

Alloy Design and Optimization of Heat Treatment Cycle to Develop Ultra High Strength with Superior Ductility Bainitic Steel

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Abstract

Bainitic steel with YS > 600 MPa, UTS > 1100 MPa and TE > 20 % was developed in the present study with alloy design and optimization of heat treatment cycle. The steel with a chemical composition (wt.%) 0.2C-1.7%Si-2%Mn-0.9%Cr-0.2%C-0.18%Mo-0.07Nb-0.04Ti was induction melted and forged followed by slicing to 2mm thick sheets. The sheets were then subjected to inter critical annealing with 70% austenite and fully austenitized (above A₃) followed by salt bath holding in the range of 350 to 480°C. YS, UTS and TE decreased in the case of inter critical annealing followed by increase in salt bath holding temperature due to the coarser lath structure of the bainites whereas the UTS increased with drastic decrease in the elongation and no significant change in the YS for the steel austenitized above A₃ followed by increase in bainitic holding temperature due to the transformation of globular type bainite to lath type bainites and their further coarsening. It was found that the steel austenitized above A₃ followed by salt bath holding at 350°C gives the best properties due to the presence of granular type bainite in the range of 50-55% along with fine precipitates in addition to the TRIP effect for the presence of 10% retained austenite. Presence of fine precipitates of micro alloying elements further improves the mechanical properties of the steel.

Keywords: bainitic steel, heat treatment, granular bainite, lath bainite, ultra high strength and superior ductility

1. Introduction

Bainitic steel are known to exhibit high strength and hardness and also a high level of toughness (F G Caballero, et al., 2006). The detrimental effect of cementite can be suppressed by addition of silicon above 1.5% which has very low solubility in cementite and greatly retard its growth from austenite (F G Caballero, et al., 2006; F G Caballero, et al., 2001; E. Jezierska, J. Dworecka & K. Rozniatowski, 2014). Hence, the rejected carbon from the bainitic ferrite enriches the residual austenite to stabilize it to room temperature. The existence of retained austenite promotes the TRIP effect during deformation to give ultra high strength and ductility aided by fine gran structure in the steel (Chengjia Shang, Xinlai He & Huaxin Hou, 2010). Suppressing the cementite formation to develop carbide free bainites and nano bainites with the nano size precipitation and fine bainitic laths with the addition of suitable alloying elements in the steel are growing demand for their excellent mechanical properties (V. C. Igwezie & P. C. Agu, 2014; Avanish Kumar & Aparna Singh, 2021). There are several terminology in bainitic steels such as carbide free bainite, trip aided bainitic ferrite (Gopal Sanyal, 2023; Jianhao Yan, et al., 2024; K. Hausmann, D. Krizan, A. Pichler & E. Werner, 2013), lower bainite (H.K.D.H. Bhadeshia, 1980; G. Spanos, H.S. Fang & H.I. Aaronson, 1990), upper bainite (G. Spanos, H.S. Fang & H.I. Aaronson, 1990; T. Furuhashi, H. Kawata, S. Morito & T. Maki, 2006; P. Retzl & E. Kozeschnik, 2024), granular bainite (Z.X. Qiao, et al., 2009; David De-Castro, et al., 2022; R. M. Jentner, et al., 2023), lath bainite and plate (C P Luo & Jiangwen Liu, 2006; Adam Skowronek, et al., 2022), nano bainite etc (Avanish Kumar & Aparna Singh, 2021) which makes confusion to the researchers although some of them implies the same group of the bainitic steels.

Lower bainite consists of fine needle like plates or laths with the carbides precipitated within the laths. The

microstructure of upper bainite consists of fine plates of ferrite grow in clusters called sheaves. Within each sheaf the plates are parallel and of identical crystallographic orientation, each with a well-defined crystallographic habit. The individual plates in a sheaf are often called the 'sub-units' of bainite. They are usually separated by low-misorientation boundaries or by cementite particles (H.K.D.H. Bhadeshia, 1980; G. Spanos, H.S. Fang & H.I. Aaronson, 1990; T. Furuhashi, et al., 2006; P. Retzl, E. Kozeschnik, 2024).

Granular bainite is a term frequently used to describe the bainite that occurs during continuous cooling transformation, widely used in industry, where most steels undergo non-isothermal heat treatments. Microstructure of granular bainite forms gradually during cooling to form coarse structure of the sheaves of bainite which under optical microstructure appears as blocks of bainite and austenite. A characteristic of granular bainite is the lack of carbides in the microstructure where carbon that is partitioned from the bainitic ferrite stabilises the residual austenite, so that the final microstructure contains both retained austenite and some high carbon martensite in addition to the bainitic ferrite (Z.X. Qiao, et al., 2009; David De-Castro, et al., 2022; R. M. Jentner, S. P. Tsai, et al., 2023).

