




## Article

# On the Q&P Potential of a Commercial Spring Steel

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**Abstract:** Over the last years heat treatment concept of “quenching and partitioning” (Q&P) has reached popularity for its ability to precisely adjust material properties to desired values. Mostly, Q&P process are applied on tailor-made materials with high purities or prototype alloys. The research in hand presents the whole routine of how to investigate the potential of a commercial 0.54C-1.45Si-0.71Mn spring steel in terms of Q&P heat treatment from lab scale in dilatometer measurements to widely used inductive heat treatment on larger scale. In order to obtain the small process window for this material we were focusing on the interplay of the formed microstructure and the resulting mechanical properties in hardness measurements, compression tests as well as tensile tests. After full austenitizing, three different Q&P processing routes were applied. Microstructural analyses by optical microscopy, Scanning Electron Microscopy (SEM) and Electron Backscatter Diffraction (EBSD) exhibit a condition with 6.4% and 15% volume fraction of fine distributed retained austenite. Interestingly, the 15% of retained austenite developed during the partitioning heat treatment. Contradictory to our expectations, tensile and compression testing were showing that the 6.4% condition achieved improved mechanical properties compared to the 15% retained austenite condition. The remarkable conclusion is that not only volume fraction and fine distribution of retained austenite determines the potential of improving mechanical properties by Q&P in commercial alloys: also the process step when the retained austenite is developing as well as occurring parallel formation of carbides may strongly influence this potential.

**Keywords:** quenching and partitioning (Q&P); heat treatment; spring steel; TRIP effect; retained austenite



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## 1. Introduction

The heat treatment concept of quenching and partitioning (Q&P) offers a lot of potential for numerous fields of application. Combining high strength and good ductility, Q&P treatment is predestined for application on safety-relevant automotive parts.

Excellent properties of quenched and partitioned steels are based on the distinctive microstructure containing martensite and retained austenite, the latter of which was stabilized to room temperature by carbon diffusion from supersaturated martensite. After full or partial austenitization, quenching to a defined temperature between martensite start ( $M_s$ ) and martensite finish ( $M_f$ ) leads to the presence of untransformed austenite within the microstructure. Stabilization of retained austenite is achieved by the following partitioning. Partitioning temperature is either the same as quenching temperature (one-step

process) or above (two-step process). Aim of the Q&P treatment is to induce formation of a fine-grained microstructure containing tempered martensite laths surrounded by small areas of stabilized retained austenite leading to good formability of the material [1–4]. In the event of mechanical loading, austenite additionally transforms into martensite, which enhances material strength [4–9]. This effect is known as transformation induced plasticity (TRIP-effect).

The achievable maximal amount of retained austenite is not exclusively limited by quenching temperature but can increase during partitioning by austenite reversion [9–11]. The carbon partitioning from martensite to martensite-austenite interfaces leads to a change in local chemical composition and hence an increase of driving force for austenite reversion. Yuan et al. [10] described austenite reversion as an effect of “kinetic freezing” of carbon at martensite-austenite interface based on the lower diffusion range of carbon in austenite than in martensite. However, austenite reversion is a complex process depending on many other factors such as initial fraction and type of martensite, dislocations, alloy composition, process parameters of heat treatment and more [9–12].

Typical chemical composition for Q&P steels contains Silicon for prevention of cementite formation and thus benefiting stabilization of austenite through carbon partitioning [13]. However, other studies mention that, even with a high amount of silicon, carbide precipitation takes place during partitioning [10,14–16]. While formation of cementite is known to have negative effect on the success of Q&P treatment, the role of transition carbide precipitation is not entirely clarified. The circumstance, that any formation of carbides involves bonding of carbon, leads to the assumption that the amount of carbon available for austenite stabilization is diminished by carbide precipitation [13,16].

Former studies on 60Si2CrVA already showed potential of Q&P compared to commercial quenching and tempering (Q&T) [17–19] for high Si content spring steels. The motivation of this study is to improve the conventional Q&T properties for a lower alloyed spring steel (reduced Cr content), with shorter processing times and without any molten salts or tempered oil baths. In this way, the potential towards industrial application of the process should be underlined.

