AN INVESTIGATION OF BAINITIC TRANSFORMATION IN LOW CARBON ALLOY AND HIGH CARBON RAILWAY STEELS

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ABSTRACT

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In this thesis study, bainitic transformation in high carbon railway steel and boron steel was investigated. Bainitic structure known as having high strength and high toughness at the same time. Mechanical properties of bainite were compared with the tempered martensite phase which is commonly used phase in industry. R260 and 32CrB4 steels were used in this study. Different heat treatments were applied to these steels for bainitic transformation. Austenitization was done at 850°C for R260 steel and at 880°C for 32CrB4 steel. It is followed by an isothermal holding at just above M_s temperatures. Studies show that, R260 steel do not complete bainitic transformation at 205°C (just above M_s) by isothermal holding for 7 days. 32CrB4 steel, on the other hand, can be transformed to fully bainite by holding 2 hours at 350°C, which is just above M_s. Two-step bainite transformation was applied in order to obtain secondary fine bainite sheaves. After holding 2 minutes at 350°C, sample was quenched to 300°C and hold 5.5 hours isothermally. Mechanical properties of two-step bainite transformation becomes higher relative to fully bainite phase. First step of two-step bainite transformation was investigated by holding 2 minutes at 350°C followed by water quenching. Additionally, tempering was applied at 450°C for 2 hours. Hardness testing, tensile testing and notched bar Charpy impact testing were applied to investigate the mechanical properties. 1410 MPa of tensile strength, 13.23% of tensile strain and 50.5 Joules of impact energy were obtained in two-step bainite transformation.

Keywords: Bainite, Two-Step Bainite Transformation, 32CrB4, R260

ÖZ

DÜŞÜK KARBONLU ALAŞIMLI VE YÜKSEK KARBONLU TREN RAY ÇELİKLERİNDE BEYNİT DÖNÜŞÜMÜN İNCELENMESİ

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Bu tez çalışmasında yüksek karbonlu tren rayı çeliği ve orta karbonlu bor çeliğinin beynitik dönüşümleri incelenmiştir. Beynitik yapı yüksek mukavemet ve yüksek tokluğa aynı anda sahip olmasıyla bilinir. Beynitin mekanik özellikleri, endüstride yaygın olarak kullanılan temperlenmiş martenzit ile karşılaştırılmıştır. Bu çalışmada R260 ve 32CrB4 çelikleri kullanılmıştır. Bu çeliklerin beynitik dönüşümü için çeşitli ısıl işlemler uygulanmıştır. Östenitleme R260 çeliği için 850°C'de, 32CrB4 çeliği için ise 880°C'de yapılmıştır. Bunu martenzit başlangıç sıcaklığının hemen üzerinde izotermal bekleme izlemiştir. Çalışmalar göstermiştir ki, R260 çeliği 7 gün boyunca 205°C'de izotermal olarak tutulduğunda beynitik dönüşüm tamamlanmamaktadır. Diğer taraftan, 32CrB4 çeliği martenzit başlama sıcaklığının hemen üstü olan 350°C'de 2 saat tutulduğunda tümüyle beynit fazı oluşmaktadır. Daha ince yapılı ikincil beynit demeti oluşturmak için iki aşamalı beynitik dönüşüm uygulanmıştır. 350°C'de 2 dakika tutulduktan sonra 300°C'de 5.5 saat tutulmuştur. İki aşamalı beynit dönüşümü, tümüyle beynit olan yapıya göre daha yüksek mekanik özellikler göstermiştir. İki aşamalı beynit dönüşümünün ilk aşamasını incelemek amacı ile 2 dakika 350°C'de tutulan numuneye su verilmiştir. Ayrıca 2 saat boyunca 450°C'de tavlama da uygulanmıştır. Mekanik özelliklerin incelenmesi için sertlik testi, çekme testi ve çentik darbe testi yapılmıştır. İki aşamalı beynit dönüşümü 1410 MPa çekme mukavemeti, 13.23 % çekme gerinimi ve 50.5 Joule darbe dayanımı sonuçlarını vermiştir.

Anahtar kelimeler: Beynit, iki aşamalı beynit dönüşümü, 32CrB4, R260

To My Precious Family

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LIST OF ABBREVIATIONS

M_s: Martensite Start Temperature

M_f: Martensite Finish Temperature

B_s: Bainite start temperature

SEM: Scanning Electron Microscope

FCC: Face Centered Cubic

BCT: Body Centered Tetragonal

BCC: Body Centered Cubic

γ: Austenite

α: Ferrite

B: Bainite

M: Martensite

B+M: Bainite + Martensite

CVN: Charpy V-notch

AISI: American Iron and Steel Institute

ASTM: American Society for Testing and Materials

HV: Vickers Pyramid Number

T_0: The temperature at which both bainitic ferrite and austenite of similar composition have identical free energy

CHAPTER 1

INTRODUCTION

Bainitic steels have superior properties relative to pearlitic steels which are used widely in industry. Apart from special purpose steels; automotive, defense, construction industries and many other areas in industry use mostly pearlitic steels for a long time. Bainite phase mixture was found in 1920s by Davenport and Bain, and started to be mentioned in books in 1947 [1]. Looking at the use of steel throughout history, bainitic steels began to take place in industry very lately. There are a lot of properties waiting to be discovered about bainitic steels.

Conventional steels used in industry provide either high strength low ductility or high ductility low strength. Bainite on the other hand, can provide both high strength and high ductility at the same time. Tempered martensite is the closest phase in terms of mechanical properties, but still bainitic steels have higher ductility than martensitic steels. Bain mentioned about this property as “tougher for the same hardness than tempered martensite” [2]. Besides, tempered martensite requires a second heat treatment step after austenitization and quenching. Bainite is obtained by quenching to bainite start temperature and holding isothermally until having fully bainitic structure. Secondary heat treatment step is eliminated since annealing is not required to increase toughness, so for economic aspect, bainite production is cheaper than tempered martensite production.

Bainite is a microstructure that consists of a non-lamellar mixture of ferrite and cementite [3]. Temperature range of bainite formation is below where fine pearlite forms and just above martensite start temperature. Bainite can be stated as a transition between these two structures and this can be interpreted as bainite has strengths of these two phases; hardness and tensile strength of martensite and ductility of pearlite are met at bainitic structure. When industry is reviewed from the point of potential usage areas of bainitic steel, railways and automotive industry are among the examples. In railway applications, pearlitic steels have been used commonly and wear problem occurs after a long-term usage. Bainitic steel has higher hardness relative to pearlitic steel and bainitic steels have been used on railway rails lately, owing to solve wear problem. In automotive industry, strength and energy absorbing capacity of a metal are important criteria for safety concerns. For example, A-pillar toughness should be high in order to absorb energy during an accident. Bainitic steels answer the need of high toughness requirement.

Bainitic transformation of low carbon steels were studied in detail in literature but mostly focused on Si, Mo, Mn, Ni or Al added alloys. Boron containing low-to-medium carbon steel 32CrB4 have not been studied in terms of bainitic transformation. It is also observed that continuous cooling process was applied mostly in these studies and being a common process in industry can be the reason. Isothermal cooling, on the other hand, was studied relatively few.

There is a trend in which bainitic steel is used instead of pearlitic steel in railways due to higher wear resistance, weldability, good fatigue resistance and machinability. Bainitic transformation of high-carbon railway steel R260 has not been studied in literature by applying isothermal cooling.

Multi-step isothermal transformation is another intriguing subject for being a promising way to obtain higher strength and ductility by eliminating blocky retained austenite or martensite phase, and forming secondary and finer bainitic phase. For high-carbon steel, two-step transformation is held by Hase et al. [4], and they observe an improvement in mechanical properties but surprisingly austenite percentage was higher than one-step transformation. The difference was austenitic phase had finer structure. For medium carbon steel, multi-step isothermal bainitic transformation experiments were carried out by Wang et al. [5] with the aim of increasing in mechanical properties of bainitic steel and they achieved it by increasing bainite volume fraction and also reducing the size of bainite plate thickness.

In this study, boron containing low carbon steel 32CrB4 and high-carbon railway steel R260 were heat treated to obtain bainitic structure, and mechanical properties were examined. In opposite to previous studies, the aim in this study is to use ISOTHERMAL COOLING for obtaining bainitic structures. By this way the microstructure can be designed more strictly and 100% bainitic or pre-determined ratios of martensitic/bainitic microstructures can be obtained. This would make possible to determine the mechanical behavior of phases much more clearly. The aim is also to show that bainite can have high strength level as tempered martensite but more ductile relative to it, while their hardness values are similar. Heat treatments were designed according to the compositions of steels. Two-step isothermal transformation is also applied to 32CrB4 steel, as one of the way for reaching to the goal of this study. Time and temperature are main variables of heat treatment that controls the final structure.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1. Martensite

Martensite is a non-equilibrium phase that transforms from austenite by rapid quenching to relatively low temperatures. Cooling rate is very high and because of that, diffusionless transformation takes place. Carbon atoms do not have time to diffuse and they stay as interstitial impurities, which makes martensite a carbon supersaturated solid solution. Shear mechanism plays a role at this transformation, large number of atoms make a cooperative movement and there is only a slight displacement for an atom from its neighbors. Face-centered cubic (FCC) austenite becomes body-centered tetragonal (BCT) martensite as Figure 1 shows, which is an elongated body-centered cube from one dimension, because of the trapped carbon atom.

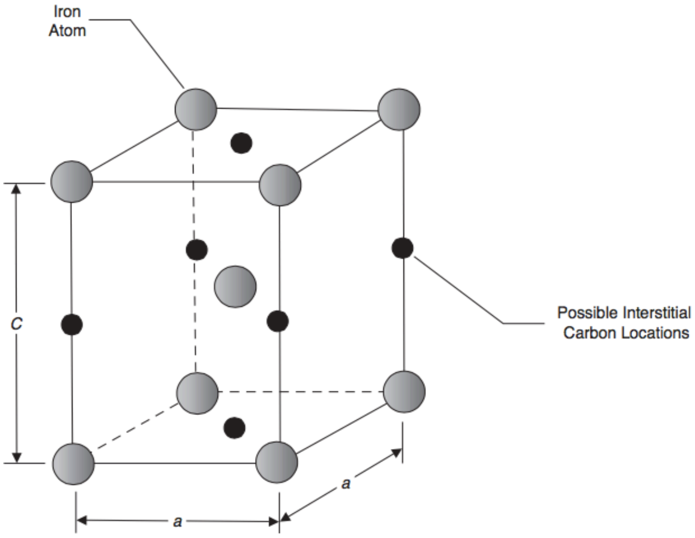


Figure 1. BCT structure of martensite and possible interstitial locations for carbon atom [6]

During martensite formation, lattice distortion occurs due to the shearing. This distortion creates a tilted surface relief as shown in Figure 2, and this movement is parallel to a certain crystallographic plane, called as habit plane, which is {111} for iron atoms. There are additional deformations of martensite, resulted from maintaining an unrotated and undistorted habit plane in the bulk austenite. All of these deformations create high density of dislocations in martensite. Transforming from FCC to relatively less densely packed BCT, results in a volumetric expansion and hardening. Due to the high dislocation density, fine structure and expansion, martensite becomes very strong phase [6].

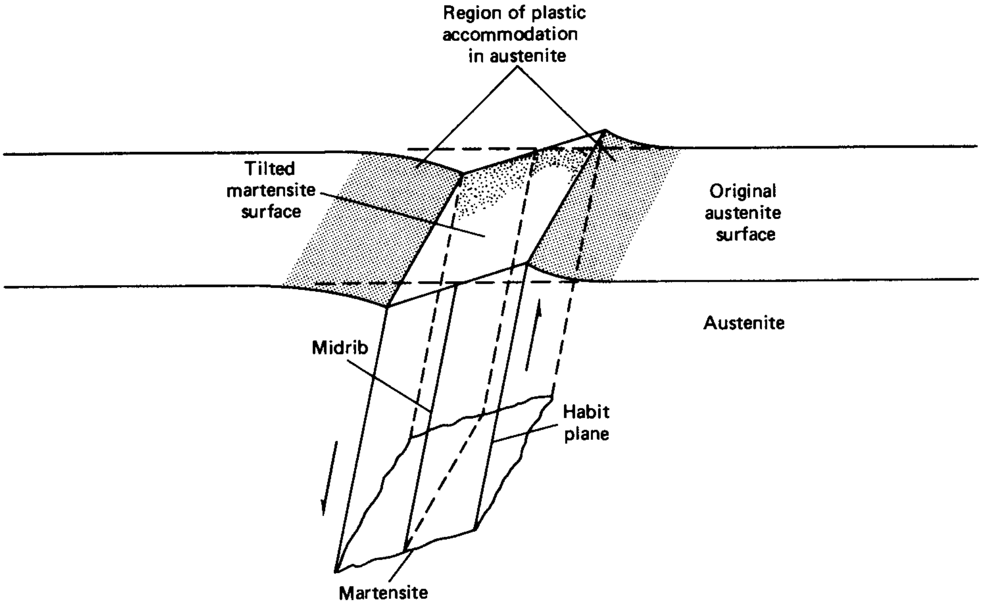


Figure 2. Tilted surface as a result of shear mechanism of martensite formation [6]

As-quenched martensite has very high strength, however it is poor in toughness and fracture resistance, that makes it not proper for most of the applications. Internal stresses introduced during transformation have weakening effect on material. In order to decrease brittleness and increase ductility, after quenching operation, tempering is applied to martensite.

Tempering is a heat treatment applied to quenched or deformed steels in order to relieve stresses. Steel is heated to a temperature below the eutectoid transformation temperature for a specified time. Temperature range for tempering is between 250°C and 650°C, time interval is usually 2 hours but there is not a specified time, since the mechanism depends on diffusion, longer holding time decreases the hardness. Martensite phase transforms to a tempered martensite by diffusion (Figure 3). Single phase BCT martensite decomposes into stable ferrite and cementite phases. Cementite particles are extremely small and embedded into a ferrite matrix. By tempering the martensite, ductility is gained but there is a sacrifice from strength, but still tempered martensite has higher strength than pearlite with a sufficient ductility level [7].

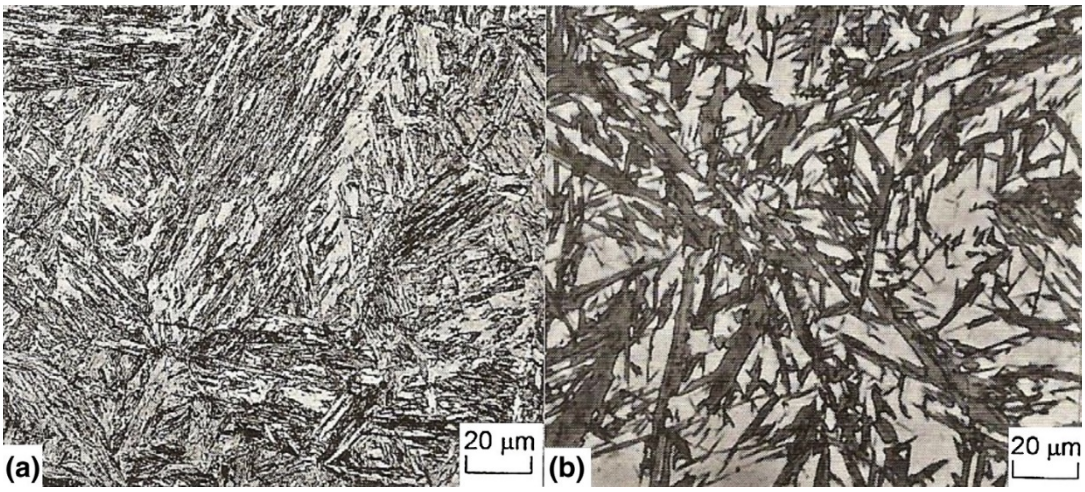


Figure 3. (a) Tempered martensite of 4130 steel, (b) plate martensite of high-carbon steel [8]

Because of the fact that martensite is a non-equilibrium phase, it does not have a representation on iron-carbon phase diagram. Instead, it is shown on TTT diagram and since it forms suddenly, martensite start (M_s) and finish (M_f) temperatures are defined as a constant temperature. In order to form fully martensitic structure, quenching rate should be high enough to not to cross TTT nose, and also pass M_f temperature and M_f should be above room temperature. If M_f is lower than room temperature, than all of the austenite can not transform to martensite, the untransformed austenite is called as retained austenite. If cooling line crosses to TTT nose, then other structures like pearlite or bainite will form together with the martensite [9].

2.2. Bainite

Bainite is a transformation product of austenite, obtained by cooling with a high rate relative to equilibrium phase transformations which makes bainite a non-equilibrium phase. Equilibrium phases are formed by very slow cooling of a metal and phases at iron-carbon phase diagram can formed by this way. In industry, such slow cooling processes are not applied due to economic concerns. Cooling suddenly from elevated temperatures to much lower temperatures produces non-equilibrium phases and bainite is one of them [10]. Time temperature diagrams are used for foreseeing which phase is going to be obtained by cooling to specific temperature at a certain time. Bainite is placed between pearlite and martensite at time temperature transformation diagrams and cooling rates of these phases are also in the same order. Cooling rate of pearlite allows a diffusion controlled transformation but martensite has such a high cooling rate that diffusion is not possible, because of that, diffusionless transformation occurs during martensite formation [11].

For many alloy steels, bainite forms as a result of isothermal transformation. Pearlite and bainite transformation temperature ranges are separated by alloying element addition (Figure 4). For plain carbon steels, on the other hand, transformation temperature ranges overlap and during continuous cooling, pearlite and bainite can form successively if cooling rate is set suitably [6].

Bainitic microstructure is composed of ferrite and cementite, as it is in pearlitic microstructure. There are differences in ferrite and cementite phase structures of pearlite and bainite, which make them two different structures. Bainitic ferrite is in acicular shape, similar to the laths of martensite, and it does not develop a lamellar structure as in pearlite. Cementite phase in bainite is very fine and chunky [6].

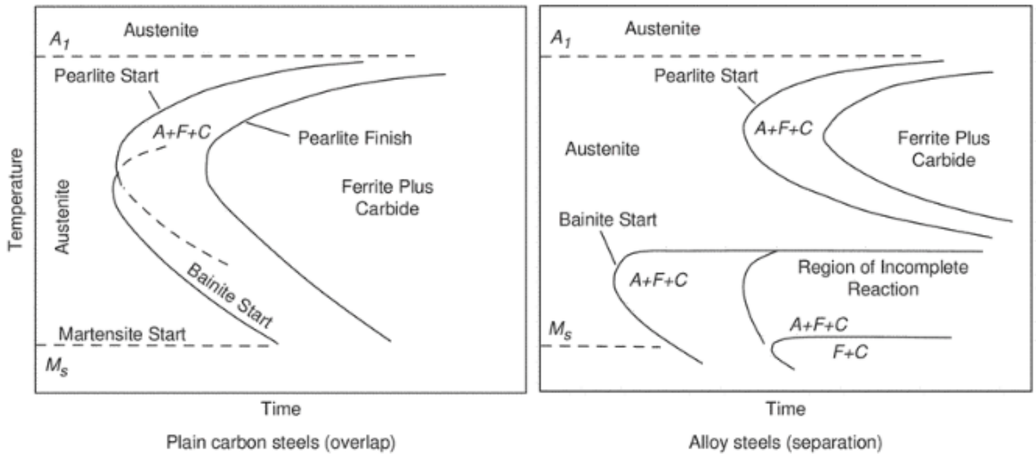


Figure 4. Effect of alloying elements on TTT diagram [6]

Ferrite plate aggregates in bainite are called sheaves and each sheaf is called sub-unit (Figure 5). Sub-units are connected with each other and their crystallographic orientations are the same. The shape of sheaf is wedge-like, larger at one end where nucleation is started and becomes smaller gradually towards to tip. Sub-units of a sheaf have nearly equal dimensions and since they nucleate close to tip of the existing sub-units, they reach to a certain size. Davenport stated that if sub-units are in lath shape, close-packed direction of ferrite becomes the longest, which has minimum angle with parent close-packed austenite. Size of a sheaf depends on transformation temperature; thickness/length aspect ratio of a sheaf decreases as transformation temperature decreases [1].

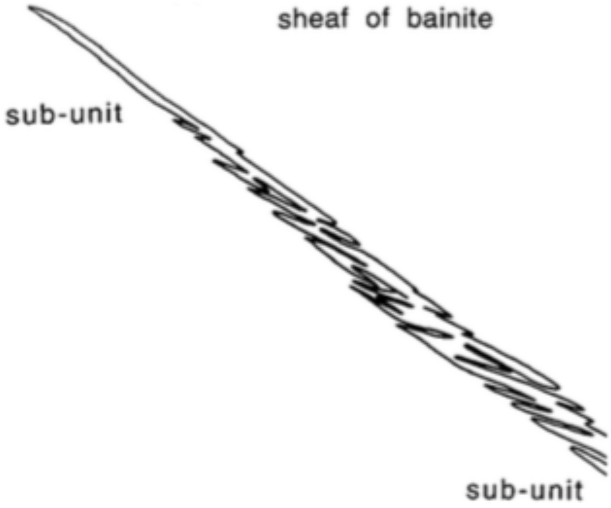


Figure 5. Bainite sheaf illustration [1]

Crystal structure of austenite is FCC and when temperature is decreased sufficiently to increase free energy change that creates driving force, BCC ferrite transformation occurs. Bainite is classified as upper and lower bainite by Mehl [12] based on the transformation temperature. As the names imply, upper side of bainite transformation temperature range belongs to upper bainite and the lower part belongs to lower bainite.

2.2.1. Upper Bainite

Upper bainite forms at higher temperatures, typically below 500°C. At TTT diagram, bainite transformation temperature range starts just below the pearlite transformation range [10]. After austenitization, quenching to upper bainite range and holding at that area isothermally gives an upper bainite structure. Figure 6 shows the upper bainite region, and an isothermal cooling path that produces upper bainite structure. Ferrite plates start to nucleate at austenite grain boundaries and they grow parallel to each other. Cementite grows between ferrite plates and in the same direction with ferrite, as a result they have elongated shape [6]. Since ferrite has low carbon solubility, carbon partitioning from bainitic ferrite to austenite takes place. Carbon content of austenite becomes increased as a result of carbon partitioning and carbides precipitate from austenite (Figure 7). Bainitic ferrite does not contain precipitated carbides [13].

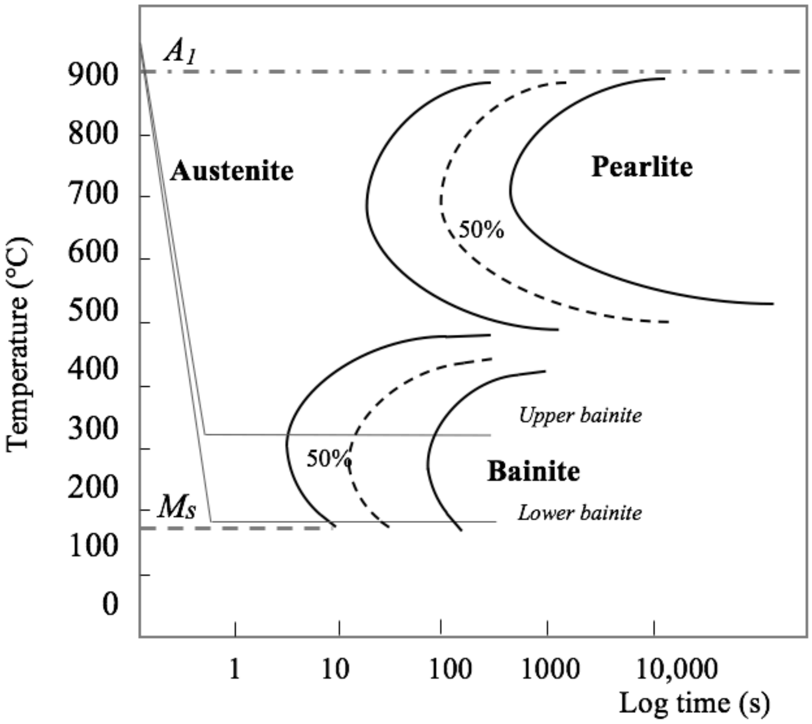


Figure 6. TTT diagram showing upper and lower bainite regions and example of isothermal cooling paths

After nucleation of ferrite plates, growth takes place and shape change occurs at the transformed regions. Shape change creates a strain with a shear component and it is called as invariant-plane strain. Martensite formation also produces strain but for bainite transformation, temperature is higher. Strains are relieved by plastic deformation of the contiguous austenite and as a result dislocation density becomes increased at that areas. High dislocation density stops the movable transformation interface and thus growth of bainitic ferrite stops. This process explains why there is a certain size of bainite plates and they are usually smaller than grains they formed from [14].

Cementite phase percentage and microstructure depends on the carbon concentration. If alloy has low concentrations, separated particles of cementite phase will form; if alloy has high concentration, continuous and distinct cementite phase will form between ferrite laths [14]. In terms of upper and lower bainite, elongated cementite particles are seen at upper bainite and small and discrete particles are seen at lower bainite [15].

