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# Isothermal Transformation Behavior and Microstructural Evolution of Micro-Alloyed Steel

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## Abstract

In this present study, the transformation products in micro-alloyed steel have been examined as well as isothermal decomposition of austenite into various phase formation. The rapid cooling from austenitizing temperature 1200°C to 14 different isothermal temperatures between 750 and 100°C with 50°C intervals were carried out by using dilatometric strain dilatometer on thermo-mechanical simulator. The heat treatments were delayed at different times to examine the microstructure evolution at all isothermal temperatures. The transformation kinetics was recorded during isothermal treatments and designed an isothermal transformation diagram, which is verified by microstructural changes. The results show that the initial microstructure which consists of proeutectoid ferrite and pearlite transforms into a combination of proeutectoid ferrite, pearlite, widmanstätten ferrite, upper or lower bainite, or martensite phases. The austenite grain size has been found to be decreased with a decrease in the isothermal holding temperature. The nose temperature was achieved at isothermal temperature 500°C which have been taking the least time for start and end of transformation of phases. It is also worth noticing that the start and end transformation times were observed decreasing with a decrease in the isothermal holding temperatures and after the nose transformation again gradually increased.

**Keywords:** isothermal transformation, micro-alloyed steel, microstructure, recrystallization, bainite, ITT diagram

## 1. Introduction

Excellent combination of mechanical and corrosion resistance properties is an initial requirement for high strength low alloyed steel plates which have to be used for the construction of bridge, pipe lines, and ship hull applications [1]. Generally, basic steels do not have the sufficient combination of properties for a direct use for such types of applications. Desirable structural properties may become possible by changes in the chemical composition of steels or by heat treatments such as continuous/isothermal heat treatments with/without thermo-mechanical processing of steels. The compassionate evolved will support in scheming isothermal heat treatment cycles during production of such steels. During steel processing as well as fabrication through welding, the phase transformation has always been a significant research topic for steel users and metallurgists. Isothermal heat treatment

is subsequently possible after casting process in industry on nominal cost. Better properties offer to users to reduce the overall cost for structures by reducing the thickness of steel plates. Presently, most of researchers focus on isothermal transformation treatments for medium and high grade carbon steel [2–5] but they lack such information in wide range of isothermal temperature on micro-alloyed steel.

In the present study, effort has been made to design isothermal transformation (ITT) phase diagram for micro-alloyed steel. Isothermal heat treatments are experimented by using physical thermo-mechanical simulator in an isothermal temperature range from 750 to 100°C. Microstructural evolution is characterized by optical microscopy.

2. Experimental details

The present micro-alloyed steel are provided by the Steel Authority of India Ltd. (SAIL), the chemical composition was predicted by using Thermo Jarrell Ash spark emission spectroscopy as displayed in **Table 1**. Present steel is familiar by the name of SAILMA steel.