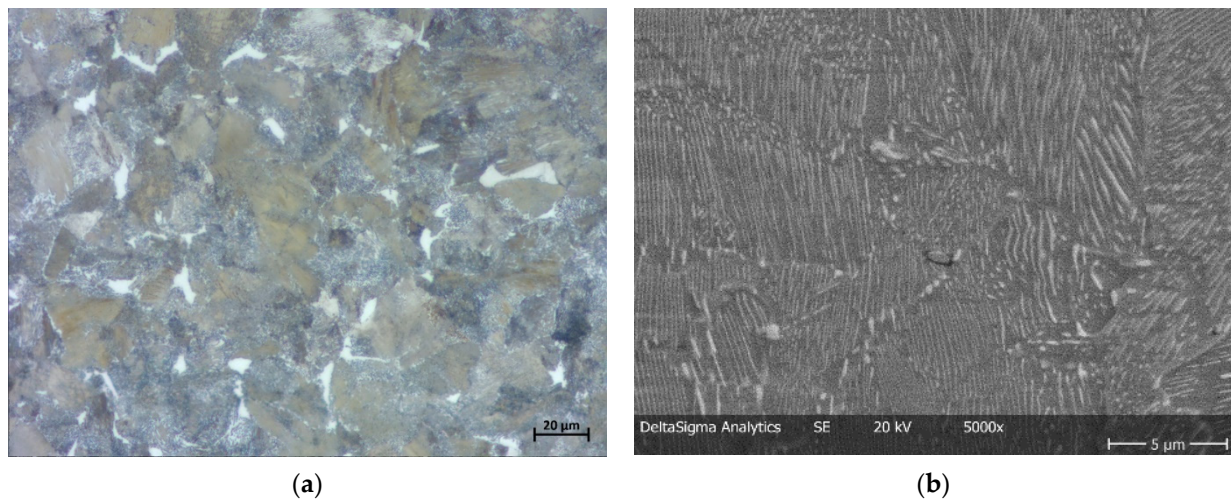
Investigation of mechanical properties is, besides hardness testing, usually carried out by tensile tests in the first place. Former studies show high potential in Q&P treatment for medium carbon steels, reaching total elongation beyond 10% and tensile strength up to  $R_m = 2000$  MPa [9,17–22].

## 2. Materials and Methods

The chemical composition of the material investigated in this study is given in Table 1. This steel is commercially distributed according to DIN EN 10,089 under the digits 1.7102, in SAE with the digits 9254 and in JIS under SUP 12. Compared to many other studies of Q&P, we were focusing on a material that is widely available worldwide and is not just casted within an unrealistic specification for a small scientific study. Please note that all occurring impurities of commercial steels strongly influence the size of the Q&P processing window. Nevertheless, we were facing this challenge to prove the potential of this material. In Figure 1, the initial condition of the microstructure is shown (find details for preparation and devices below). The microsection exhibits a typical needle-shape dark-etched pearlitic microstructure with more bright ferritic spots in between. This micro-structure is a characteristic of a non-heat-treated steel with 0.54% carbon steel.

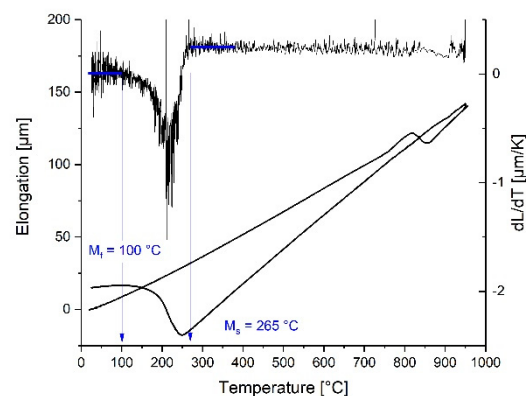
**Table 1.** Chemical composition (wt.-%) of the investigated material.

C	Si	Mn	Cr	Ni	Cu	P	S	Fe
0.54	1.45	0.71	0.63	0.04	0.06	<0.01	<0.01	Bal.



**Figure 1.** Micrographs of the initial material showing primary pearlitic microstructure (needles) with a few ferritic areas (bright spots): (a) optical microscopy and with increased magnification in (b) SEM micrograph of the same condition.

