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Reversed austenite for enhancing ductility of martensitic stainless steel

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Abstract. The novel heat treatment concept, “quenching and partitioning” (Q&P) has been developed for high strength steels with enhanced formability. This heat treatment involves quenching of austenite to a temperature between martensite start and finish, to receive a several amount of retained austenite. During the subsequent annealing treatment, the so called partitioning, the retained austenite is stabilized due to carbon diffusion, which results in enhanced formability and strength regarding strain induced austenite to martensite transformation. In this study a Q&P heat treatment was applied to a Fe-0.45C-0.65Mn-0.34Si-13.95Cr stainless martensite. Thereby the initial quench end temperature and the partitioning time were varied to characterize their influence on microstructural evolution. The microstructural changes were analysed by dilatometer measurements, X-ray diffraction and scanning electron microscopy, including electron back-scatter diffraction. Compression testing was made to examine the mechanical behaviour. It was found that an increasing partitioning time up to 30 min leads to an enhanced formability without loss in strength due to a higher amount of stabilized retained and reversed austenite as well as precipitation hardening.

1. Introduction

Martensitic stainless steels possess high strength and hardness, as well as a cost advantage compared with Ni-containing stainless steel. Thus, they are preferred for application as machine parts that are used in mildly corrosive environments, for example edge tools or pump shafts. The ductility of these steels can be improved by tempering at high temperatures (600 – 650 °C) [1, 2]. This heat treatment is associated with a decreasing strength and corrosion resistance. Furthermore there is the sensibility to temper embrittlement [3, 4]. Recent research focusses on enhancing the ductility without loss in strength by a low temperature heat treatment.

For this purpose SPEER et al. suggest a heat treatment, named quenching and partitioning (Q&P). This two-step heat treatment starts with quenching of homogenized (or partial in the case of austenitizing in the two phase region) austenite to a temperature between martensite start (M_s) and martensite finish (M_f) to remain an amount of austenite within a martensitic (ferritic) matrix. During the following partitioning this microstructure is treated either at, or above, the quench temperature, which results in carbon (C) diffusion from supersaturated martensite into retained austenite. The C-enrichment stabilizes the retained metastable austenite to room temperature [1, 5 – 7]. Q&P steels combine ultrahigh strength (up to 2000 MPa), which results from the martensitic matrix and with a high ductility, attributed to the transformation induced plasticity (TRIP) of austenite to martensite under loading condition [6, 8, 9].



Regarding the alloy composition C has the biggest effect on properties of Q&P steels because of its influence on transformation behaviour, martensite type and austenite stability. Furthermore commonly used steel for Q&P heat treatment include Manganese (Mn) to stabilize austenite and Silicon (Si) to suppress carbide forming [10, 11].

Regarding medium carbon stainless martensite YUAN et al. investigated a modified Q&P process with low temperature partitioning at 300 – 500 °C. The alloy composition (Fe-0.44C-13.6Cr) lowers the M_f temperature below room temperature leading to a high fraction of retained austenite without interrupted quenching. Furthermore they found that subsequent low temperature partitioning (400 °C) leads to a stabilization of the retained austenite and also to an increasing fraction of austenite. This is effected by a local carbon enrichment at martensite – martensite and martensite – austenite boundaries, which results in a local reversion of martensite to austenite [12], figure 1.

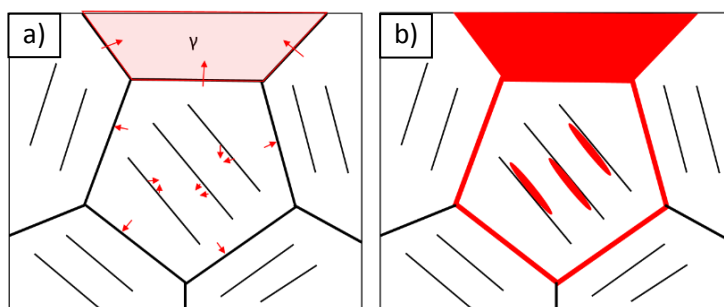


Figure 1. Austenite reversion during low temperature partitioning: a) diffusion paths of C (red arrows); b) microstructure after final quenching (austenite coloured red).

