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To cite this article: V Kurup *et al* 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **430** 012045

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The influence of full and partial austenitization temperatures on the quench and partition heat treatment process for an advanced high strength steel

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Abstract. The effect of full and partial austenitization on the microstructural evolution of a medium carbon steel with 2.2 mass %Si was studied during the quench and partitioning heat treatment process. The quench and partition process is designed to ensure that carbon partitions to retained austenite to obtain martensite and carbon-stabilized retained austenite. The full and partial austenitization temperatures were established after determining the A_{c3} and A_{c1} temperature from dilatometer runs. Heat treatment was carried out in a Bähr Dilatometer and replicated in salt bath furnaces. Phase transformations were studied by dilatometry, scanning electron microscopy and EBSD analysis. Hardness tests were carried out to evaluate the mechanical properties. The results indicate that full austenitization treatment at 940 °C resulted in thin films of retained austenite along with a lath martensite structure. The partial austenitization at 800 °C resulted in plate-like martensite with blocky retained austenite. The full austenitization gave rise to a higher volume fraction of retained austenite of about 22% which led to better mechanical properties.

1. Introduction

The average use of advanced high strength steel (AHSS) in car body applications has significantly grown over the last 20 years [1]. Numerous methods for obtaining third-generation AHSS have been proposed. The quench and partitioning process (Q&P) is considered as one of the alternatives for manufacturing AHSS [2] [3][4] [5]. During the Q&P heat treatment, austenite or austenite-ferrite mixture is quenched to an optimal quench temperature (QT) between M_s and M_f to get the desired fraction of martensite and retained austenite. This is followed by a partitioning heat treatment (PT) where the carbon from supersaturated martensite is transported to untransformed retained austenite to stabilize it. The methodology to determine optimum quench temperature has been reported elsewhere [5]. The original Q&P process is based on two important concepts: (a) constrained carbon equilibrium (CCE) and (b) athermal martensite transformation. The CCE condition implies that interstitial carbon partition from martensite to retained austenite and there is no phase boundary motion [6][5].

The Q&P steels are medium carbon steels with a composition similar to TRIP type steel [7]. The carbon content is kept low in order to avoid welding related problems. Si is added to prevent the formation of carbide precipitates in order to ensure that sufficient carbon is available to stabilize the retained austenite at room temperature [8]. Mn is an austenite former and also helps in increasing hardenability of the steel. [9]. In addition to Si, Al is also added to suppress the carbide formation [10].



Santofimia et al found that by increasing the Mn content above 3.5 mass% the initial microstructure after hot rolling consisted of martensite and polygonal ferrite. The application of Q&P heat treatment to such microstructures resulted in good mechanical properties. [11]

When the steel is subjected to a partial austenitization, the desired amount of ferrite and austenite is obtained by controlling the inter-critical temperature. The subsequent Q&P process changes the volume fraction and components of the microstructure. Partial annealing followed by the Q&P process has been reported for high Al steel. [11]

Therefore, in this work, a medium carbon steel with a high silicon content of 2.2 mass% was subjected to Q&P process after both full and partial austenitization. The effect of partial and full austenitization was evaluated by the evolution of microstructure and hardness test. The optimal Q&P process after both partial and full austenitization was studied.

2. Experimental Procedure

The experimental steel used in the present work had the nominal composition of 0.27C- 2.8Mn- 2.2Si- 0.5Al-0.75Cr and balance Fe. All composition values are in mass percentage. The steel was induction melted and cast into an ingot. It was reheated to 1200 °C to homogenize and then hot rolled and forged into 13 mm diameter rods. Si and Al were added to ensure prevention of carbide formation so that enough carbon remains in martensite to stabilize the austenite during the Q&P heat treatment. Two different sets of heat treatments were carried out, see Figure 1.