Determination of the necessary martensite start temperature ( $M_s$ ) and martensite finish temperature ( $M_f$ ) was carried out by dilatometric investigation to set limits of the process window. The dilatometric graph for quenching to room temperature and determined transformation temperatures are given in Figure 2. The characteristic changes in the length of the specimen indicates the martensite start temperature at 265 °C and the martensite finish temperature at 100 °C. For more details to the dilatometric investigation, see [23].



**Figure 2.** Dilatometric graph for hardening process of 0.54C-1.45Si-0.71Mn, after austenitization at 950 °C for 100 s. Derivative curve allows determination of transformation temperatures  $M_s$  and  $M_f$ . The characteristic changes in the length of the specimen indicates the martensite start temperature at 265 °C and the martensite finish temperature at 100 °C.

In order to obtain enough amount of material for detailed mechanical characterization we were transferring the results of the lab scale dilatometric measurements into a larger scale with a novel induction-based heat treatment equipment of DeltaSigma Analytics, combining induction furnace and contact cooling system. A complex control unit is regulating the heat treatment process in real time on the specimen's temperature monitored with several thermocouples. A mentionable benefit of the heat treatment set-up is that unhealthy molten salt baths can be avoided during the quenching as well as the partitioning. Specimens had dimensions of 18.5 mm in diameter and 10 mm in height. Austenitizing was carried out at 950 °C for 100 s. Afterwards the specimens were quenched to 175 °C and 200 °C, respectively. The following partitioning was carried out at three different partitioning temperatures (PT): 250 °C, 300 °C and 400 °C. The partitioning time (Pt) was

set to 600 s in all cases. Table 2 gives an overview of the different processing routes. The short duration of the Q&P heat treatment is addressed to industrial needs.

**Table 2.** Overview of processing parameters (quenching temperature QT, partitioning temperature PT and partitioning time Pt) for Q&P treatments.

Specimen	QT [°C]	PT [°C]	Pt [s]
Quenched	RT	-	-
Q&P 1	175	250	600
Q&P 2	200	300	600
Q&P 3	200	400	600

Microstructural investigations were carried out using optical microscopy (Axio-Lab, Zeiss, Oberkochen, Germany) and Scanning Electron Microscopy (SEM, Zeiss DSM, Oberkochen, Germany). The SEM was also used for fracture surface analysis without any further preparation. Electron Backscatter Diffraction (EBSD) was applied with a SEM (Scios DualBeam, FEI, Hillsboro, USA) to determine fractions of retained austenite. Areas of poor signal quality (Confidence Index CI < 0.1) were excluded. The microstructure of each specimen was investigated by optical microscopy and scanning electron microscopy (SEM) using 3% HNO<sub>3</sub> (Nital) etchant and Klemm I color etchant as contrasting method after grinding and polishing. Vickers microhardness testing was performed on a Buehler Wilson VH3300.

Tensile and compression tests were carried out in a conventional testing machine (ZWICK/ROELL Zmart Pro Universal 100 kN) [24] with an initial strain rate of 10<sup>-3</sup> s<sup>-1</sup> at room temperature. Testing was displacement controlled. While during compression testing only the internal displacement values of the testing machine was used, we were using a DIC system for the real time strain measurement during tensile testing [25]. This was required due to the miniature tensile specimen [26] that were used in this study and the need of an accurate measurement of the yielding. For each Q&P processing route, three (for compression tests) or four (for tensile tests) specimens were tested. Specimens for tensile tests had dimensions of 15 mm in length (5 mm gauge length), 2 mm in width and 1 mm in thickness, see [26]. Specimens for compression tests had 6 mm in diameter and 6 mm in height. All specimens were machine wired from the Q&P heat treated material.