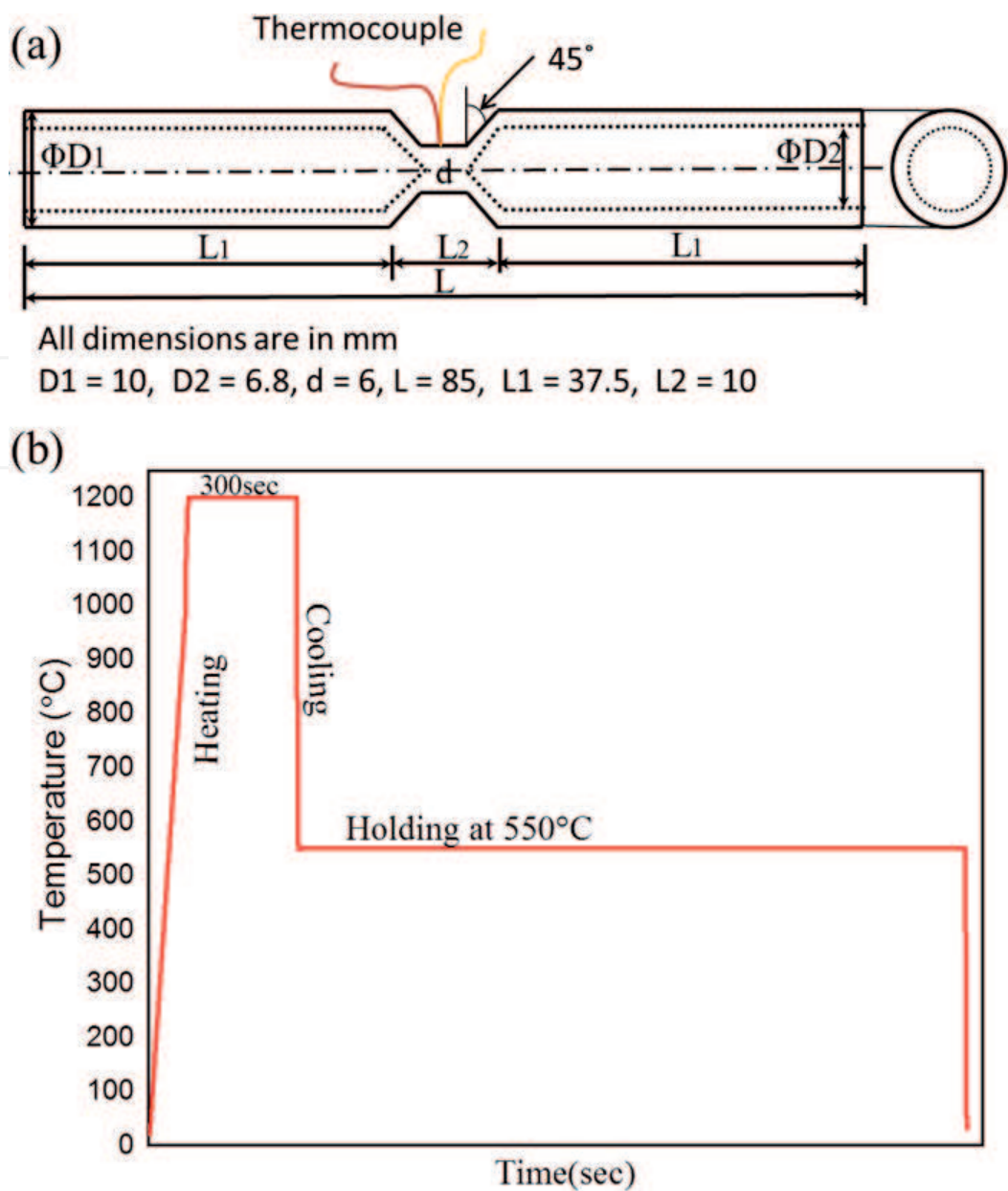
Samples were taken from the transverse direction of a 18 mm thick hot rolled plate. Samples were treated for isothermal physical simulation by the thermo-mechanical simulator Gleeble®3800. Resistance heating was provided by K-type (Chromel-Alumel) thermocouples to the samples by a machine working based on conductive heat transfer model. A Linear Variable Displacement Transducer, LVDT dilatometer (quartz based) was fitted above the sample (where the thermocouple was welded at mid position) to acquire diametric strain changes during thermal cycles. External water quenching was arranged on both side of the hollow region of sample to freeze the microstructure and achieve the desire cooling rates as per ASTM isothermal treatments. Desired cooling rate in programmed and actual thermal cycles was achieved in less than 1 s between the solutionizing temperature and the isothermal temperatures.

For isothermal treatment simulation, the samples were first heated by 5°C/s to the solutionizing temperature 1200°C for 300 s and followed by 200°C/s down to 14 isothermal temperatures between 750 and 100°C with a temperature gap of 50°C. The samples were kept at these isothermal temperatures until completion of phase transformation estimated through dilation strain changes. Subsequently, the sample was water quenched to room temperature by external water quenching arrangements. Details of sample design and isothermal treatment cycle for one isothermal temperature 550°C are shown in **Figure 1**.

Isothermal simulated samples were partitioned cross-sectionally from the position of thermocouples for microstructural examination. One piece of partitioned surface of sample was ground and polished according to standard technique of metallography and later etched by a 2% Nital etchant. Optical micrographs of base material and simulated IT samples were characterized by using optical microscope Leica DMI 5000 M equipped with digital imaging facility. The grain size of ferrite was measured by linear intercept method using Image J software. Average grain size of 10 readings is reported in the present work.

C	Si	Mn	P	S	Al	Nb	V	Fe
0.13	0.25	1.53	0.03	0.007	0.02	0.043	0.01	Bal.

