

# Morphology Feature of Nanostructure Bainitic Steel after Tempering Treatment

C. Y. Chen, C. C. Chen, J. S. Lin

**Abstract**—The microstructure characterization of tempered nanocrystalline bainitic steel is investigated in the present study. It is found that two types of plastic relaxation, dislocation debris and nanotwin, occurs in the displacive transformation due to relatively low transformation temperature and high carbon content. Because most carbon atoms trap in the dislocation, high dislocation density can be sustained during the tempering process. More carbides only can be found in the high tempered temperature due to intense recovery progression.

**Keywords**—Nanostructure Bainitic Steel, Tempered, TEM, Nano-Twin, Dislocation Debris, Accommodation.

## I. INTRODUCTION

RECENTLY, nanostructure bainitic steel has attracted a great deal of attention due to their high ultimate tensile strength of about 2.3 GPa combined with very large toughness (30 MPa m<sup>1/2</sup>) and can be produced through very simple and cheap manufacturing process [1], [2]. Therefore the state-of-art of nanostructure bainitic steel has great potential to use in the automobile industry [3], railway lines [4] and armour [5]. This excellent mechanical property is mainly attributed to unusually high dislocation density ( $2 \times 10^{15} \sim 6 \times 10^{15}/\text{m}^2$ ) exists within bainite plate and extremely slender bainite plate (20~40nm thickness) with carbon-enriched thin film retained austenite (10~20nm) dispersed between them [6], [7]. The composition design philosophy of this new generation bainitic steel emphasized that addition of about 2 wt% of silicon to the high carbon steel can avoid precipitation of cementite in the steel due to poor solubility of silicon element in the cementite. Without cementite phase within the bainite microstructure, it can get high toughness because of decrease in void-initiating phase (cementite) in the steel [8]. On the other hand; addition of molybdenum is to prevent temper embrittlement due to phosphorus. Beside above alloy design concept, bainite transformation occurs in this novel nanostructure steel can be depressed to as low as 200°C or lower due to its high carbon content (~1wt%), which is benefit for forming nanoscale crystals of bainite ferrite plate. As pointed out by Chang and Bhadeshia [9], the size of bainite ferrite plate would be influenced heavily by the plastic accommodation ability of bainite plate and its proximity retained austenite. In generally

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speaking, shape change accompanying bainite transformation can be usually accommodated by high dislocation density and other defects [9]-[12].

In the present study, the tempering microstructure of nanocrystalline bainitic steel associated with its plastic accommodation characterization was studied by tradition TEM and high resolution TEM (HRTEM). The results have provided new insight on the microstructure evolution of novel bainitic steel during tempering treatment.

## II. EXPERIMENTAL PROCEDURE

The chemical composition of the alloy studied, measured after homogenization at 1200°C for 2 days in a vacuum chamber, was (wt%) Fe- 0.96 C-1.41 Si-1.93 Mn-1.22 Cr-0.26 Mo-0.10V. Cylindrical samples of 3mm in diameter and 4 cm in length were machined from the homogenized material. A nanostructure bainite steel was obtained by austenitising for 15 min at 1000°C followed by isothermal transformation at 200°C for 10days before quenching into water. The specimens were then tempered at 425°C and 475°C for a variety of time periods.

The samples were mainly characterized by optical microscopy (OM), transmission electron microscopy (TEM, JEM-2000), and field emission gun transmission electron microscopy (FEG-TEM Tecnai F30) equipped with a nanometer probe energy dispersive spectrometer (EDS). A hardness measurement of the specimens to be examined optically was taken using a Vickers hardness tester with a load of 3kg. In order to realize the precipitation status in the ferrite, for each steel and processing condition, measurements from 100 grains were used to plot the final microhardness distribution. Thin foil specimens were prepared for transmission electron microscopy from 0.25mm thick discs slit from the specimens used in the dilatometric experiments. The discs were thinned to 0.5mm by abrasion on silicon carbide paper and then electropolished in a twin jet electropolisher using a solution of 5 vol.% perchloric acid+ 25 vol.% glycerol + 70 vol.% ethanol at -2°C and 30 V potential.