### 3. Results and Discussion

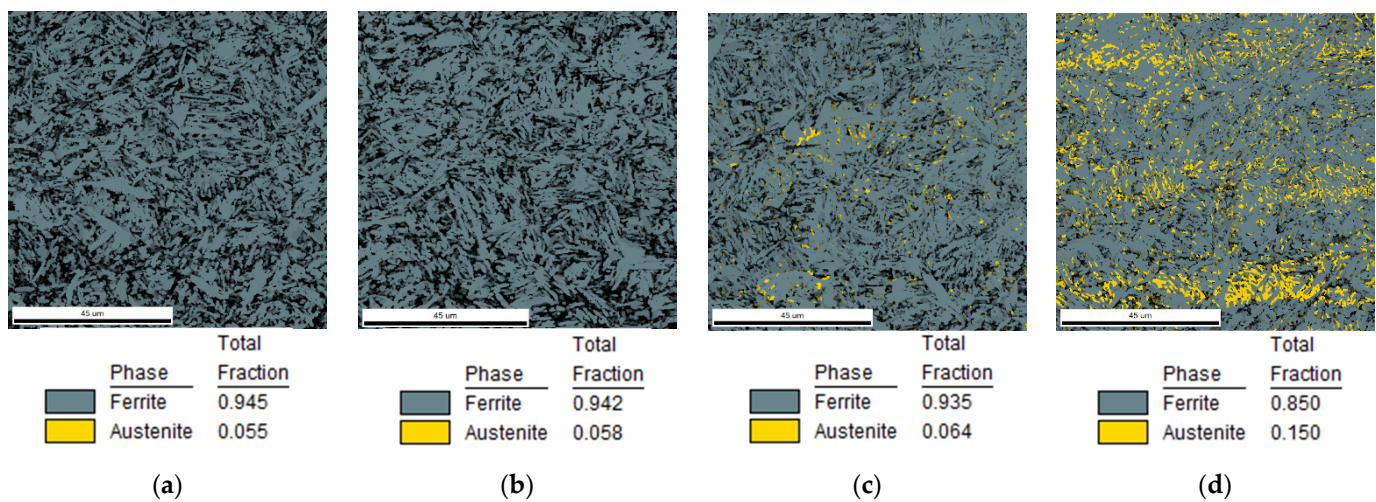
#### 3.1. Microstructure

Q&P treatment microstructure was analyzed by EBSD. Phase maps of specimens of all heat treatment conditions are shown in Figure 3. Please note that fractions of austenite small than 1 pixel cannot be displayed in such overview plots.

After quenching to room temperature, the microstructure consists of martensite with only a small amount of retained austenite (5.5%). Although, the measured temperature for M<sub>f</sub> (100 °C) is above the quenching temperature (room temperature) there are some small grains of austenite retained in the microstructure, which is stabilized by residual compressive stress.

It is reasonable to assume, that the fraction of retained austenite after quenching to 175 °C is higher than it is for quenching to room temperature. However, after quenching to 175 °C and partitioning at 250 °C, austenitic fraction is only slightly higher (5.8%). Since the mobility of C is low during the subsequent partitioning at 250 °C, partitioning seems to be insufficient to stabilize all retained austenite. During final cooling non-stabilized austenite transforms into fresh martensite.





**Figure 3.** EBSD phase maps ( $CI > 0.1$ ) of specimens: (a) Q quenched to room temperature; (b) Q&P 1 quenching temperature  $QT = 175\text{ }^{\circ}\text{C}$  and partitioning temperature  $PT = 250\text{ }^{\circ}\text{C}$ ; (c) Q&P 2  $QT = 200\text{ }^{\circ}\text{C}$ ,  $PT = 300\text{ }^{\circ}\text{C}$ ; (d) Q&P 3  $QT = 200\text{ }^{\circ}\text{C}$ ,  $PT = 400\text{ }^{\circ}\text{C}$ . The yellow fraction shows the retained austenite in the microstructure (fcc) while the grey shows the ferrite (bcc). This area is not considered in the calculation of phase fractions. The amount of retained austenite is increasing from 5.5% up to 15%. Furthermore, a fine distribution of the retained austenite can be observed in c and d. Interestingly, the increased amount of retained austenite in d must have developed during the partitioning heat treatment in order to experience the same quenching treatment such as c.

