transformation rate



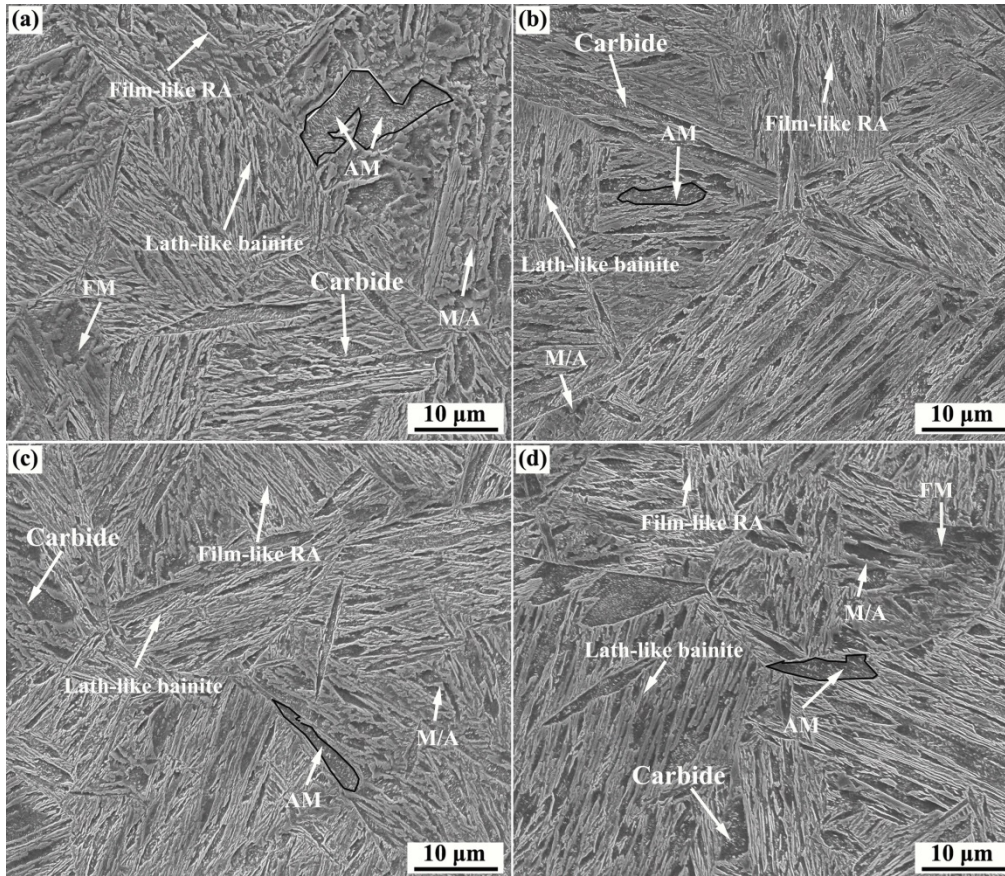
1

2

3

4

Figure. 4. PAGS for samples austenitized at: (a) 1000 °C; (b) 1100 °C; (c) 1200 °C
and (d) 1250 °C



