

## Effect of Warm Rolling on Transformation Kinetics, Microstructure and Mechanical Properties of Nanostructured Bainite: Postprint

**Authors:** He Jianguo, Zhao Aimin, Huang Yao, Zhichao, Zhao Fuqing

**Date:** 2023-03-18T00:00:00+00:00

### Abstract

Nanobainitic steel was prepared using a warm rolling plus isothermal heat treatment process, and the effect of deformation temperature on the nanobainitic transformation rate was investigated. The results indicate that the bainitic isothermal transformation time of deformed supercooled austenite at 503 K was reduced from 50 h for conventional isothermal quenching to 20 h, and the nanobainitic steel exhibited a tensile strength of 2127 MPa and an elongation of 4%. Deformation of supercooled austenite within the experimental temperature range promoted the nanobainitic transformation, and the transformation rate increased with decreasing deformation temperature. When the deformation amount of supercooled austenite exceeded 30%, the retained austenite microstructure was significantly refined, and blocky retained austenite was completely transformed into a film-like morphology. The warm rolling process can accelerate low-temperature bainitic transformation without deteriorating other mechanical properties, thereby reducing heat treatment time and production costs.

### Full Text

## Effect of Warm Rolling Process on Phase Transformation, Microstructure and Mechanical Properties of Nano-bainite Steel

HE Jianguo<sup>1, 2</sup>, ZHAO Aimin<sup>1, 2</sup>, HUANG Yao<sup>3</sup>, ZHI Chao<sup>1, 2</sup>, ZHAO Fuqing<sup>1, 2</sup>

<sup>1</sup> Metallurgical Engineering Research Institute, University of Science and Technology Beijing, Beijing 100083, China

<sup>2</sup> Beijing Laboratory for Modern Transportation Advanced Metal Materials and

Processing Technology, Beijing 100083, China

<sup>3</sup> China Electric Power Research Institute, Beijing 100192, China

*Supported by National Natural Science Foundation of China Nos. 51271035 & 51371032.*

**Abstract:** Nanostructured bainite steel with an ultimate tensile strength of 2127 MPa and elongation of 4% was successfully produced through warm rolling combined with isothermal heat treatment. The influence of deformation temperature on nano-bainite transformation kinetics was systematically investigated. The results demonstrate that with appropriate warm deformation, the time required for supercooled austenite transformation into bainite at 503 K can be reduced from 50 h (conventional austempering) to just 20 h. Deformation of supercooled austenite across the entire experimental temperature range promoted nano-bainite transformation, with the transformation rate increasing as deformation temperature decreased. When the deformation exceeded 30%, the retained austenite microstructure was significantly refined, and all blocky retained austenite transformed into a film-like morphology. The warm rolling process can accelerate low-temperature bainite transformation without deteriorating other mechanical properties, thereby shortening heat treatment duration and reducing production costs.

**Keywords:** metallic materials, nanobainite, warm rolling, transformation kinetics, microstructure, mechanical properties

---

## 1. Introduction

Nanostructured bainitic steel, developed by Bhadeshia and colleagues based on transformation strengthening theory, has attracted considerable attention due to its exceptional combination of ultra-high tensile strength ( $>2000$  MPa) and fracture toughness ( $30 \text{ MPa} \cdot \text{m}^{1/2}$ ) [1-5]. The properties of nano-bainitic steel are closely related to the thickness of bainitic ferrite laths and the morphology of retained austenite—thinner laths yield higher strength, while reduced blocky retained austenite improves toughness [6-9]. Previous studies have shown that nanometer-thick bainitic plates can only be obtained through isothermal treatment at very low temperatures (473-523 K), but such low-temperature processing requires substantial time and cost [4]. While application of large compressive stresses (200 MPa) can significantly enhance bainite transformation kinetics [10], this approach is difficult to implement for industrial-scale plate production. Although low-level deformation can accelerate martensitic transformation at low temperatures, the optimal deformation temperature remains controversial [11,12].

Current research on nano-bainitic steel has primarily focused on microstructural characterization and isothermal processing parameters [13-15]. Therefore, investigating the effects of deformation temperature and other parameters on the

transformation kinetics and microstructural properties of ultra-high-strength nano-bainitic steel holds both theoretical significance and practical guidance for thermomechanical processing and steel production. This study employs warm rolling to accelerate low-temperature bainite transformation kinetics and examines its influence on the transformation rate, microstructure, and properties of nano-bainitic steel.

