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Very hard synchrotron x-ray radiation as an advanced characterization method applied to advanced high-strength steels

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Abstract. Innovative steel materials of the third generation of advanced high-strength steel (AHSS) are based on complex multiphase microstructures on a submicron scale, which are adjusted in a heat treatment procedure. Established methods for microstructural characterization are usually applied after the heat treatment process (ex-situ) at room temperature and comprise amongst others X-ray analysis based on laboratory tubes with photon energies of several keV. The corresponding penetration depths are on the micron scale. Additionally, the results may be affected by the metallographic preparation process. Using very hard synchrotron X-ray radiation with photon energies of up to 100 keV, penetration depths in the millimetre range are realized and macroscopic volumes (mm³) can be investigated. Furthermore the photon flux of synchrotron sources is several orders of magnitude higher compared to laboratory tubes. Consequently in-situ measurements during a heat treatment process can be performed. Using the example of the standardized multiphase TRIP steel HCT690T, a microstructural investigation with high energy synchrotron X-ray radiation is discussed and compared to established diffraction methods using Co- and Cu-K_α-radiation. In-situ diffraction measurements during a heat treatment are exemplarily shown.

Introduction

The third generation of AHSS is of great interest in the automotive sector. The complex microstructures of AHSS consist of multiple phases, e. g. polygonal ferrite (bcc), bainite (bcc), martensite (bct) and metastable retained austenite (fcc). Their phase features are adjusted in heat treatment processes and determine the mechanical properties. Here retained austenite is of special interest as it promotes ductility due to its martensitic transformation during plastic deformation (transformation induced plasticity (TRIP) effect). Industrialized TRIP steels contain ca. 85 % polygonal and/or bainitic ferrite, up to 15 % retained austenite and traces of martensite [1,2,3].

Substantially improved mechanical properties are predicted for duplex microstructures consisting of martensite and retained austenite. An adequate processing concept called 'Quenching and Partitioning' (Q&P) was presented in 2003, aiming at the substitution of ferritic constituents in TRIP steels by carbon depleted martensite [4,5,6]. The heat treatment involves heating to high temperatures, fast cooling to foster diffusionless martensite transformation and isothermal holding for carbon partitioning to austenite (for details cf. *Rieger et. al.*, this conference). The investigation of the resulting retained austenite in carbon-depleted martensite is a challenging task.

Methods of phase analysis

The microstructural composition of steel is traditionally developed by metallographic preparation procedures involving grinding, polishing and etching techniques, using mainly optical and electron microscopy. All these methods are usually applied only at room temperature, and only the surface region with potential preparation artefacts can be observed [3,7,8]. For multiphase steels like AHSS X-ray diffraction is an established methodology for the quantitative determination of the austenite (face centered cubic, fcc) phase fraction compared to the eventually tetragonally distorted body centered (bcc) phases. The amount of the respective phases is quantified by comparison of the integrated intensities of the corresponding peaks in the diffractogram [9,10,11,12]. A more exact way to determine multiple phase compositions is the Rietveld method, which takes into account the full pattern of the measurement. A theoretical diffractogram is simulated based on the crystal structure of each phase. Then the calculated parameters are mathematically fitted to the experimental data [13,14].

Typically, for X-ray diffraction measurements of steels, laboratory X-ray tubes with K_α-radiation energies of several keV [15,16,17] are used. Common target materials are Mo (17.4 keV), Co (6.9 keV) and Cu (8.0 keV) [15]. The corresponding penetration depth of the radiation is on the micron scale and depends strongly on the X-ray wavelength, Fig. 1.Hence only a small part of the sample region is accessible for evaluation with laboratory X-ray tubes. Using synchrotron radiation with photon energies above 60 keV, the penetration depth is already in the millimeter range. Thus a significant and representative sample volume can be investigated, even the transmission of a steel sheet is possible. Consequently macroscopic specimens may be investigated without prior preparation process and associated artefacts. Whereas the usable wavelength of an X-ray tube is fixed by the target material, at a synchrotron source the X-ray wavelength can be tuned in a very broad range. Moreover, the photon flux at synchrotron sources is several orders of magnitude higher than at X-ray tubes, allowing for in-situ measurements the during the heat treatment process.

Experimental Setup

The results of in-situ diffraction experiments with high energy synchrotron radiation at the HARWI-II beamline at DESY were compared to laboratory diffraction experiments with Cu and Co

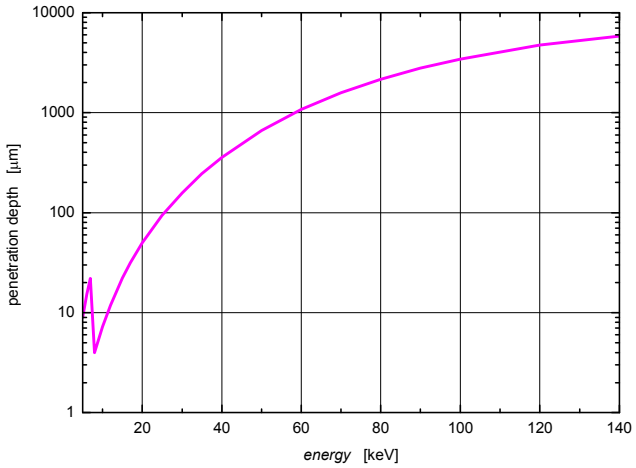

Fig. 1: X-ray penetration depth for steel as a function of photon energy. The dip is caused by the Fe K-edge at 7.1 keV.

radiation at room temperature. The experimental setups are summarized in Table 1. The standardized TRIP steel HCT690T [18] was utilized for the experiments, Table 2. Specimens were cut from industrially produced sheet of dimensions 7 mm x 4 mm x 1.3 mm.

