Effect of Twinning on the Mechanical Properties of Bainitic Steels

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By

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CERTIFICATE

I, Vivek Kumar Singh, hereby declare that the work presented in this thesis entitled "**Effect of Twinning on the Mechanical Properties of Bainitic steels**" in fulfillment of the requirement for the award of degree of Master of Engineering (PE) submitted to Department of Mechanical Engineering, **Thapar Institute of Engineering and Technology (deemed to be university), Patiala** is an authentic record of work carried out under the supervision of **Dr. Tarun Nanda** Associate Professor, MED, TIET, Patiala, **Dr. Ajay Batish,** Professor, MED, TIET, Patiala and **Dr. Debasis Poddar**, Principal Researcher, R&D, Tata Steel, Jamshedpur from July 2016 to July 2018. The matter presented in this report has not been submitted either in part or full to any other university or institute for the award of any degree.

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ABSTRACT

Bainitic steels are able to provide ultra-high strengths but show limited ductility. Authors have worked to develop bainitic steels with different chemical compositions and various heat treatment processing routes. In most of the studies, the toughness values have been achieved because of the very high strength values obtained. However, limited ductility has been a concern in these developed steels resulting in poor formability. Very limited literature is available where authors have developed bainitic grades with good combination of high strength-high ductility. There is a necessity to develop high toughness bainitic steels with improved ductility and hence improved formability. The main objective of the present dissertation work was to develop bainitic steel with high strength and high ductility by obtaining maximum volume fraction of bainite phase mixture in the microstructure, reducing the cementite/carbide content in bainite, and obtaining bainitic-ferrite as thin-plates. To achieve this objective, the steel chemistry-heat treatment combination was very carefully designed. The thermo-mechanical processing comprised of hot forging and hot rolling (mechanical portion of processing) followed by austempering (heat treatment or thermo-portion of thermo-mechanical processing). Bainite was obtained by isothermal holding at a low transformation temperature. The soaking time for isothermal transformation at the bainitic transformation temperature (350°C) was varied over a wide range in three distinct periods of 1h, 1 day, and 3 days to study the behavior and mechanical properties of bainitic-ferrite and RA obtained in the microstructure under different conditions. The results of tensile testing showed that microstructure comprising of high volume fraction of bainite (in the form of thin bainitic-ferrite) along with some amount of stable retained austenite results in good strength-ductility combination in bainitic steel. The bainitic steel (1 day sample) comprising of bainite (76.45%) and retained austenite (23.55%) showed the best properties with high strength (1507.29 MPa) and good ductility (30.0%). Characterization results showed thin plates of bainite-ferrite uniformly distributed throughout the micrograph along with RA which was present in two forms viz. fine austenite and blocky austenite. Thin films of RA in-between the sheaves of bainite-ferrite helped to improve the mechanical properties. For the given thermo-mechanical processing conditions applied to the steel chemistry, the microstructure of the bainitic steel with best properties showed presence of stacking faults (SFs)/twins in the microstructure.

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Nomenclature

Acronym	**Full Form**
AHSS	Advance High Strength Steel
ASTM	American Society for Testing and Materials
AISI	American Iron and Steel Institute
AT	Austenization Temperature
BF	Bainitic-Ferrite
CCT	Continues Cooling Curve
CRSS	Critical Resolved Shear Stress
DP	Dual Phase
DT	Deformation Temperature
EBSD	Electron Backscatter Diffraction
EDM	Electrical Discharge Machine
FCC	Face-Centered Cubic
HRTEM	High Resolution Transmission Electron Microscopy
LSCM	Laser Scanning Confocal Microscopy
RA	Retained Austenite
SEM	Scanning Electron Microscopy
SFs	Stacking Faults
SFE	Stacking Fault Energy
SADP	Surface Area Diffraction Pattern
TEM	Transmission Electron Microscopy
TRIP	Transformation Induced Plasticity
TWIP	Twin Induced Plasticity
TT	Transformation Temperature
TTT	Time-Temperature-Transformation
UTS	Universal Tensile Strength
XRD	X-Ray Diffraction
YS	Yield Strength

CHAPTER 1
INTRODUCTION

1.1 General

Steels can be argued to be the most important and useful of all materials because of their relatively low price and the wide range of desirable properties which can be controlled by varying the steel chemistry and processing parameters. The mechanical properties depend to a reasonable extent on the microstructure. Phase transformations are therefore important in designing the microstructures for specific applications. Several techniques (heat treatment, work hardening etc.) are used to improve properties of steels [1, 2].

Traditional single phase steels either have good strength (e.g. martensitic steels) or good formability (e.g. ferritic steels). However, multi-phase steels like DP steels (dual-phase), TRIP steels (transformation-induced-plasticity steels) can provide remarkable properties with good high strength-good formability combination [3]. The current demand for armor vehicles requires materials with high strength along with high ductility (so as to possess good toughness). This requires processing of novel materials with extraordinary combination of strength and ductility. A reasonable amount of success has been achieved by development of a new category of steels called, 'Advanced High Strength Steels (AHSS)'. Another category which comes to rescue for these type of applications are the, 'bainitic steels'. The development of bainitic steels has become an area of interest in the armor steel industry, railway industry, construction industry etc. because of its ultra- high strength [4]. The ductility of bainite has always beena major concern for several years now. In recent years, developments have been made in this regard by processing carbide free bainitic steels [4, 5, 6, 7].

1.2 Bainitic steels

In this section, the main focus is on bainitic grade steel and its properties. Bainite forms by the decomposition of austenite between B_S and B_F temperature range. Bainite microstructure consists of aggregates of ferrite plates, separated by un-transformed austenite, martensite, or cementite. The clusters of plates grow in a parallel fashion and are called sheaves [1, 2]. The individual ferrite plates belonging to a sheaf are known as 'sub-units' of bainite. In carbide-free bainitic steels, the bainitic-ferrite and retained austenite plates can be observed clearly and separately in the microstructure (see Figure 1.1). In the microstructure, bainite-ferrite plates strengthen the steel and the stable retained austenite enhances the overall ductility [4, 6].The form in which retained austenite is present is important in this context. The phase can be present in the microstructure in two forms: (a) blocky and (b) film type. It is reported that the best elongation behavior is observed when RA is present as films in-between sub-units of bainite and not in the form of blocks between sheaves of bainitic-ferrite [8, 9]. Literature shows that the bainitic steels currently development is able to provide ultra-high strengths (of the order of 1790 MPa) but show limited ductility (of the order of 11–14%) [5, 6]. Though, the required toughness is obtained in these steels due to the exceptional strength, limited ductility results in poor formability.

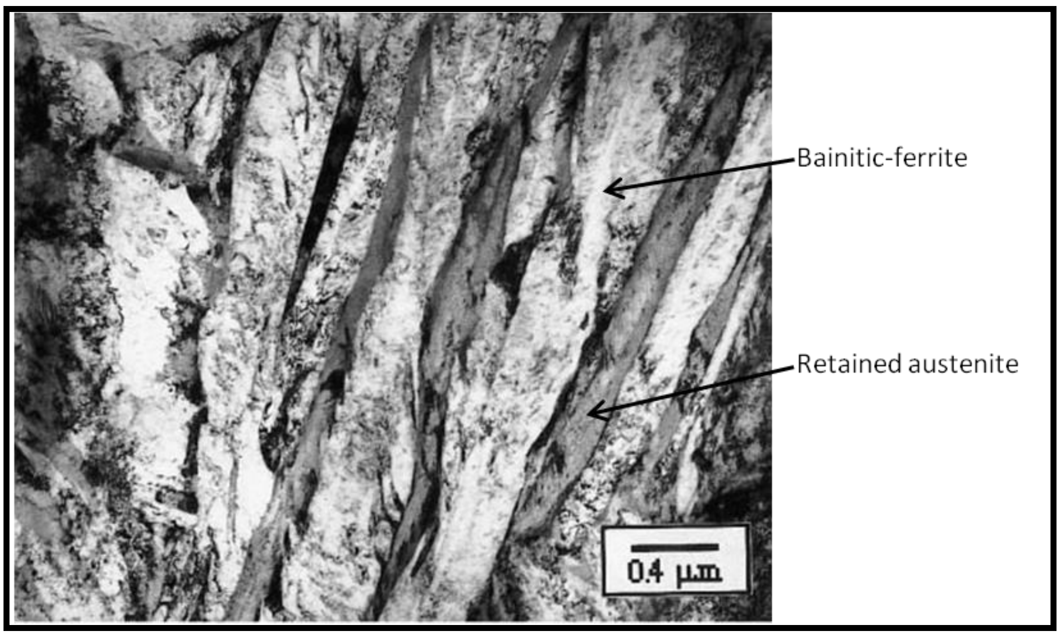


Figure 1.1 Bainite microstructure consisting of bainitic-ferrite and retained austenite [10].

1.3 Bainitic transformation

The bainitic transformation temperature range is in-between the transformation temperature range of pearlite and martensite. Bainitic structure can be generated either through isothermal transformation or through continuous cooling below the bainite-start (B_S) temperature as shown in Figure 1.2.

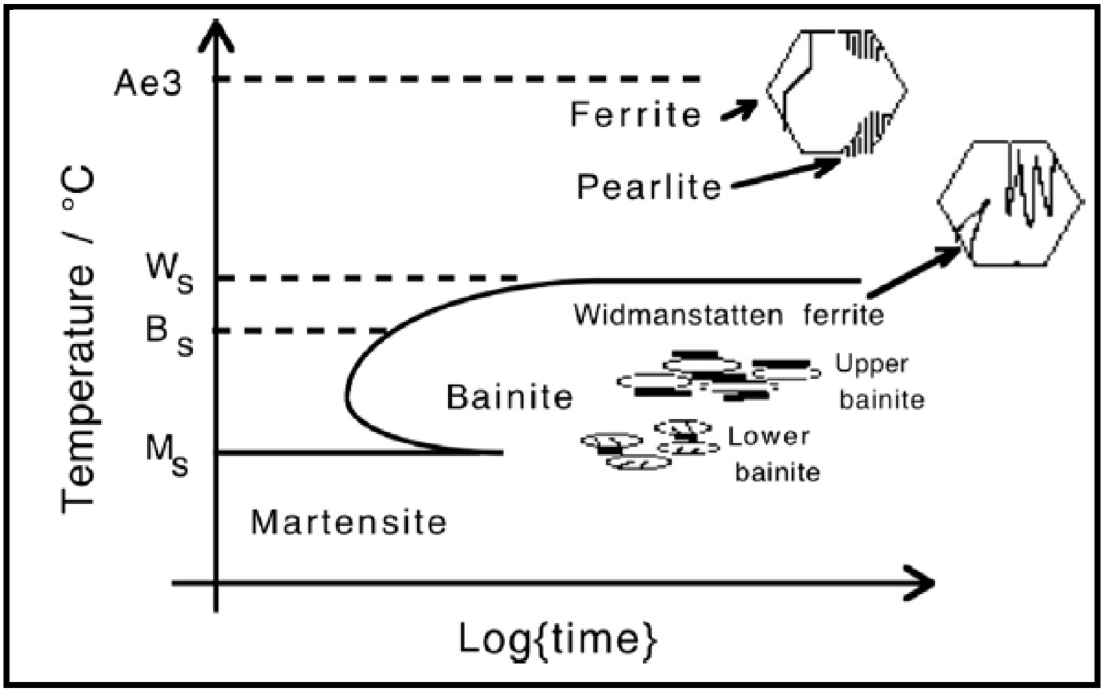


Figure 1.2 TTT diagram showing different domains of transformation [10].

Bainite transformation takes place through a displacive mechanism, without disrupting the relative order of the atoms in the parent phase (i.e. austenite). It is done by generating the unit cell of ferrite by homogeneous deformation of parent austenite phase. When such shape deformation occurs in bulk, the accommodation leads to high strain energy. To release this energy, ferrite plates opt for the thin plate morphology called bainitic-ferrite. In displacive mode of transformation, the resulting phase maintains the atomic sequence as had existed in the parent phase. The transformation of parent phase was also possible by breaking the bonds between the atoms and rearranging them into the structure to maintain the overall shape. Such type of transformation is termed as reconstructive transformation which is observed during austenite to pearlite transformation. Both the types of transformation mechanisms (as discussed here) are shown in Figure 1.3.

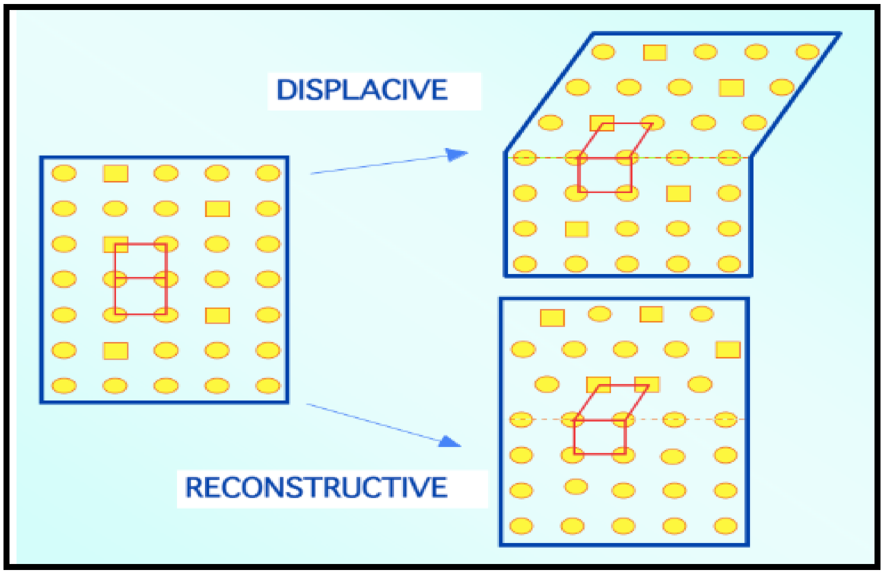


Figure 1.3 Displacive and reconstructive modes of transformation [11].

The nucleus of bainite is ferrite. During bainitic transformation in steels, the scale, the type of structure, and the strength obtained directly depend on the movement of atoms [1]. Bainite is generally observed in two main forms based on morphology (i. upper or feathery bainite, and ii. lower or acicular bainite). The type of morphology obtained in bainite during austenite decomposition depends on the initial composition of steel and also on the transformation temperature for bainitic transformation during heat treatment [4].

1.4 Retained austenite

RA is un-transformed austenite contained in the microstructure of steel under room temperature conditions. The properties displayed by steels containing RA are dependent on its stability. The stability depends on various factors like carbon content in RA, morphology of RA etc. [12]. Researchers have reported that stability of RA is more at lower bainitic transformation temperatures [13, 14]. During rapid cooling to the bainitic transformation temperature and the subsequent holding at this temperature, the transformation reaction proceeds by growth of bainitic-ferrite plates resulting in carbon enrichment and modification of the morphology of residual austenite. In bainitic steels, RA is present either in the form of thin films between the sub-units of sheaves of bainite-ferrite, or as coarser blocks between different sheaves. Both forms of RA are carbon enriched but the thin film morphology is relatively more enriched because of its geometrical isolation between the plates of ferrite [15]. It is reported that presence of blocky morphology of retained austenite is detrimental to toughness [16, 17]. In a given bainitic steel, if the RA present is un-stable, it has tendency to transform to martensite under external stresses. On the other hand, if the available RA is stable, the martensitic transformation becomes sluggish and results in twinning rather than phase transformation.

1.5 Twinning and stacking faults

Twining is an indispensable and unavoidable mechanism of deformation in polycrystalline materials. It is well known that formation of twins in steels increases the elongation or ductility with nominal decrement in tensile strength. In twining, each atom moves by only a fraction of inter-atomic distance relative to other atoms in the plane. In some metals, the occurrence of twins takes place during plastic deformation which is called mechanical twining. However, some other metals exhibit twining after their heat treatment, which is called annealing twinning. In this process, a region of a crystal undergoes a homogeneous shear that produces the original crystal structure in a new orientation (see Figure 1.4) [18]. Hollow circles and black solid circles refer to positions of atoms before and after twinning respectively. The atoms above X-Y are mirror images of atoms below. This result in atoms of the original crystal ('parent') and those of the product crystal ('twin') being mirror images of each other by reflection in a composition plane.

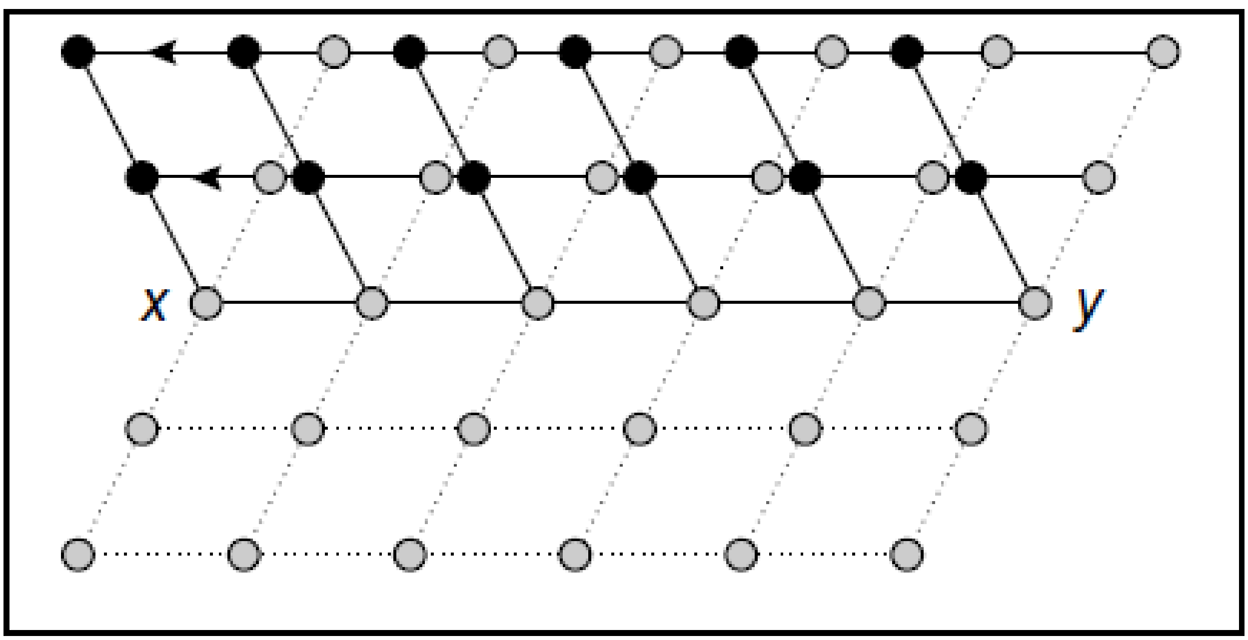


Figure 1.4 Atom arrangement in a twin related structure [19].

Twins produce kinematical barriers to dislocation motion [20]. The rate of nucleation of twins depends on dislocation density, twin nucleus size, and the stacking fault energy (SFE) through critical twin stress. Literature reports that for SFE <18 $mJmol^{-1}$, 'ε' martensitic transformation occurs while for 18$mJmol^{-1}$<SFE<35 $mJmol^{-1}$, twinning occurs [21]. In general, twins are easily achieved in low stacking fault metals and alloys where a limited number of slip systems are available for plastic deformation. Stacking fault (a planar defect) is a local region in the crystal where regular stacking sequence is interrupted [19]. The associated energy per unit area of fault is known as the stacking-fault energy [19].

Twin is the preferential deformation mode of crystal when the shear stress required to slip (critical resolved shear stress, CRSS) is higher than the twining stress [02].

1.6 Dislocations

Dislocations are non-equilibrium defects which get developed in a crystal during solidification/phase transformation/or during deformation. Dislocation movement under the influence of applied external stress/load results in plastic strain. It is based on the fact that when a dislocation moves, two atoms on sites adjacent across the plane of motion are displaced relative to each other by the Burgers vector b (see Figure 1.5) [18].

