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Alloying effects on microstructure formation of dual phase steels

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ABSTRACT

In dual-phase (DP) steels, inherited microstructures and elemental distributions affect the kinetics and morphology of phase transformation phenomena and the mechanical properties of the final material. In order to study the inheritance process, we selected two model materials with the same average DP steel composition but with different initial microstructures, created by coiling at different temperatures after hot rolling. These samples were submitted to a DP-steel heat treatment consisting of a short isothermal annealing in the pure austenite region and a quenching process. The evolution of microstructure, chemical composition and mechanical properties (hardness) during this treatment was investigated.

The initial samples had a bainitic–martensitic $(B + M)$ microstructure for the material coiled at lower temperature and a ferritic–pearlitic $(P + F)$ microstructure for that coiled at higher temperature. The P + F microstructure had a much more inhomogeneous distribution of substitutional elements (in particular of Mn) and of carbon. After complete heat treatment, both materials showed a typical DP microstructure (martensite islands embedded in ferrite) but the P + F material showed lower hardness compared to the $B + M$ material. It was found that the inhomogeneous elemental distribution prevailed in the $P + F$ material.

The inheritance process was studied by combining measurements of the elemental distribution by Wavelength-Dispersive X-ray spectroscopy (WDX), simulations of the evolution of the elemental composition via the DICTRA (diffusion-controlled reactions) computer programme, dilatometry to observe the kinetics of phase transformation, and observation and quantification of the microstructures by Electron Backscatter Diffraction (EBSD) measurements. For the P + F material it was found that the $\alpha-\gamma$ transformation during annealing is slowed down in regions of lower Mn content and is therefore not completed. During the subsequent cooling the incompletely autenitized material does not require ferrite nucleation and the γ – α transformation starts at relative high temperatures. For B + M, in contrast, nucleation of ferrite is needed and the transformation starts at lower temperatures. As a result the B + M material develops a higher martensite content as well as a higher density of geometrically necessary dislocations (GNDs). It is speculated that for the B + M material the γ - α transformation occurs through a bainitic (i.e. partly displacive) process while the transformation at higher temperatures in the P + F material proceeds exclusively in a diffusive way.

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1. Introduction

Dual-phase steels consist of martensite islands embedded in a ferrite matrix. Typically, the martensite volume fraction is around 15–25% [\[1–5\].](#page--1-0) They are synthesized from a rather lean elemental composition (e.g. approx. 0.2 wt.% C, 1.5 wt.% Mn and 0.25% Si [\[6\]](#page--1-0)) via an intercritical heat treatment in the austenite (γ)–ferrite (α) two-phase region followed by quenching the material. The good mechanical properties $[1,7,8]$ combined with low costs make DP steels attractive as structural design materials. A number of

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works have addressed the optimization of the thermomechanical heat treatment [\[3,9–11\]](#page--1-0) and the resulting microstructures [\[7,12–](#page--1-0) [14\]](#page--1-0) and properties [\[12,15–18\]](#page--1-0) of these steels. For instance, Calcagnotto et al. used different heat treatments to lower the grain size of ferrite, since this improves the toughness of the DP-steel and the capability to absorb impact energy $[13]$. Also the initial microstructure of the steel before intercritical annealing controls, in different ways, the properties of a dual phase steel [\[7,19–25\].](#page--1-0)

Different initial microstructures may be related to different elemental distribution in the material and may influence the properties of the final material. The heterogeneous spatial distribution of substitutional elements, in particular, is of great importance for the properties of DP-steels. Manganese crystal-segregations from

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solidification, for example, are stretched out during rolling, leading to microstructures where the carbon-rich phases like pearlite, bainite or martensite are confined to those band-like areas which have a higher Mn-content. [\[23–26\].](#page--1-0) An inhomogeneous manganese distribution is not only found on the micrometer scale, where it is caused by solidification segregations, but also on the sub-micro- to nanometer scale. Here it is related to globular cementite or lamellar cementite in pearlite [\[22,27–29\].](#page--1-0) Cai et al. [\[22\]](#page--1-0) described the influence of different starting microstructures on the formation of a dual phase steel. To obtain the starting microstructures, they applied several heat treatments to different samples of the same type of steel. One of the applied heat treatments led to a pearlite microstructure, in which the cementite lamellae were enriched with Mn. They report that the cementite in pearlite must have formed under conditions of low carbon supersaturation, since these conditions are the only ones at which substantial partitioning of Mn can occur. During the intercritical annealing, which they applied to the pearlite-containing microstructure, these Mn-enrichments did not dissolve completely and after complete heat treatment a duplex microstructure consisting of ferrite and austenite/martensite was present. Calcagnotto et al. [\[30\]](#page--1-0) described the influence of globular cementite on the formation of a dual phase steel. They showed, that Mn-enrichments, caused by globular cementite, survive an intercritical annealing and enrichments can be found afterwards in austenite where they raise the hardenability. Apart from these two papers, little information related to the inheritance of elemental distribution has been found in the literature.