**Table 1.**  
*The chemical composition of micro-alloyed steel under investigation, in wt%.*



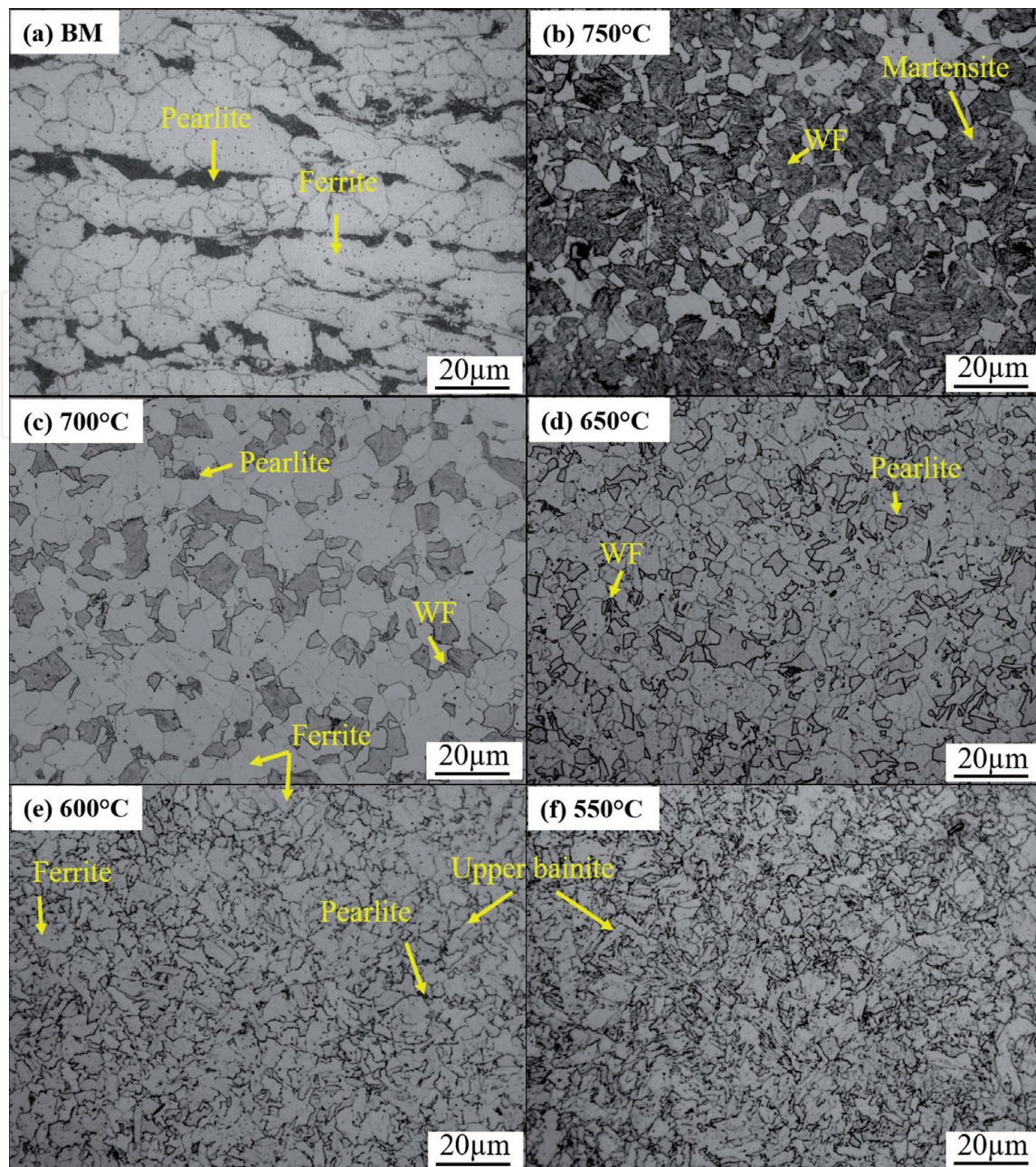
**Figure 1.**  
Schematic diagrams: (a) sample design and (b) isothermal treatment cycle for isothermal temperature 550 $^\circ\text{C}$ .

### 3. Results and discussion

#### 3.1 Microstructural evolution

The optical micrographs for base metal and isothermally simulated steel samples are shown in **Figures 2** and **3**. The base metal micrograph consists of pro-eutectoid ferrite and pearlite as shown by arrow in **Figure 2a**. The pearlite structure is found to be in banded form in the direction of rolling [6]. The average grain size of pro-eutectoid ferrite is found to be in range of  $10 \pm 3 \mu\text{m}$ . The average grain size of ferrite is found to be decrease with decreasing isothermal temperature as shown in **Figure 4**. At 750 $^\circ\text{C}$ , the banded structure of pearlite is completely dissolved and new structure is formed which consists of widmanstätten ferrite (WF), pearlite and very small amount of martensite (**Figure 2b**). Rest bright phase fraction reveals as the pro-eutectoid ferrite. It is worthy noticed that the material has not undergone any transformation due to fast cooling rate more than 175 $^\circ\text{C/s}$  to 750 $^\circ\text{C}$ . The austenite grain boundaries are completely illuminated through ferrite