2. Materials and methods

2.1 Material

The chemical composition of the alloy used to apply a Q&P process was analyzed by optical emission spectroscopy (table 1). The heat treatment was performed with cylindrical specimen cut from soft-annealed round bars.

Table 1. Alloy composition of the material in wt.-%.

Material number	C	Mn	Si	Cr	P	S	Fe
1.4034	0.45	0.65	0.34	13.95	0.01	< 0.001	Bal.

The choice of austenitizing parameters for the investigations was based on thermodynamic considerations involving temperature and the resulting microstructure. The thermodynamically stable carbon and chromium content in the austenitic phase solution were calculated as a function of temperature with the software Thermo-Calc (figure 2) [13]. The transformation from bcc to fcc enhances the solubility of carbon. Further increase of temperature leads to a higher degree of carbide dissolution. Thus the carbon and chromium content in the austenitic solid solution is increased until the complete dissolution of carbides is achieved at about 1100°C. The applied temperature was raised up to 1150 °C to dissolve the large carbides within the soft-annealed raw material.

Two initial cooling end temperatures (QT1 – 25 °C and QT2 – 100 °C) were chosen for dilatometer investigations. Room temperature (25 °C) was determined as QT1 for comparability with literature. To retain a higher austenite fraction QT2 was at 100 °C. The quenching end temperature was limited to QT1 for further experiments, because of the results from dilatometer measurements.

After initial quenching, the partitioning treatment was conducted at 400 °C. To investigate the time dependency of the austenite reversion and its influence on mechanical behavior the partitioning time was varied (0 min – as quenched, 1 min and 30 min). The medium for final cooling was water, too.

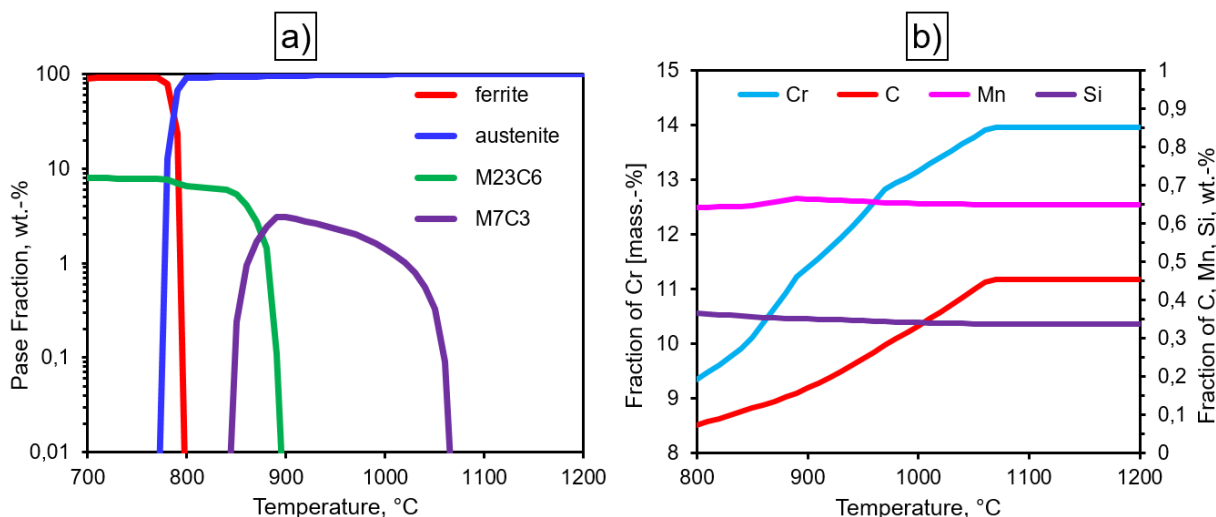


Figure 2. Thermo-Calc calculation of a) temperature dependent phase transformation and b) the accompanied element distribution (right).