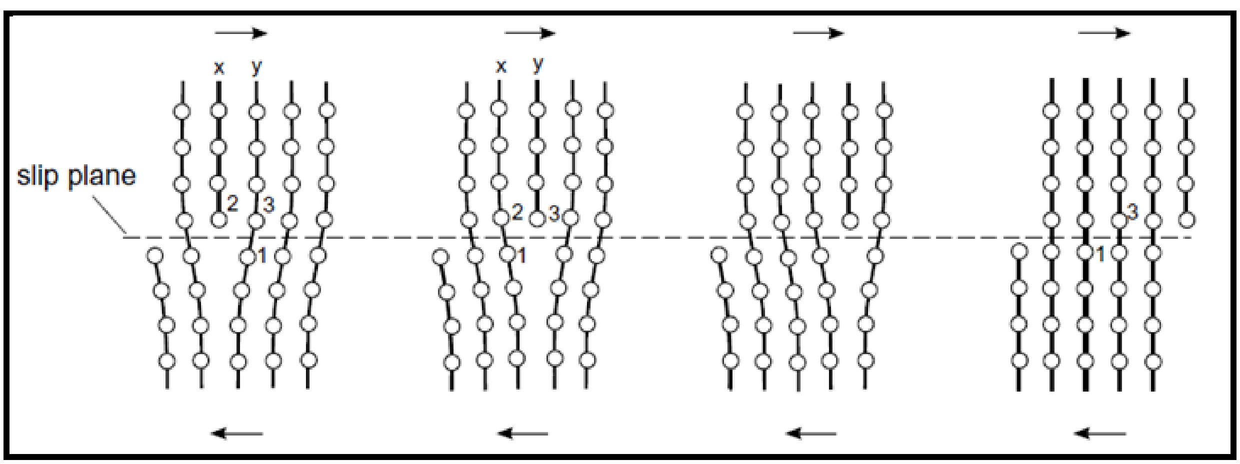


Figure 1.5 Schematic showing movement of an edge dislocation. Arrow represents applied shear stress [18].

Atoms of the crystal containing a dislocation are displaced from their perfect lattice sites and cause distortion. This distortion produces a stress field in the crystal around the dislocation. This is the reason for development of large residual internal stresses in the material which has high dislocation density.

CHAPTER 2

LITERATURE REVIEW

2.1 International status

Caballero *et al.* [5-6] designed a new high strength bainitic steel. The authors designed a carbide free bainitic steel which possessed impressive combination of strength and toughness. Alloy was designed using phase transformation theory by aiming maximum transformation to bainitic-ferrite. The alloy composition that was used in the study is shown in Table 2.1.

Table 2.1 Chemical composition of the designed alloys [6].

Alloy	C	Si	Mn	Cr	Mo	Ni	V
Mn	0.30	1.5	2.00	1.30	0.25	…	0.1
Ni1	0.30	1.5	…	1.44	0.25	3.5	0.1
Ni2	0.30	1.5	…	1.44	0.25	3.5	…

Every element had its specific purpose like silicon was used to suppress the formation of cementite precipitates, molybdenum was added to reduce the impurity embrittlement, and vanadium was used to restrict the grain growth at high temperature and so on. TTT and CCT diagram were plotted to obtain the mixed microstructure of bainite and austenite.

The alloys were prepared by casting followed by forging to get the desired thickness. Forged samples were homogenized at 1200 ℃ for 2 days and again forged down to 50 mm final thickness. These samples were then immediately hot pressed up to 25 mm thickness after austenization at 900 ℃ for 2 h. The overall processing route is given in Figure 2.1.

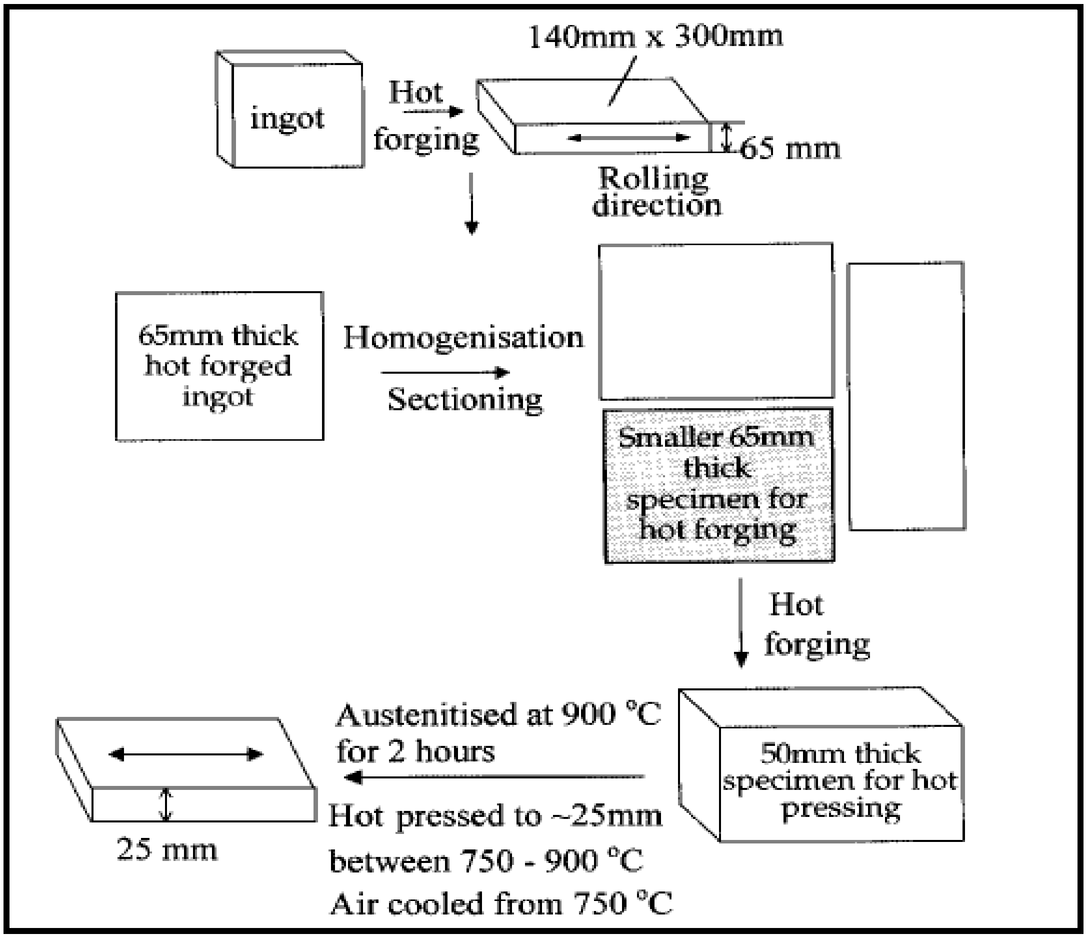


Figure 2.1 Processing route for the designed steel [6].

Quantitative X-ray analysis was done to determine the volume fraction of different phases. Optical, SEM, TEM characterization was carried out for further analysis. The quantitative study is shown in Table 2.2.

Table 2.2 Quantitative data of resultant microstructures [6].

Alloy	Volume fraction of bainitic ferrite V_B	Volume fraction of retained austenite V_γ	Volume fraction of martensite $V_{\alpha'}$	C content of austenite x_γ, wt-%	Hardness, HV30
Mn	0·26±0·01	0·07±0·01	0·67±0·02	0·55	597±2
Ni1	0·62±0·05	0·12±0·01	0·26±0·04	0·92	493±5
Ni2	0·81±0·06	0·11±0·01	0·08±0·05	1·03	536±6

From the studies it was confirmed that the size of bainitic-ferrite plates was near about 10 μm in length with 0.2 μm thickness which was the main reason behind the high strength of bainite.

Further tensile test and fracture toughness testing were carried out; results are shown in Table 2.3.

Table 2.3 Tensile and fracture toughness properties of designed alloy [6].

Alloy	YS, MPa	UTS, MPa	El., %	RA, %	K_{max}, MPa m$^{1/2}$	J_{max}, MPa m	K_{Jmax}, MPa m$^{1/2}$
Mn	1167	1790	13	44	...	...	...
Ni1	1150	1725	14	55	125	0·114	160
Ni2	1100	1625	14	59	128	0·134	174

Garcia *et al.* [7] investigated the possibility of isothermal formation of bainitic steels in a low temperature range. The aim was to process bainitic microstructure at low temperature for particular structural applications. The authors studied a Fe-Mn-Si steel and observed that bainite can be achieved by isothermal transformation at 400 K. The authors observed that this transformation takes place via a displacive transformation mechanism. The time taken for nucleation at this given low temperature can take many days, but such transformation leads to formation of enormously thin platelets of bainite. In the experiment, authors found the bainite-start (B_S) and martensite-start (M_S) temperatures as a function of composition of steel. The carbon concentrations of bainite and austenite were determined using standard X–ray analysis. It was said that the carbon may be trapped at defects. The bainite plate size was found to be exceptionally fine, amounting to tens of nanometers, especially at the lowest transformation temperatures. Stereological measurements showed a plate thickness of 32±3 nm for transformation at 200 °C and 49±4 nm at 250°C.

Bhadeshia [4] compared the properties of newly developed carbide-free bainitic steel with the conventional pearlitic rail steel (see Figure 2.2). The author studied the choreography of atoms during bainitic transformation in steels as the scale, extent of structure, and strength of steel directly depend on the movement of atoms. This information was used as the theory for formation of bainite in steels that can be used to process alloys for applications such as cover rails, protective armor. In this research, the author particularly worked on silicon-rich steels where upper bainite formed without any precipitation of cementite. Bainitic steels of this kind are economical. It required mainly sufficient silicon to suppress cementite but on the other hand large blocks of austenite between the sheaves of bainite-ferrite. Blocky austenite is quite unstable and transforms to high carbon, un-tempered martensite under the control of applied stress, thereby making the steel prone to embrittlement.

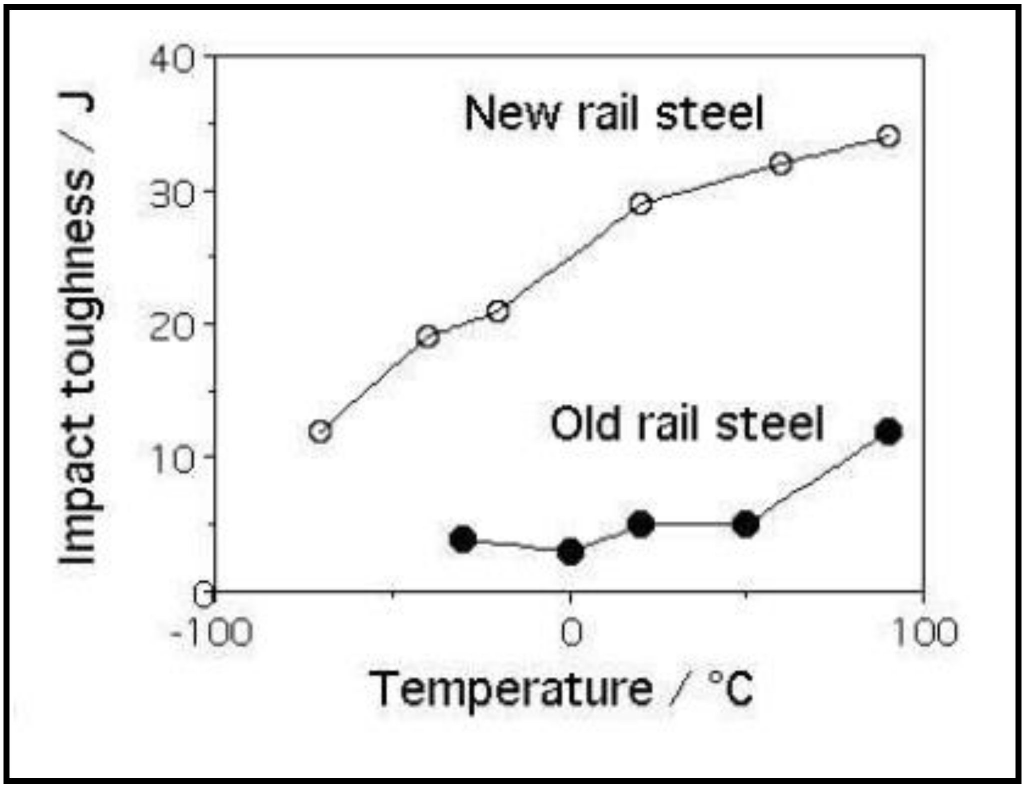


Figure 2.2 Toughness comparison between carbide-free bainitic rail steel (new rail steel) and ordinary pearlitic rail steel [4].

Mateo *et al.* [9] studied the mechanical properties of steel containing low-temperature transformed bainite. The microstructure consisted of bainite-ferrite and retained austenite. The bainitic-ferrite fraction, its carbon content, and the dislocation density increased as the transformation temperature decreased. There were two typical features of bainite obtained by transformation at low temperature. The first feature was the thin plates of ferrite without carbon, and the second was the large amount of carbon which remained intact inside bainitic-ferrite. The increase in obtained strength induced at lower temperature was because of thinner bainitic-ferrite plates. It was shown that strength of the low-temperature bainite was because of extremely fine plates of bainitic-ferrite. It was therefore anticipated that the strength of resulting steel and thickness of bainite-ferrite plates present in its microstructure varied reciprocal to each other. At elevated transformation temperature, the bainitic-ferrite volume fraction decreased as its thickness increased, leading to smaller contribution from both of these strengthening mechanisms. Toughness and ductility were mainly controlled by the fraction of retained austenite present in microstructure. Authors concluded that the slender plates of bainitic-ferrite in combination with high dislocation density in bainite-ferrite were the main strengthening mechanisms for the developed bainitic steel.

Hua *et al.* [22] investigated the microstructure and mechanical properties of low carbon high manganese steel (see Table 2.4). Tensile test was carried out followed by micro hardness testing. Microstructure was studied by optical and TEM microscopy. X-ray diffraction analysis was used to identify the phase structure. Authors found that No.1 steel had higher strength but No.2 steel had higher elongation, at the same deformation condition.

Table 2.4 Chemical composition of tested steels [22].

Steel	Mn	Al	Si	C	Fe
No. 1	23. 8	2. 7	3. 0	6×10^{-4}	Balance
No. 2	33. 0	2. 93	3. 0	6×10^{-4}	Balance

It was found that TRIP and TWIP effects co-existed in No.1 steel while only TWIP effect appeared in No.2 steel, at room temperature. This implied that TRIP effect could obviously increase the strength of steel but TWIP effect was favorable to increase the plasticity of material shown in Figure 2.3.

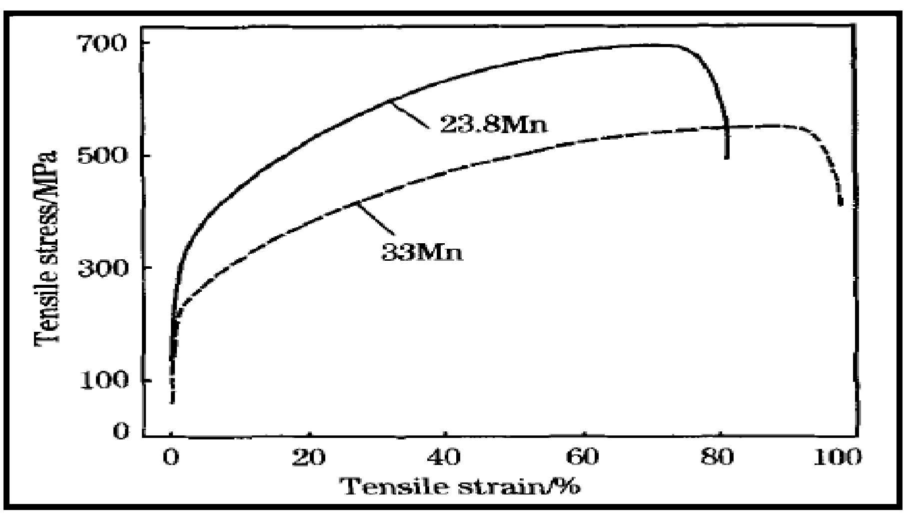


Figure 2.3 Engineering stress and strain curve [22]

Saedi *et al.* [23] produced different microstructures comprising of full bainite, bainite-ferrite, and martensite-ferrite in AISI 4340 steel to investigate their mechanical properties. All specimens were homogenized at 1000 °Cfor 2 h prior to heat treatment process. Different phases were obtained by different heat treatment routes (see Table 2.5).

Table 2.5 Different heat treatment cycles used to get different microstructures [23].

	Microstructure	Heat treatment
A	Full bainite (B)	Austenitizing at 850 °C (60 min), austempering at 300 °C (60 min).
B	Bainite–34% ferrite (BF)	Austenitizing at 850 °C (60 min), isothermal transformation at 700 °C (100 min), austempering at 300 °C (60 min), air cooling.
C	Martensite–34% ferrite (MF)	Austenitizing at 850 °C (60 min), isothermal transformation at 700 °C (100 min), oil-quenching.

Volume fraction of each phase was calculated by using image analyzer software. Mechanical testing (tensile and Charpy test) was done to study the properties of materials at room temperature and hardness values were measured under load of 150 kg. It was observed that bainite-ferrite microstructure provided greater ductility as compared to other microstructures. Hardness was nearly same for all the microstructures. Bainite and ferrite microstructure showed 46% more charpy impact energy than full bainite and 71% more than martensite and ferrite microstructure. Tensile tests showed that the elongation of bainite and ferrite microstructure was 9% and 41% more than the full bainite and martensite and ferrite microstructures respectively.

Zhou e*t al.* [24] designed two types of carbide free bainitic steels with different types of heat treatment processes viz. (a) austempering and (b) continuous cooling process, to study the effect of Cr on bainitic transformation, final microstructure, and properties of low carbon bainitic steel. The composition of steels is shown in Table 2.6. The steels were developed and cast in the form of 50 kg ingots using vacuum furnace followed by hot-rolling and then air-cooling to room temperature.

Table 2.6 Chemical composition of the designed steels [24].

Steels	C	Si	Mn	Cr	Mo	N	P	S
Cr-free	0.218	1.831	2.021	/	0.227	<0.003	<0.006	<0.003
Cr-added	0.221	1.792	1.983	1.002	0.229	<0.003	<0.006	<0.003

Heat treatment processes were carried out on a Gleeble 3500 simulator. Two different kinds of thermal treatment were planned for the two steels, i.e. austempering and continuous cooling as shown in Figure 2.4.

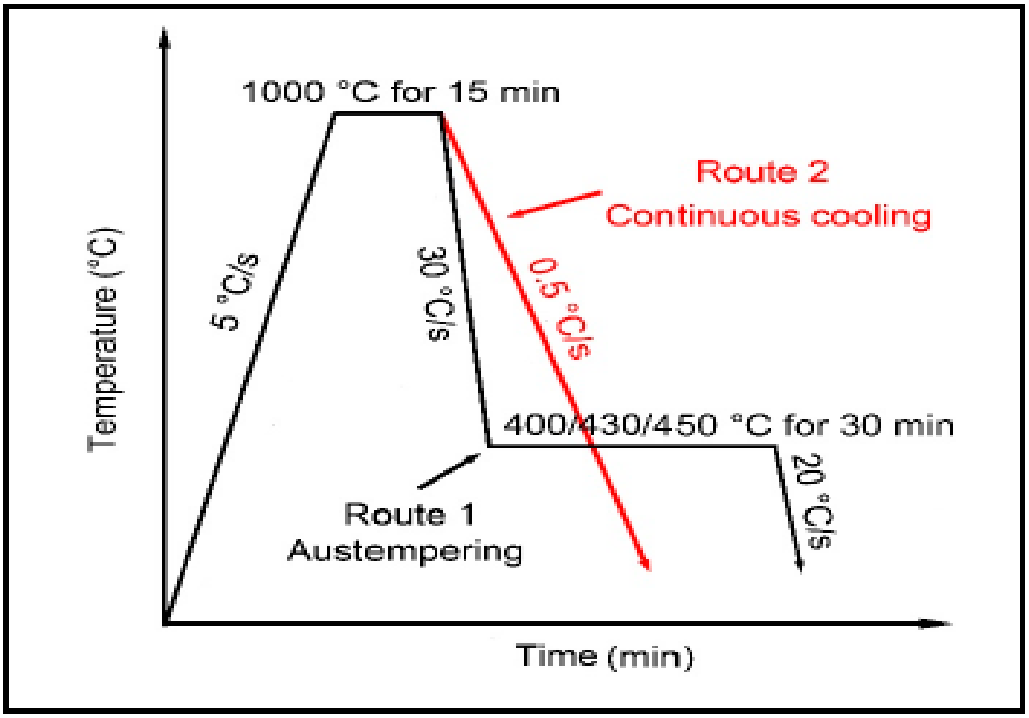


Figure 2.4 Heat treatment route for different steels [24].

