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‘Quenching and Partitioning’ - an in-situ approach to characterize the process kinetics and the final microstructure of TRIP-assisted steel

Thomas Rieger^{1,a}, Klaus Herrmann^{1,b}, Dagmar Carmele^{2,c}, Stephan Meyer^{2,d},
Thomas Lippmann^{3,e}, Andreas Stark^{3,f}, Wolfgang Bleck^{1,g}, Uwe Klemradt^{2,h}

¹Department of Ferrous Metallurgy, RWTH Aachen University, D-52056 Aachen, Germany

²II. Institute of Physics B, RWTH Aachen University, D- 52056 Aachen, Germany

³Institute of Materials Research, Helmholtz-Zentrum Geesthacht (HZG),
Max-Planck-Straße 1, D-21502 Geesthacht, Germany

^arieger@iehk.rwth-aachen.de, ^bherrmann@iehk.rwth-aachen.de,

^ccarmele@physik.rwth-aachen.de, ^dmeyer@physik.rwth-aachen.de, ^ethomas.lippmann@hzg.de,

^fandreas.stark@hzg.de, ^gbleck@iehk.rwth-aachen.de, ^hklemradt@physik.rwth-aachen.de

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Abstract. The ‘Quenching and Partitioning’ (Q&P) concept aims to increase the strength level of conventional TRIP-assisted advanced high strength steel (AHSS) by replacing ferritic constituents by tempered martensite. The Q&P heat treatment process involves austenitization and interrupted quenching followed by carbon partitioning from martensite to austenite at elevated temperatures. The final microstructure is traditionally investigated at room temperature after metallographic preparation by microscopy and x-ray analysis with laboratory tubes. Besides other disadvantages the established characterization methods are not adequate to observe the development of the microstructure during Q&P treatment.

In the present work the microstructural evolution during Q&P processing was monitored by in-situ diffraction experiments using very hard (100 keV) synchrotron x-ray radiation. Debye-Scherrer rings were recorded as a function of time and temperature during the heat treatment in a state-of-the-art dilatometer (type Bähr DIL805AD) at the Engineering Materials Science beamline HARWI-II (HZG outstation at Deutsches Elektronensynchrotron (DESY), Hamburg). The diffraction patterns contain quantitative information on the phases present in the sample (for more details cf. Abstract Carmele et al, this conference). The evolution of the austenite phase fraction during the partitioning treatment at the quench temperature (1-step Q&P) is discussed exemplarily for a Si-based TRIP steel with additions of Ni.

Introduction

The heat treatment called ‘Quenching and Partitioning’ (Q&P) has gained much scientific interest since its first publication in 2003 [1]. The concept intends the substitution of polygonal and bainitic ferrite in TRIP-assisted steels by carbon decreased martensite. The martensitic constituents are reflected in an increased strength. A satisfying ductility is assured by the TRIP-assisted strain hardening. The process starts with intercritical / full austenizing and quenching to the quench temperature QT between martensite start M_s and finish M_f , **Fig. 1**. It is followed by isothermal holding at QT (1 Step Q&P) or at an elevated partitioning temperature PT (2 Step Q&P). During the isothermal holding, carbon diffuses from martensite into austenite decreasing its M_s and M_f temperatures. Hence the austenite remains stable during the concluding quenching to room temperature [2, 3, 4].

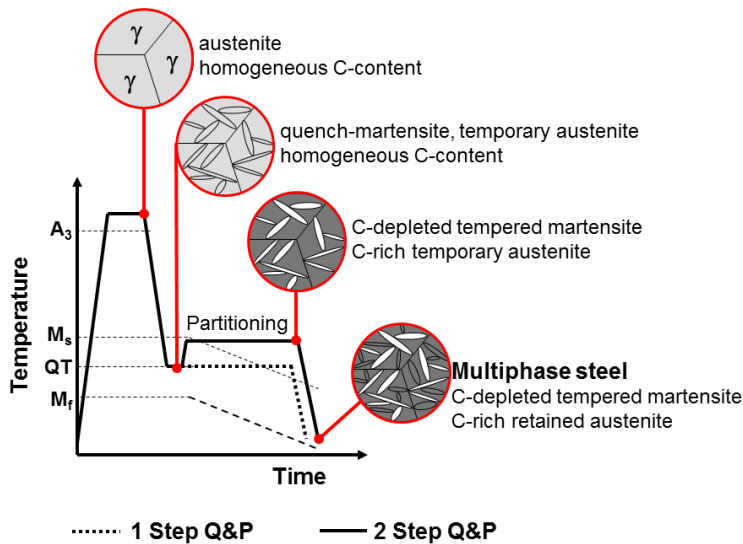


Fig. 1 Schematic microstructural evolution during Q&P heat treatment process.