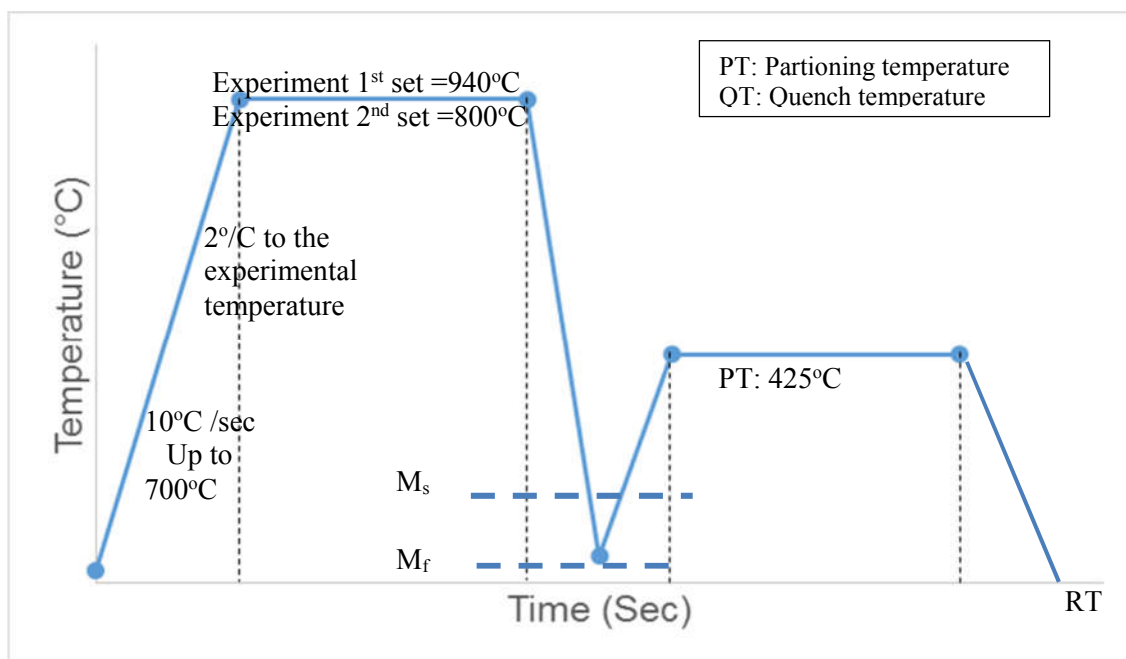


Figure 1. Q&P heat treatment cycle for the 1st and 2nd set of experiments

The Bähr Dilatometer 850D™ was used to measure the M_s and critical temperatures A_{c1} and A_{c3} . The tests were carried following ASTM A-1033-04 [12]. Thermodynamic analysis was carried out using Thermo-Calc 2017b computer software for phase analysis at different austenitization temperatures [13]. Figure (2) shows the temperature–phase fraction phase diagram generated from Thermo-Calc [13].

Heat treatments were carried out in the dilatometer machine. The first set of tests comprised of the heating sample above the A_{c3} temperature of 940 °C and thereafter quenching using helium to the calculated optimum quench temperature as determined by the Speer model [5]. The samples were then reheated to the partitioning temperature of 425 °C and isothermally held for 200 s before quenching to

room temperature. The quenching temperature was varied 40 °C above and below the calculated optimum quench temperature.

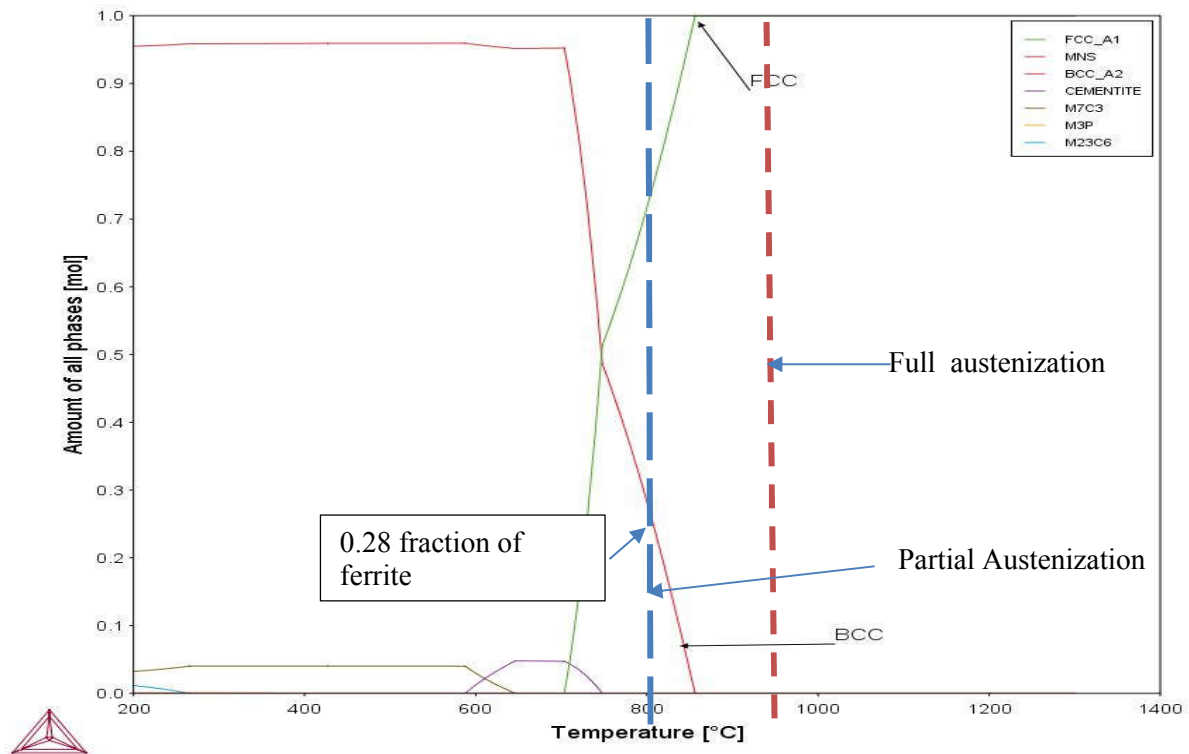


Figure 2. Temperature – volume phase fraction of the alloy as calculated by Thermo-Calc[13]

In the second set, the samples were heated in the two-phase region at 800 °C, between A_{c3} and A_{c1} , quenched to the optimum quench temperature, and partitioned as 1st set. The partial austenitization induces ferrite formation and also increases the carbon content of the austenite. The optimal quench temperature was theoretically calculated for both sets of experiments using the respective M_s temperatures and thermodynamic data.

