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Characterisation of the Microstructure of X80 Heavy Plate for Pipeline Applications using the EBSD Method

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Abstract
Modern high-strength heavy plates used in the production of UOE pipes with a yield stress of above 550 MPa are generally produced by thermomechanical rolling followed by accelerated cooling (TMCP). This processing route results in a microstructure that consists predominantly of bainite. The combination of high strength and high toughness of these steels is a result of the microstructure realised by TMCP and is influenced by cooling conditions. Classical light-optical characterisation of these steels is at its limits because the size of the features is too small to allow reliable quantitative results. Therefore alternative methods have to be used to obtain an understanding of the influence of processing conditions on the microstructure.

The microstructure of X80 heavy plates produced at Salzgitter Mannesmann Grobblech GmbH (MGB) and of plates rolled using a laboratory rolling mill was characterised using the electron backscatter diffraction (EBSD) method at the Salzgitter Mannesmann Forschung GmbH (SZMF), both in combination with an electron microprobe and a high-resolution scanning electron microscope. Measurements carried out on identical X80 heavy plate samples with both systems made it possible to assess the quality of the results obtained. With this method, it is possible to gain quantitative information on the grain size, misorientation within grains and the misorientation between neighbouring grains. Knowledge of the misorientation distribution, which is influenced by processing parameters, makes it possible to classify the bainite modifications observed.

EBSD measurements of plates rolled on the laboratory rolling mill followed by accelerated cooling showed that a variation of the cooling conditions has a direct influence on parameters that are accessible through the EBSD method. A correlation between mechanical properties and these parameters was also possible. These results can be used as a guideline for the definition of the processing window for heavy plate production depending on the required plate properties.

1. Introduction

Salzgitter Mannesmann Grobblech, a member of the Salzgitter group, runs a 5.1-meter heavy plate rolling mill which is specialized on the production of heavy plate for pipe. More than 90 percent of its more than 750,000 tons annual plate production is delivered to large diameter pipe mills all over the world. The most important and biggest customer is the German pipe producer EUROPIPE which is a joint venture of the two steel manufacturers Salzgitter and Dillinger Hütte. MGB developed and improves ultra high strength heavy plates mainly for this customer. All important steps of this development take place within companies integrated or at least partly integrated in the Salzgitter group: the steel is produced at Hüttenwerke Krupp Mannesmann (HKM), the plates are rolled at MGB, the pipes are manufactured at EUROPIPE, bends can be manufactured at the MGB pipe bending plant. Last but not least the whole research and development for this production chain is performed in cooperation with Salzgitter Mannesmann Forschung (SZMF) which is the development centre of the Salzgitter group.

Since the beginning of the eighties of the 20th century, plates, pipes and pipe bends of steel grade API X80 were developed and produced at Mannesmann and EUROPIPE. Since these days, the

X80 products were steadily improved for example in terms of toughness and weldability. Today they are daily business for both companies. Figure 1 gives an impression of the produced tonnage of heavy plates to date as well as the number of produced pipe bends in API Grade X80.

In the beginning of the nineties, both partners started to work on the development of API X100 plates and pipes. While X80 today is daily business for both companies, X100 plates and pipes were produced on trial scale up to now. Some 1000 tons of X100 plates and pipes of different dimensions were manufactured so far.

