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The Influence of Cooling Rate between M_s and M_f on the Mechanical Properties of Low Alloy 42SiCr Steel Treated by the Q-P Process

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Abstract: A series of experiments was conducted by quenching and partitioning (Q-P) heat-treated alloys to investigate the effect of cooling intensity on the mechanical properties of low alloy steel 42SiCr. By applying a conventional heat treatment, reasonable high strength can be achieved; however, the alloys become more brittle. To obtain an optimal balance, advanced heat treatment methods like the Q-P process can be used. It consists of quenching to temperatures between martensite start and martensite finish temperatures and holding, which leads to the stabilization of untransformed austenite by carbon partitioning. The martensitic microstructure is then formed with a small volume fraction of retained austenite embedded on a microscopic scale. The material's deformability can be significantly improved by using such heat treatment processes. Moreover, to improve advanced high strength properties (AHSS), an additional Q-P process can be applied, which leads to erasing the influence of cold forming as well as enhancement of the mechanical properties. Several combinations of the Q-P process with/without partitioning were performed with various cooling rates for both heat treatment methods. Ultimate Tensile Strength (UTS), Ductility and Hardness (HV10), as well as the microstructure of the alloys, are compared to evaluate the cooling intensity effects. The cooling rate is found not to be a significant factor influencing mechanical properties, which is a crucial point for practical material heat treatment.

Keywords: 42SiCr; quenching and partitioning; cooling intensity; advanced high-strength steel; mechanical properties; microstructure; quenching rate

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1. Introduction

A sustainable approach requires the development of new or modified steel grades which can lead to advanced modifications of heat and other treatments resulting in weight saving, fuel economy, and higher mechanical performance. In recent decades, the increased safety and reliability of structures and components exposed to different types of static and cycling service loading has been in high demand, which has pushed researchers to modify steel alloys to match environmental and economic demands as well as elevated mechanical properties [1]. This approach was activated in 2003 when several researchers found it effective in generating microstructures containing retained austenite (RA) [2–6]. The potential for the best combination of strength and ductility is due to the presence of RA, which can be achieved via deformation-induced processes like the transformation-induced plasticity (TRIP) effect during straining. The investigation of Q-P processing was later extended to carbon-containing steels. This starts with the partial transformation of austenite to á′-martensite by quenching austenite to a temperature between martensite start (M_s) and finish (M_f). In the next step, partitioning is activated by holding at a temperature equal to or higher than the quenching temperature (QT). This allows diffusion of carbon from supersaturated martensite into austenite, leading to RA stabilisation [7]. This method is widely used for carbon and low-alloyed steels [8,9], while research on stainless steels is gradually growing [10–12].

Generally, the Q-P process comprises four successive steps: annealing, quenching, partitioning, and final cooling. In the initial steps (annealing and quenching), the steel undergoes austenitisation (full or partial), which is also called annealing. It is then quenched to a temperature (QT) between martensite start temperature (M_s) and martensite finish temperature (M_f), during which the austenite partially transforms into martensite (quenching). Then the steel is held at QT or can be heated to a temperature (PT) higher than the QT. This allows the enrichment of carbon (C) in the austenite by redistribution from the surrounding martensite (partitioning). Finally, the steel is cooled to room temperature (final cooling). During this last step, additional fresh martensite may be formed if the C enrichment in the austenite is insufficient to ensure its thermal stability.