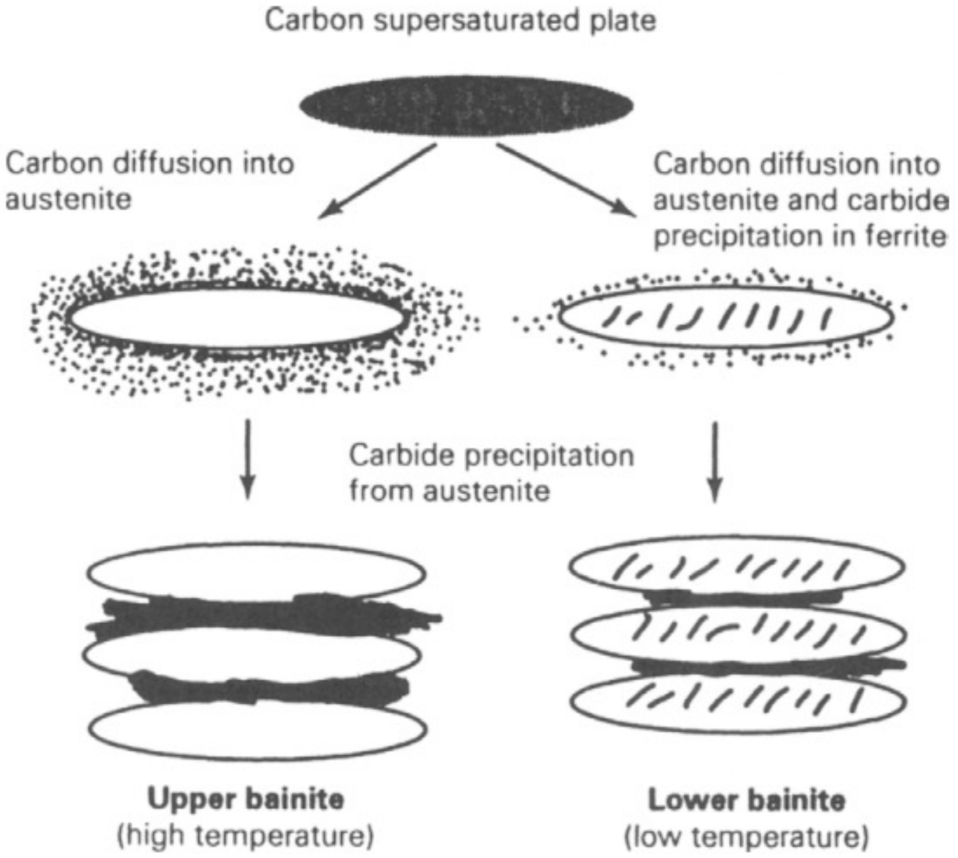
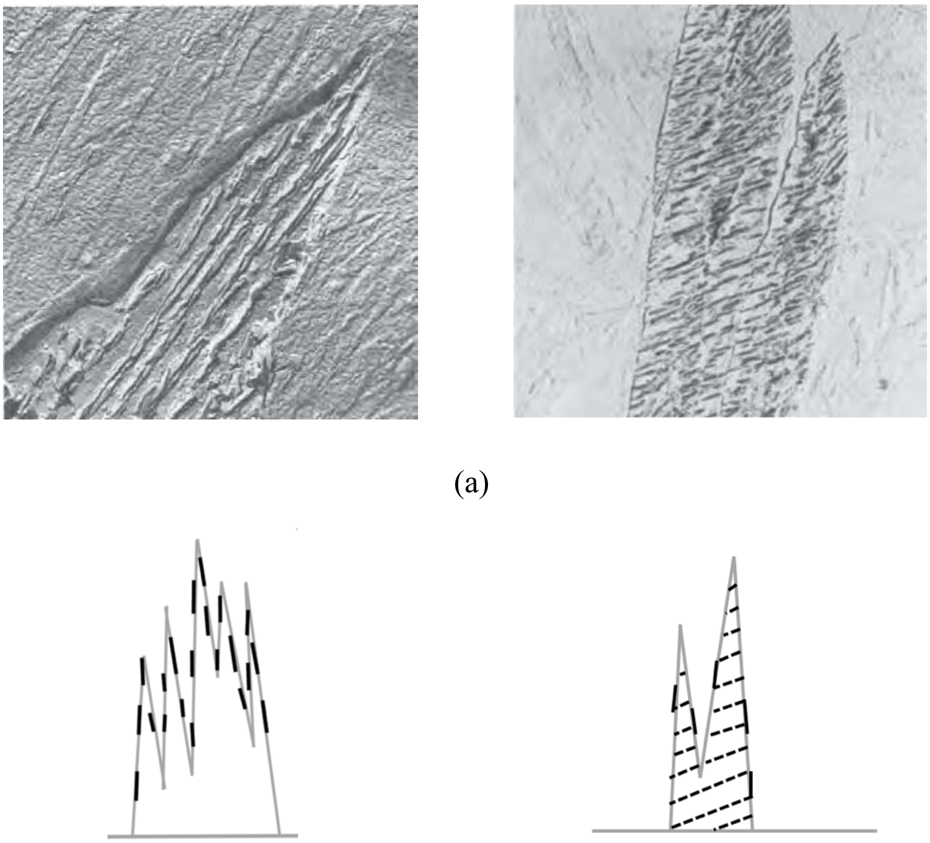


Figure 7. Carbon diffusion and precipitation for upper and lower bainite [14]

Upper bainite microstructure described as feathery and coarser relative to lower bainite, as it can be seen in Figure 8 [6], if carbon concentration is decreased, structure becomes coarser. In terms of subunits, on the other hand, upper bainite has smaller structure relative to lower bainite [16]. For microscopic analyzes, bainite structure seems dark after etching, and if there are light areas, they are either untransformed austenite or martensite. The reason why austenite transformation does not take place is that, carbon partitioning increases the carbon percentage of austenite and martensite start temperature for this new composition becomes lowered below the room temperature. After heat treatment of bainitic transformation, alloy is cooled in air as the last step and new alloy does not have sufficient undercooling to transform [7]. If alloy can be cooled below the martensite start temperature, then martensite forms. These potential second phases do not form if bainitic transformation is completed and alloy becomes fully bainitic. Some other phases such as pearlite also can co-exist with bainite, especially continuous cooling is applied.



Upper bainite Lower bainite

(b)

Figure 8. (a) Electron microscope images of upper and lower bainite microstructures [6] and (b) sketch of upper and lower bainite microstructures and position of carbides and precipitation sites.

2.2.2. Lower Bainite

Lower bainite transformation occurs at low temperatures and the lowest temperature at which transformation can occur is the martensite start temperature. Ferrite plates are fine and acicular rather than feathery and the major difference between upper and lower bainite is that carbides precipitate inside the lower bainitic ferrite plates. There are two mechanisms for carbide precipitation, first one is from carbon saturated austenite, the same as described at upper bainite, and second is from supersaturated ferrite. Since transformation temperature is low, carbon diffusion from ferrite to austenite is slower and ferrite plates become carbon enriched causing cementite precipitation to occur inside ferritic plates [6].

Carbide amount partitioned to the austenite is very few and because of this, carbide precipitations in austenite are very small in size in lower bainite. Upper bainite, on the other hand, has larger cementite particles and large carbides are source for crack initiation [14]. Fine bainitic ferrites in lower bainite have different orientations and high angle grain boundaries separate them, whereas low-angle grain boundaries exist in upper bainite and ferrites are nearly parallel to each other [17]. These two phenomena explain why lower bainite has more strength and toughness than upper bainite has. Transformation temperature effect on tensile strength of low-carbon bainite can be seen in Figure 9.

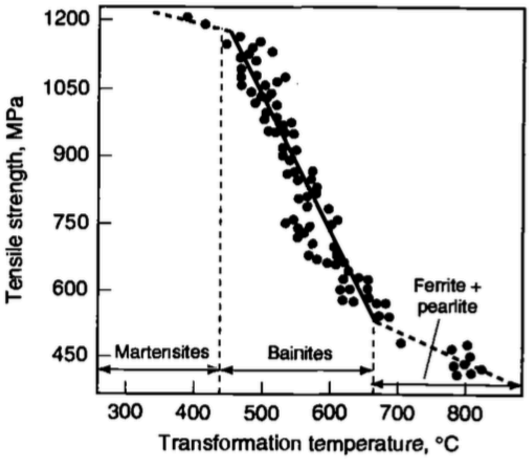


Figure 9. Diagram showing the effect of transformation temperature on tensile strength of low-carbon steel [18]

Lower bainite can appear very similar to tempered martensite since they both have fine carbide. The difference is that cementite particles in bainite precipitate about 60° to the major axis of the bainite plate whereas carbides of tempered martensite precipitate in a multivariant Widmanstätten arrays. There are also exceptions such as Widmanstätten arrays can be found in lower bainite if carbon concentration is high or transformation temperature is low. If alloy has low carbon, martensite phase can form an array similar to that of lower bainite after tempering [14] [19].

Abbaszadeh *et al.* held a study in order to see the effects of bainite on mechanical properties of mixed bainite-martensite microstructure. They used D6AC steel that is composed of a 0.46%C-0.75%Mn-1.1% Cr-0.25% Si-0.6%Ni-1.0%Mo-0.1%V. First, they austenitized all samples and then quenched them in three different ways; oil quenching, quenching in salt bath at 330°C and at 425°C isothermally for various holding times. Then they were all tempered at 200°C for 2 hours. After mechanical tests were held, they found that lower bainite-martensite mix shows higher yield and tensile strength than fully martensitic (it should be noticed that samples were tempered) microstructure. Charpy V-notch (CVN) impact energy and ductility is improved as bainite percentage is increased. Upper bainite-martensite mix, on the other hand, has lower strength and also low CVN impact properties relative to fully martensitic microstructure. They attributed the lower bainite-martensite mix having high mechanical properties to finer and uniform carbide distribution, lower thickness of bainitic ferrite plate and higher dislocation density in lower bainite [20].

2.3. Transformation Mechanism of Bainite

There is an ongoing controversy about the transformation mechanism of bainite, one theory asserts that growth of bainite is diffusion controlled transformation and the other one says that displacive, diffusionless mechanism is responsible for bainite growth.

Diffusionless growth is explained by shear mechanism, many atoms make a cooperative glide-type movement, displacement of an atom relative to its neighbor is very slight. Martensite forms via diffusionless mechanism and this theory for bainite is explained by showing the similarities of martensite and bainite formation and their morphologies. Nucleation is explained with carbon partitioning, growth is displacive and stopped by adjacent austenite since there is a plastic relaxation. Size of a sub-unit is restricted by frictional stress related with plastic deformation caused by a shape change. In this case, propagation of a sheaf is dominated by nucleation, at the tip of the completed sub-unit.

Diffusive theory, on the other hand, tells that both nucleation and growth have reconstructive mechanism. Longitudinal growth is greater than latitudinal growth, resulting that dentations to form. Bainitic ferrite and cementite grows at the same time since carbon partitioning increases the driving force for cementite formation. Metallographic similarities between bainite and other diffusively formed structures are again used as a supporting evidence, also AFM and high-resolution measurements were done. Both theories have their evidences and because of this, transformation mechanism is still not understood well and cannot be described completely [16].

Theoretically, bainite forms above M_s temperature, nevertheless, there are studies proving that bainitic transformation can occur by isothermal holding approximately 10°C below M_s temperature. After it was mentioned that bainitic structure could form in such conditions, TEM examinations proved that lower bainite exists in microstructure formed by holding isothermally below M_s temperature, but formed together with martensite. Growth rate of bainite below M_s is observed slower than both growth rates of martensite and bainite above M_s [21]. Samanta *et. al.* found that transformation mechanism below M_s is similar with the displacive transformation mechanism model and they also mentioned that martensite and bainite from the same parent austenite grain having common crystallographic orientation. In the light of these study, they suggest that bainite transformation mechanism is displacive [22].

2.4. Basic Heat Treatment of Bainite Formation

Steels are heat treated in order to obtain desired mechanical properties by changing the microstructure. Bainite is obtained by a heat treatment process that contains austenitization, cooling to bainite start temperature (B_s) and then air cooling, respectively.

Bainite is a product of an austenite decomposition, and this is why material should be heated first to elevated temperatures. After austenitization, material should be cooled. Cooling can be continuous or isothermal, for conventional applications continuous cooling is favored mostly, due to economic concerns. Continuous cooling has some disadvantages in terms of the obtained bainite percentage. During cooling, there could be an interception with pearlite nose and small amount of pearlite can form. Quenching to B_s and holding isothermally, on the other hand, allows to directly step into the bainite transformation range. When B_s temperature is reached, eutectoid reaction takes place, and bainitic ferrite forms:

$$\gamma\ (0.76\text{wt\%C}) \rightleftharpoons \alpha\ (0.022\text{wt\%C}) + Fe_3C\ (6.7\text{wt\%C})$$

Isothermal holding should be long enough to transform all austenite to bainite, and required time interval is decided according to TTT diagram of the alloy. After that, steel can be cooled in air [9].

Quenching can be done in different mediums such as water, water spray, water-based solutions or oil, but they can cause distortions, cracks or non-uniform hardness. Also, vapor layer forms at the surface of the material and retards cooling for mediums that has a low melting point. Molten salt, on the other hand, has many advantages with respect to those other quenchants.

Nitride-based salts have wide operating temperature range, between 150 to 595°C, and molten salt has no vapor phase, meaning that problem caused by vapor phase is eliminated. Salt temperature can be controlled and severity of medium becomes proper for material. They have excellent thermal and chemical stability, which means that it is proper for serial quenching operations and also parts rapidly attain temperature equalization. They enable isothermal holding at a specified temperature without fluctuation. After quenching is completed, residual salt can be cleaned by washing with water easily. There are several limitations of using salt bath. It can not be used below its melting point, which is usually 150°C. It should be considered that salts have oxidizing nature.

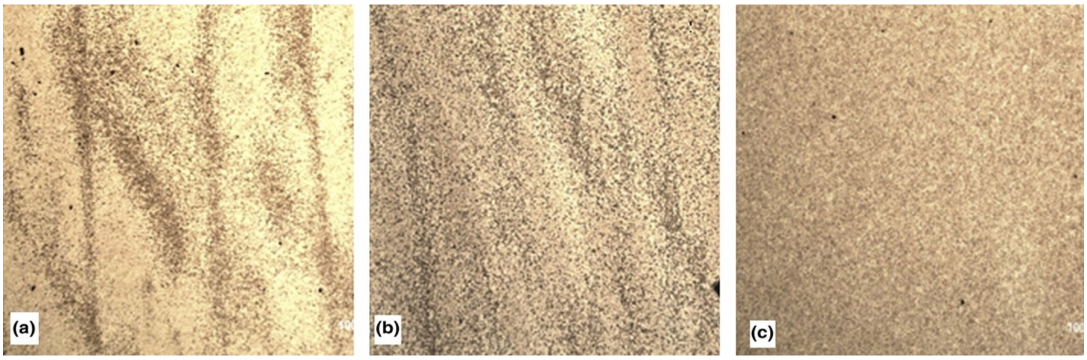


Figure 10. Micrographs of bainitic transformation of AISI O1 steel, (a) after 10 min. (b) after 20 min. (c) after 40 min. [23]

After isothermal heat treatment, banded structure can develop. Figure 10 shows bainitic transformation period of AISI O1 steel which is austenitized and cooled by quenching to 320°C in salt bath. Banded structure can be seen in the beginning of cooling as it is shown in Figure 10a, it is a result of segregation effect differences. Banded structure disappears gradually as shown in Figure 10b and 10c [23].

2.5. Multi-step Isothermal Transformation

When austenite is quenched to just above M_s temperature and held isothermally, bainite transformation occurs, but at the end, obtained structure may not be fully bainitic. There can be blocky retained austenite or martensite phases can be seen. These phases increase the brittleness of the material and decreases the toughness.

Multi-step quenching is a way to increase the bainite percentage and decrease the blocky austenite/martensite phases.

In one-step quenching process, carbon partitioning from bainite to austenite increases the carbon concentration of austenite, and M_s temperature of this new alloy becomes lowered. After a while, bainite transformation stops, in order to make alloy continue to transform, isothermal holding temperature should be lowered too. Wang *et al*. states that second quenching temperature should be lower than the M_s of the original alloy but higher than the M_s of carbon enriched untransformed austenite (Figure 11). With this second quenching process, blocky austenite will be reduced, and toughness of the material will be increased [5]. Blocky austenite is reduced because by lowering the isothermal holding temperature, austenite with increased carbon content can now complete the transformation to bainitic ferrite. This subsequently formed bainitic ferrite structures are finer than the first ones because of the high carbon content.

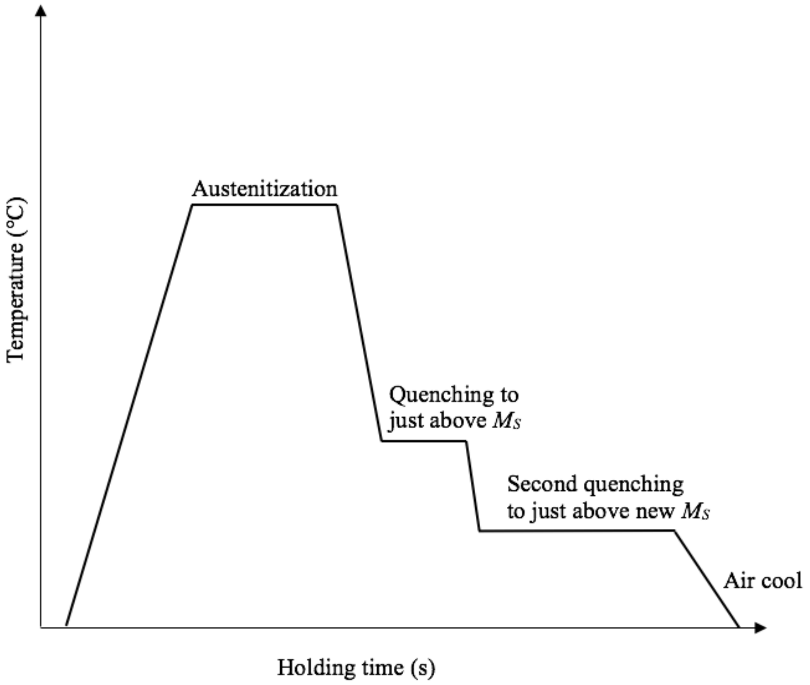


Figure 11. Schematic heat treatment path for two-step quenching process

Wang et al. used a steel with a composition of Fe - 0.30C - 1.46Si - 1.97Mn - 1.50Ni - 0.30Cr - 0.96Cu - 0.25Mo in wt.%. Carbon percentage of their steel is nearly the same as the steel 32CrB4 used in this study. Their experiments include conventional bainitic transformation, two-step quenching and three step quenching transformations. Microstructures of obtained steels can be seen in Figure 12 [5].

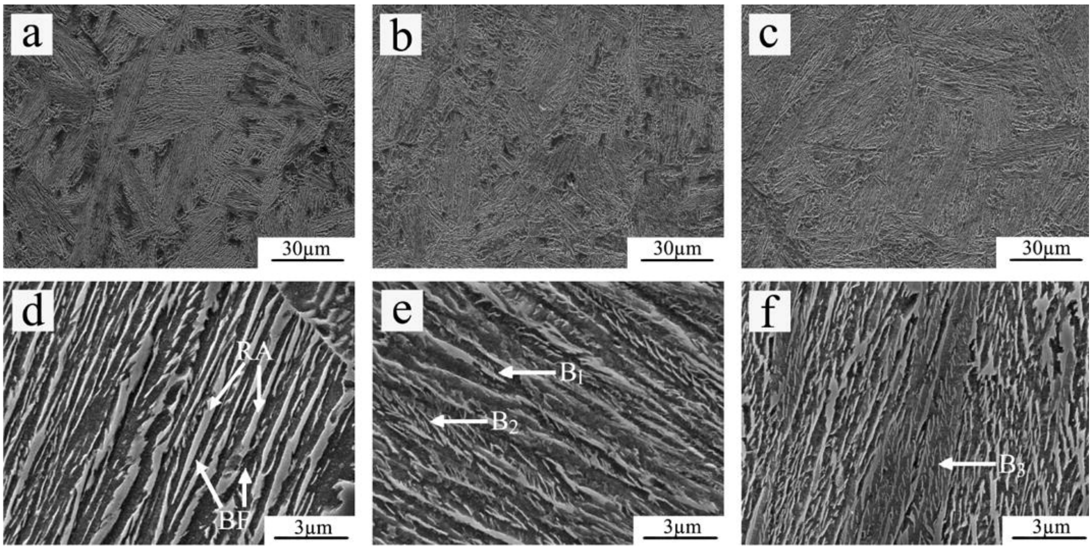


Figure 12. SEM images of (a)(d) conventional bainitic transformation, (b)(e) two-step quenching bainitic transformation, (c)(f) three-step quenching bainitic transformation [5].

Hase *et al.* studied to step isothermal transformation for bainite formation. A high-carbon steel with Si, Mn, Al, Cr, Co alloying elements, first austenitized and then cooled with 25°C/s rate to temperature between 250-350°C, hold for 18 hours. After that, they quenched alloy to 250°C and hold for 18 hours, then cooled with 50°C/s to room temperature. They found that two-step quenching is beneficial for mechanical properties; they achieve ductility of 40% of total elongation, 63 MPa $m^{-1/2}$ toughness at an ultimate tensile strength of 1.5 GPa. It is also interesting that austenite percentage of two-step quenching process is higher than one-step quenching has, which is not an expected result but this increases the toughness since refinement of the blocky austenite is achieved. They also stated that two-step quenching leads to a bimodal size distribution of bainite plate [4].

2.6. 32CrB4 Steel

32CrB4 is a chromium and boron alloyed low-carbon steel and proper for cold heading and cold extrusion, with a subsequent quenching and tempering. Typical application fields of this alloy are ship, vehicle, airplane, railway, bridge, pressure vessel, machine tools.

Tensile strength and hardness values obtained at room temperature after quenching at 870°C in water, according to EN 10263-4: 2003 for 32CrB4 can be seen at Table 1.

Table 1. Hardness and tensile strength values of tempered samples according to EN 10263-4 [24]

HRC	52	51	49	48	46	44	41	39	35	32	29	22
UTS (N/mm^2)	1880	1820	1700	1640	1520	1430	1300	1220	1080	1010	930	790
Tempering at (°C)	100	150	200	250	300	350	400	450	500	550	600	650

Medium-carbon steels have carbon concentration between nearly 0.25 and 0.60 wt%. They are used in areas where high mechanical properties are needed. Plain medium-carbon steels have low hardenability but with addition of chromium, nickel and molybdenum, steel can be hardened. Mechanical properties are enhanced by heat treatment processes.

Medium carbon steels are mostly used for rods, screws, gears, shafts, filters, machined metal parts, components that requires combination of high strength, toughness and wear resistance [9], [25]. 32CrB4 steel is used mostly for rods and screws, machine and metal parts, filters, injector, shafts and gears. In literature, only the effect of deformation on CCT diagram of 32CrB4 steel was studied [26]; bainite transformation, on the other hand, has not been studied as far as gathered from the literature.

2.6.1. Effect of Boron

Boron is an interstitial element with a high diffusivity in austenite and very low solubility in ferrite solid-solution (<0.003%). Main effect of boron to heat treatable steels is to enhance their hardness. Machinability relative to the boron-free steels at the same hardness level is increased. Quench cracking and heat treatment distortions are reduced for boron steels [27].

Boron affects bainitic transformation in a very beneficial way that early commercial development of bainitic steels relied on it. Nucleation of allotriomorphic ferrite at the austenite grain boundaries is retarded more than that of bainitic ferrite by boron atoms (Figure 13). The reason why bainitic ferrite is affected less than allotriomorphic ferrite has not been studied, but Bhadeshia associated this effect with the fact that bainite nucleates autocatalytically. Growth of bainite is in the form of sheaves of small platelets and after the initial nucleation at the grain boundaries, most of the platelets nucleate autocatalytically [1].

Since boron has high diffusivity in austenite and solubility of boron is reduced by cooling the alloy, as soon as cooling begins, boron starts to segregate to the austenite grain boundaries, even before austenite to bainite transformation starts. Grain boundaries are preferential nucleation sites for ferrite, and boron has a reducing effect on the nucleation rate of ferrite. This is because of possible nucleation sites are occupied by boron atoms and grain boundary energy becomes lowered. If grains become finer, effect of boron will be higher, so austenitization at high temperature for long time will reduce the effect of boron. Also, self-diffusion coefficient of iron at grain boundaries is reduced by boron, which leads to the decrease in ferrite nucleation. [28].

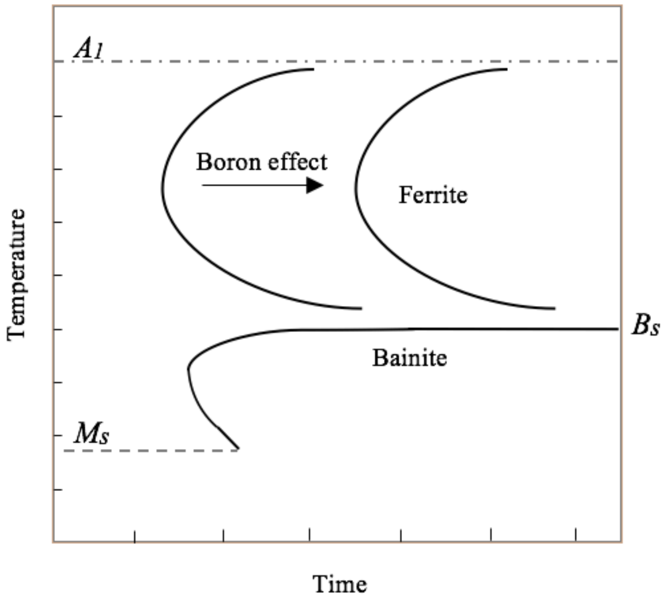


Figure 13. Effect of boron on TTT diagram of a steel.

