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Feasibility of 0.02% Nb-Based Microalloyed Steel for the Application of One-Step Quenching and Partitioning Heat Treatment

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Abstract

To attain an enhanced combination of mechanical properties for low alloyed steel, the current study has been made to fulfill that growing need in the industry. Its results are introduced within this paper. One step Quenching and Partitioning (Q&P) heat treatment has been applied on Niobium-based microalloyed steel alloy with 0.2 %C, in the form of 2 mm thickness sheets. The target of this study is to investigate the viability of applying that significantly recommended, results-wise, heat treatment on the highly well-suited alloy steel samples, to achieve the main target of enhanced properties. A single temperature of 275˚C was used as quenching and Partitioning temperature. Four Partitioning periods (30, 200, 500, and 1000 Seconds) were used for soaking at the same temperature. The results were analyzed in the light of microstructural investigation and mechanical testing. All applied cycles did not enhance the strength but moderately improved the ductility and toughness, mainly caused by the slightly high soaking temperature used. Niobium impact of grain refining was apparent through all cycles. The cycle of 500 Seconds Partitioning time obtained optimum values at that particular temperature. The 1000 Seconds Cycle obtained the worst combination of properties. A set of recommendations are set. More research is required at this point, where a lower Partitioning temperature is advised. In the light of the applied combination of parameters, the Partitioning period at such temperature is advised to be between 500 and 1000 Seconds. A high probability that periods closer to 500 than 1000 Seconds will produce better results. More research is needed between those two values of Partitioning time to precisely determine the optimum time at that temperature on that specific alloy.

Keywords

Nb-Based Microalloyed Steel, Advanced High Strength Steel, Quenching and

Partitioning, Retained Austenite, Martensite Transformation, Automotive Applications

1. Introduction

The steel-based industry in general and the Automotive industry always aspired to continuously enhance mechanical properties, such as strength, ductility, toughness, and other relevant properties. That combination of properties is the interpretation of the persistent need for better fuel efficiency, and higher passenger safety, which are the significant targets concerning Automotive industry development. There have been many trials within different research paths to take another step forward towards that aspiration.

The Advanced High Strength Steels (AHSS) are a product of that research. The use of AHSS has been progressively increasing, mainly in the Automotive industry, amongst others. The high fulfillment capability and promising potential of AHSS towards the industry's defined needs are the reason for that increase of use. More elaborately, using AHSS in the Automotive industry provided a combination of enhanced properties [1] [2], such as high strength, high formability (ductility), high capacity of energy absorption, lower vehicle weight, more economical fuel consumption, lower cost, enhanced crash resistance, and higher passenger safety. Based on that combination of privileges, AHSS are deployed within the Automotive industry in several places through the vehicle body [1] [2] [3], such as Body In White components (BIW), sills, reinforcements in the bumper, hood, doors, and other parts.

AHSS, as a family of steels, consists of three generations [2]. Each generation has its distinctive combination of characteristics. The first generation consists of several steel grades, such as Dual Phase (DP), Complex Phase (CP), and Transformation Induced Plasticity (TRIP) steels. The first generation is generally distinguished with low strength and ductility levels compared to the two other generations. The second generation of AHSS is featured by an enhanced combination of high strength and ductility with a downside of high cost and processing difficulty. Twinning induced plasticity (TWIP), and Austenitic stainless steel (AUST-SS), are two grades within the second generation AHSS.

On one hand, we have the first generation of AHSS with a good combination of mechanical properties; on the other, we have the second generation with a much superior combination, but with industrial limitations. The third generation of AHSS achieved the needed balance between acquiring enhanced mechanical properties and processing combination with more economical cost. Third-generation AHSS consists of several grades [3], such as medium-Mn steels and Quenched and Partitioned (Q&P) steel. Q&P steel is the main interest in the present work.

