Microstructural Characterization of Quenched and Partitioned Commercial Medium Carbon Steel

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Abstract. In order to achieve the desired properties, the microstructure of metals is often modified by heat treatments. A suitable combination of high strength and good ductility can be achieved by adjusting the amounts of martensite and retained austenite through a Quenching and Partitioning (Q&P) process. Controlling these material properties offers new potentials in the production of lightweight steel parts. This paper presents a basic study of heat treatment conditions with subsequent microstructural characterization of quenched and partitioned commercial 0.54C-1.45Si-0.71Mn spring steel. Beginning from full austenitization, different quenching and partitioning parameters were applied. Microstructural characterization was performed using optical microscopy in combination with different etching methods as well as Scanning Electron Microscopy (SEM) and Electron Backscattered Diffraction (EBSD). It is shown that the intended dual phase microstructure with a specific phase fraction of austenite can be produced. Those results and consideration of hardness measurements show that the investigated commercial spring steel is a promising candidate for improving mechanical properties by a Q&P process.

1 Introduction

Advanced high strength steels offer new possibilities in lightweight construction. Especially for automotive parts increasing strength while keeping ductility at sufficient values can allow significant weight reduction. Over the last years quenching and partitioning (Q&P) process has become a popular heat treatment process for producing excellent materials properties, combining high strength and good ductility. The aimed properties are achieved by an individual material-dependent processing route, starting from full or partial austenitization followed by quenching to a defined temperature between martensite start (Mₜ) and martensite finish (Mₙ) temperature. The resulting microstructure contains supersaturated martensite as well as a fraction of retained austenite. During the following low temperature tempering (partitioning) the retained austenite is stabilized to room temperature by carbon diffusion from martensite [1-3]. Q&P heat treatment is either performed as one or two step process. Figure 1 shows the processing route for each variant [4]. Due to this heat treatment the materials properties develop from brittle (as quenched) to a high strength and ductile mechanical behavior (partitioned). During the low temperature tempering carbon escapes from the supersaturated martensite, which becomes formable with only a small decrease in strength. The carbon enriches the retained austenite. As a consequence, Mₜ of the retained austenite is lowered below room temperature. The
austenite fraction enhances the formability of the material. Furthermore, the austenite transforms into martensite under mechanical loading (TRIP effect), which increases the materials strength [5, 6].

Steels used for Q&P heat treatment must contain a certain amount of carbon for hardenability. Additionally, Silicon is needed to prevent carbide formation and Manganese for expanding gamma phase area [7-10]. An appropriate Q&P heat treatment can lead to a unique combination of tensile strength and total elongation. For low carbon steel $R_m = 1047$ MPa and very high elongation of 15.5 % can be reached [11]. Medium carbon steels reach $R_m = 2000$ MPa and a total elongation beyond 10 % [12-15].

![Figure 1. Schematic processing variants of Q&P heat treatments with quenching temperature QT defined as $M_s > QT > M_f$ [4].](image)

0.54C-1.45Si-0.71Mn is a material that is already widely used in constructing automotive parts and may be a key for industrial application of the Q&P process. The chemical composition especially with a comparatively high amount of silicon makes this kind of steel a promising candidate for Q&P heat treatment. The present study examines appropriate heat treatment parameters for Q&P by the investigation of parameter dependent microstructure development.

2 Experimental procedure
The material investigated in this study is a 0.54C-1.45Si-0.71Mn spring steel and provided from our customer thyssenkrupp. Chemical composition is given in Table 1. Q&P heat treatment was performed as a dilatometric study. Dilatometric specimens were produced by electrical discharge machining (EDM) and had dimensions of 4 mm in diameter and 10 mm in length. The austenitization was performed under vacuum at 950 °C for 100 s. First, the transformation temperatures were measured by hardening the material to room temperature using He gas as quenching medium. Derived from the measured $M_s$ and $M_f$ temperatures for Q&P heat treatment the samples were quenched to either 175 °C or 200 °C to vary the fraction of retained austenite. For partitioning the samples were reheated to the partitioning temperature (PT) of 250 °C for QT = 175 °C and 300 °C and 400 °C, respectively, for QT = 200 °C. The partitioning time (Pt) was fixed to 600 s. An overview is given in Table 2.

The microstructure of each specimen was investigated by optical microscopy and scanning electron microscopy (SEM) using 3 % HNO$_3$ (Nital) etchant and Klemm I color etchant as contrasting method after grinding and polishing. Volume fractions of retained austenite were analyzed by electron backscatter diffraction (EBSD), whereas areas with poor signal quality (Confidence Index below 0.1) were excluded. For all EBSD measurements FEI Scios DualBeam scanning electron microscope was used. Vickers micro hardness testing was performed on a Buehler Wilson VH3300.
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. |

Table 2. Overview of chosen parameters for Q&P heat treatment.

| Specimen | QT [°C] | PT [°C] | Pt [s] |
|----------|---------|---------|--------|
| Hardened | RT      | -       | -      |
| T2.1     | 175     | 250     | 600    |
| T2.2     | 200     | 300     | 600    |
| T2.3     | 200     | 400     | 600    |

3 Results

3.1 Transformation temperatures
As can be seen in Figure 2 the initial material is primary perlitic with embedded islands of ferrite. The dilatometric curve for the measurement of transformation temperatures is shown in Figure 3. Therefore, the hardening process was performed by quenching to room temperature. The investigated steel 0.54C-1.45Si-0.71Mn is characterized by $M_s = 265$ °C and $M_f = 100$ °C. Hence, for Q&P the quenching temperature was set to be 175 °C and 200 °C, respectively, to retain different amounts of austenite.

