#### **International Journal of Advanced Materials Research**

Vol. 5, No. 1, 2019, pp. 12-19

http://www.aiscience.org/journal/ijamr

ISSN: 2381-6805 (Print); ISSN: 2381-6813 (Online)



# Phase Transformation Kinetics and Microstructure Evolution in a Medium Carbon Nano-structured Bainitic Steel During Isothermal Annealing Treatment

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#### **Abstract**

To examine the influence of isothermal annealing treatment both below and above Ms temperature on the transformation kinetics and microstructure evolution in a medium carbon nano-structured bainitic steel, dilatometry studies were conducted in a Thermo-mechanical simulator (Gleeble-3500 C) in a steel of compostion C: 0.49%, Mn: 1.46%, S: 0.019%, P: 0.003%, Si: 1.78%, Cr: 1.12% & Mo: 0.17% having Ms temperature ~270°C. The heat treatment cycle comprised of heating the samples at 5°C/s to austenitizing temperature of 850°C, soaking at 850°C for 180 seconds, cooling at 20°C/s upto the isothermal annealing temperature varying from 180°C to 360°C, holding at these temperatures for 8 hrs. followed by natural cooling upto room temperature. Microstructure at and above Ms temperature comprised lower bainite (accompanied by carbide precipitation) alongwith retained austenite and martensite-austenite (MA) blocks, while below Ms temperature, besides tempered martensite, features of typical lower bainite were observed alongwith carbides aligned in ferrite platelets. Phase transformation kinetics was analysed through dilatometry plots. It has been found that formation of prior athermal martensite causes an acceleration at the start of the subsequent isothermal transformations below Ms temperature compared to those above Ms temperature, where prior martensite does not form, which is reflected in high incubation time for bainitic transformation in such cases.

#### **Keywords**

Nano-structured Bainitic Steel, Phase Transformation Kinetics, Thermo-mechanical Simulation

Received: February 23, 2019 / Accepted: March 26, 2019 / Published online: May 5, 2019

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#### 1. Introduction

In recent years, Nano-structured bainitic steels have been developed by Bhadeshia, Caballero, Garcia-Mateo and coworkers [1]. These steels are processed by austenitisation followed by austempering at temperatures well below 300°C resulting in a microstructure comprising alternate layers of nano-scale bainitic ferrite as well as retained austenite. Nano-structured bainitic structure so obtained is

free of the carbide formation due to significant amount of Si (> 1.5%) present in the steel. The bainitic lath size varies from 40 to 100 nm. This kind of nano-bainitic microstructure is achieved in steels having carbon: ~1.0% and Si >1.5% through isothermal annealing by holding for a period ranging from days to weeks. The mechanical properties achieved in these steels are quite remarkable with tensile strength varying from 1.6-2.5 GPa, hardness in the range of 650-700 HV and fracture toughness: 30-50 MPa/m², depending on the steel chemistry and isothermal

\* Corresponding author E-mail address: skshukla@sail-rdcis.com (S. K. Shukla) transformation temperatures employed [2]. Though, the strength properties of nano-structured bainitic steels are excellent but the austempering time is often 10:s or 100:s of hours which makes their production more complicated. Since the carbon content of these nano-bainitic steels is quite high, so the weldability of these steels is an issue. Hence, efforts are required to achieve the comparable level of mechanical properties in steel having low/medium carbon content. Efforts made on developing low cabon nano-structured bainitic steel have not resulted in satisfactory results due to various reasons like merger of Bs and Ms temperature, coalescence of bainite etc. However, efforts may be made in developing medium carbon (0.4-0.6%) nano-structured bainitic steels with mechanical properties comparable to conventional nano-bainitic steel. Another issue which needs be addressed is the slow transformation kinetics of these steel. This must be done without the addition of costly alloying elements, only through modification in heat treatment techniques. Some studies have been recently reported [3, 9], where transformation kinetics has been found get enhanced through formation of prior martensite in the structure. However, still controversies exists regarding transformation products formed during isothermal annealing around Ms temperature and the factors affecting their kinetics and morphology. Therefore, objective of the present work was to examine the influence of isothermal annealing treatment both below and above Ms temperature on transformation kinetics and microstructure evolution in a medium carbon nano-structured bainitic steel.