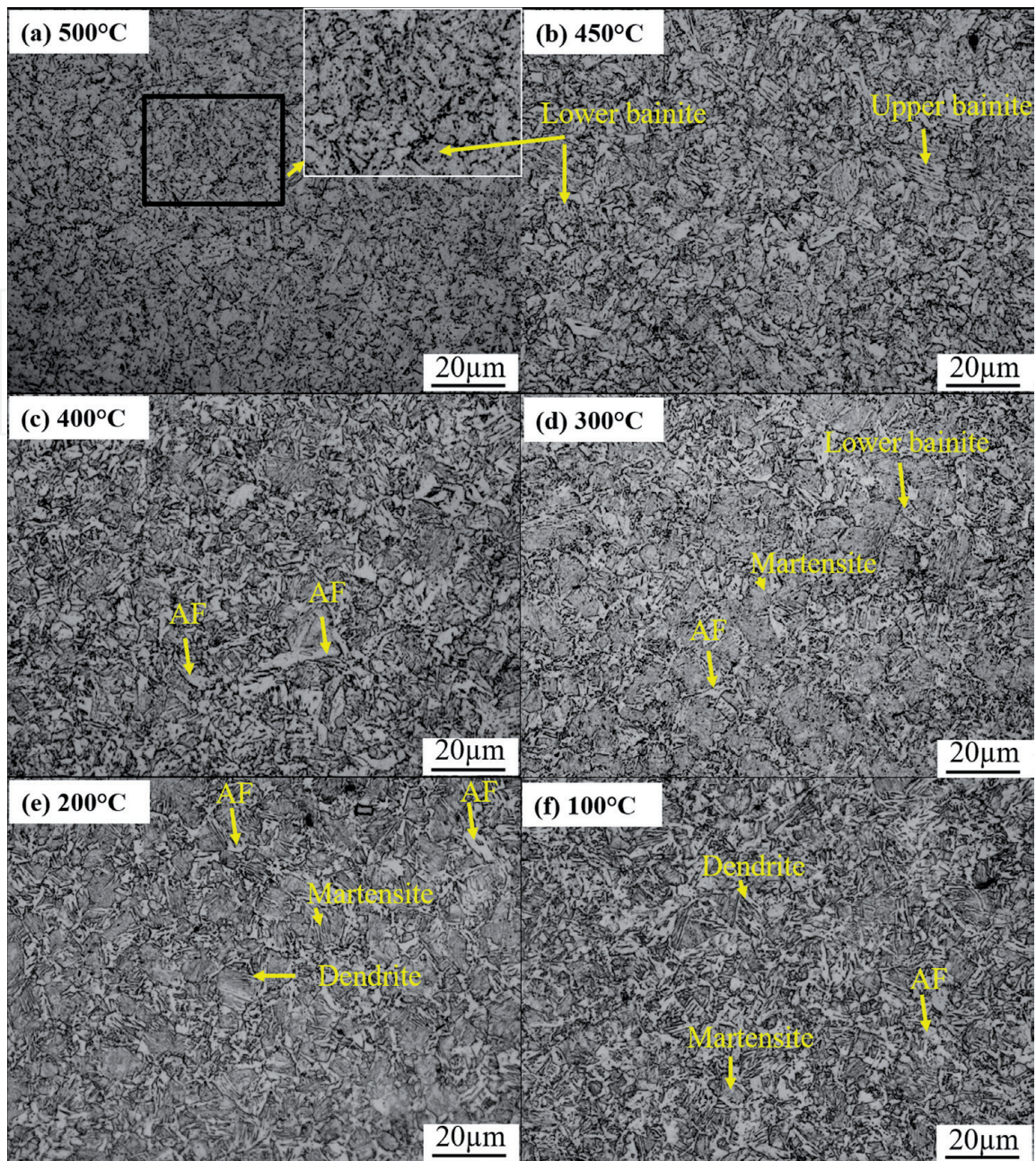


**Figure 2.**

*Optical micrographs of micro-alloyed steel: (a) base metal steel and (b–f) isothermally treated samples subjected to temperatures between 750 and 550°C.*

allotriomorphs and retained austenite has reconstructed into pearlite as can see in **Figure 2b**. This pearlite reaction fundamentally occurs at high temperature between 720 and 550°C, which is governed by the diffusion process [7]. In addition, the conventional growth occurs of widmanstätten ferrite under para-equilibrium must correspond either to interface friction control or to carbon diffusion control [8]. This widmanstätten ferrite nucleates at austenite grain boundaries and grows as plates into austenite interior in reconstructive mode [8]. The diffusion of carbon in austenite ahead of plate tip gives maximum lengthening rate due to the capillarity effects. This structure is in agreement with other micro-alloyed steels [9, 10]. The average grain size of ferrite also reformed due to solutionizing treatment and uniform grain size is found to be 8 µm at 750°C. **Figure 2c** shows the amount of widmanstätten ferrite is found to be increasing due to instability of the pearlite phase at 700°C. As the isothermal temperature decreases, the rest amount of enriched carbon islands is fully dissolved in soft pearlite phase region as shown in **Figure 2d**. The ferrite grain size is found to be decreasing gradually