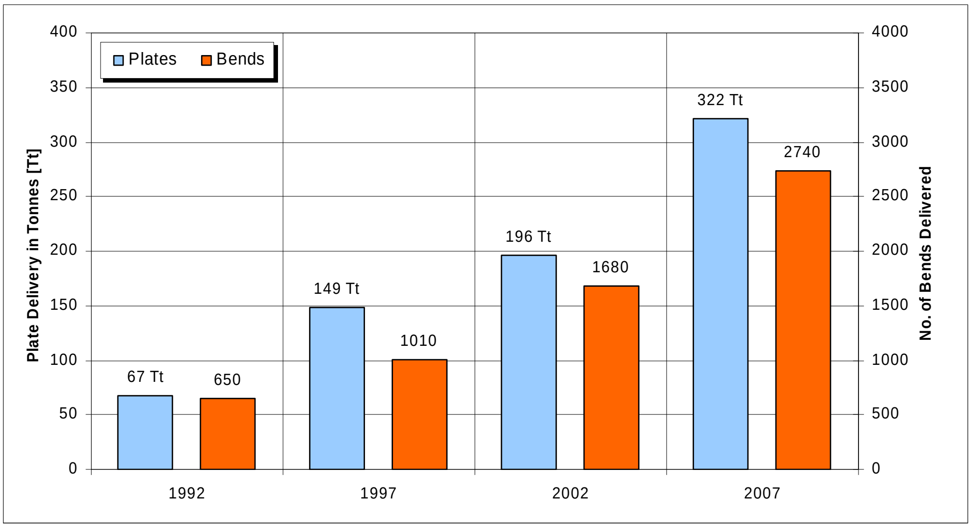

Figure1: Development of X80 production at MRM GmbH

X80 is produced by thermomechanical rolling with final rolling temperatures above the A_{r3}-temperature followed by accelerated cooling (TMCP). The microstructure that is typically obtained using this procedure consists predominantly of bainite with minor volume fractions of ferrite and martensite, as shown exemplarily in the light-optical micrograph in Figure 2. Light-optical microscopy offers only limited possibilities for quantitative characterisation because of the low size of the features.

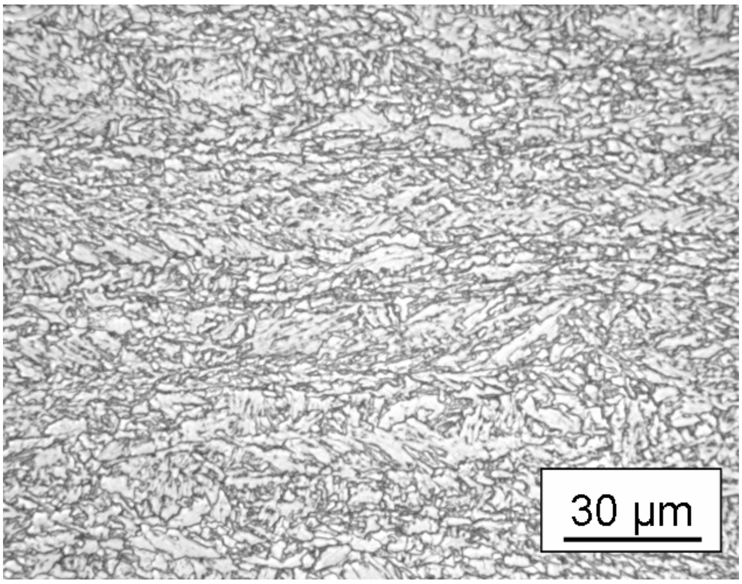

Figure 2: Typical microstructure of a sample with an intermediate cooling stop temperature at a magnification of 500:1

Electron backscatter diffraction (EBSD) is a method that has recently gained significance for the characterisation of the microstructure of bainitic steels, e.g. [1]. In the following sections, the parameters used at the SZMF and results of the ongoing activities are illustrated and discussed in light of the mechanical properties.

2. Output of EBSD measurements used for characterisation

The EBSD method provides information of the local crystal orientation and is by now a well established tool for the crystallographic analysis of material [2-5]. By generating a map out of EBSD spot measurements the orientation differences or misorientations can be calculated. Applying this calculation local misorientations as well as grain boundaries can be accurately distinguished.

The first characteristic value is the cell size of the microstructure. It is defined as a grain size which is effective for certain mechanical properties like yield strength, fracture strain or fracture toughness. In difference to the grain size measured by light optical microscopy on an etched sample, the cell size is based on crystallographic principles. The values differ significantly from grain size values especially for bainitic microstructures because light optical microscopy provides the former austenite grain size while EBSD the size of every area surrounded by a significant misorientation.

The misorientation distribution consist of the number fraction of misorientation of all misorientations between 5° and 65°. The misorientation distribution is characteristic for the transformation mode generating the steel microstructure. While a ferritic microstructure generated by diffusion controlled transformation reveals a random distribution of misorientations with a maximum at 45°, a massive transformation like the bainitic generates characteristic peaks in the misorientation distribution (Figure 3). The characteristic misorientation angles find the origin in the nucleation of the massive transformation. The transformed variants of bainite roughly follow the crystallographic relationships postulated by Kurdjumov and Sachs [6]. This results in discrete angles of misorientation possibly created between two bainite packages [1, 7]. The 60° peak is more pronounced in a microstructure which is closer to a martensitic transformation (low transformation temperatues) than in a granular or upper bainite. As a qualitative measure, the shape of the misorientation curve is applicable. A quantification on the basis of this distribution is of high interest but also of a high uncertainty.