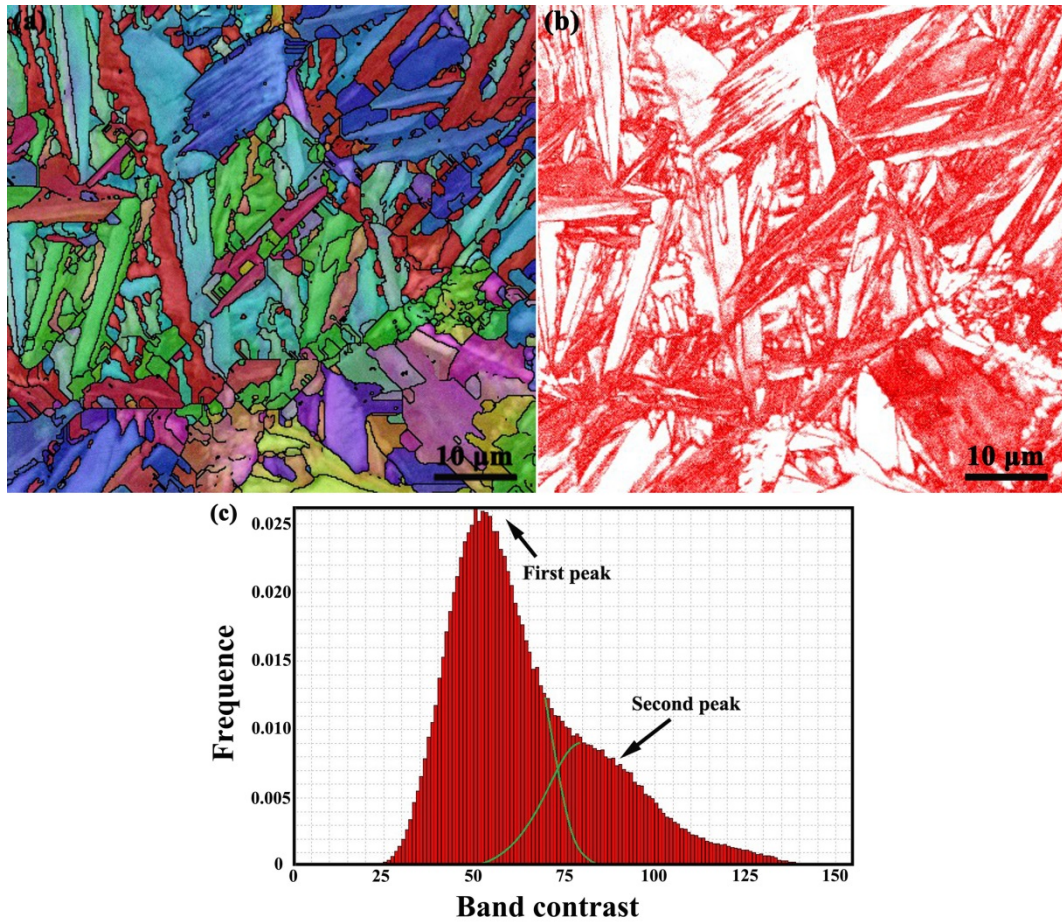
1

2

Figure. 5. Typical SEM microstructures after different austenization temperatures:

3

(a) 1000 °C, (b) 1100 °C, (c) 1200 °C and (d) 1250 °C

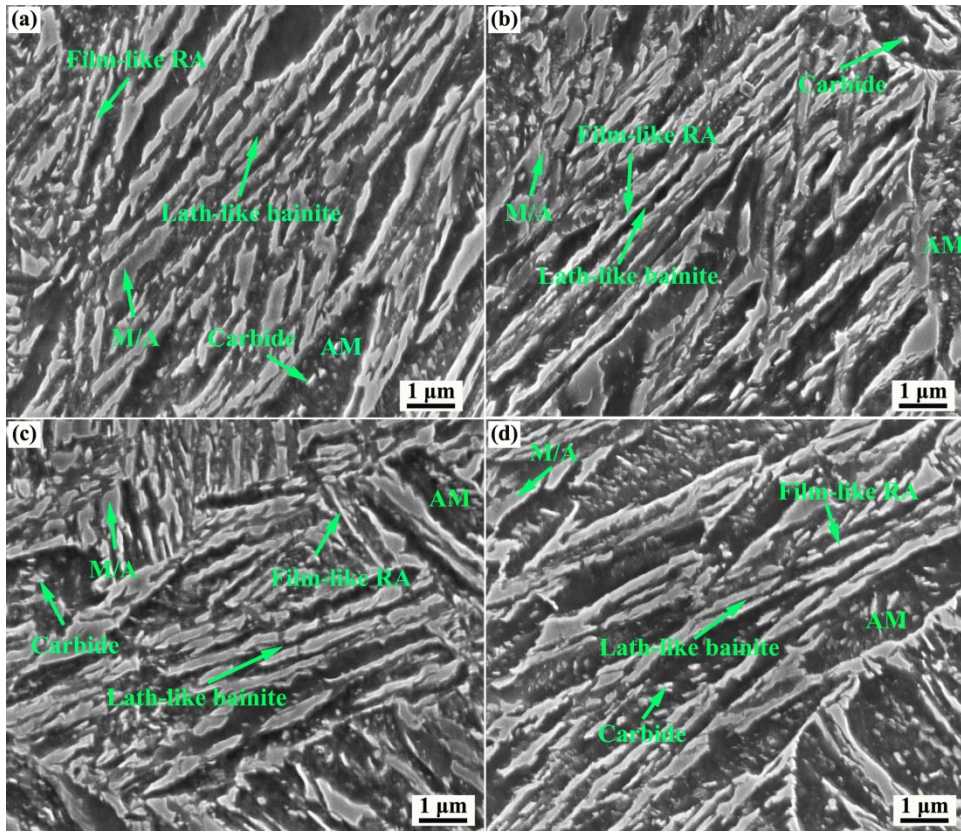


1

2 **Figure. 6.** An example of EBSD measurement of sample austenitized at 1250 °C: (a)

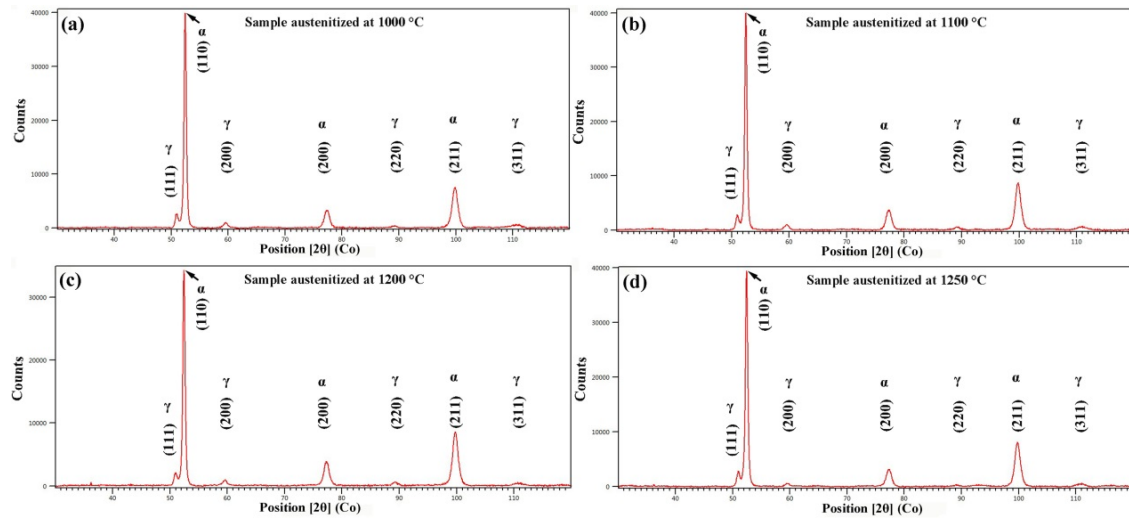
3 crystallographic orientation; (b) the diagram of frequency distribution; and (c) the

4 curves of frequency versus band contrast



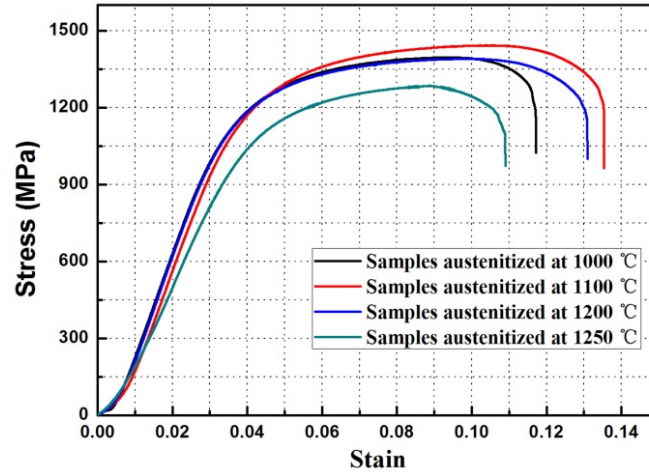
1
2
3
4

Figure. 7. The microstructures with larger magnification after different austenization temperatures: (a) 1000 °C and (b) 1100 °C; (c) 1200 °C and (d) 1250 °C