Various investigations were carried out through SEM microscopy, X-ray diffraction etc. Tensile testing was carried out to evaluate the mechanical properties of designed steels. The mechanical properties obtained are shown in Table 2.7 and Figure 2.5 shows the stress-strain curve respectively.

Table 2.7 Tensile test results of designed steels [24].

Steel	YS (MPa)	TS (MPa)	TE (%)	PSE (GPa%)
Cr-free	662 ± 13	1054 ± 15	13.2 ± 0.8	13.9 ± 0.56
Cr-added	812 ± 15	1145 ± 21	6.9 ± 0.2	7.9 ± 0.15

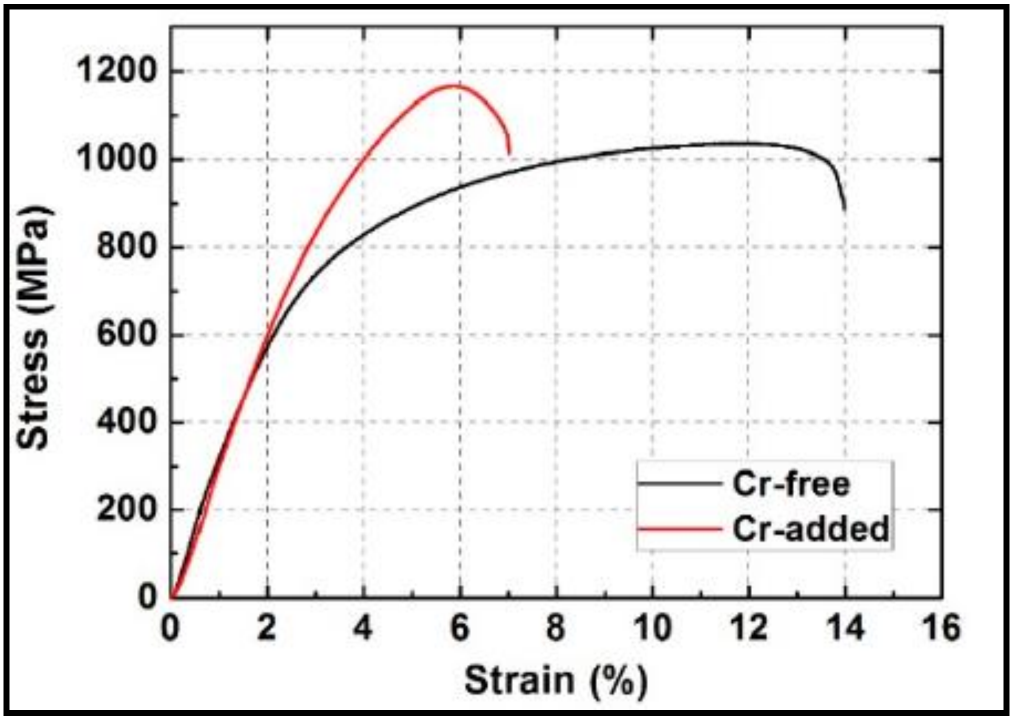


Figure 2.5 Stress-strain curve of designed steels [24].

It was observed that chromium addition hindered the isothermal bainitic transformation kinetics due to the decline in driving force for nucleation and growth of bainite; also it was noticed that Cr addition increased the strength and elongation for austempering process concurrently at lower temperature range. For continuous cooling thermal process, it was observed that the amount of RA decreased with increase in yield strength, but the total elongation got decreased in Cr added steel because of large amounts of martensite.

Hu *et al*. [25] studied the effect of cobalt and aluminum addition to steel on bainitic transformation. Quenched and tempered martensitic steel had many drawbacks like responsive to crack propagation during quenching, high energy consuming in manufacturing etc. To overcome these drawbacks, carbide free bainitic steels were developed. The novel high strength-high toughness carbide free bainitic steels were designed and microstructure of nano-size bainitic-ferrite and retained austenite was obtained by low temperature transformation. This transformation can be increased by increasing the free energy change between austenite and ferrite phases. In the light of this, composition of alloy was modified by addition of cobalt and aluminum. It increased the dynamic force at constant temperature which resulted in refinement of microstructure in two ways (a) by promoting formation of greater quantity of bainitic-ferrite by eliminating islands of retained austenite, and (b) by refining the size of bainitic-ferrite plates which increases the density of thin plates and overall it shifts the TTT diagram to the left side.

Sarizam [26] discussed the influence of holding temperature on the mechanism of bainitic transformation in steels. Specimens of 5 mm diameter with 1 mm thickness were prepared for the said purpose. Specimens were austenized at 1350 ℃ for 60 s before isothermal holding. Isothermal holding at temperatures 560 ℃ and 580 ℃ for 3600 s was done and the behavior of bainitic transformation was studied with the help of laser scanning confocal microscopy (LSCM) and the heat treated specimens were analyzed by electron backscattered diffraction (EBSD). From the result and discussion of this research, the authors concluded that transformation temperature shows a linear relationship with average block size. Higher transformation temperature resulted in larger value of average block size. Thus, finer microstructure of bainitic-ferrite can be obtained at lower transformation temperatures.

Hu *et al.* [27] developed a method to stabilize and refine the retained austenite present in bainitic steels. The authors stated that carbon enriched retained austenite generally forms during low temperature bainitic transformation in the range of 200–300 ℃. Generally, there are two types of retained austenite (based on size) formed during transformation. RA with size in excess of 1 µm is called blocky austenite and that with size less than 1 µm is called fine retained austenite. Presence of blocky austenite is more prone to transformation to martensite under the external applied load. So elimination of this type of austenite is very important in order to get good mechanical properties by presence of bainite. In their research work, the authors developed two different methods to get fine retained austenite by eliminating the blocky one. The methods included (a) two stage bainitic transformation (B+B), and (b) bainitic transformation followed by partitioning process and then water quenching to room temperature (B+P+Q). Schematic diagram for both the processes is shown in Figure 2.6.

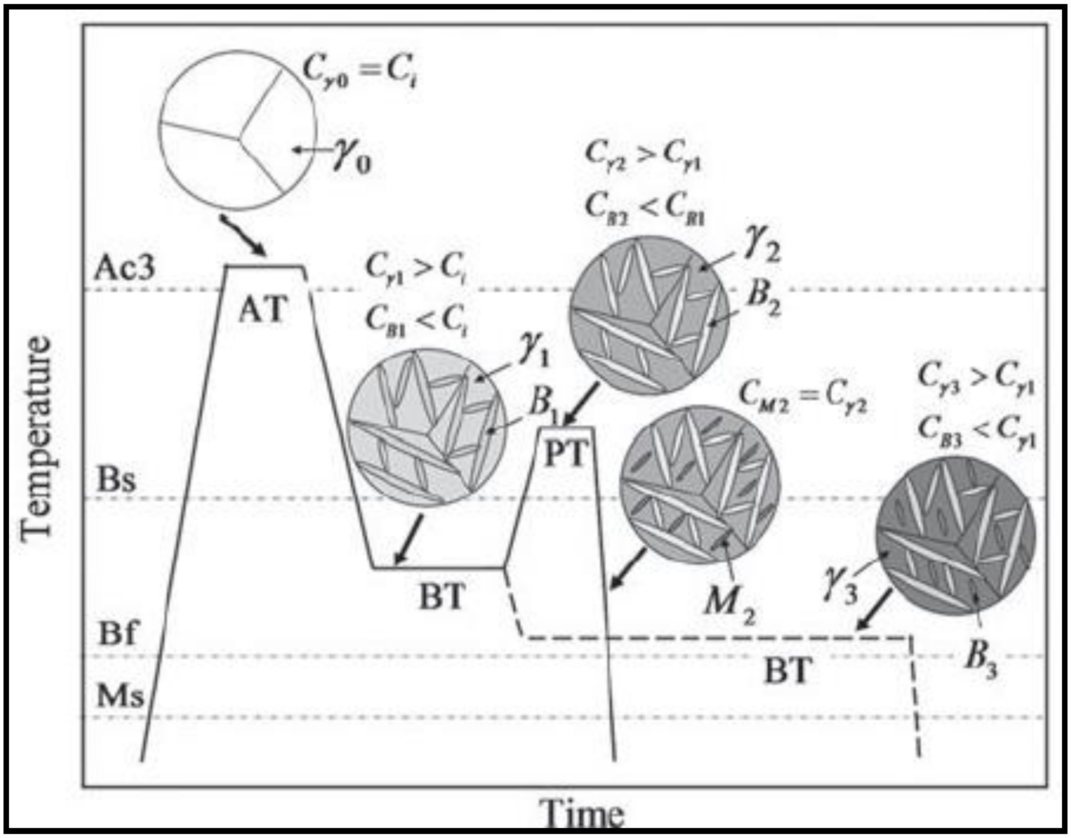


Figure 2.6 Schematic diagram of (B+B) and (B+P+Q) process [27].

It was shown that refinement of RA was done due to formation of ultra-fine bainite in both the processes. The level of RA refinement was found in both the processes but it was concluded that (B+P+Q) approach took much shorter cycle time as compared to the (B+B) process.

Grajcar [28] determined the influence of isothermal holding on the bainitic transformation and also on the mechanical stability of retained austenite. Medium carbon steel was taken for the experimental examination. From the austenization temperature, samples were quenched to a temperature range of 250 to 550 ℃ with a holding time of 600 and 1800 s. Figure 2.7 shows the various heat treatment cycles opted for in the current research.

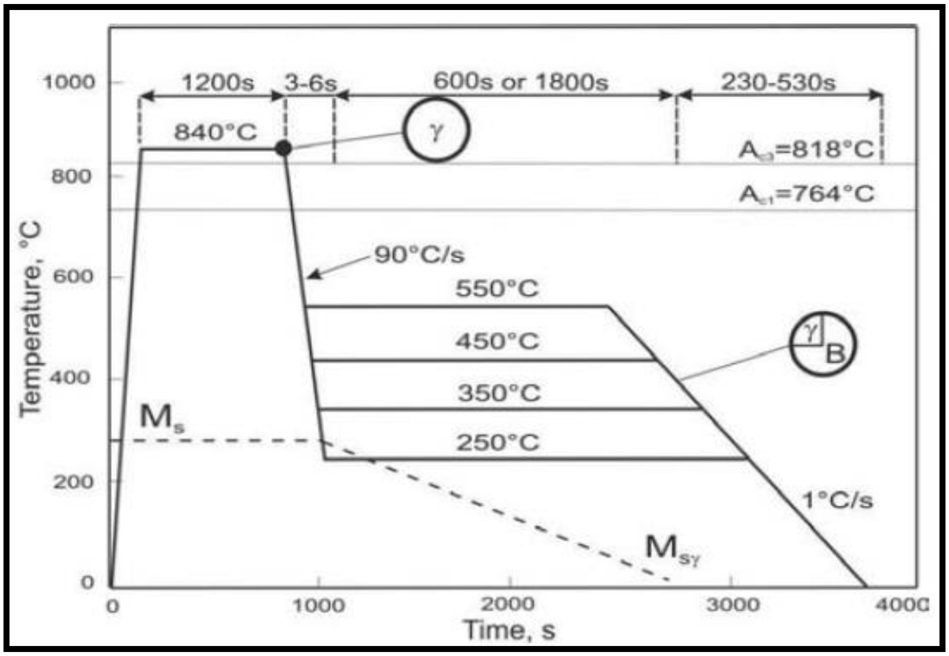


Figure 2.7 Heat treatment of investigated steel [28].

19% volume fraction was obtained after isothermal quenching at 250℃ with a matrix of bainitic-ferrite. It was observed that by increasing the isothermal holding temperature to 350 ℃ or holding time to 1800 s, lower bainite forms due to initiation of carbide precipitation. On further increment in temperature up to 550 ℃ resulted in the change in morphology of the matrix from lower to upper bainite with a drop of retained austenite stability. Regular size of retained austenite grains was obtained as 3 µm whereas maximum austenite phase was there in the form of thin films in between the sheaves of bainite.

Shen *et al.* [29] examined the influence of various factors related to retained austenite like volume fraction, morphology, carbon content on the strength and ductility of nano-structured TRIP steel. The authors examined eight heat treatment processes in order to get the final desired microstructure shown in Figure 2.8a-b. Seven of these heat treatment processes included isothermal bainitic holding whereas in one process, sample was just air cooled to room temperature after austenization.

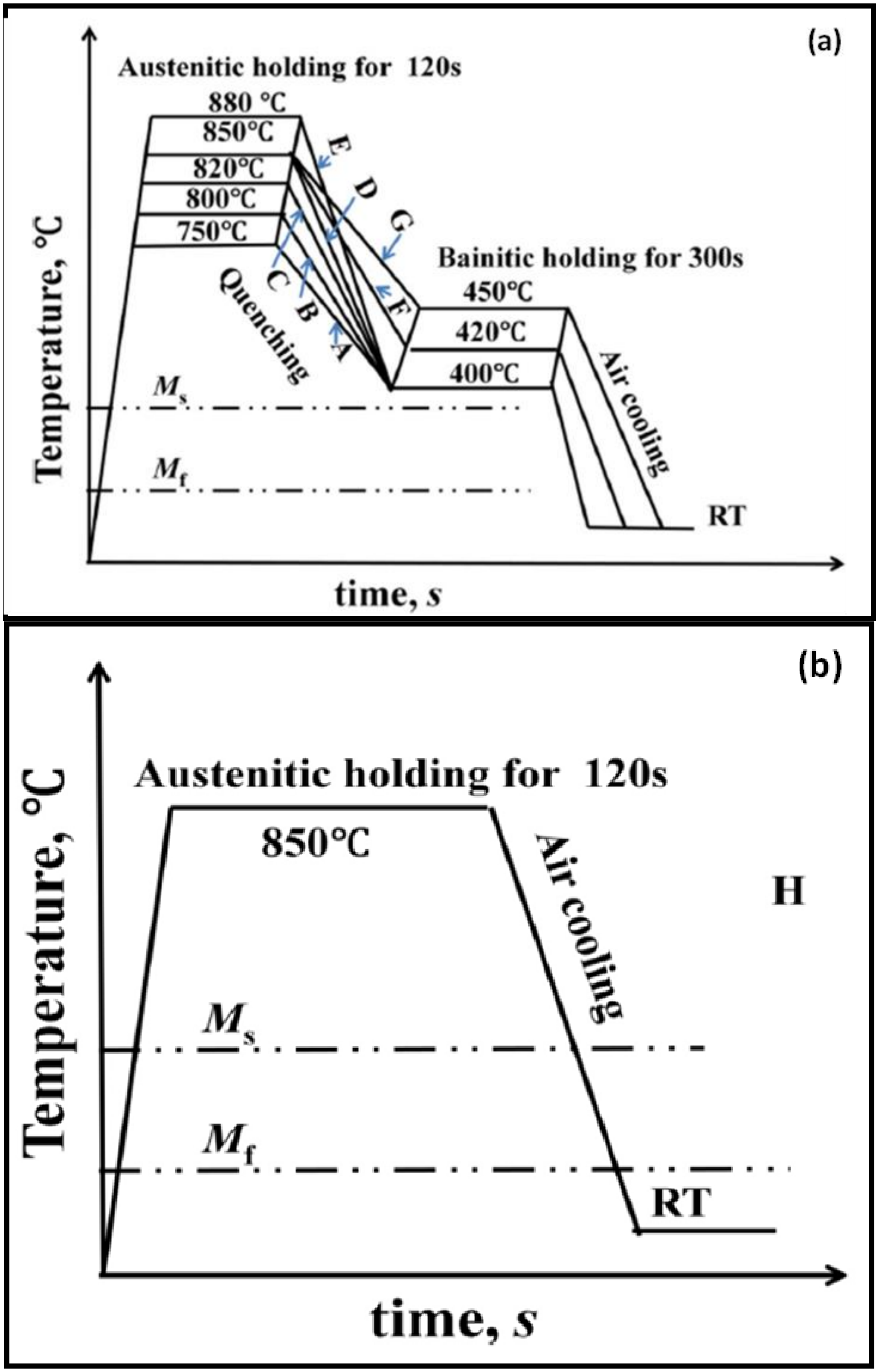
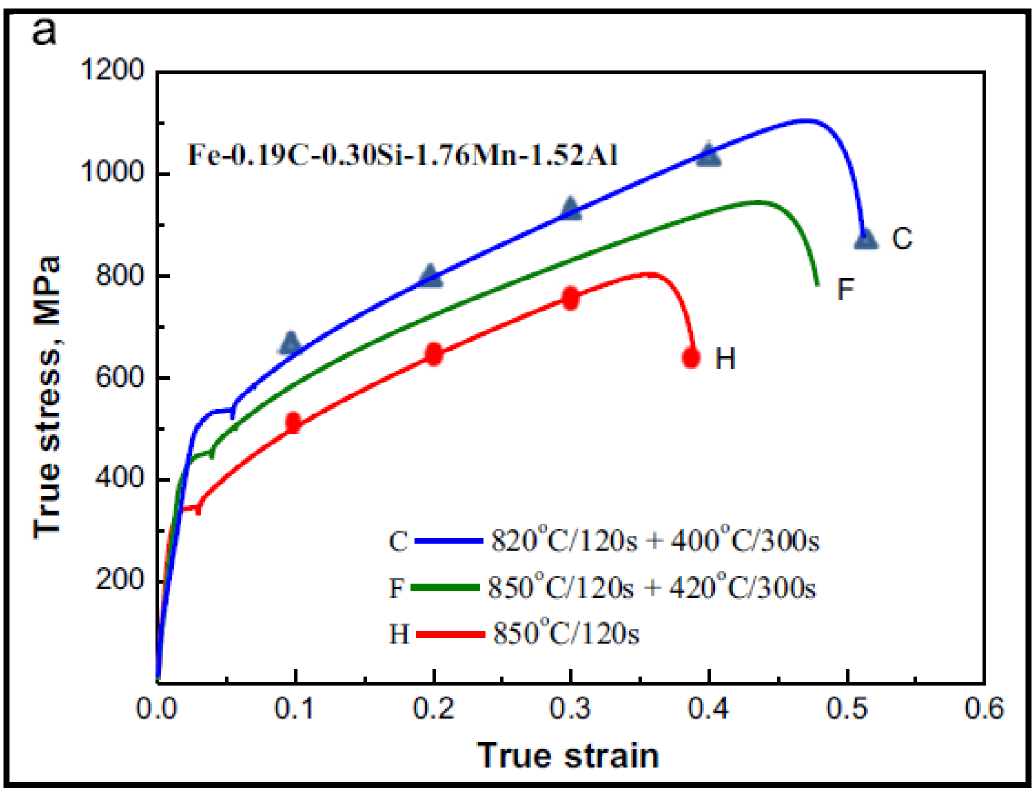


Figure 2.8 Various heat treatment processes used (a) process A-G (b) process H [29].