There are several advantages of using Q-P heat treatment on a 42SiCr steel in a flexible manufacturing chain with integrated incremental bending for the safe production of parts [13]. The authors and their partners have been investigating the feasibility of Q-P steels for several years. The first results shows that it is possible to reach the necessary cooling rates for the Q-P steel in the Hot Metal Gas Forming (HMGF) process [14]. The fatigue strength of 42SiCr steel tubes with a high wall thickness of 4 mm was assessed while the potential of structural Q-P steel parts under cyclic loading caused by HMGF is demonstrated by the fatigue strength of over 1350 MPa (R = 0.1) [15]. Ostash et al. studied the influence of various modes of heat treatment by Q-P treatments on the mechanical properties of 65G steel, which is regarded as a model wheel steel. They showed that after the modified Q-P the mechanical characteristics of the steel improved to a greater extent than after traditional heat treatment. It was shown that by using Q-P the relaxation of stresses of the second kind in the bulk of martensite and bainite lathes takes place and leads to better mechanical properties and improved cyclic fracture toughness [16]. Jirková et al. (2019) examined the effects of various processing methods and partitioning times on a 42SiCr sample with a wall thickness of 4 mm and a simple geometry [17]. It became clear that the cooling rate attained in the tubes during the HMGF process is insufficient to achieve high strength and high ductility due to the high wall thickness. A hardness of 450–550 HV10 was achieved in these cases. However, our findings unequivocally demonstrate that in order to fully utilise Q-P heat treatment in a forming process, knowledge of the local cooling rates over the entire part and their impact on the microstructure is required. Because of this, the current study aims for the first time to study the effect of the various local cooling rates on the quenching process to temperatures between martensitic temperature start (M_s) and martensitic temperature finish (M_f) in a Q-P treated 42SiCr steel. Additionally, it is shown that local variations in mechanical characteristics and microstructures rely on the area's temperature history. Thus, it is possible to clearly identify the cooling rates required to utilise the full capability of Q-P treatment in industrial forming processes. To support the previous findings, the partitioning effect is also studied in combination with different cooling intensities.

2. Experimental Procedure

2.1. Material Preparation

The low alloy 42SiCr steel is suitable particularly for manufacturing transport vehicle components like shafts, pins, screws or springs. The chemical composition is mainly 0.42% C, 2% Si, 0.59% Mn and 1.33% Cr (weight%). The increased level of Si leads to better mechanical properties than similar steels in this category. This allows the reduction of carbide precipitation and enables diffusion of carbon to residual austenite. Moreover, the manganese improves the carbon solubility in austenite, leading to the occurrence of pearlite [18]. Thus, the material's composition provides reasonable cold-forming properties within the initial pearlitic state and subsequently enables the phase transformations of the Q-P heat treatment.

Previous research suggested a carbon content of 0.42%, stabilising the austenite phase during partitioning [19–21]. Mainly, the carbon strengthens the martensite phase and increases the stability of austenite. However, this improvement follows from the supersaturated martensite due to incomplete quenching. That is why the suggested amount of manganese should be used for the austenite stabilisation in the present alloying composition of 42SiCr with outstanding properties. The chemical composition of 42SiCr steel alloy used in this study, as well as the calculated carbon equivalent (CEV), are presented in Table 1.

Table 1. Chemical composition and CEV of 42SiCr steel used in this work.

Element	C	Si	Cr	Mn	Fe	CEV
wt%	0.42	2.0	1.3	0.68	Bal.	0.82

As described earlier, the material contains silicon to protect the carbon from cementite forming. Moreover, it stabilises the requested austenite phase. In the same way as manganese, silicon increases both the final product's strength and ductility. The last element, chromium, is mainly used to improve hardening by lowering the martensite formation temperature to within a reasonable range [22,23].

In the present study, the as-received material was cast and hot rolled to a thickness of 10 mm. Then it was reduced to 2.5 mm by cold rolling with interstage annealing followed by grinding to 1.5 mm. This allowed the material to form a pearlitic microstructure with a low fraction of ferrite. In this way, the alloy reaches a sufficient ductility for cold forming. Finally, specimens of a gauge length of L0 = 20 mm are cut using a waterjet for tensile testing according to DIN 50125.

2.2. Q-P Treatment

The Q-P heat treatments are performed to regenerate the plastic deformability and improve the mechanical properties of the alloy. The Q-P treatment is used to develop Advanced High Strength Steel (AHSS) material characteristics and improve the material's ductility. The primary function of Q-P processing is to transform the initial pearlitic microstructure into a predominantly martensitic microstructure including a certain fraction of retained austenite. Several Q-P processes are applied to investigate each parameter's effect on the material's mechanical properties.

All the samples are annealed to 950 °C to reach austenitization. Then each sample is quenched individually to a quenching temperature QT slightly higher than the finish M_f temperature to retain an increased part of the austenite. The martensitic transformation phase usually starts at M_s = 298 °C for 42SiCr steel, while M_f is around 178 °C [24,25]. Based on the authors experience with this alloy, over 90% of the transformation is completed at about 200 °C, so the QT is considered as 200 °C for this study. Then the specimens are re-heated to the partitioning temperature PT and held for the partitioning time t_p. This allows redistribution of carbon elements to stabilise the retained austenite.