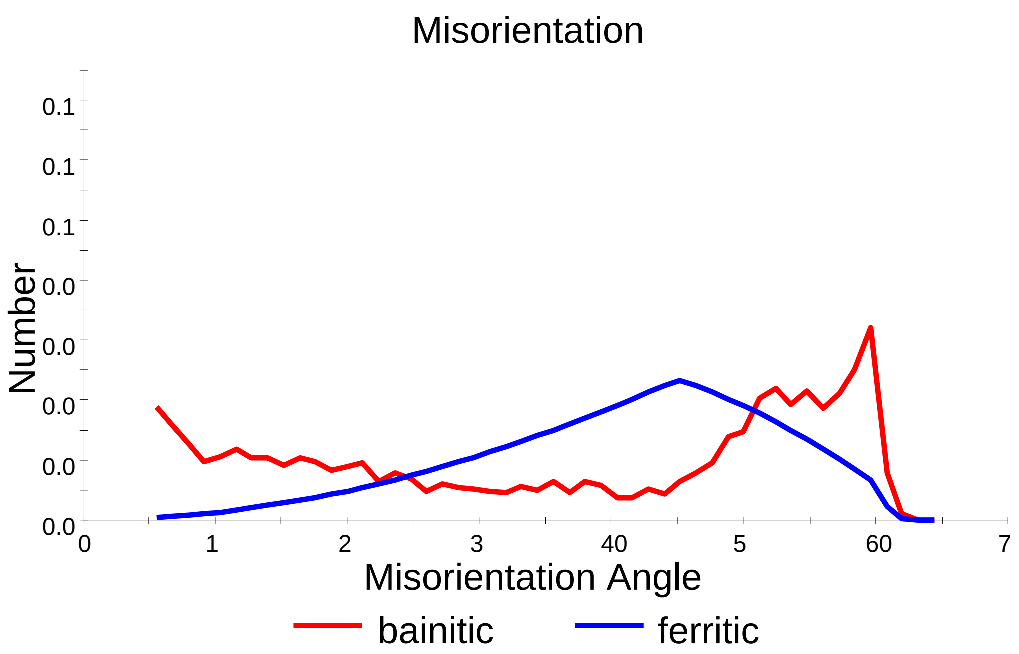

Figure 3: Comparison of the schematic representation of the misorientation distribution measured by EBSD for microstructures generated by bainitic (red) or diffusion controlled transformation (blue).

The third characteristic value extracted from the EBSD data is the kernel average misorientation. The calculation of the average misorientation angle of each measurement point to its neighbours provides information on the local plastic crystal deformation. Assuming no significant influence by elastic residual stresses, this deformation can only be generated by arrays of dislocations. This allows us to use the measure of kernel average misorientation as a qualitative value to characterize the distribution of the dislocation density. The bainitic microstructure of continuously cooled steels shows a dependency of the dislocation density on the generation temperature of the bainite. Following this, the cool stop temperature of the TM rolling as well as the position in the sheet thickness are expected to influence this value. A higher cooling stop temperature and a position closer to the sheet centre will promote a microstructure with lower dislocation densities due to changed nucleation and growth of the bainite [8] as well as recovery effects.

3. Experimental Investigations

Two alloying strategies that were investigated at the SZMF are shown in Table 1. These differ in the use of the elements vanadium and chromium, on the one hand, or copper and nickel, on the other hand. Laboratory rolling trials were carried out with accelerated cooling, in which the wall thickness was 20 mm, the final rolling temperature and the intermediate thickness were held constant, while the cooling stop temperature was varied between 400°C and 650°C with cooling rates above 10 K/s.

	C	Si	Mn	Nb	Ti	others	CE(IIW)
Type 1	0.06	0.30	1.90	0.06	<0.025	V, Cr	0.43
Type 2	0.06	0.30	1.90	0.06	<0.025	Cu, Ni	0.44

Table 1: Possible alloying concepts for the grade X80 and the resulting carbon equivalents

According to the ISO specification 3183 [9], a minimum yield strength of 555 MPa and an ultimate tensile strength of 625 MPa are required for this grade in transverse direction. Representative results of transverse round bar tensile tests are presented in Figure 4. The results show that the requirements can be fulfilled with both alloying concepts and that in both cases a decrease of the cooling stop temperature leads to an increase in the yield strength but also to an increase in the Y/T ratio. Transverse Charpy impact tests were carried out between -20°C and -80°C on V-notch specimens and showed that the mean impact energy remained above 200 J in all cases down to a temperature of -60°C, i.e. sufficient toughness was achieved with both alloying concepts.