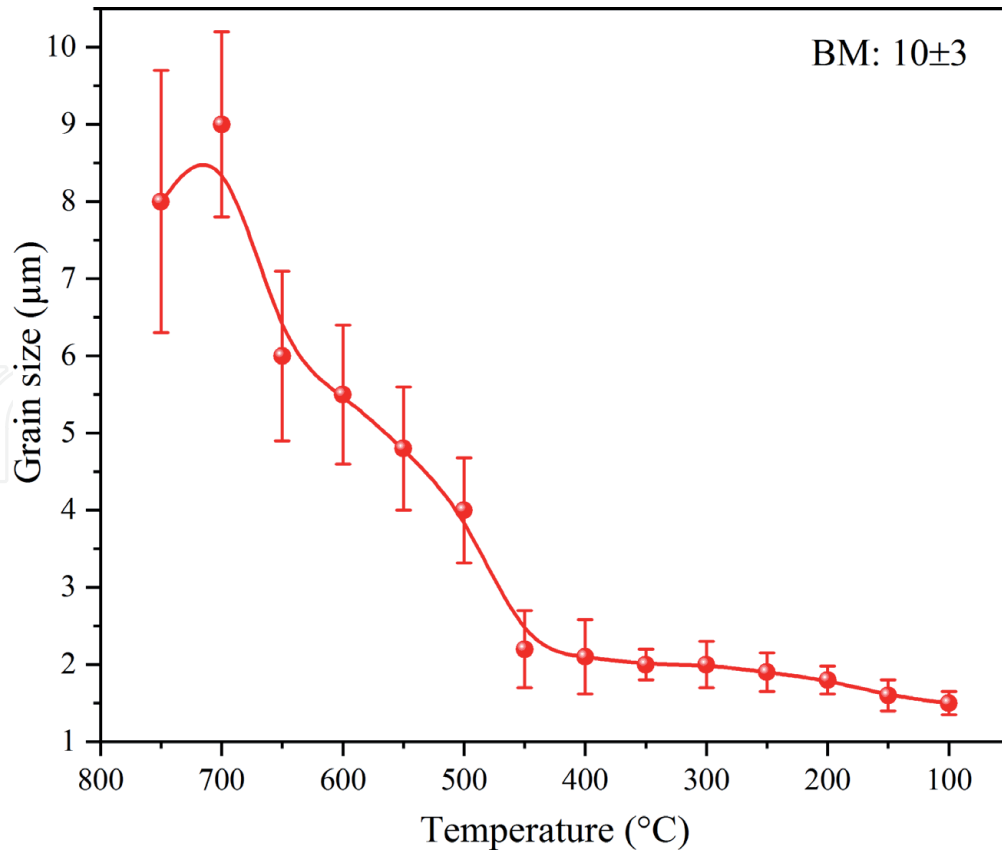




**Figure 3.**  
 Optical micrographs of isothermally treated samples subjected to temperatures between 500 and 100°C.

with a decreasing isothermal temperature (**Figure 4**). Carbon is extracted from pearlite and it transforms into residual austenite or cementite or carbides along the austenite grain boundary. This formation of ferrite laths and separation by residual austenite or cementite or carbides is known as upper bainite [5, 11, 12]. The fraction of residual austenite, etc., phases observed are decrease with a decrease in isothermal temperature (600°C) as can see in **Figure 2e**. The amount of widmanstätten ferrite is decreased with decreasing in isothermal temperature. At 550°C, the microstructure is fully transformed into upper bainite however; a small amount of proeutectoid ferrite is also observed (**Figure 2f**). Upper and lower bainite structures have formed and grown by displacive mechanism [13]. Bainite formed as an aggregate of small plates of ferrite (sub-units). These ferrite plates or laths grow in the form of clusters, which are known as sheaves. These plates are parallel and there is an identical crystallographic orientation in every sheaf. The carbon-enriched residual austenite or martensite is found between plates of ferrite in upper bainite and it is free of precipitation of carbides. However, carbide precipitates precipitate inside ferrite plates in lower bainite [11].