The carbide free bainite or TRIP aided bainitic ferrite where precipitation of cementite during bainitic transformation can be suppressed by alloying the steel with silicon. The carbon that is rejected from the bainitic ferrite enriches the residual austenite, thereby stabilising it down to ambient temperature. The microstructure obtained consists of bainitic ferrite laths interwoven with thin films of untransformed retained austenite (Gopal Sanyal, 2023; Jianhao Yan, et al., 2024; K. Hausmann, et al., 2013; F.G. Caballero, 2012).

Nano bainite microstructure is an elegant mixture of interwoven plates of bainitic ferrite and thin films of C-enriched retained austenite, both with tens of nm in thickness (Avanish Kumar & Aparna Singh, 2021; X.F. Yu, et al., 2022).

2. Experimental Plan

A steel was designed and subjected to induction melting and forging to get billet of 70*70 mm² cross section for the development of ultra high strength bainitic steel. The billet was then sliced to 2 mm thick sheets in an EDM machine to further heat treatment in a muffle furnace with the arrangement of salt bath. The chemical composition of the steel was obtained through SPECTRO make OES. The critical temperatures of the steel was obtained by constructing the CCT, TTT diagram through JMatPro Software. The phase field and volume fraction of phase were obtained through Thermo-Calc software. The steel samples were subjected to inter critical annealing at 800°C (with the aim of two phase of 70% austenite and 30% ferrite) and above Ac₃ temperature at 880°C (with the aim of 100% austenite) for 5 min followed by salt bath quenching and holding for 5 min in the temperatures such as 350, 400, 450 and 480°C and then water quenched. Subsize tensile specimens were made from the heat treated samples according to ASTM E8 and tensile tested in a Zwick/Roell make 250 kN tensile testing machine at standard strain rate of 0.008/s. Microstructure observations were conducted through an optodigital Olympus make optical microstructure and Hitachi make scanning electron microscope after sample preparation with the standard metallography technique.

3. Results and Discussion

3.1 Chemical Composition

Chemical composition of the steel is shown in Table 1. The steel is having 0.2%C and 2% Mn which helps in austenite stabilization and hardenability and the 1.6% Si which suppresses the cementite formation additionally helps in austenite stabilization in the steel (Lucia Morales-Rivas, 2022). Around 0.9% Cr facilitates metal carbide formation and significantly improve the hardness and hardenability, while Cu improves the ductility, and Mo gives solid solution strength and reduces the susceptibility of temper embrittlement in the steel. In addition to hardenability C, Mn and Cr helps in controlling the transformation temperatures (Radhakanta Rana, et al., 2024). C, Si and Mo acts as the most powerful austenite solid solution strengtheners. Micro alloying such as Nb and Ti helps in grain refinement in the hot deformation stage to give fine grain structure (E. Jezierska, J. Dworecka & K. Rozniatowski, 2014). Hence, with the alloying and micro alloying of the steel resulted in increase in the strength and ductility of the steel.

The TTT diagram of the steel with the critical temperatures and heat treatment cycle is shown in Figure 1. The phase diagram of the steel and volume fraction of phase obtained through Thermo-Calc software is shown in Figure 2. The steel subjected to austenitization at 800 and 880°C where 800°C corresponds to 70% austenite and 880°C corresponds to 100% austenite.

Table 1. Chemical composition of the steel (wt. %)

C	Mn	S	P	Si	Al	Cr	Ni	Cu	Nb	V	Ti	Mo	B
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0.213	2.07	0.037	0.056	1.71	0.025	0.899	0.045	0.213	0.069	0.005	0.038	0.176	0.0017
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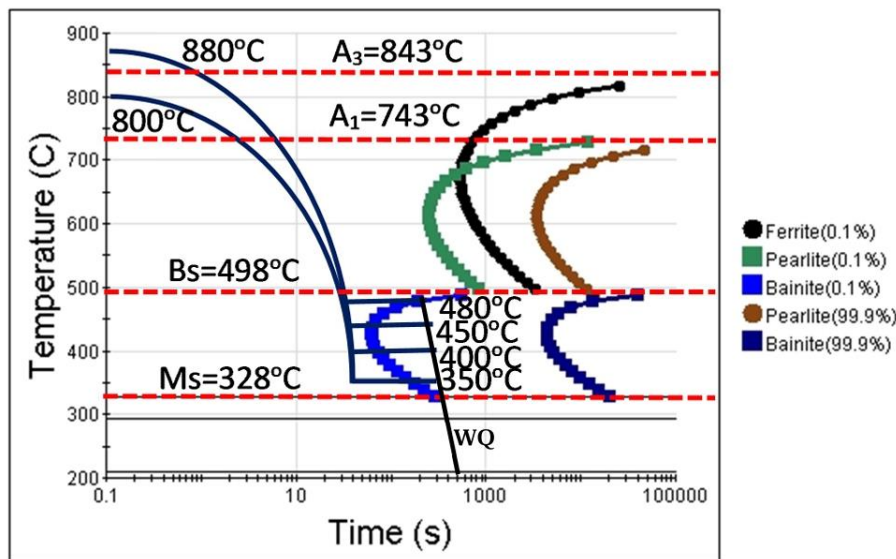


