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Novel method for refinement of retained austenite in micro/nano-structured bainitic steels

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Abstract

A comparative study was conducted to assess the effects of two different heat treatments on the amount and morphology of the retained austenite in a micro/nano-structured bainitic steel. The heat treatments used in this work were two-stage bainitic transformation and bainitic-partitioning transformation. Both methods resulted in the generation of a multi-phase microstructure containing nanoscale bainitic ferrite, and/or fresh martensitic phases and much finer retained austenite. Both heat treatments were verified to be effective in refining the retained austenite in micro/nano-structured bainite and increasing the hardness. However, the bainitic transformation followed by partitioning cycle was proved to be a more viable approach than the two-stage bainitic transformation due to much shorter processing time, i.e. ~2 hours compared to ~4 day, respectively.

Key words: Retained austenite, austenite refinement, quenching & partitioning, two-stage bainitic transformation, micro/nano-structured bainitic steel

Introduction

The microstructure of micro/nano-structured bainitic steels consists of nano-structured bainitic ferrites plates (20~65 nm thickness) and carbon-enriched retained austenite films (≥ 20 vol%), which are formed during low temperature bainitic transformation typically ranging from 200 °C to 300 °C¹⁻⁵. Generally, the presence of retained austenite grains larger than 1 μm in this type of steel is undesirable⁶, because the blocky retained austenite is prone to transform into brittle martensite under the influence of residual stresses or externally applied loads. Hase *et al.*⁷ reported that the blocks of retained austenite could be eliminated by adopting a two-stage bainitic transformation (B+B). However, such treatments need long cycle time (*usually e.g.* in excess of 20 hrs⁷, even if Co and Al were added to accelerate the bainitic transformation^{3,7}).

Previous works on the martensitic steels showed that the carbon from the supersaturated martensite can migrate to the untransformed austenite during partitioning stage⁸⁻¹¹. The carbon enriched retained austenite becomes more stable as a result of this relatively short process (usually ≤ 0.5 hrs)¹². Therefore, it appears that for the Q&P treatment, the microstructure can be refined and the transformation time can be considerably shortened, compared to the low temperature micro/nano-structured bainitic transformation. In the present work, conventional micro/nano-structured bainitic transformation combined with partitioning, and quenching process was conducted in order to develop a micro/nano-structured bainitic steel with even finer and more stable retained austenite grains. The heat treatment sequence consisted of a low temperature bainitic transformation followed by a partitioning process at an intermediate temperature, and finally water quenching to room temperature (B+P+Q). The effect of such multi-stage heat treatment on the microstructure of a micro/nano-structured bainitic steel was investigated by using optical, scanning and transmission electron microscopy methods.

Experimental

The chemical composition of the steel used in this work is given in **Table 1**. The high C content was used to reduce the transformation temperature and obtain the nanoscale bainitic ferrite. The Si was enough to prevent the precipitation of cementite. The Mn and Cr improved the stability of austenite and the Mo eliminated the brittleness. The high Ti content was utilized to refine the austenite grain and to form Ti carbides during intermediate temperature service. The as-received steel was homogenized at 1200 °C for 48 hrs in a vacuum furnace followed by furnace cooling to room temperature. All homogenized specimens were austenitized at 1000 °C. One set of samples went through an initial bainitic transformation at 300 °C for 24 hrs followed by second bainitic transformation at 200 °C for 48 and 96 hrs. Another set of the austenitised samples went through the same bainitic transformation at 300 °C for 24 hrs first and then were kept at 450 °C for 0.5 and 2 hrs, before water quenching to room temperature (B+P+Q).

Scanning electron microscope (Sirion 200) and transmission electron microscope (JEM 2010 HT) were used to examine the microstructures and also to determine the distribution, size and morphology of the retained austenite, bainitic ferrite and martensite constituent phases. The thickness t of lath-shape phases was determined by measuring the mean linear intercept $L = \pi t / 2$ in a direction normal to the each set of plates¹³. All specimens for microstructure observations were ground and polished using standard techniques and etched in 4 vol% nital solution. TEM specimens were machined into 3 mm diameter rods, which were sliced into 100 µm thick discs. Each slice disc was ground down to 50 µm in thickness by using 2000 grit silicon carbide paper, followed by electro-polishing at 50 V by using a twin-jet unit electro-polisher at room temperature. The electrolyte consisted of 5 % perchloric acid, 15 % glycerol and 80 % methanol.