EBSD measurements were carried out 5 mm below the surface (S=0.5) on samples of type 1 and type 2 in order to gain a better understanding of the effect of cooling parameters on the resulting microstructure and mechanical properties.

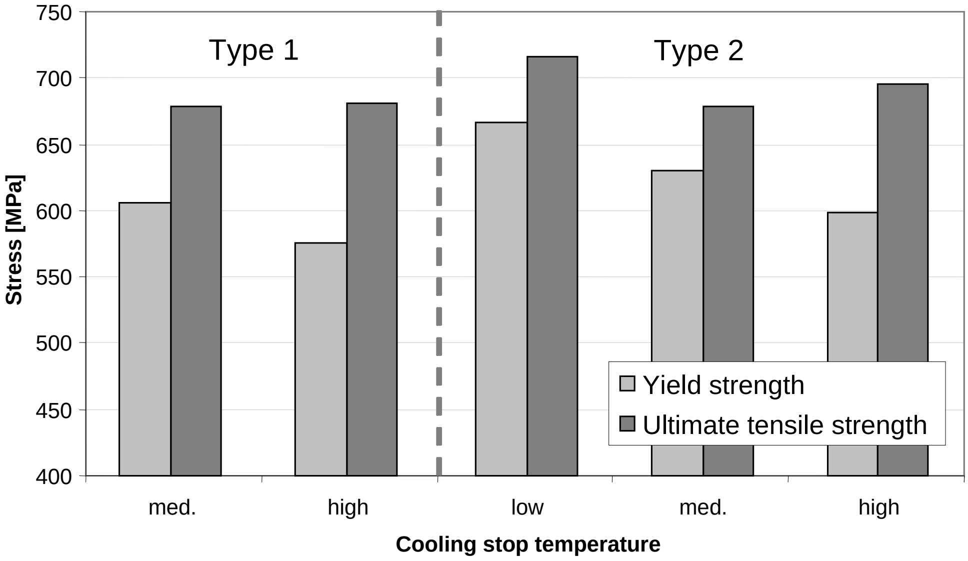

Figure 4: Results of round bar tensile tests for the plates produced in the laboratory rolling trials

The average cell size was found to increase from 2.8 µm to 4.2 µm with increasing cooling stop temperature while the kernel average misorientation decreased with increasing cooling stop temperature, as shown in Figure 5. If accelerated cooling is interrupted at the cooling stop temperature, the metastable austenite that has not yet transformed to bainite will transform during air-cooling. For a fixed composition, the nucleation rate increases with decreasing cooling stop temperature, while the growth rate decreases [10-11]. The lower cell size observed at lower cooling stop temperatures is therefore plausible. The increase of the kernel average misorientation with decreasing cooling stop temperature is connected with the increase in dislocation density with decreasing temperature of transformation [8].

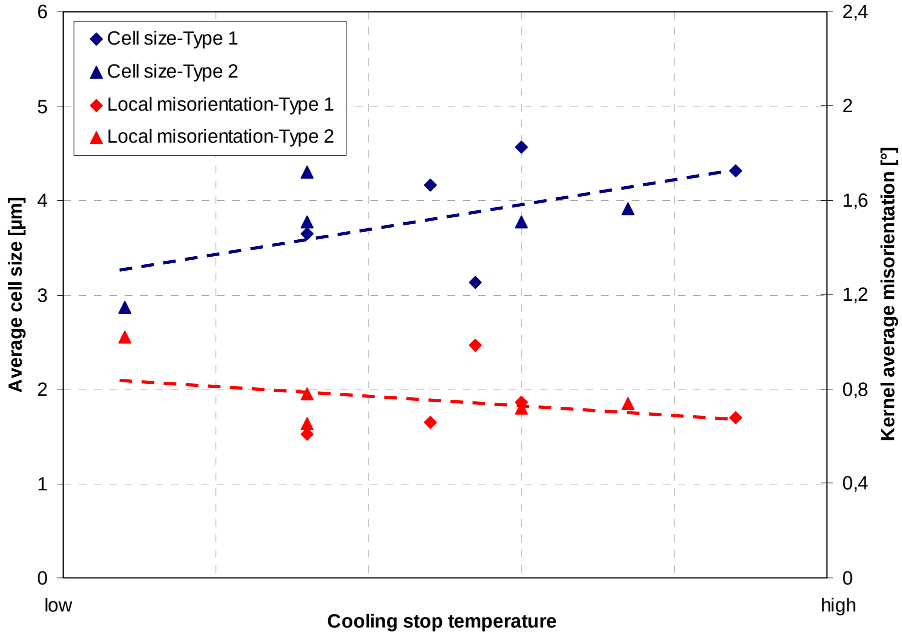

Figure 5: Influence of the cooling stop temperature on the average cell size and kernel average misorientation