Figure 1. TTT diagram obtained through JMat Pro Software with the critical temperatures; B_s , M_s , A_1 and A_3 along with the heat treatment cycle

The steel was held in the salt bath in the temperature from 350 to 480°C to vary the bainite content in the steel from lower bainite to upper bainite with the variation in their volume fraction and morphology which changes the mechanical properties of the steel.

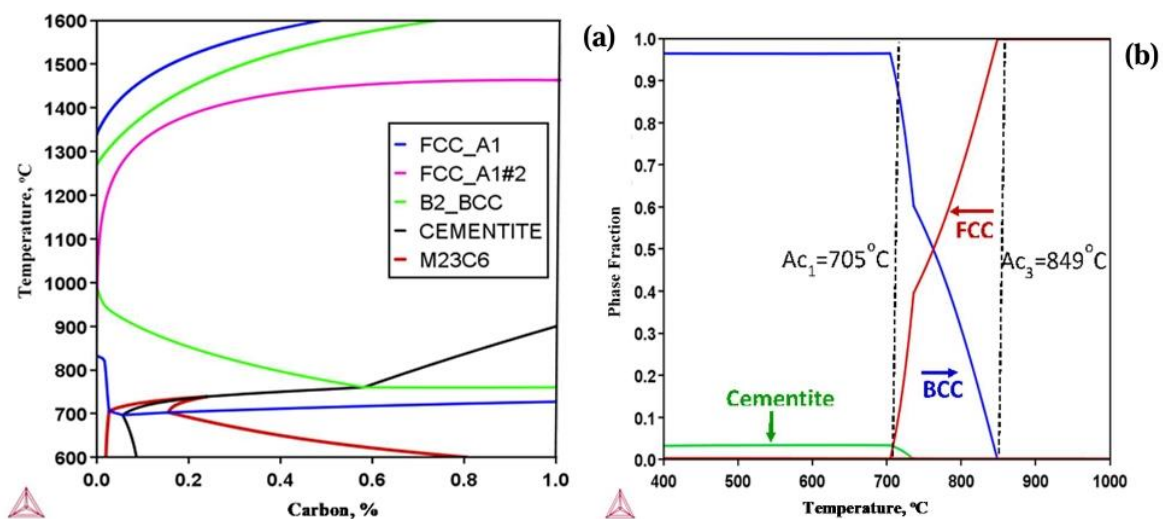


Figure 2. (a) Phase diagram and (b) Phase volume fraction of the steel obtained through Thermo-Calc software

3.2 Mechanical Properties

Figure 3 shows the engineering stress strain diagram of the steel inter critical annealed followed by salt bath holding in the temperature range of 350 to 480°C. The mechanical properties of the steel at different bainitic holding temperature have been summarized in Table 2. The mechanical properties such as YS, UTS and TE were plotted against the bainitic holding temperature shown in Figure 4. There is very high ultimate tensile strength (>1100MPaUTS) and very low total elongation (<5%) found in the as-hot deformed steel. Bainitic holding at 350°C resulted in about 50MPa decrease in strength while increase in yield strength around 30MPa and total elongation to >16%. With further increase in bainitic holding temperature no significant change in the properties

found. A very small decrease in yield strengths and elongation found with the increase in the bainitic holding temperature. The hardness of the steels was increased with increase in the bainitic holding temperature.