## 2. Experimental Methods

The experimental steel had a composition (mass fraction, %) of Fe-0.91C-1.65Si-2.07Mn-1.26Cr-0.25Mo-1.56Co-0.78Al and was melted in a vacuum induction furnace. The ingot was cast as a 50 mm diameter  $\times$  600 mm cylinder, then forged into 80 mm  $\times$  80 mm  $\times$  80 mm billets. All billets underwent homogenization annealing at 1552 K for 24 h, followed by hot rolling with a starting temperature of 1273 K and finishing temperature of 1173 K, and finally air-cooled to produce 6 mm thick plates for subsequent warm rolling.

Thermal expansion experiments were conducted using a DIL805 high-temperature dilatometer on cylindrical specimens (4 mm diameter  $\times$  10 mm length) to study the transformation kinetics of nano-bainitic steel during conventional isothermal treatment at 503 K for 60 h. Gleeble 3500 thermomechanical simulations were performed on  $\Phi$ 6 mm  $\times$  15 mm specimens. Samples were austenitized at 1173 K for 15 min, rapidly cooled to various temperatures between 503-873 K for 20% compression deformation, then quenched to 503 K and held for 6 h. Real-time dilatometric data were recorded during holding to investigate the effect of austenite deformation temperature on low-temperature bainite transformation kinetics. The experimental procedure is schematically illustrated in [Figure 1: see original paper].

Based on the in-situ thermomechanical simulation results, warm rolling processes were designed with varying total reductions achieved by changing the number of rolling passes within the same temperature range to study the effect of strain on nano-bainitic microstructure and properties. The rolling schedule is shown in [Figure 2: see original paper]. The process involved heating 6 mm thick hot-rolled plates to 1173 K, austenitizing, rapidly cooling to 873 K to initiate warm rolling with a pass reduction of 0.5 mm, then cooling to 503 K and isothermally holding in a furnace at 503 K for 20 h.

Metallographic specimens were sectioned from the mid-thickness of warm-rolled plates along the rolling direction. Microstructural observation was performed using a ZEISS SUPRA55 field-emission scanning electron microscope (SEM). Transmission electron microscopy (TEM) samples were prepared from the same location, mechanically ground to 50  $\mu$ m thickness, then electropolished at room temperature using a twin-jet polisher with 5% perchloric acid-ethanol solution at 20-30 V. Fine microstructural details were examined using a JEOL JEM-2000 TEM. Tensile properties were measured at room temperature on specimens with gauge dimensions of 25 mm  $\times$  6 mm  $\times$  2 mm at a crosshead speed of 1 mm/min.

Vickers hardness was measured using a 430SVD tester with a 9.8 N load, with average values taken from five measurements. The volume fraction of retained austenite and its carbon content were determined using the method described in reference [17].

## 2.1 Low-Temperature Isothermal Transformation Rate of Nano-bainite

[Figure 3: see original paper] presents the dilatometric curves recorded by the high-temperature dilatometer during isothermal nano-bainite transformation. The expansion curve (L-change) shows no significant change during the first 5 h of holding, after which the expansion gradually increases, indicating the onset of bainite transformation. The curve approaches a plateau after 50 h, suggesting completion of bainite transformation. These results indicate that bainite transformation at 503 K requires approximately 50 h, with an incubation period of about 5 h.

Differentiating the expansion-time curve yields the expansion rate-time curve shown in [Figure 3: see original paper]. The transformation rate reaches a maximum of  $1.24 \times 10^{-3} \text{ m} \cdot \text{s}^{-1}$  at 10 h 26 min, then gradually decreases with prolonged holding time. After 50 h, the expansion rate approaches zero, confirming that bainite transformation ceases. In summary, conventional isothermal quenching of high-carbon, silicon-rich nano-bainitic steel requires at least 50 h to complete bainite transformation at 503 K.

## 2.2 Effect of Deformation Temperature on Low-Temperature Transformation Kinetics

To shorten the isothermal holding time in the bainite region, this study proposes a novel process involving warm rolling prior to isothermal treatment. Given existing controversies regarding the effect of deformation temperature on bainite transformation kinetics, Gleeble thermomechanical simulation was first employed to investigate nano-bainite transformation kinetics after austenite deformation at various temperatures, thereby establishing appropriate warm rolling parameters.