**Figure 4.**  
The variation of ferrite grain size of micro-alloyed steel with isothermal temperature.

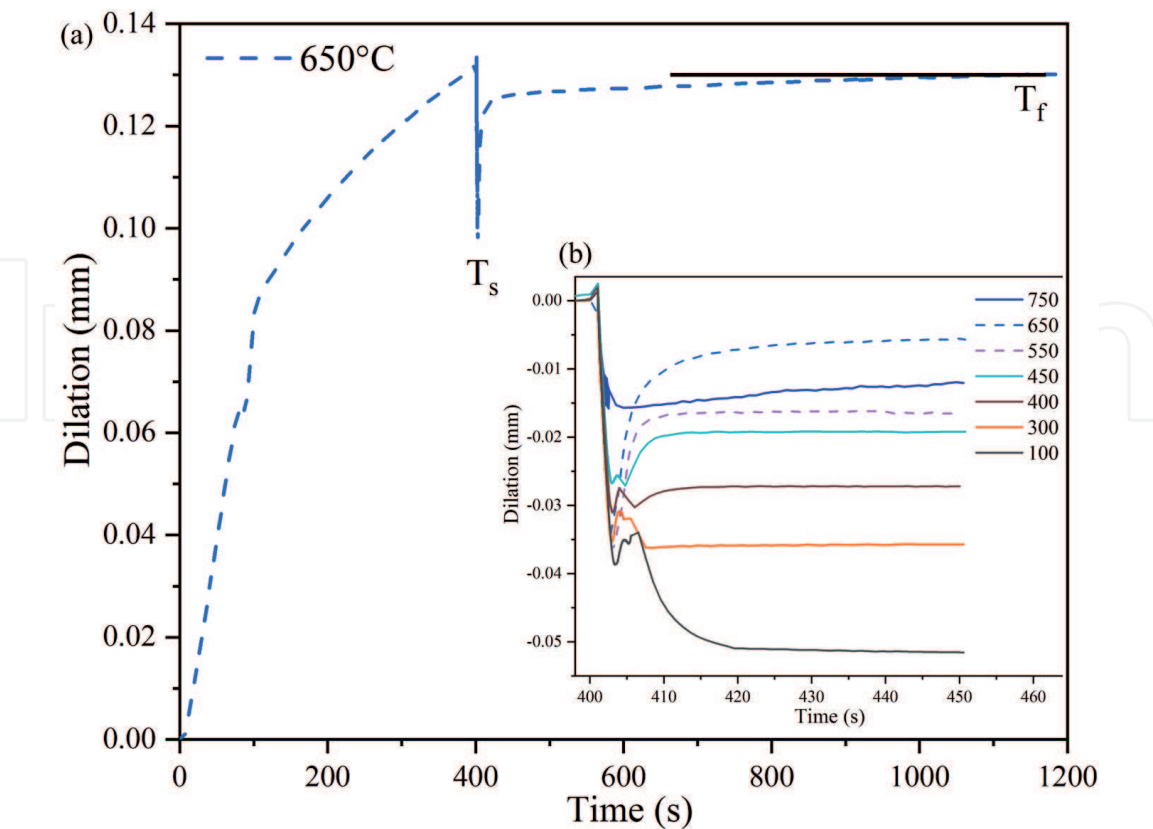
As there is a decrease in temperature (500°C), the amount of upper bainite is found to be decreasing and more refined due to the lower transformation temperature, and subsequently lower bainite reveals (**Figure 3a**). At 450°C, the fraction is fully transformed into lower bainite with small amount of upper bainite as can see in **Figure 3b**. It is also noticed that lower austenite grain size leads to the rate of higher amounts of bainite transformed with refinement in ferrite plates under the same isothermal temperature. At temperature 400°C, the microstructure consists of lower bainite and martensite with small amount of allotriomorphic ferrite (AF) around the austenite grain boundaries (**Figure 3c**). This microstructure is similar to allotriomorphic transformation process as reported for high temperature (at around 720°C) in the work of Ashrafi et al. [14]. The several nucleation sites form from the austenite grain boundary and simultaneously hard impingement also occurs between sheaves of bainite inside austenite grains [15]. As there is a decrease in the isothermal temperature (from 300 to 100°C), the fully lath martensite structure is observed with allotriomorphic ferrite (AF) at austenite grain boundaries. This allotriomorphic ferrite (AF) has a very similar morphology to a dendrite structure. It is acknowledged that the nucleation of ferrites form preferable locations at primary austenite grain boundaries due to the effect of boundary energy (**Figure 3d–f**). However, the martensite formed due to rapid cooling rate of austenite form of iron so that carbon atoms do not have sufficient time to diffuse out the crystal structure. Since, the nucleation of martensite is diffusionless. Laterally, the fraction of allotriomorphic ferrite (AF) observed is coarser with decreasing in martensitic transformation temperature region.