A number of square samples (10 mm×10 mm×2 mm) were ground, polished and slightly etched in 4 vol% nital for the phase characterization by X-ray diffractometry (Xpert Pro MPD, operating at 40 kV and 45 mA, with Cu K α radiation). The 2θ scanning angles were varied from 20° to 100° with a stepping angle of 0.03342°. Finally, the volume fraction of retained austenite was calculated by measuring the

integrated intensities of the (111), (200), (220) and (311) austenite peaks, and comparing them with the (110), (002), (112) and (022) bainite peaks¹⁴⁻¹⁵, with an error of about ± 0.015 vol%. The carbon concentration in austenite was estimated from the measured lattice parameters¹⁵⁻¹⁶ with an accuracy of about ± 0.10 wt%. Each hardness value, reported in this work, is the average of at least ten Vickers tests (1 kg).

Results

Fig. 1 shows the microstructure of a sample after a single-stage isothermal transformation at 300°C for 24 hrs. The high magnification SEM and TEM micrographs show that the microstructure consists of nano-size bainitic-ferrite plates (~100 nm) and some retained austenite. The retained austenite appears as ultrafine (<100 nm) films or micron-size blocks (>1000 nm) between the bainite sheaves.

Fig. 2 shows optical and SEM micrographs of the samples which went through a two-stage bainitic transformation (B+B) and those subjected to bainitic transformation then partitioning and finally quenching stages (B+P+Q). The absence of large blocks of retained austenite in the samples subjected to partitioning cycle is clear. Nevertheless, **Figs. 2b** and **2d** show the volume fractions of retained austenite in the samples subjected to the two-stage heat treatments (B+B) or three-stage cycle (B+P+Q) are comparable.

The high magnification SEM micrographs in **Fig. 3** show that increasing the bainitic transformation time from 48 to 96 hrs (compare **Figs. 3a** and **3b**) or partitioning time from 0.5 to 2 hrs (compare **Figs. 3c** and **3d**) had no significant effect on the microstructure of the test samples. These micrographs also prove that a long B+B cycle can be replaced by the much shorter B+P+Q cycle without compromising the resultant microstructure. This is an important finding in terms of potential economic benefits of the B+P+Q cycle, developed in this work.

The thicknesses of the retained austenite blocks are given in **Table 2**. In the samples with single-stage heat treatment (300 °C for 24 hrs), the thickness of the

retained austenite blocks was just under 3000 nm. In the samples subjected to 2 stage, B+B and 3 stage B+P+Q thermal cycles, the thickness of the retained austenite blocks was reduced noticeably. **Table 2** also shows that increasing the treatment time in B+B samples or increasing partitioning time in B+P+Q samples further reduced the thickness of retained austenite blocks.

The reference sample which was treated in a single-stage bainitic transformation had an average hardness of 454 HV1 and contained 44 vol% retained austenite. As expected, the B+B and B+P+Q treated samples had higher hardness (532 and 540 HV1, respectively) and lower amounts of retained austenite (23 and 28 vol%, respectively), compared to the reference sample. It was also noticed that the longer the bainitic transformation or partitioning time, the higher the hardness. In addition, the B+B and B+P+Q treated samples had slightly higher carbon contents in their austenite grains compared with those in the reference sample (2.02~2.14 wt% in B+B, 1.83~1.96 wt% in B+P+Q and 1.79 wt% in the reference sample).