It is known that trace amount of boron should be added into alloy to benefit from it. There is a critical boron content and if it is exceeded, it becomes detrimental for alloy. Borocarbide $M_{23}(C,B)_6$ starts to precipitate at the grain boundaries and becomes a potential nucleation site for ferrite, which reduces the hardenability [29]. The concentration range that providing maximum effect of boron is between 0.0005 to 0.003%.

2.6.2. Effect of Chromium

Chromium forms the basis for stainless steels by increasing corrosion and oxidation resistance. It is a substitutional element for steels and increase the hardness. Wear resistance becomes improved by addition of chromium since it is one of the strong carbide forming elements. At high temperatures, softening is delayed by chromium, so it increases strength at high-temperature [6] [25].

When effects of chromium on bainite is considered, addition of chromium is mainly for increase hardenability of steel, so that bainite transformation at lower cooling rates can be done more easily [30]. Brecke and Xu states that chromium reduces the kinetics of lower bainite transformation, obtained by continuous cooling [31]. It also contributes to depressing the B_s temperature and shifting T_0 curve to lower carbon concentrations [11].

2.6.3. Effect of Carbon

Carbon is the main and the most important alloying element for steel since it forms cementite, Fe_3C. Pearlite, bainite, martensite, spheroidite, and other carbides require carbon. All mechanical properties are affected from carbon content. High carbon content increases strength and hardness (also hardenability), but decreases toughness and ductility [32]. Other alloying elements and heat treatments are applied for altering mechanical properties of steel according to needs. Bainite start temperature is lowered very effectively by carbon. Lowered B_s leads to microstructure to form in finer scale [11].

Long *et al.* used a steel that has the same carbon percentage with the 32CrB4 steel in their study. Composition of their steel was given as Fe - 0.34C - 0.01Si - 1.61Mn - 1.24Cr - 0.96Ni - 0.45Mo - 0.04Al - 0.002S - 0.005P - 0.003N in wt%. After austenitization, they quenched to M_s + 10°C, which is 350°C and hold isothermally for 1 hour. Figure 14b and 14d shows microstructure they obtained by using this steel. The resultant microstructure contains bainitic ferrite and carbides [33].

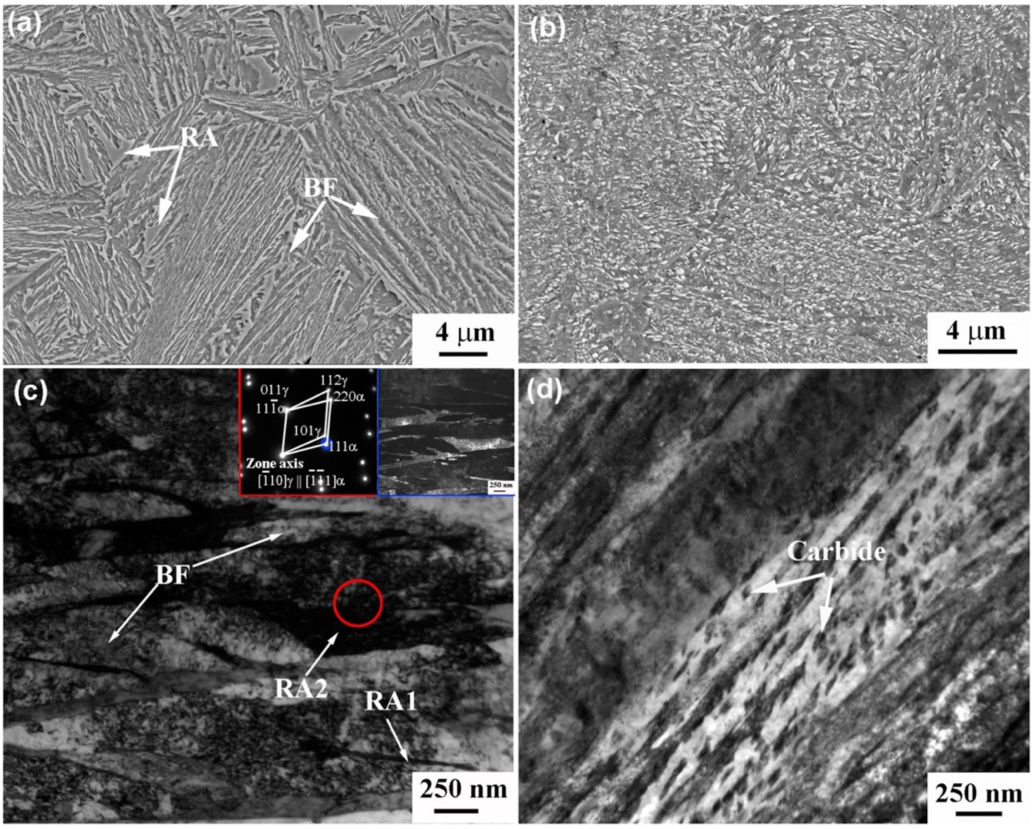


Figure 14. SEM and TEM micrographs of (a)(c) carbide-free bainite structure, (b)(d) carbide-bearing lower bainite structure, studied by Long *et al.*, by using 0.34C% [33].

2.7. Railway Steel R260

R260 is a high-carbon alloy steel used at railway rails. Mechanical properties of the alloy are shown in Table 2.

Table 2. Mechanical properties of R260 steel

Alloy	Tensile strength (MPa)	Yield Strength (MPa)	Elongation (%)	BHN Hardness
R260 according to EN 13674-1	≥ 880	-	≥10	260 - 300
R260 according to a study of Johnson *et al.* [34]	951	731	15.5	282

High-carbon steels have a carbon content between 0.60wt% and 1.4wt%. They are the hardest and strongest steels relative to other carbon steels. High carbon content is added in order to improve wear characteristics. For railway applications, high wear resistance is one of the most required specifications. Rail steel also should have a resistance to contact failures and low-temperature brittle fracture, which are all related to basic strength and plasticity properties such as yield strength, ultimate tensile strength, percent elongation. Microstructure is one of the key parameter that can control these properties.

High carbon steels have a tendency for cementite precipitation as thin films at the austenite grain boundaries when transform to bainite. Toughness of steel becomes lowered by the effect of these thin films. The growth of thin films can be suppressed by decreasing the transformation temperature [1].

An example of high-carbon bainite can be seen in Figure 15, by using Fe-0.6C-2Si-1Mn (in wt%) steel. Figure 15a shows lath-shaped upper bainite formed at 450°C and Figure 15b shows plate-shape lower bainite formed at 350°C [35].

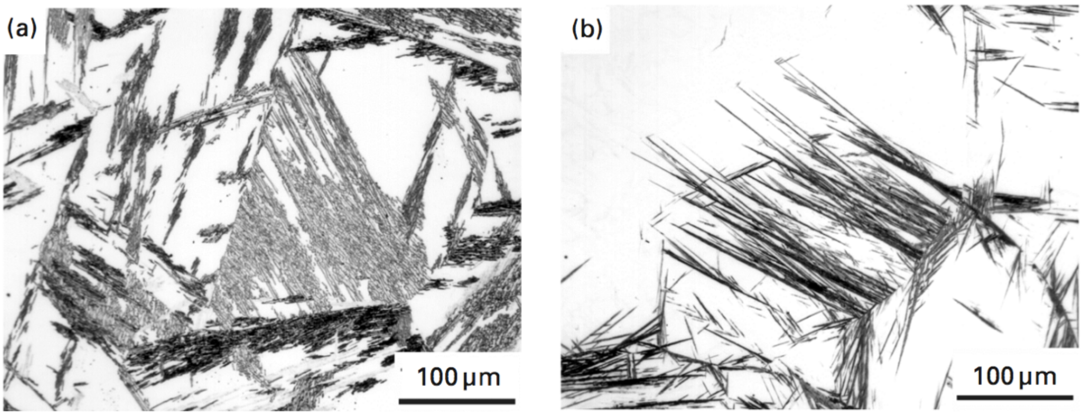


Figure 15. Optical micrographs of (a) upper bainite (b) lower bainite of high-carbon steel [35]

Baradari et al. used Fe - 0.66C - 0.80Si - 0.99Mn - 0.84Al - 0.95Cr - 0.19Mo - 1.53Co (in wt%) steel in their study. After they austenitized samples, quenching and isothermal holding processes were held in salt bath at 300, 330 and 360°C for different holding times. At the end, bainite and retained austenite phases were obtained. Figure 16 shows optical micrographs of these three samples [36].

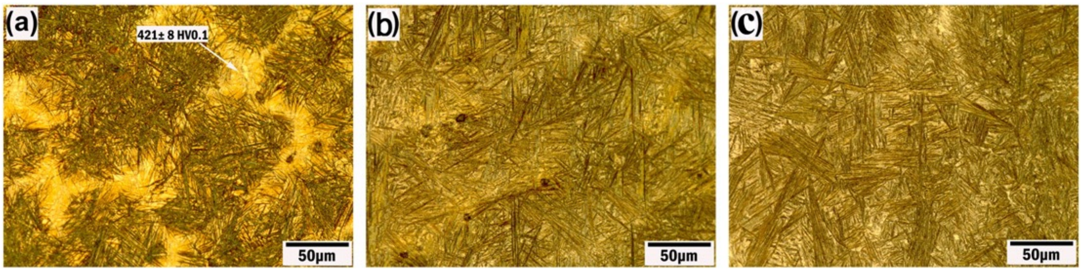


Figure 16. Optical micrographs of samples subjected to isothermal holding at (a) 300°C for 8 h (b) 330°C for 4 h and (c) 360°C for 1 h [36].

R260 is a conventional railway steel that has pearlitic structure. If bainitic structure can be obtained by using R260 steel, then those required specifications will be answered satisfactorily relative to current used state. Bainite transformation of R260 steel was not studied in literature and in this study, bainite transformation just above M_s will be investigated. By obtaining bainite phase, strength and hardness will be higher, which leads to an increase in wear resistance, and toughness is not going to be lowered as much as it is in martensite. It can respond to needs, with a small sacrifice from toughness.

As mentioned before, bainite transformation improves the tensile strength and toughness of steels at the same time, by heat treating the steel. These improvements also affect the rolling contact fatigue, wear resistance and machinability properties positively. Most steel alloys can be transformed to bainite by applying right heat treatments. In this study, bainite transformation of low carbon steel 32CrB4 and high carbon steel R260 were investigated. Both steels were not studied before and this study may contribute to literature and develop a scientific background to industry about the bainite transformation, transformation variables and resultant mechanical properties.

CHAPTER 3

MATERIALS AND METHODS

3.1. Materials

Two different grades of steel are used in this study. First one is a high carbon steel which is used in railway rails, the grade R260. It was directly supplied from Turkish State Railways. Chemical composition is given in Table 3.

Table 3. Chemical composition of steel R260 in wt% according to EN 13674-1: 2006 [37] and to chemical analysis.

Steel	%C	%Si	%Mn	%P	%S	%Cr	%Al	%V
R260	0.62-0.80	0.15-0.58	0.70-1.20	≤0.025	0.008-0.025	≤0.15	≤0.004	≤0.03
Chemical analysis of R260	0.77	0.32	1.03	0.02	0.05	0.06	0.009	0.006

The second steel is a boron added medium carbon alloy steel, 32CrB4. Steel was supplied from Asil Çelik and received as a 30mm diameter round bar. The standard chemical composition is given in Table 4.

Table 4. Chemical composition of steel 32CrB4 in wt% according to EN 10263-4: 2003 [24] and to chemical analysis.

Steel	%C	%Si	%Mn	%P	%S	%Cr	%Cu	%B
32CrB4	0.30-0.34	≤0.30	0.60-0.90	≤0.025	≤0.025	0.90-1.20	0.25	0.0008-0.005
Chemical Analysis of 32CrB4	0.36	0.24	0.82	0.008	≤0.0003	1.16	0.09	0.003

3.1.1. Spectral Analysis

Spectral analyses were done in order to obtain composition of steels and compare with the information gathered from literature. Spectral analyses were performed in "Optical Emission Spectrometer WAS Foundrymaster" located in METU Foundry, Metal Processing and Automotive Materials Laboratory. Spectral analysis of steels R260 and 32CrB4 are shown in Table 3 and 4.

3.2. Heat Treatment Process

3.2.1. Furnaces and Salt Baths

Protherm muffle furnace is used for austenitization at elevated temperatures and for tempering. Protherm salt bath is used for quenching austenitized samples to selected temperatures and holding isothermally at desired temperature for bainitic transformations. The AS 135 Tempering Salt (PETROFER) is used in salt bath. It consists of alkaline nitrates and nitrites with a melting point of 135°C, and a working range of 160°C-550°C. Two salt bath furnaces were used: One of them is for smaller samples such as microstructural analysis samples, which has 0.75 liter melting pot.

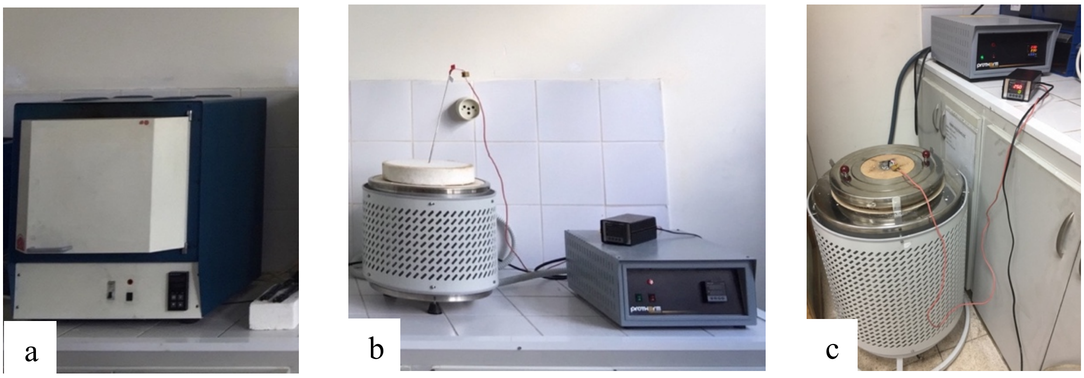


Figure 17. Furnaces used in this study; (a) muffle furnace, (b) small salt bath, (c) large salt bath.

In this study, 15x10x10 mm^3 sized samples were used at the small salt bath. The other one is for larger samples such as tensile test specimens, high capacity salt bath furnace having a capacity of 8.5 liter is used for minimizing the temperature fluctuations. In Figure 17, the muffle furnace and two salt baths with different capacity are shown.

3.2.2. Heat Treatment Parameters

TTT diagrams of steels were constructed by using JMatPro software (Figure 19 and 20). Also, a diagram given by Herian *et al.* [38] is taken from literature for R260 steel (Figure 18), C-shaped curve was extrapolated by dashed lines in this thesis.

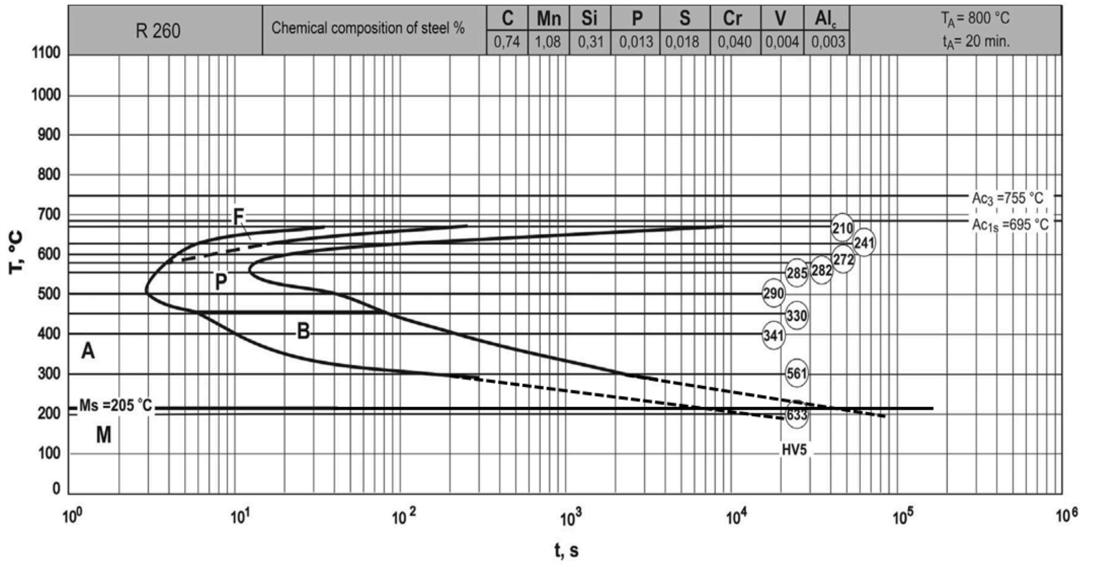


Figure 18. TTT diagram of R260 railway steel [38]

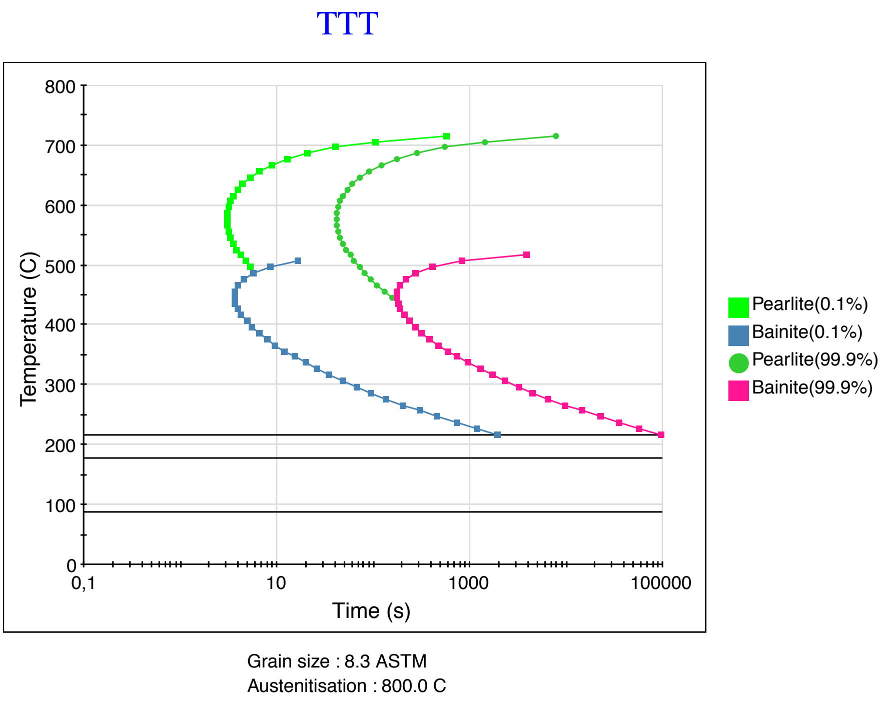


Figure 19. TTT diagram of R260 railway steel constructed by using JMatPro.

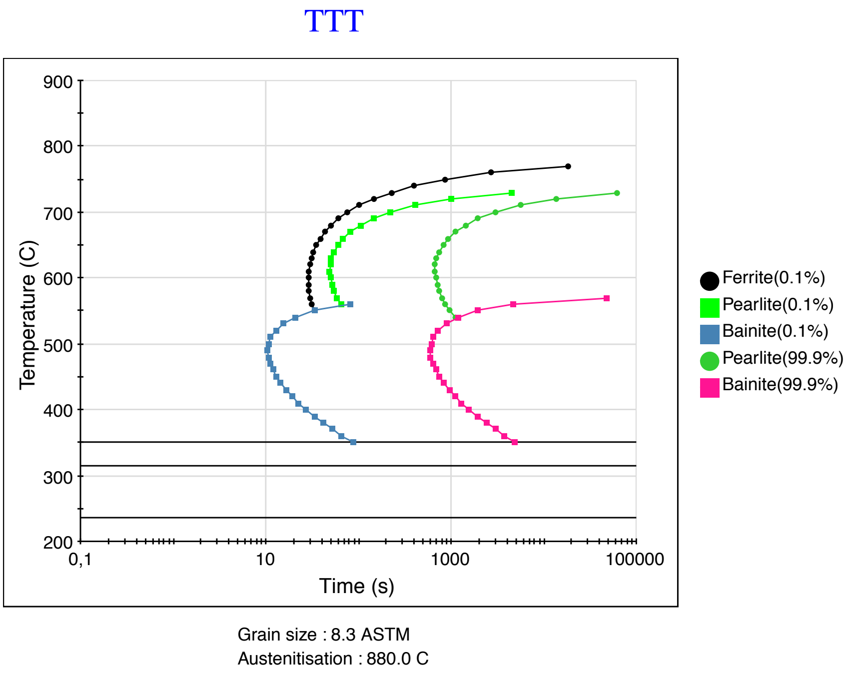


Figure 20. TTT diagram of 32CrB4 steel constructed by using JMatPro.

$1x1x2cm^3$ samples were cut from main steels by using Metkon Metacut 251 Abrasive Cutter. Muffle furnace was heated and fixed to austenitization temperature and salt bath furnace was heated and fixed to isothermal holding temperature. Austenitization and M_s temperatures were accepted as 850°C and 205°C for R260 steel; 880°C and 350°C for 32CrB4 steel, respectively. For bainite transformation, isothermal holding temperatures were set to just above M_s. Austenitization was done for 30 minutes and after that, samples were directly quenched to the salt bath and isothermally hold for a definite period of time according to the TTT diagram.

For two-step bainitic transformation, pre-heated and fixed second salt bath furnace was used. After first step, sample was quenched to lower temperature and held isothermally. In order to obtain martensitic structure, after austenitization, samples were quenched to water or oil at the room temperature.

Preliminary heat treatments were done for 32CrB4 steel and according to results, variables were chosen for 5 different heat treatment paths. After that, mechanical test specimens were heat treated by following the same experimental procedure.

3.3. Microstructural Characterization

Optical and electron microscopes were used for microstructural characterization. Sample preparation process is same for the both microscopes. First, in order to eliminate the decarburized region, 2 mm width steel was cut and removed from the surface (Figure 21), by using Metkon Metacut 251 Abrasive Cutter. After cutting, samples were mounted into bakelite for easy handling during sample preparation, mounting was done by using Metkon Ecopress 100. Grinding was done to achieve smooth surface by using Metkon Grip0 2V Grinder - Polisher and by using 120-200-400-600-800-1200 grit sand papers respectively. After grinding, samples were polished by Mecapol P230 Polisher, 6μ and 1μ of Metkon Diapat-P water-based polycrystalline diamond suspension were used. After polishing, 2% nital solution was used for etching.

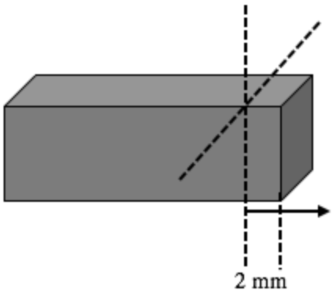


Figure 21. Sketch of a sample for microstructure examination, 2 mm part is cut from sample to remove decarburized region.

After sample preparation was completed, optical microscopy examinations were done with Huvitz Digital Microscope HDS-5800 and SEM examinations were conducted by Jeol 6400 Scanning Electron Microscope and FEI 430 Nano Scanning Electron Microscope.

3.4. Phase Area Fraction Measurement

Optic micrographs of a sample are used for phase area fraction measurement. Different phases have different colors that allows to create a color contrast and by counting pixels of each color, area fraction was measured digitally. Software Argenit Kameram was used for this measurement.

3.5. Hardness Testing

Hardness values of samples were measured by using Emco Universal Digital Hardness Testing Machine on Vickers scale. Tests were done under the load of 30 kgf.

3.6. Tensile Testing

Tensile test specimens were prepared according to ASTM E8/E8M – 16a standard. Specimens were round with 8mm diameter and 40 mm gage length (Figure 22). INSTRON 5582 Universal Testing Machine was used for tension tests. For each heat treatment, 2 specimens were tested.

Figure 22. Tension test specimen of 32CrBr steel

3.7. Notched Bar Charpy Impact Test

Samples were prepared according to ASTM E23 – 16b, with dimensions 10x10x55 mm^3 and a V shape notch (Figure 23). For each heat treatment 2 specimens were used. TINIUS-OLSEN pendulum type Charpy impact tester was used for tests. Fracture surfaces were analyzed.

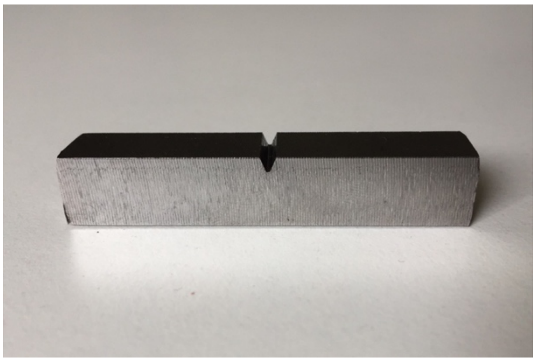


Figure 23. V-notched bar Charpy impact test specimen of 32CrB4 steel

CHAPTER 4

RESULTS

4.1. Bainitic Transformation in R260 Steel

4.1.1. Microstructural Characterization of R260 Steel and TTT Diagram Evaluation

While heat treatment paths were being decided, TTT diagrams were used as a guide. Figure 18 shows the one gathered from literature, C shaped curve is extrapolated in order to find start time of bainite transformation, just above M_s. Also, a software called JMatPro was used to construct a TTT diagram, which can be seen in Figure 19.

