Identifying and quantifying microstructures in low-alloyed steels: a correlative approach

D. Britz, J. Webel, A.S. Schneider, F. Mücklich

Quantification of the microstructural constituents of complex multiphase steels is one of the most challenging tasks in modern metallography. Classical methods like light and scanning electron microscopy as well as electron backscatter diffraction (EBSD) provide insights into the microstructure of steels. However, these methods are mainly based on morphological or in the case of EBSD crystallographic orientation differences and thus have limitations in separating microstructural constituents, which differ only in dislocation density such as proeutectoid and bainitic ferrite. In order to get a reliable quantification and accordingly a better understanding of the microstructure-property relationship, different characterization techniques have to be combined in a correlative approach.

In the presented work, the results from classical metallography and EBSD were correlated to characterize different microstructures of a low-alloy steel produced by thermomechanical rolling and accelerated cooling. Different microstructures ranging from ferritic-pearlitic to ferritic-bainitic were realized in this steel by varying the finish cooling temperature. The results show that through the correlation of the different methods, a quantification of the different microstructures is possible.

**KEYWORDS:** MICROSTRUCTURE - LOW ALLOYED STEELS - BAINITE - CORRELATIVE MICROSCOPY - METALLOGRAPHY - EBSD

INTRODUCTION

Classical metallography for modern steels with fine-grained microstructures and microstructural constituents in the sub-micron range is limited due to the resolution of light optical microscopy (LOM). In particular, bainitic microstructures are a challenge to characterize and nor light optical microscopy neither scanning electron microscopy (SEM) are able to determine the amount of bainite in a multiphase steel: because of its complex appearance and morphology. Recently, a lot of studies have aimed to identify microstructures in steels based on their dislocation characteristics via electron backscatter diffraction (EBSD) [1]–[4]. For example, Kang et al. [3] showed that by analyzing the image quality (IQ), a separation of martensite from ferrite is possible. However, these techniques led to a huge spread in the quantification results due to variations in the experimental setup and post processing, especially because of a missing reference for the chosen thresholds [2], [5]–[8]. A more promising approach is the combination of different methods, like the combination of EBSD and local hardness measurements [9], [10] to gain a quantification of the different phases. In contrast to a single characterization technique, the correlative approach has the advantage of combining information of different micrographs and information depths as well as the possibility to cross validate the data and derive thresholds for the phase separation. Furthermore, the correlation of techniques such as LOM and EBSD allows combining morphological parameters of different phase constituents with local misorientation information. This is very interesting as it merges the well-known classical metallurgical information with high-resolution information of the substructures of the microstructure.

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Although several quantification methods have recently been developed and have shown to work for simple microstructures, there is still a strong demand for techniques enabling to characterize and quantify complex steel microstructures. Therefore, the goal of the presented work is to test a correlative approach based on EBSD and light microscopy and develop an appropriate quantification routine especially for bainitic and proeutectoid ferrite.

**EXPERIMENTAL**

**Material**

For this study a low alloyed thermo-mechanically rolled steel (TM-steel) with a carbon equivalent of 0.42 wt% was used. To produce samples with a dual phase microstructure consisting of a ferritic matrix and different second phase constituents, a plate was finish rolled in the dual phase regime and cooled with varying intensity across the plate length resulting in a wedge shape finish cooling temperature profile, as shown in Fig. 1. From the plate, samples were taken at the head and the center position, with two different finish cooling temperatures (FCT); one with a high FCT to get a microstructure consisting of ferrite and pearlite (FCT1) and one with a low FCT to have a ferritic microstructure with bainite as second phase (FCT2). The advantage of this procedure is that it ensures that the chemical composition, the prior austenite grains as well as the rolling conditions are the same and just the final cooling temperatures vary for the two samples.