Discussion

The experimental outcome of this work was checked against the general theory of phase transformation in steel. MUCG83.Mod program [17] was used to estimate the free energy available for the transformation of retained austenite to ferritic/bainitic phases as a function of carbon content in the retained austenite. Using the same program, the effect of carbon content on the corresponding TTT phase diagram was also modeled. **Fig. 4** shows that the driving force for decomposition of the retained austenite is reduced as its carbon content is increased. Consequently, the C-curve is shifted to the right hand side of the TTT-diagram. In the other words, the higher carbon content in the retained austenite is longer in the bainitic transformation time.

In all samples, some bainitic ferrite plates and carbon-enriched untransformed retained austenite were evolved in the course of initial isothermal transformation at 300°C. Further transformation of the carbon-enriched retained austenite was possible either by keeping the sample at lower temperature to trigger a secondary bainitic

transformation (B+B) or quenching it in water to produce fine martensitic plates after a short carbon homogenization period at 450°C (B+P+Q). In the second stage of the two-stage bainitic transformation (B+B), some nano-scale bainitic sheaves were formed from the untransformed austenite as a result of keeping the steel at a lower temperature (**Fig. 5a**). Whereas in the B+P+Q process, the leftover austenite from the first stage, transformed to fine plates of martensite during the final quenching stage (**Fig. 5b**). The fresh bainite or martensite plates are very fine due to the large driving forces¹⁸ available for the decomposition of carbon-enriched retained austenite at low temperatures.

Fig. 5b shows the thickness of martensitic plates in a B+P+Q sample is about 10~20 nm, which is much thinner than ~100 nm for the bainitic ferrite plates in a bainite sample. Consequently, large blocks of retained austenite were fragmented and divided by the newly formed martensite sheaves; hence a much finer microstructure was obtained. **Fig. 6** schematically shows the microstructural evolution of prior austenite and the change in its carbon content during various heat treatments in a micro/nano-structured bainitic steel. Unlike the diffusionless martensitic transformation, it is commonly accepted that carbon partitioning occurs during the bainitic transformation. According to the T⁰ line theory, the maximum carbon concentration in austenite under para-equilibrium condition decreases when the temperature increases. In the present work, Si was added into the steel to suppress the precipitation of cementite during bainite transformation at lower temperature. The bainitic ferrite formed at lower temperature is a solid solution with saturated carbon atoms. During the followed partitioning at higher temperature (450°C), the carbon atoms have a higher mobility and can continue to diffuse from bainitic ferrite to form carbon-enriched austenite and thus improve the stability of austenite¹⁹. It is also possible that the carbon concentration in retained austenite is homogenised during the partitioning stage. This could stabilise the carbon-lean blocky retained austenite. The thin retained austenite plates entrapped between sub-units of bainitic ferrites have higher carbon contents than the blocky retained austenite located between the sheaves of bainite²⁰. Both types of retained austenite could transform to bainite or martensite.

Haseet *al.* ⁷ reported that the austenite blocks were subdivided by newly generated bainite in various orientations following a B+B thermal treatment in a micro/nano-structured bainitic steel. Moreover, the refined retained austenite through the B+B treatment, significantly improved the toughness of the micro/nano-structured bainitic steel. Our previous work ²¹ has revealed that multi-step micro/nano-structured bainitic transformation can improve the strength, ductility as well as toughness owing to the refinement and reduction of blocky martensite/retained austenite constituents in medium carbon steels.

Most recently, Luo *al.* ²² reported that a multi-phase microstructure contained martensite and nanostructured bainite (nanoscale bainitic ferrite and austenite) was obtained by QPB (quenching - partitioning & bainitic transformation) process of bainitic steel, and the blocky austenite was rare after the novel quench and partitioning treatment. In the present work, fine fresh martensite plates produced by the B+P+Q treatment exhibit features and properties similar to those of the fine bainite plates produced by the B+B treatment. Nevertheless, B+P+Q seem to be a more viable process due to shorter processing time required as compared to that in a B+B cycle. A comprehensive investigation on the mechanical properties of micro/nano-structured bainitic steels produced by B+B and B+P+Q processes will be carried out in the future work.