## III. RESULTS AND DISCUSSION

The initial microstructure of nanocrystalline bainitic steel before followed tempering treatment is the outcome of isothermal decomposition of austenite at 200°C for 10 days. Figs. 1 (a) and (b) have shown the optical and transmission electron micrograph consisting of the slender and tiny bainitic ferrite plates separated by thin film retained austenite and large area of blocky retained austenite. The tiny thickness of bainite plates measured from TEM micrographs can be ascribed to high carbon content and extremely low transformation

temperature. Although no carbide precipitation within bainitic ferrite can be observed in this initial microstructure, other studies used advanced atom probe facility had found out carbon enrich region (i.e. cementite) distributed inside the bainitic ferrite plate and to proof the lower bainite structure characteristic [6], [13], [14]. More details of this initial microstructure can be seen in other publications [14].

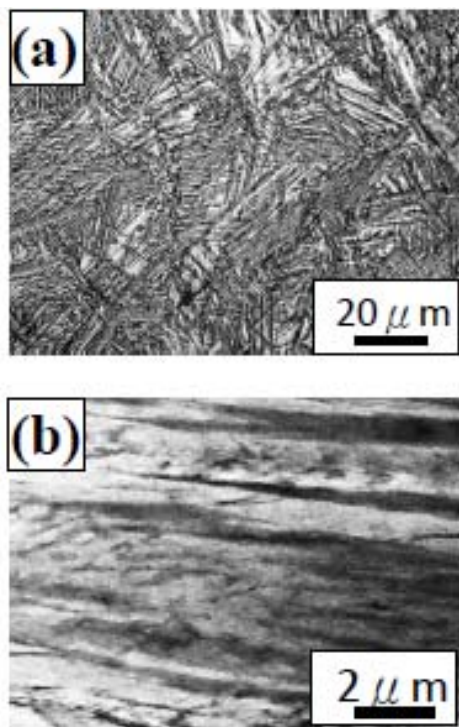


Fig. 1 Micro-image of (a) OM and (b) TEM image of nanostructure bainitic steel formation at 200°C for 10 days

Fig. 2 (a) has shown dislocation debris distributed around the austenite-bainitic ferrite interface. This dislocation debris morphology is the result of the displacive transformation, which causes shape deformation and needs accommodation of surrounding matrix by further plastic relaxation [15]. Figs. 2 (b)-(d) have revealed another accommodation morphology, nanotwin structure with thickness about 10~20nm, dispersed in the retained austenite. This nanotwin structure usually appears in the blocky austenite not in the thin film austenite mainly due to different chemical composition of retained austenite.. Besides nanotwin structure in the blocky austenite, other researches also discovered martensitic transformation occurring in this blocky austenite region due to its relatively high Ms Temperature arising from relatively low carbon content [16].