Further optical, XRD, SEM, EBSD, TEM and magnetic measurements were done to analyze the final microstructure. It was observed that with increase in annealing temperature, volume fraction of RA increased monotonically, because of low fraction of primary ferrite resulting from the higher austenitizing temperature. It was noticed that difference in carbon concentration within austenite affects its thermal stability. It was also observed that the usual carbon content of RA somewhat decreased with the overall increment in volume fraction of RA. It was quite possible that RA with maximum carbon concentration (steel C) ought to possess the highest stability which enabled lower and more gradual martensite transformation during deformation resulting in best combination of strength and ductility. On the other side, some RA grains with high carbon content might not be transformed into martensite during deformation. This confirmed the fact that increase in carbon content decreases the starting point of martensitic transformation and provides thermal stability to RA. Figure 2.9a-b shows the graphical representation of mechanical properties of the designed steel.



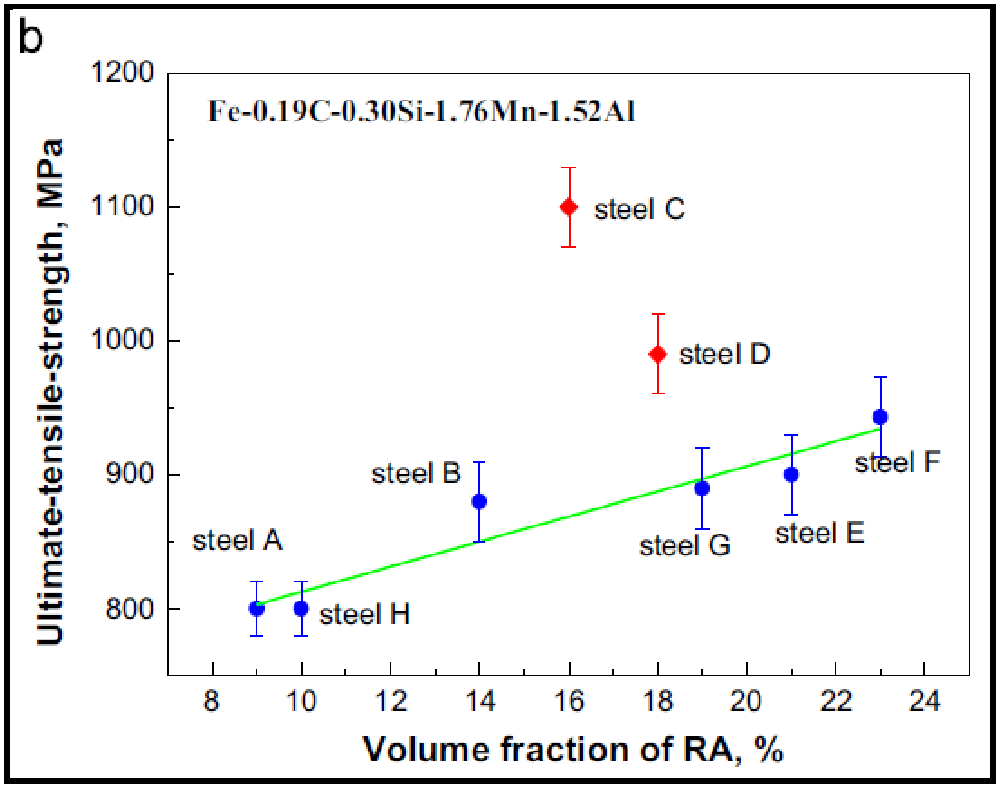


Figure 2.9 (a) True stress-true strain curve for designed steel (b) ultimate tensile strength vs volume fraction of RA [29].

The following main conclusions were drawn from the above work:

a) Both strength and ductility of TRIP-assisted steels can be enhanced by altering the heat treatment processes, which leads to design of ultrafine-lamellar RA with high mechanical stability against straining.

b) The morphology and carbon content of austenite decides its stability.

c) By controlling the morphology, distribution, and stability of RA, mechanical properties of TRIP-assisted steels can be improved to achieve high strength and high ductility simultaneously.

2.2 National status

Jha *et al.* [30] studied the stability of retained austenite and conducted its microstructure analysis against various thermal conditions. The existence of retained austenite as a supplementary phase in dual phase steels (usually a ferrite-martensite mixture) is favorable as it improves toughness of the steel because of transformation induced plasticity (TRIP) effect. Retained austenite content in the steel can be increased by opting for a suitable heat treatment process. Five temperatures in the range of 100–500 ℃ with a difference of 100 ℃ were chosen for subsequent tempering process. The holding time was maintained as 3600 s at all the temperatures. The volume fraction of retained austenite as an outcome of tempering at different temperatures was examined by X-ray diffractometer. The main conclusion by the authors was that the shape and size of retained austenite governs its decomposition behavior.

Nanda *et al.* [3] reviewed the different processing and mechanical properties of various advanced high strength steels (AHSS).Microstructures and mechanical properties achieved under various processing circumstances were discussed. A detailed study on TWIP mechanism was provided in the review. TWIP steel grades justify their position in second generation of AHSS because their microstructure consists of significant amount of retained austenite. These types of steels show exceptional combination of strength along with ductility possessing tensile strength near about 1000 MPa with ductility in the range of 50%. TWIP steels usually consist of iron (Fe), manganese (Mn), or nickel (Ni, 15–35%), silicon (Si, 1– 3%), and aluminum (Al, 1–3%). Ni, Mn are mostly used as austenite stabilizers which can retain the austenite even at room temperature. The authors concluded that in TWIP steels, strength increases due to twin boundaries. Fine grain size and high volume fraction of twins can be achieved at high deformation temperature. Authors concluded that finer grain size and increased twinning fraction in the microstructure improves the ductility and strength. Figure 2.10 shows the different types of twins.

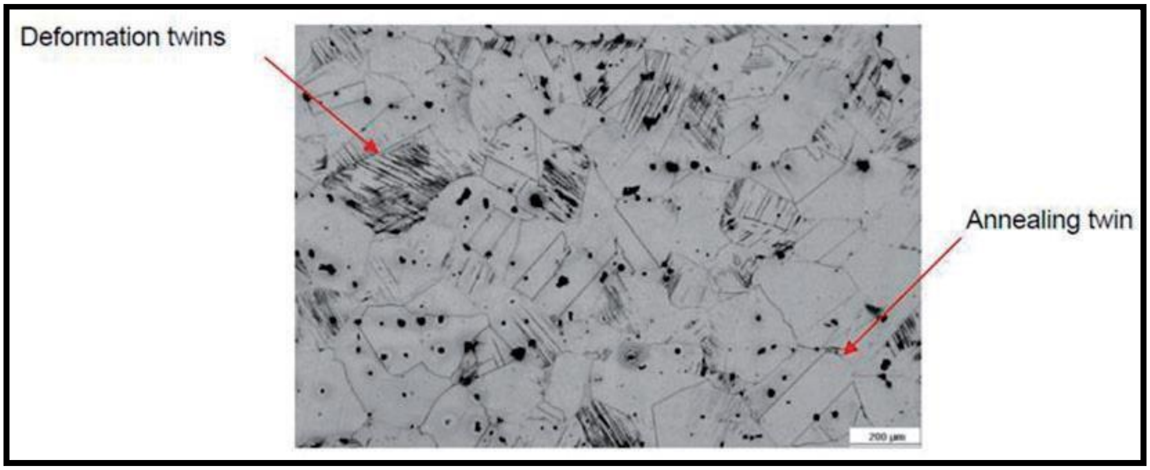


Figure 2.10 TWIP steel microstructure shows the deformed and annealed twins [3].

Nationwide very limited work has being done in this area. It's a new concept and the present dissertation work is step in this new direction.

2.3 Summary of the existing literature

The work done by various authors can be summarized as follows:

1. Researchers have developed bainitic grade steels with different alloy steel compositions and processing parameters to obtain desired properties having extremely high strength but limited ductility [5, 6, 22]. Further, Bhadeshia [4] was able to develop a carbide-free bainitic steel with improved toughness.

2. A few authors have studied the TRIP and TWIP effects in low carbon bainitic steels. The authors concluded that TRIP effect enhanced the strength of the steel while TWIP effect enhanced the ductility of steel [3, 22].

3. Authors have discussed the effect of thickness of bainite-ferrite plates and stability of retained austenite on the strength of bainitic steels. The authors reported that at low transformation temperatures, thinner bainitic plates are obtained. The ferrite plate thickness varies linearly with the reciprocal of the strength of bainitic steel. Further, the shape and size of retained austenite governs its stability and hence the decomposition behavior. The authors stated that stability of retained austenite is dependent on various factors including composition of steel, orientation of the phases etc. [9, 30].

2.4 Limitations of the existing literature

1. Authors have worked to develop bainitic steels with different chemical compositions and various heat treatment processing routes. In most of the studies, the toughness values have been achieved because of very high strength values obtained. However, limited ductility has been a concern in these developed steels resulting in poor formability. Very limited literature is available where authors have developed bainitic grades with good combination of high strength-high ductility. There is a necessity to develop high toughness bainitic steels with improved ductility and hence improved formability.

2. With regards to retained austenite present in the steel, authors have mainly transformed this phase into martensite via TRIP effect to obtain enhanced strength in bainitic steels. There is a lack of literature reporting on increasing the stability of retained austenite so that the same does not transform to martensite but rather suffers twinning to provide enhanced ductility in bainitic steels (i.e. TWIP effect).

CHAPTER 3
DESIGN OF THE STUDY

3.1 General

This chapter presents the design and structure of the present dissertation work. It covers the objective of research work, the key issues, the research methodology and details of experimental set-up etc. It also provides brief details of various machines/equipment and software used in the research work.

3.2 Objective of present research

Bainite phase-mixture is obtained by transformation of austenite in the bainitic transformation temperature range. Every steel composition has its own typical 'C' type curve with well-defined B_s and B_f temperatures. Bainite phase mixture consists of non-lamellar lath-shaped fine aggregates of bainitic-ferrite plates (called bainitic-ferrite) with some cementite/carbide particles [1, 11]. Depending on carbide precipitation, bainite forms in two different ways and is referred to as (a) upper bainite, and (b) lower bainite. Upper bainite forms at a relatively higher transformation temperature range and the cementite/carbide particles are present in-between the sheaves of ferrite plates (bainitic-ferrite) in the resulting bainitic structure. Upper bainite appears like feathers (of a bird) and hence is known as feathery bainite. Lower bainite (acicular bainite) forms at relatively lower transformation temperature range and the cementite/carbide particles are present within the bainite-ferrite plates due to low carbon diffusibility [1, 11, 31].

Fine (sized) plates of bainitic-ferrite result in high strength and make the bainitic steel useful for many applications like armour steel, rail steel etc. However, presence of carbide precipitates in bainite reduces the elongation capacity of the material leading to poor ductility. This restricts further applications of bainitic steels [4, 7, 25]. The formation/precipitation of cementite/carbide particles during bainitic transformation (which results in lower ductility, as discussed above) can be reduced/avoided by adding some alloying elements [4, 5, 9] and/or making suitable changes in the thermo-mechanical processing route used to process bainitic steels [24, 28]. Literature reports that addition of alloying elements like Si, Al, Co, Mo, Cr etc. may be useful in achieving this goal. Silicon addition to steel composition reduces/eliminates formation of carbide precipitation during bainite formation, and the resulting steel shows high resistance to cleavage fracture/void formation because of absence of fine carbides. Further, the carbon rejected by bainitic-ferrite plates as a result of silicon addition (i.e. carbon which otherwise would have formed cementite/carbides) also enhances the stability of retained austenite formed in bainitic steels. Si addition also helps in ferrite strengthening [4, 5, 6]. Al addition helps in regulating the stacking fault energy of the material and results in formation of stable (non-blocky type) retained austenite in the final microstructure [26]. Cobalt (Co) addition increases the rate of reaction for bainitic transformation. Co and Al play a combined role in refining the steel microstructure by reducing the thickness of bainite-ferrite and also increasing the volume fraction of bainite [4, 5, 6, 7, 9, 24, 25].

With regards to heat treatment, literature reports that researchers have used different thermo-mechanical processing routes to obtain bainite steels [5, 6, 24]. It is observed from literature review that the austenization temperature (AT: 800–1200℃) and deformation temperature (DT: 750–900℃) values used by various authors can be suitably modified to a slightly higher range. Further, with regards to transformation temperature (TT) for bainite formation, authors have used a wide range of temperatures (TT: 250–550 ℃). Bainite transformation temperature can be judiciously chosen for the formation of bainite with thin bainite-ferrite plates to obtain superior properties [5, 6, 24, 28, 32]. With relatively lower austenization and deformation temperatures (as are used in literature), the transformation temperature difference for bainitic-ferrite formation is low. As a result, the size of bainite-ferrite gets enhanced leading to formation of lesser number of bainite-ferrite plates (i.e. as a result, fine plates of bainite-ferrite are less). So, to obtain bainitic microstructure with fine bainite-ferrite plates and stable retained austenite, the AT, DT, and TT need to be suitably modified. In bainitic steel microstructure, presence of fine bainitic-ferrite plates provide high strength whereas inter-weaved thin films of high carbon retained austenite control the ductility [5, 6, 24, 25, 31]. Bainitic transformation is a displacive and diffusionless transformation in nature which provides unique features to the resulting microstructure including high dislocation density, heterogeneous distribution of carbon in retained austenite, and nano twins. High volume fraction of fine plates of bainitic-ferrite in combination with high dislocation density improves, both the strength as well as overall elongation of the steel [31, 33]. Further, it is observed from literature review (of bainitic steel processing) that researchers have utilized combinations of steel chemistry-processing route resulting in incomplete transformation of austenite to bainite [5, 6, 23]. This incomplete transformation to bainite results in presence of other phases like martensite, retained austenite etc. in the final microstructure of bainitic steels. Presence of martensite improves the strength but is very detrimental to overall ductility of the material [5, 6]. In the light of aforesaid, there is scope of improving the strength-ductility combination of bainitic steels by making suitable modifications to steel chemistry and thermo-mechanical processing routes generally used for processing of bainitic steels.

Thus, the main objective was to develop bainitic steel with high strength and high ductility by (a) obtaining maximum volume fraction of bainite phase mixture in the microstructure (by reducing fraction of non-bainitic phases), (b) reducing the cementite/carbide content in bainite, and (c) obtaining bainitic-ferrite as thin-plates. To achieve this objective, the steel chemistry-heat treatment combination was very carefully designed. The thermo-mechanical processing comprised of hot forging and hot rolling (mechanical portion of processing) followed by austempering (heat treatment or thermo- portion of thermo-mechanical processing).

Thus, the main objective of the present research work was ***to develop bainitic steel with high strength-ductility combination through thermo-mechanical processing***.

3.3 Materials and methods

The details pertaining to the starting material and the experimental procedure followed in the present work are described in this section.

3.3.1 Starting material

The starting material was an as-cast alloy received in the form of a cylindrical ingot (diameter: 60 mm, length: 185 mm) as shown in Figure 3.1.

Figure 3.1 Starting material received in the form of as-cast alloy.

It was developed as a 15 kg induction melt at CSIR-NML, Jamshedpur. The alloy chemistry of this starting material included C, Mn, Si, Ni, Co, Cr, Cu, Mo, V, and Al (composition details available in Indian Patent No. 201831011864 filed on 29/03/2018).

3.3.2 Software used

Software used in the present work is discussed as follows:

A. JMat-Pro

JMat-Pro software (JMat-Pro 10.2; developed by Sente Software Ltd, Guildford U.K) was used to develop phase fraction diagram, CCT diagram, and TTT diagram for the given steel chemistry. These diagrams were used to obtain information with regards to upper and lower critical temperatures and transformation temperatures for the given steel.

B. ImageJ

It is a public domain Java image processing program (developed by NIH, MacintoshInc.USA). It can analyze, edit, display, save and print 8-bit, 16-bit, and 32-bit images. It can read several image formats like TIFF, GIF, JPEG, BMP etc. Quantitative analysis for determining the phase fraction of individual phases present in the microstructure of processed steels was done using this package.

C. ORIGIN 8.0

Origin (developed by ORIGINLabs: Northampton, Massachusetts, USA) is a data analysis and graphing software. It offers advanced analysis tools and applications for peak fitting, surface fitting, statistics, and signal processing. In the present work, the software was used for plotting graphs of the extracted data from tensile testing etc.

3.3.3 Materials processing

The experimental procedure in the present research work mainly comprised of thermo-mechanical processing of the as-cast alloy, followed by tensile testing, and characterization.

The as-received alloy was subjected to hot deformation (comprising of hot forging and hot rolling) along with heat treatment steps. The sequence of steps followed during the thermo-mechanical processing of the material is described in Figure 3.2. The details of these steps are described in the following sections.

The as-cast cylindrical ingot (d = 60 mm, l = 185 mm) was cut to obtain a rectangular section of 113×52×24 (all dimensions in mm). The casted/cut material (113×52×24, all in mm) was now subjected to the following sequence of steps.

A. Austenization: For austenization, the steel specimen (113×52×24, all in mm) was heated to a temperature (AT=1300 ℃) significantly above its upper critical temperature (A_{C3}= 830 ℃) in a muffle furnace. To determine the critical temperatures, JMat-Pro software was used to develop TTT, CCT, and phase fraction diagrams for the given steel composition. The steel specimen was soaked at this temperature (AT=1300 ℃) for a period of 3 h [5, 6, 7]. This high temperature heating and subsequent soaking of the material prior to subjecting it to hot working processes (forging and rolling) was performed with the purpose to (a) obtain uniform temperature throughout the cross-section of the material, (b) homogenize the chemical composition throughout the material (to eliminate any cored/inter-dendritic segregated zones), and (c) obtain austenitic microstructure in the steel which is most favorable for hot working of steels [1, 5, 6, 7, 24, 25, 26, 32].

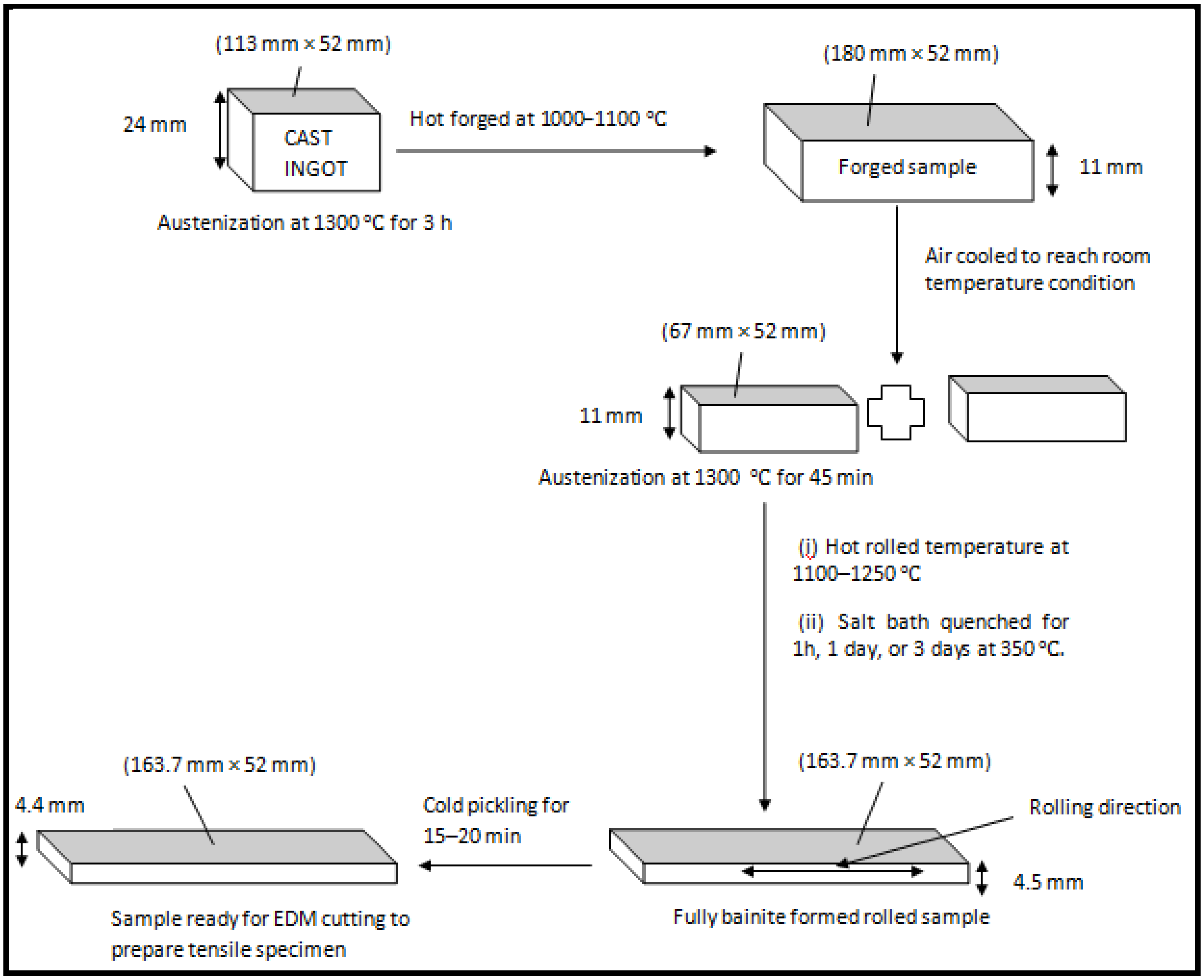


Figure 3.2 Thermo-mechanical processing route used in the present research for developing bainitic steel.