The cooling intensity variation from 1.56% to 200% is investigated to focus on the cooling rate between M_s and M_f. To cover this range, over 300 samples are tested and the most significant intensities are summarized in Table 2 showing the variation of Q-P parameters used in this study. The cooling intensity is expressed by an increase or decrease of the cooling time and it is compared to the standard rate of 20 s corresponding to 100% cooling intensity (Figure 1). Figure 1 shows the scheme of the Q-P treatment where:

M_s is the martensitic start temperature
M_f is the martensitic finish temperature
QT is the quenching temperature
PT is the partitioning temperature
t_h is the heat-up time
t_p is the partitioning time

Table 2. Q-P process parameter.

No.	QT [°C]	PT [°C]	t_p [s]	Cooling Intensity [%]	Cooling Time [s]
QP1	200	250	600	100	20
QP2	200	250	600	200	10
QP3	200	250	600	50	40
QP4	200	250	600	25	80
QP5	200	250	600	12.5	160
QP6	200	250	600	6.25	320
QP7	200	250	600	3.12	640
QP8	200	250	600	1.56	1280
QP11	200	RT	-	100	20
QP22	200	RT	-	200	10
QP33	200	RT	-	50	40
QP44	200	RT	-	25	80
QP55	200	RT	-	12.5	160
QP66	200	RT	-	6.25	320
QP77	200	RT	-	3.12	640
QP88	200	RT	-	1.56	1280

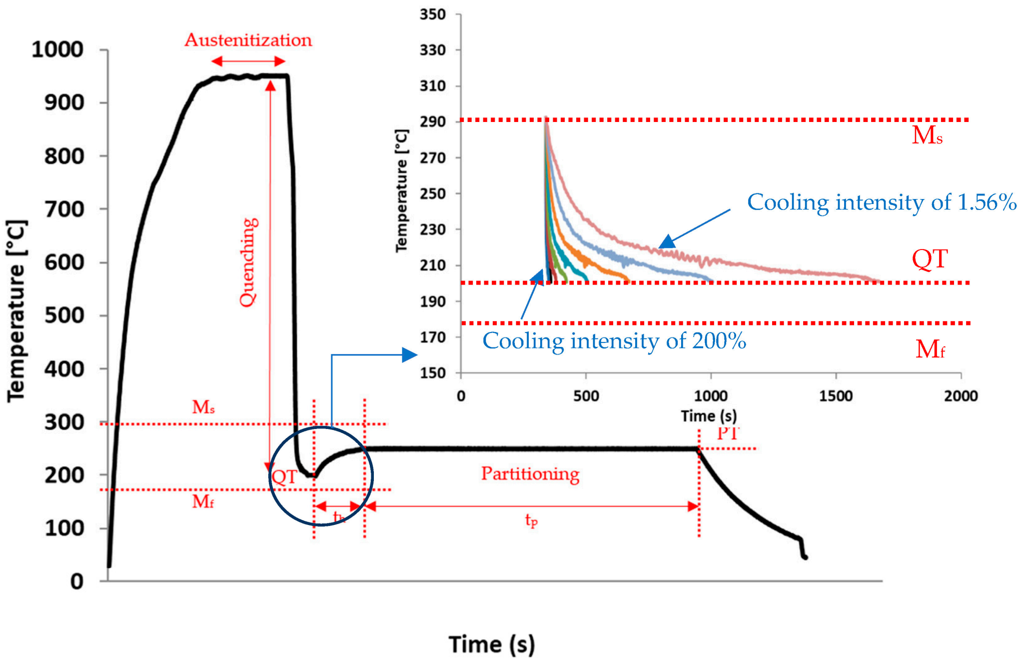


Figure 1. Schematic illustration of the Q-P process as a qualitative temperature over time plot.

2.3. Sample Preparation

To optimise the sample preparation process, standard tensile samples (gauge length 20 mm, width 2 mm, and average thickness 2.5 mm) are prepared to fit in the tensile-test machine. The samples are cut in a longitudinal direction (parallel to the rolling direction), and the thickness of the specimens is approximately 1.5 mm after grinding.