Conclusions

The effect of a two-stage bainite transformation (B+B) and bainite transformation followed by partitioning/quenching process (B+P+Q) on the microstructure of a micro/nano-structured bainitic steel was investigated. The refinement of the retained austenite occurred due to the formation of ultrafine bainite or ultrafine martensitic plates in the B+B and B+P+Q treated samples, respectively. The level of austenite refinement and increase in the hardness were comparable using these thermal cycles although, the newly developed B+P+Q approach had a much shorter cycle time *e.g.* hours compared to days.

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Table 1 Chemical composition of investigated steel (wt.%)

C	Si	Mn	Cr	Mo	Ti	Fe
0.95	0.91	1.30	2.30	0.99	0.17	Bal.

Table 2 Thickness (RA), volume fraction (V_γ), carbon content (C_γ) of retained austenite, and hardness of steel investigated in this work. BT and PT represent the secondary bainite transformation and partitioning temperatures, respectively.

Samples	Single-stage	B+B (BT=200 °C)		B+P+Q (PT=450 °C)	
		2 days	4 days	0.5 hrs	2 hrs
Block RA, nm	2970±1520	2690±1320	1880±640	2770±1030	1990±560
V_γ , vol%	44	34	23	38	28
C_γ , wt%	1.79	2.02	2.14	1.96	1.83
Hardness, HV1	454±8	476±14	532±12	506±14	540±12

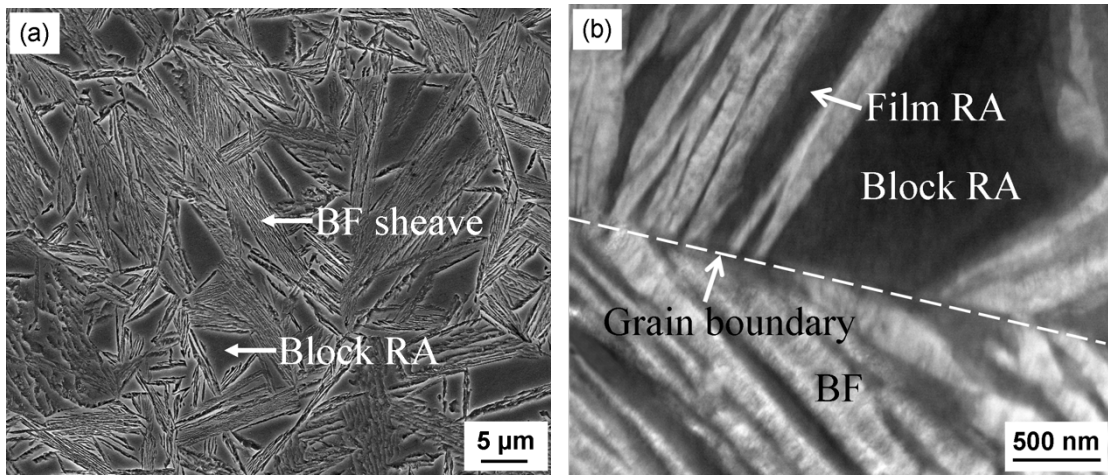


Fig. 1: (a) SEM and (b) TEM micrographs of a reference steel sample after bainitic transformation at 300 °C for 24 hrs (single-stage treatment).