The influence of the cell size on the yield and tensile strength is illustrated in Figure 6. Both were found to increase with decreasing cell size. A linear relationship is found if the square root of the inverse average cell size is plotted versus the yield strength, i.e. the Hall-Petch relationship appears to hold in the present case. The yield and tensile strength was found to increase with increasing kernel average misorientation, as shown in Figure 7. The correlation between the uniform elongation and the cell size was not as clear, as illustrated in Figure 8. It appears that the uniform elongation increases with the cell size, but the considerable scatter of values does not fully justify this conclusion.

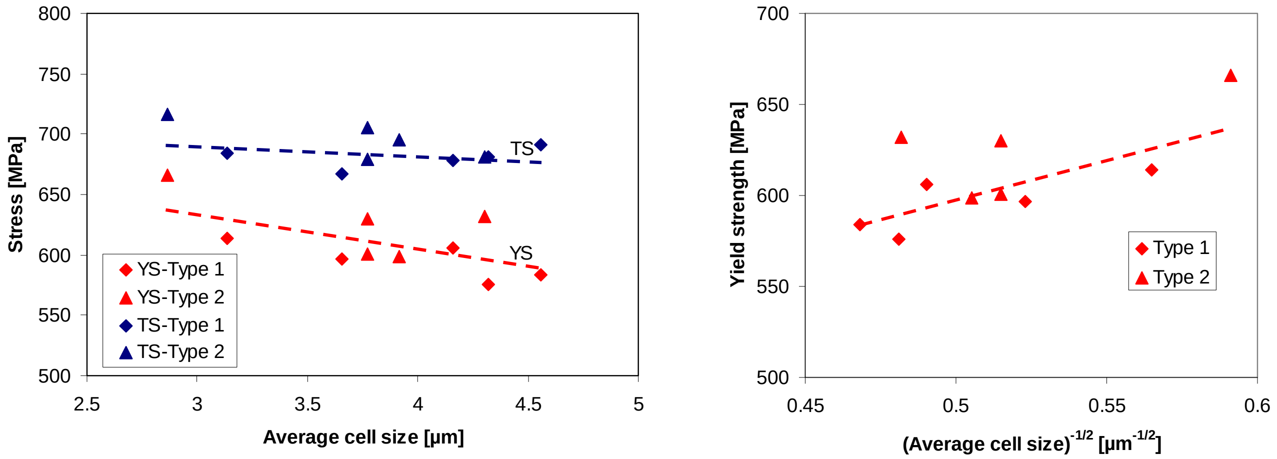

Figure 6: Influence of the average cell size on yield and tensile strength (left) and analysis of yield strength according to the Hall-Petch relationship (right)

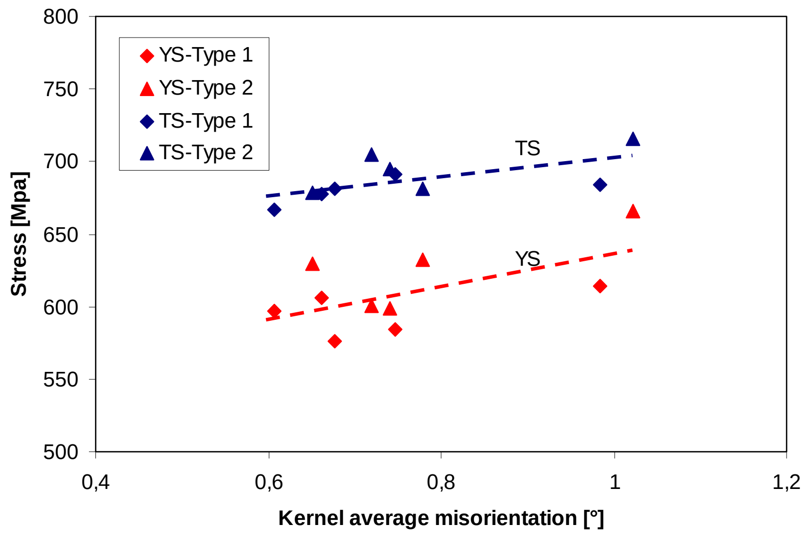

Figure 7: Correlation between Kernel average misorientation and yield and tensile strength