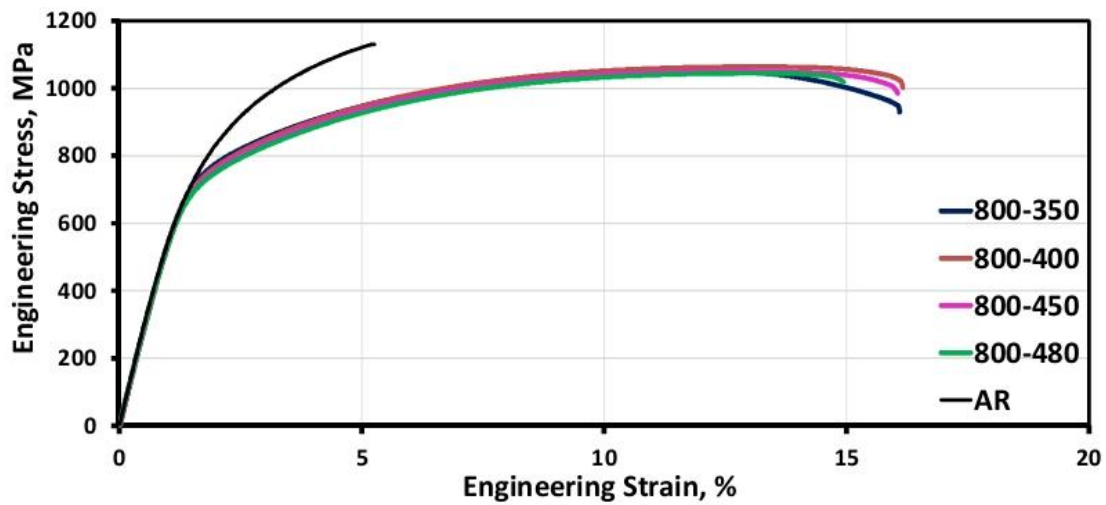


Figure 3. Engineering stress-strain diagram of the inter-critical austenitization followed by bainitic holding steel

Table 2. Mechanical properties in inter critical annealing followed by bainitic holding

Austempering Temp. °C	Austenitization Temperature- 800°C					
	YS, MPa	UTS, MPa	TE, %	YR	UTS*TE, GPa.%	Hv0.5
AR	642	1101	4.75	0.58	5.25	438
350	676	1053	15.55	0.64	16.38	402
400	661	1061	14.17	0.62	15.04	407
450	648	1055	15.83	0.61	16.70	418
480	633	1033	12.76	0.61	13.21	502

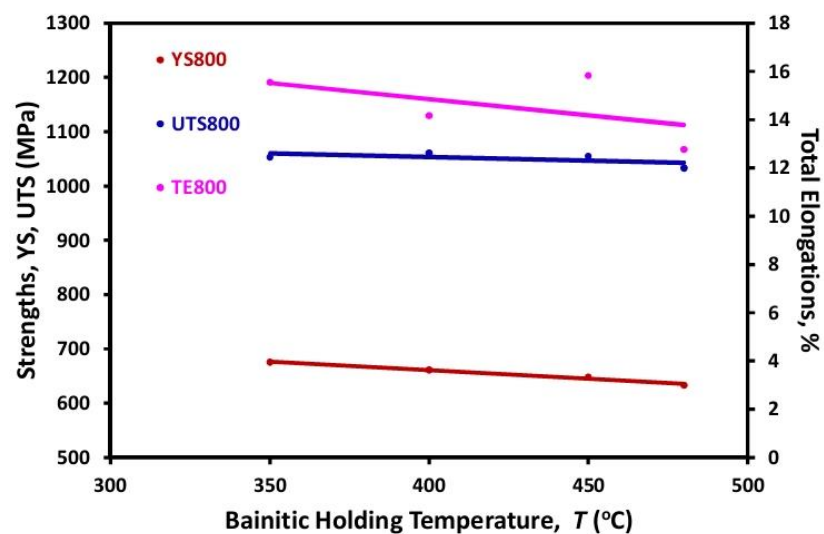


Figure 4. Change in YS, UTS and TE of the inter-critical austenitization of the steel followed by bainitic holding

The steel showed ultra high strength >1000MPa, yield strength >630MPa and total elongation >12.5% in all the

bainitic holding temperature with the best properties at the lowest bainitic holding temperature of 350°C with YS>670MPa, UTS>1050MPa and TE>15.5% with the yield ratio >0.60 with the UTS*TE>16GPa. %.

Figure 5 shows the engineering stress strain diagram of the steel after fully austenitized at 880°C followed by bainitic holding between 350-480°C. The mechanical properties of the steel have been summarized in Table 3 and the change in the properties has been shown in Figure 6. Unlike inter critical annealing the ultimate tensile strength has been increased with a drastic decrease in the total elongation of the steel with increase in bainitic holding temperature with no significant variation in the yield strength. The best properties achieved in the fully austenitized condition followed by the lowest bainitic holding temperature of 350°C with the yield strength>620MPa, UTS>1100MPa and TE>22% with the yield ratio >0.55 and UTS*TE>24.5GPa.% qualifying for the third generation AHSS.