**Sample preparation**

For EBSD it is crucial to obtain an extremely smooth sample surface with a minimum of plastic deformation. For this purpose, the samples were first ground starting with 80 grit SiC abrasive paper and successively using higher grits, ending at 2500. Afterwards the surface of the samples were polished using diamond paste with grains of 6 µm, 3 µm, 1 µm and finally an pH neutral oxide polishing called OP-AN from Struers. The different preparation steps with the specific duration are listed in Tab. 1.
Tab. 1 - Grinding and Polishing steps for the correlative investigation

<table>
<thead>
<tr>
<th>Grinding-/Polishing step</th>
<th>Duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>80 SiC</td>
<td>Until flat</td>
</tr>
<tr>
<td>180 SiC</td>
<td>1 min</td>
</tr>
<tr>
<td>320 SiC</td>
<td>1 min</td>
</tr>
<tr>
<td>600 SiC</td>
<td>1 min</td>
</tr>
<tr>
<td>1200 SiC</td>
<td>1 min</td>
</tr>
<tr>
<td>2500 SiC</td>
<td>1 min</td>
</tr>
<tr>
<td>6 µm Diamond</td>
<td>12 min</td>
</tr>
<tr>
<td>3 µm Diamond</td>
<td>5 min</td>
</tr>
<tr>
<td>1 µm Diamond</td>
<td>5 min</td>
</tr>
<tr>
<td>OP-AN</td>
<td>4 min + 2 min H₂O</td>
</tr>
</tbody>
</table>

In order to contrast the microstructure, for the different microscopy techniques were applied: a Pikral etching (3 g picric acid in 100 ml ethanol) and a modified Beraha etching (3 g potassium metabisulfite in 100 ml water).

**Methods**

The microstructure of the samples was characterized in a correlative approach using EBSD and light microscopy. This means that the two techniques were applied at the same sample position, marked by a triangular configuration of Vickers indents, in order to enable a direct comparison of the results.

In the first step, the samples were measured with EBSD using a Zeiss Merlin scanning electron microscope equipped with an EDAX Hikari system and operated at 20 kV and 20nA. Afterwards the same position was etched and imaged with a Zeiss Axio Imager 2 light optical microscope and an Olympus LEXT OLS4100 laser scanning microscope. In the further course of the work, the results from the light optical microscope and the laser scanning microscope were termed as LOM.

**RESULTS**

**Microstructure**

Fig. 2 and Fig. 3 show the microstructure of the two different samples (FCT1 and FCT2) imaged with classical light optical and scanning electron microscopy (SEM). For the optical microscopy, the samples were etched with Pikral, while for scanning electron microscopy a solution of modified Beraha etching was used for etching.

![Fig. 2](image)

(a) LOM micrograph of the Pikral etched FCT1 sample; b) SEM micrograph of the same sample, etched with a modified Beraha etching.
It can be seen that the microstructure of FCT1 is composed of a ferritic matrix with equiaxed grains and a pearlitic second phase. In contrast, the microstructure of FCT2 is more irregular and exhibits a ferritic matrix and a bainitic second phase characterized by an agglomeration of non-oriented cementite, partly next to grain boundaries.

Quantification
Quantification of the microstructure just based on LOM and SEM is not possible for the sample with FCT2, because the cementite is not always located in isolated grains but extends to a certain degree in the ferrite grains. Thus, a threshold value segmentation cannot be used to separate ferrite from bainite. A quantification of the bainitic structures based on EBSD measurements is also challenging, because there is no difference in the crystallography between the ferrite within the bainite and the proeutectoid ferrite. Furthermore it is not possible to map the very small cementite needles with the EBSD system. Therefore, a correlation between LOM and EBSD as a mean to quantify these structures was tested. As target parameter for EBSD the grain orientation spread (GOS) was chosen. This parameter describes the average misorientation of all pixels in a grain to the mean misorientation of the grain [6], which might be a suitable parameter to separate the different ferrite types based on their different dislocation densities and the related internal crystallographic misorientations. To find a good threshold for the separation of ferrite from bainite for this parameter, the sample with the ferritic-pearlitic microstructure (FCT1) was used. The microstructure of this sample exhibits no bainite and a clear boundary between ferrite and pearlite. Because both samples (FCT1 and FCT2) come from the same mother plate and only the final cooling temperature is different, the GOS threshold determined for the sample with the higher FCT should also apply for the sample with the lower FCT. With the purpose of determining the threshold $\theta$, the GOS angle was varied until the amount of second phase matched the amount determined by a reconstruction of the LOM micrograph of the same sample position, where the pearlite can clearly be seen. Everything below the threshold can then be assigned to proeutectoid ferrite, whereas everything over the threshold belongs to the second phase. With this approach, the threshold for the GOS was determined as 1.5°. In Fig. 4 a, the result of this EBSD based quantification is shown for the sample with the ferritic-bainitic microstructure. For a better comparison, the binarised micrograph from the LOM image in Fig. 4 b is overlaid on the EBSD map and grain boundaries are color coded for the differentiation of the GOS threshold. All grains with a GOS-threshold smaller than 1.5° are shown with green boundaries and grain boundaries of grains with a GOS-threshold larger than 1.5° are depicted in red.
All the cementite agglomerations visible in Fig. 4a are located within the red surrounded grains. This suggests that the grains with red boundaries represent bainitic grains. To further investigate the chosen GOS-threshold as a morphological parameter, the ratio between the convex perimeter of a grain and the total grain boundary length for the two GOS-fractions of the sample is shown in Fig. 5.