Austenitization temperature is given as 800°C at TTT diagrams. Also, martensite start temperature is given as 205 and 210°C at both TTT diagrams. When the bainite transformation time is examined in diagrams at just above M_s, it is seen that there is an uncertainty at the one that is gathered from literature. In order to have an idea, extrapolation is done. Bainite transformation starts nearly after 2 hours and is completed in 9 hours. On the other hand, according to the digital constructed TTT diagram, transformation starts after 40 minutes and completed in 28 hours. In order to find the duration, experiments at different periods were held.

In view of these examinations, the austenitization of R260 is carried out at 850°C for 30 minutes and the M_s temperature is accepted as 205°C in this study. Start and finish times of bainite transformation at just above M_s, are accepted as 2 hours and 28 hours respectively. Table 5 shows the summary of critical phase transformation points of R260 steel.

Table 5. Phase transformation data of R260 steel

Phase	Phase Transformation Status	Phase Transformation Temperature (°C)	Required Time for Phase Transformation
Martensite	M_s	205	-
	M_f	90	-
Bainite	0% Bainite	205	2 h
	100% Bainite	205	28 h

Schematic cooling paths for fully martensitic and fully bainitic transformation are shown on digital constructed TTT diagram of R260 in Figure 24.

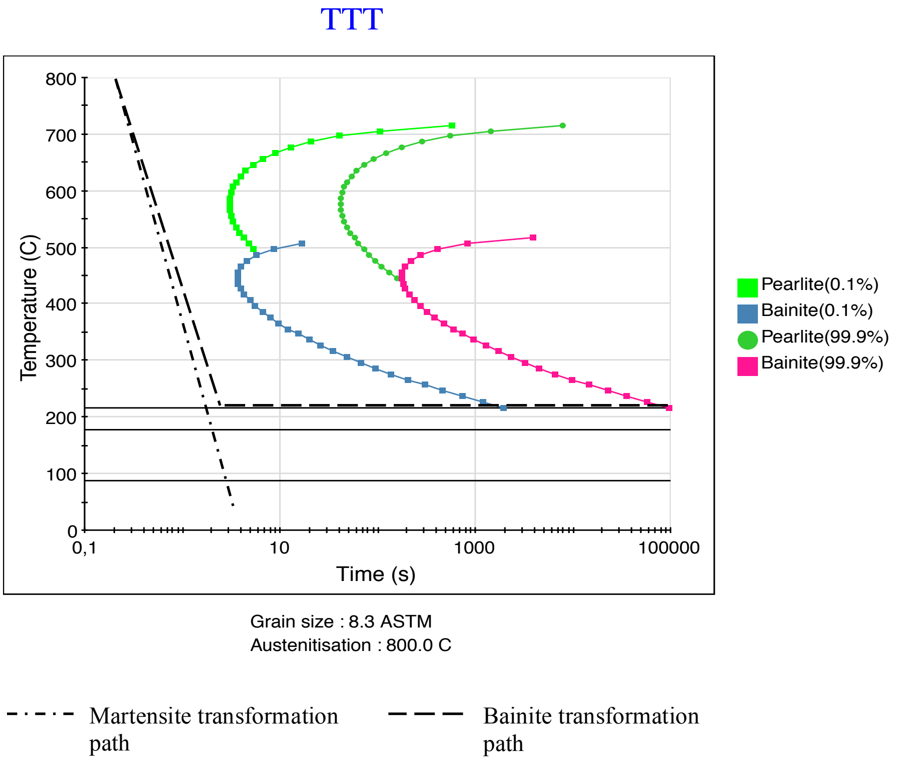


Figure 24. Schematic cooling paths on digital constructed TTT diagram of R260

After quenching from 850°C to water at room temperature, the microstructure of R260 consists of 100% martensite (Figure 25).

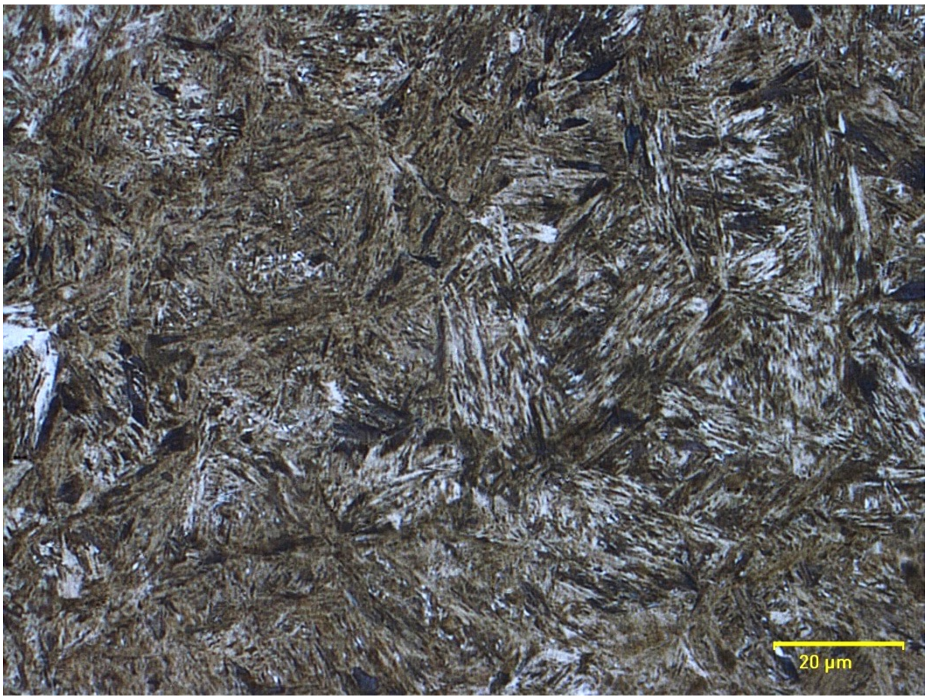


Figure 25. Optical micrograph of martensite phase in R260 steel.

It was mentioned that, just above M_s is the lowest possible temperature for fully bainitic transformation. Before that, transformation below M_s was experimented by austenitizing at 850°C for 30 minutes followed by quenching to 170°C and holding isothermally for 3 hours, in order to verify that 170°C is below M_s and also see that if there is any bainitic transformation takes place. According to the microstructural analysis of this experiment shown in Figure 26, bainite forms at the grain boundaries as dark colonies and the rest of the matrix transformed to martensite.

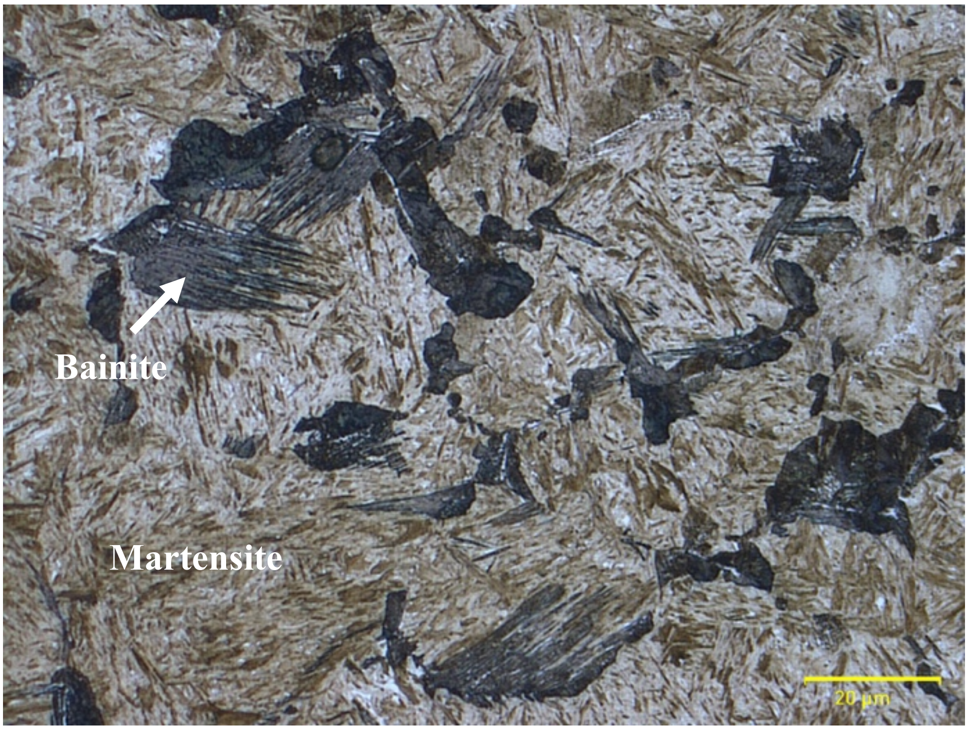


Figure 26. Optical micrograph of R260 steel, quenched to 170°C and hold isothermally for 3 hours.

After an austenitization treatment at 850°C for 30 minutes and quenching into the salt bath at 205°C for 3 hours, it is seen that 100% bainite could not be formed (Figure 27). It is seen that bainite phase is present at the grain boundaries in the form of dark colonies. The rest of the matrix is transformed to martensite. 205°C is M_s temperature of R260 steel and it is the lowest possible temperature for fully bainitic transformation in theory.

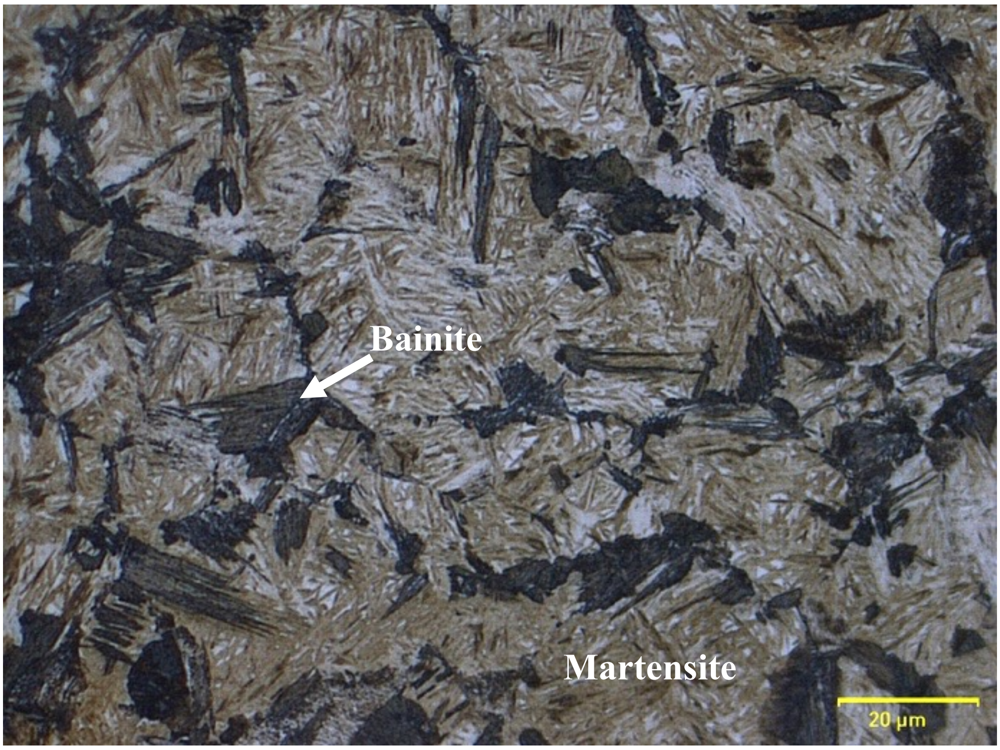


Figure 27. Optical micrograph of R260 steel quenched to 205°C and hold isothermally for 3 hours.

In order to examine the bainite transformation at longer holding times, the time in salt bath is increased from 3 hours (Figure 27) to 3 and 7 days (Figure 28). A considerable change in microstructure was not observed; bainite at grain boundaries and martensite phase coexist.

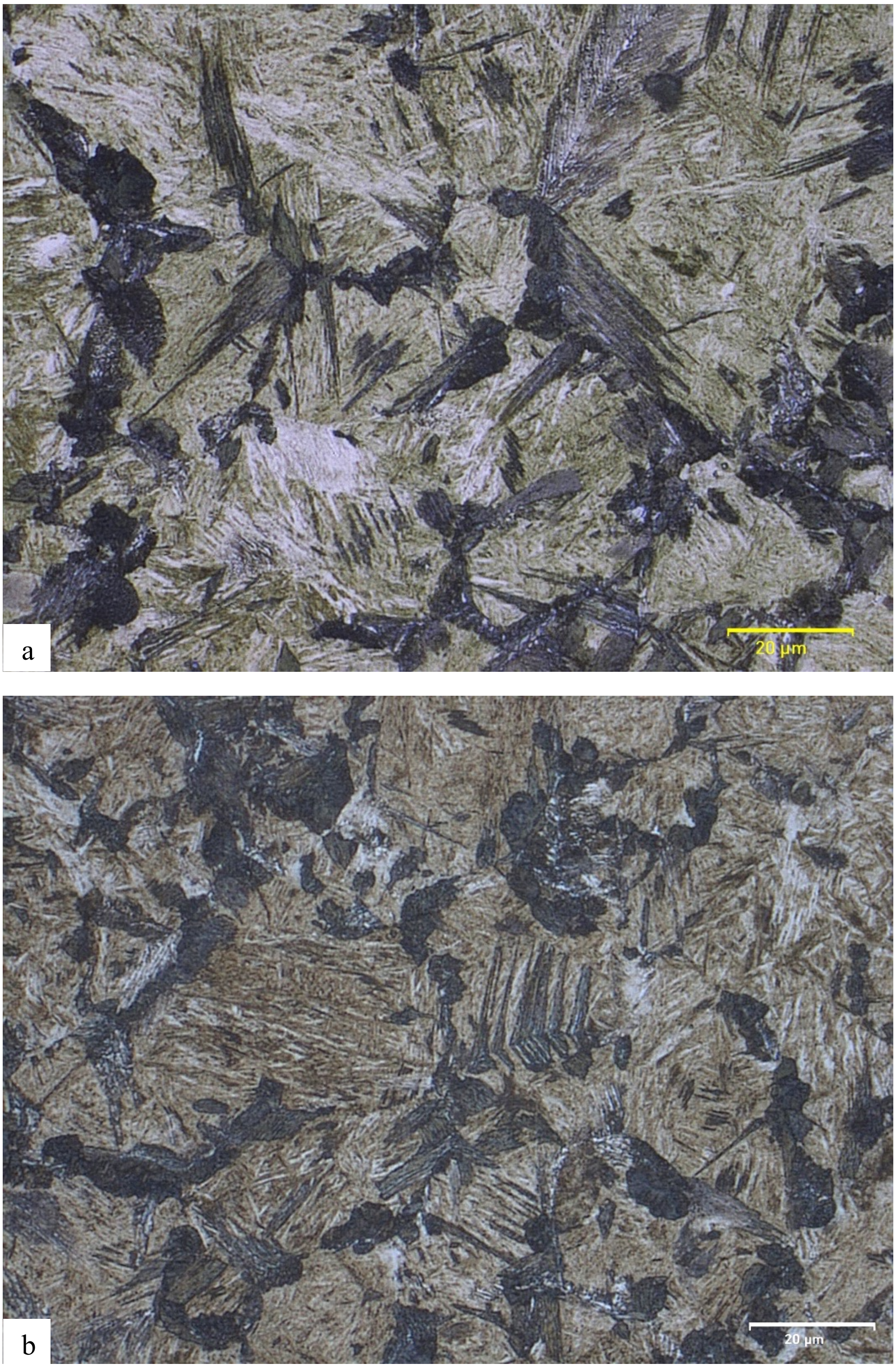


Figure 28. Optical micrographs of R260 steel; (a) quenched to 205°C and hold isothermally for 3 days, (b) quenched to 205°C and hold isothermally for 7 days.

For R260 steel, when the bainite transformation temperature is increased from 205°C to either 220°C, 250°C or 280°C the amount of bainite formed at grain boundaries starts to increase. Optical images of steels transformed at 220°C, 250°C and 280°C can be seen in Figure 29. The increase in the amount of bainite become very pronounced especially at 250°C and 280°C (Figure 29b and 29c). The TTT diagram of the R260 steel dictates the formation of 100% bainite after an isothermal treatment of 28 hours at 205°C. However, the bainitic transformation is not complete even after a 7-days of treatment at 205°C (Figure 28c) or after a 3-hours treatment at 250°C (Figure 29b). These results indicate that the bainitic transformation in R260 is very sluggish. Also, it can be dictated that the M_s temperature obtained from the calculated TTT diagrams (205°C) is substantially different than the real M_s temperature. Also, there are cases where bainitic transformation can stop after a definite time period and named as "Incomplete Transformation". In any case, due to the difficulties in obtaining 100% bainite, the examination of R260 steel is stopped and no further experiments were carried out.

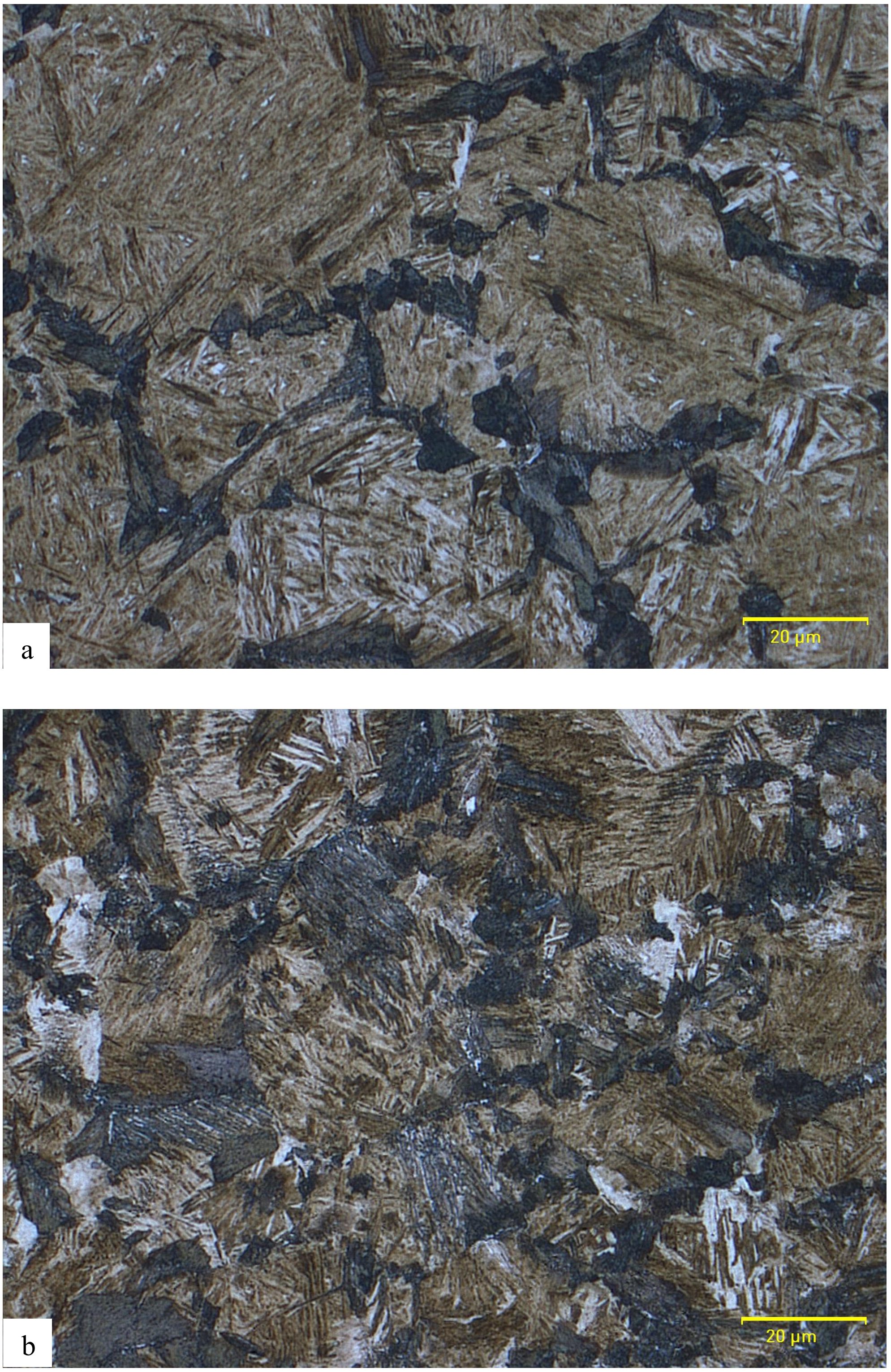


Figure 29. Optical micrographs of R260 steel; isothermally treated for 3 hours after quenched to (a) 220°C, (b) 250°C and (c) quenched to 280°C and transformed isothermally for 1 hour.

Figure 29. (Cont.)

4.1.2. SEM Studies of R260 Steel

SEM images enable to differentiate bainite and martensite very clearly. Bainite phase forms only at the grain boundaries with a limited size and the rest of the structure is martensite (Figure 30). A holding time as long as 7 days did not change the amount of bainite, indicating that 205°C is below the real M_s temperature.

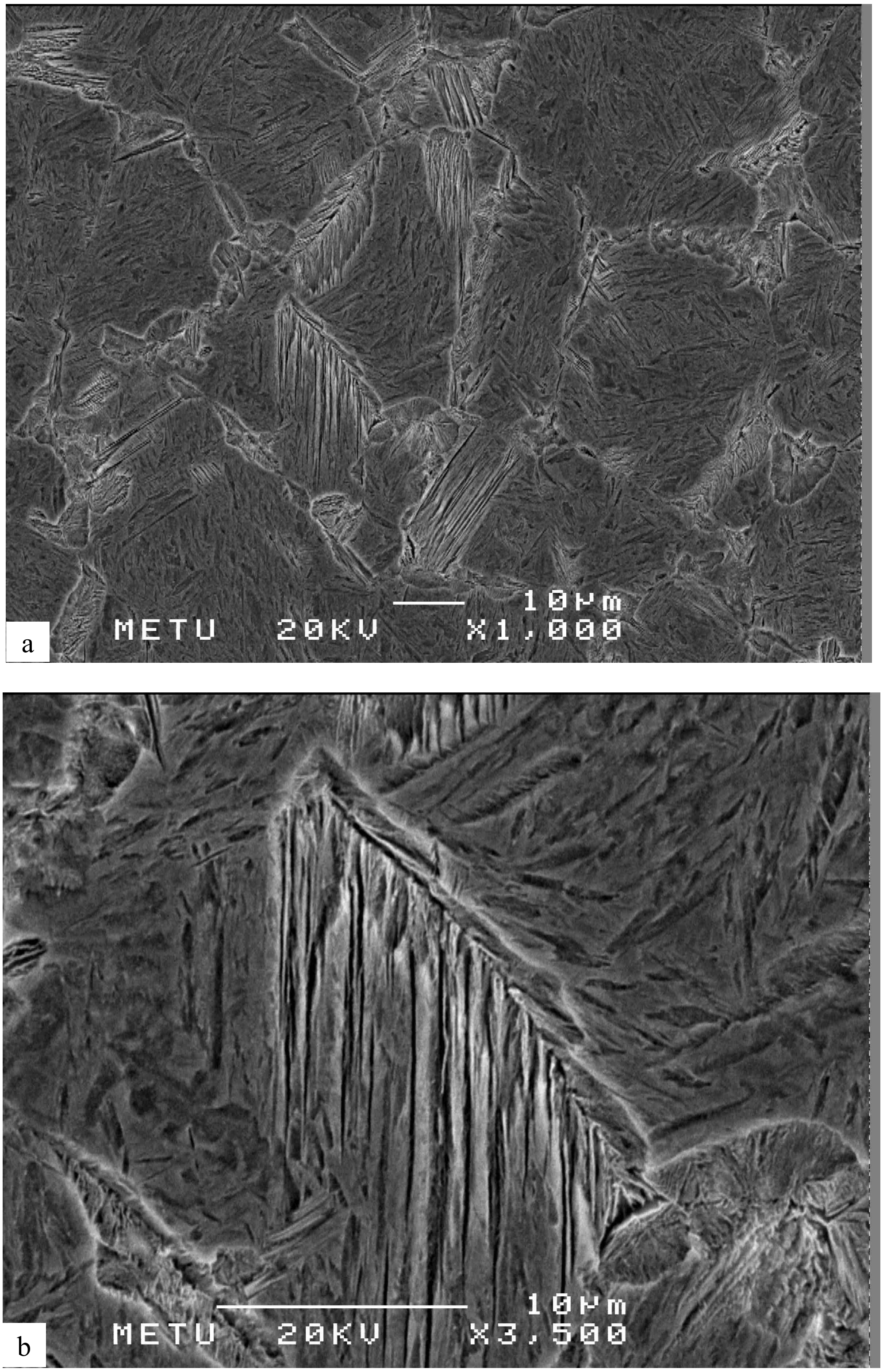


Figure 30. SEM images of R260 steel, (a) quenched to 170°C and hold 3 hours isothermally, bainite phase at the grain boundaries and martensite phase, (b) magnified bainite phase of image (a), (c) quenched to 210°C and hold for 3 days isothermally.