[Figure 4: see original paper] shows the bainite transformation kinetics curves at 503 K after compressing supercooled austenite 20% at different temperatures. The dilatometric curves reveal that deformation at 873 K has minimal effect on transformation rate, which remains essentially consistent with undeformed material. However, as the deformation temperature of supercooled austenite decreases, the bainite transformation incubation period shortens markedly: after deformation at 773 K and 673 K, the incubation periods are 3 h and 0.7 h, respectively, while deformation at 573 K and 503 K reduces the incubation period to less than 0.5 h.

[Figure 5: see original paper] presents the bainite transformation rate-time curves for supercooled austenite after compression deformation and isothermal

holding at 503 K. The “No-” curve represents the transformation rate of undeformed material, which remains zero throughout the holding period, indicating no bainite transformation occurred. In contrast, the curve for material deformed at 873 K shows a distinct change at 5 h 25 min, with the transformation rate increasing sharply from zero to  $4 \times 10^{-4} \text{ m} \cdot \text{s}^{-1}$ , marking the end of the incubation period and onset of transformation. These results demonstrate that compressive deformation of supercooled austenite at 873 K can shorten the bainite transformation incubation period. Compared with other curves in [Figure 5: see original paper], deformation at lower temperatures (503 K or 573 K) exerts a more pronounced effect on transformation rate, with the deformed supercooled austenite reaching maximum transformation rates after 3 h of holding, measuring  $4 \times 10^{-3} \text{ m} \cdot \text{s}^{-1}$  and  $3 \times 10^{-3} \text{ m} \cdot \text{s}^{-1}$ , respectively.

In summary, deformation processing within the 503-873 K temperature range can accelerate low-temperature isothermal bainite transformation at 503 K, with transformation completing in over 6 h. Consequently, the warm rolling process was designed with a starting temperature of 873 K and finishing temperature of 503 K. Additionally, to investigate the effect of different deformation levels at the same rolling temperature on low-temperature nano-bainitic microstructure and properties, three warm rolling schedules with total reductions of 25%, 33%, and 50% were designed.

### 2.3 Microstructure of Warm-Rolled Nano-bainitic Steel

The nano-bainitic microstructures produced by different processes are shown in [Figure 6: see original paper]. [Figure 6: see original paper]a presents the typical room-temperature microstructure of conventionally austempered nano-bainitic steel after 2 weeks of isothermal transformation at 503 K, while [Figure 6: see original paper]b-d show microstructures after isothermal holding at 503 K for 20 h following warm rolling with 3 passes (25% reduction), 4 passes (33% reduction), and 6 passes (50% reduction), respectively. All three warm-rolled processes yielded fully nano-bainitic microstructures without coarse blocky retained austenite, indicating that low-temperature bainite transformation can be completed within 20 h after warm rolling.

[Figure 6: see original paper]a illustrates the typical nano-bainitic morphology, featuring blocky untransformed austenite (average intercept size  $\sim 3 \text{ }\mu\text{m}$ ) distributed in the bainitic matrix, with these retained austenite islands exhibiting triangular or quadrangular shapes [10,16]. In contrast, [Figure 6: see original paper]b-d reveal that blocky untransformed austenite disappears after warm rolling, with nano-bainitic lath thickness decreasing and becoming distributed throughout the matrix. Since austenite deformation occurred below the recrystallization temperature, the deformed austenite could not recrystallize and retained its elongated grain structure from warm rolling. When total reductions reached 33% and 50%, the microstructure became extremely refined, as shown in [Figure 6: see original paper]c and d. The inter-lath regions consist of film-like retained austenite with even finer thickness.

The fine structure of warm-rolled nano-bainite is shown in [Figure 7: see original paper]. The microstructure consists of carbide-free bainite. Unlike conventional lower bainite, nano-bainite comprises alternating lamellae of bainitic ferrite and film-like retained austenite. The TEM images in [Figure 7: see original paper], corresponding to the microstructures in [Figure 6: see original paper]c and d, show bainitic ferrite laths (white regions) approximately 50 nm wide. With increasing deformation, the bainitic laths become thinner.

#### 2.4 Mechanical Properties of Warm-Rolled Nano-bainitic Steel

The mechanical properties of warm-rolled nano-bainitic steel compared with conventionally austempered material are listed in . As a reference, conventionally austempered nano-bainitic steel (Aus) was prepared with extended isothermal holding at 503 K for 2 weeks to ensure complete austenite transformation, yielding a tensile strength of 2040 MPa, total elongation of 6.1%, and retained austenite content of 32.01%. When the total warm rolling reduction reached 50%, the material achieved the highest tensile strength of 2127 MPa with 4% total elongation and 34.49% retained austenite. Combined with hardness data and the microstructural analysis above, although all three rolling processes produced fully nano-bainitic microstructures, increasing warm rolling reduction refined the microstructure further and enhanced austenite work hardening, manifested as improved tensile strength and hardness.