2.2 Characterization methods

Mechanical properties were determined by compression testing at room temperature along the rolling direction of the specimen. The samples for compression testing had a height of 9 mm and a diameter of 6 mm. The testing was carried out with a TIRAtest 28100 universal tension and compression testing machine.

Dilatometer measurements were made with DIL805A with sub-zero expansion. The volume fraction of austenite after heat treatment was measured by X-ray diffraction (XRD). The samples were prepared by standard grinding and polishing procedure and finally etched with V2A etchant. The measurements were realized with a PANalytical X'Pert PRO diffractometer using Cu K α radiation. The data were collected over a 2θ range of 30 – 120° with a step size of 0.017°.

The austenite distribution was measured by electron back-scatter diffraction (EBSD). Therefore the specimen were prepared by standard grinding and polishing with a subsequent vibration polishing using Buehler MasterMet2 polisher slurry to achieve a surface without residual stresses due to preparation. EBSD measurements were performed with EDAX/TSL OIM DC 5 and evaluated with OIM Analysis 5 (clean up via grain dilation; parameters: grain tolerance angle: 5°, minimum grain size: 10 pixel). Scanning electron microscopy (SEM) was conducted with a FEI XL30 ESEM-FEG operating with an accelerating voltage of 25 kV.

3. Results and discussion

3.1 Transformation behaviour

Figure 3 shows a martensite start temperature of ~170 °C. Regarding enhanced formability of Q&P steels a certain volume fraction of stabilized retained austenite is needed [15, 16]. Therefore two initial quench end temperatures (QT), leading to different fractions of retained austenite, are examined concerning phase development during partitioning. Figure 4 shows the results of this investigation. For initial quenching to room temperature (figure 4 a) there are no phase transformation during final quenching from partitioning temperature (400 °C) as expected. On the contrary a sample quenched to 100 °C during the initial cooling exhibits a martensitic transformation during the final cooling

(figure 4 b). It is supposed from that data that the stabilizing effect of carbon diffusion during the partitioning treatment is insufficient for such a high portion of austenite remaining when quenching Fe-0.45C-0.65Mn-0.34Si-13.95Cr to elevated temperature. The formation of “fresh” (newly formed) untempered martensite is expected to decrease the formability. Thus the further work on mechanical properties and microstructural evolution will focus the Q&P process with QT1 (25 °C).

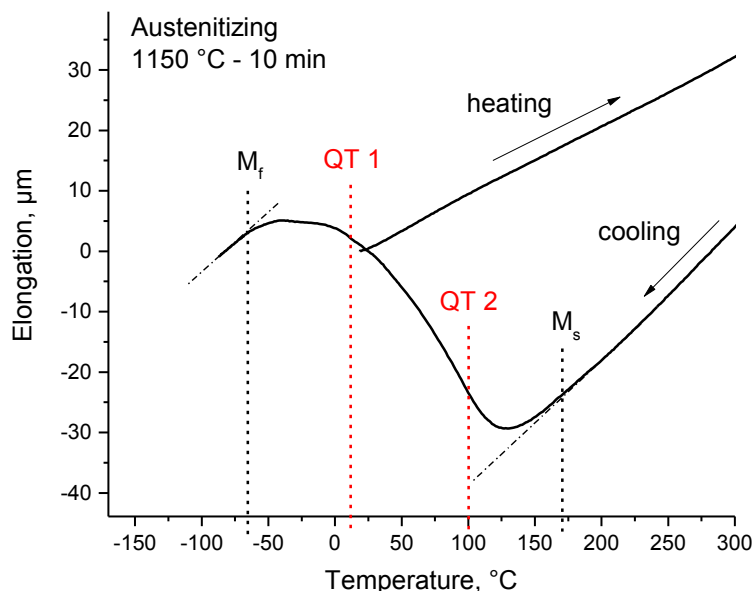


Figure 3. Dilatometer measurement of transformation behavior of Fe-0.45C-0.65Mn-0.34Si-13.59Cr during quenching from 1150 °C to -100 °C; determined initial quench end temperatures marked with red dashed lines.