B. Hot forging: After austenization, the steel specimen was subjected to hot forging. The purpose was to (a) obtain equi-axed and refined grains, (b) remove anisotropy and defects like porosity etc., and (c) obtain desired dimensions for hot rolling[1, 2].For forging, the specimen after austenization (1300 °C, 3 h) was immediately transferred to the forging press. The set-up used for forging contained a forging hammer of 0.5T capacity. During forging, the temperature of the specimen was in the range of 1000–1100°C. Forging was continued to obtain final dimensions of 180×52×11 (all in mm) in the specimen. After the required dimensions were obtained, the specimen was allowed to cool in air to reach room temperature conditions. Figure 3.3 shows the steel specimen before and after being subjected to forging.

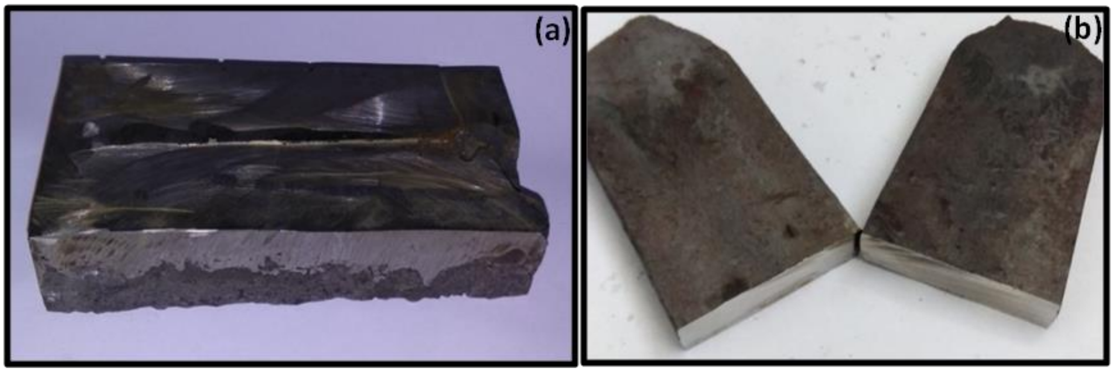


Figure 3.3 Actual photos of the steel specimen (a) before forging, and (b) after forging.

C. Hot forging was followed by hot rolling. Hot rolling was done to achieve the final thickness in the specimen as was required per ASTM E8 standard for the tensile test. Hot rolling also incorporated high dislocation density during deformation [1, 2].

For rolling, the forged specimen (180×52×11, all in mm) was allowed to cool down to room temperature under ambient conditions. The forged specimen was cut into two specimens, each of size 67×52×11 (all in mm). One of these specimens was used and it was subjected to hot rolling under plain strain conditions.

For hot rolling, the specimen was again subjected to austenization at 1300 ℃ for 45 min [6, 7, 24].After austenization (1300 ℃, 45 min), the specimen was immediately transferred to the hot rolling mill. During hot rolling, the temperature of the specimen was in the range of 1100–1250 ℃. Hot rolling was performed in 05 passes with roller speed of 27 rpm to obtain final dimensions of 163.7×52×4.5(all in mm) in the specimen. After the required dimensions were obtained, the specimen was suddenly quenched to 350 ℃ using a salt bath tub. The details of salt bath quenching are provided in the next section. The specimen, before and after hot rolling, is shown in Figure 3.4.

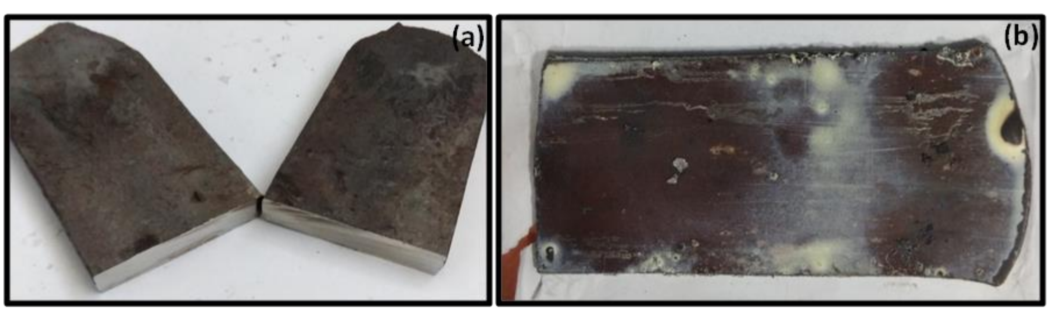


Figure 3.4 Actual photos of the steel specimen (a) before rolling, and (b) after rolling.

D. Salt bath quenching: During hot rolling, after the required dimensions were obtained (4.5 mm thickness), the specimen was quenched to bainitic transformation temperature using a salt bath comprising of 50% $NaNO_3$ and 50% KNO_3 solution for austempering. This salt bath was maintained at a temperature at which bainitic transformation was desired. In the present work, the B_S and B_f temperatures were predicted as 450 ℃ and 330 ℃ respectively. For bainitic transformation, isothermal transformation temperature of 350 ℃ was selected. Thus, for the present work, bainitic transformation temperature for austempering was considered as 350 ℃ and salt bath was maintained at this temperature. The specimen was held at the bainitic transformation temperature of 350 ℃for three different soaking periods of (i) 1 h, (ii) 1 day, and (iii) 3 days [7, 24, 28]. After holding the specimen in the salt bath solution for the stipulated time period (1h/1 day/or 3 days), the specimen was taken out of the salt bath and air cooled to room temperature. It was expected that in each case, the desired bainitic microstructure would have formed in the treated steel specimens.

E. Pickling: Salt bath quenching resulted in formation of salt scaling on the surface of thermo-mechanically processed specimens. The scales on various specimens were removed by pickling. For this, the specimens were immersed in a container with pickling solution (comprising of aqueous solution containing 50% HCl) for 15–20 min. After picking, there was a small decrease in the value of specimen thickness (decreased from 4.5 mm to ~ 4.4 mm).

F. Preparation of tensile samples: After complete removal of scales from the surface of thermo-mechanically processed specimens, tensile samples were prepared using wire EDM.

3.3.4 Tensile testing of heat treated specimens

The processed bainitic steel specimens were machined using wire EDM process to prepare tensile specimens (ASTM standard E8/E8M; gauge length of tensile specimens = 25 mm).Testing was done on Instron 8862 system (capacity: 100 kN; cross-head velocity: 1mm/min). The tensile specimen (l = 25 mm, t = 4.4 mm, b = 5.92 mm) used in the present research is shown in Figure 3.5.

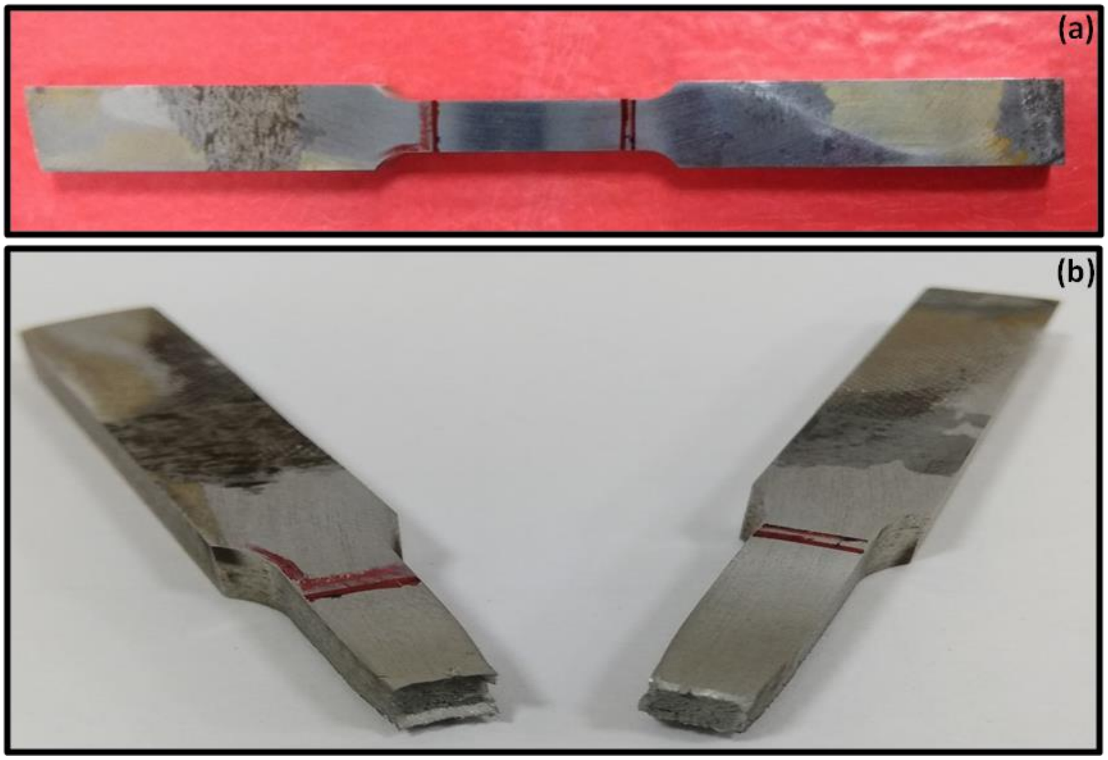


Figure 3.5 Tensile specimen used in the present research shown (a) before testing (b) after testing.

3.4 Sample preparation for metallography

The main steps are discussed in brief as follows:

3.4.1 Cutting

Cutting of samples for mounting was done by automatic precision cutting machine (Make: Mecatome T210, Japan). Sample was fixed in between the two jaws in which one was fixed and the other was movable as per requirement of the sample size. Speed of 2500 rpm with a feed rate of 0.01 mm/s was used during the cutting of sample. The cutting machine used in the present work is shown in Figure 3.6.

Figure 3.6 Precision cutting machine (Courtesy: Tata Steels, Jamshedpur).

3.4.2 Mounting

The use of mounting of samples was to facilitate their easy handling. Mounting was done by non-conductive mould powder PHENOLIC (green hot epoxy resin).Specimens were hot mounted at a temperature of 170 ℃ for 300 s heating time as well as 300 s cooling time at a pressure of 900 daN. The edges of the mounted samples were rounded to minimize the damage to grinding/polishing discs. The mounting press (Make: PRESI.SA-11, Mecapress 3, France)was used in the present work is shown in Figure 3.7.

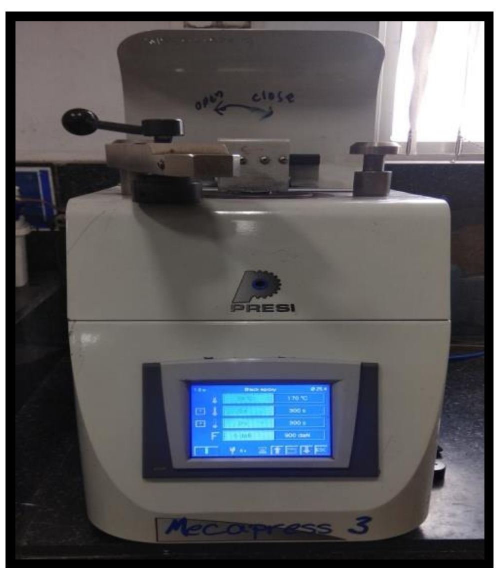


Figure 3.7 Mounting machine (Courtesy: Tata Steels, Jamshedpur).

3.4.3 Grinding

The surface to be examined by microscope was polished on a rotating wheel containing SiC abrasive papers of successive finer grades such as 120, 180, 320, 600, 1000 and 1200. The sample was grinded to eliminate the scratches and pits from the surface. Pits have to be eliminated by the paper abrasive, otherwise it is difficult to eliminate the pits during cloth polishing. Over-heating of sample was avoided by use of sufficient water coolant throughout the grinding so that the final microstructure does not get distorted. A speed of 100–200 rpm was used for both grinding/polishing disks whereas speed of 50–120 rpm was used for specimen holder rotating in the opposite direction to the grinding wheel. Force of 0.10– 0.80 daN was used during the grinding depending on the grade of paper abrasive. Each abrasive paper required 300–900 s to complete the grinding.

3.4.4 Polishing

Polishing was done just after the grinding to remove the finer scratches from the sample. It used the same set-up as was used in grinding with only difference that during polishing special clothes were used instead of paper abrasive. Soft clothes of velvet, canvas or selvet can be used for the said purpose. Instead of water coolant, polishing medium like alumina, colloidal, or diamond slurry can be used. Diamond slurry of different grade size was used in present work for polishing i.e. 6 µm,3 µm, and 1µm. The polishing cloth was washed out with water before the start of polishing to remove any chances of contaminants which may cause deep scratches on surfaces. All the polishing parameters were same as in grinding. Polishing machine/grinding machine (Make: PRESI, Mecatech 234, Germany) shown in Figure 3.8 was used in the present work.

Figure 3.8 Grinding and polishing machine (Courtesy: Tata Steels, Jamshedpur).

3.4.5 Etching

Etching is done to disclose the microstructure of the selected metal/alloy through selective chemical attack. Cleaning of sample by alcohol is required before etching. Etchant must be chosen carefully and prepared accurately. Etchant can be applied by means of a cotton bud wiped over the surface for a few times (required precautions must be taken while etching, as very small difference occurs between etching and over-etching). Nital (2% nitric acid (HNO_3) in ethanol) was used in the present work. The specimens were instantly washed with alcohol after applying the etchant and were then dried by hot air drier.

3.4.6 Leveler

The surface of the sample to be examined optically must be flat and leveled, otherwise viewing surface area would be out of focus. By using leveling press, this problem can be solved, as it presses the mounted specimen into clay on a microscope slide and makes it flat. A piece of paper was used to coat the surface of the specimen to avoid the scratches. In the present work, a leveling machine (Make: Leica Microsystems, Wetzlar, Germany) as shown in Figure 3.9 was used.

Figure 3.9 Leveling machine (Courtesy: Tata Steels, Jamshedpur).

3.4.7 Characterization of the heat treated specimens

The microstructural details of the thermo-mechanically processed specimens were characterized by using optical microscopy, scanning electron microscopy, and transmission electron microscopy.

A. Optical microscope

The purpose of the optical microscope is to magnifying small samples assisted by visible light and a system of lenses. Investigations of plane cross-sections of metallic materials are done by incident light. Due to the difference in the refractive indices, they appear in different grey or color shades. The optical microscope (Make: Leica DM2500 Microsystems, Wetzlar, Germany) was used in the present work (see Figure 3.10).

Figure 3.10 Optical microscope (Courtesy: Tata Steels, Jamshedpur).

B. Scanning electron microscope (SEM)

Scanning electron microscope (SEM) is an electron microscope which uses high-energy beam of electrons to scan the sample and provides the corresponding details of images. Figure 3.11 shows the scanning electron microscope (Make: QUANTA FEG SEM 650; Field Emission Inc., Hillsboro, Oregon USA) used in the present study.

Figure 3.11 Scanning electron microscope (Courtesy: Tata Steels, Jamshedpur).

C. Transmission electron microscope (TEM)

In transmission electron microscope (TEM), a high energy beam of electrons is flashed through an especially thin sample and the interactions between electrons and the atoms can be used to study the features i.e. the crystal structure, dislocations, and grain boundaries. Figure 3.12a is showing the TEM used in current work (Make: JEOL-2100f, Tokyo, Japan).

ATEM specimen should be thin so as to transmit sufficient electrons to form an image with minimum energy loss. Therefore, specimen preparation is an important phase of TEM analysis. Specimen needs a separate holder through which it is inserted in the TEM (see in Figure 3.12b). For most of the materials, a general preparation technique is ultrasonic disk cutting, dimpling, and ion-milling. In the current work, specimen of 3mm diameter with 0.08mm thickness was prepared. Later on twin-jet electro polishing and etching was done to reduce the thickness up to 0.06mm by means of a solution which contained 5% perchloric acid and 95% methanol at a temperature approximately -38 ℃.

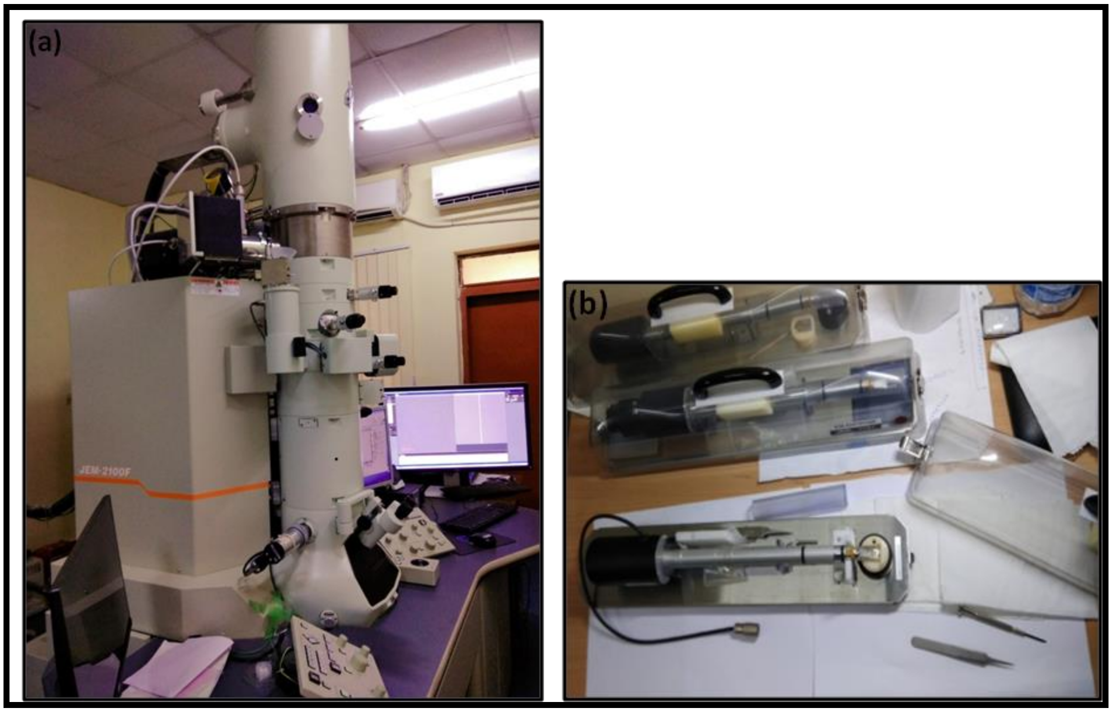


Figure 3.12 (a) Transmission electron microscope, and (b)specimen holder (Courtesy: IIT Kharagpur).