The heat-treated samples were analyzed using x-ray diffraction (XRD). The samples were analyzed using a PANalytical X'Pert Pro powder diffractometer in θ - θ configuration with an X'Celerator detector and variable divergence and fixed receiving slits with Fe filtered $Co-K\alpha$ radiation ($\lambda=1.789\text{\AA}$). The phases were identified using X'Pert Highscore Plus computer software. The phase quantification was carried out using the Rietveld method [14]. The sample with the highest amount of retained austenite was analyzed by scanning electron microscope (SEM) and electron backscatter diffraction (EBSD). The step size used for SEM-EBSD was 60 nm. Vickers hardness test was carried out to evaluate the hardness of the alloy under different heat treatment conditions. The hardness test was done on all samples with a load of 500 grams. Ten hardness tests were taken lengthwise in the middle of each sample in order to obtain more accurate average hardness values.

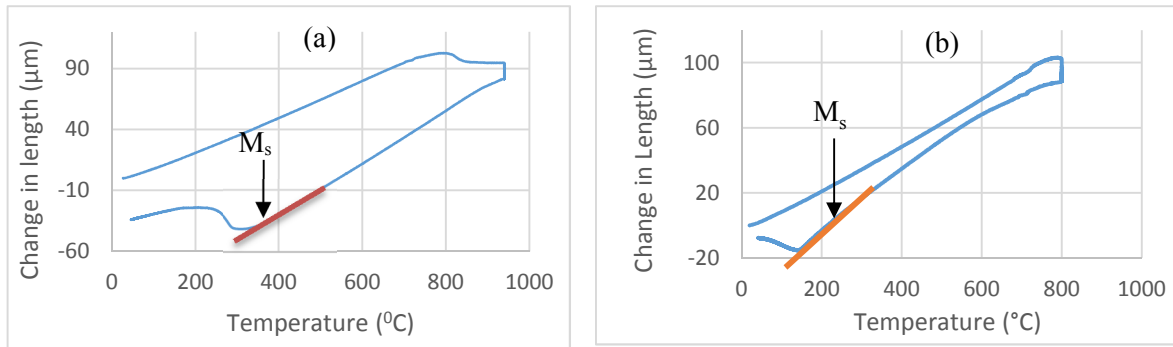
3. Results and Discussion

3.1. Critical temperatures

The A_{c1} , A_{c3} and M_s temperatures of the steels are shown in Table 1. Figure 3 shows change in length versus temperature graphs used for the estimation of M_s temperature.

Table 1. Critical temperatures obtained from dilatometer experiments

Critical temperatures	Full Austenitization at 940 °C	Partial Austenitization 800 °C
A _{C1}	765	765
A _{C3}	855	855
M _s	370	275

**Figure 3:** Change in length versus temperature graph (a) M_s temperature of steel after full austenitization at 940 °C. (b) M_s temperature of steel partially austenitized at 800 °C

The optimal quench temperature calculated using the Speer method [5] which use the KM equation that gives the fraction of martensite at the quench temperature [15]:

$$f_m = 1 - e^{-\alpha(M_s - QT)} \quad (1)$$

where f_m the fraction of martensite transformed and M_s is the martensite start temperature and QT is the quench temperature. α is the rate parameter in this case taken to be 0.011 [15] The M_s Equations, which were used are as follows:

$$M_s = 545 - 330 \times C + 2 \times Al - 14 \times Cr - 23 \times Mn - 7 \times Si \quad (2)$$

$$M_s = 565 - 600(1 - e^{-0.96 \times C}) - 31 \times Mn - 13 \times Si - 10 \times Cr \quad (3)$$

The exponential equation (3) and equation (2) were used to calculate the theoretical M_s temperature for the steel austenitized at 940 and 800 °C respectively. The theoretical optimal quench temperature for the fully austenitized sample was found to be 225 °C and for the partially austenitized sample, it was 183 °C. The optimal quench temperature is important in the design of Q&P heat treatment cycles as this temperature determines the volume fraction of martensite formed and the potential level of carbon enrichment of austenite during the partitioning process. This carbon enrichment increases the thermal stability of austenite and its M_s temperature decreases which leads to less fresh martensite on second quenching.

3.2. XRD measurements

The XRD analysis of the as-cast structure revealed the presence of martensite and 8% retained austenite. Figure 4 shows the XRD pattern of the alloy with partial austenitization at 800 °C and quenched to 183 °C.