2.4. Testing Equipment and Procedure

The mechanical tests are conducted using a UHL/VMHT hardness tester (Walter Uhl, Asslar, Germany) and a servo-hydraulic MTS thermomechanical simulator (MTS, Minnesota, MN, USA). Custom clamps are manufactured (UWB, Pilsen, Czech Republic) to hold the samples on the servo-hydraulic MTS. The strain rate of all the tensile tests is 1×10^3 s^{-1}. The average values of the ultimate tensile strength (UTS) and ductility (A) are statistically determined after testing three samples from each batch. The polished sample heads are subjected to hardness testing with a 10 kg weight and 11 s loading period. Three measurements are used to calculate the average value. Samples are prepared from the heat treated materials for metallographic analysis. A scanning electron microscope (SEM), Tescan LYRA 3 (Tescan, Brno, Czech Republic), is used for the metallographic analysis of the samples. All the samples are prepared using standard grinding and polishing processes. The last polishing step is performed with oxide polishing suspension (OPS). To characterize the rather fine microstructure, a magnification of 5 k× is used.

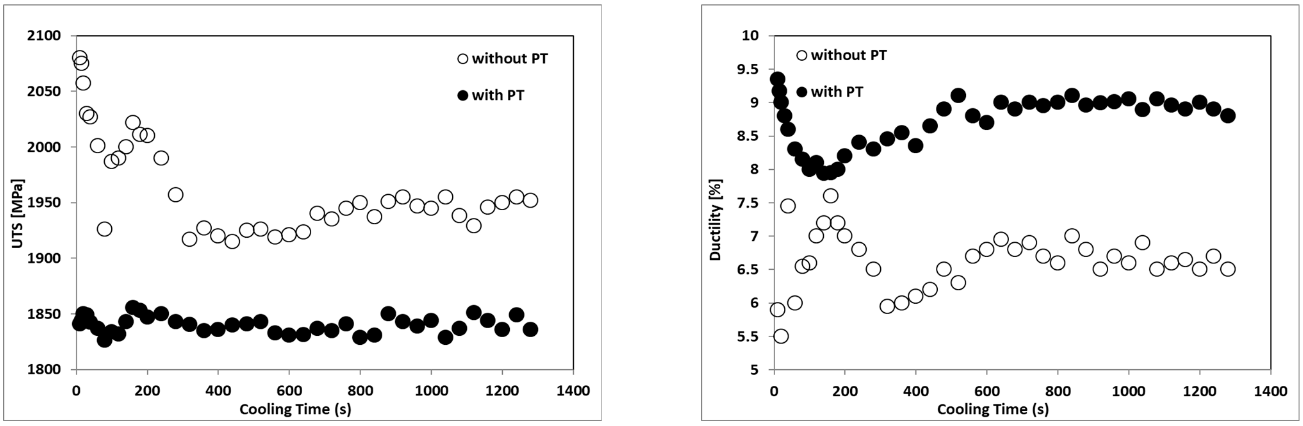
3. Results and Discussion

As mentioned above, the application of the unique Q-P process strengthens the steel and keeps its ductility within a reasonable region. This characterization is achieved by varying the cooling rate between M_s and M_f. Based on the previous research [26–28], the temperatures M_s and M_f achieve the values of around 300 °C and 200 °C respectively. The cooling intensity is changed to observe its influence on the mechanical properties.

The thickness of the component or, more generally, the cooling rates, must be taken into consideration in any forming process with combined Q-P treatment. For example, thinner wall thicknesses would experience quick quenching and, as a result, a fast drop to QT temperature. As a result, hardly any partitioning occurs. On the other hand, for thick walls the cooling rates drastically slow down and partitioning is allowed. In these cases, the Q-P treatment is necessary to unify the mechanical properties of the part.

3.1. Mechanical Properties

Figure 2 shows the ultimate tensile strength (UTS) and ductility obtained from the tensile tests for various cooling intensities subjected/not subjected to partitioning. The ultimate tensile strength (UTS) values are defined as the stresses corresponding to the highest point of the stress-strain curve. The UTS is usually characterized as the ability of a material to resist loading due to tension. This parameter applies to all types of materials, thus only UTS is used as a comparative parameter for this study.



(**a**) (**b**)

Figure 2. (**a**) Ultimate Tensile Strength and (**b**) Ductility versus cooling time.