Attaining the desired enhanced combination of properties through Q&P is done by reaching a suitably ranged combination of Martensite phase, Ferrite phase, and enhanced levels of Retained Austenite. Achieving that combination is done by a specified sequence of thermal treatment. Q&P thermal treatment starts with proper steel Austenization. The material is heated (with a moderate rate) to a temperature above Ac_3 and held at that temperature for a suitable period according to the sample size. The next step is quenching to a temperature within the M_S-M_F range to get a Martensite and untransformed Austenite phase mixture mainly. Partitioning and final quenching to room temperature are the final steps in the Q&P thermal treatment sequence. The Partitioning step is done either at the same Quenching temperature, so-called One step Q&P, or at a higher temperature than Quenching temperature, so-called Two-step Q&P. Partitioning aims to stabilize the untransformed Austenite through carbon enrichment by partial carbon depletion of the Martensite phase and transporting carbon into untransformed Austenite, which increases the chemical stability of Austenite phase and eventually attaining enhanced levels of Retained Austenite at room temperature after the final quenching step in Q&P. Of the two sequences of Q&P, One step Quenching and Partitioning thermal treatment is the focus of the research in hand, because of its ease of application and higher cost-efficiency [3] [4].

For the first time, Q&P heat treatment was introduced through J. Speer *et al.* [5]. Through that work, the defined sequence of the treatment was set and explained. A thermodynamical model was also introduced to simulate the Q&P treatment on any suitable chemically alloyed steel to recommend the optimum conditions for performing the treatment. The model was built on the metastable equilibrium Constrained Paraequilibrium condition [6] and its relevant assumptions. The recommendation of the treatment conditions through the model is based on the optimum combination of phase fractions, which is determined at the endpoint of the treatment. The combination of inherited steel properties, and the microstructure, after the application of Q&P, was explained through earlier work [1] [7] [8].

Several research attempts have been made in the light of the proposed work of J. Speer *et al.* [5] and validated the proposed findings. A group of concepts was adopted to explain the results of applying the treatment. The chemical alloying role was highlighted [9] [10], especially silicon, Manganese, Aluminum, and several other elements. The focus was mainly on chemical elements' role in suppressing different reactions (carbides precipitation, mainly) competing against carbon enrichment from martensite into untransformed Austenite during the Partitioning stage. The role of Niobium (Nb) was highlighted in previous work [11] [12], and the focus was mainly on the grain refinement effect. Other concepts were highlighted, such as Partitioning kinetics [13], stability of Retained Austenite [14], effect of multiphase-microstructure and attained properties relationship [15], the effect of Partitioning temperature and time [16] [17].

Starting with the proven superior beneficial use of Q&P steel on an industrial scale [18] [19] as the main reason for pursuing this research. This study is focused on investigating the effect of applying the relatively economic One Step

Q&P heat treatment on the sheet-type samples of the well-suited chemically composed Microalloyed steel on hand [7] [20]. The Partitioning was applied at 275˚C, which is a relatively high temperature (under M_s) for better Partitioning kinetics, based on previous literature recommendations [20] [21]. A wide range of Partitioning time (30 - 1000 Seconds) was applied to investigate its effect on microstructure and mechanical properties development. Highlighting the effect of chemical alloying, especially Niobium (Nb), was the main interest. In the light of previously mentioned research and other documented work [22] [23] [24], the results were investigated using metallographic and mechanical testing. Means of light optical microscopy, and tensile testing were used.

2. Material and Methods

2.1. Material

The approximate chemical composition of the investigated alloy in the As Received condition is presented in **Table 1**. The chemical analysis was done using an optical emission spectrometer (Model: Spectro-Ametec, Germany, Technique: Hyper-PMT + CCD, Standard: ASTM A751-14a (ASTM E415-15)). This material is classified as hypo-eutectoid Micro-alloyed steel with low carbon alloying (0.192%) and other alloying elements, which in total follows the typical chemical composition ranges of Quenching and Partitioning steel, which grants this alloy the potential for attaining the characteristics of advanced high strength low alloyed steels and shows promise in resulting excellent enhanced combination of physical and mechanical properties after going through the novel heat treatment of Quenching and Partitioning. The material was received as rolled sheets of 2 mm thickness, 250 mm length, and 200 mm width. The used specimens to perform the applied One Step Quenching and Partitioning heat treatment cycles were cut from the basic received sheets dimensions to the standard sub-sized tensile specimen.