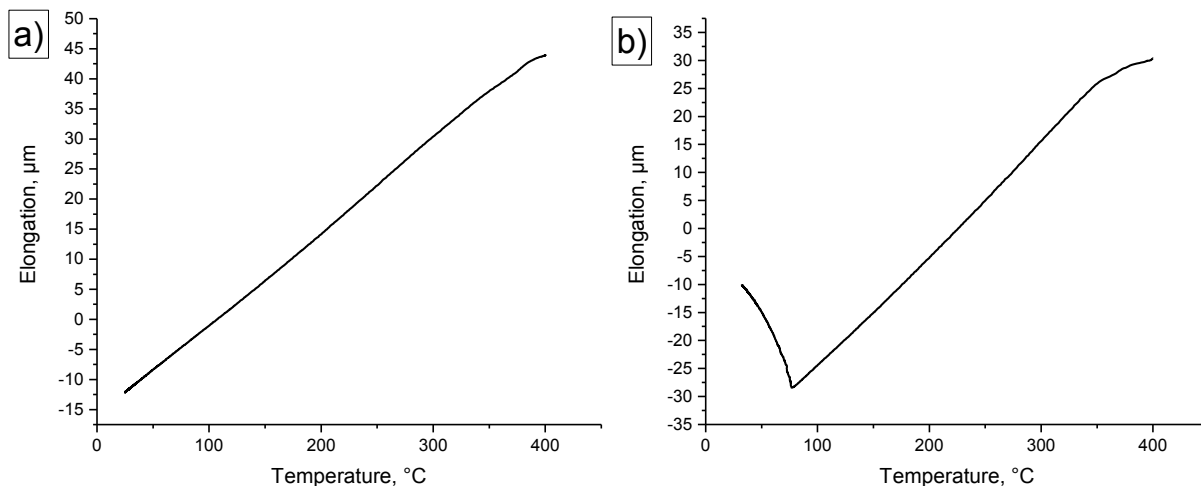


Figure 4. Dilatometer measurements of transformation behaviour during final cooling to room temperature after partitioning: a) austenitizing → quenching to QT1 (25 °C) → partitioning and b) austenitizing → quenching to QT2 (100 °C) → partitioning.

3.2 Mechanical properties

The results of compression testing are summarized in table 2. It has to be considered that all tests stopped at the machine maximum force of 100 kN before fracture of the samples. So an evaluation of total elongation and compression strength is hindered.

What can be seen from the results is a decrease of compression yield strength with increasing partitioning time. For the as quenched states a yield strength of 1885 MPa is achieved. For partitioned states a loss of ~100 MPa (1 min partitioned) and ~300 MPa (30 min partitioned) is registered. While the maximum compressive strength is nearly the same for all states (~3500 MPa), the elongation when reaching the machine limit increases with increasing partitioning time from 13.7 % (as quenched state) to 21.7 % (1 min partitioned) and 34.1 % (30 min partitioned). The enhancement of elongation behaviour is attributed to the evolution of austenitic phase and the softening of martensite as further investigations will prove.

Table 2. Compression behaviour: comparison of an as quenched with a 1 min and a 30 min partitioned sample.

state	$R_{p0.2}$ (MPa)	σ_{max} (MPa)	ϵ_{max} (%)
as quenched	1885	3366*	13.7
1 min partitioned	1788	3525*	21.7
30 min partitioned	1602	3602*	34.1