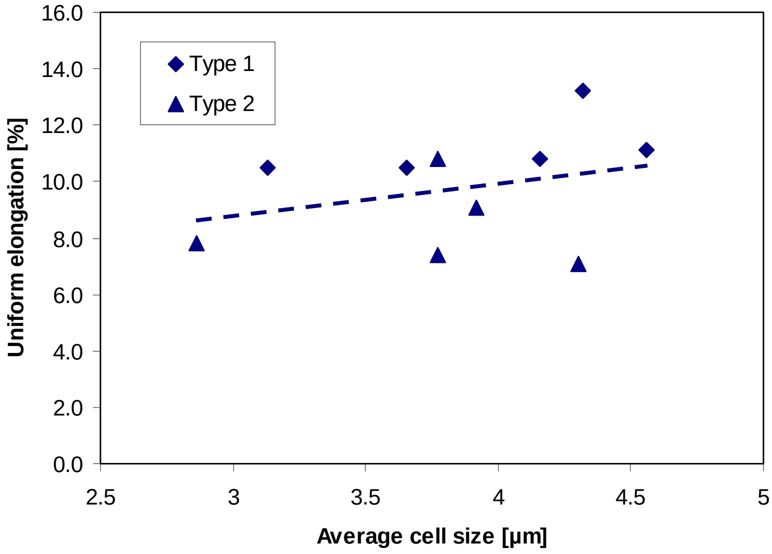

Figure 8: Correlation between average cell size and uniform elongation

The intention of the experiments was to bridge the gap in the understanding of the effect of cooling parameters on mechanical properties and microstructure evolution. As shown above, it was possible to correlate mechanical properties with the selected output parameters of EBSD measurements, which can aid in the selection of cooling parameters during production. In the following section, the results of EBSD measurements of samples from production are discussed and the results are compared with those obtained using material rolled on the laboratory scale.

4. EBSD measurements of samples from heavy plate production

17 T tons of X80 heavy plate (20.5 mm wall thickness and 3730 mm width) were produced with both alloying concepts at MGB. An overview of the required mechanical properties for this order is shown in Table 2. The distribution of the yield and tensile strength that was achieved in large scale production is shown in Figure 9. Similar strength levels were obtained with both alloying concepts which were well above the minimum requirements. The toughness was also well above the required level.

	SMYS MPa	**UMTS MPa**	**CVN* J**	**BDWT* %**	**Elong. %**	**Y/T**
transverse	555	625	115/150	75/85	18	0,90

Table 2: Required mechanical properties of X80, *****design temperature for CVN and BDWT 0°C