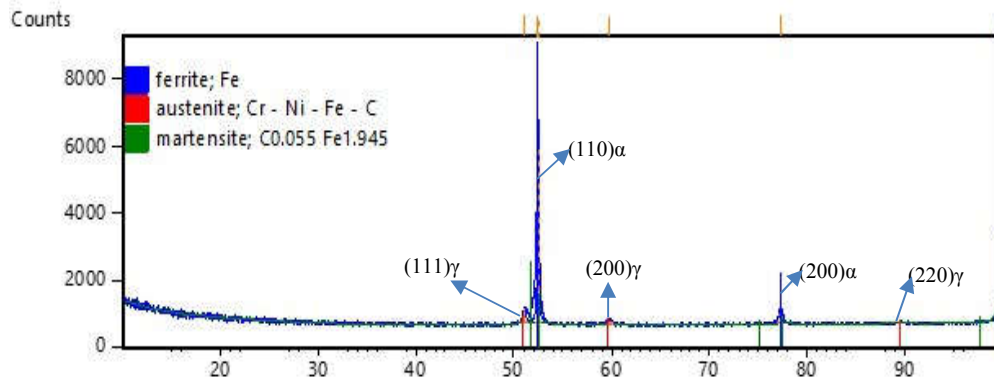


Figure 4. XRD pattern of alloy heat treat at 800°C and quenched at 183°C

Table 1. Volume fraction of retained austenite for different Q&P heat treatment, measured by XRD

<u>Partial austenitization 800 °C</u>		<u>Full austenitization 940 °C</u>	
<u>Quench temperature</u> (°C)	<u>Retained austenite</u> (%)	<u>Quench temperature</u> (°C)	<u>Retained austenite</u> (%)
143	20.3	185	17.3
183	11.5	225	22.4
223	9.9	265	19.6

Table 2 shows the amount of retained austenite at each quench temperature under partial and full austenitization condition. For steel with full austenitization, the optimal quench temperature of 225 °C resulted in maximum retained austenite of 22.4%. However, in the case of the partially austenitized sample, maximum retained austenite was obtained at 143 °C rather than the calculated 183 °C and was found to be 20.3%. This may be because not enough martensite was formed to supply carbon atoms to stabilize the retained austenite. Therefore, the unstable retained austenite transformed into fresh martensite during the second quenching stage.

From the optimal temperature calculations and XRD results in Table 2, the effect of quench temperature on the Q&P heat treatment is shown in Figures 5 and 6. It can be seen that as the quench temperature increased, the volume fraction of martensite decreased while that of austenite increased. Along with that, carbon concentration in austenite also decreased immensely. The theoretical carbon content in the retained austenite was higher in the steel which was partially annealed. From the iron-carbon phase diagram[13], it can be seen that carbon content in the austenite in the two-phase region is more, which points to the high carbon content in the retained austenite, which was partially austenitized. The dots in figures 6 and 7 indicate the experimental retained austenite results from XRD.

3.3 Microstructural characterization

Scanning electron microscope (SEM) was used to study the Q&P heat-treated samples.

Figures 7 (a) and (b) shows coarse and rounded martensite structure along with the ferrite phase from partially austenitized steel. The ferrite phase can be distinguished by the absence of carbides in it. The difference in morphology of martensite between partial and full austenitization is attributed to the higher carbon content in the former which leads to more of plate-like martensite than the lath-like structure of the latter. For the steel which was heat treated at 940 °C with Ms temperature of 370 °C, when quenched to 225 °C, exhibits a higher driving force (ΔT). This leads to a high nucleation rate resulting in the finer structure of martensite. On the contrary, the higher carbon content martensite of the partially austenitized steel leads to lower Ms temperature of 275 °C, see Figure 3. Therefore, when quenched to 183 °C, the ΔT is lower which leads to lower nucleation rate resulting in coarse plate-like martensite. As the quench, the temperature decreases to 143 °C the ΔT increases so it results in a finer structure in comparison to

the steel quench to 183 °C. The morphology of the martensite is dependent on the carbon content of the austenite. Figure 7 (c) shows the microstructure of the fully austenitized steel which consists of very fine lath martensite with some carbides. The structure consists of two types of martensite 1) secondary martensite which formed in the first quench and 2) fresh martensite formed during a final quench of the heat treatment. Secondary martensite is tempered during the partitioning process, which shows the presence of carbides in it. Retained austenite is also present but is not easily identified in the micrograph.