### 3. Discussion

The ultra-high strength of nano-bainitic steel originates from its ultra-refined microstructure, with strength strictly correlated to bainitic ferrite lath thickness [10]. When bainitic ferrite lath thickness increases from 20 nm to 100 nm, tensile strength decreases from 2300 MPa to 1800 MPa, while elongation increases from 4% to 20% with increasing retained austenite lath thickness [6-8]. These relationships hold only when the matrix consists entirely of nano-bainite. Incomplete bainite transformation leads to metastable retained austenite transforming to martensite upon cooling to room temperature, introducing brittle phases that reduce both strength and ductility [9]. Based on the microstructural evidence and mechanical properties presented earlier, low-temperature nano-bainite transformation is essentially complete within 20 h after warm rolling, accounting for the superior mechanical properties. This confirms that warm rolling significantly accelerates nano-bainite transformation without compromising mechanical performance.

X-ray diffraction was used to quantify retained austenite content and calculate its carbon concentration via lattice parameter analysis, with results shown in [Figure 8: see original paper]. All four processes produced similar retained austenite contents, though slight increases were observed when warm rolling reductions exceeded 33%. Literature analysis suggests that compressive deformation of supercooled austenite at higher temperatures ( $>873$  K) may increase mechanical stability, stabilizing more austenite at room temperature [12].

At 25% total reduction (WR3 process), the retained austenite content was 32.04%, identical to conventionally austempered material. This indicates maximum bainite transformation was achieved, with no further transformation upon extended holding. However, both strength and hardness were lower compared with conventional austempering. Microstructural analysis reveals that although blocky retained austenite was finer ( $\sim 0.5 \mu\text{m}$ ) after warm rolling, its non-uniform distribution and extensive agglomeration degraded comprehensive mechanical properties [17].

At 33% total reduction (WR4 process), blocky retained austenite disappeared completely, leaving only film-like retained austenite and nano-bainite ([Figure 6: see original paper]c). X-ray diffraction results show the highest retained austenite content of 37.08% but the lowest carbon concentration (1.25%) among all processes, yielding excellent comprehensive properties: tensile strength of 1890 MPa and total elongation of 14.4%.

At 50% total reduction (WR5 process), nano-bainitic ferrite lath thickness of 20-50 nm produced the maximum strength of 2127 MPa, consistent with literature reports [18]. This demonstrates that the combined warm rolling and isothermal process can shorten bainite region holding time without sacrificing mechanical properties.

Since the supercooled austenite deformation temperatures in this study were relatively low—the second rolling pass already occurring below 773 K, with most deformation below 673 K—the effect of low-temperature deformation on austenite mechanical stability exhibits new characteristics. The WR4 process yielded the highest retained austenite content (37.08%) with 1.25% carbon, while the Aus process contained 32.01% retained austenite with 1.72% carbon, WR3 contained 32.04% with 1.50% carbon, and WR6 contained 34.49% with 1.74% carbon. Literature [17] indicates that when total retained austenite content is similar, film-like austenite with low carbon content (50-150 nm width) provides good ductility, consistent with our results.

The mechanism by which warm rolling deformation significantly accelerates nano-bainite formation can be attributed to two factors: (1) deformation of supercooled austenite at lower temperatures accumulates substantial dislocations that provide nucleation sites for transformation, and (2) the stored deformation energy in low-temperature austenite is greater than that at high temperatures for equivalent strain levels, thereby increasing the driving force for low-temperature bainite transformation. Both factors contribute to shortened incubation periods and accelerated transformation rates in deformed supercooled austenite [16].

#### 4. Conclusions

1. Thermomechanical processing significantly accelerates low-temperature nano-bainite transformation in the experimental steel. The improved warm rolling-plus-isothermal process can produce nano-bainitic steel

with 2127 MPa strength and 650 HV hardness within 20 h, whereas conventional austempering requires at least 50 h to achieve comparable strength levels.