*tension when reaching the force limit of the testing machine

3.3 Phase fraction

Figure 5 shows the results from XRD measurements. For the as quenched state there is a small peak of austenite. This is shifted compared to the reference data (red bars), because of substitutional elements and residual stresses as a consequence of phase transformation. The partitioned states are characterized by a time dependent increasing of integral intensity of austenite peaks and decreased integral intensity of ferrite peaks. Hence, the partitioning treatment results in a higher austenite fraction, which grows with increasing partitioning time. Notable is the significant increasing of austenite fraction even for the 1 min partitioned state. Such a high amount of austenite causes the significant enhanced elongation seen in results of compression testing. The carbon enrichment of austenite during the partitioning process additionally enables the TRIP effect under loading condition. While the peak shift compared with reference data remains unaffected, the width of peaks becomes smaller with increasing partitioning time. This signalizes a decrease of second and third order stresses which is a result of relaxation of martensite, whereas the first order stresses are constant. Because of the used Cu K α radiation no quantitative analysis of phase fractions was possible.

3.4 SEM investigations

The EBSD investigation of the as quenched state, figure 6 a), proves the existence of retained austenite islands with an area fraction of 9 % within the microstructure. Compared to that the 1 min partitioned sample (figure 6 b)) shows a smaller austenite fraction, which is not confirm with XRD results. This is attributed to circumstance of small size of reversed austenite in the initial state of partitioning. The 30 min partitioned sample (figure 6 c)) is characterised by an austenite fraction of 39 %. Moreover two types of austenite can be found. There are islands of coarse austenite and dispersed needle shaped austenite located on former martensite – martensite boundaries. In agreement with the investigations of YUAN et al. [12] the coarse one can be characterised as stabilized retained austenite and the needle shaped austenite represent reversed austenite. So the EBSD investigations confirm that the growth of austenite fraction due to reversion during the partitioning treatment is a time dependent process. It is expected that the carbon diffusion to austenite during partitioning leads to a softening of remaining martensite which enhances the formability of the specimen further. A correlation between Image Quality and stress level development due to the partitioning couldn't be observed.

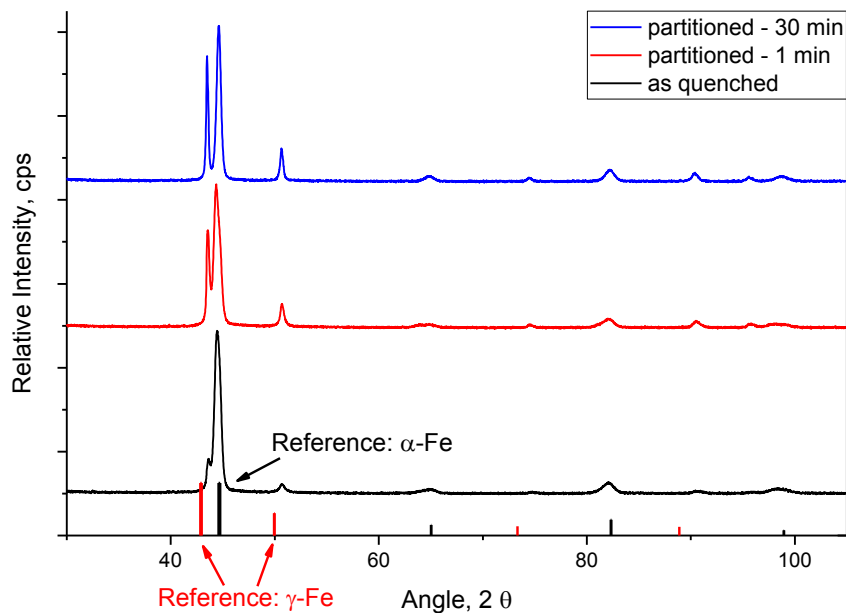


Figure 5. XRD measurements: comparison of an as quenched with a 1 min and 30 min partitioned sample.