Critical transformation temperatures determination was vital to proceed with performing the heat treatment. Ac_1, Ac_3, and M_s, which are the main temperature of interest, were combinedly determined empirically through Equations (1)-(3) [25] [26] [27] [28], and Thermo-Calc Software, TCFE10 Steels/Fe-alloys database [29]. Eventually, the approximated values of Ac_1, Ac_3, and M_s, were calculated to be 720˚C, 1000˚C, and 403˚C, respectively.

$$\mathrm{Ac_1} = 742 - 29\mathrm{C} - 14\mathrm{Mn} + 13\mathrm{Si} + 16\mathrm{Cr} - 17\mathrm{Ni} - 16\mathrm{Mo} + 45\mathrm{V} + 36\mathrm{Cu} \tag{1}$$

$$\begin{aligned}\mathrm{Ac_3} = {} & 955 - 350\mathrm{C} - 25\mathrm{Mn} + 51\mathrm{Si} + 106\mathrm{Nb} + 100\mathrm{Ti} + 68\mathrm{Al} \\ & - 11\mathrm{Cr} - 33\mathrm{Ni} - 16\mathrm{Cu} + 67\mathrm{Mo}\end{aligned} \tag{2}$$

$$\mathrm{Ms} = 565 - 31\mathrm{Mn} - 13\mathrm{Si} - 10\mathrm{Cr} - 18\mathrm{Ni} - 12\mathrm{Mo} - 600\left[1 - \exp\left(-0.96\mathrm{C}\right)\right] \tag{3}$$

Table 1. Chemical composition of the investigated alloy (wt%).

C	Nb	Si	Al	Mn	Cr	Ni	V	Ti	Mo	Cu
0.192	0.021	0.686	1.47	1.62	0.039	0.031	0.0058	0.0063	0.0031	0.031

2.2. Defined Thermal Profile Design of the Applied One-Step Q&P Cycles

Figure 1 demonstrates the sequence of the Four different One Step Q&P heat treatment cycles conducted on the investigated alloy specimens. First, the sub-sized steel tensile specimen was heated to an Austenization temperature of 1100˚C for 20 minutes. The specimen was then quenched to a temperature under Ms of 275˚C in a salt bath furnace and then soaked in the salt bath at the same temperature for a period known as the Partitioning step. Four different periods (30, 200, 500, and 1000 Seconds) were selected and applied as Partitioning periods. The quenching to room temperature in distilled water was the final step of all applied cycles. The used specimens to perform the applied One Step Quenching and Partitioning heat treatment cycles were cut from the basic received sheets dimensions to the standard sub-sized tensile specimen. The sub-sized specimen was cut from the As Received sheets using a computer numerically controlled water jet machine, which provides high accuracy cutting with negligible heat so that the specimen's microstructure cannot be affected or altered.

2.3. Metallographic Examination

Small samples were cut for microstructural investigation from a specific rectangular piece attached to the tensile specimens on which each cycle was applied. Standard steps of microstructure samples preparation of grinding, polishing, and 2% Nital etching (5 - 10 Seconds), were applied, followed by an inverted type of light optical microscope. The As Received sample went through the same metallographic preparation methodology.

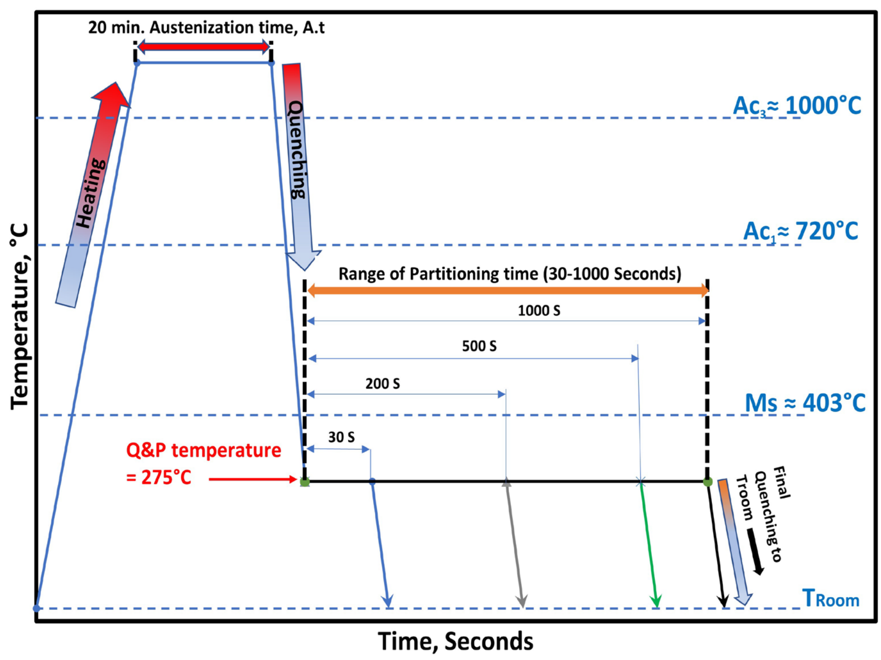

Figure 1. Thermal profile schematic of the performed one step Q&P heat treatment cycles.