## 2. Experimental Work

A heat of composition C: 0.49%, Mn: 1.46%, Si: 1.78%, Cr: 1.12%, Mo: 0.17%, S: 0.019%, P: 0.003% was made in the laboratory and hot rolled into 12 mm plates. Samples of size 10 mm dia. x 90 mm length were prepared (parallel to the rolling direction) for dilatometry test in Thermo-mechanical simulator (Gleeble-3500 C) to determine critical transformation temperatures and to do isothermal annealing experiments. Two types of heat treatments cycles were applied:

1. Quenching experiment was conducted to determine the Ms as well as Ac1 & Ac3 temperatures. The treatment consisted of heating the specimen @5°C/s to a fully austenitizing temperature of 1000°C and holding at this temp. for 5 minutes, followed by quenching by water to room temperature. From this treatment, Ms, Ac1 and Ac3 temperatures were determined.

2. Isothermal annealing treatments were conducted at different temperatures around Ms temperature to evaluate prior martensite formation effect on the subsequent phase formation during isothermal annealing treatment. The heat treatment cycle comprised heating the samples at 5°C/s to a austenitizing temperature of 850°C, austenitization at 850° for 180 seconds, cooling at 20°C/s upto the isothermal annealing temperature varying from 180°C to 360°C, as depicted in Figure 1, where the specimens were isothermally annealed for 8 hrs.. Finally, the specimens were subjected to natural cooling upto room temperature.

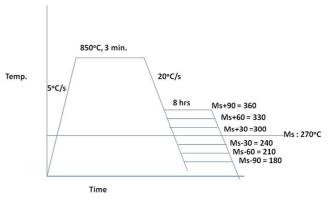


Figure 1. Schematic representation of isothermal annealing treatment.

The experimental specimens were prepared by following conventional polishing techniques for microstructural analysis. Nital 2% etchant was used to observe the microstructures through Optical Microscopy Specimens were also examined with a Zeiss EVO-MA10 SEM using a 20 kV electron beam. X-ray diffraction (XRD) analysis was conducted in a PANalytical 'XPERT PRO' diffractometer to determine retained austenite (RA) volume fraction. CuKa radiation was used in the 20 scan range of 30 to 110 deg with a step size of 0.03 deg. Integrated area technique was used to determine austenite and ferrite fractions using (111), (200) and (311) austenite peaks and (110), (200) and (211) ferrite peaks. Hardness of the dilatometry specimens were measured by Vickers Hardness Tester by applying 10 kgf load and six individual test values were considered for calculating average hardness value of the sample.

#### 3. Results & Discussion

# 3.1. Determination of Transformation Temperatures

Dilatometry plot generated during the first set of experiment test is shown in Figure 2.

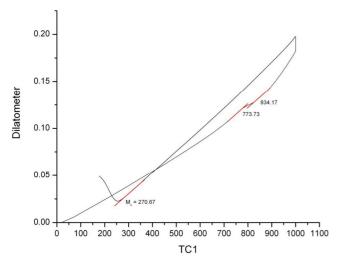


Figure 2. Dilatation plot for the investigated steel.

Tangents were drawn at the critical dilation points to determine transformation temperatures.  $Ac_1$ ,  $Ac_3$  &  $M_s$  temperatures were found to be 773.7, 834.2 and 270.7°C respectively.

#### 3.2. Phase Transformation Kinetics Through Dilatometry

In Figure 3a & b, dilatometry curves of all the seven isothermal annealing treatment are shown. Figure 3a shows the dilation verses temperature plot. Three distinct regions can be seen: (I) cooling to isothermal annealing temperature subsequent to austenitisation treatment, (II) isothermal holding at annealing temperature and (III) natural cooling to room temperature..

240 C

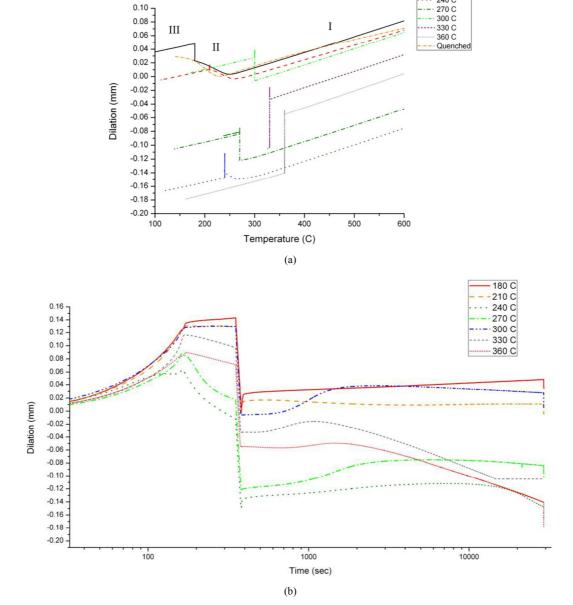


Figure 3. Dilation as function of (a) temperature and (b) time.