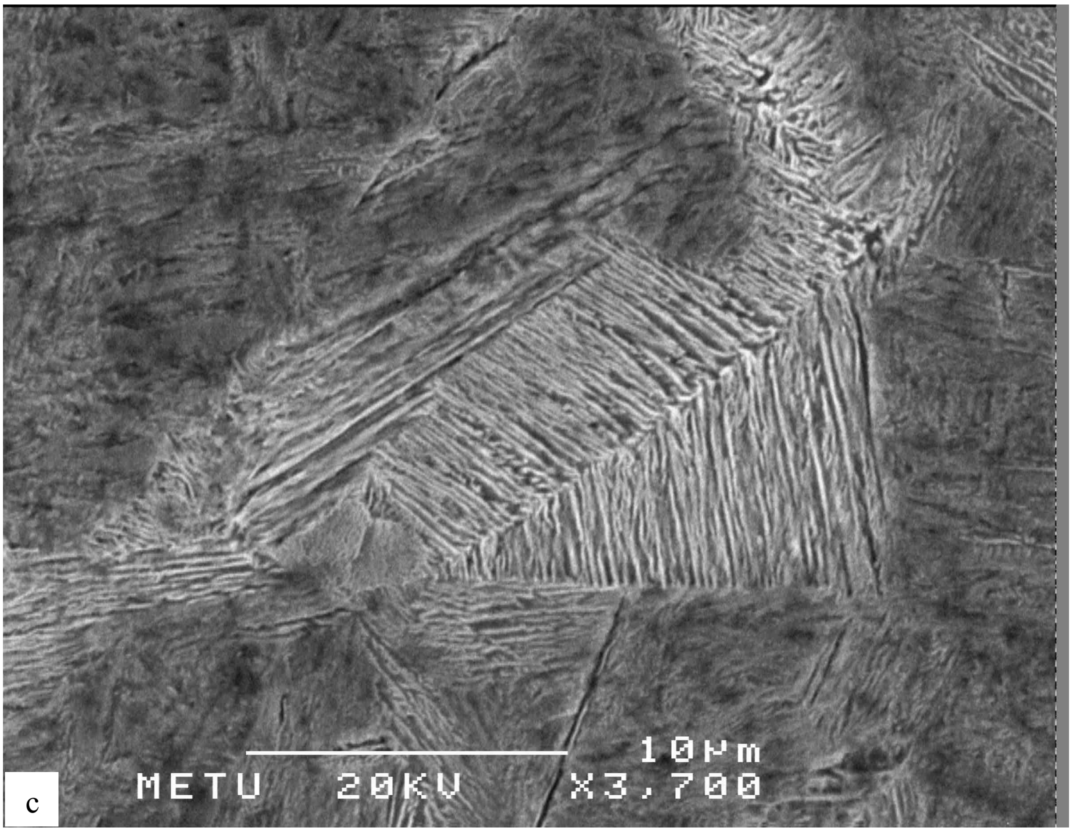


Figure 30. (Cont.)

Table 6 gives the summary of isothermal holding temperatures and times in salt bath for R260 steel samples. After isothermal holding was completed, samples were cooled in air.

Table 6. Isothermal holding times and temperatures for 5 samples of R260 steel

Quenching Temperature (°C)	170°C	205°C	220°C	255°C	280°C
Holding time	3 hours	3 hours 3 days 7 days	3 hours	3 hours	1 hour

4.1.3. Hardness Test Results of R260 Steel

Hardness test results of heat treated railway steel R260 specimens are given in Table 7.

Table 7. Hardness test results of R260 steel

Quenching Medium	Isothermal Holding		Hardness HV30
	Temperature (°C)	Time	
Water	-	-	663 ± 10
Oil	-	-	372 ± 12
Salt Bath	170	3 hours	568 ± 10
Salt Bath	205	3 hours	544 ± 4
Salt Bath	205	3 days	540 ± 5
Salt Bath	205	7 days	576 ± 4
Salt Bath	220	3 hours	542 ± 6
Salt Bath	250	3 hours	482 ± 4
Salt Bath	280	1 hour	354 ± 9

As seen in Table 7, the hardness seems to be around 540 HV even after 3 days at 205°C. On the other hand, a treatment of 7 days at 205°C yields 570 HV which is not very consistent and it seems that the exact determination of M_s in R260 is difficult. The reason for inconsistent results can be due to "Incomplete Transformation" phenomena for bainitic transformation, which can be observed for several alloys.

4.2. Bainitic Transformation in 32CrB4 Steel

4.2.1. Microstructural Characterization of 32CrB4 Steel and TTT Diagram Evaluation

32CrB4 steel was used in the study of Kawulok *et al.*; they used a CCT diagram constructed by a software QTSteel and a CCT diagram constructed according to dilatation curves as Figure 31 shows. When M_s temperature is compared, it can be seen that Kawulok *et al.* found as 355°C according to their two CCT diagrams [26] . In this study, the calculated TTT diagram gives the M_s temperature as 350°C. So, the M_s temperature is taken as 350°C in this study. Austenitization temperature was stated as 850°C in the study of Kawulok *et al.*, in this study austenitization was held at 880°C in order to ensure that austenitization takes place.

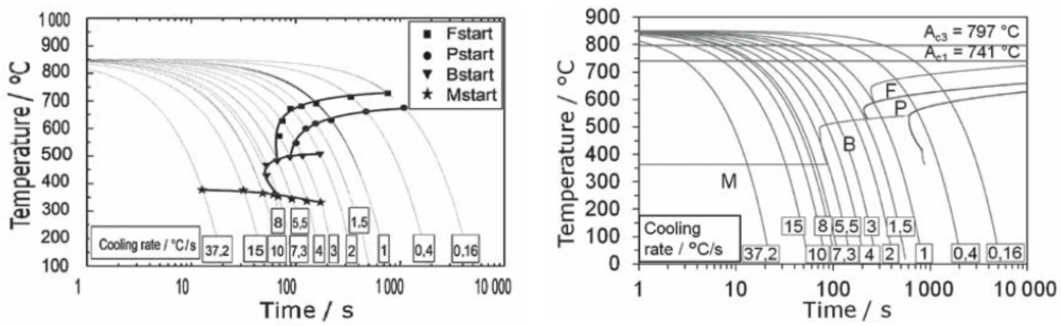


Figure 31. CCT diagram of 32CrB4, (a) drawn by experimental data and (b) drawn by software [26].

Bainite transformation at 350°C starts in 1 minute and completes in 85 minutes as calculated TTT diagram dictates. At 300°C, bainite transformation starts in 2 minutes and completes in 5.5 hours. Since 300°C is below M_s, in order to find the transformation temperatures, the C shaped curve is extrapolated below M_s (Figure 32). In the light of these information, summary of the critical transformation points is given in Table 8.

Table 8. Phase transformation data of 32CrB4 steel

Phase	Phase Transformation Status	Phase Transformation Temperature (°C)	Required Time for Phase Transformation
Martensite	M_s	350	-
	M_f	240	-
Bainite	0% Bainite	350	1 min
	100% Bainite	350	85 min
Bainite	0% Bainite	300	2 min
	100% Bainite	300	5.5 h

Cooling paths for fully martensite, fully bainite and two-step bainite transformations are shown schematically in Figure 32.

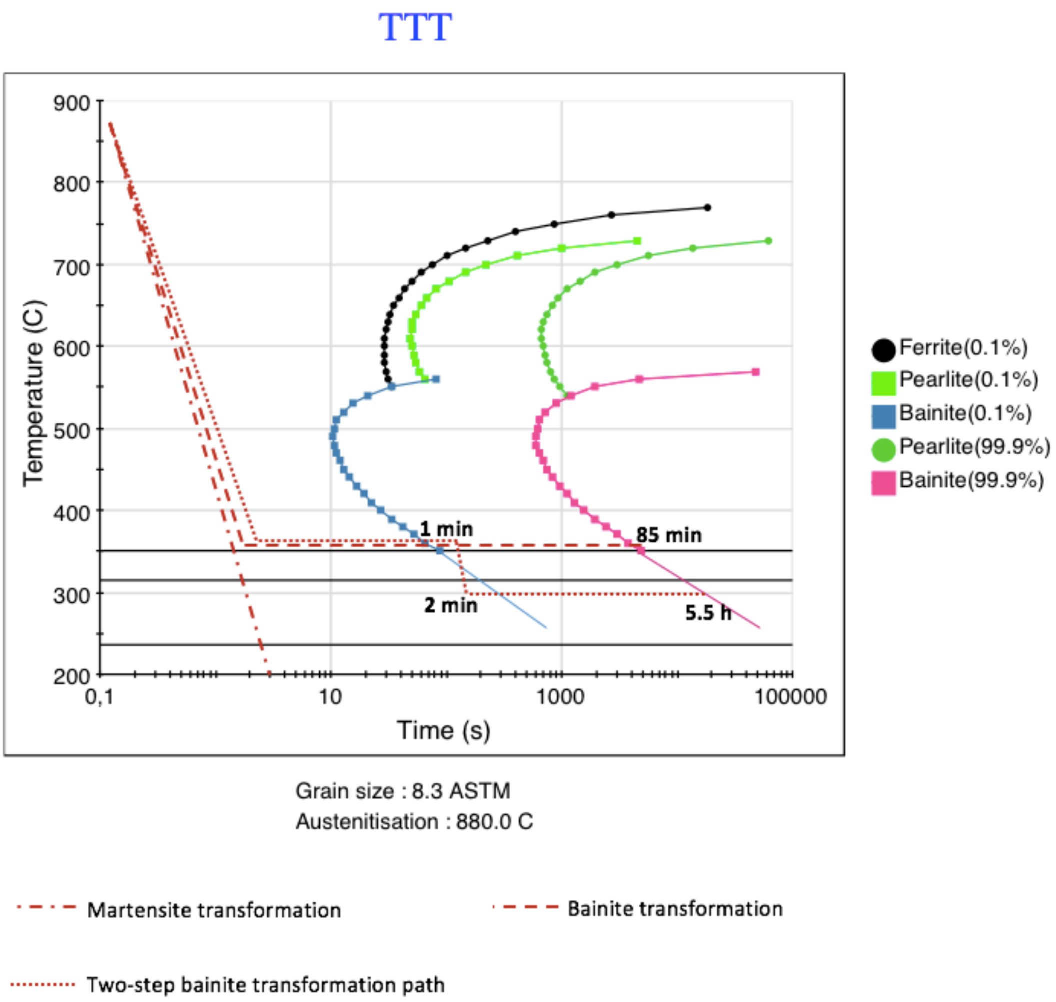


Figure 32. Schematic sketch of cooling paths of martensite, bainite and two-step bainite transformations for 32CrB4 steel

Preliminary heat treatments were performed based on the variables given in Table 8 and they are shown in Table 9. Microstructural examinations were done for these preliminary heat treatments and main heat treatments were determined according to these analyses. All tempering operations were made for 2 hours.

Table 9. Preliminary heat treatments for steel 32CrB4

1st Quenching			2nd Quenching			Tempering
Medium	**Temp. (°C)**	**Isothermal Holding Time**	**Medium**	**Temp. (°C)**	**Isothermal Holding Time**	**Temp. (°C)**
Water	RT	-	-	-	-	-
Oil	RT	-	-	-	-	-
Oil	RT	-	-	-	-	400
Oil	RT	-	-	-	-	450
Oil	RT	-	-	-	-	500
Salt bath	350	1 hour	-	-	-	-
Salt bath	350	2 hours	-	-	-	-
Salt bath	350	2 hours	Water	RT	-	-
Salt bath	350	3 days	-	-	-	-
Salt bath	350	5 min	Salt bath	320	1h30min	-
Salt bath	350	2 min	Salt bath	300	5h30min	-
Salt bath	350	2 min	Salt bath	300	5h30min	450
Salt bath	350	2 min	Water	RT	-	-
Salt bath	350	2 min	Water	RT	-	450
Salt bath	350	1 min	Water	RT	-	-
Salt bath	300	15 min	Water	RT	-	-

Microstructural analysis of 32CrB4 steel shows that, with applied heat treatment procedures, either 100% martensite, 100% bainite or a mixture of bainite-martensite phase can be obtained.

First water quenching and oil quenching were done at room temperature and 100% martensite phase was obtained, as shown in Figure 33.

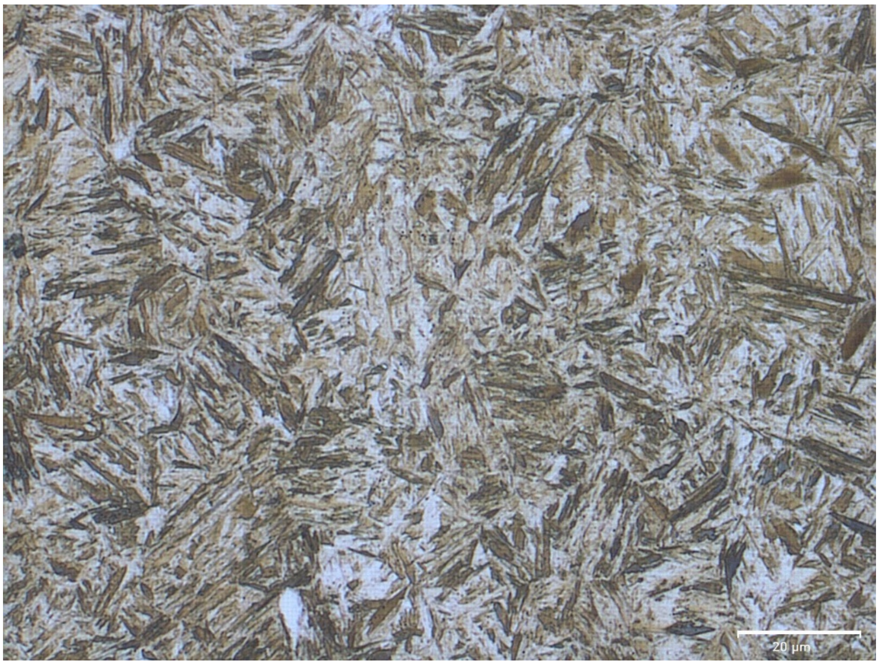


Figure 33. Optical image of fully martensite phase of 32CrB4 steel, obtained by water quenching

Martensite phase obtained by oil quenching was tempered at 450°C for 2 hours and Figure 34 shows the optical micrograph of the tempered martensite phase.

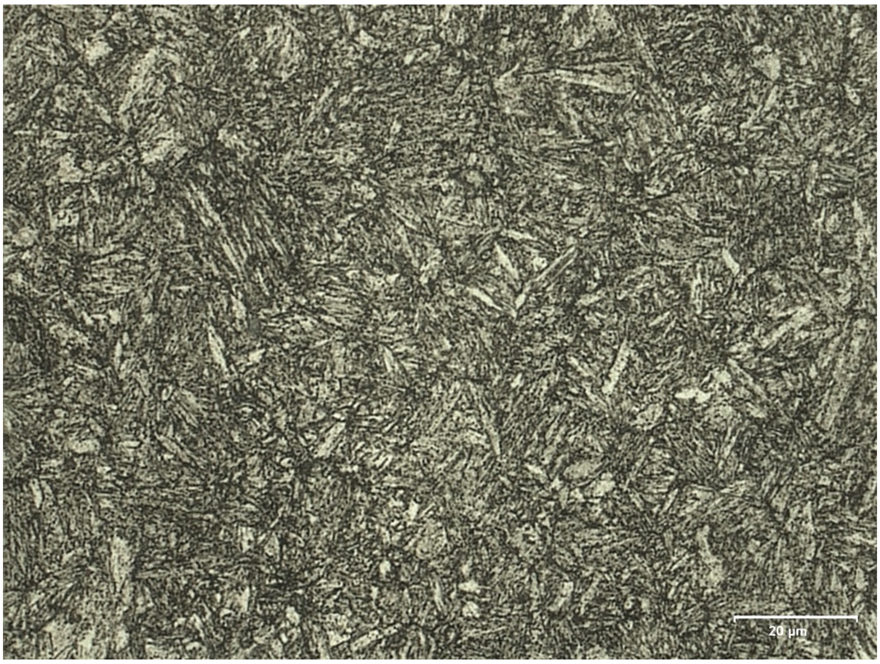


Figure 34. Optical image of tempered martensite of 32CrB4 steel.

SEM images of tempered martensite phase can be seen in Figure 35. It is clear that there is no parallel sheave formation which is typical for bainite phase mixture (Figure 35b).

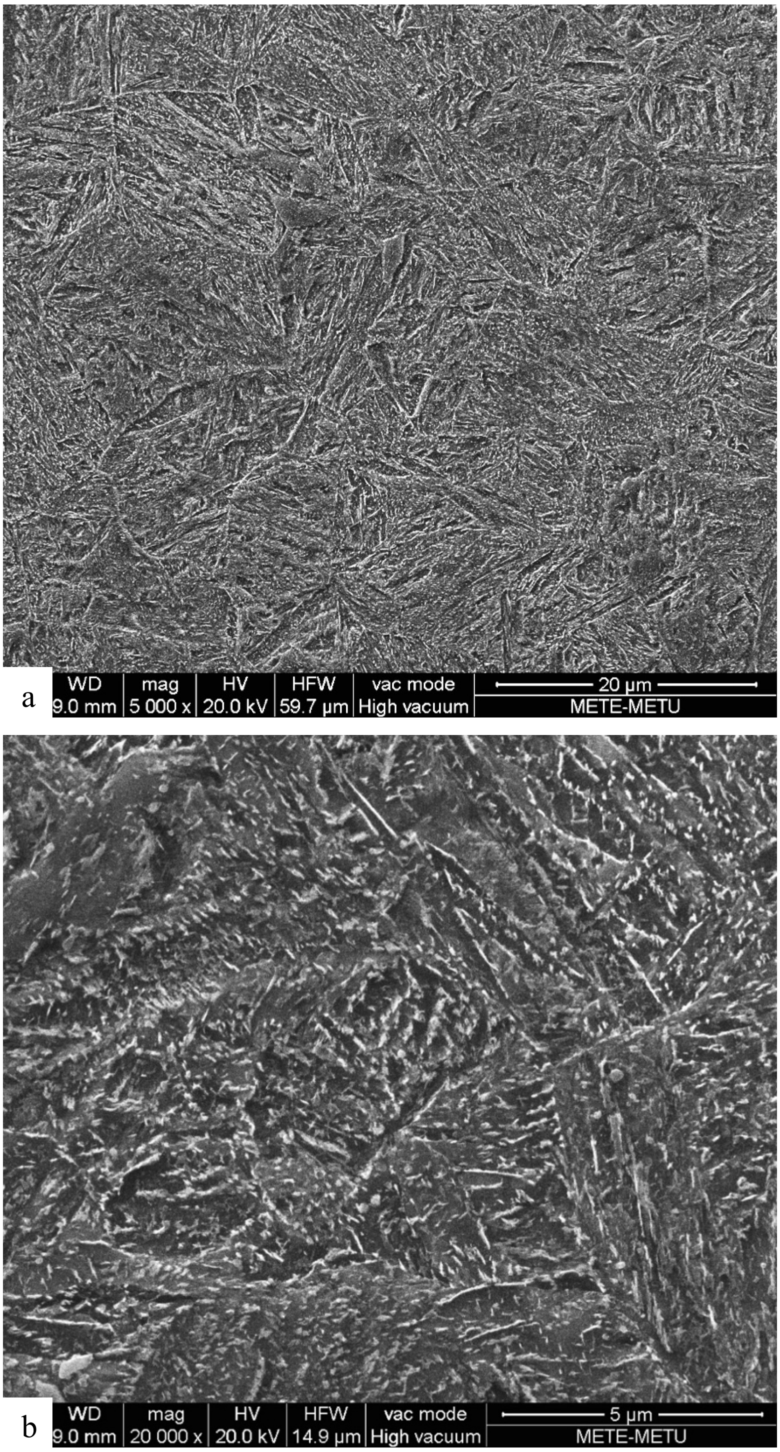


Figure 35. SEM images of 32CrB4 steel, oil quenched and tempered at 450°C for 2 hours. (b) At high magnification, the carbide precipitation can be seen.

It is known that martensite phase appears as light yellow and bainite appears as dark gray after etching [3]. A heat treatment, which consists of quenching around M_s and holding isothermally for 1 minute and then quenching to room temperature enables to differentiate bainite from martensite phase in 32CrB4 (Figure 36).

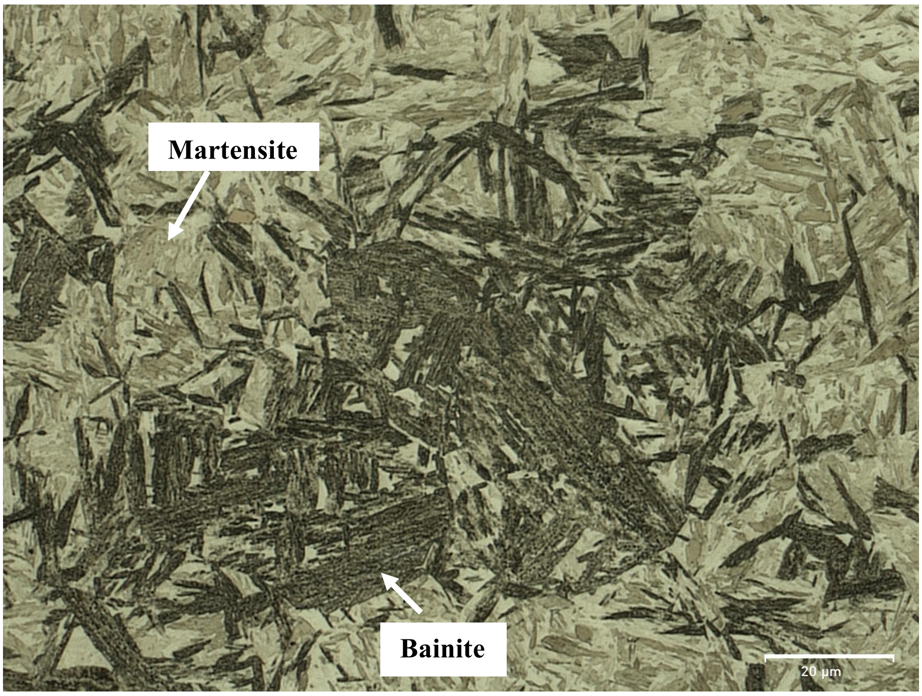


Figure 36. Optical micrograph of 32CrB4 steel, quenched to 350°C and hold for 1 minute and then water quenched. Light contrasted acicular phase is martensite and the dark, feathery constituent is bainite.

It is clear that light appearing phase in Figure 36 is martensite. It should be noted that, for this heat treatment, martensite needles can be observed very easily due to the short holding time and water quenching. The other heat treatment processes, on the other hand, have longer holding time periods. Longer holding time changes the martensite microstructure due to tempering. Although it is not a tempering process, long holding periods can have a tempering effect on martensite and give darker color than expected. This yields a morphology that is very similar to bainite microstructure and it becomes impossible to separate martensite from bainite even under SEM.

The other possibility considered for the second phase is retained austenite, however retained austenite is observed in steels that have a higher carbon content than 0.6%C which is higher than 32CrB4 steel. Also, martensite finish temperature is 240°C which is substantially above room temperature.

According to the calculated TTT diagram of the 32CrB4 steel, quenching to 350°C and holding isothermally for 85 minutes, 2 hours and 3 days should give 100% bainite phase, as bainitic transformation finishes in 85 minutes. Figure 37 belongs to the specimen hold at 350°C for 1hr, 2hrs and 3 days which are 100% bainitic. Though not certain, several light-appearing areas are seen under optical microscope, which can be martensite islands (Figure 37). One possibility for these martensite islands can be the due to that the real M_s temperature for this steel is somewhat higher than 350°C and some amount of martensite is formed at the quenching stage to 350°C. Another possibility can be due to an incomplete bainite reaction.