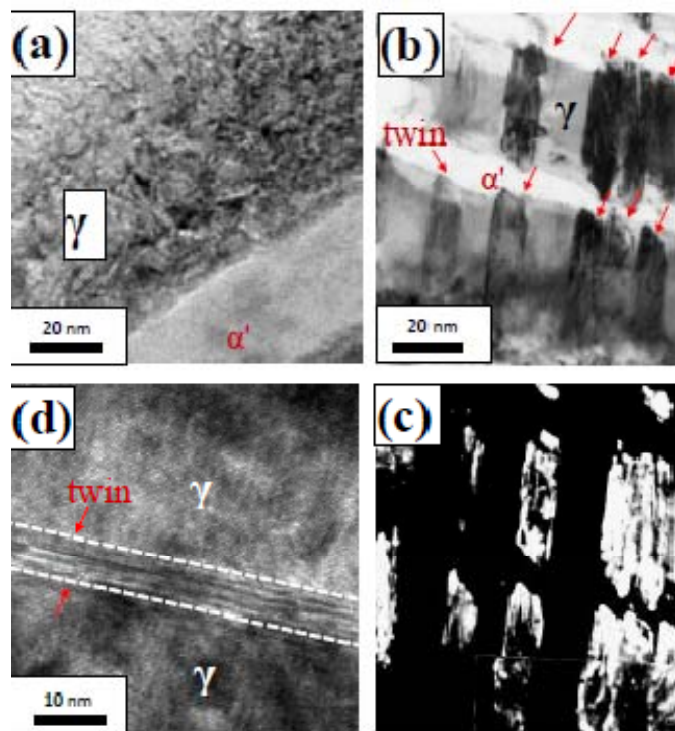


Fig. 2 TEM images of nanostructure bainitic steel transformation at 200°C for 10 days; (a) high magnification TEM bright field image show dislocation debris dispersed around the interface between austenite and ferrite, (b) and (c) bright field image and corresponding dark field image of nanotwin residues in the austenite region, (d) HRTEM image about 8nm of the nanotwin embedded in the austenite region

Fig. 3 shows the morphology of nanostructure bainitic steel after tempering at 475°C for 60min. The cementite and eta carbides precipitate along interphase between the thin film austenite and bainitic ferrite plate. Fig. 4 presents the carbides precipitation in the austenite and bainitic ferrite region in the various tempering condition. Whatever tempering condition it is, fewer precipitation carbides can be found in the bainitic ferrite. However more and large size of precipitation carbide appeared in the interphase boundary between thin film austenite and bainitic ferrite plate during long time tempering at 475°C.

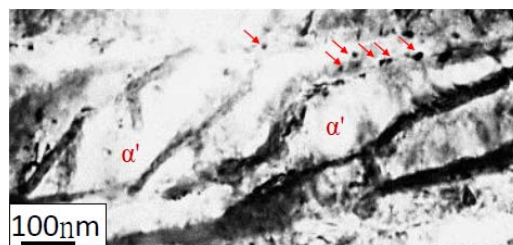


Fig. 3 Bright field TEM image of nanostructure bainitic steel after tempering at 475°C for 60min showing the distribution of cementite and other transition carbides in the interphase between thin film austenite and bainitic ferrite region

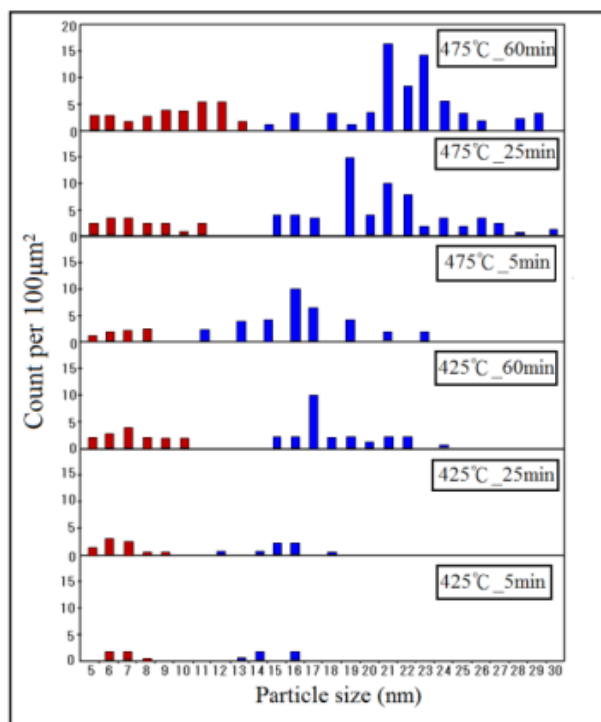


Fig. 4 Size distribution of carbides in the tempered steels showing increase in size and quantity with long tempered time and higher tempered temperature (■ Thin film retained austenite precipitates, ■ Bainitic ferrite plate precipitates)