The scope of this study is to understand the influence of an inhomogeneous local distribution of Mn, related to individual carbide precipitates and to cementite in pearlite, on the reaustenization process of a dual-phase steel and on its mechanical properties after final heat treatment. Therefore, two different initial microstructures were created from a steel with typical DP-steel composition by using different coiling regimes after hot rolling. Afterwards, the same heat treatment, including an isothermal annealing above the AC3 temperature, was applied to both materials. The evolution of microstructure and elemental distribution was investigated using light optical microscopy (LOM), Scanning Electron Microscopy (SEM) with Electron Backscatter Diffraction (EBSD), Energy Dispersive X-ray spectroscopy (EDX), Wavelength Dispersive X-ray spectroscopy (WDX), dilatometry and hardness measurements. This was done at different stages during the heat treatment. The evolution of local Mn-enrichments during the heat treatment and the influence on the local phase transformation was furthermore investigated by thermodynamical simulations. For equilibrium calculations, ThermoCalc (TC) was used and simulations diffusional phase transformations were performed with DICTRA (TC) [\[31,32\]](#page--1-0).

2. Experimental methods

2.1. Material production

A commercial hot rolled steel with a typical DP-steel composition of 0.14 wt.% C, 1.9 wt.% Mn and 0.4 wt.% Cr, 0.25 wt.% Si was used as initial material in the form of 3.7 mm thick plates. Different initial microstructures were created by a variation of the coiling temperature after hot rolling during large-scale industrial production of the material. Note that the exact coiling temperatures cannot be given but they are below the AC1 temperature and differ by about 100 K. For the microstructural investigations and heat treatments, cylinders were cut from the material with a diameter of 4 mm and a length of 9 mm. The rolling direction was parallel to the cylinder axis. Since the total thickness of the sheet was slightly lower than 4 mm the cylinder had flat surfaces perpendicular to the normal direction.

All heat treatments were performed in a dilatometer (DIL 805A/D produced by TA Instruments) equipped with an induction heater. During heat treatments, an argon atmosphere was established in the dilatometer to prevent the material from oxidization. The thermal treatment is sketched in [Fig. 1](#page--1-0). A maximum temperature of 840 \degree C was applied for roughly 90 s. In total, the heat treatment took less than 10 min. In order to follow the evolution of the properties of the material, samples from 3 different stages were observed, as shown in [Fig. 1](#page--1-0): the initial material (point 1), material quenched after the isothermal holding (point 2) and final material after complete heat treatment (point 3).

2.2. Microstructural investigations

For microstructural investigations specimens were cut along the normal direction–rolling direction plane. Specimens were ground, polished until 3 µm and etched with 1 pct. Nital. Both, LOM and SEM were used for microstructural investigations.

The average austenite content was measured using X-ray diffraction (XRD). A Seifert Type ID 3003XRD system with a Meteor0D detector produced by General Electric was employed. The spectra were evaluated using the MAUD software package in version 2.33 [\[33\].](#page--1-0) The specimens were ground, polished until 3 µm and polished with Oxide Polishing Suspension (OPS).

EBSD was used to observe the distribution of ferrite, martensite and austenite. Specimens were prepared in the same way as those of LOM and XRD-analyses. A Zeiss-Crossbeam XB 1540-SEM equipped with an EDX silicon drift detector (Apollo XL) and an EBSD system with a Hikari camera provided by EDAX were used. The measurements were performed at an accelerating voltage of 15 kV and mostly with 100 nm step size. EBSD data were analysed with TSL OIM software version 6.2. Measurement points with a confidence index lower than 0.1 were excluded from the measure-ment [\[34\]](#page--1-0), whereby the confidence index is a criterion of the reliability of the indexing of a given EBSD pattern.

The distinction between ferrite and austenite is straightforward, as they differ in crystal structure. In contrast, the separation between martensite and ferrite by EBSD is more challenging, since the crystal structure of martensite and ferrite cannot be distinguished by conventional EBSD. In this paper, both were separated using the grain average image quality (IQ), as is described in detail by Pinard et al. $[35]$. The IQ is a measure for the quality of a diffraction pattern [\[36\]](#page--1-0). Martensite has a higher defect density than ferrite and shows, therefore, a clearly lower IQ value than ferrite. To exclude the effect of grain boundaries, which show a low IQ as well, the ''grain average (GA) IQ''-value was used. Since, in the present material, martensite and ferrite do not yield very different IQ values, the GA-IQ-value distribution is not sharply bimodal. Selecting the correct IQ threshold value for the differentiation between ferrite and martensite, therefore, is not unique. Thus, upper and lower values for the martensite content were determined and the average of both was set as the martensite content. As error for this procedure we selected half of the difference between upper and lower values. A quantitative and statistically representative measurement of the average martensite content of the material was performed by using a large area EBSD scan as described by Davut et al. [\[37\]](#page--1-0).

The performed heat treatment led to the formation of geometrically necessary dislocations (GND). Their density was determined using kernel average misorientation (KAM) calculations. In OIM data analysis (version 6.2) the KAM value is calculated from orientation maps as the average over all misorientation angles determined between a centre pixel and all its neighbours, thereby excluding pairs with larger misorientations than a defined

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