A general overview presented in Figure 2 demonstrates that the partitioning process only slightly decreases the UTS while significantly increasing the ductility. The non-partitioned component exhibits the highest UTS, as expected. The UTS of 2057 MPa and corresponding ductility of about 5.5% is achieved for 100% cooling intensity (cooling time 20 s). With the highest cooling intensity (the shortest cooling time of 10 s), the material without the partitioning process shows the highest UTS of 2080 MPa and slightly higher ductility of 5.8%. A certain optimum of the cooling intensity can be found for the cooling times between 150 and 200 s, when both the UTS and the ductility obtain local maxima. Cooling times between 300 and 400 s should be avoided, as both the UTS and the ductility achieve local minima.

The material that was partitioned for 600 s at 250 °C has an approximate UTS of 1841 MPa, independent of the cooling time. With respect to the ductility, cooling times between 150 and 200 s should be avoided. It is generally accepted that internal stresses in the martensitic microstructure are reduced by carbon diffusion from quenched martensite into the remaining austenite, slightly reducing yield strength but significantly enhancing ductility. The partitioning is also linked to the nucleation and development of a larger number of carbides, which subsequently impair ductility. In terms of a proper balance between strength and ductility, some short partitioning times undoubtedly produce useful results. It should also be taken into consideration that a shorter partitioning time is more energy-efficient than a longer one.

As explained earlier, the main task of partitioning is to increase the ductility, which usually corresponds to a slight decrease in the UTS. As a result, the UTS shows a decrease of 5%, while the ductility shows a 30% increase. Furthermore, the UTS is not dependent on the cooling rate, thanks to subsequent partitioning, even at the higher cooling intensity.

Figure 3 shows data comparing both UTS and ductility in dependence on the cooling intensity. 100% cooling intensity is taken as a reference condition for comparison of all the other tests and it corresponds to 20 s of cooling between M_s and M_f. The reference condition (100% cooling rate) is dotted in the graph to distinguish it from the other data. It can be seen from Figure 3a that the change of the cooling intensity has only a slight impact on the UTS. It varies within 7% from 2057 MPa to 1917 MPa without partitioning and there is almost no change after partitioning. The high value of UTS can be attributed to the formation of lath martensite. For a low alloy steel with a carbon content below 1%, the formation of lath martensite is predictable, but it significantly depends on the quenching rate influencing the fraction of the retained austenite. Further discussion dealing with the microstructure is presented in the next section.