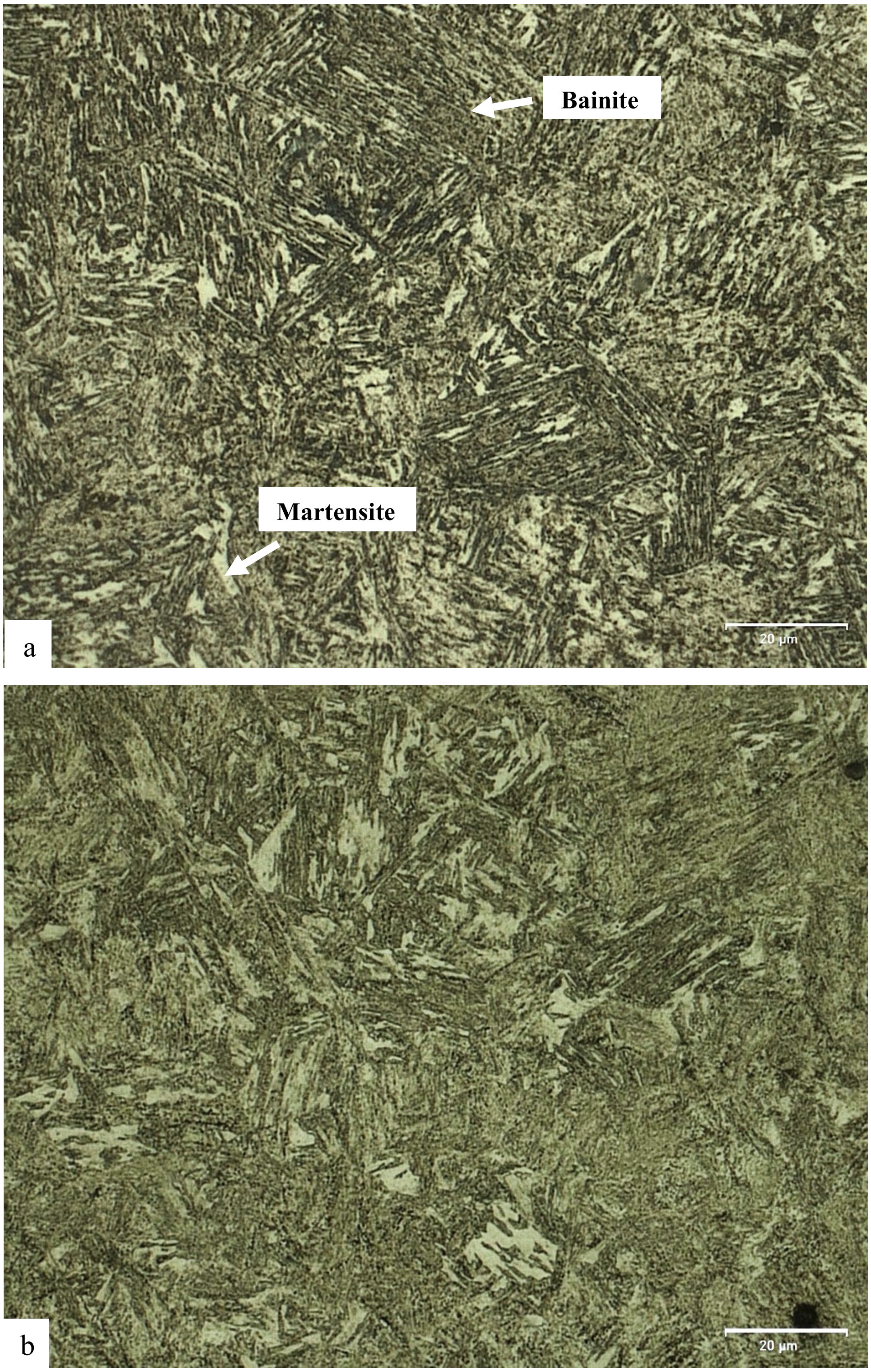


Figure 37. Optical micrographs of fully bainitic phase mixture in 32CrB4 steel quenched to 350°C and hold isothermally (a) 1 hour, (b) 2 hours, (c) 3 days.

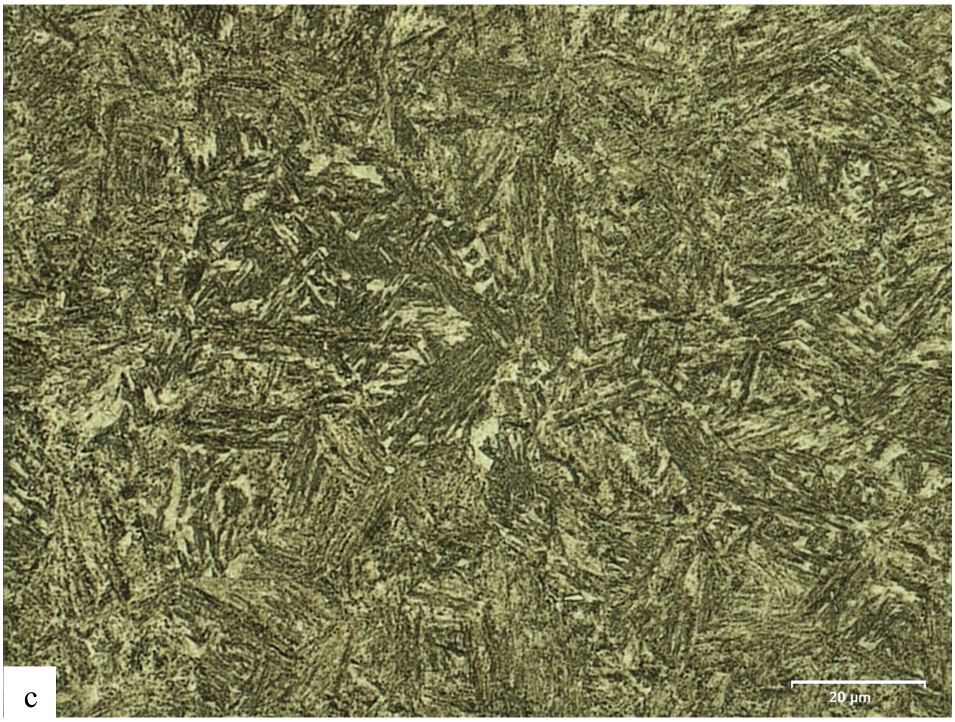


Figure 37. (Cont.)

SEM analyses of 2 hours isothermal holding at 350°C also shows that microstructure consists of nearly fully bainite phase as shown in Figure 38 and 39. Figure 38a shows that, all of the austenite phase was transformed to bainite since the structure is homogeneous. In Figure 38b and 38c, grains boundaries and bainite structure inside grains are seen very clearly. Though nearly 100% bainitic, isolated small islands of martensite can also be seen after 2 hours at 350°C in Figure 38b and 38c.

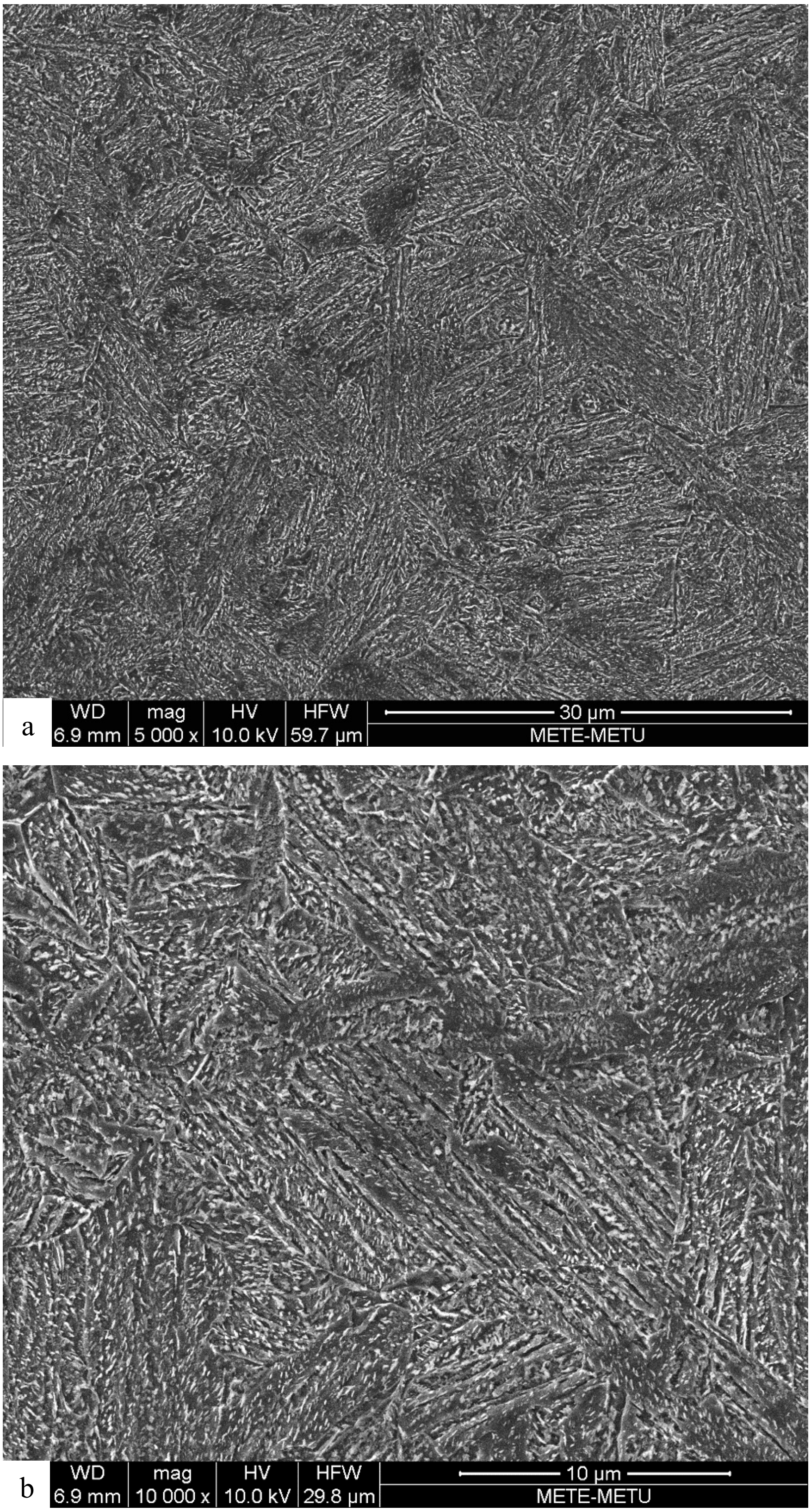


Figure 38. SEM images of 32CrB4 steel quenched to 350°C and hold isothermally for 2 hours. The microstructure is nearly fully bainitic.

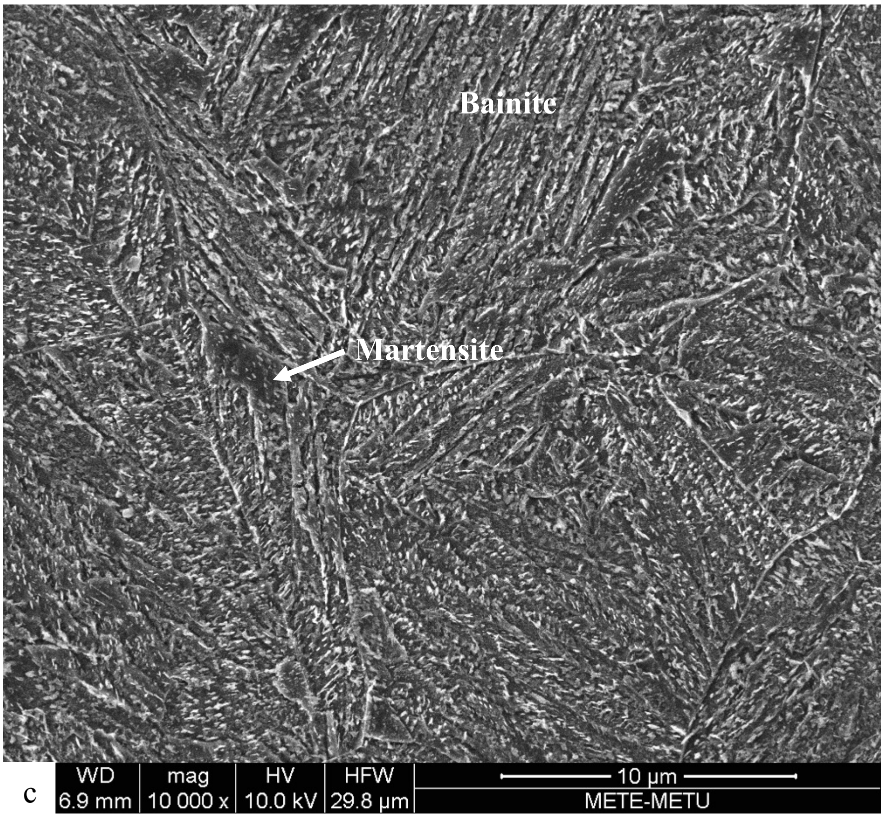


Figure 38. (Cont.)

In Figure 39, bainite microstructure appears in the form of acicular and parallel oriented sheaves. At higher magnifications carbide precipitation can be seen within bainite sheaves, in Figure 39c.

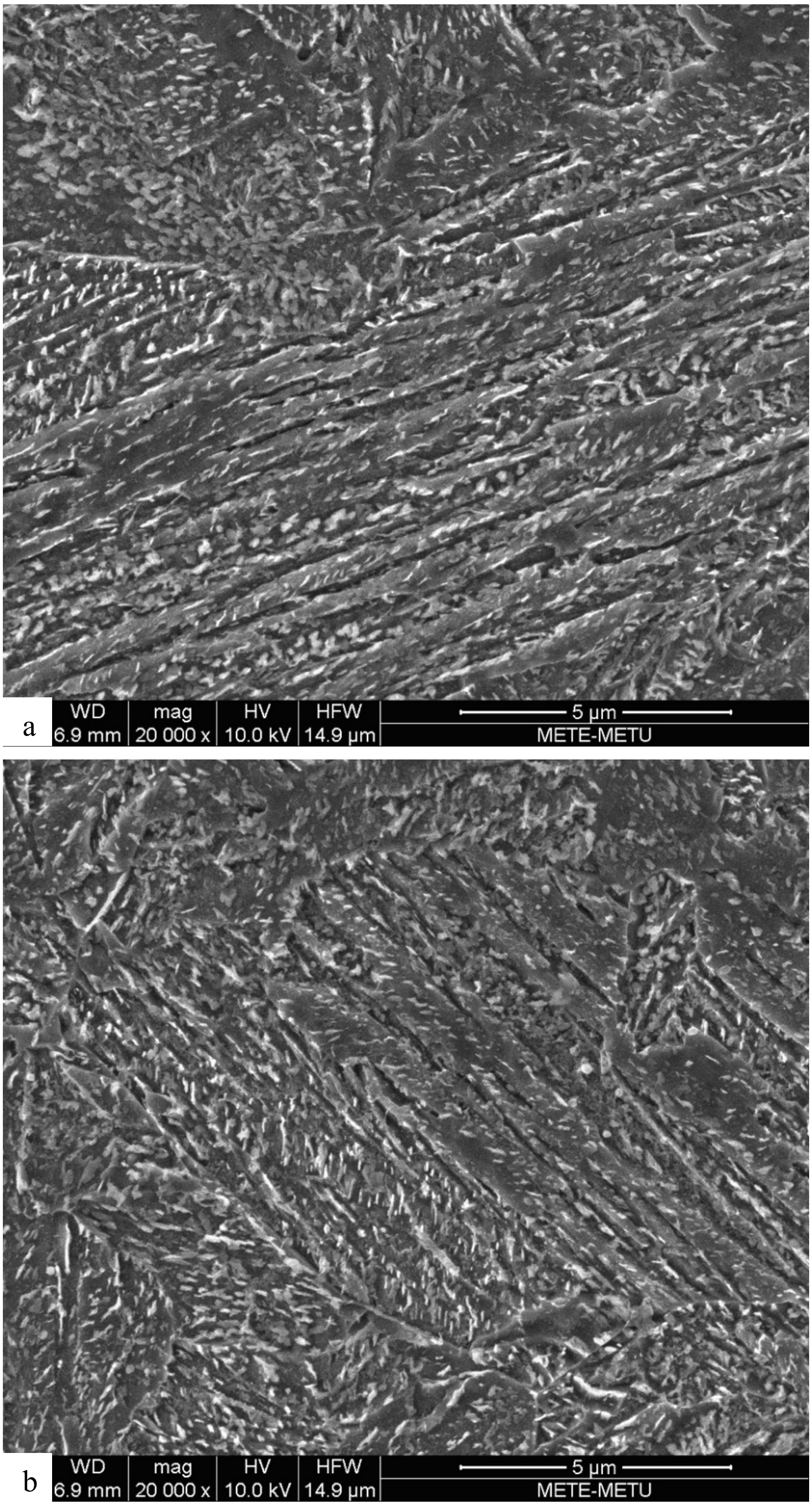


Figure 39. SEM images of 32CrB4 steel quenched to 350°C and hold isothermally for 2 hours. (a) and (b) The microstructure is nearly fully bainitic. (c) The small white precipitates are fine carbides (within bainite sheaves).

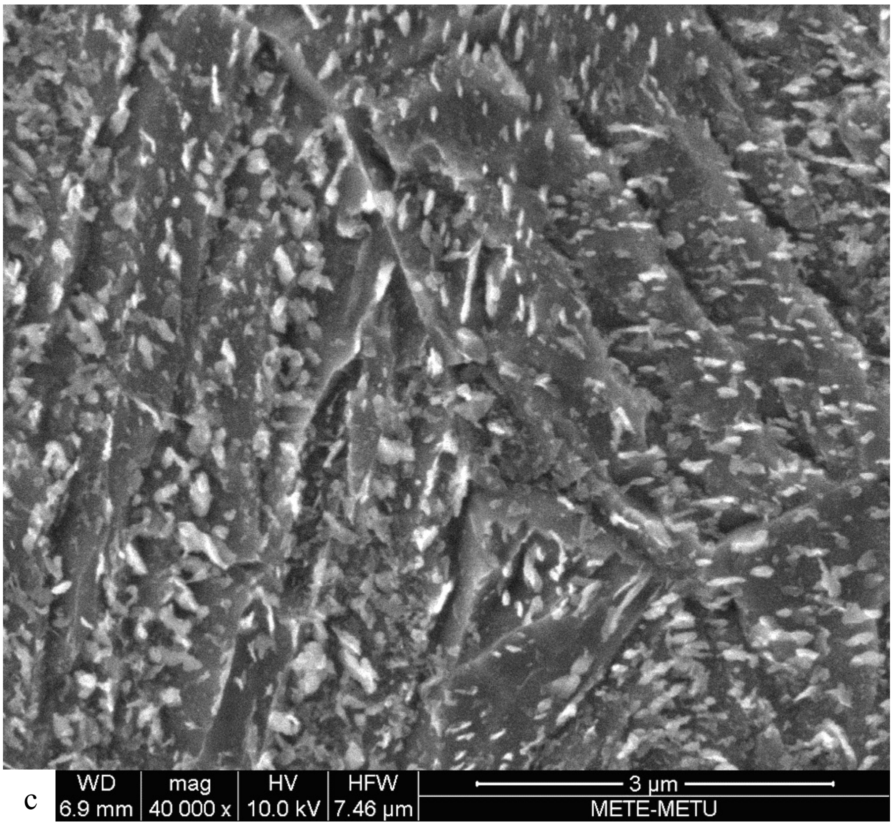


Figure 39. (Cont.)

For two-step bainite transformations, the first step of quenching and isothermal holding temperature was 350°C for all samples since M_s is 350°C. The second step of quenching and isothermal holding temperatures were 320°C and 300°C. 90 minutes for 320°C and 330 minutes for 300°C isothermal holdings were applied.

At first step, 2 minutes at 350°C produces nearly 85% bainite and the rest stays as austenite with a higher carbon content. In order to find the transformed bainite percentage at the end of first step, sample was water quenched and untransformed austenite is transformed to martensite. According to results of area fraction measurement of phases for this heat treatment, nearly 85% of austenite transforms to bainite in 2 minutes at 350°C (Figure 41). SEM images of bainite and martensite phase mixture obtained by quenching to 350°C and hold isothermally for 2 minutes and then quenched to water at room temperature can be seen in Figure 40. The martensite islands can be differentiated easily from bainite phase: Bainite sheaves have fine carbide precipitation.

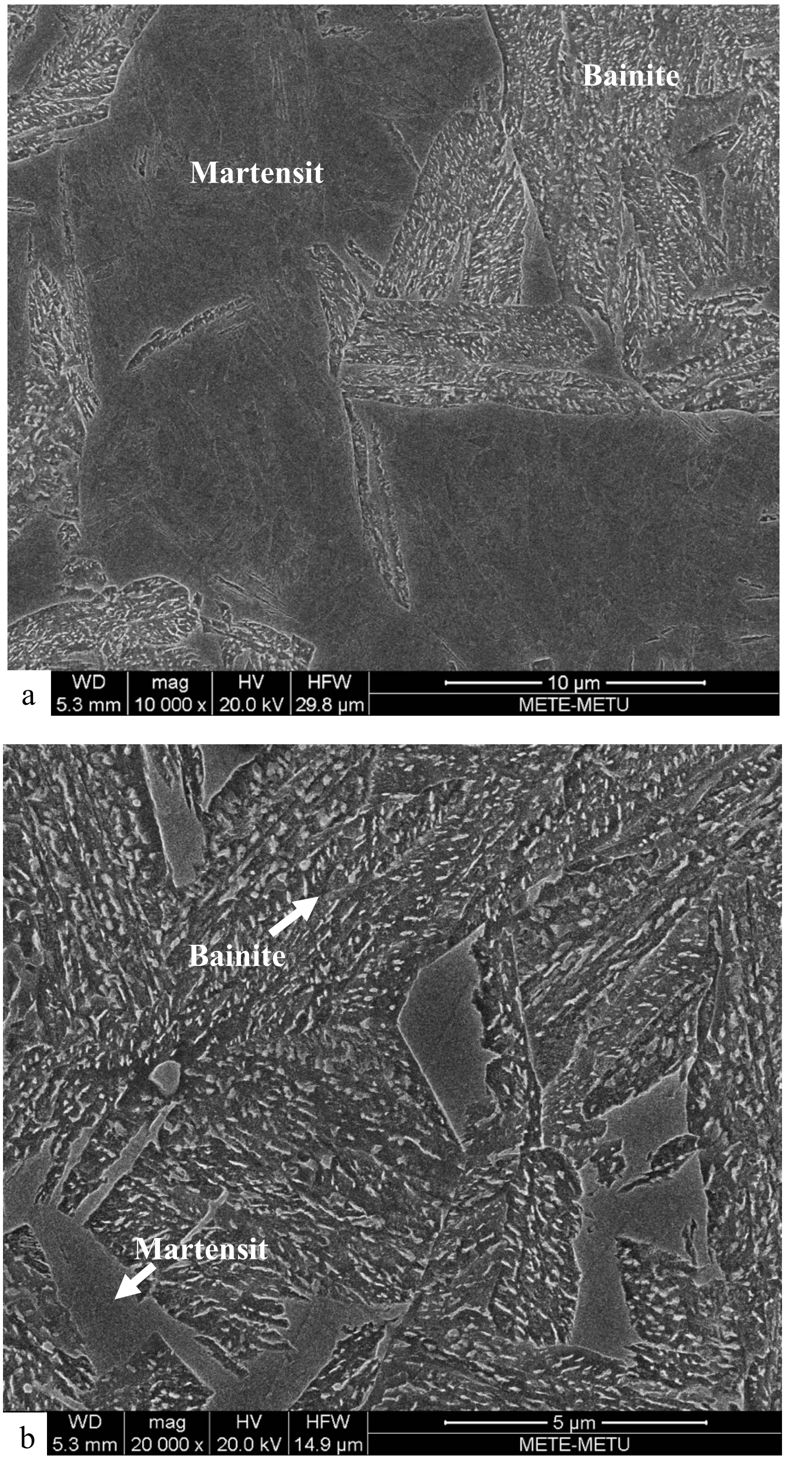


Figure 40. Bainite and martensite phase mixture of 32CrB4 steel obtained by quenching to 350°C and hold isothermally for 2 minutes and then quenched to water at room temperature. (c) Image from tensile test specimen.

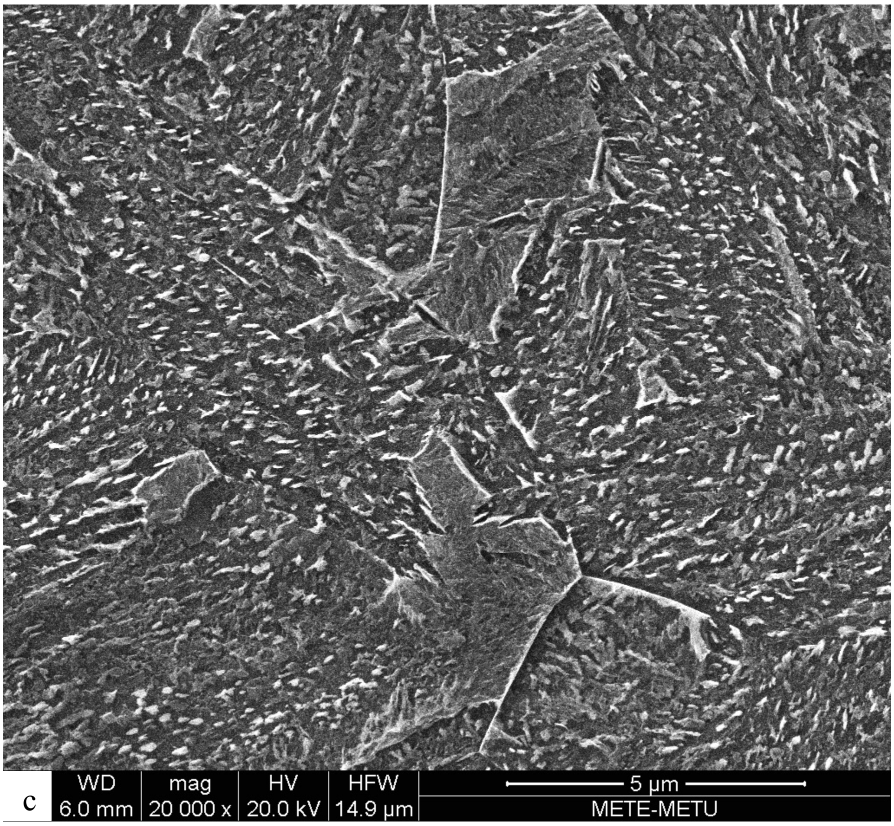


Figure 40. (cont.)