2.4. Tensile Testing

Mechanical uniaxial tensile testing was performed on all sub-sized tensile specimens of all applied quenching, and Partitioning heat treatment cycles besides the As Received tensile specimen. As mentioned before, the tensile specimens were cut before the application of heat treatment cycles. They were cut to a profile, which follows the Standard (ASTM-E8/E8M) [30] for a plate-type test. Each specimen had the As Received thickness of 2 mm. The overall length is 110 mm. Each grip section was 35 mm long and 10 mm wide. The radius of each fillet was 6.37 mm. The gauge length was 25 mm long and 6 mm wide (as shown in **Figure 2**). All tested tensile specimens were submitted to the same tensile testing conditions.

3. Results and Discussion

3.1. Metallographic Investigation

Figure 3 demonstrates the optical micrographs of As Received condition and developed Q&P steel one step partitioned for different times of 30, 200, 500, and 1000 Seconds. The As Received sample was composed of martensite (black area), Ferrite (white area), and a percent of Bainite. The heat-treated specimens' microstructure has composed of tempered Martensite (black area), Ferrite (white area), and predicted Retained Austenite (γ_r) (a light gray area), and a percent of carbide-free Bainite (α_b) (a dark gray area). The optical micrographs also indicated that increasing Partitioning time from 30 to 1000 Seconds displayed a slight change in the morphology and size of fine coexisted phases. In comparison, the long Partitioning time of 1000 Seconds resulted in an increasing amount of Martensite. The formation of the carbide-free Bainite phase in this microstructure may be attributed to the slow decomposition of (ε) carbide in the Martensite during the Partitioning stage, leading to incomplete carbon diffusion into untransformed Austenite. As a result, a small fraction of Bainite or bainitic Ferrite is created in the final microstructure with Retained Austenite and tempered martensite [31]. The optical micrographs of this investigated Q&P steel demonstrated a general overview of the microstructure.

3.2. Tensile Testing Results

Figure 4 demonstrates the engineering Stress-Strain curves of heat-treated

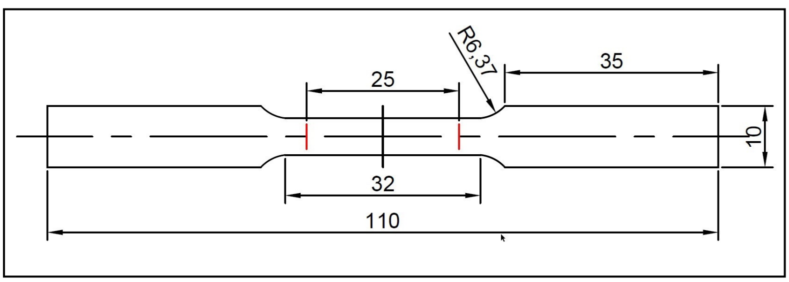

Figure 2. Schematic of the used standard sub-sized tensile test specimen.