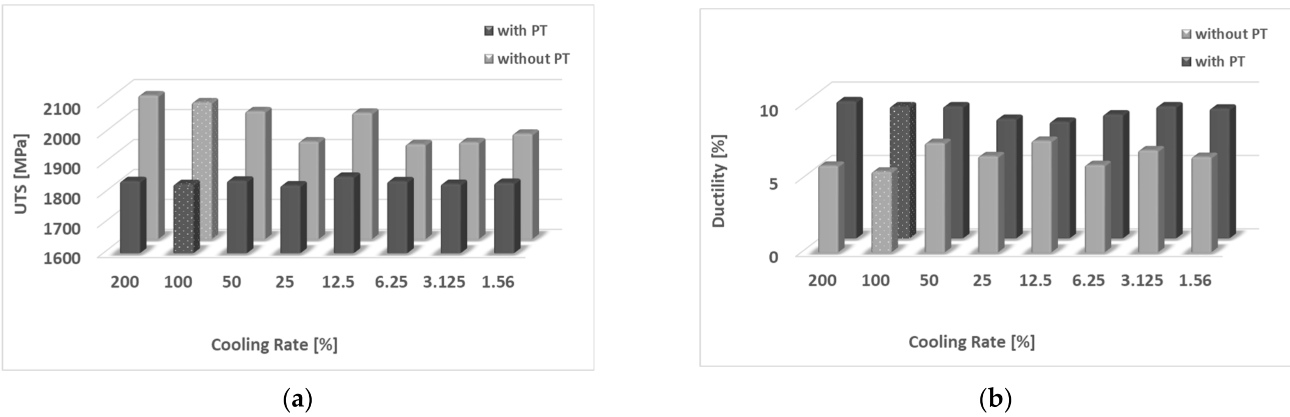


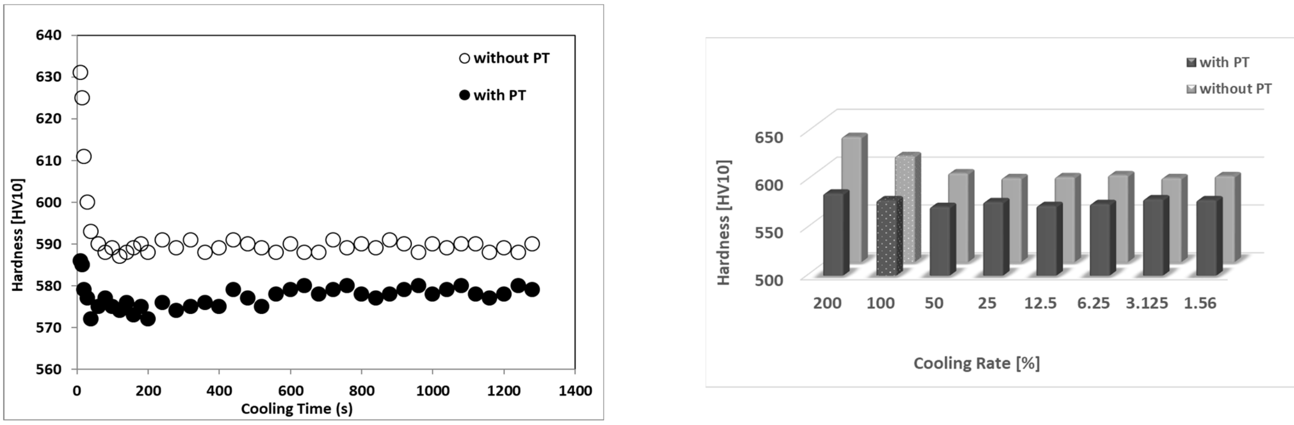
Figure 3. (**a**) Ultimate Tensile Strength and (**b**) Ductility versus selected cooling rate.

Figure 3b compares the ductility values for different cooling rates for cases with and without partitioning. The results show that cooling rates below 100% with partitioning have almost no influence on the material's ductility, while it fluctuates from 5.5% to 7.45% for the samples without partitioning. Increasing the cooling intensity has almost no influence on the ductility, with and without partitioning.

Figure 4 presents the dependence of hardness on cooling rates for with and without partitioning. From Figure 4a it can be seen that after a sufficient increase of the cooling time, the cooling time has almost no effect on the hardness of the material, even without partitioning. For short cooling times, the hardness increases quite significantly with decreasing cooling time for with and without partitioning. For a better overview, Figure 4b shows the effect of cooling intensity on the hardness value.

As expected, the samples with partitioning exhibit lower hardness. Despite rather different peripheral cooling and deformation conditions, the experimental results show that hardness is still significantly higher than the hardness values (450–550 HV1) found by

Jirková et al. (2019) for thick-walled (4 mm) tubes of the same steel [17]. This indicates a crucial influence of rapid local cooling rates throughout the forming process on the part's mechanical qualities and stresses the importance of the sophisticated Q-P heat treatment.