2. Plastic deformation of supercooled austenite within the 873-503 K temperature range accelerates low-temperature nano-bainite transformation kinetics. Deformation at higher temperatures shortens the transformation incubation period, while deformation at lower temperatures—with greater stored energy—both shortens incubation and accelerates bainite transformation, resulting in finer microstructures.
3. The isothermal bainite transformation rate in the experimental steel reaches its maximum after 10 h 26 min of holding, with transformation essentially complete after 50 h. Plastic deformation of supercooled austenite below 673 K reduces the bainite incubation period from 6 h to less than 0.5 h and enables the transformation rate to reach its maximum within 3 h, demonstrating the remarkable accelerating effect of warm rolling on low-temperature nano-bainite transformation.

## References

1. F. G. Caballero, H. K. D. H. Bhadeshia, *Design of novel high strength bainitic steels—Part 1*, Materials Science and Technology, 17(5), 512 (2001).
2. F. G. Caballero, H. K. D. H. Bhadeshia, K. Mawella, D. G. Jones and P. Brown, *Design of novel high strength bainitic steels—Part 2*, Materials Science and Technology, 17(5), 517 (2001).
3. F. G. Caballero, H. K. D. H. Bhadeshia, K. Mawella, D. G. Jones and P. Brown, *Very strong low temperature bainite*, Materials Science and Technology, 18(3), 279 (2002).
4. C. Garcia Mateo, F. G. Caballero, H. K. D. H. Bhadeshia, *Development of hard bainite*, ISIJ International, 43, 1238 (2003).
5. F. G. Caballero, H. K. D. H. Bhadeshia, *Very strong bainite*, Current Opinion in Solid State and Materials Science, 8(3), 251 (2004).
6. C. G. Mateo, H. K. D. H. Bhadeshia, F. G. Caballero, *Mechanical properties of low-temperature bainite*, Materials Science Forum, 500, 495 (2005).
7. H. K. D. H. Bhadeshia, *Large chunks of very strong steel*, Materials Science and Technology, 21(11), 1293 (2005).
8. H. K. D. H. Bhadeshia, *Nanostructured bainite*, Proceedings of the Royal Society A: Mathematical, Physical and Engineering Science, 466(2113), 3 (2010).
9. H. K. D. H. Bhadeshia, *The first bulk nanostructured metal*, Science and Technology of Advanced Materials, 14(1), 14202 (2013).

10. K. Hase, C. G. Mateo, H. K. D. H. Bhadeshia, *Bainite formation influenced by large stress*, Materials Science and Technology, 20(12), 1499 (2004).
11. W. Gong, Y. Tomota, M. S. Koo and Y. Adachi, *Effect of ausforming on nanobainite steel*, Scripta Materialia, 63(8), 819 (2010).
12. W. Gong, Y. Tomota, Y. Adachi, A. M. Paradowska, J. F. Kelleher and S. Y. Zhang, *Effects of ausforming temperature on bainite transformation, microstructure and variant selection in nanobainite steel*, Acta Materialia, 61(11), 4142 (2013).
13. F. G. Caballero, M. K. Miller, C. G. Mateo, *Atom probe tomography analysis of precipitation during tempering of a nanostructured bainitic steel*, Metallurgical and Materials Transactions A, 42(12), 3660 (2011).
14. M. N. Yoozbashi, S. Yazdani and T. S. Wang, *Design of a new nanostructured, high-Si bainitic steel with lower cost production*, Materials & Design, 32(6), 3248 (2011).
15. S. Khare, K. Lee, H. K. D. H. Bhadeshia, *Carbide-Free bainite: compromise between rate of transformation and properties*, Metallurgical and Materials Transactions A, 41(4), 922 (2010).
16. Y. Huang, A. M. Zhao, J. G. He, X. P. Wang, Z. G. Wang and L. Qi, *Microstructure, crystallography and nucleation mechanism of NANO-BAIN steel*, International Journal of Minerals, Metallurgy, and Materials, 20(12), 1155 (2013).
17. C. G. Mateo, M. Peet, F. G. Caballero and H. K. D. H. Bhadeshia, *Tempering of hard mixture of bainitic ferrite and austenite*, Materials Science and Technology, 20(7), 814 (2004).
18. C. G. Mateo, F. G. Caballero, *The role of retained austenite on tensile properties of steels with bainitic microstructures*, Materials Transactions, 46(8), 1839 (2005).

*Note: Figure translations are in progress. See original paper for figures.*

*Source: ChinaXiv – Machine translation. Verify with original.*