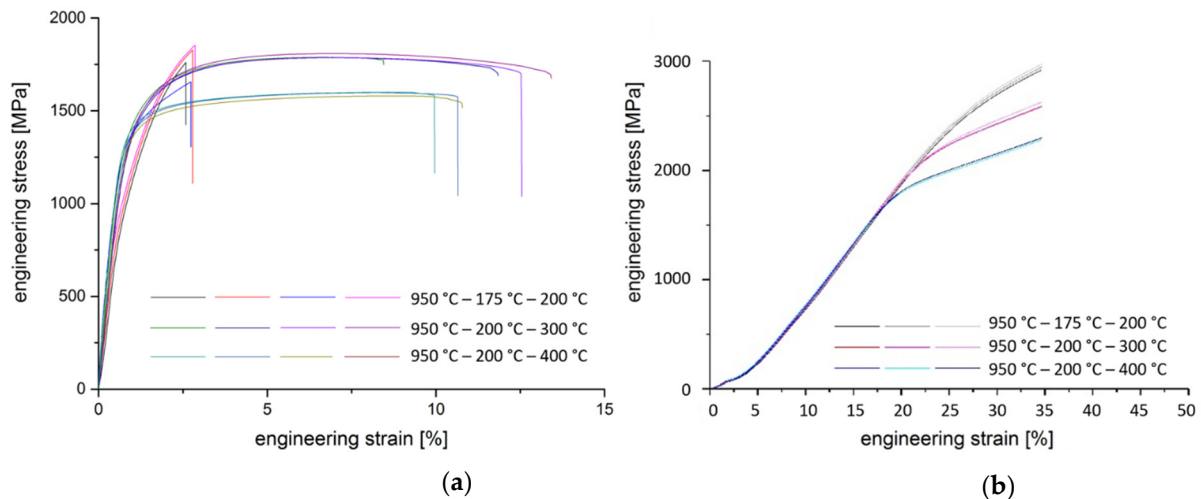
With increased quenching temperature and increased partitioning temperature, fraction of austenite increases. Interestingly, the amount of retained austenite strongly differs between the condition from Figure 3c,d. While the quenching temperature was the same those two conditions only differ in partitioning temperature. On one hand, higher partitioning temperature might stabilize higher amount of retained austenite. In this case, however, from dilatometric curves [23] it might be assumed that most retained austenite was already stabilized at  $300\text{ }^{\circ}\text{C}$ , since there is no evidence for martensitic conversion of non-stabilized retained austenite at final quenching. This leads to the assumption that the higher amount of austenite in Figure 3d might also result from reversion of martensite to austenite during partitioning at  $400\text{ }^{\circ}\text{C}$ .

Effects of reversed austenite on mechanical properties is not yet well investigated. However, composition, morphology and size of the austenitic fractions in any case have strong influence on mechanical properties (occurrence of TRIP-effect). Newly developing retained austenite developing during partitioning may not be stable and may also not contribute to the outstanding performance of mechanical testing of Q&P heat treated steels.

### 3.2. Mechanical Properties

In the pre-work of this paper we were already publishing the hardness values of the three different conditions in comparison to the only quenched condition with a correlation to the amount of retained austenite [20]. The Q condition exhibits the highest values of hardness ( $774 \pm 15\text{ HV}0.1$ ) and the lowest amount of retained austenite ( $\sim 5.5\%$ ). This amount is still remarkable for a Q condition and emphasizes the role of Si as stabilizer for austenite in the alloy. Compared to Q the Q&P 1 condition ( $QT = 175\text{ }^{\circ}\text{C}$ ,  $PT = 250\text{ }^{\circ}\text{C}$ ) exhibits a reduced hardness ( $640 \pm 14\text{ HV}0.1$ ) while the amount of retained austenite is only slightly increased ( $\sim 5.8\%$ ). Retained austenite values for the Q&P 2 condition ( $QT = 175\text{ }^{\circ}\text{C}$ ,  $PT = 300\text{ }^{\circ}\text{C}$ ) are again slightly increased to  $\sim 6.4\%$  which goes in line with a decrease of hardness to  $579 \pm 8\text{ HV}0.1$ . The Q&P 3 condition with  $QT = 175\text{ }^{\circ}\text{C}$ ,  $PT = 400\text{ }^{\circ}\text{C}$  exhibits 15% retained austenite and a hardness of  $508 \pm 11\text{ HV}0.1$ . Compared to other studies the values are very promising and are already a sign for the potential of this heat treatment for that material. Anyway, for industrial application, more detailed mechanical properties are required. In order to obtain them tensile and compression tests were performed on the

three different Q&P conditions. In Figure 4a, the results are from the tensile testing are shown, in Figure 4b those from compression testing. Please note the difference in Young's modulus which is related to the different strain measurements for tensile and compression tests, see chapter 2. Please also note that the compression tests are running quick into the maximum load of the testing device for compression tests. That is why the curves are ending after only comparatively small plastic deformation.