For the diffraction experiments using Cu- and Co-K_α-radiation, the specimens were embedded in a cold hardening resin, grinded, polished with a polishing suspension (3 µm) and electrolytic polishing. State-of-the-art diffractometers in Bragg-Brentano geometry were used. The Engineering Materials Science beamline HARWI-II is located at Hasylab (DESY) in Hamburg and an outstation of the Helmholtz-Centre Geesthacht. A transmission geometry setup was used with image plate detectors and an exposure time of 1 s. The measured diffraction patterns were azimuthally integrated to powder diffractograms. Temperature dependent results are presented for one specimen which was heat treated at HARWI-II beamline in a state-of-the-art dilatometer (Bähr Thermoanalyse 805 AD) with inductive heating (dT/dT = 25 K/s) and gas quenching (Ar, dT/dT < 40 K/s). The time-temperature cycle is shown in Fig. 2 with four marks corresponding to the in-situ recording of diffraction patterns during the heat treatment. In order to compare the measurements at HARWI-II and those with Co radiation quantitatively, the phase fraction of retained austenite was calculated at room temperature (initial condition) from a Rietveld refinement.

Table 1 Used energies and wavelengths, *[16] T: transmission, R: reflection. RT: room temperature.

Source	Energy [keV]	Wavelength [Å]	Geometry	Heat treatment
HARWI-II beamline	100.0	0.124	T	RT / in-situ Q&P
X-ray tube (Cu target*)	8.0	1.542	R	RT
X-ray tube (Co target*)	6.9	1.790	R	RT

Table 2 Chemical composition of the investigated steel, mass content in % (*n.a. – not analyzed*).

Alloy	C	Si	Mn	P	S	Al	N
HCT690T	0.248	0.036	1.623	<0.120	<0.015	1.246	n.a.

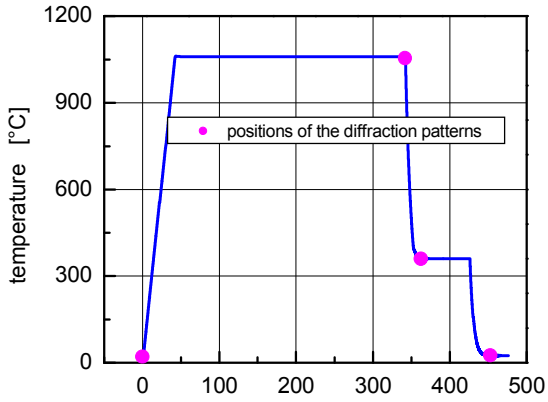

Fig. 2: Heat treatment process of the HCT690CT sample with marked positions of the four analyzed in-situ diffraction patterns (cf. Fig. 4).

Results and Discussion

A comparison of the diffractograms measured with different X-ray sources is shown in Fig. 3. The abscissa are scaled in units of sin θ / λ to account for the different wavelengths. While the diffractogram measured at the HARWI-II beamline covers a very large range and resolves many high order peaks, the diffractograms measured with tubes are necessarily much more limited. Additionally, it can be seen for the use of Cu-K_α-radiation that several peaks disappear in a background mainly caused by Fe fluorescence.

Exemplarily, a Rietveld refinement was made for the diffractograms measured with Co-K_α-radiation and synchrotron radiation. The results of the phase fractions are shown in Table 3. The volume fraction of the retained austenite measured with the laboratory tube is significantly smaller. Possible reasons are the different penetration depths of microns compared to a few millimeters, or a martensitic transformation of metastable austenite during the metallographic preparation.