3.5 Machines and equipment

3.5.1 Muffle furnace

Muffle furnace was used to get the high austenization temperatures/deformation temperatures during thermo-mechanical processing in the present work. The muffle furnace used in the present work (Make: Naskar & Co, Kolkata, India) is shown in Figure 3.13. Temperature still 1400 ℃ can be achieved from the same furnace.

Figure 3.13 Muffle furnace (Courtsey: CSIR-NML, Jamshedpur).

3.5.2 Forging hammer

Pneumatic forging hammer (Make: Massey, 1959, UK) was used for forging in the present research work. The hammer was generally operated by hand lever or foot pedal. The work piece was placed on lower anvil and the upper ram made strikes on the work piece through a height of 1–2 m and pressed the work piece into the desired shape and size. Forged hammer with a capacity of 0.5 T was used in the present work and is shown in Figure 3.14.

Figure 3.14 Forging hammer (Courtesy: CSIR-NML, Jamshedpur).

3.5.3 Rolling mill

Rolling mill (Make: Loewy, IHI-1967, USA) can be used for cold rolling/hot rolling depending on the requirement. In the present work, it was used for hot rolling was used. The purpose of hot rolling was to reduce the thickness by maintaining plain strain conditions. Hot rolling was carried out in the temperature range of 1100–1250℃. The input conditions used for hot rolling were power of 15 kW with current and voltage as 32 A and 415 V respectively. The rpm of the rollers depends on the material and its rolling temperature; it can go up to 32 rpm. Depth of draft was calculated by theoretical calculations. The draft scale contained 250 divisions and each division resulted in 0.025mm reduction in thickness.

The rolling mill used in the present research is shown in Figure 3.15.

Figure 3.15 Rolling mill(Courtsey: CSIR-NML, Jamshedpur).

3.5.4 Salt bath

Salt bath (Make: Naskar & Co, Kolkata, India) was used for isothermal holding at the bainitic transformation temperature. A rectangular furnace contained a cylindrical pot (100 mm diameter and 210 mm length) inside the furnace. Salt bath pot used in the present work is shown in Figure 3.16. Salt mixture of50% $NaNO_3$ with 50% KNO_3 by weight percent was melted into the cylindrical pot and maintained at the required temperature i.e. 350℃. Maximum temperature of 1300℃ can be achieved in this salt bath.

Figure 3.16 Salt bath (Courtesy: CSIR-NML, Jamshedpur).

3.5.5 Wire EDM

Wire EDM (Make: Fanuc Robocut, Japan) machine was used to cut the tensile samples. Dielectric fluid (distilled water) was used as the working medium. Figure 3.17 shows the wire EDM used in the present work.

Figure 3.17 Wire EDM (Courtesy: Tata Steels, Jamshedpur)

3.5.6 Tensile testing machine

The tensile testing machine (Make: Instron 8862 System, Instron Engineering Corporation, Norwood, USA) with a loading capacity of 100 kN was used (see Figure 3.18).

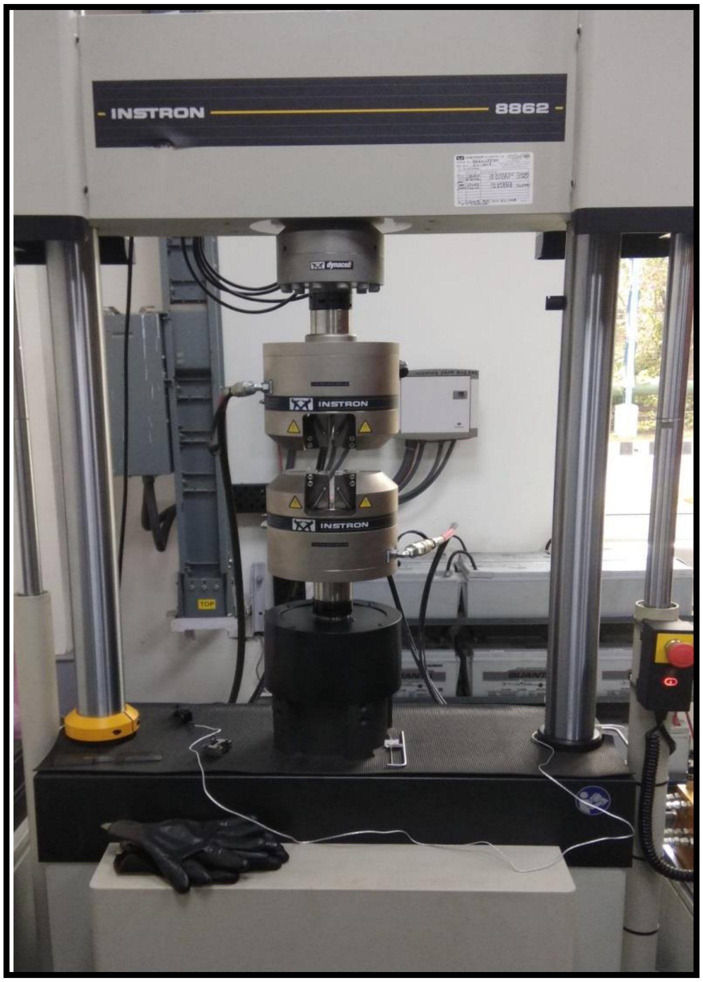


Figure 3.18 Tensile testing machine (Courtesy: Tata Steels, Jamshedpur).

The next chapter presents the results and their discussion.

CHAPTER 4
RESULTS AND DISCUSSION

4.1 General

This chapter presents the main results of experimental work that was carried out in the present dissertation work. The chapter deals with the microstructure analysis at various stages. It also discusses the results of mechanical processing routes through which bainite formed in the thermo-mechanically processed steel specimens. Finally, the results of testing are presented and discussed.

4.2 Microstructure of the starting material

The initial microsctructure of the as-cast received steel is presented and discussed in this section. Microstructure of the starting material (as-cast steel specimen) showed a typical honeycomb like microstructure. Figure 4.1 presents the optical micrograph of the as-received cast material. Microstructure mainly consisted of ferrite phase (as black solid phase) with various alloy precipitates (as white interfaces at boundaries between the solid phases).

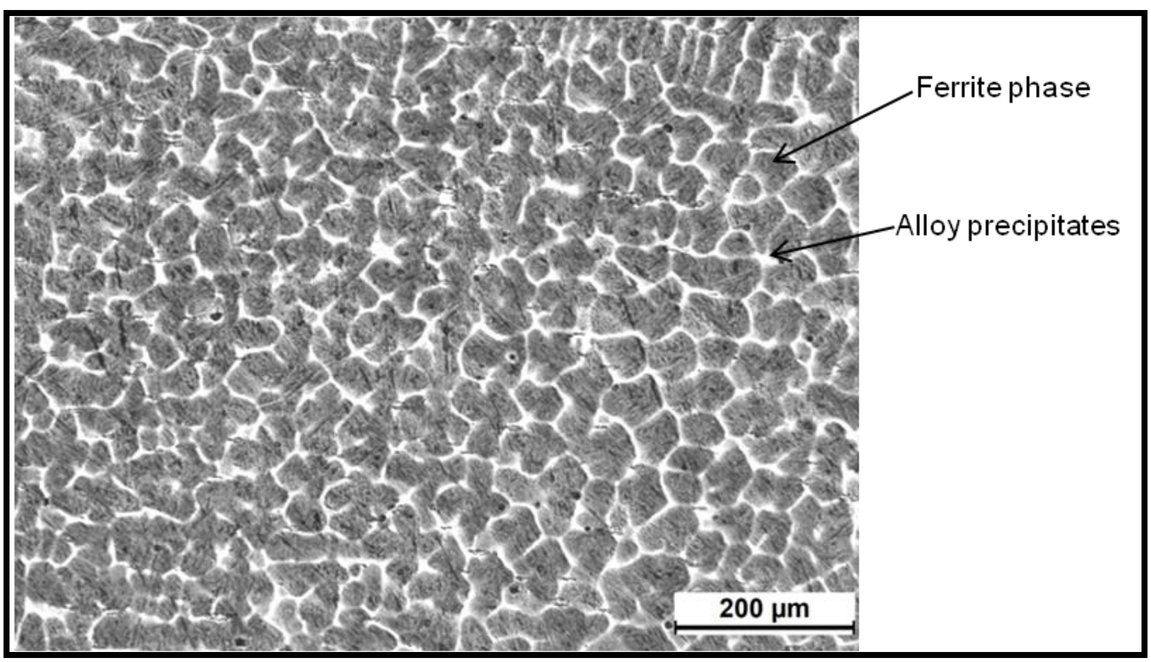


Figure 4.1 Optical microstructure of the starting material (as-cast alloy).

4.3 Prediction of heat treatment parameters

The various heat treament parameters of the given steel composition (A_{C3} , B_S , and B_f temperatures) required for thermo-mechanical processing were predicted by constructing phase fraction diagram, TTT, and CCT diagrams for the given steel chemistry using JMat-Pro software. The results are presented in this section:

The upper critical temperature for austenization (A_{C3}) was predicted from the phase fraction diagram as 830 ℃. The B_S and B_f temperatures were predicted as 450 ℃ and 330 ℃ respectively using CCT and TTT diagrams. For austempering, the isothermal transformation temperature for bainitc transformation was selected as 350 ℃(in the low temperature band).

The thermo-mechanical processing route from hot forging till complete formation of final bainitic microstructure in the steel is shown in Figure 4.2.

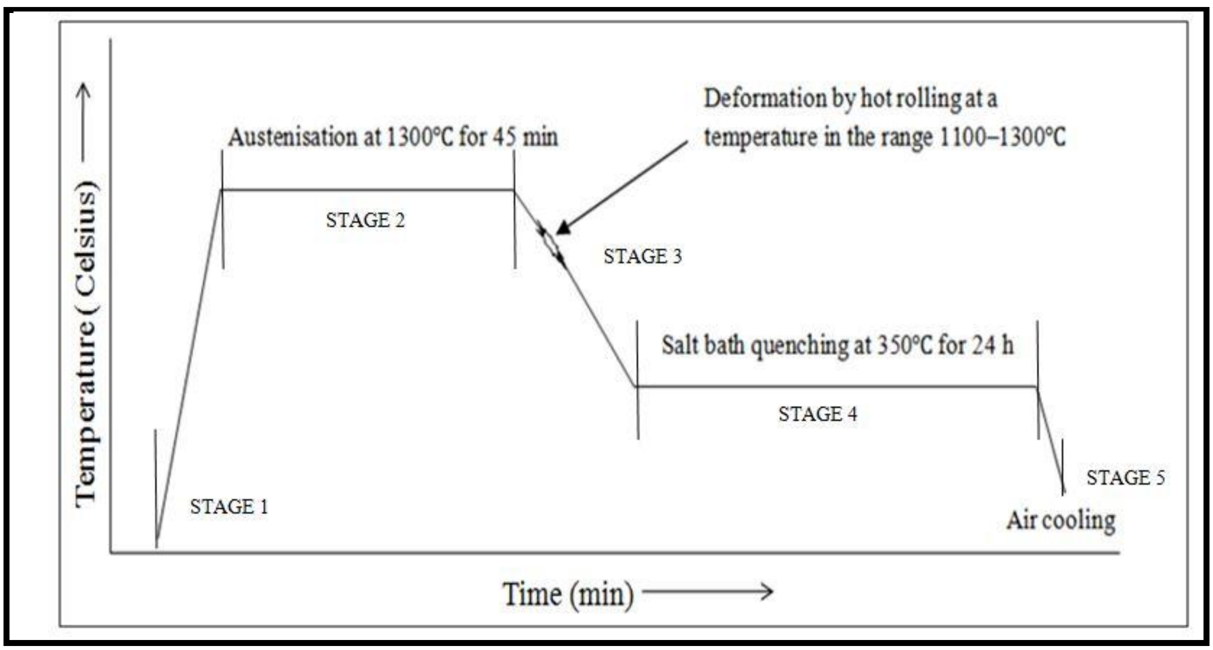


Figure 4.2 Thermo-mechanical cycle of the proposed bainitic steel.

4.4 Microstructure of the hot forged specimen

The as-cast specimen (starting material) was subjected to austenization at 1300 ℃ for 3 h. Thereafter, it was subjected to hot forging. Figure 4.3 presents the optical micrograph of the hot forged steel specimen.

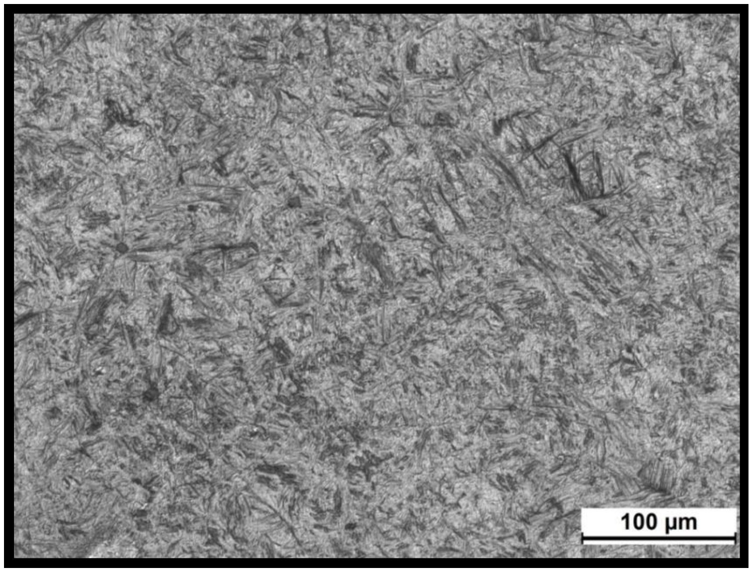
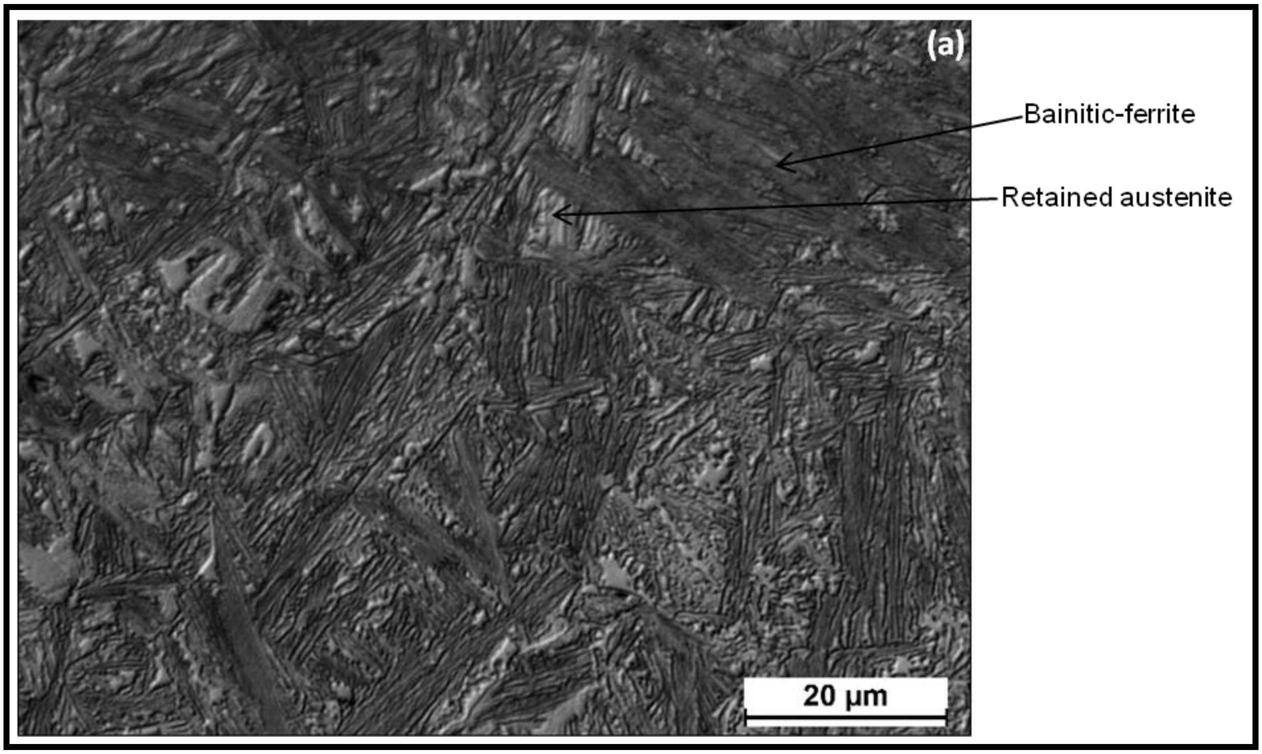
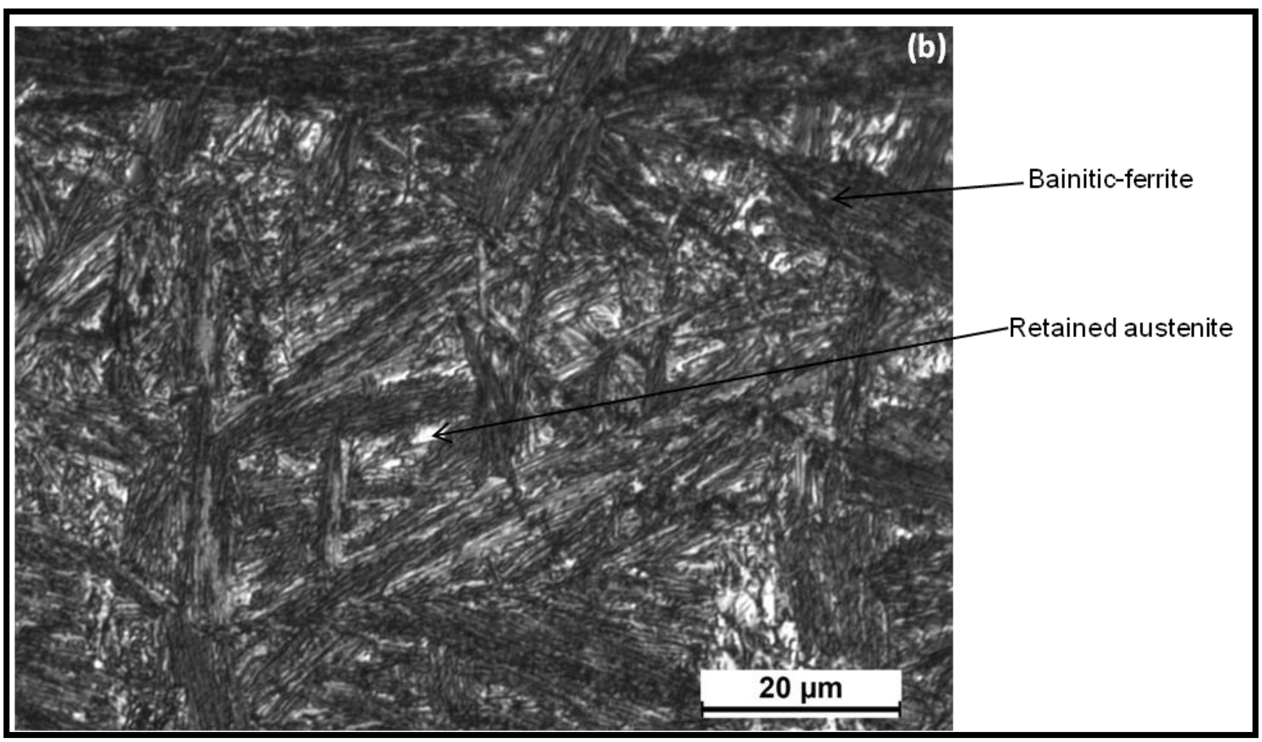


Figure 4.3 Optical micrograph of the specimen subjected to austenization followed by hot forging.