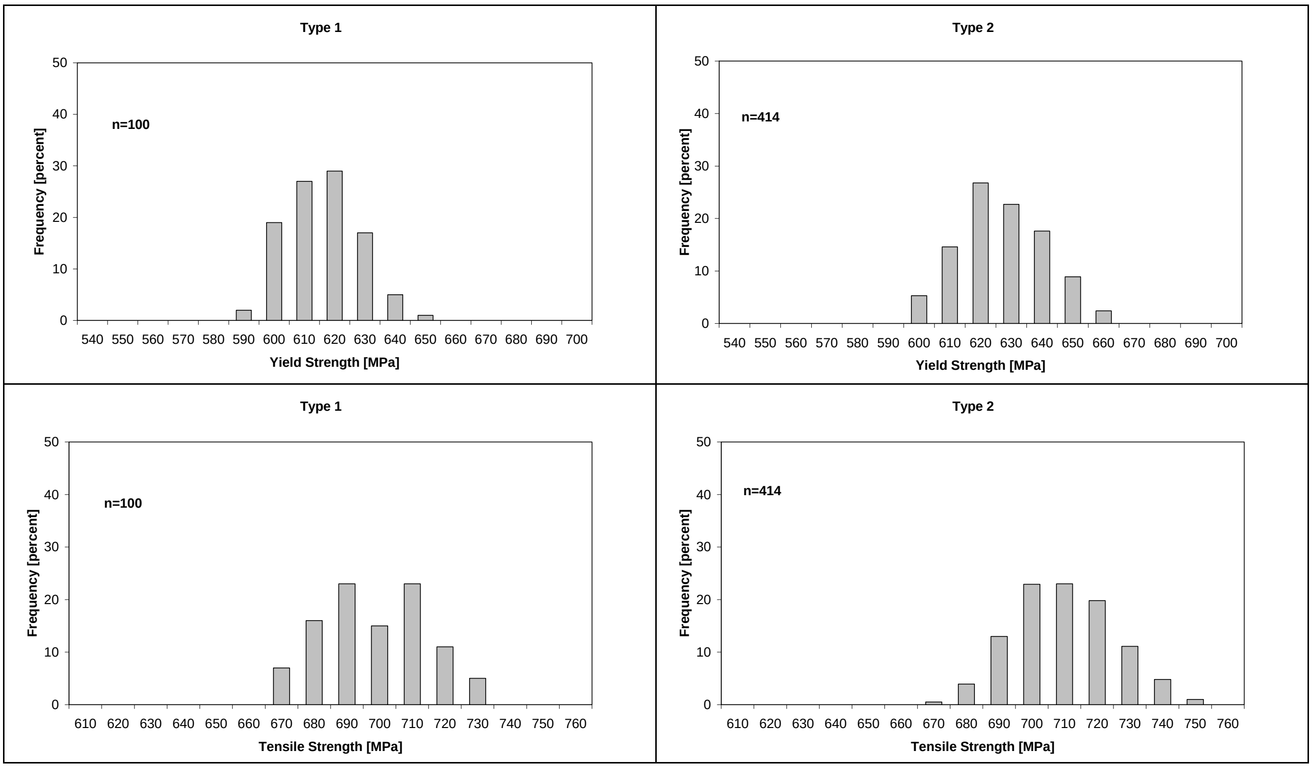

Figure 9: Strength properties for alloying concept type I and type II

In order to compare the results of the laboratory trials with samples taken from full-scale production, EBSD measurements were carried out on samples taken from heavy plate material. These were performed slightly below the surface (S=0.98), 5 mm below the surface (S=0.5) and at the midwall position (S=0). The cell size and the kernel average misorientation at these positions is illustrated in Figure 10. While the cell size increased from 2.4 µm to 3.4 µm with increasing distance from the plate surface, the kernel average misorientation decreased from about 0.90° to 0.65°.

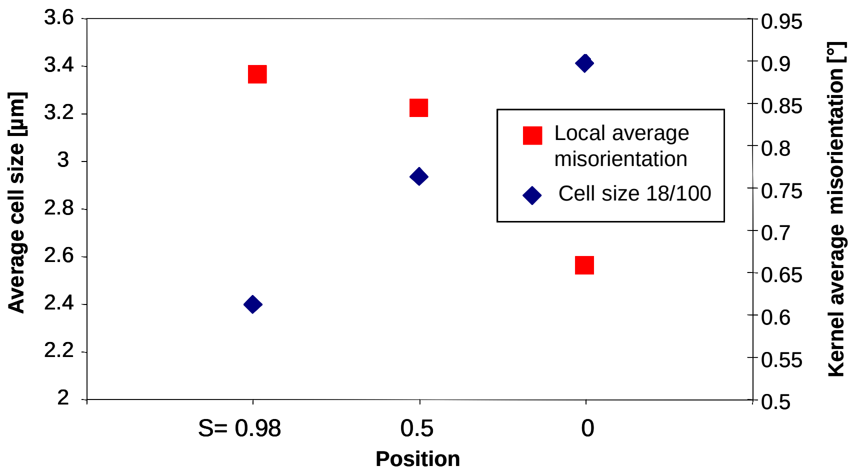

Figure 10: Average cell size and kernel average misorientation of heavy plate material at different distances below the surface

A comparison of the kernel average misorientation and grain boundary misorientation distributions is shown in the map in Figure 11. Blue areas in the kernel average misorientation charts correspond to regions with a lower and yellow and red regions have a higher kernel average misorientation. The fraction of areas with a higher kernel average misorientation decreases clearly with increasing distance from the surface of the plate. This is related to the fact the cooling rate during accelerated cooling is highest and the cooling stop temperature is lowest close to the surface compared to positions further below the surface.