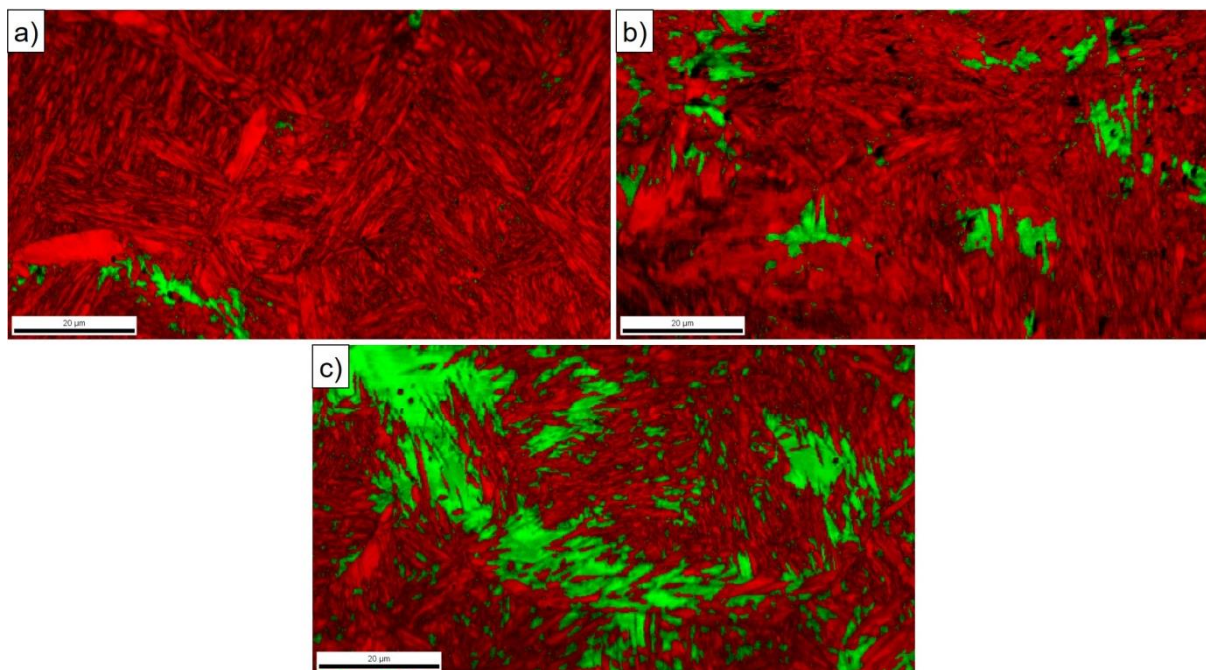


Figure 6. EBSD maps (Image Quality overlay by phase classification - fcc (austenite): green, bcc (martensite): red): comparison of a) an as quenched with b) a 1 min and c) a 30 min partitioned sample.

SEM investigations on the 1 min partitioned state, figure 7 a), are confirm to the EBSD results in such a way that this sample has a microstructure similar to the as quenched state, showing a martensitic matrix and austenitic islands. Additionally globular pores with white surroundings can be found in the SEM pictures. The high Cr content of these white edges, examined with EDX, figure 7 d), indicates the presence of carbides breaking out during sample preparation. These carbides remain from the large

carbides of the soft-annealed raw material, which means that the austenitizing time was insufficient. Thus less C is available for the austenite stabilizing and reversion, which is expected to restrict the maximum of austenite phase fraction due to partitioning. Also the 30 min partitioned sample shows these kind of pores resulting from carbide break-out, figure 7 b). When raising the magnitude, figure 7 c), nm-sized white needles within the martensite laths become visible. Similar investigations of LU et al. characterized them as M_3C [14]. The fact, that the earlier stage of partitioning not includes this small carbide needles, define them to be precipitations as a consequence of extended partitioning time. Thus a precipitation hardening should improve the strength of the 30 min partitioned state in case of having an optimum size regarding OROWAN mechanism [17]. The extent of the influence of carbide precipitations on strength development cannot be clarified in this study and has to be investigated in future work.