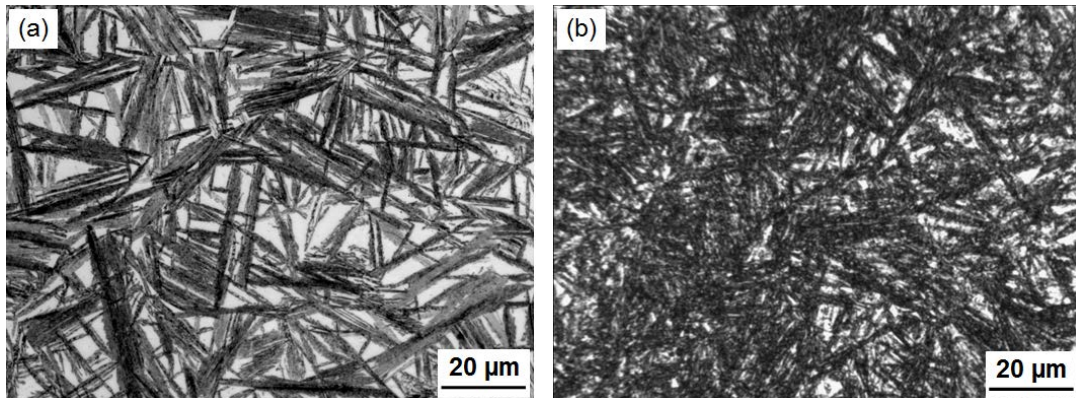


Fig. 2: Optical micrographs show (a) steel sample transformed to bainite by a single-stage heat treatment and (b) sample subjected to the same bainitic transformation followed by a partitioning /quenching cycle – note reduction in the volume of retained austenite blocks (bright phases) in the latter.

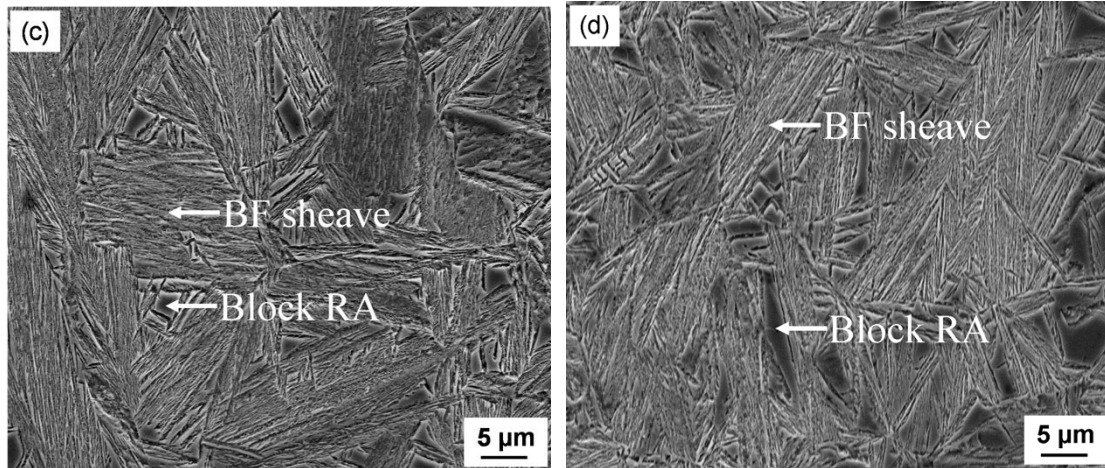


Fig. 3: SEM micrographs show comparable microstructures of steel samples made by (a) two stage bainitic transformation (B+B) and (b) a bainitic transformation followed by a partitioning /quenching cycle (B+P+Q) – see text for process conditions.