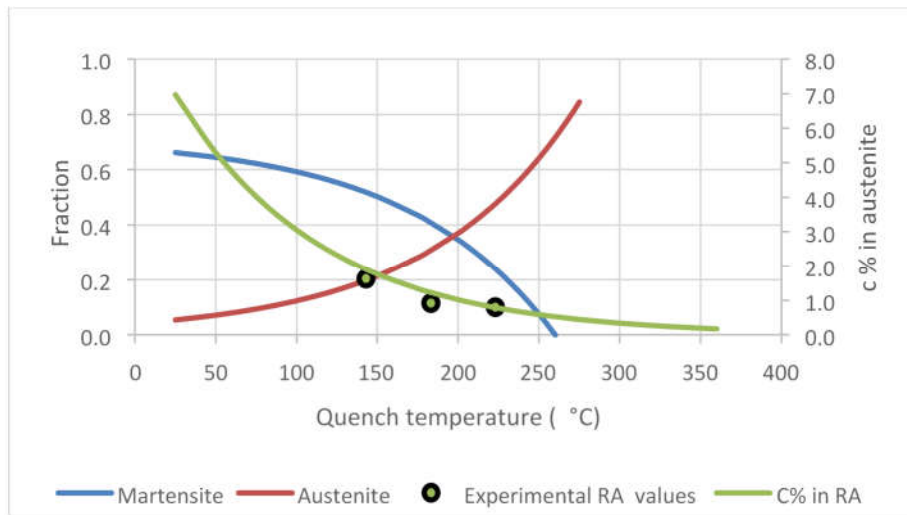


Figure 5. Fraction of martensite and austenite at various quench temperatures for steel with partial austenitization at 800°C

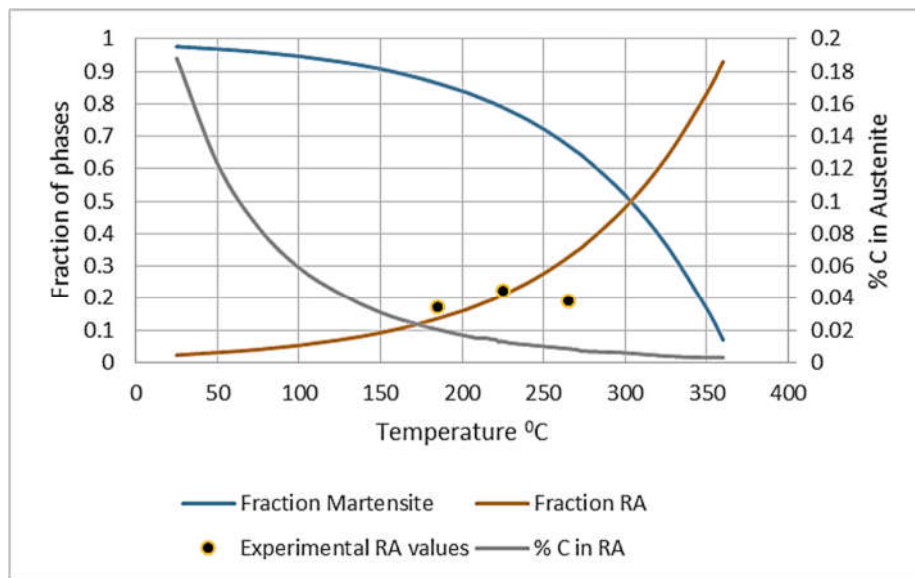


Figure 6. Fraction of martensite and austenite at various quench temperatures for with full austenitization at 940°C

The Retained austenite though present cannot be easily distinguished from the SEM images. Therefore, SEM -EBSD was carried out to see the size, morphology and distribution of retained austenite. Figures 8 (a) and (b) show the EBSD of steel that underwent full austenitization and partial austenitization respectively. It can be seen that some fraction of retained austenite is in the form of thin

films and is distributed between the martensite lath phase while the coarse blocky retained austenite is situated along the prior austenite grain boundaries. On the contrary, for the partially austenitized steel, the retained austenite is mainly coarse and blocky. Longer partitioning time was necessary to stabilize the blocky retained austenite. As a result, less retained austenite is obtained after the Q&P process in partially austenitized steel.