In Figure 3b, dilation plot as a function of time is shown. The curves corresponding to isothermal annealing temperature at 270 and 300°C shows austenite transformation to lower bainite (Figure 3a) during the isothermal annealing at these temperatures, indicated by an expansion in the dilation curve. Deviation from athermal and isothermal transformation is clearly visible in Figure 3b. The time required for initiation (incubation time) of bainitic transformation is ~500 s for isothermal annealing temperature of 270°C, while it ~400 s for isothermal annealing temperature of 300°C. Incubation time further decrease to ~200 s for temperature of 330°C, while for 360°C, there is no incubation time needed, the transformation starts immediately to bainite. However, after ~500 sec., dilation plot for both 330 and 360°C shows a decrease in dilation. This could be due to carbide precipitation in the bainitic ferrite phase. Further, it is clear from the dilation plot (Figure 3a) that no transformation is occurring during cooling from austenitisation to isothermal annealing temperature and cooling to RT. Also, it can be seen that bainitic transformation is almost finished (dilation almost constant) when isothermal annealing is interrupted.

However, when isothermal annealing was carried out below Ms temperature, prior martensitic transformation was observed when samples were fast cooled to below Ms, as shown in Figure 3a. The initial martensite (IM) fraction was determined both from dilatometry plot (by applying lever rule) and by using Koistinen-Marburger relationship {Fm =1exp(-0.011 (Ms-QT))}, where Fm is martensite volume fraction, QT is quenching temperature. From the dilatometry plot, the IM values were found as 0.24, 0.50 and 0.78 at 240, 210 and 180°C respectively. While from Koistinen-Marburger relationship, IM values were estimated as 0.28, 0.46 and 0.76 at 240, 210 and 180°C respectively. This shows that both estimated and measured values are quite close, thereby implying that Koistinen-Marburger empirical relationship is valid in this case. The dilation plot was observed to show an expansion trend during isothermal annealing below Ms temperature. In the Figure 3b, it can be clearly observed that there is no incubation time required for bainitic formation at isothermal annealing temperatures below Ms temperature. Also, dilation plots clearly shows that presence of prior martensite formation leads to formation of lower bainitic ferrite structure due to austenite decomposition during isothermal holding below Ms temperature [10-11]. As the kinetics of diffusion less bainite formation is controlled by nucleation, the presence of prior martensite in the structure leads to more nucleation sites before the isothermal annealing treatment and this results in enhanced transformation kinetics. The final deviation in dilatation plot, subsequent to isothermal holding may be because of formation of some fresh martensite (FM) which occurs due to

transformation of small volume of retained austenite (RA).