From the distribution of the morphological fraction demonstrated in Fig. 5 it can be deduced that the grains with a GOS value smaller than 1.5° are more roundish than the grains with a GOS larger than 1.5°. This supports the visual impression from the microscope images (Fig. 2b and Fig. 3b), where two different grain shapes can be identified. To quantify the amount of cementite in the grains after the separation with the GOS threshold value, the fraction of cementite per grain was plotted in Fig. 6. It can clearly be seen that almost all the cementite is located in the grains with a GOS larger than 1.5°, which supports that these grains represent the bainitic phase.

The total amount of bainite quantified using this correlative approach for the FCT2 sample is 48.3%. In addition to this, a manual segmentation of the bainite of the LOM micrograph from the same measuring field was carried out, which is very time consuming.
Thermomechanical processing

... consuming and subjective. This manual segmentation yielded a bainite amount of 45%, which is in good agreement with the results from the correlative approach.

DISCUSSION

Up to date, the quantification of bainite is still a challenge in the steel community [1]. Because of the complex structure of bainite, there is no successful metallographic approach to quantify this phase. In this study, it was demonstrated that by combining EBSD with LOM in a correlative approach and defining a threshold GOS value, the bainite content in a low carbon steel can be quantified. The GOS was chosen as parameter to separate the bainitic phase from the proeutectoid ferrite, as it can be used to reveal the different dislocation densities of the two phases. The higher dislocation density of the bainitic ferrite is related to its formation process [11]. As bainite forms in a shear transformation it contains a high density of geometrical necessary dislocations (GNDs) to accommodate the lattice strain imposed by the transformation. In contrast, the proeutectoid ferrite, which forms in reconstructive diffusion process, exhibits a low dislocation density. Both phases with different densities of GNDs can be differentiated by the GOS, if the right threshold value is available. This value was determined on a ferritic-pearlitic reference sample from the same mother plate and by the correlation of LOM and EBSD results. Another suitable parameter to separate bainite from ferrite is the kernel average misorientation (KAM), which was proposed by Zaefferer et. al. [7]. In the presented work, the GOS was preferred, because the threshold is already the discrimination of whole grains and not only parts of a grain like it is the case for the KAM. However, there are several issues that have to be considered if the GOS is used, which becomes apparent if the results of the manual segmentation (45%) and the GOS segmentation (48.3%) are compared.

For example, a larger grain always has a higher grain orientation spread than a small one, although the orientation gradient is the same in both grains. Therefore, the larger the proeutectoid ferrite grains are, the more difficult it gets to separate ferrite from bainite and the higher is the inaccuracy of this approach. This was confirmed on a pure iron sample, which showed an average grain size of more than 100 μm [13]. Accordingly, the bigger ferrite grains in the FCT2 sample might be one of the reasons for the slightly higher amount of bainite compared to the manual segmentation. Another reason for the difference between the two segmentation methods could be the better resolution of the EBSD-system relative to LOM. As a consequence, smaller grains as well as grains with a very small amount of cementite, which is the criterion for the LOM segmentation, are only taken into account in the GOS based approach.

CONCLUSION

The goal of the presented work was to find a method to separate proeutectoid and bainitic ferrite in a dual-phase steel. Instead of using the common methods independently, a correlative approach based on EBSD and LOM was applied. Using the grain orientation spread in EBSD, a threshold to quantify the bainite could be defined. This threshold was determined by using a reference sample of the same plate with a ferritic-pearlitic microstructure. With a manual quantification based on LOM, the threshold could be cross validated and a derivation of morphological parameters was possible.

REFERENCES