Area fraction analysis in Figure 41 shows that approximately 85% of austenite transforms to bainite in 2 minutes at 350°C. The remaining austenite (15%) transforms to martensite due to water quenching.

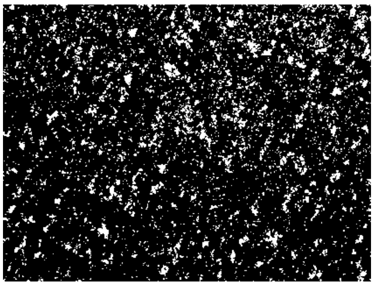
Optical Image	**Digitally Colored Image**	**% Dark Area (%Bainite Phase)**
		85% ± 2%

Figure 41. Area fraction of sample obtained by 2 minutes at 350°C followed by water quenching.

Two-step bainite transformation produces bainite in two different bainite sheave width: Coarser ones will be formed at 350°C and finer ones will be formed at 300°C. The morphology of 300°C bainite and 350°C bainite could not be differentiated. SEM images of two-step bainite transformation at 350°C for 2 minutes followed by 300°C for 5.5 hours can be seen in Figure 42.

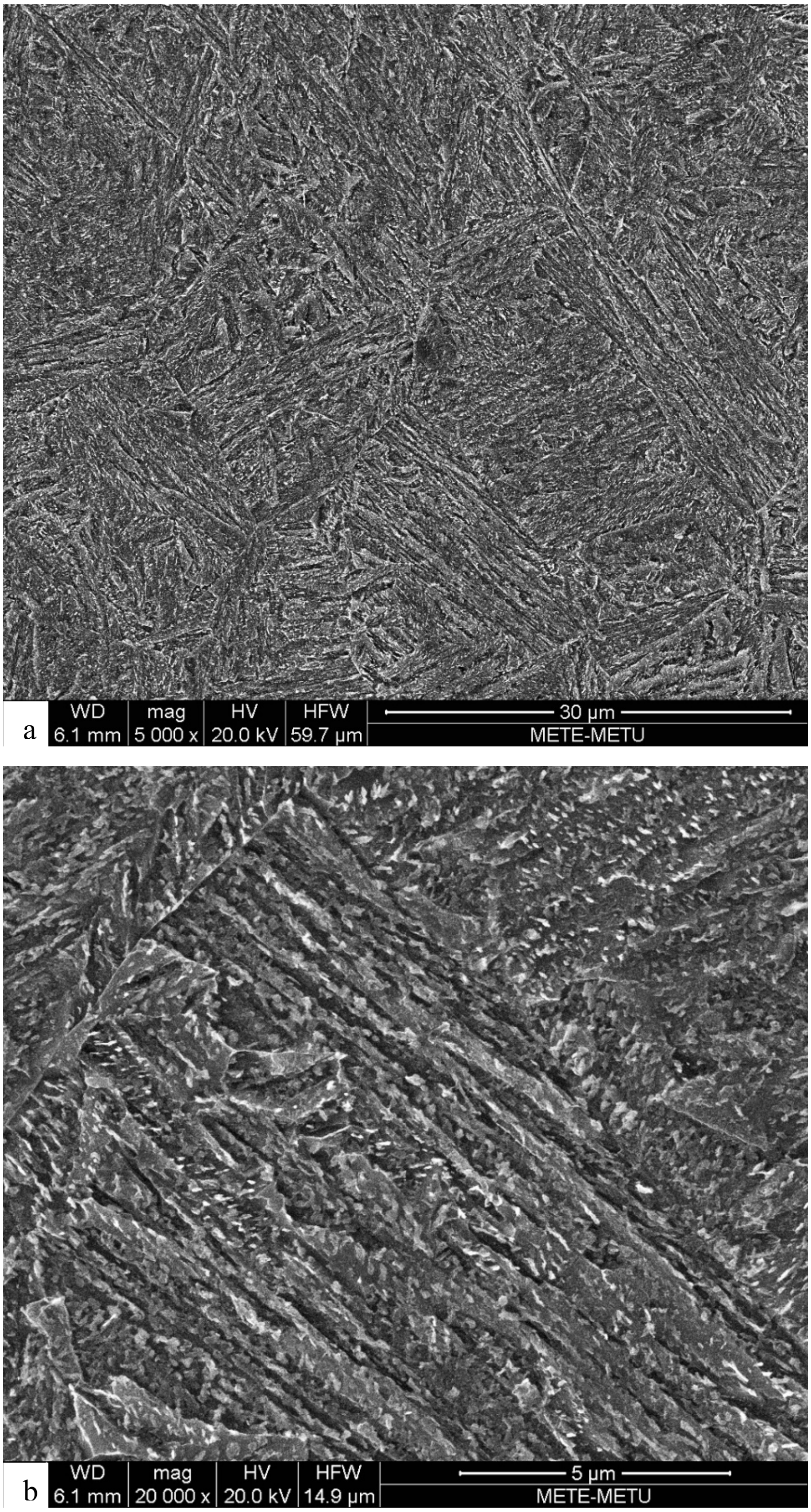


Figure 42. SEM images of two-step quenched specimen (350°C for 2 minutes and 300°C for 5.5 hours). The bainite phase can be seen easily. However, the martensite phase could not be differentiated.

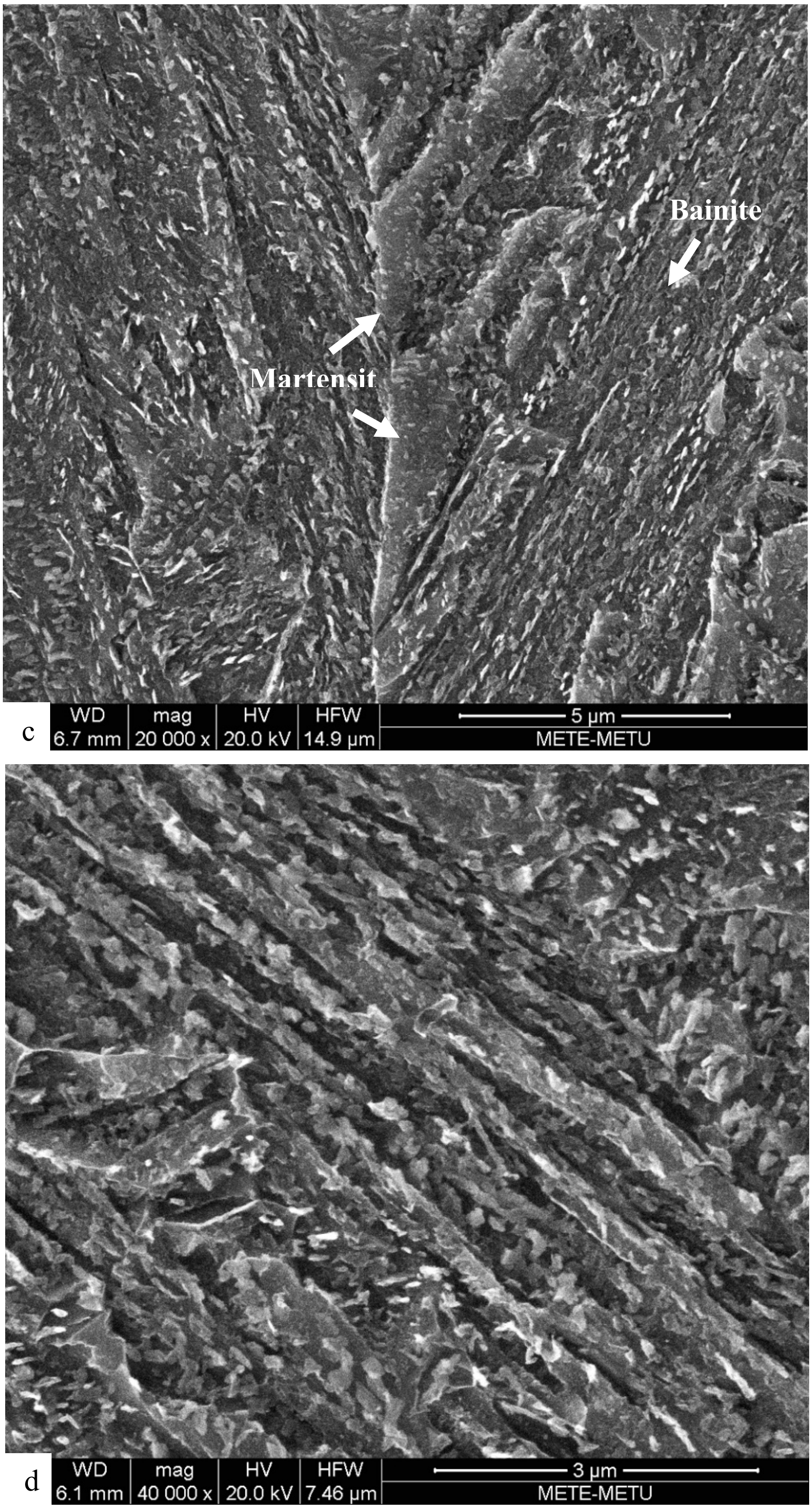


Figure 42. (Cont.)

Bainite and martensite phase mixture obtained by holding 2 minutes at 350°C and then water quenched were tempered at 450°C for 2 hours. After tempering, carbide precipitation took place within martensite islands (Figure 43a and 43b) and sharp martensite corners became rounded (Figure 43a). Bainite and tempered martensite phase mixture was obtained.

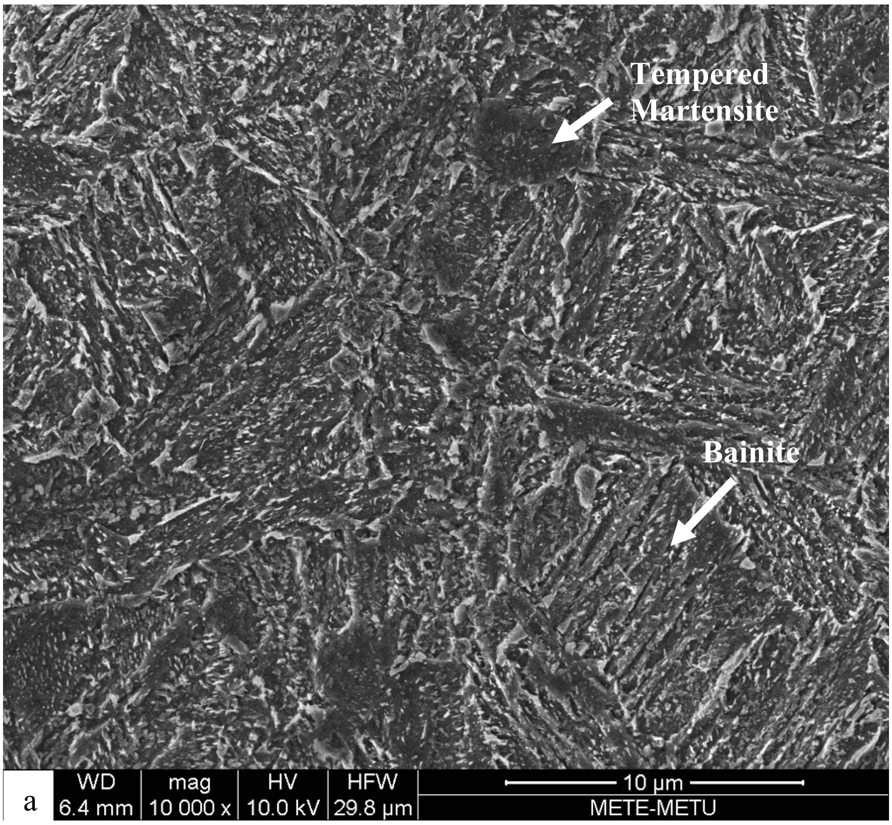


Figure 43. Bainite and tempered martensite phase mixture of 32CrB4 steel obtained by quenching to 350°C and hold isothermally for 2 minutes and water quenching followed by tempering at 450°C for 2 hours.

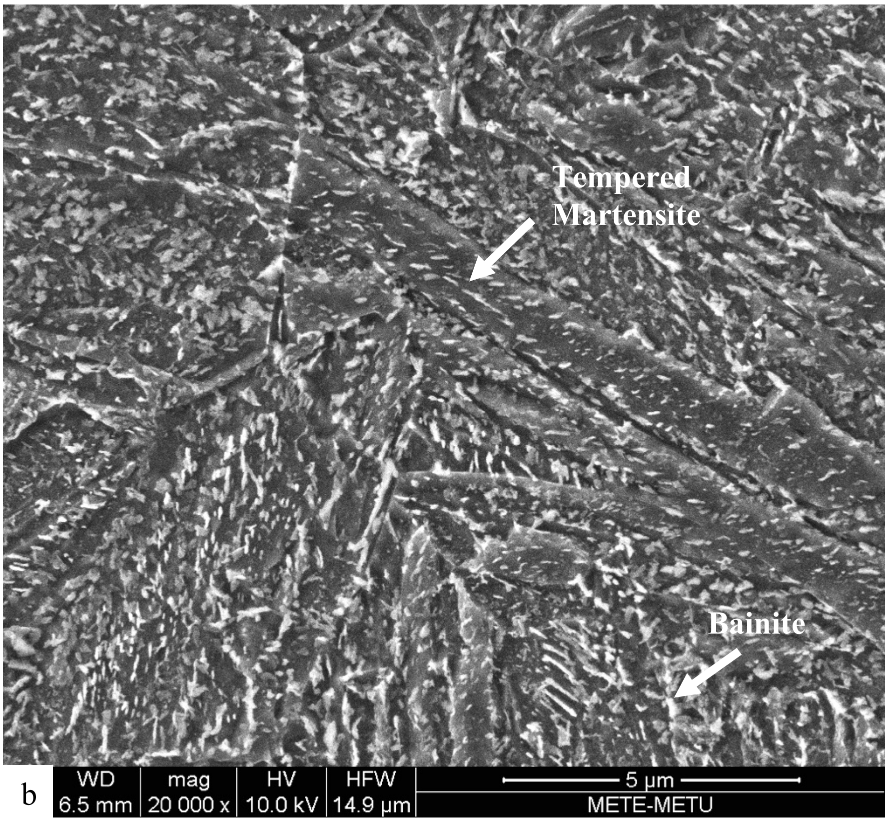


Figure 43. (Cont.)

4.3. Mechanical Tests

4.3.1. Comparison of Hardnesses of Bainite and Martensite

Hardness test results of heat treated 32CrB4 specimens are shown in Table 10. While hardness results of preliminary heat treatments were analyzed, microstructural characterization also taken into consideration.

When 32CrB4 steel is quenched in water a hardness of 618HV is obtained. The hardness was lower, i.e., 565HV when oil is used instead of water as a quenching medium. However, oil quenching is selected for the experiments as there is a lower risk of cracking. Tempering the quenched 32CrB4 steel in the range 400°C-500°C yielded a range of hardness between 465HV-370HV.

An isothermal treatment at 350°C (just above M_s) gives a hardness value of 435HV which is the hardness of 100% bainitic microstructure. Either waiting 1 hour or 3 days at 350°C does not affect the hardness too much and yields around 440HV. Water quenching after 2 hours did not change the hardness, may be explained as transformation completes after 2 hours. Since a bainitic transformation at 350°C (2 hours) and tempered martensite at 450°C for 2 hours have comparable hardnesses, 450°C was chosen as a tempering temperature for the mechanical testing specimens.

Table 10. Hardness test results of 32CrB4 steel

1st Quenching			**2nd Quenching**			**Tempering**	**HV30**
Medium	**Temp. (°C)**	**Isothermal Holding Time**	**Medium**	**Temp. (°C)**	**Isothermal Holding Time**	**Temp. (°C)**	
Water	RT	-	-	-	-	-	618 ± 3
Oil	RT	-	-	-	-	-	565 ± 3
Oil	RT	-	-	-	-	400	464 ± 8
Oil	**RT**	**-**	**-**	**-**	**-**	**450**	**433 ± 1**
Oil	RT	-	-	-	-	500	371 ± 4
Salt	350	1 hour	-	-	-	-	445 ± 3
Salt	**350**	**2 hours**	**-**	**-**	**-**	**-**	**437 ± 4**
Salt	350	2 hours	Water	RT	-	-	437 ± 9
Salt	350	3 days	-	-	-	-	441 ± 2
Salt	350	5 min	Salt	320	1h30min	-	438 ± 8
Salt	**350**	**2 min**	**Salt**	**300**	**5h30min**	**-**	**471 ± 10**
Salt	**350**	**2 min**	**Salt**	**300**	**5h30min**	**450**	**370 ± 1**
Salt	**350**	**2 min**	**Water**	**RT**	**-**	**-**	**472 ± 8**
Salt	**350**	**2 min**	**Water**	**RT**	**-**	**450**	**405 ± 11**
Salt	300	15 min	Water	RT	-	-	522 ± 10

4.3.2. Hardnesses of Two-Step Bainite Transformation

In two-step bainite transformation experiments the aim was compare the behavior of bainite-martensite phase mixtures with that of 100% bainitic and 100% martensitic structures. Also, it is known that the morphology and mechanical properties of bainite is affected from the transformation temperature. Therefore, by consecutive isothermal treatments at two different temperatures may form two different bainite and may have a positive effect on mechanical properties.

Two-step bainite transformation gives a hardness range from 370HV to 470HV. If the sample is isothermally treated first at 350°C for 2 minutes and then at 300°C for 5.5 hours, a hardness of 470HV is obtained. This is higher than 100% bainitic structure. At TTT diagram given in Figure 32, bainite transformation is completed at 5.5 hours at 300°C. When second holding temperature was changed to 320°C and hold for 3 hours 40 minutes, 438HV is obtained. If the sample which was held at 350°C (2mins.) and at 300°C (5.5hrs) is tempered at 450°C for 2 hours the hardness drops from 470HV to 370HV. This most probably indicates that a treatment of 350°C (2 min.) and 300°C (5.5 hours) yields some amount of martensite phase.

Directly quenching to 300°C, which is the second step temperature of two-step transformation, and holding 15 minutes after austenitization, and followed by water quenching produces 522HV. It has lower hardness than oil quenched specimen, which is 565HV.

Bainite-martensite phase mixture obtained by holding at 350°C (2 minutes) and then water quenching gives 472HV and tempering this specimen at 450°C for 2 hours lowers the hardness to 405HV. It is remarkable that, two-step bainite transformation reaches to hardness value of bainite-martensite phase mixture which is 472HV, by holding 2 minutes at 350°C and 5.5 hours at 300°C (Figure 44).

Microstructural characterization and hardness results of preliminary heat treatments were considered together in order to decide main heat treatments. Table 11 gives the time and temperature information of selected heat treatment paths.

Table 11. Heat treatments of 32CrB4 steel specimens.

1st Quenching			2nd Quenching			Tempering
Medium	**Temp. (°C)**	**Isothermal Holding Time**	**Medium**	**Temp. (°C)**	**Isothermal Holding Time**	**Temp. (°C)**
Oil	RT	-	-	-	-	450
Salt bath	350	2 hours	-	-	-	-
Salt bath	350	2 min	Salt bath	300	5h30min	-
Salt bath	350	2 min	Water	RT	-	-
Salt bath	350	2 min	Water	RT	-	450

Figure 44 summarizes the hardness of selected heat treatments.

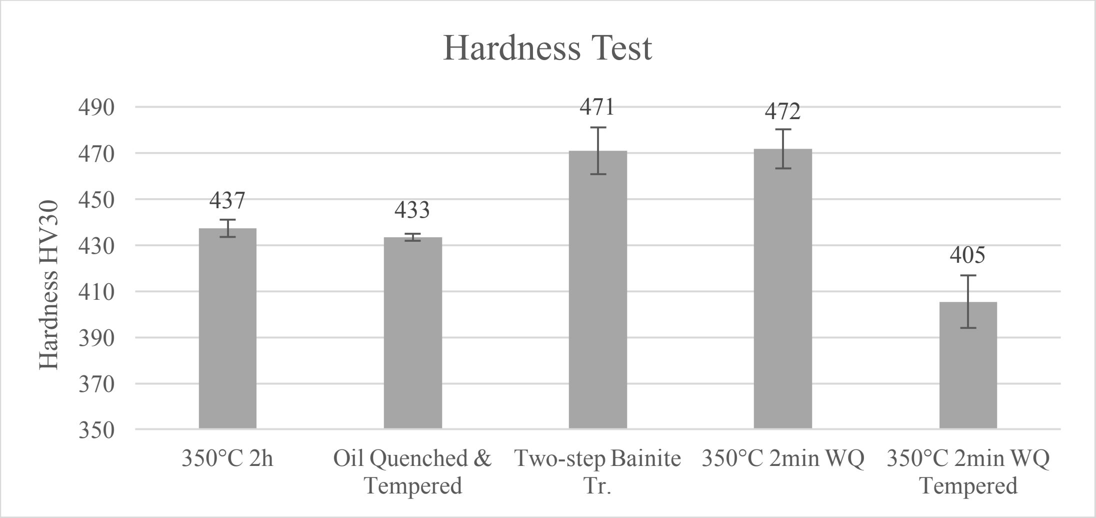


Figure 44. Graph of 32CrB4 steel hardness for selected heat treatments

4.3.3. Tensile Testing

Tensile test results of 5 heat treated samples are given in Figure 45. Table 12 shows numerical results of tensile test.

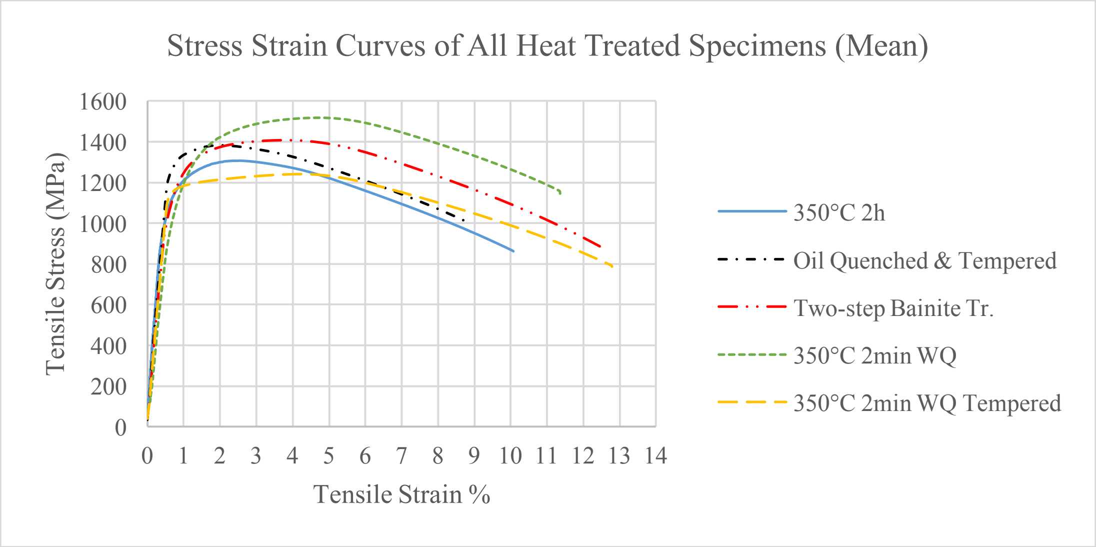


Figure 45. Stress strain curves of heat treated 32CrB4 steel specimens with mean values for each heat treatment process

Table 12. Tension test results of heat treated 32CrB4 steel specimens with mean values for each heat treatment process

Heat Treatment	**Phase**	**Yield Stress (0.2% Offset) (MPa)**	**Ultimate Tensile Stress (MPa)**	**Tensile Strain (%)**	$\frac{\mathbf{TS}}{\mathbf{YS}}$
350°C 2h	100%B	1125	1326	11.17	1.18
Oil Quenched & Tempered	100%M	1319	1397	10.03	1.06
Two-step Bainite Tr.	B+M	1158	1410	13.23	1.22
350°C 2min WQ	85%B+15%M	1165	1520	11.53	1.30
350°C 2min WQ Tempered	85%B+15%TM	1166	1241	13.47	1.10

Tempered martensite shows highest yield strength with 1319 MPa and fully bainite phase has the lowest with 1125 MPa. Two-step bainite transformation produces 1158 MPa yield strength and it can be said that improves the yield strength of bainite phase. 85%Bainite-15%Martensite phase mixture has 1165 MPa yield strength and tempering did not change this value.

Ultimate (or maximum) tensile strength (UTS) of fully bainite is lower than that of tempered martensite. Two-step bainite transformation improves effectively the UTS of bainite. Bainite-martensite phase mixture has the highest UTS and tempering this mixture decreases the UTS remarkably.

Percent elongation of tempered martensite is the lowest with 10% whereas fully bainitic structure yielded slightly higher % elongation, i.e., 11.2%. Two-step bainite transformation gives 13.23% tensile strain. Bainite-martensite phase mixture has 11.53% and tempering increased it to 13.47% which makes it the highest tensile strain.

4.3.4. Notched Bar Charpy Impact Test

Charpy impact test results are given in Figure 46.