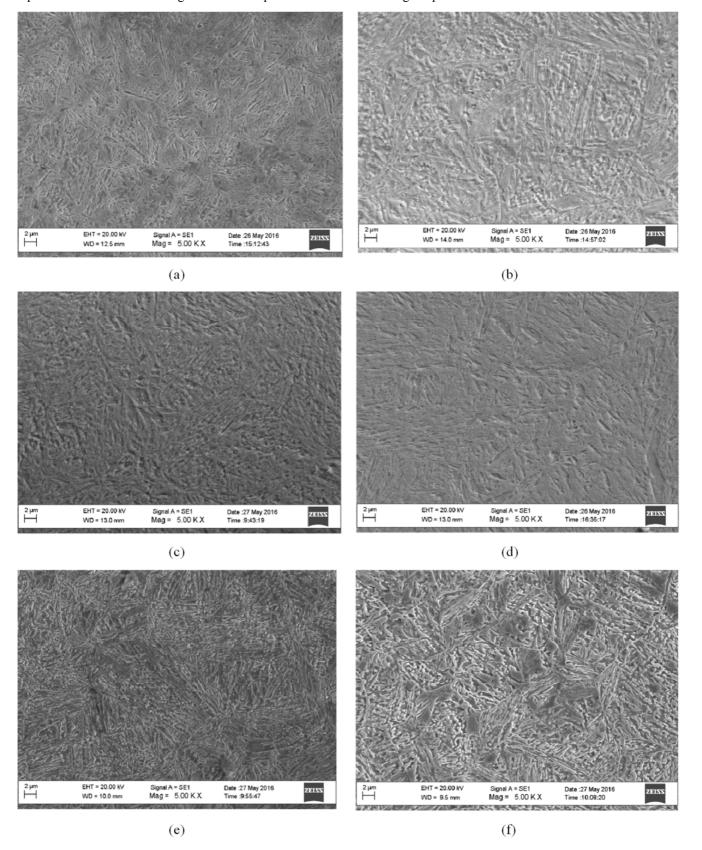
#### 3.3. Microstructure Evolution

Scanning electron micrographs of isothermally treated samples are shown in Figure 4. It was observed that the samples which were isothermally treated above Ms temperature, the lower bainite is the prominent phase in the structure (Figure 4(e) to (g)). The rod-like morphology of carbide precipitation was observed in lower bainite. Besides bainite, additional microstructural features such as RA and MA blocks have also been observed. The formation of RA and MA occurs as some fraction of untransformed austenite remains after isothermal holding is over and out of this certain fraction of retained austenite gets transformed (depending on RA stability) to fresh martensite (FM) during final stage of cooling and some fraction of RA still found in the final microstructure. The blocky type phase of FM was observed, the same type of microstructural features has also been reported by other researches [12, 13]. Micrographs of the specimens, isothermally annealed at 180-240°C, are shown in Figure 5a-c. In these cases, the microstructures comprises IM fractions of 0.28, 0.46 and 0.76 respectively, which were formed during fast cooling of the specimens subsequent to austenitising treatment. Subsequently, during isothermal holding for 8 h, the IM undergo tempering process. Therefore, the IM in subsequent sections is termed as tempered matensite (TM), which can be clearly observed in the micrographs. The carbide precipitation in these micrographs is also visible, however, the morphology of these precipitates seems to be different as compared to those observed in micrographs of samples isothermally annealed above Ms temperature.

In addition to TM, lower bainite as well as ferrite platelets with carbides of rod-like morphology are also observed in the micrographs. The above observations support the argument that lower bainite formation takes place during the isothermal annealing below Ms temperature. Since the silicon content of steel is quite high (~1.78%), cementite formation may be somewhat gets inhibited at lower isothermal annealing temperature. However, the carbide precipitation was clearly observed in the specimens which were isothermally annealed above Ms temperature (particularly at 330 and 360°C). Presence of RA at interlath/blocky positions was observed particularly in specimens which were isothermally annealed above Ms temperature (300-360°C). Also, some fraction of FM was also observed in specimens which were isothermally annealed at 330°C and 360°C, however, their presence was absent in specimens which were annealed at temperatures below Ms temperature (very small deviation observed in dilatation). Another reason of FM absence in these specimens

could be the slow cooling rate (free cooling and not quenching) employed during final cooling from isothermal annealing temperature to RT. Slow cooling rate in these specimens was

also due to low temperature gradient (temperature difference between start and final temperature), as the isothermal annealing temperature was also lower in these cases.



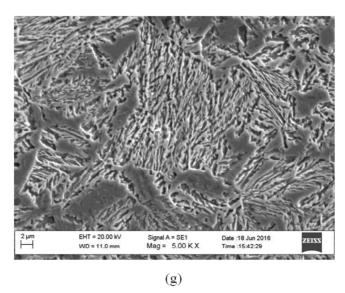


Figure 4. Scanning electron micrographs of the specimens isothermally annealed for eight hours at temperatures below Ms (a)  $180^{\circ}$ C (b)  $210^{\circ}$ C and (c)  $240^{\circ}$ C, just at Ms (d)  $270^{\circ}$ C, and above Ms (e)  $300^{\circ}$ C, (f)  $330^{\circ}$ C and (g)  $360^{\circ}$ C.

#### 3.4. X-ray Diffraction Analysis

To determine experimentally the phase fractions of bainitic ferrite, retained austenite, and fresh martensite /tempered martensite in the isothermally annealed specimens, X-ray diffraction analysis (using Diffrac Plus Eva software) and dilatation plots were analysed. X-ray diffraction graphs of all the seven samples have been shown in the Figure 5.