4.5 Thermo-mechanical processing

After forging, thermo-mechanical processing took place in which material was heated to its austenization temperature of 1300 ℃ and provided 45 min soaking time. The purpose was that all precipitates get dissolved uniformly and the material gets completely homogenized. After complete soaking, the material was subjected to hot deformation process. During hot rolling the material went through 55% reduction in thickness. Hot rolled sample was suddenly quenched to 350 ℃ in salt bath for isothermal transformation of bainite. During quenching, austenite which has FCC crystal structure underwent instant super cooling (i.e. from 1200 ℃ to 350 ℃) which induces huge stresses within the crystal structure, resulting in displacive mode of transformation. When such shape deformation occurs in bulk of polycrystalline steel, its accommodation leads to high strain energy, and to minimize it, the ferrite phase adopts a thin-plate like shape called bainitic-ferrite [11]. In between these thin plates of bainitic-ferrite there is some un-transformed austenite, also called retained austenite (RA) which does not get transformed to bainitic-ferrite due to unfavorable thermo-dynamical conditions. Bainitic-ferrite plates form without any diffusion of carbon, but after complete transformation, the carbon partitions into RA [11]. The soaking time for isothermal transformation at the bainitic transformation temperature (350℃) was varied over a wide range in the present work (short; moderate; and also very large). The soaking time was varied in three distinct periods of 1h, 1 day, and 3 days to study the behavior and mechanical properties of bainitic-ferrite and RA obtained in different conditions. The microstructure obtained in each case is shown in Figure 4.4a-c.





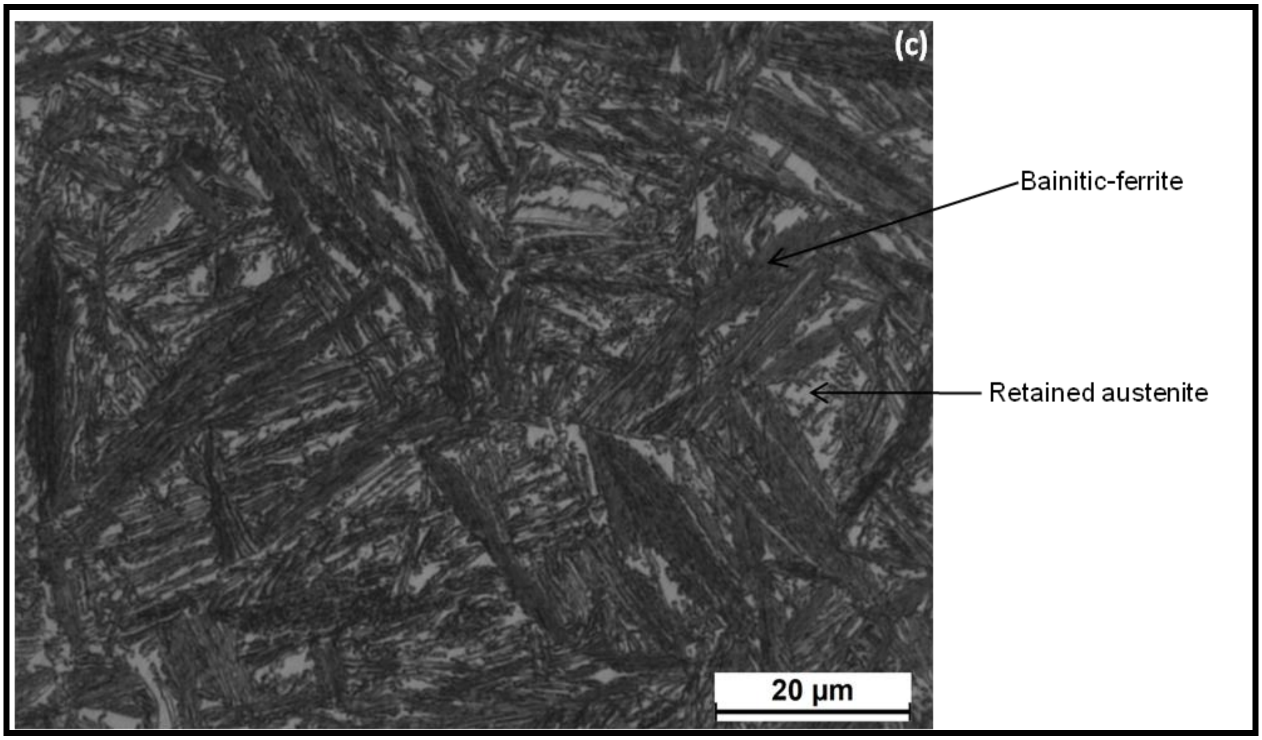


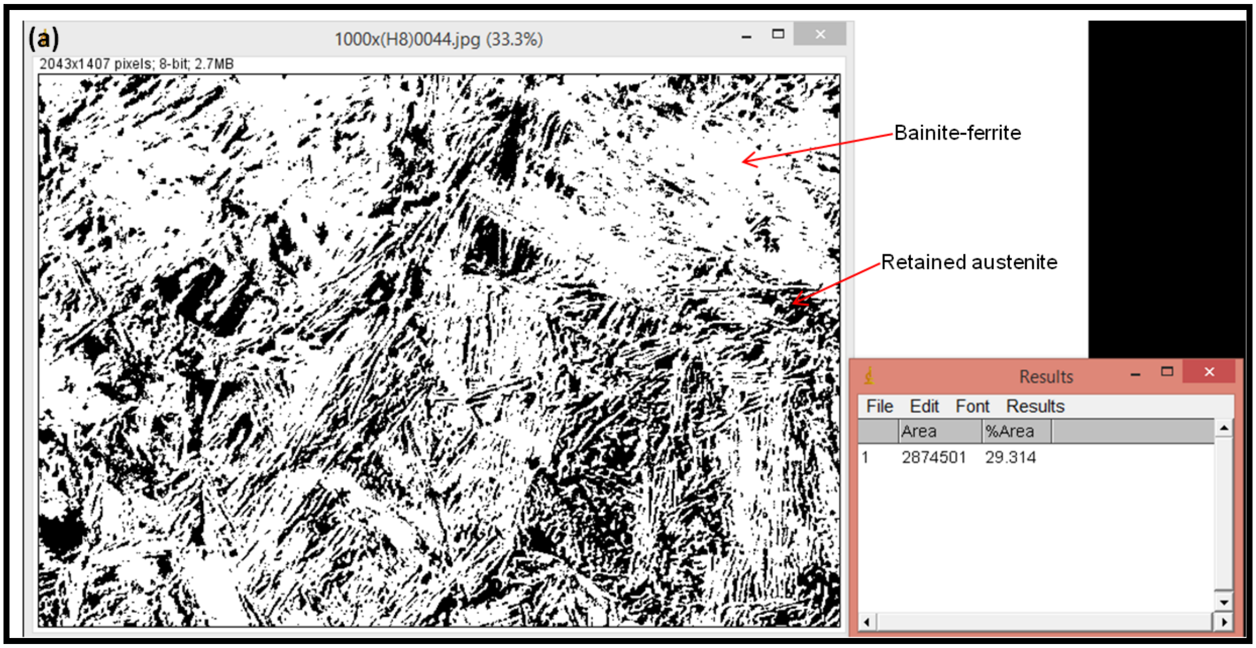
Figure 4.4 Optical microstructure of specimens with soaking time of (a) 1 h (b) 24 h (c) 72 h.

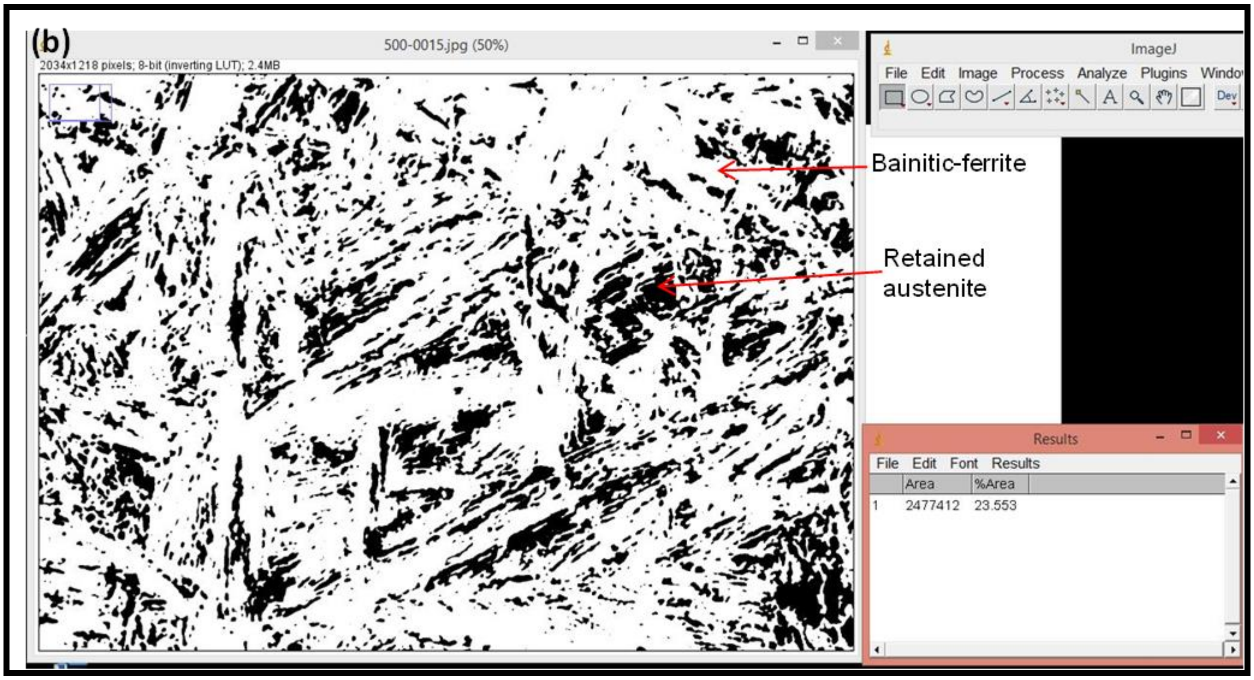
Figure 4.4a–c shows the thin needle like structure of bainitic-ferrite found enormously in all the specimens obtained under the three conditions. The density of apparently acicular like structure sheaves was very high and the sheaves were randomly oriented throughout the structure. Volume fraction of the thin bainitic-ferrite was measured using ImageJ software. Phase fraction measurements showed most of the microstructure covered with thin plates of bainitic-ferrite with some amount of residual retained austenite. The actual distribution of bainitic-ferrite and residual austenite is shown in Table 4.1 with the help of ImageJ analysis shown in Figure 4.5a-c.

Table 4.1 Volume fraction of phases in final microstructure of various specimens.

Holding time at the bainitic transformation temperature (i.e. 350 ℃)	Volume fraction of bainitic-ferrite (%)	Volume fraction of retained austenite (%)
1 h	**70.686**	**29.314**
24 h	**76.447**	**23.553**
72 h	**77.862**	**22.138**

Figure 4.5 shows the procedure to measure volume fraction of final phases by ImageJ. White color shows the dominating bainite-ferrite phase whereas stable retained austenite was found as non-uniformly distributed white patches within the microstructure.





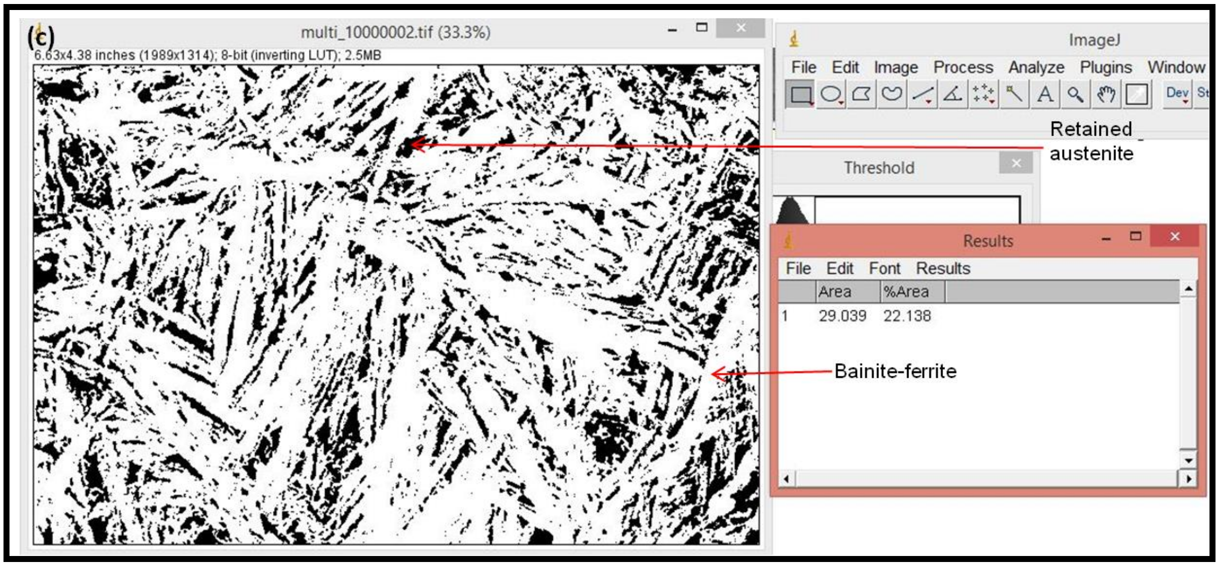
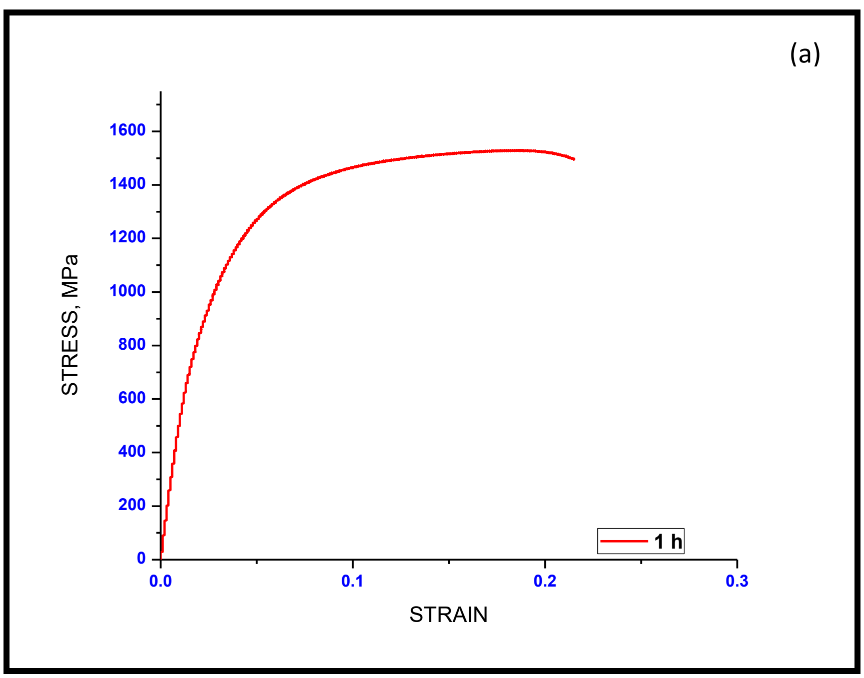
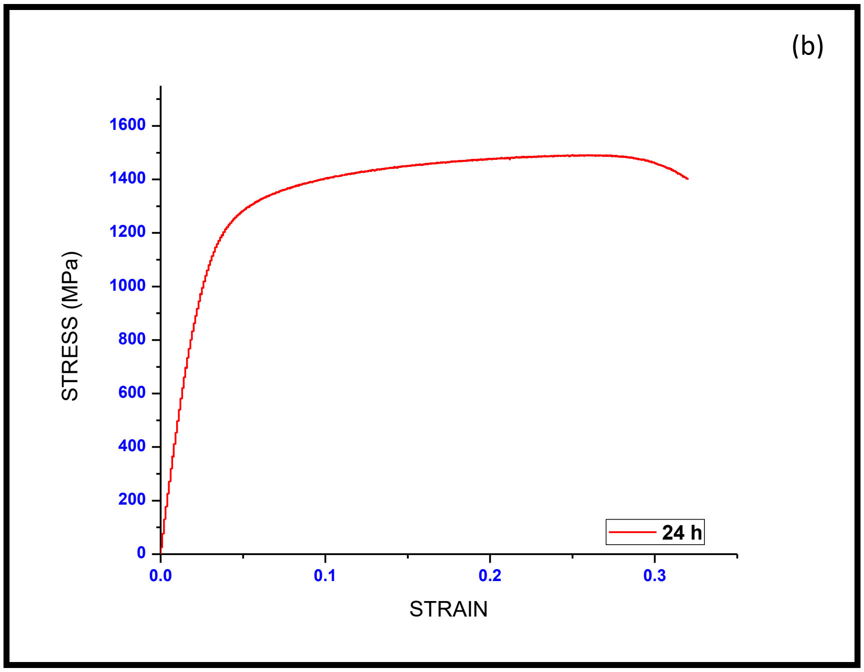


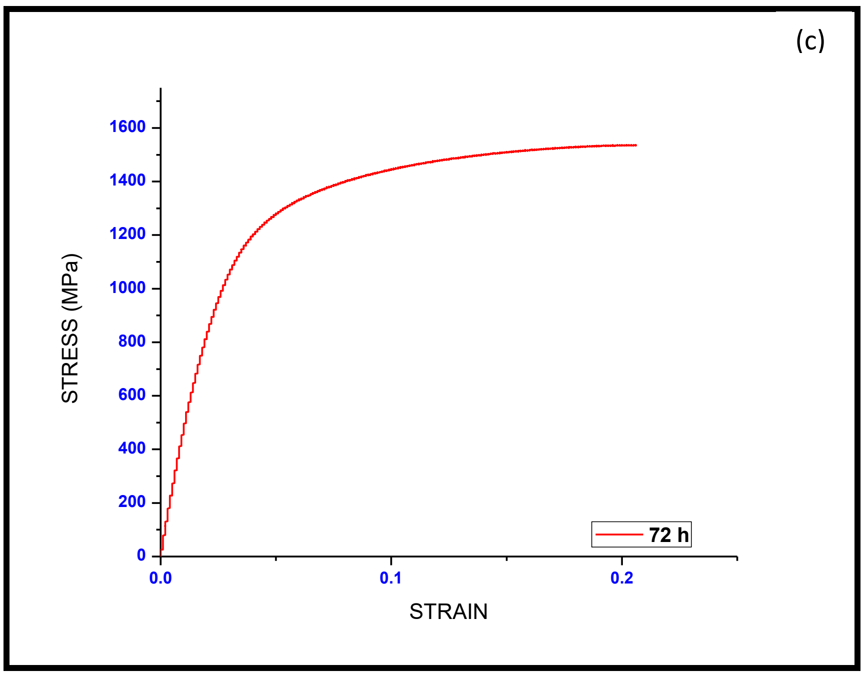
Figure 4.5 Volume fraction analysis using ImageJ for samples with soaking period of (a) one hour, (b) one day, and (c) three days.

4.6 Tensile testing results

Tensile testing results of all the three types of samples (1 h, 24 h, 72 h of soaking time) are presented in Figure 4.6a-d. The tensile properties obtained in the three cases are presented in Table 4.2.