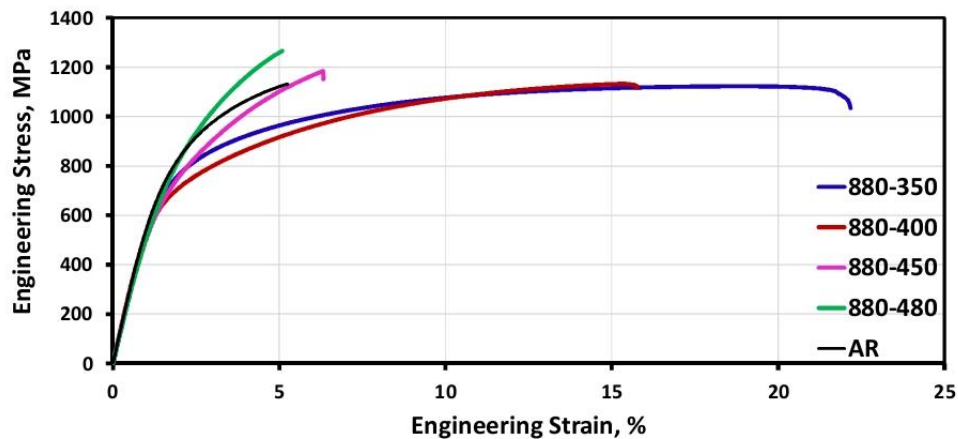


Figure 5. Engineering stress-strain diagram of the fully austenitized followed by bainitic holding steel

Table 3. Mechanical properties in austenitization above A_3 followed by bainitic holding

Austempering Temp. °C	Austenitization Temperature- 880°C					Hv0.5
	YS, MPa	UTS, MPa	TE, %	YR	UTS*TE, GPa.%	
350	625	1115	22.20	0.56	24.76	375
400	561	1126	15.42	0.50	17.37	404
450	562	1107	5.25	0.51	5.89	387
480	604	1273	5.21	0.47	6.63	389

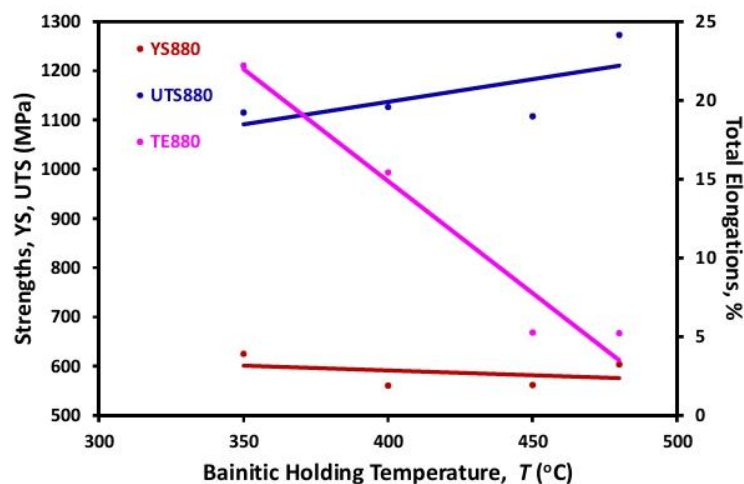


Figure 6. Change in YS, UTS, TE of the fully austenitized steel followed by bainitic holding

3.3 Microstructure

Optical microstructure and SEM micrograph of the hot deformed steel is shown in Figure 7 (a) & (b) respectively. The steel shows bainitic/martensitic microstructure with the interfaces of the plates/laths are very sharp resulted in the lower ductility of the steel although the strength of the steel is very high ($>1100\text{MPa}$).