**Figure 4.** Stress-strain diagrams derived from specimens treated with different Q&P processing routes: (a) tensile stress-strain diagram; (b) compressive stress-strain diagram. Q&P 1 condition (950 °C–175 °C–200 °C) exhibit the highest yield strength in a and b, but brittle characteristic with almost no plastic deformation. Q&P 2 condition (950 °C–200 °C–300 °C) exhibits high yield strength with a pronounced work hardening and strongly increased total strains in a and slightly decreased yield strengths under compression load compared to first condition in (b). Q&P 3 condition (950 °C–200 °C–400 °C) exhibits high yield strength with a less pronounced work hardening and slightly decreased total strains compared to second condition and lowest yielding under compression (b).

The Q&P 1 condition (950 °C–175 °C–200 °C) exhibits yield strengths ( $R_{P0.2}$ ) of  $1038 \pm 30$  MPa, ultimate tensile strengths ( $R_m$ ) of  $1774 \pm 88$  MPa and total elongations (A) of  $1.56 \pm 0.39\%$ , while yielding under compression occurred at  $2054 \pm 24$  MPa. This enormous strength differential (SD) effect is typical for martensitic microstructures [27]. A comparable amount of retained austenite like the only quenched condition (see Figure 3a,b) as well as the highest value of hardness are supporting this assumption. We need to conclude that this Q&P treatment failed in order to compare those results with data from the actual applied quenching and tempering (QT) heat treatment where values of  $R_{P0.2} = 1300$  MPa,  $R_m = 1450$ – $1750$  MPa and  $A = 6\%$  are achieved [<https://portal.totalmateria.com/de/search/quick/materials/1041714/mechanical>, 8 August 2021].

The Q&P 2 condition (950 °C–200 °C–300 °C) exhibits remarkable properties with  $R_{P0.2} = 1338 \pm 14$  MPa,  $R_m = 1792 \pm 11$  MPa and  $A = 10.64 \pm 2.11\%$  in the tensile testing and  $R_{P0.2} = 1825 \pm 8$  MPa in compression testing. This condition with only 6.4% fine distributed retained austenite (see Figure 3c) results in the smallest SD effect. Compared to the commercial QT condition we achieved an increase of  $R_{P0.2}$  of ~3%, of  $R_m$  of more than 4% and of A of ~77%. The remarkable improvement in mechanical properties can be related to the stabilized retained austenite in this condition which transforming induced by plastic deformation (TRIP effect). Compared to the study in [18] the result for yielding and ultimate tensile strength are comparable, those for strain are almost doubled even though our material exhibits less carbon, less retained austenite after QP treatment and was much shorter tempered. One specimen with  $R_{P0.2} = 1354$  MPa,  $R_m = 1808$  MPa and  $A = 12.5\%$  was outstanding and underlined the potential of Q&P heat treatment for this material, especially with the fact that the whole inductive heat treatment was lasting less than 8 min overall.