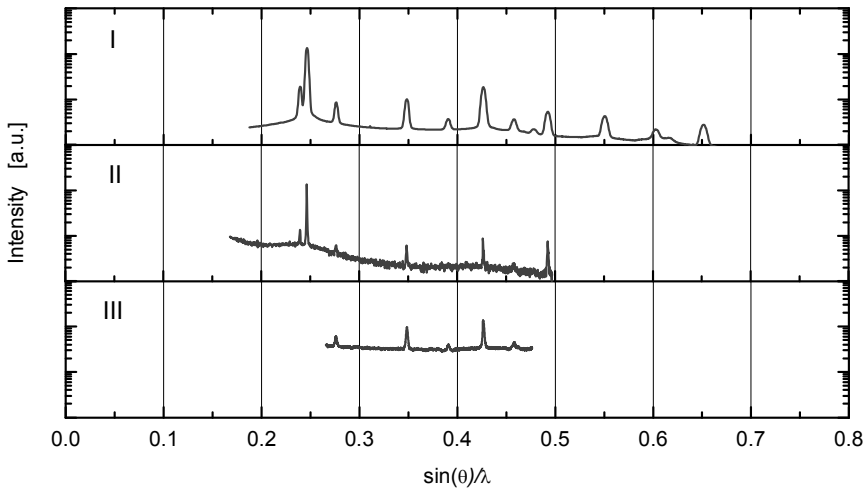

Fig. 3: Comparison of azimuthally integrated diffractograms measured at the beamline HARWI-II (I), with Co K_α-radiation (II) and with Cu K_α-radiation (III).

Table 3: Phase fraction of the different phases obtained with the Rietveld refinement method.

	fcc	bcc (Ferrite / Bainite)
RT measurement HARWI-II	16.4%	83.6%
RT measurement Co tube	13.3%	86.7%

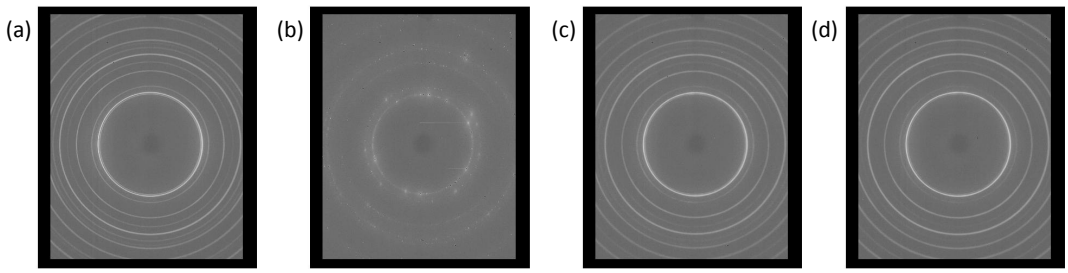

Fig. 4: Diffraction patterns of the HCT690T sample (a) at room temperature before heat treatment, (b) at the austenitizing temperature (1054.8°C), (c) at the quenching temperature (360.1°C), and (d) again at room temperature after the heat treatment.

Fig. 4 shows diffraction patterns measured in-situ during the heating treatment process. Both measurements at room temperature before and after the heating treatment as well as the diffractogram recorded at the quenching temperature (360.0°C) show obvious Debye-Scherrer rings (Fig. 4(a,c,d)). However, the pattern recorded at the austenitizing temperature (1054.8°C) only show very weak rings and a strong texture. The resulting diffractograms of the azimuthally integrated patterns a summarized in Fig. 5. Qualitatively, the diagrams clearly show a shift in the positions and intensities of the peaks and hence allow monitoring of the heat treatment. A quantitative evaluation of the phase composition can be done following the procedure described in [13], exemplarily used by *Rieger et al.* (this conference). The measurements show that diffraction information obtained during heat treatment can be analysed quantitatively using powder procedures.

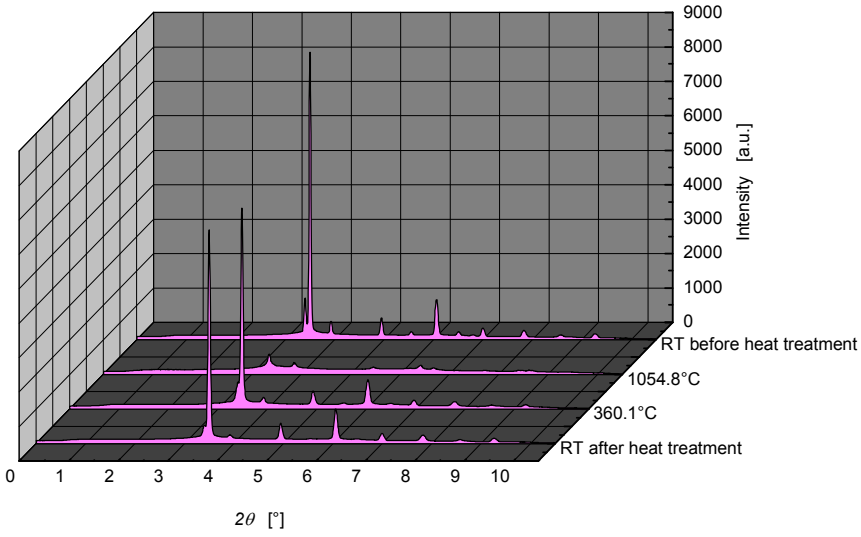

Fig. 5: Azimuthally integrated diffractograms of the diffraction patterns measured in-situ at different temperatures during the heat treatment.

Outlook

The use of high energy synchrotron radiation turns out to be an excellent tool for in-situ microstructural characterization during the heat treatment of AHSS. To exploit the whole capabilities, further quantitative investigations based on Rietveld refinements are necessary.

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