Figure 2. Micrographs of the initial material (a optical microscopy, b SEM) showing a primary perlitic matrix with ferritic islands.
Figure 3. Dilatometric graph for quenching 0.54C-1.45Si-0.71Mn for austenitization at 950 °C for 100 s (a) and the resulting martensitic microstructure for reference (b).

3.2 Microstructural evolution
From the curves of dilatometer measurement (Figure 4) can be seen, that the sample with QT = 175 °C is characterized by a gradient in slope during final cooling, indicating a martensitic transformation. The specimen with QT = 200 °C shows a constant slope during final cooling.

Figure 4. Temperature dependent elongation during Q&P (a QT = 175 °C, PT = 250 °C; b QT = 200 °C, PT = 300 °C; c QT = 200 °C, PT = 400 °C).

The microstructure after quenching (Figure 3 b) shows martensitic laths and, as expected, no evidence of austenite. Figure 5 shows optical micrographs of microstructures resulting from the selected Q&P parameters. The specimens quenched to 175 °C and partitioned at 250 °C do not show any sure indication of austenitic fractions. The sharp needle-like martensitic structure is comparable with the as-quenched stage.

Figure 5. Optical micrographs of etched surfaces of 0.54C-1.45Si-0.71Mn after Q&P (a QT = 175 °C, PT = 250 °C; b QT = 200 °C, PT = 300 °C; c QT = 200 °C, PT = 400 °C).
For the specimen quenched to 200 °C the sharp needle-like martensitic structure is less pronounced compared to the 175 °C condition. The small bright regions between the martensitic laths can be observed in both specimens, see Figure 6, and can be attributed to the formation of austenite [16]. The fraction of austenite increases with higher partitioning temperature.

**Figure 6.** Optical micrographs of colour etched surfaces of 0.54C-1.45Si-0.71Mn after Q&P (a QT = 200 °C, PT = 300 °C; b QT = 200 °C, PT = 400 °C).

**Figure 7.** EBSD phase maps (CI > 0.1) of specimen a quenched to room temperature, b quenched to 175 °C and partitioned at 250 °C, c quenched to 200 °C and partitioned at 300 °C, d quenched to 200 °C and partitioned at 400 °C.
The EBSD measurements confirmed the assumptions made from optical microscopy investigations. Figure 7 shows corresponding EBSD phase maps. At lower quenching temperature (Figure 7 b) austenite fraction is comparable to the as-quenched stage (Figure 7 a). The phase fraction of austenite increases with higher partitioning temperature (Figure 7 c and d). Thereby, the austenite is located between the martensite laths. Furthermore, the fraction of areas with poor signal quality (CI < 0.1) is lowered for the Q&P samples quenched to 200 °C, leading to the assumption that the residual stresses are lowered compared to as-quenched and quenched to 175 °C.

3.3 Mechanical properties
The results of hardness measurements are summarized in Figure 8. As expected, there is a loss of hardness when the quenching is followed by a reheating. The hardness decreases with increasing partitioning temperature. The associated hardness distribution maps can be seen in Figure 9. Each hardness profile is comparatively homogeneous.

4 Conclusions
Different Q&P processing routes were tested on commercial 0.54C-1.45Si-0.71Mn spring steel. Volume fractions of retained austenite and Vickers hardness were determined.

The microstructure of the specimen with QT = 175 °C is comparable with the as-quenched stage, meaning that the chosen parameter for Q&P are insufficient. It is possible, that the amount of retained austenite is already too low at QT = 175 °C (QT might be too close to Mf). Another possible explanation
is that PT = 250 °C could be too low for sufficient stabilisation of retained austenite due to limited carbon diffusion. Regarding the temperature dependent elongation of the sample, the last option seems to be the probable explanation, since transformation of fcc to bcc seems to take place during final cooling. Similar behaviour has been observed by Dieck et al. [15]. The specimens with QT = 200 °C are characterized by a distinct austenite fraction. In the case of PT = 400 °C an amount of 15 % could be stabilized. This leads to the assumption, that this parameter setup is appropriate for sufficient stabilising retained austenite. The carbon diffusion further causes the decreasing hardness. The amount of austenite is a function of PT and can be adjusted. Whether this stabilized austenite enhances the mechanical properties of the material will be checked by mechanical testing in further investigations as well as the impact of hydrogen embrittlement [17].

It was shown that commercial 0.54C-1.45Si-0.71Mn is a very promising candidate for excellent enhancement of mechanical properties through Q&P heat treatments. The formation of microstructures with different amounts of retained austenite was shown successfully. By designing individual processing routes the microstructure can be adjusted precisely to the desired properties.

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