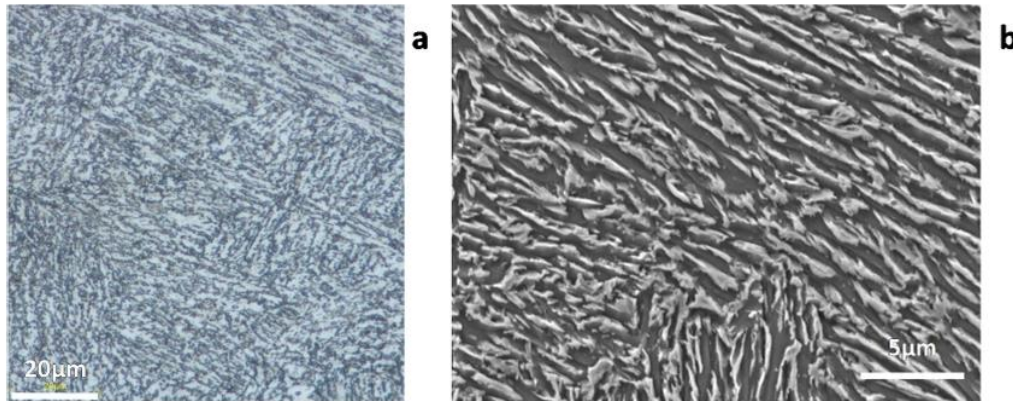


Figure 7. (a) Optical microstructure and (b) SEM micrograph of as-forged Steel

The optical microstructure and SEM micrograph as of the steel inter critical annealed at 800°C followed by bainitic holding at 350, 400, 450 and 480°C and water quenched is shown in Figure 8 (a)–(d) and (e)–(h) respectively. With the inter critical annealing at 800°C lead to introduction of 30% ferrite and in the steel followed by bainitic holding at 350°C forms lower bainite and tempered structure of the pre-existing bainite/martensite which improves the ductility of the steel above 15%. With the increase in bainitic holding temperature the structure changes from lower bainite to upper bainite with the coarser structure of the lath/plate of the bainites. However, the elongation does not drop much due to both the presence of 30% ferrite and coarsening of the bainite lath/plate structure. The yield strength of the steel decreased with the increase in bainitic holding temperature due to the tempered structure of the pre-existing bainite/martensite.

The optical microstructure and SEM micrograph as of the steel inter critical annealed at 880°C followed by bainitic holding at 350, 400, 450 and 480°C and water quenched is shown in Figure 9 (a)–(d) and (e)–(h) respectively. The steel subjected to bainitic holding at 350 and 400°C shows globular type bainite with a fully bainitic ferrite matrix to show lower bainite of the steel. The bainite content is found to be between 50-55% at 350°C bainitic holding and between 60-65% at 400°C bainitic holding. With further increase in the bainitic holding temperature the globular type bainites are transformed to lath/plate type upper bainites. The strength and elongations were greater than 1100MPa strength and $>15\%$ elongations at 400°C bainitic holding, with the best properties at 350°C with $\text{YS}>620\text{MPa}$, $\text{UTS}>1100\text{MPa}$ and $\text{TE}>22\%$ with the $\text{YR}>0.55$ and $\text{UTS}*\text{TE}>22.5\text{ GPa}\cdot\%$. With further increase in the bainitic holding temperature the volume fraction of globular type bainites increased to increase the UTS and decrease the total elongation. With further increase in the bainitic holding temperature the upper bainites were formed with the lath/plate structure with their coarsening leading to poor elongation of the steel.