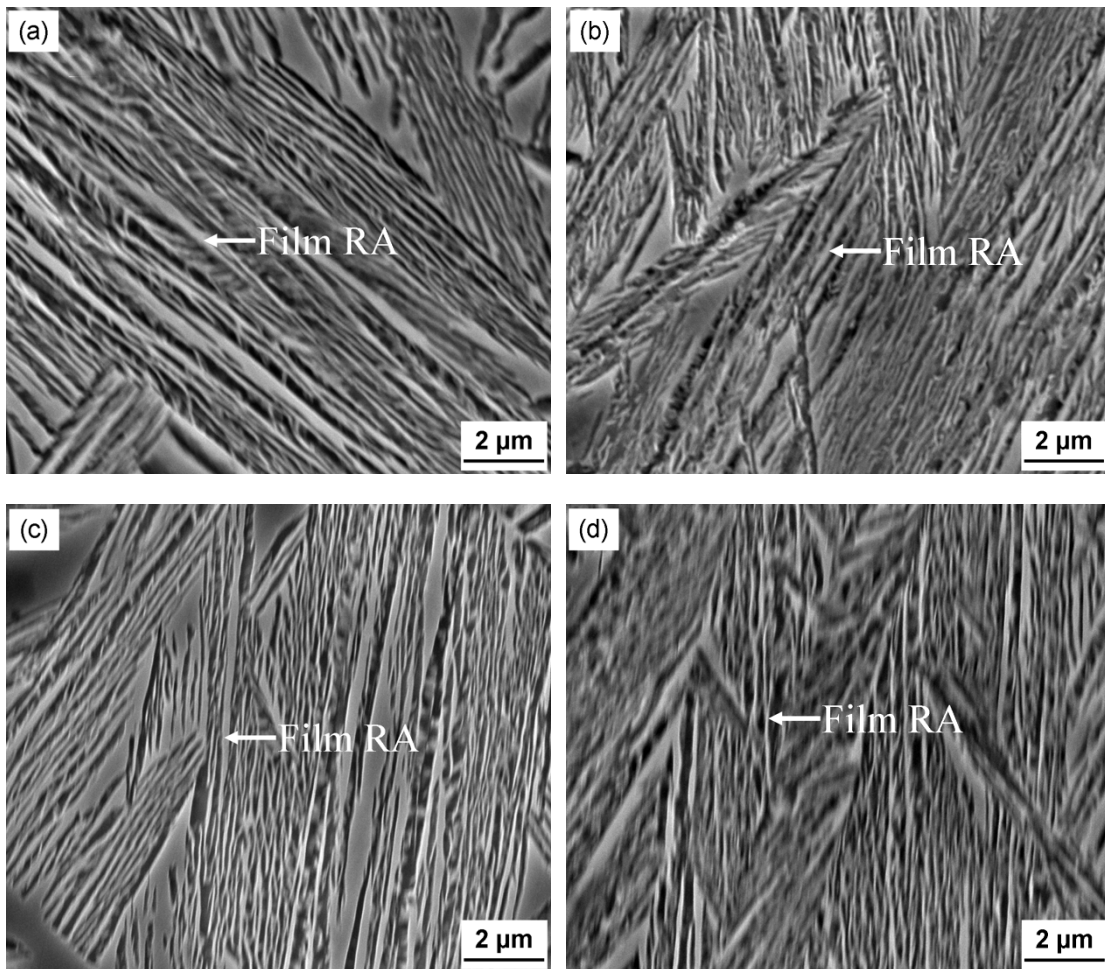


Fig. 4 SEM micrographs show sheaves of bainite are very similar in samples made in this work (a) B+B in 48 hrs, (b) B+B in 96 hrs, (c) B+P (0.5 hr)+Q and (d) B+P (2 hrs)+Q – see text for process conditions.

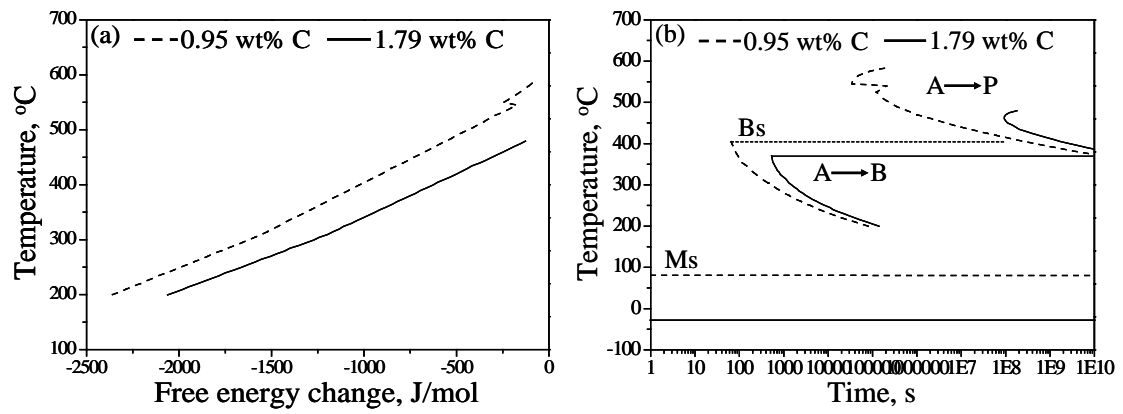


Fig. 5: Calculated effect of carbon content in untransformed austenite on (a) free energy change and (b) TTT phase diagram of the investigated steel.