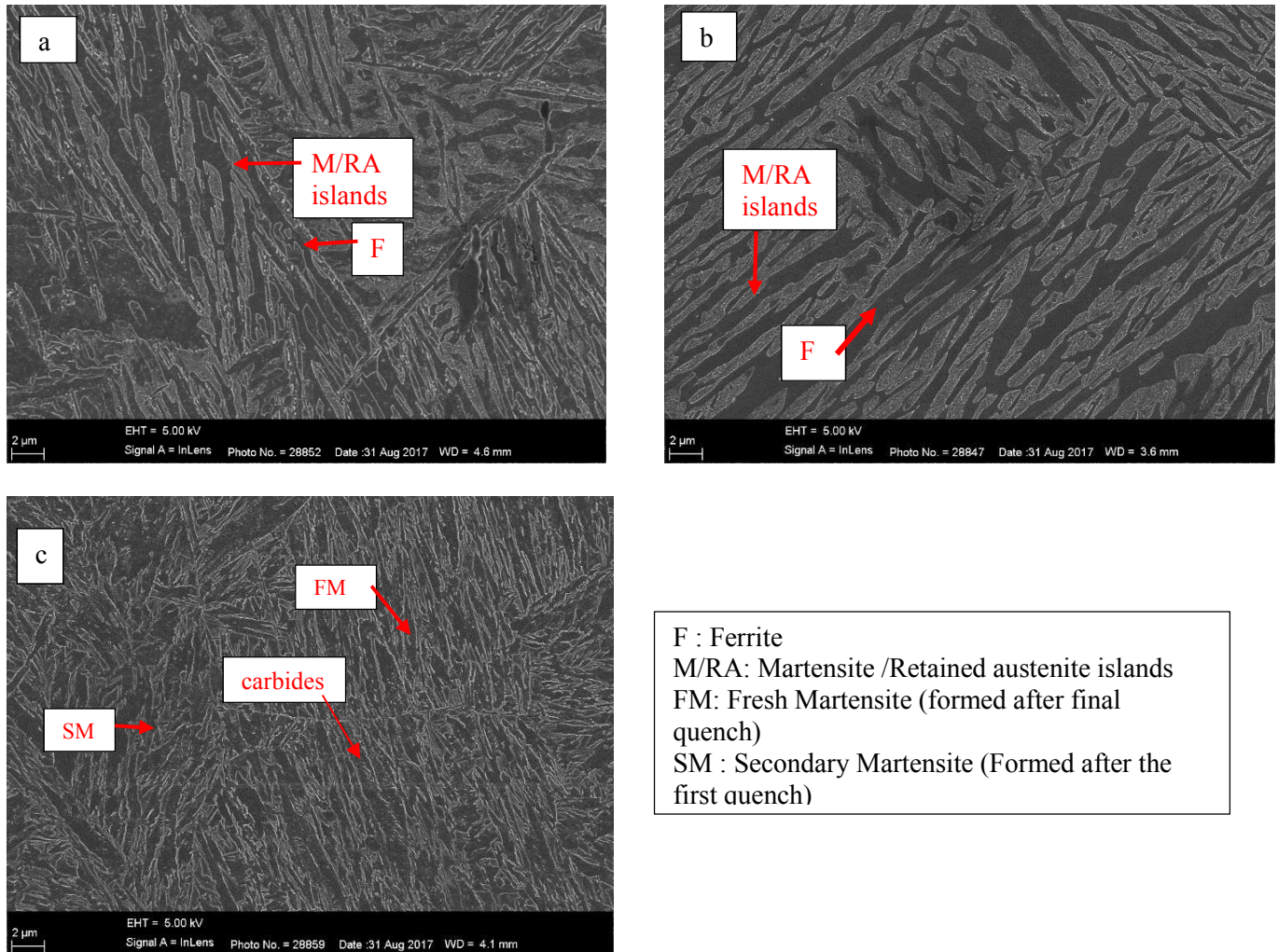


Figure 7. SEM images at 8000X a) Steel heat-treated at 800 °C and Q&P at 143 and 425 °C b) Steel heat-treated at 800 °C and Q&P at 183 and 425 °C c) Steel heat-treated at 925 °C and Q&P at 225 and 425 °C.

Figure 9 shows the hardness of full austenitization higher compared with partial austenitization. This is because the steel heat-treated at 940 °C only consisted of fine laths of martensite and thin films of retained austenite. On the contrary, the steel that was heat treated at 800 °C exhibited lower hardness because of the mixture of the soft ductile ferrite phase, plate-like martensite, and coarse blocky retained austenite. For fully austenitized steel, the hardness is insensitive to quench temperature. For partially austenitized steel, the hardness increased with an increase in quench temperature. This is attributed to the amount of the softer retained austenite which is at its maximum volume fraction at the quench temperature of 143 °C, i.e. 20.3%. In order to elucidate the discrepancy of maximum %RA between the predicted optimal quench temperature of 183 °C and the one observed at a quench temperature of

143 °C in the partially austenitized steel, the thermal profiles of the partitioning heat treatment were analysed, see Figure 10.