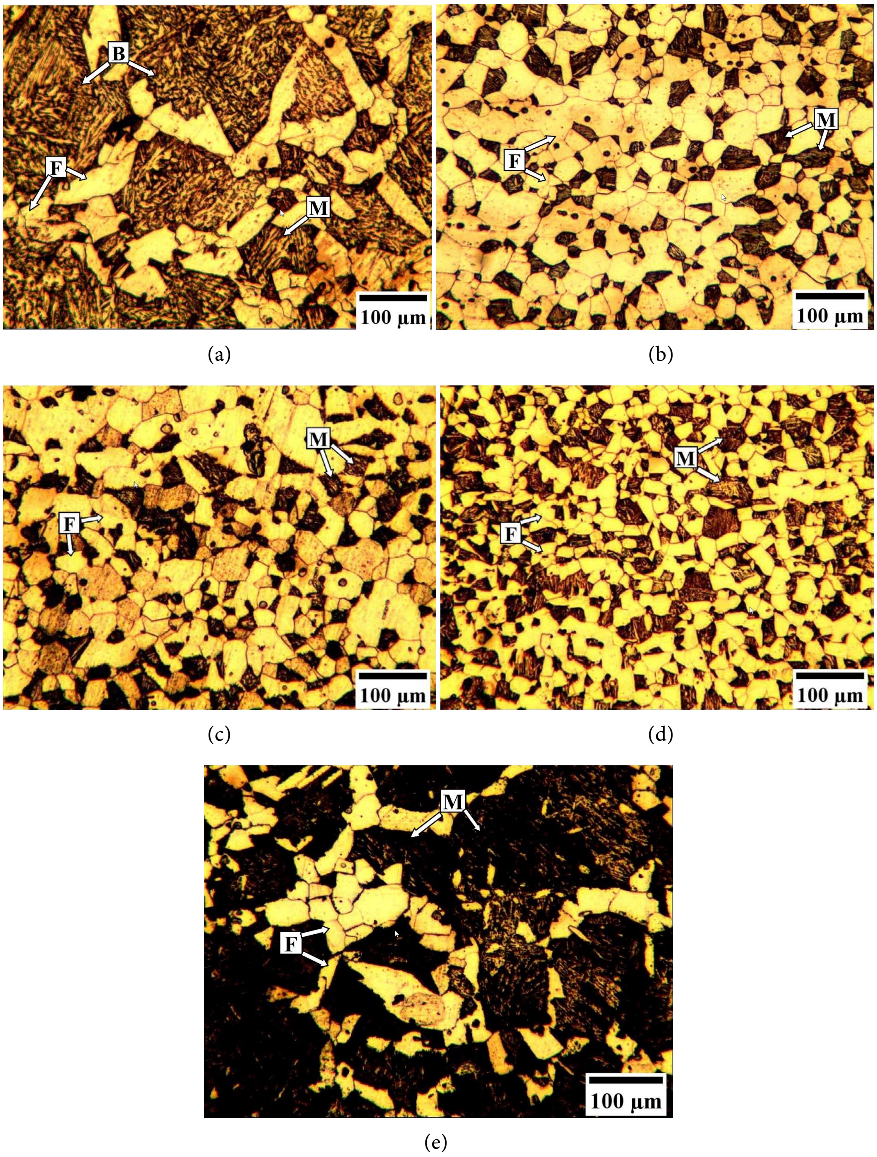

Figure 3. Optical micrographs of the As received condition and the one-step heat-treated samples at 275˚C, for different Partitioning periods; (a) As-Received, (b) 30 Seconds, (c) 200 Seconds, (d) 500 Seconds, and (e) 1000 Seconds.

specimens, and the As Received condition. As shown in this figure, all the heat-treated specimens exhibited continuous yielding behavior. This behavior may be attributed to some nitride-forming elements such as Al and Si, which decrease Carbon and Nitrogen interstitial atoms that impede dislocation movement [32]. Consequently, the mobile dislocation density is increased [32].

Table 2 demonstrates a combination of measured mechanical and microstructural properties of the As Received condition and the applied heat treatment cycles. The focus was on mechanical properties such as Yield Strength (Y.S), Ultimate Tensile Strength (U.T.S), Ductility or total percentage elongation (EL.%), Yielding ratio (Y.S/U.T.S), and the multiplication of U.T.S and Ductility as an indication of Toughness (T). The measured microstructural data were Ferrite

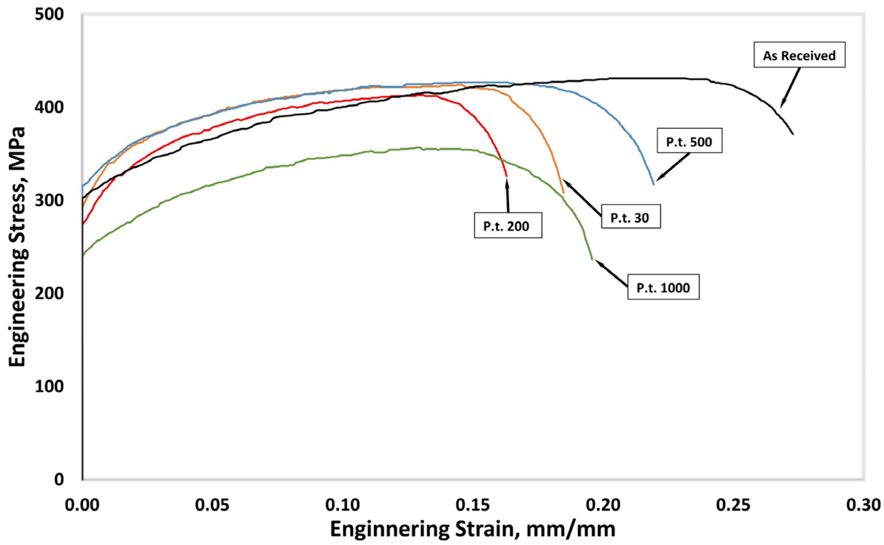

Figure 4. Engineering stress-strain curves of the As received sample and investigated steel alloy partitioned at 275˚C temperature for different time cycles.