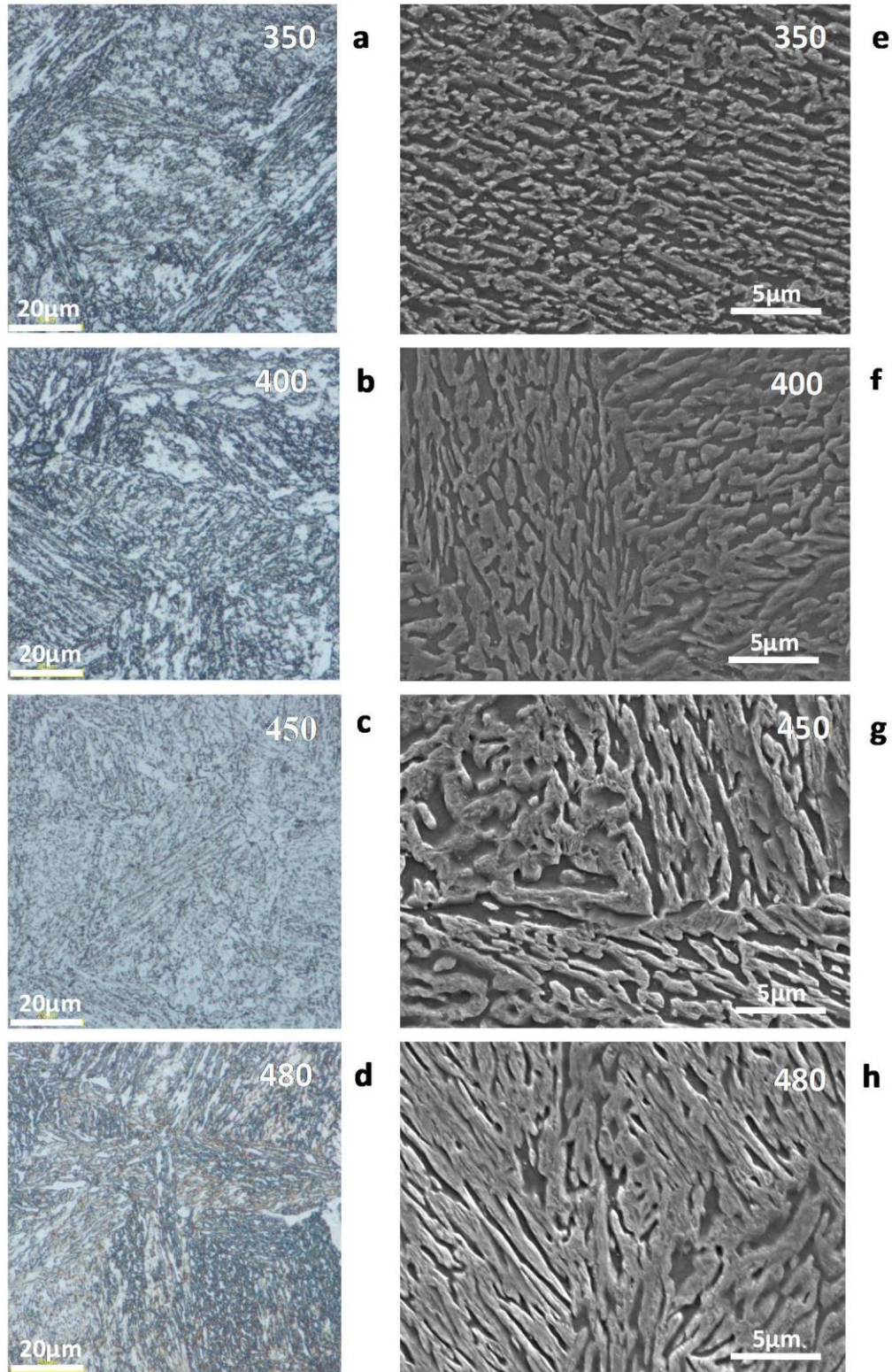


Figure 8. (a)-(d) Optical microstructure and (e)-(h) SEM micrograph of the steel IC annealed at 800°C/5min followed by bainitic holding at 350, 400, 450 and 480°C/5min respectively followed by WQ