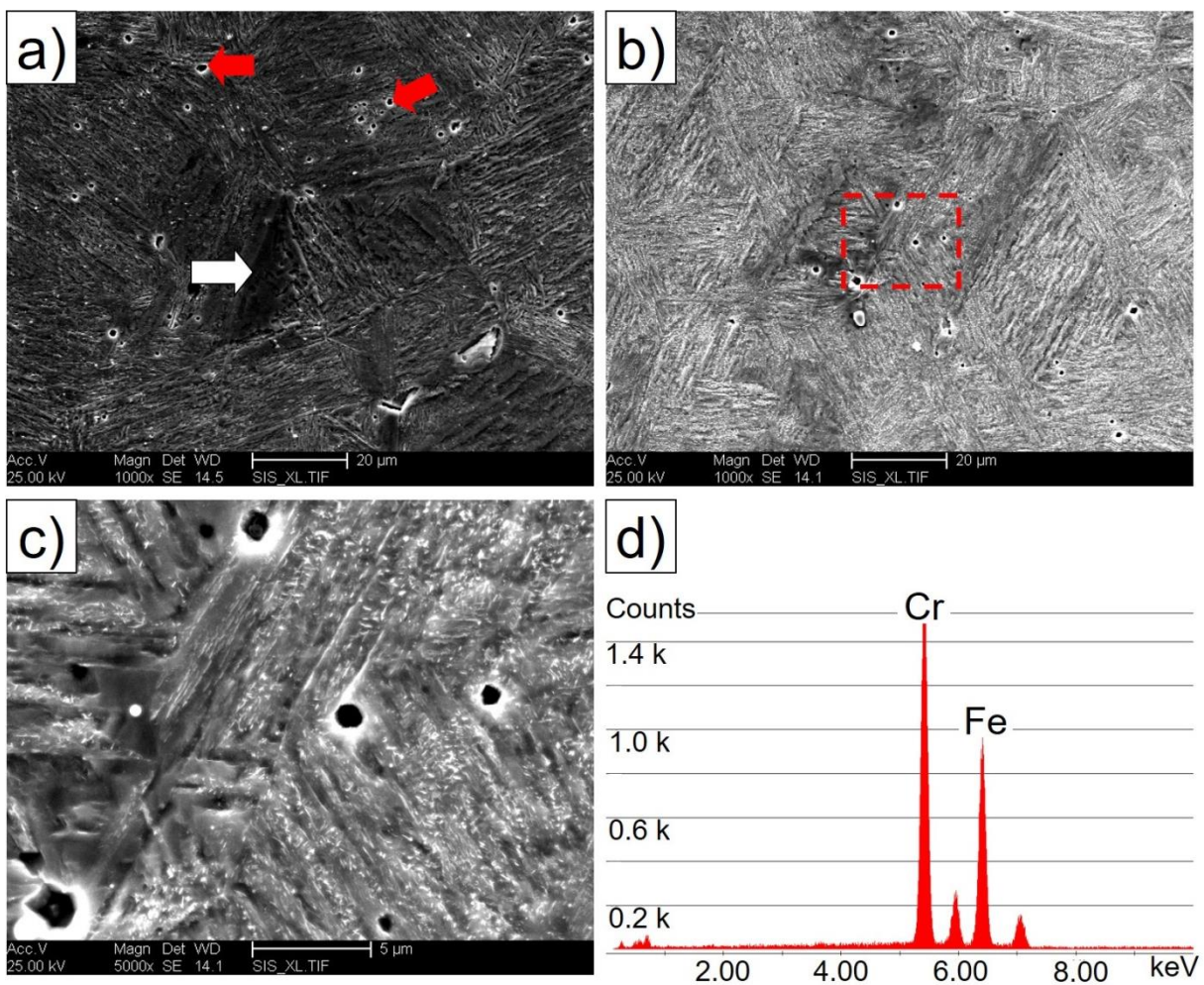


Figure 7. SEM (SE contrast) investigations on a) the 1 min partitioned state, showing austenite (white arrow and former carbide locations (red arrow), and b) the 30 min partitioned state; higher magnification of red window is shown in c). EDX analysis of the surrounding area of a pore is shown in d).