Table 2. Measured values of mechanical and microstructural properties of the As received sample and investigated steel alloy partitioned at 275˚C temperature for different time cycles.

Sample	Y.S	U.T.S	EL.%	Y.S/U.T.S	T	F.G.S	M.G.S	M.%
As Received	310.0	431.0	15.5	0.72	6680.5	81.3	--	--
P.t.30 S	293.4	423.9	16.2	0.69	6867.2	49.7	49.5	40.2
P.t.200 S	274.8	413.6	16.7	0.66	6907.5	56.5	49.6	33.6
P.t.500 S	316.0	426.9	18.7	0.74	7983.0	34.9	51.5	33.5
P.t.1000 S	238.8	357.3	19	0.67	6788.9	49.9	112.2	80.1

phase grain length as an indication of its size (F.G.S), Martensite phase grain length as an indication of its size (M.G.S), and Martensite phase volume fraction (M%).

Figure 5 shows that U.T.S and ductility are inversely proportional through the different applied Partitioning times, except 500 Seconds. Mechanical properties of different cycles show clearly that only ductility was enhanced compared to As Received condition in general, with a directly proportional relationship with increasing Partitioning time. As for U.T.S, the applied cycles of Q&P did not provide higher values than the As Received condition. As for Y.S, the only enhanced case was 200 Seconds Partitioning time, with a slight increase of 6 MPa compared to As Received condition. From the Yielding Ratio point of view, only 500 Seconds of Partitioning time improved the ratio compared to As Received condition, increasing 2%. Using the multiplication of (U.T.S * EL.%) as an indication of the material Toughness, specific results were found. All cycles increased the material Toughness compared to the As Received condition, with the directly proportional relationship with increasing Partitioning time till 500 Seconds.

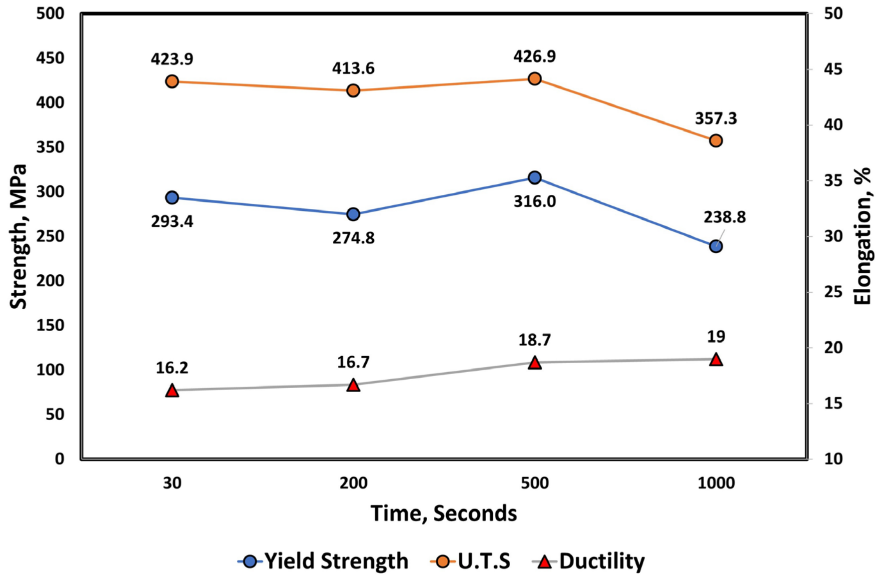

Figure 5. Combination of yield strength, ultimate tensile strength, and ductility values of the investigated steel alloy partitioned at 275˚C temperature for different time cycles.

As for 1000 Seconds, the Toughness started to decrease like U.T.S, Y.S, and Yielding Ratio.

The tensile strength does not depend on Partitioning time until 500 Seconds; then, it has decreased from 427 MPa at 500 s to 357 MPa at 1000 Seconds, while the total elongation (EL.%) has increased with increasing Partitioning time. The developed Q&P steel has exhibited maximum elongation and strength-elongation balance (Toughness) of about 19% and 7983 MPa% with an ultimate tensile strength value of 427 MPa when partitioned for 500 Seconds.