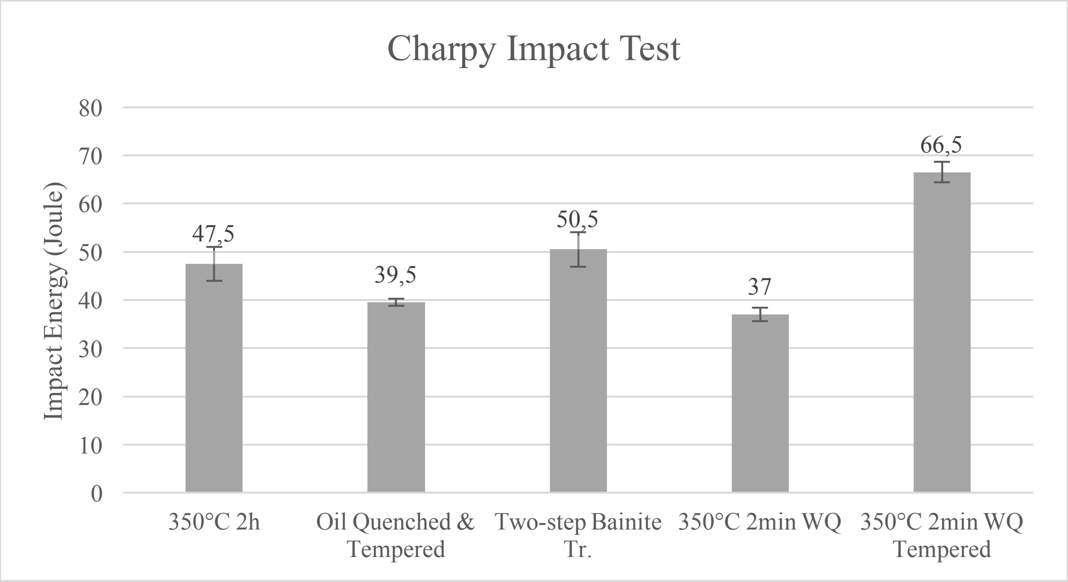


Figure 46. Charpy impact test results of heat treated 32CrB4 steel specimens

Toughness of specimens were measured by Charpy impact test. Tempered martensite absorbs 39.5 Joules impact energy and it is lower than that of fully bainite phase. Fully bainite phase has 47.5 Joules impact energy and in two-step bainitic transformation sample it increases to 50.5 Joules. Bainite-martensite phase mixture has a toughness value of 37 Joules, which is very close to that of quenched and tempered specimen. If the Bainite-martensite phase mixture is tempered, the toughness increases to 66.5 Joules.

Graph of Charpy impact energy vs. tensile strength is given in Figure 47. Bainite-martensite phase mixture, bainite-tempered martensite phase mixture and fully bainite obtained by two-step bainite transformation have superior results according to graph. Two-step bainite transformation seems to produce a phase that shows optimum mechanical results among all.

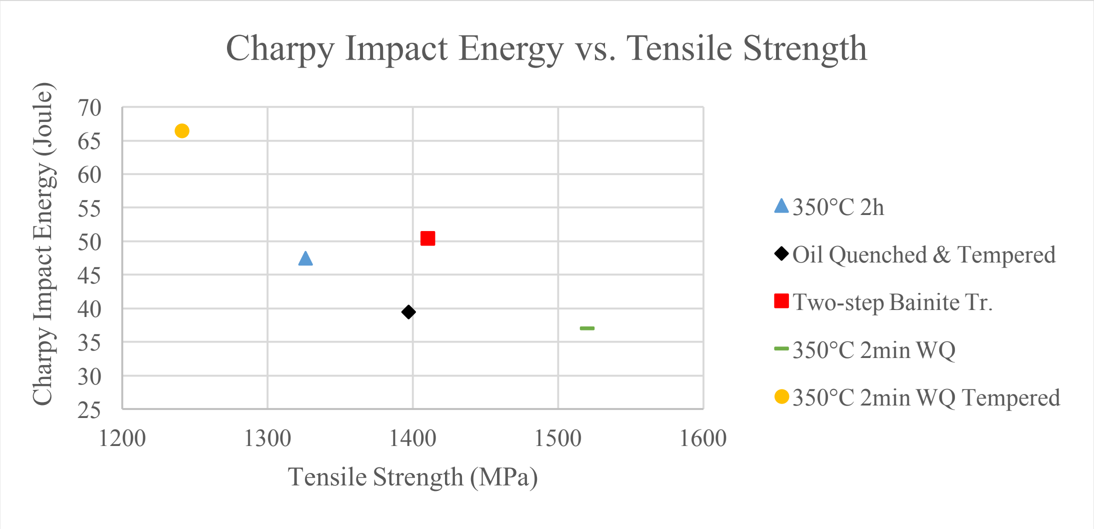


Figure 47. Graph of Charpy impact energy vs. tensile strength of heat treated 32CrB4 steel specimens.

Fracture surfaces of notched bar Charpy impact test samples were analyzed under SEM. Single-step bainite transformation images can be seen in Figure 48.

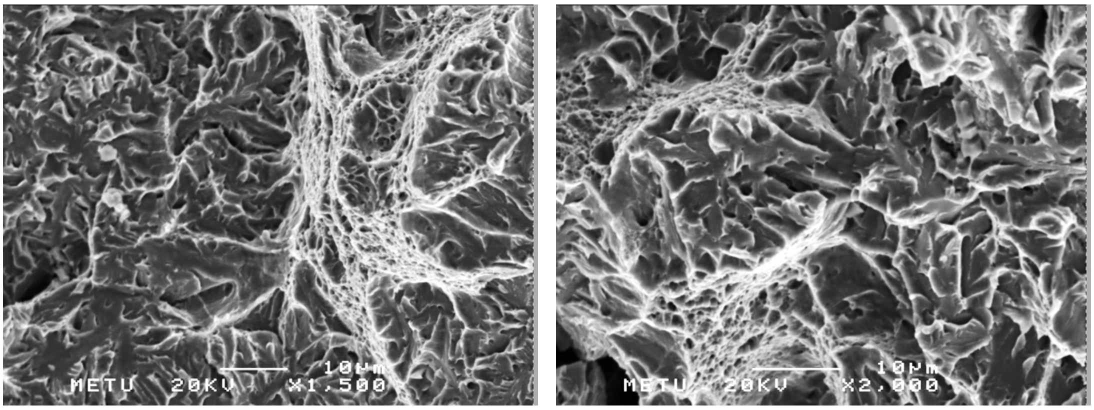


Figure 48. Fracture surface images of 32CrB4 steel quenched to 350°C and hold isothermally for 2 hours.

Fracture surface of sample hold at 350°C for 2 hours shows that facture is ductile. When two-step bainite transformation fracture surface is analyzed (Figure 50), very similar surfaces are observed with the single-step bainite transformation. They show the same fracture characteristics. Dimples and quasi-cleavage areas were observed in both Figure 48 and 49.

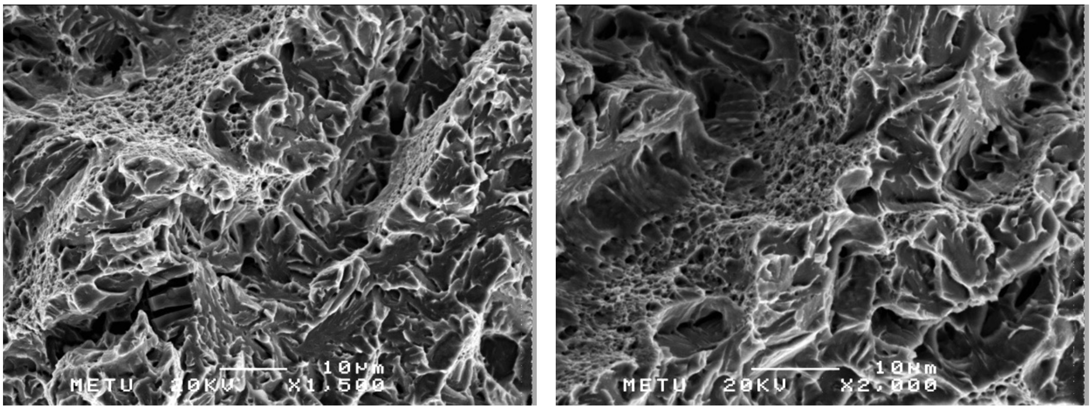


Figure 49. Fracture surface images of 32CrB4 steel that has a two-step bainite transformation

Fracture surface of sample hold 2 minutes at 350°C and then water quenched was analyzed (Figure 50), fracture can be still defined as ductile but less dimples were observed, relative to single and two-step bainite transformations.

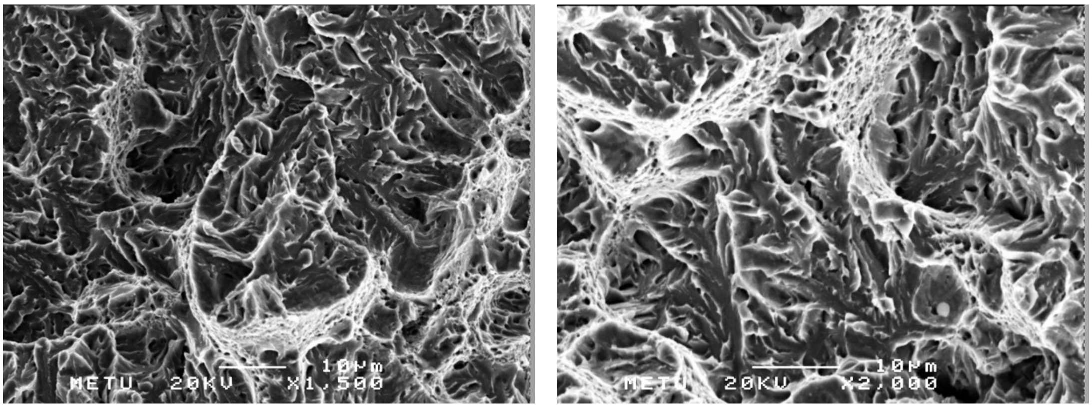


Figure 50. Fracture surface images of 32CrB4 steel that hold isothermally at 350°C for 2 minutes and then water quenched.

Tempering the 350°C 2min + WQ specimen increases the amount of ductile fracture regions most probably due to that martensite islands becomes softer, as Figure 51 shows.

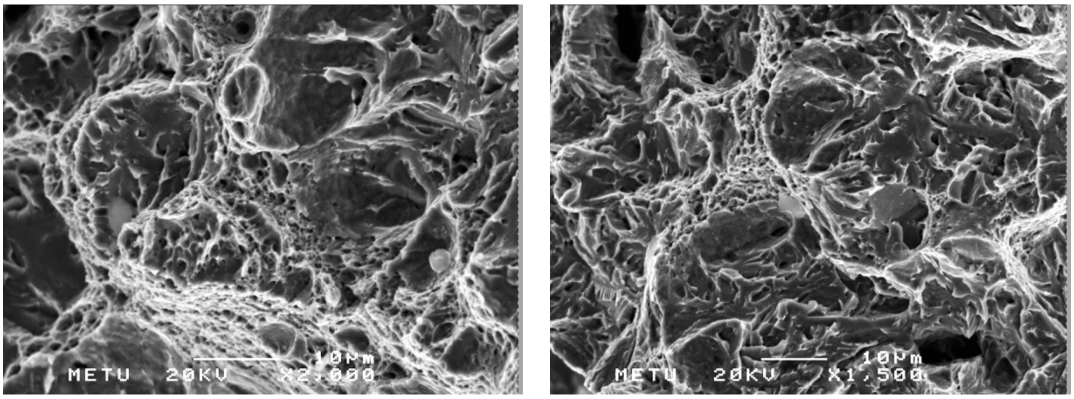


Figure 51. Fracture surface images of 32CrB4 steel that hold isothermally at 350°C for 2 minutes then water quenched and tempered.

CHAPTER 5

DISCUSSION

In this study, two steels were used to obtain bainitic structures and improve mechanical properties of bainite by altering heat treatment processes. Steels were high carbon railway steel R260 and medium carbon boron steel 32CrB4. In this chapter, experimental results of these two heat treated steels will be discussed. There are mainly two important parameters: holding time and temperature. These parameters affect the microstructure and mechanical properties of steels.

By looking at the metallographic studies of R260 steel, it is seen that bainitic structure forms only at the grain boundaries. Increasing the isothermal holding time to 7 days did not increase the percentage of bainite. Bainitic structure did not grow further, resultant structure of all heat treatments was martensitic, where bainite is seen only at the grain boundaries. In TTT diagram of R260 steel, it can be seen that bainite nose is behind the pearlite nose, and pearlitic transformation starts in 2 seconds. Alloying elements like Cr, Mo, Mn, Ni moves pearlite nose to right and bainite formation becomes easier. In steel R260, the major alloying element is Mn, which is known to promote pearlite formation. Also, being the hardenability of R260 is very low, several dark appearing phase at the grain boundaries can be also pearlite.

There are ways of increasing bainite for this alloy. First one is increasing the isothermal holding temperature and the second one is increasing the holding time. However, these ways do not coincide with aims of this study. Increasing the temperature forms upper bainite which has a lower hardness, tensile strength and toughness relative to lower bainite. Increasing holding time, on the other hand, is not economical since even 7 days were not enough to increase bainite percentage. Because of that, tension tests and Charpy impact tests were not applied to R260 steel.

Experiments of 32CrB4 steel show that, bainite can be obtained and mechanical properties of bainite can be improved with different heat treatments. All heat treatments were designed according to TTT diagram that was constructed by JMatPro software since TTT diagram of 32CrB4 was not found in literature. Also, preliminary experiments should have been carried out to confirm the constructed diagram, but only for chosen temperatures. These preliminary experiments were also used for deciding 5 different heat treatment paths, which are the basis of this thesis. According to the micrographs, constructed TTT diagram was correct. At 350°C, just above M_s, bainite transformation starts in 100 seconds and completed in 85 minutes. Isothermal holding at 350°C for 85 minutes, 2 hours, 2 hours and water quenching and 3 days were held. Microstructure and hardness values do not change after all these experiments, which proves that bainitic transformation is complete after 85 minutes as given in TTT diagram.

Spectral analysis result of 32CrB4 steel was identical with the chemical composition in literature. Microstructural studies of as-received structure show that spheroidization was applied to the sample since spherical cementite particles embedded in ferrite matrix were observed.

5.1. Microstructural Characterization of Heat Treated 32CrB4 Specimens

The fully bainite phase mixture by holding 2 hours isothermally at 350°C, is a lower bainite because 350°C is just above M_s temperature. Also, lower bainite is known as having carbide precipitates in ferrite and in Figure 39c, SEM image very clearly shows carbide precipitation in bainite sheaves. A few amounts of martensite phase also exist as martensite islands, it is distinguishable in both optical images and SEM images in Figure 37 and 38. Since quenching temperature is very close to M_s, martensite formation can be expected. Bainite phase microstructure has parallel sheaves but martensite needles have a random distribution. In SEM images, it can be differentiated by detecting the parallel orientation of sheaves. For optical images, bainite is known to be etched more than martensite and Figure 36 shows very clearly the difference of two phases. Bainite is darker than martensite. For the fully bainite phase, few martensite islands can be detected as light-yellow areas.

Two-step bainite transformation produces a similar microstructure with that of single step bainite formation. In fact, microstructure formed after second quenching should produce finer bainite sheaves; however, it is very hard to differentiate. In Figure 52, several sheaves can be seen, fully bainite phase has 0.85µm sheave width. On the other hand, in specimen transformed to bainite by two-step transformation, several sheaves have been found having a width of 0.20µm. The reason of decrease in sheave width may be explained as following: As bainite transformation continues, carbon partitioning from bainite to untransformed austenite takes place. Carbon content of austenite increases and as a result, M_s and M_f becomes lowered. This gives an opportunity for bainite transformation to continue even the temperature is lowered. At lower temperature, driving force for transformation increases and nucleation rate becomes higher. High carbon in austenite also has a contribution to finer structure since carbon atoms may resist motion of interface. Carbon atoms that could not diffuse to austenite, precipitate in ferrite.

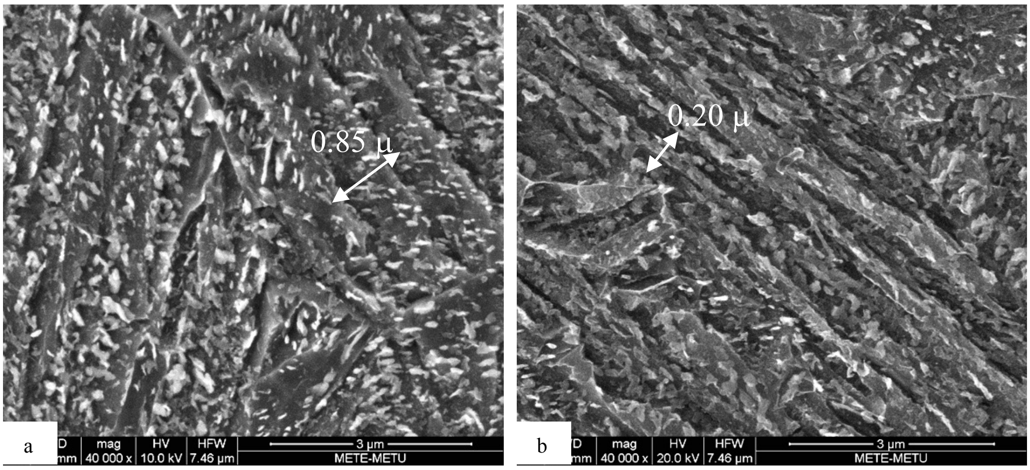


Figure 52. Bainitic structures formed by (a) single step isothermal holding at 350°C for 2 hours (b) two-step quenching and partitioning at 350°C for 2 minutes and 300°C for 5.5 hours.

Isothermal holding at 350°C for 2 minutes and then water quenching produces bainite and martensite phase mixture as it can be seen in Figure 40 very clearly. This heat treatment shows clearly how the structure of bainite has formed during the first step of two-step bainite transformation. After the bainite is formed, rest of the structure transforms to martensite since water quenching was applied. Bainite transformation starts very fast and nearly 85% of austenite transforms to bainite in 2 minutes. In this specimen, the martensite formed can be detected very easily, because there is no carbide precipitation within martensite.

After an isothermal treatment at 350°C for 2 minutes and water quenching, tempering at 450°C for 2 hours was applied. It is seen that carbides precipitate within martensite islands and sharp corners of martensite structures become rounded. This carbide precipitation in martensite makes it difficult to differentiate bainite from martensite. But SEM images show that, bainite has parallel needles and carbide precipitations are also in the same order since they are precipitate on the ferrite phase. In tempered martensite, on the other hand, carbide precipitation does not have definite orientation and they are dispersed, parallel sequence does not exist. This difference sometimes helps to differentiate bainite from martensite.

Table 13. Microstructures of heat treated 32CrB4 specimens

Heat Treatment	Microstructure
350°C 2h	Almost fully bainite + few martensite
Oil Quenched & Tempered	Tempered martensite
Two-step Bainite Transformation	Primary and secondary bainite + few martensite
350°C 2min & WQ	Bainite + martensite
350°C 2min & WQ & Tempered	Bainite + tempered martensite

5.2. Mechanical Properties of Heat Treated 32CrB4 Specimens

Hardness, tension and Charpy impact tests were done to heat treated 32CrB4 steel specimens in order to understand the effects of microstructure and phase mixtures on mechanical properties.

Fully bainite phase, obtained by holding 2 hours at 350°C, and tempered martensite, obtained by oil quenching and tempering at 450°C for 2 hours, are compared in terms of mechanical properties. It is seen that, bainite can reach to the tensile strength of tempered martensite, when transformed just above M_s temperature, thanks to its fine structure. High grain boundary area, fine and uniform distributed carbides precipitated on bainite sheaves play an important role for increasing the tensile strength of bainite. Besides, impact toughness and elongation of bainite are higher than that of tempered martensite and again, fine structure may be the explanation for high ductility.

After tempering the bainite-martensite phase mixture, obtained by holding 2 minutes at 350°C and water quenched, hardness decreases to 405HV. When compared with the hardness of oil quenched and tempered martensite (433HV), it is lower. This can be attributed to the response of martensite to tempering. Since B_s is higher than M_s, the bainite formed in isothermal transformation is autotempered. As a result, the hardness of bainite phase mixture is affected at a much lower degree with respect to martensite [1]. Therefore, it can be stated that a tempering operation of bainite-martensite phase mixture at 450°C affects the martensite component and a hardness drop is seen due to the tempering of martensite phase.

Two step bainite transformation is done to obtain bainite with two different sheave sizes. Upon transformation at 350°C for 2min, approximately 85% coarse bainite should form. The remaining austenite should be enriched in carbon, due to rejection from bainite sheaves, and consequently M_s would be shifted to lower temperatures. Then a second step of isothermal transformation at 300°C (5.5hrs) would yield a bainite with smaller sheave width. However, the tempering results indicate that the M_s

may not shifted to lower temperatures and martensite is formed upon cooling to 300°C (5.5hrs) for a second step isothermal treatment. The comparison of hardnesses of the specimens indicate the presence of martensite. An isothermal treatment of 350°C (2min) + 300°C (5.5hrs) yield a hardness of 471HV. This hardness drops to 370HV upon tempering this specimen at 450°C. It is a well-known fact that the hardness of bainite is much less sensitive to tempering than that of martensite. Therefore, a decrease in hardness from 471HV to 370HV indicates that 100% bainite could not be obtained but rather some amount of martensite is formed. When tempered, the drop in hardness of 350°C (2min) + WQ specimen is not as significant as that of 350°C (2min) + 300°C (5.5hrs) specimen. This can possibly be attributed to the stage at which martensite forms. If some amount of martensite is formed upon cooling to 300°C, then this martensite phase will be autotempered upon waiting at 300°C for 5.5hs.

By looking at the hardness values of two-step bainite transformation and bainite-martensite phase mixture obtained by holding 2 minutes at 350°C and water quenching, it can be thought that they have the same microstructure; but by considering the Charpy impact test results, it is obvious that two-step bainite transformation has high toughness. Together with the microstructural analyses, it can be concluded that the higher amount of bainite phase mixture and/or autotempering of martensite led to a much finer and tougher structure.

Wang *et al.* reported that, two-step bainite transformation gives 485HV and 42 Joules absorbed energy while conventional bainite transformation gives 467HV and 40 Joules absorbed energy. In this study, 471HV and 50,5 Joules for two-step bainite transformation and 437HV and 47,5 Joules for single step bainite transformation were observed. Higher hardness increase was measured in this study, and in terms of impact energy, difference is similar. When tensile strength and percent elongation were considered, they reported 1565 MPa and 12.5% for two-step bainite transformation and 1380 MPa and 11.7% for conventional bainite. In this study, 1410 MPa and 13.23% for two-step bainite transformation and 1326 MPa and 11.17% for single-step bainite transformation were measured. In this study, the two-step transformation yields slightly lower strength values but single step transformation yields very similar results in comparison to that of Wang *et al.*.

Fracture surface analysis of Charpy impact test specimens shows that, bainite and two-step bainite transformations have ductile fracture and their surface characteristics are very similar. When compare with the impact energy values, which are 47,5 Joules for bainite and 50,5 Joules for two-step bainite transformation, it seems reasonable. Held at 350°C for 2 minutes and then water quenched sample, on the other hand, has 37 Joules impact energy and when it was tempered, absorbed energy increases to 66,5 Joules. Fracture surface of this tempered specimen has relatively high dimples since hard martensite islands becomes soft and explains the ductile fracture. As-quenched specimen has lowest impact energy and fracture surface contains few dimples and more quasi-cleavage areas. When considering the stress-strain graph (Figure 46), 350°C (2min) + WQ specimen had more strain hardening but impact energy was lower than the expected. Low impact toughness could be explained with the existence of as-quenched martensite islands. The carbon content of this martensite islands is also higher than the original alloy composition since carbon was partitioned during the bainite transformation.

Two-step bainite transformation shows the second-best results for all mechanical tests, while the best one changes for every test. Explanation for this result is that obtained bainite-finer secondary bainite mixture meets all desired mechanical properties, high tensile strength, high elongation and high toughness (Figure 47).

CHAPTER 6

CONCLUSION

In this study, R260 steel and 32CrB4 steel were used to obtain bainitic structure. Mainly two heat treatments were done for obtaining bainite, first one is single step bainite transformation and the second one is two-step bainite transformation processes. Oil quenching and tempering was done for comparison of tempered martensite with bainite. Another two heat treatments were performed to understand the phases formed at the first stage of two-step bainite transformation. Microstructural and mechanical properties of obtained phases were investigated. The following conclusions are withdrawn from this study:

1. Fully bainitic transformation is not possible for R260 steel by holding isothermally up to 7 days at just above M_s temperature.
2. In 32CrB4 steel, bainitic transformation is complete after a 1 hour isothermal treatment at 350°C, just above the M_s temperature.
3. The hardness of 100% bainitic structure is 437HV. On the other hand, 100% martensitic structure yields an as-quenched hardness of 565HV which drops to 433HV upon tempering at 450°C for 2 hours
4. Bainite formed at M_s temperature can compete with tempered martensite in terms of mechanical properties. Having the same hardness level (433HV), tempered martensite has a higher UTS value of 1397 MPa which is higher than bainite UTS value of 1326 MPa. Beside that fact, from the tensile strain and Charpy impact toughness point of view, bainite shows superior values than tempered martensite which are 11.7%-47.5 Joules and 10.03%-39.5 Joules, respectively.
5. Two-step bainite transformation improves the mechanical properties of bainite by forming secondary bainite with finer sheaves. 1410 MPa UTS, 13.23%

tensile strain and 50,5 Joules Charpy impact energy are obtained. These properties are also superior relative to tempered martensite.

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