### 3.2 Isothermal transformation (ITT) diagram

**Figure 5** illustrates the approach to calculate time for start transformation ( $T_s$ ) and end transformation ( $T_f$ ), and between of them such as 25, 50, and 75% phase

transformation, which results are used to construct an ITT diagram (**Figure 6**). This ITT diagram is created on the basis of dilation strain data test which is in reference of phase transformation as shown in **Figure 5a** for 650°C. The phase transformation 100% is confirmed by no change in dilation w.r.t. time, it means that the amount of phase is not increasing with time at that particular isothermal temperature. The change in dilation represents to nucleation and growth phase as shown in **Figure 5b**. The fluctuation in dilation appears after start transformation of phases when temperature was lower than 550°C. It is attributed to undercooling. Further, its increase/decrease in dilation completely depends upon type of phase formation such as upper bainite/lower bainite/martensite. The incubation time is found to be decreased with decreasing in isothermal temperature. Similar trends in dilation studies are also reported in the work of Chen et al. [15]. The dilation after an incubation period is almost constant without any contraction or expansion after the start of phase transformation for isothermal temperature 400°C as can see in **Figure 5b**. Since, the most of austenite fraction is completely transformed into lower and upper bainite with small amount of martensite as confirmed from the microstructure (**Figure 3c**). Nevertheless, the dilation was found to decrease in dilation with a decrease in isothermal temperature less than 400°C. Here, both lower bainite and martensite transformation mechanism work simultaneously.

In martensitic transformation, the atom movements occur by two shears formation mechanism, i.e., long-wave as primary and shuffle type shear as secondary which take place at the same time [16]. The primary and secondary part of shears is corresponding to the Bain distortion, and the rest of the secondary shear is in agreement with the lattice invariant shear of the phenomenological theories [17]. However, widmanstätten ferrite, upper bainite, and lower bainite are formed by high-velocity shear. Since, transformation products are developed by displacive



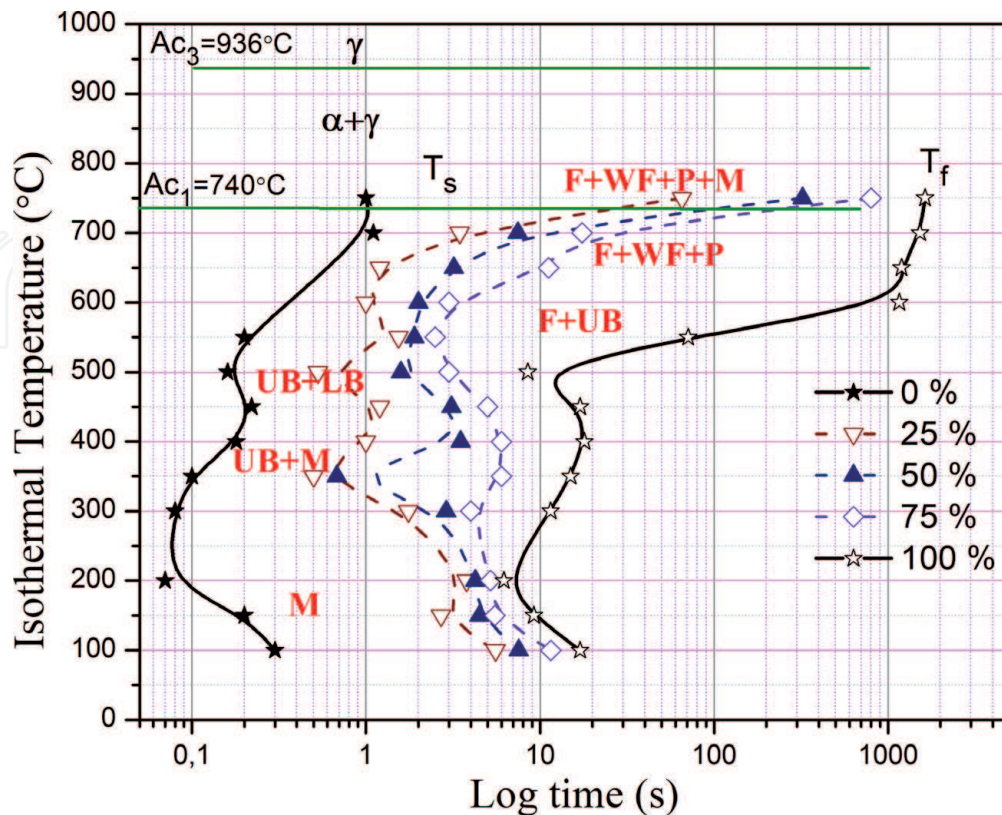
**Figure 5.** Dilation vs. log time plots of micro-alloyed steel subjected to isothermal temperatures: (a) full dilation plot curve for temperature 650°C; and (b) dilation plots for start and end transformation regions of phases for selective transformation isothermal temperatures.



mechanisms, however martensite formed by shear process. The contribution of shear process increases as the temperature decreases [11].