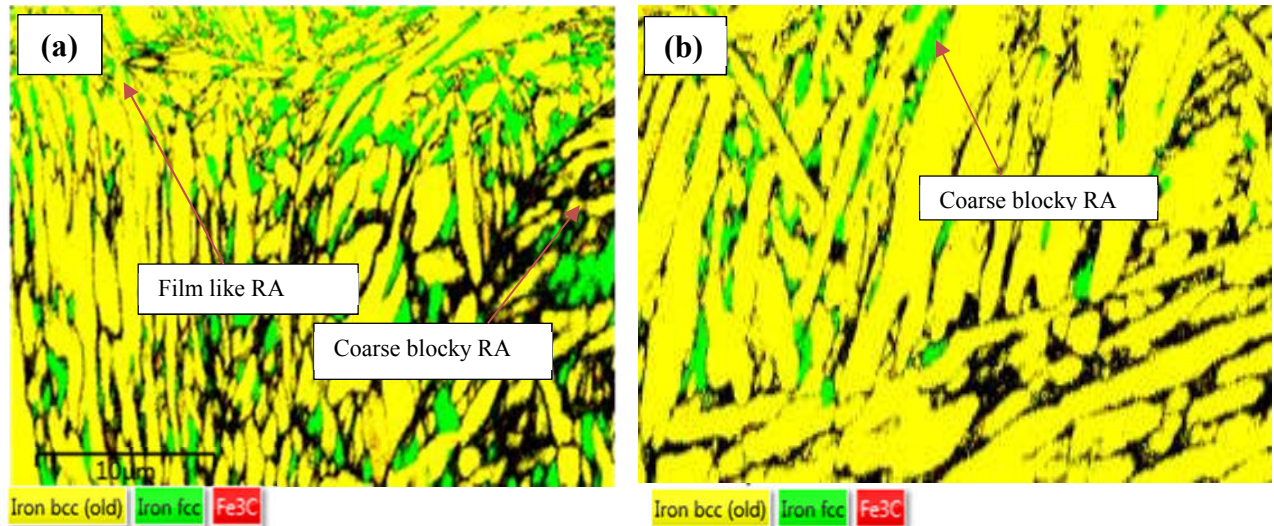


Figure 8. (a) The EBSD image of steel austenitized at 940 °C and quenched to 225 °C, (b) The EBSD image of steel partially austenitized at 800 °C and quenched at 143 °C

3.4 Mechanical properties

Vickers hardness test was done on all heat-treated samples. The average hardness values at different quench temperature under full and partial austenitization are shown in Figure 9.

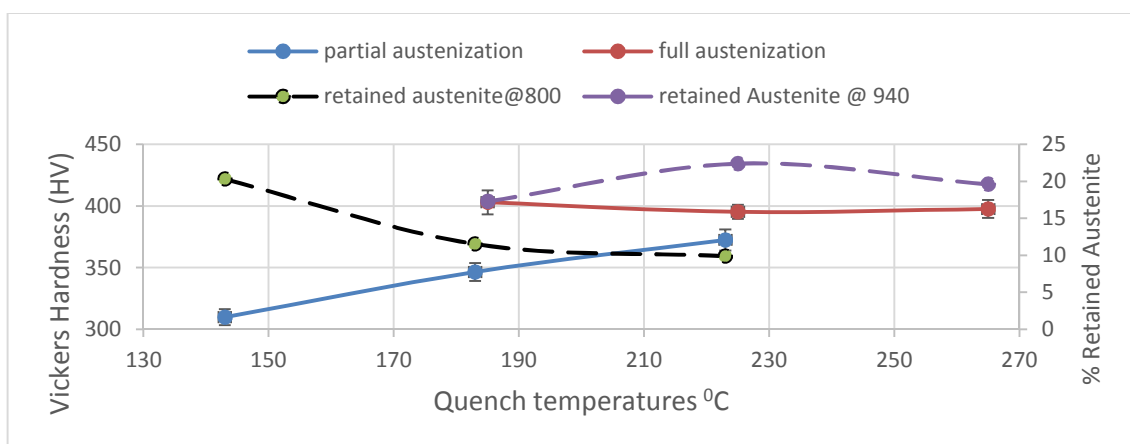


Figure 9. Hardness test at various quench temperature for the full and partial austenitisation heat treatment.

The expansion observed during partitioning in Figure 10 (a) is attributed to the phase transformation of the austenite (FCC) to bainite (BCC) after partial austenitization at 800 °C, quenching to 143°C and partitioning at 425 °C. During bainite formation, there is carbon rejection, which is taken up by the austenite phase, and this leads to the increased stability of the retained austenite. Evidence of bainite formation can be seen from the microstructure in Figure 11. After the final quench, no expansion is observed in Figure 10(a), which implies that no fresh martensite was formed.

No significant expansion was observed during partitioning in Figure 10 (b) after partial austenitization at 800 °C, quenching to 143°C and partitioning at 425 °C but the end of final quench shows expansion which is attributed to fresh martensite formation. This implies that the retained

austenite obtained during the first quench was not fully stabilized during partitioning. This unstable austenite finally decomposed into fresh martensite. Hence, the lower amount of retained austenite at 1830C than at 1430C. The same argument applies to the full austenitization, see Figure 10 (c).