Fig. 5 reveals the variation of dislocation density within the bainitic ferrite plate at various tempering conditions. With slightly decrease in the dislocation density, almost no recovery phenomenon in the bainite structure can be seen under tempering at 400°C. However, when tempering at 475°C, the trend of diminishing in dislocation density with time implies recovery prevailing in the structure. The similar consequences also discovered in the present study. Releasing carbon atoms from dislocation through moderate temperature tempering can lead to recovery of structure and precipitation of carbides in the bainite plate or thin film austenite region. In addition to cementite and other transition carbides would be detected in the present research, vanadium carbide also found in the bainitic plate after 60 min tempered at 475°C. Figs. 6 (a) and (b) show the vanadium carbide morphology and corresponding EDS analysis result. It is believed that vanadium carbide precipitated directly from ferrite matrix, since substitution atom such as vanadium would not be partitioned into austenite during displacive transformation. Fig. 7 presents the hardness variation with various tempering conditions. The increase in hardness at is mainly due to precipitation of carbide in the matrix.

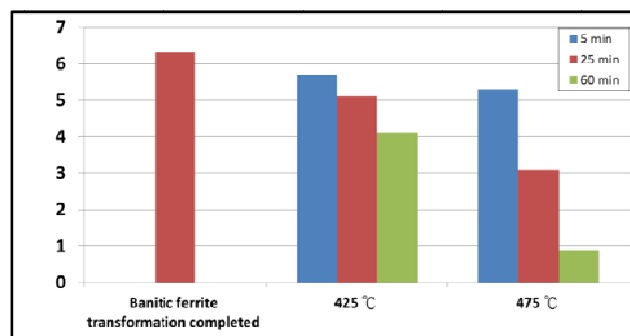


Fig. 5 Dislocation density in bainitic ferrite in nanostructure bainitic steel after tempering at various conditions revealing evidently decline in dislocation density at 475°C due to active recovery progression in the steel

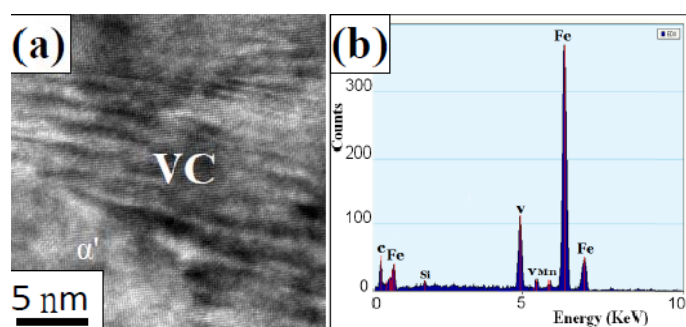


Fig. 6 (a) HRTEM image and (b) corresponding EDS analysis of vanadium carbide precipitation in the bainitic ferrite plate after tempering at 475°C for 60 min.

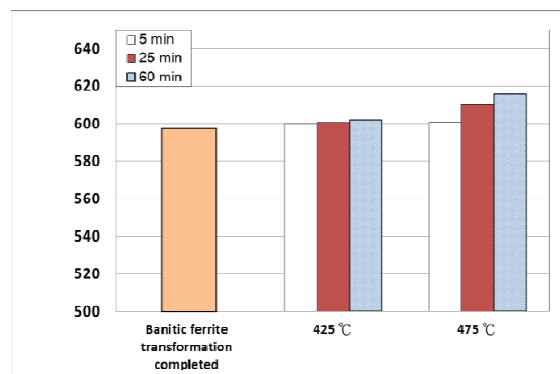


Fig. 7 Vickers hardness as a function of tempering temperature and time revealing obvious increase in hardness with time at 475°C because of carbide precipitation in the bainitic ferrite plate

#### IV. CONCLUSIONS

Through carefully examination of tempered nanostructure bainitic steel, it has found much dislocation debris connected with the austenite /bainitic ferrite interface and nanotwins in the retained austenite region. They are the consequence of plastic deformation occurring in the proximity retained austenite to accommodate the shape change when bainitic ferrite plates growth advance.