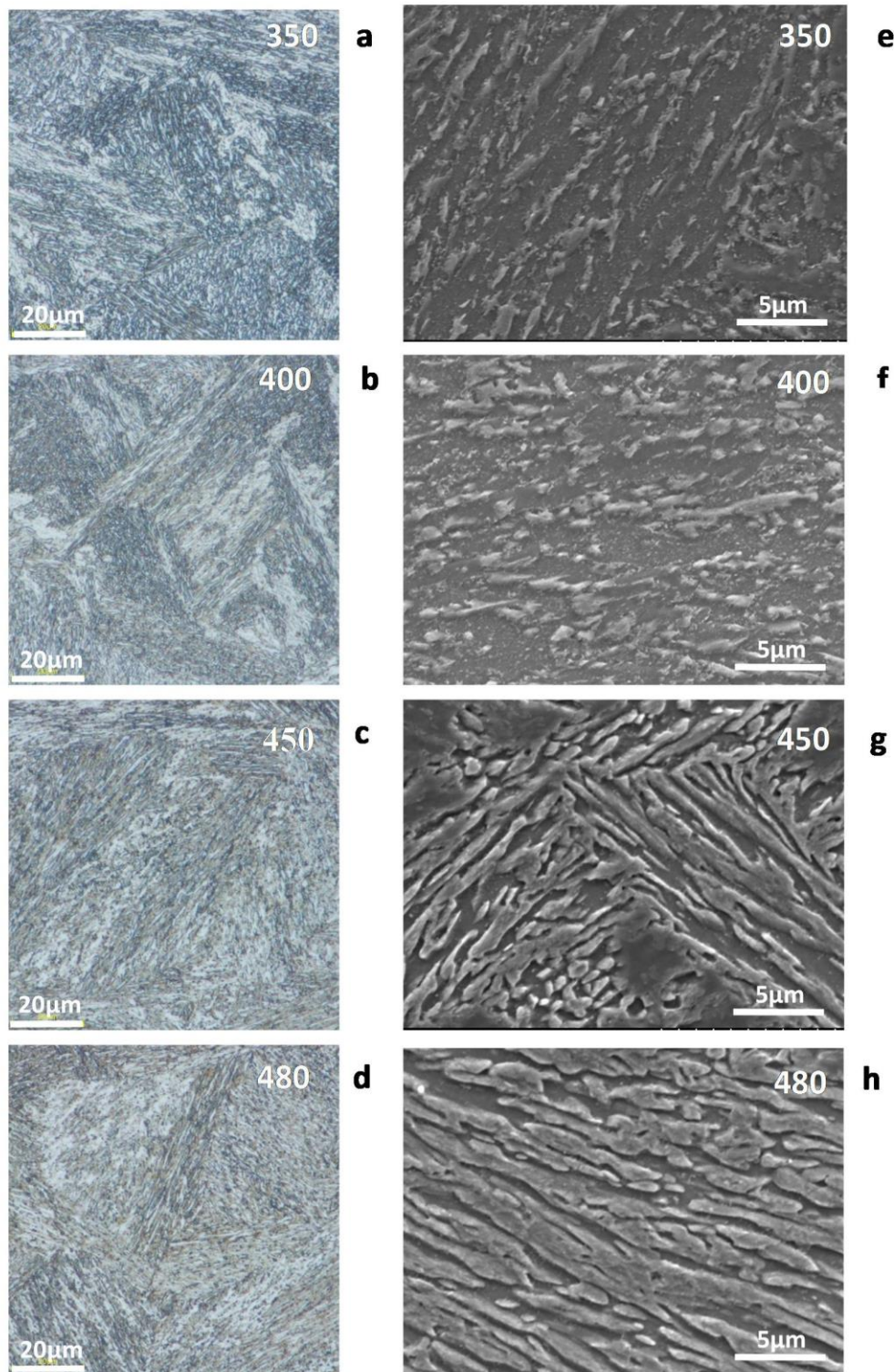


Figure 9. (a)- (d) Optical microstructure and (e)-(h) SEM micrograph of the steel austenitized at 880°C/5min followed by bainitic holding at 350, 400, 450 and 480°C/5min respectively followed by WQ

3.4 Phase Fraction/Retained Austenite Through XRD

XRD of the as-received (AR) and at selected bainitic held samples is shown in Figure 10. The retained austenite (RA) of the steel at selected bainitic holding conditions are summarized in Table 4. It can be observed that the RA is about 4.3% at the condition of inter critical annealing followed by bainitic holding at 350°C. Such bainite gives the TRIP effect to give the better strength-ductility to the steel. With increase in bainitic holding temperature the RA is getting transformed to fully bainitic structure resulting in proving reasonable strength and ductility to the steel. In the case of fully austenitized condition followed by bainitic holding at 350 and 400°C the

RA is around 10-12% which gives very good strength elongation due to the TRIP effect. After that the RA transformed to bainitic structure/ martensitic structure resulting in poor ductility of the steel.

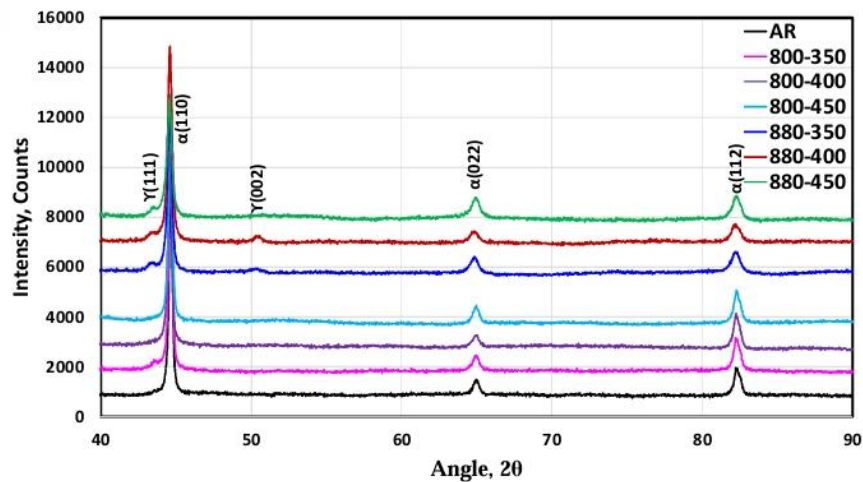


Figure 10. XRD of the as-received (AR) steel and bainitic holding in the temperature range of 350-480°C for 5min after austenitized at 800 and 880°C for 5min

Table 4. Retained austenite of the steel evaluated through XRD

Austenitization Condition	Bainitic Holding Condition	Retained Austenite, %
Inter-Critical Austenitization 800°C/5min (70% austenite)	350°C/5min/WQ	4.30
	400°C/5min/WQ	0
	450°C/5min/WQ	0
Fully Austenitization (100% austenite)	350°C/5min/WQ	10.16
	400°C/5min/WQ	12.17
	450°C/5min/WQ	4.38

4. Conclusions

A steel of composition (wt. %) 0.2C-1.7%Si-2%Mn-0.9%Cr-0.2%C-0.18%Mo-0.07Nb-0.04Ti was developed in the present study and optimized its heat treatment cycle to achieve the best level of mechanical properties by bainitic holding in a salt bath.

The steel formed is a carbide free bainite with the yield strength > 600MPa, ultra high tensile strength >1100MPa and TE>20% to give the yield ratio >0.55 and the product of UTS and TE >22 GPa.% to give the third generation AHSS properties for the optimised heat treatment condition (austenitization at 880°C/5min-salt bath bainitic holding at 350°C/5min/WQ to give globular type bainites in the range of 50-55% and 10% retained austenite which provides TRIP effect). Presence of fine precipitates of micro alloying further improves the strength of the steel by grain refinement and dislocation pinning.

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