The characterization of multiphase steels is an important task in the scientific and industrial practice since the mechanical properties are determined by the microstructural features. Established investigation methods comprise optical and electron microscopy as well as x-ray analysis with laboratory tubes. Yet the common methods are usually applied at room temperature and their accuracy is often limited by a small sampled region and preparation artifacts [5, 6, 7].

In the present paper the microstructure evolution during the heat treatment is observed by synchrotron x-ray diffraction. The evolution of the austenite phase fraction during partitioning at quench temperature (1-step Q&P) is discussed exemplarily for a Si-/Ni-alloyed TRIP steel.

Experimental

Three alloys with moderate carbon contents were investigated, **Table 1**. HCT690T is a standardized automotive TRIP steel based on aluminum additions for carbide inhibition [8]. QPSN and K1 are

Table 1 Chemical composition of investigated steels, mass content in % (*n. a.* - not analyzed).

Alloy	C	Si	Mn	P	S	Al	B	N	Ni	Cr	Ti
HCT690T	0.248	0.036	1.623	<0.120	<0.015	1.246	n.a.	n.a.	0.012	0.023	n.a.
QPSN	0.200	1.910	1.640	<0.002	<0.010	n.a.	n.a.	0.002	3.100	n.a.	n.a.
K1	0.229	1.510	1.490	0.005	n.a.	0.022	0.0031	0.001	0.020	0.014	0.010

TRIP compositions on a Si basis produced in laboratory and modified by Ni (QPSN) or Ti/B (K1) additions to decrease the critical cooling rate.

Specimens of 7 mm x 4 mm x 1.3 mm were processed in a state-of-the-art dilatometer (Bähr Thermoanalyse 805 AD) with inductive heating ($dT/dt = 25$ K/s) and gas quenching (Ar, $t_{8/5}=17$ s). The dilatometer was installed at the Engineering Materials Science beamline HARWI-II (HZG outstation at Deutsches Elektronensynchrotron (DESY), Hamburg). Debye-Scherrer patterns were recorded in-situ with a photon energy of 100 keV (wavelength 0.0124 nm): The transmission geometry of the experimental setup allowed the investigation of ca. 0.75 mm³ in an exposure time of 1 s. Powder diffractograms were azimuthally integrated to observe the microstructural evolution during the heat treatment, **Fig. 2**. The theoretical peak positions were calculated to identify the corresponding phases and diffracting lattice planes. Following the procedure described in [9], the integrated intensities were compared pairwise ($(110)_{bcc}$ or $(112)_{bcc}$ to $(111)_{fcc}$ or $(220)_{fcc}$) to obtain the average phase fraction. Details of the experiment are given by *Carmelet al.*, this publication.

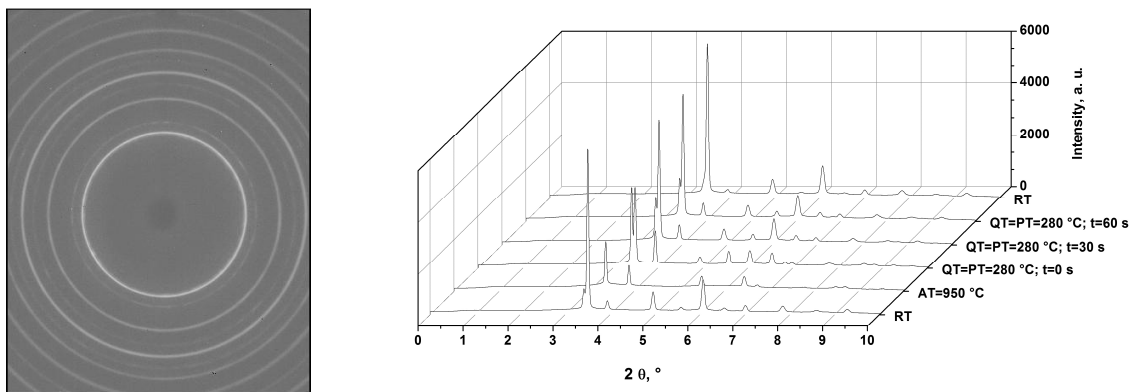


Fig. 2 X-ray scattering data and azimuthally integrated intensities during Q&P processing of QPSN. In an exemplary manner the amount of retained austenite was quantified for alloy QPSN. The phase fractions were evaluated during 1 Step Q&P at quench temperatures of 250 °C, 280 °C and 300 °C for partitioning times of 0 s (immediately after quenching), 30 s and 60 s. In **Fig. 3**, the recorded time temperature cycles are shown. Additionally the corresponding relative elongation is displayed for QT=280 °C.