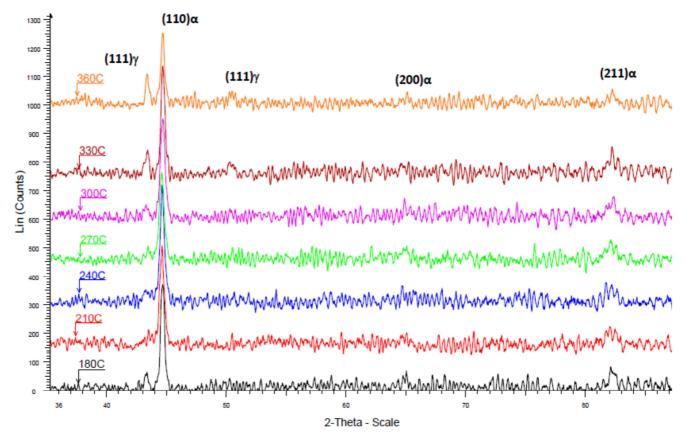
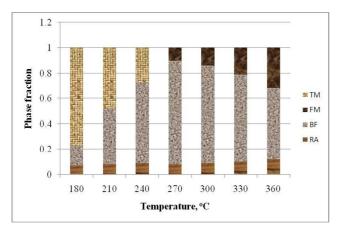


Figure 5. XRD of isothermally annealed samples.

It can be seen in the graph that major phase formed in all the samples is bainitic ferrite, while peaks of austenite are also visible. In fact, small peaks of martensite at  $2\theta$  angle of 43.9

and 82.2 can also be clearly seen in isothermally samples of above Ms temperatures. However, these peaks have got merged with the adjacent peaks of austenite and ferrite

respectively. All the XRD graphs have been shown in the same plot, so that a comparison among the seven samples can be made at a glance and qualitative information about the different phases can be drawn. Fraction of phases formed at different isothermal annealing temperatures are shown in Figure 6. In the SEM analysis, the retained austenite was observed in all the isothermally annealed specimens after completion of entire heat treatment cycle. However, the fraction of RA is quite low in all the specimens. As can be seen in Figure 6 that the RA fraction varied in the range of 7-12%. The maximum RA content of ~12% was observed in specimen, which was isothermally annealed at 360°C. The austenite stability increases as carbon gets partitioned from IM to untransformed austenite and remains there till the heat treatment cycle is over. [14, 15]. The fraction of IM in terms of TM fractions, as well as FM fractions were determined through dilation plot, while RA was measured through X-ray diffraction analysis. Phase fraction of bainitic ferrite was estimated through balance of TM, RA and FM phase fractions formed in the samples.



**Figure 6.** Transformed phase fractions during heat treatment, TM: tempered martensite, FM: fresh martensite, BF: bainitic ferrite, RA: retained austenite.

#### 3.5. Hardness Variation with Isothermal Annealing Treatment

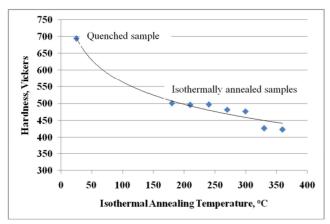


Figure 7. Hardness variation with isothermal annealing temperature.

Hardness of the isothermally annealed samples with annealing temperature is shown in Figure 7. With increase in isothermal annealing temperature, the hardness was found to decrease. This may be due to the presence of higher amount of prior matensite formation (which subsequently gets tempered during isothermal holding) in the samples which were isothermally annealed below Ms temperature. From this, it can be inferred that low temperature tempered martensite is harder compare to microstructure consisting bainite ferrite, retained austenite and fresh martensite.

#### 4. Conclusion

Bainitic ferrite and/or tempered martensite have been identified as the transformation products in isothermal annealing treatments below and just at Ms. The morphology of bainitic ferrite comprises laths with carbides. Both above and below Ms temperature, bainitic ferrite has been identified as the transformation product formed. The morphology of bainitic ferrite consists of acicular units with interspersed retained austenite. Formation of prior martensite increases the transformation rate below Ms temperature compared to isothermal transformation above Ms temperature due to absence of prior martensite formation, which is reflected in high incubation time for bainitic transformation in such cases. Further, it is found that hardness of the isothermally treated sample increases with decrease in isothermal holding temperature because of the presence of tempered martensite in the structure.

### **Acknowledgements**

The authors are grateful to the management of RDCIS for their constant support and encouragement during the course of work accomplished under this project.

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