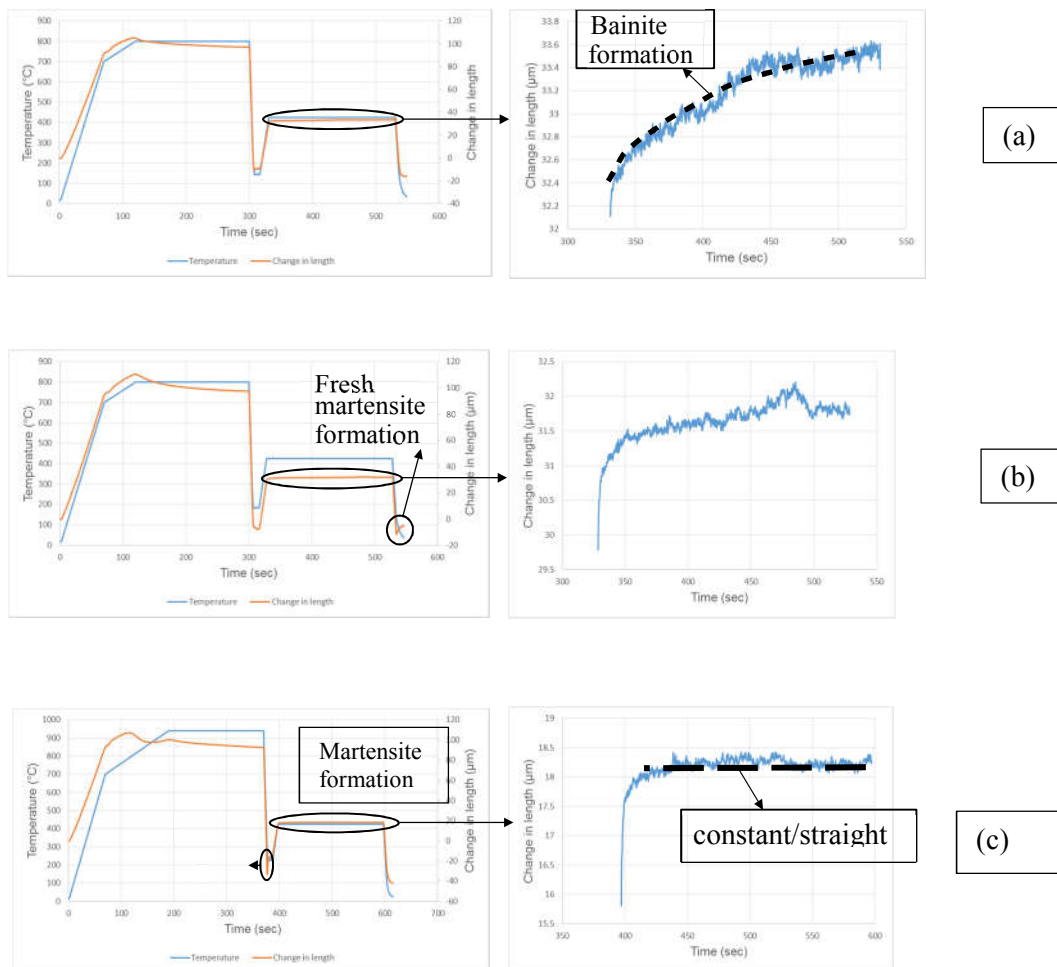


Figure 10. Q&P heat treatment (a) partially austenitized at 800 °C, quenched to 143^oC and partitioned at 425 °C, (b) partially austenitized at 800 °C, quenched to 183 °C and partitioned at 425 °C and (c) fully austenitized at 940 °C, quenched to 225 °C and partitioned at 425 °C.

4. Conclusions

- Q & P process increases the amount RA from 8 % in the as-cast alloy to 22 % in heat treated condition which shows that partitioning step help in stabilizing austenite. Full and partial austenitization before the Q&P heat treatment alters the morphology of martensite from thin lath-shaped to the coarser plate-like morphology because of the different initial carbon content in austenite.
- XRD results show maximum RA (22.4%) at 225 °C for full austenitization which confirms with optimum temperature calculation based on the CCE condition. The XRD results for the partial austenitization shows maximum RA (20.3%) at 143 °C instead of 183 °C as calculated from optimum temperature calculation. This deviation is because of bainite formation as seen from the thermal profiles of the different Q&P heat treatments.

- EBSD images show mainly thin film-like RA in case of full austenization while you have coarse blocky type RA after partial austenization. Since the size affects the stability of RA less RA is obtained in case of partial austenization
- In partial and full austenization, steel with maximum retained austenite has the lowest Vickers hardness, which would provide the maximum ductility along with strength.
- Full austenization is preferred over partial austenization for Q&P heat treatment due to the fineness in the microstructure and more amount of RA obtained

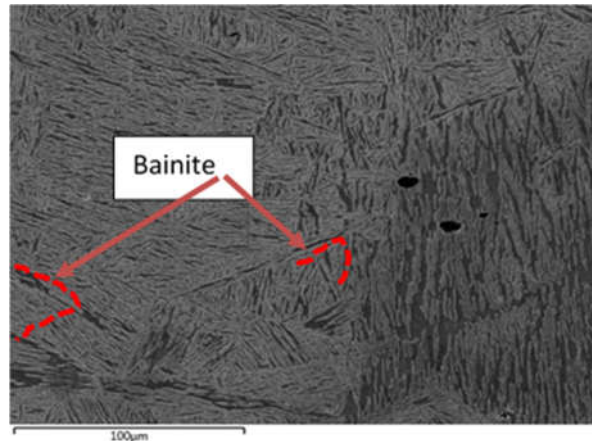


Figure 11. SEM image of sample quenched at 143 °C and partitioned at 425 °C

Acknowledgements

The authors would like to thank Ferrous Metal Development Network (FMDN), Mintek under Advanced Metal Initiative for financial support.

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