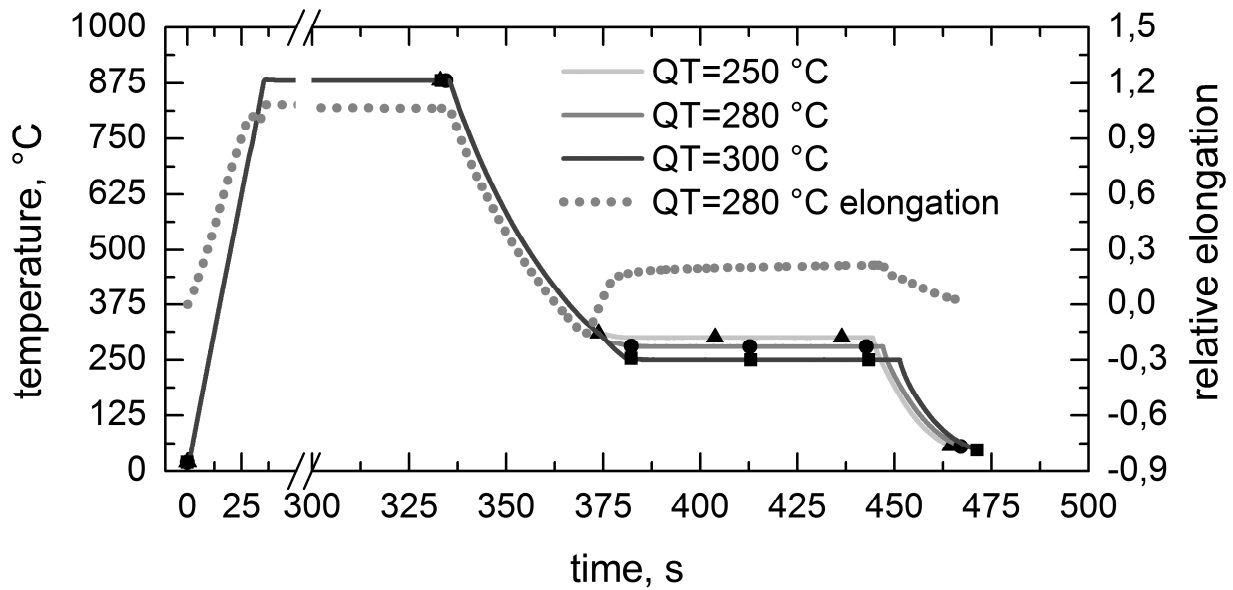


Fig. 3 Dilatometer heat treatment: time-temperature cycles and measured elongation (exemplary plot for QT 280 °C). Marker symbols indicate in-situ diffraction measurements evaluated in Fig. 4.

Results

The recorded time-temperature curves coincide during the analogous austenizing treatment and split during the quenching to the different quenching temperatures QT. The relative elongation signal (dotted line, **Fig. 3**) mirrors the temperature cycle. Deviations from a linear relation become obvious during heating to the austenizing temperature, during cooling to QT and during final cooling to RT. Furthermore an increase of the relative elongation is observed during the isothermal holding time at QT, though far less expressed compared to the deviations during quenching.

The results of the according diffraction experiments are featured in

Fig. 4. The austenite phase fraction is quantified and displayed as function of the partitioning time (t) and the quench temperature QT. A fully austenitic microstructure appears at the austenizing temperature AT. After reaching the quenching temperature (t=0 s) the austenite fraction decreases as expected with decreasing QT to 88 % at QT=300 °C and to 41 % at QT=250 °C. The austenite fraction drops significantly after isothermal holding for 30 s (t=30 s). An additional 30 s partitioning time (t=60 s) leaves the austenite fraction nearly unaltered. Remarkably, the concluding quenching operation diminishes its content for all three investigated QTs to around 6.0 %. It should be noted that the dilatometric elongation curve (dotted line, **Fig. 3**) does not distinctly show a phase transformation though more than 20 % of the austenite present at QT=280 °C / Pt 60 s disappear during cooling to room temperature RT.