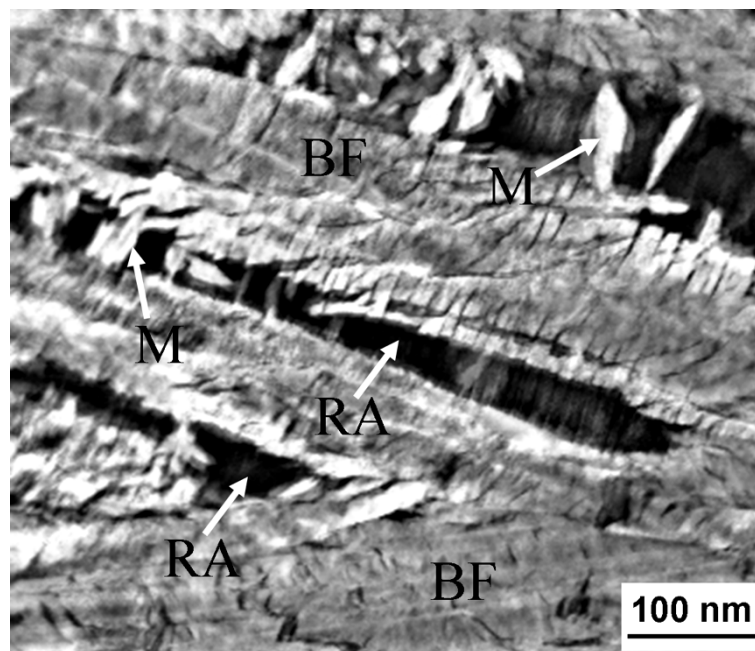


Fig. 6 TEM micrograph of a water quenched super-bainitic steel shows progressive formation of very fine martensite plates into retained austenite sheaves.

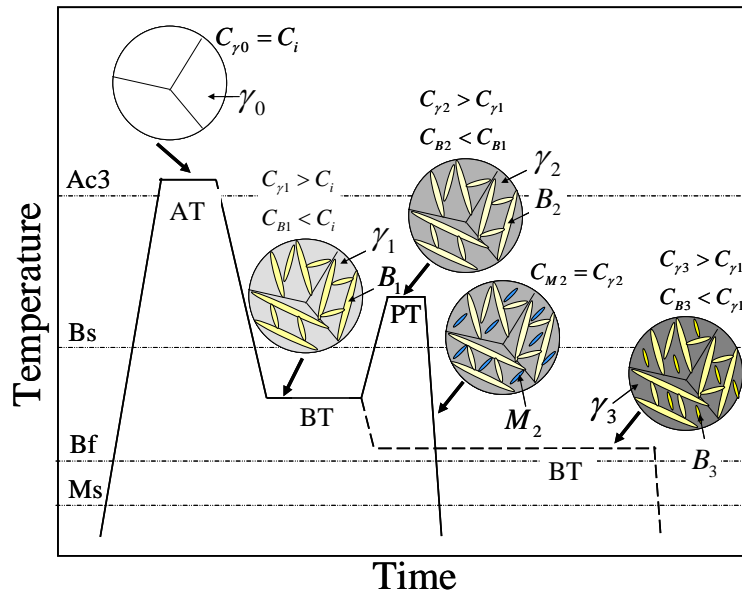


Fig. 7 Schematic diagram shows expected microstructures and carbon contents following the B+B and B+P+Q processes of a steel containing austenite (γ), bainite (B) and martensite (M). C_i , C_γ , C_B and C_M represent the carbon contents of the bulk alloy, austenite, bainite and martensite, respectively. B_1 , B_2 and B_3 represent the temperatures of first and second bainitic transformations and PT is the partitioning temperature just before quenching in water.