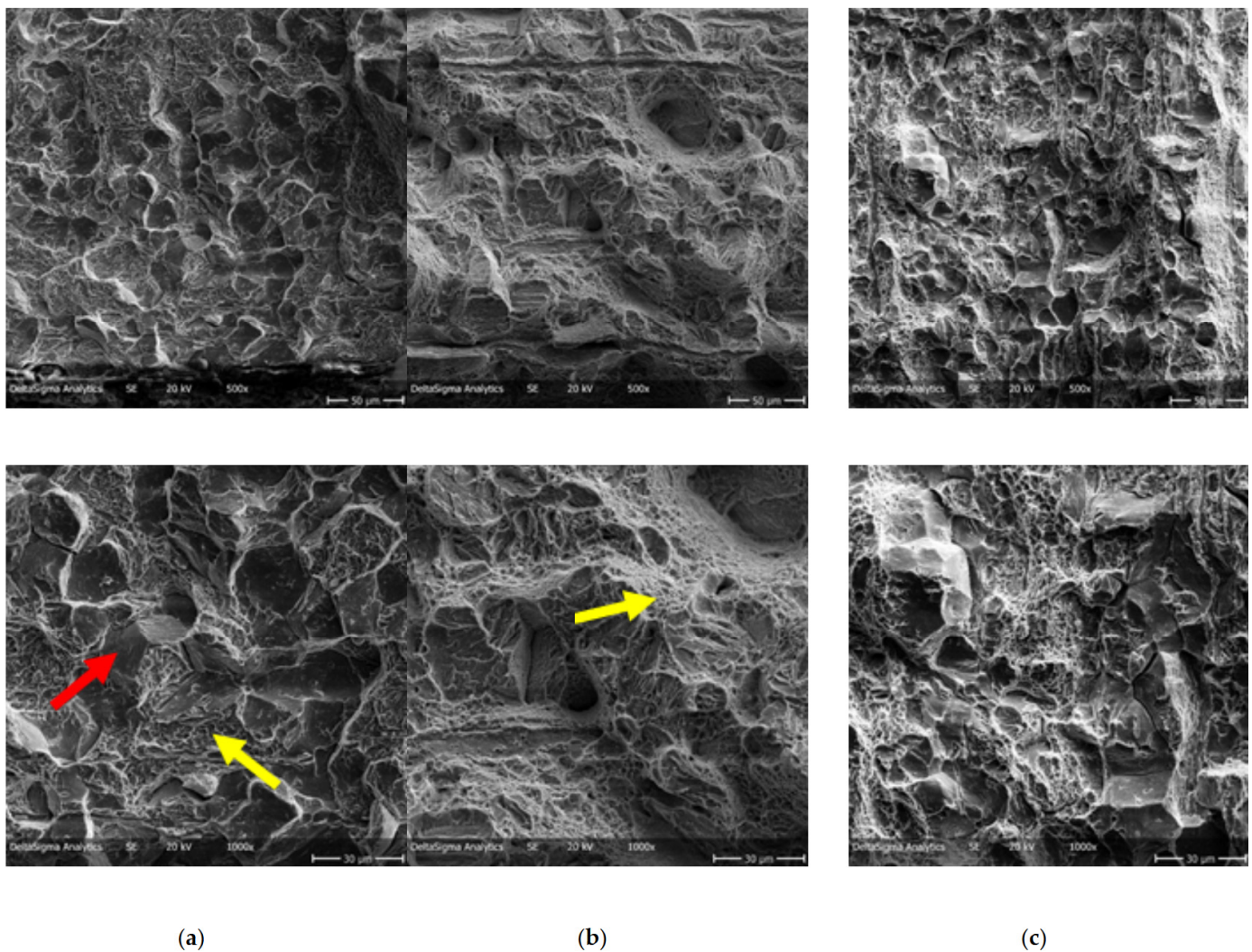
The Q&P 3 condition (950 °C–200 °C–400 °C) exhibits  $R_{p0.2} = 1349 \pm 19$  MPa,  $R_m = 1593 \pm 9$  MPa and  $A = 9.89 \pm 0.61\%$  in the tensile testing and  $R_{p0.2} = 1606 \pm 16$  MPa in compression testing. The less pronounced but still existing SD effect is an indication for a bimodal microstructure of martensite and retained austenite. This can be observed in Figure 2d. Even though we achieved with 15% the most amount of retained austenite for all conditions we cannot see enhanced mechanical properties which we were expecting after measuring the volume fraction of retained austenite. Comparing the differences in processing of second and third condition we assume that during the partitioning at 400 °C new retained austenite was developing but without any stabilization. Without the stabilization of the retained austenite, we cannot achieve mechanical properties such as for the second condition with less amount of retained austenite but obviously stabilized retained austenite. Furthermore, we assume that the partitioning at 400 °C also leads a tempering of the quenched martensite (lower carbon content in martensite) and to the formation of cementite and some brittle carbides (e.g.,  $M_7C_3$ ) which decreases both the strength and the ductility of this Q&P 3 condition compared to the Q&P 2 condition. Considering the short duration of partitioning those carbides are supposed to be very small. That is why we were not able to observe their formation in SEM clearly. Future studies with STEM or TEM may overcome this issue.