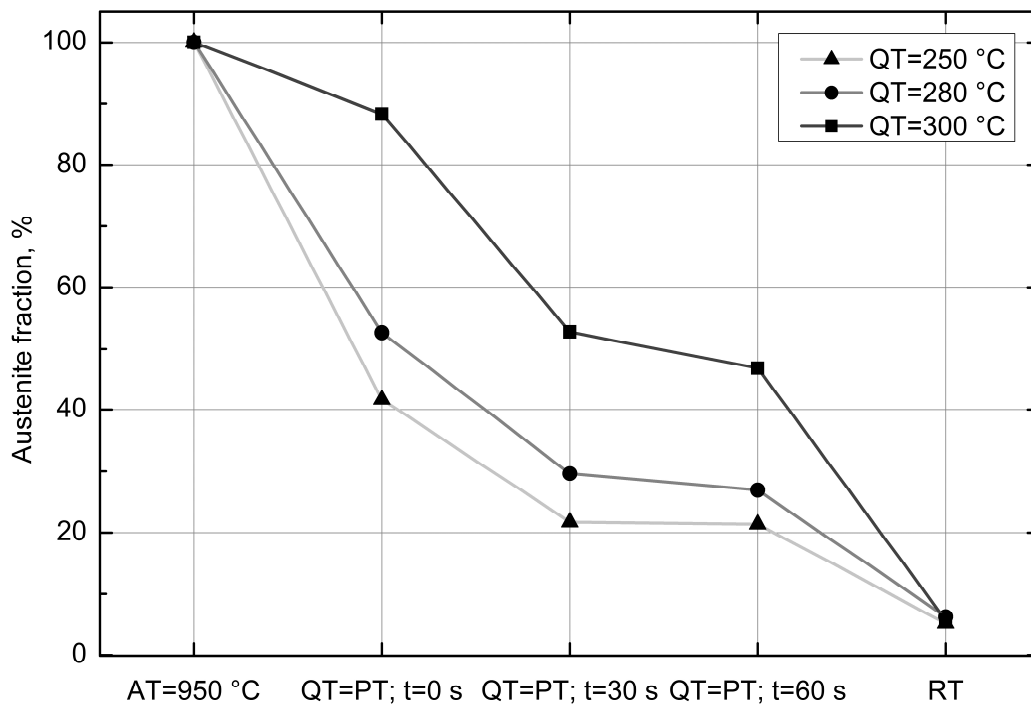


Fig. 4 Austenite fraction as function of partitioning time (t) at quench temperatures QT=250 °C / QT=280 °C / QT=300 °C. The lines are a guide to the eye.

Discussion

The coinciding time-temperature cycles during the austenizing provide strong evidence for the reproducibility of the heat treatment process. The recorded change in the relative elongation indicates the phase transformation to austenite during heating to high temperatures (AT=950 °C). Based on the associated temperature range, the observed phase transitions during cooling to QT and to RT, respectively, can be attributed to martensitic transformations. Due to the altered phase composition before and after the heat treatment the relative elongation may attain negative values. Yet the exact phase composition, i. e. the evolution of the phase fraction of austenite is unknown. The austenite phase fraction is of special interest at the very beginning of the partitioning as it defines the initial conditions for the partitioning treatment: the possible amount of austenite to be stabilized and the accessible amount of carbon in supersaturated martensite for its chemical stabilization are set.

Complementary information is obtained by the synchrotron diffraction experiments which allow for a direct measurement of the austenite content as function of time and temperature due to the short exposure time of 1 s. Starting from a fully austenitic material at AT, the austenite content was quantified at QT, during the isothermal holding and at RT. The austenitic phase fraction was constantly reduced with a faster transformation rate at higher temperatures QT. An important amount of the fcc phase transformed isothermally, which is only poorly mirrored in a slight increase

of the relative elongation measured dilatometrically. The isothermal transformation becomes negligible after the first 30 s at the holding temperature. An even shorter time may be sufficient but was not detectable during the experiments due to the restricted time resolution.

Unexpectedly, the QT does not seem to be relevant for the final austenite content at room temperature after Q&P processing for the parameter range investigated here. For a process design aiming exclusively at the volume content of retained austenite this implies a surprisingly simple and robust process. A sophisticated control of the quench temperature would not be necessary. However the reported amount of circa 6.0 % of retained austenite will probably be insufficient for a TRIP-assisted enhancement of the ductility.

Outlook

The presented methodology combines the reproducible heat treatment in a state-of-the-art dilatometer with the advantages of diffraction experiments with very hard synchrotron radiation. The effect of the heat treatment parameters on the final austenite content needs to be clarified. Further validation of the accuracy of the diffraction results and experiments with a higher time resolution would be desirable. Furthermore the change of the austenite lattice parameter during partitioning indicates carbon enrichment. Rietveld refinement of the diffraction patterns and an evaluation of the resulting mechanical properties will therefore be future tasks.

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