The grain refinement effect is clearly shown through all applied cycles, where the impact of the heat treatment itself and Niobium microalloying [11] [12] are predicted to be the leading cause. Stating the fixed value of Niobium in all applied cycles, the change in Ferrite grain size is linked to the changing Partitioning time, as shown in **Figure 6**. The Ferrite grain size is effectively refined through all applied cycles to near values, but with small deviations. A predicted justification is introduced to describe those deviations. First, as the Partitioning time increases, Ferrite grain length increases. Then the grain length decreases as the time keep increasing. Silicon is a Ferrite stabilizer and also refines the Ferrite grain size. The decrease in Ferrite grain length after 200 Seconds, is predicted to be due to the emerging effect of Silicon, as a result of increased Partitioning time (500 Seconds), and increasing Austenite nucleation rate. However, continuously increasing Partitioning time (up to 1000 Seconds) leads to grain coarsening again. Using the Ferrite grain length as a reference, cycles with 500 Seconds and 200 Seconds had the highest and lowest grain refining impact, respectively.

Martensite grain size as a reference shows the semi-constant impact of different cycles till 500 Seconds Partitioning time, with the apparent exception of cycle 1000 Seconds Partitioning time, as shown in **Figure 7**. Similarly, Martensite

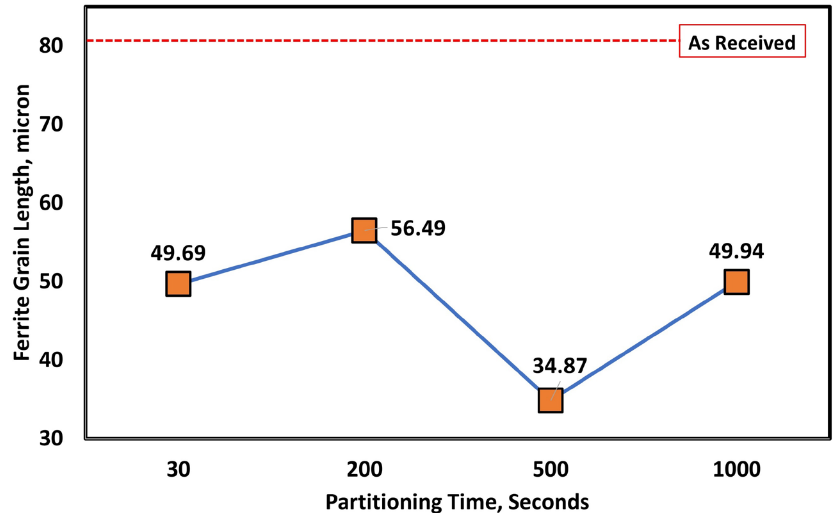

Figure 6. Ferrite grain length of the investigated steel alloy partitioned at 275˚C temperature for different time cycles.

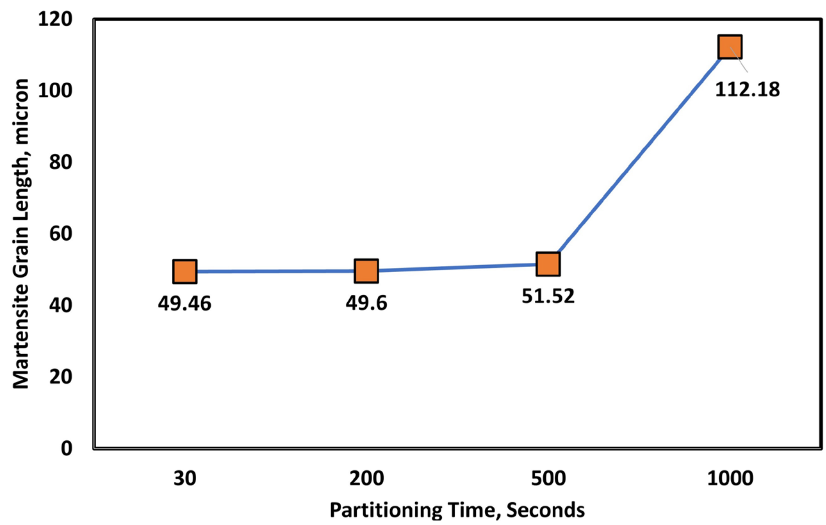

Figure 7. Martensite grain length of the investigated steel alloy partitioned at 275˚C temperature for different time cycles.