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#### REFERENCES

- [1] Caballero, F. G., Bhadeshia, H. K. D. H., Mawella, K. J. A.; Jones, D. G., and Brown, P, "Very strong low temperature bainite", *Mater. Sci. Technol.*, 2002, 18, pp. 279-281.
- [2] C. Garcia-Mateo, F. G. Caballero, and H. K. D. H. Bhadeshia, "Development of Hard Bainite", *ISIJ Int.*, 2003, 43, pp.1238-1243.
- [3] O Matsumura, Y Sakuma, and H Takechi, "Trip and its kinetic aspects in austempered 0.4C-1.5Si-0.8Mn steel", *Scripta Mater.*, 1987, 21, pp. 1301-1306.
- [4] H. K. D. H. Bhadeshia, "Steels for rails", In *Encyclopedia of materials science*, 2007, Oxford, UK:Pergamon Press/Elsevier Science, pp. 1-7.
- [5] Bhadeshia, H. K. D. H., "Large chunks of very strong steel", *Mater. Sci. Technol.*, 2005, 21, pp. 1293-1302.
- [6] F.G. Caballero, C. Garcia-Mateo, M.J. Santofimia, M.K. Miller, and Garcia de Andrés, "New experimental evidence on the incomplete transformation phenomenon in steel", *Acta Metall.*, 2009, 57, pp.8-17.
- [7] F.G. Caballero, Hung-Wei Yen, M.K. Miller, Jer-Ren Yang, J. Cornide, and C. Garcia-Mateo, "Complementary use of transmission electron microscopy and atom probe tomography for the examination of plastic accommodation in nanocrystalline bainitic steels", *Acta Metall.*, 2011, 59, pp.6117-6123.
- [8] F.G. Caballero, M.K. Miller, S.S. Babu, and C. Garcia-Mateo, "Atomic scale observations of bainite transformation in a high carbon high silicon steel", *Acta Metall.*, 2007, 55, pp.381-390.
- [9] L.C. Chang, and H. K. D. H. Bhadeshia, "Metallographic observations of bainite transformation mechanism", *Mater. Sci. Technol.*, 1995, 11, pp. 105-108.
- [10] B. P. J. Sandvik, and H. P. Nevalainen, "Structure-property relationships in commercial low-alloy bainitic-austenitic steel with high strength, ductility, and toughness", *Met. Technol.*, 1981, 8, pp.213-220.
- [11] M.-X. Zhang, and P.M. Kelly, "Crystallography of carbide-free bainite in a hard bainitic steel", *Mater. Sci. Eng. A*, 2006, 438, pp. 272-275.
- [12] Chen C. Y., Yen H. W. and Yang J. R. Nanostructure investigation on a tempered super bainitic steel. In: *The 1st international symposium on steel science*. Kyoto: ISIJ; 2007.
- [13] H.S. Hasan, M. J. Peet, and H. K. D. H. Bhadeshia, "Severe tempering of bainite generated at low transformation temperatures", *Int. J. Mater. Res.*, 2012, 103, pp. 1319-1324.
- [14] C. Garcia-Mateo, M. Peet, F. G. Caballero and H. K. D. H. Bhadeshia, "Tempering of a Hard Mixture of Bainitic Ferrite and Austenite", *Mater. Sci. Technol.*, 2004, 20, pp. 814-818.
- [15] S. Nagakura, Y. Hirotsu, M. Kusunoki, T. Suzuki, and Y. Nakamura, "Crystallographic study of the tempering of martensitic carbon steel by electron microscopy and diffraction", *Metall. Trans. A*, 1983, 14A, pp. 1025-1031.
- [16] F.G. Caballero, M.K. Miller, C. Garcia-Mateo, C. Capdevila, and S.S. Babu, "Redistribution of alloying elements during tempering of a nanocrystalline steel", *Acta Metall.*, 2008, 56, pp.188-199.