The lower ( $Ac_1$ ) and upper ( $Ac_3$ ) critical temperatures during heating at  $5^\circ\text{C/s}$  were obtained  $734$  and  $912^\circ\text{C}$  respectively from the present study. The ITT diagram's purpose is not only to collect the statistics about the nature of phases however, it also finds out the kinetics of phase transformation for the holding period at constant (isothermal) temperature. This is a very useful set of heat treatment processes to obtain the desired properties by moderation of microstructure. This ITT diagram is also known as time temperature transformation (TTT) diagram. An ITT diagram plotted based on isothermal temperature heat treatment is displayed in **Figure 6**. The initial microstructure was consisting of proeutectoid ferrite and pearlite which transforms into proeutectoid ferrite, pearlite, and other new phases such as widmanstätten ferrite, upper/lower bainite and transformed martensite (**Figures 2 and 3**). It is worth noticing from **Figure 6** that the time of start transformation decreases with decreasing isothermal temperature until  $250^\circ\text{C}$ , and further starts increasing. The start transformation time is delayed by around  $12$  s and takes longer time to finish (end) transformation when the temperature is close to the lower critical temperature as can see in **Figure 6**. As decreasing in isothermal temperature, both  $T_s$  and  $T_f$  phase transformation times are observed decreasing. The nose of ITT is found to be at  $500^\circ\text{C}$ , where there was the shorter delay (less than  $1$  s) between the start and end transformation of phases. An analysis of this ITT diagram is divided into five regions which show a combination of phase's microstructure (**Figure 6**) as confirmed by the microstructural evolution (**Figures 2 and 3**).

IT $750^\circ\text{C}$  ( $>Ac_1$ ), the start transformation occurred after  $12$  s (incubation period) and the end transformation (100% transformation of phases) after  $1080$  s, which is predicted in the form of diametric changes in mm during experimental studies. The microstructure consists of proeutectoid ferrite, widmanstätten ferrite, pearlite and martensite at high temperature isothermal holding. Small volume fraction of



**Figure 6.**  
Isothermal transformation diagram of micro-alloyed steel.

martensite was established at this isothermal temperature. These transformation products are developed by displacive and reconstructive mechanisms, however martensite formed by shear process which is also part of displacive mechanism.

Ac1–IT650°C denotes the formation of phases with both reconstructive and displacive transformations. The microstructural evolution is the combination of pro-eutectoid ferrite and pearlite, widmanstätten ferrite (WF), no more martensite. Start to end transformation time is found to be decreased gradually due to the formation of phases without any delayed.

IT650–500°C, the microstructural evolution was observed through the formation of upper bainite, formed by displacive or diffusionless mechanism. The ferrite plates in upper bainite are separated by the cementite as cleared from the microstructure. Nevertheless, a small fraction of lower bainite is observed. The nose temperature was achieved at isothermal temperature 500°C that has been taking the least time between the start and end transformation of phases. Later it starts increasing again gradually in incubation time with a decrease in IT temperature (**Figure 6**).

IT450–300°C, the lower bainite structure is the combination of ferrite plates with carbide and these plates are separated by retained austenite or cementite or martensite. The needle type morphology of ferrites is observed along the grain boundary in dendrite form due to higher shear strains.

IT300–100°C is once more a displacive and diffusional transformation though with tiny or no carbide precipitation which is attributed to low IT. These microstructures are transformed into fully martensite. Lower bainite amount has been found to be decreasing with isothermal temperature. These transformation products are developed by shear process mechanism.

#### 4. Conclusions

The following outputs are constructed from the present work on micro-alloyed steel.

- The initial microstructure of base steel consists of proeutectoid ferrite and pearlite. During isothermal heat treatments, the microstructure transforms into a combination of possibly proeutectoid ferrite, pearlite, widmanstätten ferrite (WF), upper or lower bainite, or martensite phases. The austenite grain size has been found to be decreasing with a decrease in the isothermal holding temperature.
- At IT750°C, the microstructure consists of proeutectoid ferrite, widmanstätten ferrite, pearlite and martensite due to high temperature isothermal holding. Small volume fraction of martensite was found due to isothermal temperature above the critical temperature. These transformation products are developed by displacive and reproductive mechanisms, however martensite formed by shear process.
- IT700–600°C shows a significant fraction of allotriomorphic ferrite, thus the pearlite phase has apparently to be softer. However, the volume fraction of pearlite is decreased with temperature. A fraction of carbon content was observed along the grain boundary, which was extracted from the ferrite during nucleation and growth of phases.
- Most of the austenite fraction transforms into upper bainite in medium range of isothermal temperature (600–450°C). In IT450–300°C, the austenite



transforms into predominantly lower bainite with increment in martensite. At less than 300°C, the microstructure was observed to be a martensite structure and acicular martensitic dendrite structure at austenite grain boundary.

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