1
2
3

Figure. 8. Diffraction patterns of different samples

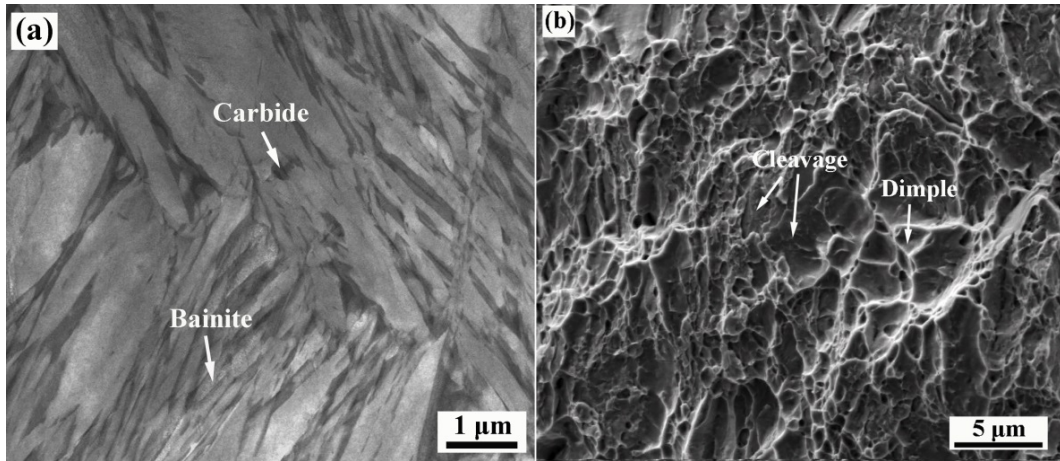


1

2

Figure. 9. The tensile curves of the samples austenitized at different temperatures

3



1
2
3
4

Figure. 10. Sample austenitized at 1250 °C: (a) TEM microstructure and (b) fracture morphology

1 **Figure captions**

2 **Figure. 1.** The experimental procedures.

3 **Figure. 2.** Examples: (a) the programmed and thermocouple temperature versus time;
4 (b) dilatation versus temperature during the whole transformation process and (c) The
5 determination of M_s by the method proposed by Bhadeshia et al. [30].

6 **Figure. 3.** (a) The curves of the total dilatation versus time; (b) the curves of D_B versus
7 time; (c) the change trends of D_{AM} and D_B versus austenization temperature; and (d) the
8 curves of dilatation change rate versus time indicating the bainite transformation rate

9 **Figure. 4.** PAGS for samples austenitized at: (a) 1000 °C; (b) 1100 °C; (c) 1200 °C and
10 (d) 1250 °C.

11 **Figure. 5.** Typical SEM microstructures after different austenization temperatures: (a)
12 1000 °C, (b) 1100 °C, (c) 1200 °C and (d) 1250 °C.

13 **Figure. 6.** An example of EBSD measurement of sample austenitized at 1250 °C: (a)
14 crystallographic orientation; (b) the diagram of frequency distribution; and (c) the
15 curves of frequency versus band contrast.

16 **Figure. 7.** The microstructures with larger magnification after different austenization
17 temperatures: (a) 1000 °C and (b) 1100 °C; (c) 1200 °C and (d) 1250 °C.

18 **Figure. 8.** Diffraction patterns of different samples.

19 **Figure. 9.** The tensile curves of the samples austenitized at different temperatures.

20 **Figure. 10.** Sample austenitized at 1250 °C: (a) TEM microstructure and (b) fracture
21 morphology.