4. Conclusions and outlook

Quenching and partitioning heat treatment was applied to Fe-0.45C-0.65Mn-0.34Si-13.95Cr. The phase development based on different end temperatures of initial quenching was studied by dilatometer measurements. The mechanical properties were determined by compression testing and the microstructural evolution was investigated with XRD and SEM (EBSD). The results were summarized as follows:

- (1) When initial quenching to QT2 (100 °C) the carbon partitioning is insufficient to stabilize the elevated volume fraction of retained austenite compared with an initial quenching to 25 °C. As a consequence unstabilized retained austenite transforms into fresh martensite during final cooling.
- (2) An additional partitioning at 400 °C of the QT1 (25 °C) state leads to an enhanced elongation behaviour in compression testing. However, there is a loss in compression yield strength. Although the enhancement of elongation increases when increasing the partitioning time up to 30 min.
- (3) The austenite fraction increases with the time of partitioning treatment. This effect is based on the reversion of martensite to austenite due to carbon diffusion out of supersaturated martensite and local enrichment on martensite-martensite and martensite-austenite boundaries. A softening of the remaining martensite was connected with this process.
- (4) A partitioning at 400 °C leads to a time dependent precipitation of carbides, which has an additional strengthening effect beside TRIP, i.e. compensation of strength loss by austenite formation (ductility gain).

Acknowledgment

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References

- [1] Edmonds D V, He K, Rizzo F C, De Cooman B C, Matlock D K and Speer J G 2006 *Mater. Sci. Eng. A* **438-440** 25–34
- [2] Krauss G and Repas P E 1992 *Fundamentals of Aging and Tempering in Bainitic and Martensitic Steel Products* (Warrendale: Iron and Steel Society)
- [3] Irvine K J and Crowe D J and 1979 *The Metallurgical Evolution of Stainless Steels* ed F B Pickering (Ohio: American Society for Metals) pp 43–62
- [4] Gaunkar G V P, Huntz A M and Lacombe P 1980 *Met. Sci.* **14** 241–52
- [5] Speer J G, Rizzo Assunção F C, Matlock D K and Edmonds D V 2005 *Mater. Res.* **8** 417–23
- [6] Wang L and Speer J G 2013 *Metallogr. Microstruct. Anal.* **2** 268–81
- [7] Santofimia M J, Zhao L and Sietsma J 2011 *Metall. Mater. Trans. A* **42** 3620–6
- [8] Torralba J M, Navarro A and Campos M 2013 *Mater. Sci. Eng. A* **537** 253–6
- [9] De Moor E, Lacroix S, Clarke A J, Penning J and Speer J G 2008 *Metall. Mater. Trans. A* **39** 2586–9
- [10] Hsu T Y and Jin X 2011 Ultra-high Strength Steel Treated by Using Quenching–Partitioning–Tempering Process *Advanced Steels* ed Y Weng et al. (Beijing: Springer) pp 67–73
- [11] Fonstein N 2015 *Advanced High Strength Sheet Steels* (Chicago: Springer) pp 350–4
- [12] Yuan L, Ponge D, Wittig J, Choi P, Jiménez J A and Raabe D 2012 *Act. Mat.* **60** 2790–804
- [13] Thermo-Calc Software with limited academic version of Thermo-Calc database version 3.1 (accessed on March 2014)
- [14] Lu S-Y, Yao K-F, Chen Y-B, Wang M-H, Chen N and Ge X-Y 2016 *Corr. Sci.* **103** 95–104
- [15] Hao Q, Qin S, Liu Y, Zou X, Chen N and Rong Y 2016 *Mater. Sci. Eng. A* **671** 135–46
- [16] Lee C G, Kim S-J, Lee T-H and Lee S 2004 *Mater. Sci. Eng. A* **371** 16–23
- [17] Rösler J, Harders H and Bäker M 2012 *Mechanisches Verhalten der Werkstoffe* (Wiesbaden: Springer Vieweg) pp 206–11