### 3.3. SEM Analysis of Fracture Surfaces

Figure 5 shows SEM images of fracture surfaces from tensile tested specimens. First (Figure 5a) and third (Figure 5c) condition specimens show both areas of ductile fracture and brittle fracture. The second condition specimen, however, shows mainly ductile fracture. Since data from tensile testing showed that second condition specimens reached highest elongation levels, appearance of the fracture surfaces correspond well to derived data. In all cases fracture surfaces showed intergranular fracture. No evidence for the existence of carbides within the fracture surfaces could be found. However, existence of carbides cannot be excluded, since precipitation size is often within nanometer range.

Considering all results of the research it should be pointed out Q&P obtains a remarkable potential for commercial steels as well. Though, the process window for enhanced mechanical properties is small and even small deviation in process route can lead to worse properties. Interestingly, the condition with 15% of austenite developed during the partitioning heat treatment. Contradictory to our expectations, tensile and compression testing were showing that the 6.4% condition achieved improved mechanical properties compared to the 15% austenite condition. The remarkable conclusion beyond the accompanying TRIP effect during mechanical testing is that not only volume fraction and fine distribution of retained austenite determines the potential of improving mechanical properties by Q&P in commercial alloys: also the process step when the retained austenite is developing (and stabilizing or not) as well as occurring parallel formation of carbides may strongly influence this potential.





**Figure 5.** Fracture surfaces of tensile specimens: (a) Q&P 1; (b) Q&P 2; (c) Q&P 3: While condition 1 (a) and 3 (c) show both, areas of ductile fracture (yellow arrow) and brittle fracture (red arrow), condition 2 (c) mainly shows ductile fracture.

#### 4. Conclusions

In the present work, a study on the potential of quenching and portioning heat treatment (Q&P) of a commercial spring steel 0.54C-1.45Si-0.71Mn was performed successfully. The most promising candidates from a dilatometric and SEM/EBSD study in a pre-work were investigated regarding their mechanical properties with the help of tensile and compression tests. The main conclusion can be summarized as follows:

1. The Q&P 1 condition (950 °C–175 °C–200 °C) exhibits results in mechanical testing and fracture surface analysis comparable to an only quenched brittle condition. Almost the same content and distribution of retained austenite from those two conditions can be an explanation for this result.
2. The Q&P 2 condition (950 °C–200 °C–300 °C) exhibits the most remarkable results compared to the commercial heat treatment of this alloy. While the yield strength as well as the ultimate tensile strength were only slightly increasing, we were achieving a strong increase in ductility (+77%) compared to the actual performed heat treatment. Those observations can be rationalized with an increase and fine distribution of the retained martensite of this condition.
3. The Q&P 3 condition (950 °C–200 °C–400 °C) was the most promising candidate from the view of amount of retained austenite in the microstructure after Q&P heat treatment. Considering the same quenching temperatures as Q&P 2 condition it



is obvious that the increase in amount of retained austenite occurred during the partitioning. In order to obtain stabilization of retained austenite and not to obtain more austenite the resulting mechanical properties were not as good as the Q&P 2 condition. Furthermore, higher temperatures at partitioning leads to more tempering of the quenched martensite.

4. One can assume that not only volume fraction and fine distribution of retained austenite determines the potential of improving mechanical properties by Q&P in commercial alloys: also the process step when the retained austenite is developing as well as occurring parallel formation of carbides may strongly influence this potential.

For an industrial application we can strongly recommend a heat treatment route following our Q&P 2 parameters. Considering the overall process time of less than 8 min for both improved strength and ductility values while avoiding molten salt baths is emphasizing the potential of Q&P for commercial spring steels.

**Author Contributions:** M.H., A.W. and S.D. were the main authors during the original draft preparation. S.D. was responsible for all microstructural features. A.W. and S.D. were performing the heat treatments. M.H., P.L. and T.G. were responsible for the mechanical testing. P.L. and T.G. assisted in writing the manuscript. T.L., H.N., T.H. and S.W. supervised the work and reviewed and edited the manuscript. S.W. furthermore had the original idea of using this material for Q&P process. H.N. and S.W. were funding this research from internal research budget of thyssenkrupp Presta Chemnitz GmbH. All authors have read and agreed to the published version of the manuscript.

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