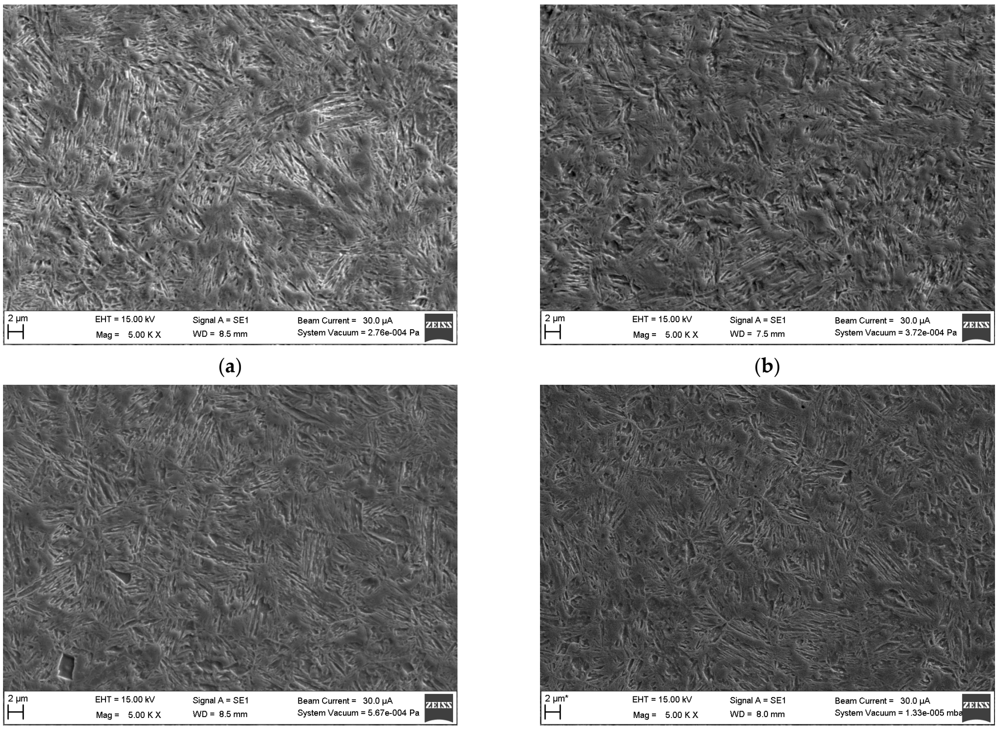


(**a**) (**b**)

Figure 4. Dependence of hardness (HV10) on various cooling times (**a**) complete set of data (**b**) selected range.

3.2. Metallographic Analysis

The microstructure after different heat treatment conditions and with PT is presented in Figure 5 for 50%, 25%, 200%, and 3.12% of the cooling rate. All conditions lead to martensite and retained austenite in the whole specimen, which is in agreement with investigations [23,24]. A hardness of 560–580 HV10 is measured for all cooling rate conditions, confirming the domination of lath martensite. The ductility of the sample with 200% cooling rate is significantly higher than those with 50% and 25% cooling rates.



(**c**) (**d**)

Figure 5. Microstructures from scanning electron microscope imaging of the standard Q-P with (**a**) half cooling rate, (**b**) quarter cooling rate, (**c**) double cooling, and (**d**) 3.12% cooling from 292 °C to 200 °C.

The martensitic reaction begins during cooling, when the austenite reaches the martensite start temperature (Ms), and the parent austenite becomes thermodynamically unstable. The differences in the fraction of the retained austenite (RA) cannot be detected in Figure 5a–d, which can be attributed to high enough cooling rate for all cases. It means that most of the austenite transforms into martensite before reaching the M_f temperature. The difference is more pronounced in the formation of the coarser and finer martensite laths. A lower quench rate leads to a slightly finer microstructure. For all conditions, no other phases than martensite and retained austenite are observed due to the high enough cooling rate. It should be noted that the cooling rate has an influence on the fractions of martensite and retained austenite but for the ranges of cooling rates and the composition of the steel in this study, the dominant phase was martensite for all cases. Small changes observed in the mechanical properties allow the assumption that the fractions of martensite and retained austenite do not vary noticeably.

4. Conclusions

The findings of this investigation show the added value of the various cooling rates between M_s and QT within the Q-P process since it makes it possible to combine high strength with good ductility providing a high energy absorption potential. The results show how crucial it is to understand the influence of the local cooling rates for various Q-P processed 42SiCr10 steel. Different dimensions/thicknesses and shapes of parts usually lead to different cooling rates/intensities, which sometimes becomes an issue when forming these materials. The results of this study show that the cooling rate has almost no significant effect on the mechanical properties of 42SiCr steel alloy when it undergoes a complete Q-P process. The results also indicate almost no significant changes in the microstructures as all lead to the formation of lath martensite. Although the samples with no partitioning phase show some variations in mechanical properties, only the maximum variation of 5% in UTS and 3% in ductility is determined and the hardness values exhibit no significant effect. Increasing the cooling intensity up to 200% increases the UTS, ductility and hardness value to a maximum of 2080 Pa (1% increase), 5.9% (0.4% increase), and 631 (3% increase), respectively. Conversely, decreasing the cooling intensity changes the mechanical properties by less than 5%.

In conclusion, for the Q-P treatment combined with different cooling intensities, the following findings on the impact of the local cooling rates may be obtained. Similar conclusions can also be qualitatively applied to other forming processes with an integrated Q-P heat treatment:

(1) It is possible to produce profile parts with good mechanical qualities by combining the appropriate quenching rate to QT with the proper Q-P heat treatment.
(2) Varying the cooling rate between M_s and QT influences the final mechanical properties more intensively without partitioning than with partitioning.
(3) Partitioning periods of 600 s are sufficient for the Q-P steel guaranteeing outstanding properties in the whole profile of the part.

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