(b)
STRESS (MPa)
1600
1400
1200
1000
800
600
400
200
0
0.0
0.1
0.2
0.3
STRAIN
24 h


(c)
STRESS (MPa)
1600
1400
1200
1000
800
600
400
200
0
0.0
0.1
0.2
STRAIN
72 h

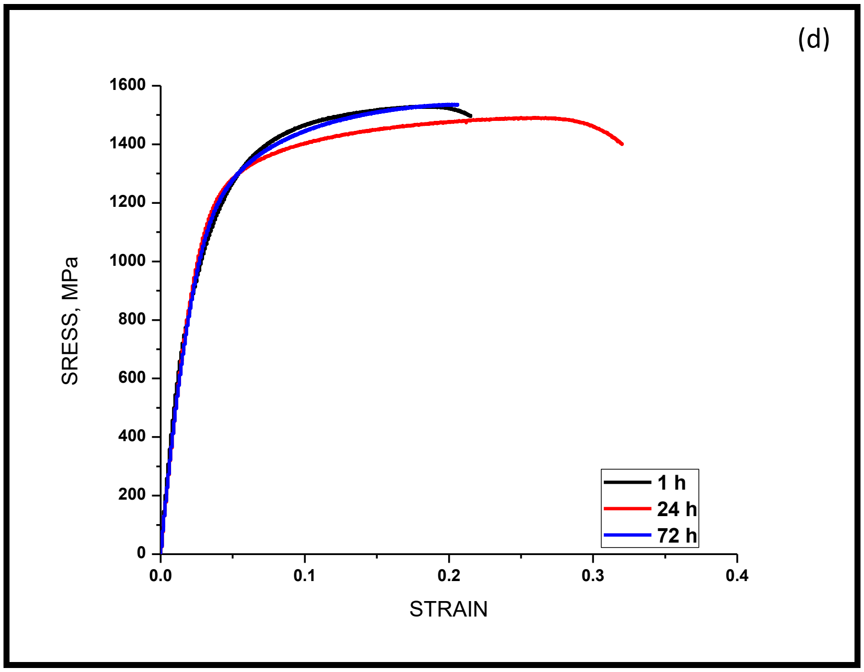


Figure 4.6 Stress-strain curves for specimens held at transformation temperature for (a) 1 hour, (b) 1 day,(c) 3 days, and (d) the graph showing all the curves.

Table 4.2 Tensile properties obtained in various specimens.

Soaking time at transformation temperature	Ultimate tensile stress (MPa)	Percentage elongation (%)
1 h	**1499.65**	**20 ± 2**
1 day	**1507.29**	**30 ± 2**
3 days	**1536.377**	**18 ± 2**

It is shown clearly that the strength in each case is very high and almost equal. This is because of presence of high volume fraction of thin bainitic-ferrite which imparts high strength to the steel. Further, austenite has a great role in increasing the elongation/ductility of steel. The reasons for difference in mechanical properties (especially percentage elongation) in the three specimens are discussed as follows:

A. It was observed that holding time of 1 h at the isothermal bainitic transformation temperature was sufficient for austenite to bainitic transformation. The results demonstrate that further increase in holding time will not increase the volume fraction of bainite-ferrite. However, holding time plays a great role in diffusion of carbon (and other alloying elements present, if any) into austenite plate and determines the level of mechanical stability of the austenite plate in addition to other substitution alloying elements.

B. The result showed that there is an optimum fraction of stable retained austenite which provides best combination of high strength-good ductility in bainitic steels. If retained austenite is unstable, it can readily transform to martensite during straining, thereby affects the elongation of the steels. On the other hand, presence of retained austenite in amounts more than the optimum or presence of over-saturated austenite can decrease the strength.

C. It is also possible that austenite may not undergo any phase change; nevertheless, there may be crystallographic alteration in the microstructure, i.e. presence of stacking faults (SFs) and twins in the microstructure. This occurs when stacking fault energy of stable austenite is good enough to form the SFs and twins.

D. The results of tensile testing indicated similar strength in all the three samples. This indicated that for the sample with 1 h holding time, some low volume fraction fine martensite might be generated. This low martensite fraction could not affect the strength considerably (as compared to other samples of 1 day and 3 days); nevertheless, the presence of martesite impaired the ductility of this sample (1 h holding time). On the other hands, for the sample with 3 days holding, there is a high possibility of containing some low volume fraction of carbide due to considerable carbon diffusion for the prolong time. This carbide precipitation might have affected/impaired the elongation of this sample (3 days holding).

E. For the current work, one of the main aim was to incorporate the SFs or twins in the bainitic steel to achieve high strength in combination with high ductility. The current results demonstrate a good combination of high strength and high ductility, especially for the 1 day holding. There might be fine twins in the austenite, nevertheless, detail HRTEM will be carried out for future investigation. However, the streaks in the SADP (see Figure 4.7) relates to the typical presence of SFs.

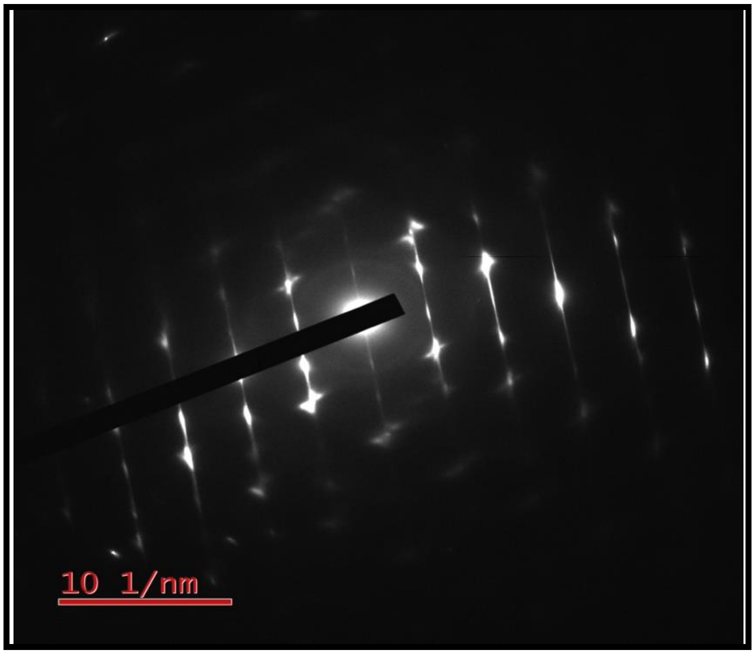


Figure 4.7 Surface area diffraction pattern (SADP) by TEM analysis showing streaks of SFs.

4.7 Microstructural analysis

Specimen held for 1 day at the quenching temperature showed the best strength-ductility combination of 1.5 GPa-30%. Further microstructure analysis was conducted for this specimen only. The result are presented and discussed as follows.

4.7.1 Fractographic analysis using SEM

Fractured surface of tensile specimen of the 24 h sample was analyzed using SEM microscopy (see Figure 4.8). A large number of small dimples were observed signifying large plastic deformation and hence justifying the high value of ductility obtained in this specimen.

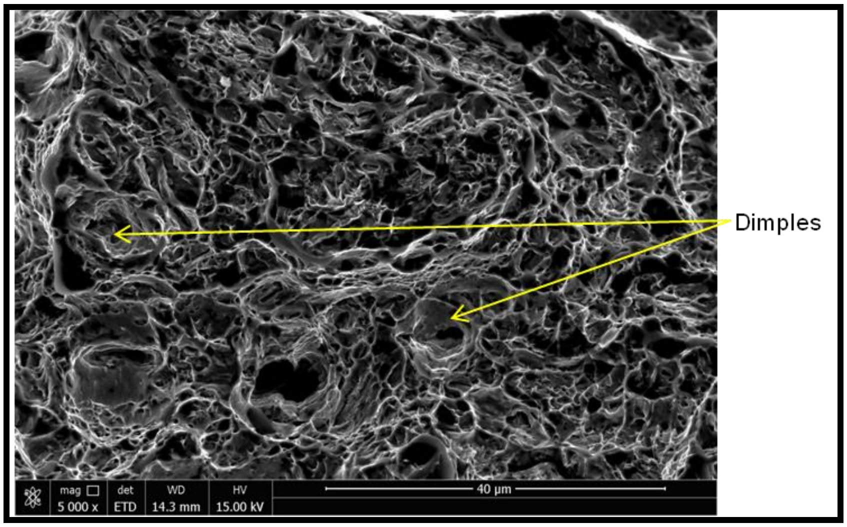


Figure 4.8 SEM micrograph of fracture surface for the tensile specimen held at the bainitic transformation temperature for 1 day.

4.7.2 Microstructural analysis using SEM

SEM analysis was done to study the morphology and distribution of bainite-ferrite (BF) and retained austenite (RA). Figure 4.8 shows details of morphology of bainite-ferrite and retained austenite and also their distribution in the microstructure. The thin plates of bainite-ferrite were found uniformly distributed throughout the micrograph. RA was present in two forms (a) fine austenite, and (b) blocky austenite. Thin films of RA in-between the sheaves of bainite-ferrite help to improve the mechanical properties whereas the high volume fraction of RA films lead to improved ductility [29]. Very low fraction of blocky austenite was also present in the microstructure which is a matter of concern (scope for future work). Blocky austenite leads to inhomogeneous deformation causing drop in ductility [6, 29]. Figure 4.9 shows the SEM image micrograph for the 1 day specimen.

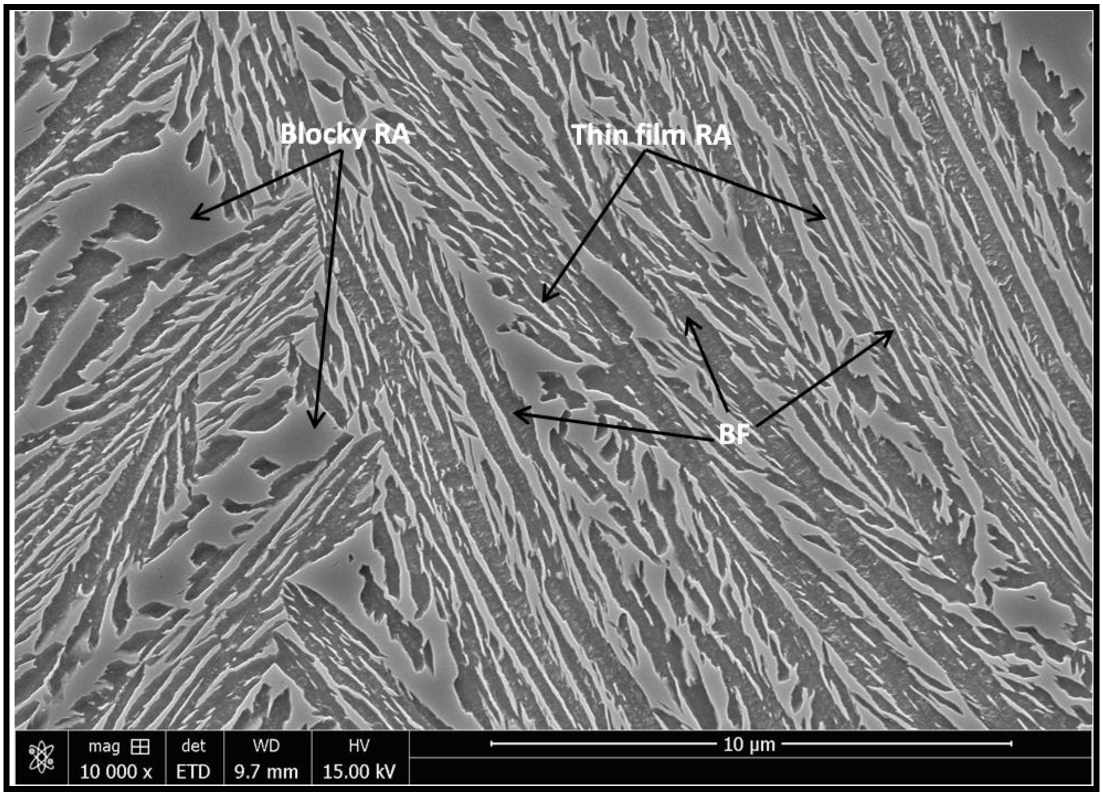


Figure 4.9 SEM micrograph showing microstructural details for the 1 day specimen. BF represents bainite-ferrite and RA is retained austenite.

4.7.3 Microstructural analysis using TEM

Finally, TEM analysis of the 1 day specimen was done. TEM micrograph confirmed that the microstructure was free from carbides as was expected because of Si presence in the steel chemistry. Figure 4.10 shows the TEM micrograph for the 1 day specimen.

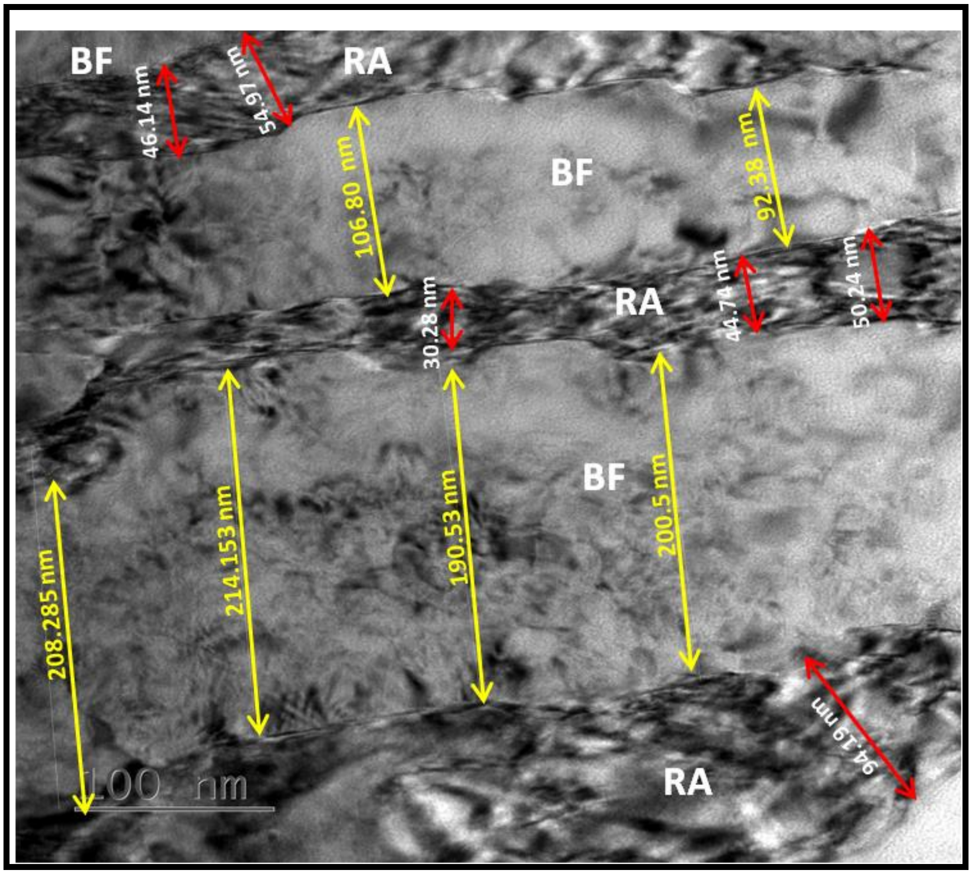


Figure 4.10 TEM micrograph showing microstructural details for the 1 day specimen. BF represents bainite-ferrite and RA represents retained austenite.

The micrograph further confirmed that the low temperature transformation utilized in the present work resulted in formation of very fine micro-scale bainitic-ferrite with thickness in the range of 0.10–0.20 µm. Also, ultra-fine thin plates (0.03–0.05 µm) of retained austenite were observed. It is reported that such fine RA plates result in superior mechanical properties [29].

CHAPTER 5

CONCLUSIONS

5.1 General

In the present dissertation work, bainite was obtained by isothermal holding at the transformation temperature. The resultant bainitic steel showed improved combination of strength and percentage elongation.

5.2 Results and conclusions

The main results and the conclusions to be drawn from the present experimental work are as follows:

A. Microstructure of the starting material

- Microstructure of the starting material (as-cast steel specimen) showed a typical honeycomb like microstructure. The microstructure mainly consisted of ferrite phase with various alloy precipitates.

B. Prediction of heat treatment parameters

- The upper critical temperature of the given steel was predicted as 830 ℃. B_S and B_f temperatures were predicted as 450 ℃ and 330 ℃ respectively. For austempering, a low isothermal transformation temperature of 350 ℃ was selected for bainitc transformation.

C. Thermo-mechanical processing

- For thermo-mechanical processing, the steel was heated to its austenization temperature of 1300 ℃ and provided 45 min soaking time. After complete soaking, the material was subjected to hot deformation process. During hot rolling, the material went through 55% reduction in thickness. Hot rolled sample was suddenly quenched to 350 ℃ in salt bath for isothermal transformation of bainite.
- The soaking time for isothermal transformation at the bainitic transformation temperature (350℃) was varied over a wide range in three distinct periods of 1h, 1 day, and 3 days to

study the behavior and mechanical properties of bainitic-ferrite and RA obtained in the microstructure under different conditions.

- The density of acicular shaped bainitic sheaves was very high and these were randomly oriented throughout the microstructure. Bainite phase mixture mainly comprised of thin plates of bainitic-ferrite and was the primary phase in microstructure along with residual retained austenite.

D. Tensile testing

- The results of tensile testing showed that microstructure comprising of high volume fraction of bainite (in the form of thin bainitic-ferrite) along with some amount of stable retained austenite results in good strength-ductility combination in bainitic steel. The bainitic steel (1 day sample) comprising of bainite (76.45%) and retained austenite (23.55%) showed the best properties with high strength (1507.29 MPa) and good ductility (30.0%).
- The results showed that for a given steel chemistry and thermo-mechanical processing, there is an optimum soaking time period (here, 1 day) at the isothermal bainitic transformation temperature which provides best combination of bainite volume fraction and retained austenite stability for improved properties.
- For the given thermo-mechanical processing conditions applied to the steel chemistry, the microstructure of the bainitic steel with best properties showed presence of stacking faults (SFs)/twins in the microstructure.

E. Microstructural analysis

- Fractographic analysis of fractured surface of tensile tested specimen showed a large number of small dimples. The presence of dimples signified large plastic deformation and hence justified the high value of ductility obtained in the one day quenched specimen.
- SEM micrograph showed thin plates of bainite-ferrite uniformly distributed throughout the micrograph. RA was present in two forms (a) fine austenite, and (b) blocky austenite. Thin films of RA in-between the sheaves of bainite-ferrite helped to improve the mechanical properties.
- TEM micrograph showed that the microstructure comprised of very fine micro-scale bainitic-ferrite with thickness in the range of 0.10–0.20 µm. Also, ultra fine thin plates (0.03–0.05

μm) of retained austenite were observed. TEM micrograph confirmed that the microstructure of 1 day quenched specimen was free from carbides.

5.3 Major conclusions and recommendations

Attempts to develop bainitic steel with high strength-good ductility combination were successful using the thermo-mechanical processing route. This novel work provided good mechanical properties in the given steel because of the judicious mix of steel chemistry and process parameters of the thermo-mechanical processing treatment. The presence of optimum concentration of constituent phases of bainite and retained austenite, the morphology and stability of retained austenite, and the presence of SF's/twins in the microstructure were important factors resulting in development of bainitic steel with very high strength of 1.5 GPa and good percentage elongation of 30%.

5.4 Scope of future work

In the current work, bainite was obtained in the steel at a low transformation temperature (350°C). Micrographs showed presence of some blocky retained austenite in the microstructure. This morphology of retained austenite is detrimental to the mechanical properties. Bainite transformation mainly depends on the transformation temperature along with isothermal holding time. Future work can be conducted to explore the transformation temperature-soaking time combinations which can result in elimination of this un-desirable blocky retained austenite from the microstructure of bainitic steel for further improvement in mechanical properties. Also, future work can be carried out to confirm more clearly the presence of SF's/twins in the bainitic steel microstructure.

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