volume fraction as a reference shows a semi-constant impact till 500 Seconds Partitioning time, with a slightly higher increase in the 30 Seconds cycle, which is logically justified due to the shortest applied Partitioning period. The frequent pattern of obviously anomalous impact of 1000 Seconds Partitioning time is clearly shown with the highest Martensite volume fraction provided, as shown in Figure 8. The high amount of martensite is justified due to combining two different types of existing Martensite due to the high applied Partitioning time. The first type is the initial Martensite due to the first Quenching stage. The second type is Fresh Martensite due to the long Partitioning period at a relatively high temperature (275˚C), which could cause the decomposition of the already stabilized Austenite grains into other microconstituents, to be eventually transformed into Fresh (Twinned) Martensite upon Final quenching stage [7] [20].

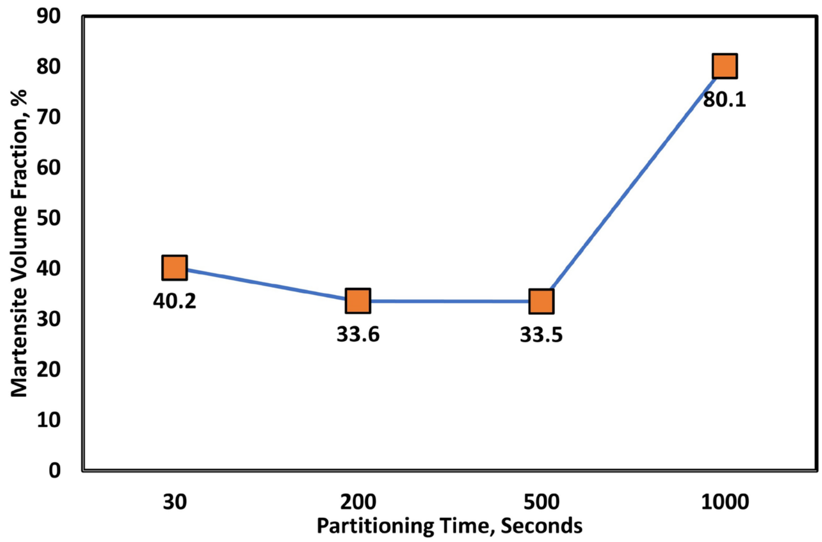

Figure 8. Martensite phase volume fraction of the investigated steel alloy partitioned at 275˚C temperature for different time cycles.

As another summarization of the results in the light of microstructural investigation, all applied Partitioning periods, with Niobium alloying effect as a constant helping factor, had nearly the same beneficial effect of grain refining, and subsequentially increasing strength, except for 1000 Seconds Cycle. The exact impact is repeated in resulted Martensite volume fraction, with 1000 Seconds similarly excluded.

4. Summary and Conclusions

In this work, heat treatment cycles of 0.2 C-1.62 Mn-0.69 Si-1.47 Al-0.021 Nb, Wt% steel alloy were performed to determine heat treatment conditions for developing a novel Q&P steel. The heat treatment process comprised Quenching and Partitioning at 275˚C for different times (30 up to 1000 Seconds). The results showed that:

- The heat treatment process of studied Q&P steel partitioned at different times resulted in Martensite, Retained Austenite, and carbide free Bainite in the microstructure. A slight difference in the microstructure has been observed at Partitioning time from 30 to 1000 Seconds.
- Concerning mechanical testing, all the applied partitioning periods had nearly the same effect till the 500 Seconds Cycle. Compared with the received condition, all applied cycles did not enhance the strength but increasing ductility.
- The developed Q&P steel has exhibited maximum elongation and strength-elongation balance of about 19% and 7983 MPa% with an ultimate tensile strength value of 427 MPa when partitioned for 500 Seconds.
- The volume fraction of martensite has increased with increasing Partitioning time due to combining two different types of existing Martensite due to the high applied Partitioning time.

- Concerning the applied combination of parameters, the Partitioning period at such temperature is advised to be between 500 and 1000 Seconds, with the high probability that periods closer to 500 than 1000 Seconds will produce better results.
- More research is needed between those two values of Partitioning time (500 and 1000 Seconds) to precisely determine the optimum time at that temperature on that specific alloy.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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