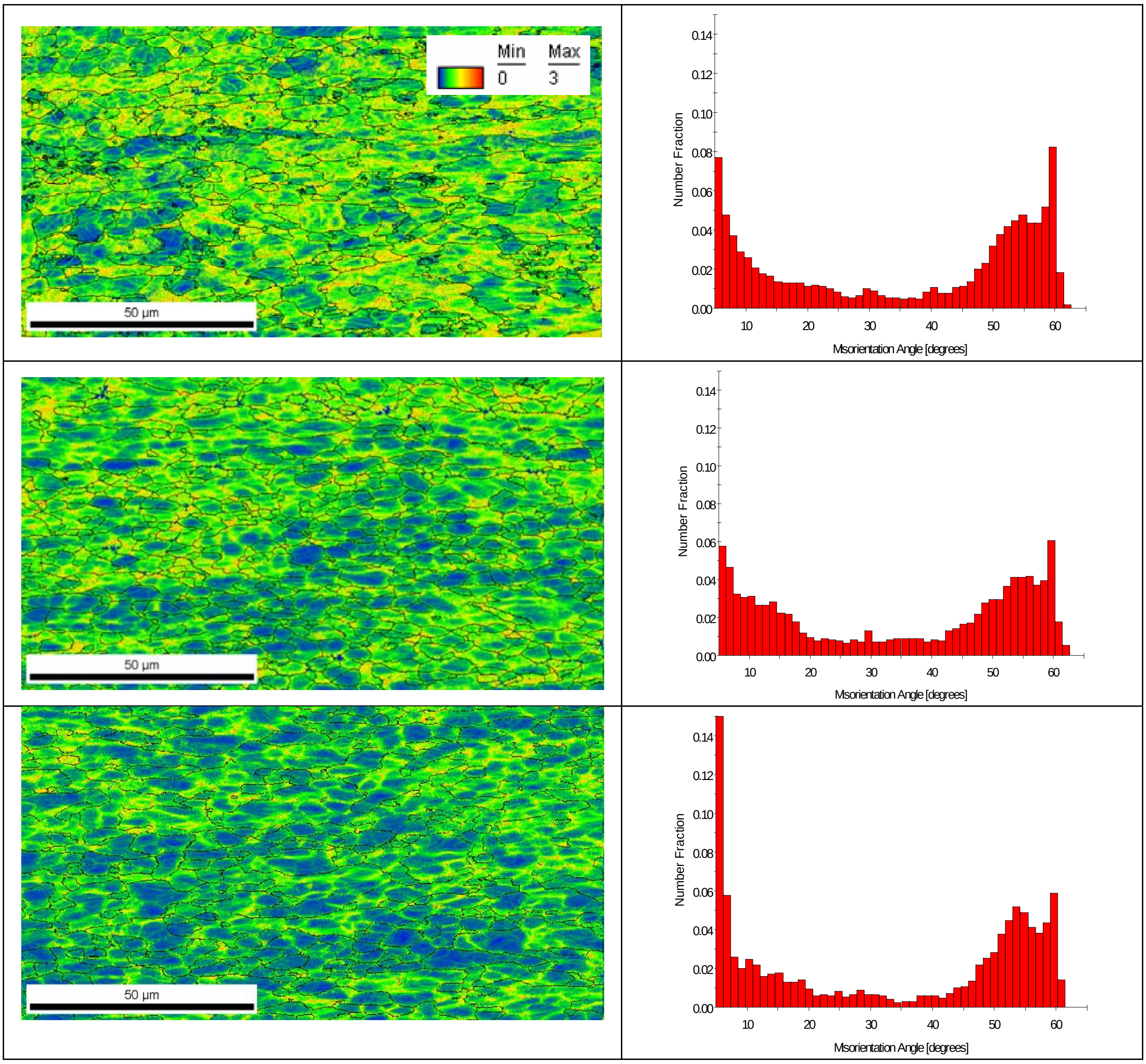

Figure 11: Kernel average misorientation between 0° and 3° (left) and grain boundary misorientation distribution (right) of heavy plate material at different distances below the surface: S=0.98 (top), S=0.5 (centre) and S=0 (bottom)

Grain boundary misorientation distributions are shown in Figure 11 for the same positions. A pronounced peak at a misorientation of 60° is observed for S=0.98, while the peak height was lower in the case of the other two positions. This indicates a fraction of transformation products with a Kurdjumow-Sachs relationship with respect to the austenite, i.e. lower bainite or martensite. This is consistent with the higher kernel average misorientation close to the surface of the plate.

5. Comparison of the microstructure and mechanical properties obtained after laboratory rolling and in the production

The misorientation distributions of material rolled at the SZMF and at the heavy plate mill measured at S=0.5 are compared in Figure 12. The peak height at 60° is slightly higher in the case of the production sample. Nevertheless, the agreement between both distributions is considered adequate, i.e. the accelerated cooling conditions at the laboratory are comparable to those of the heavy plate mill.

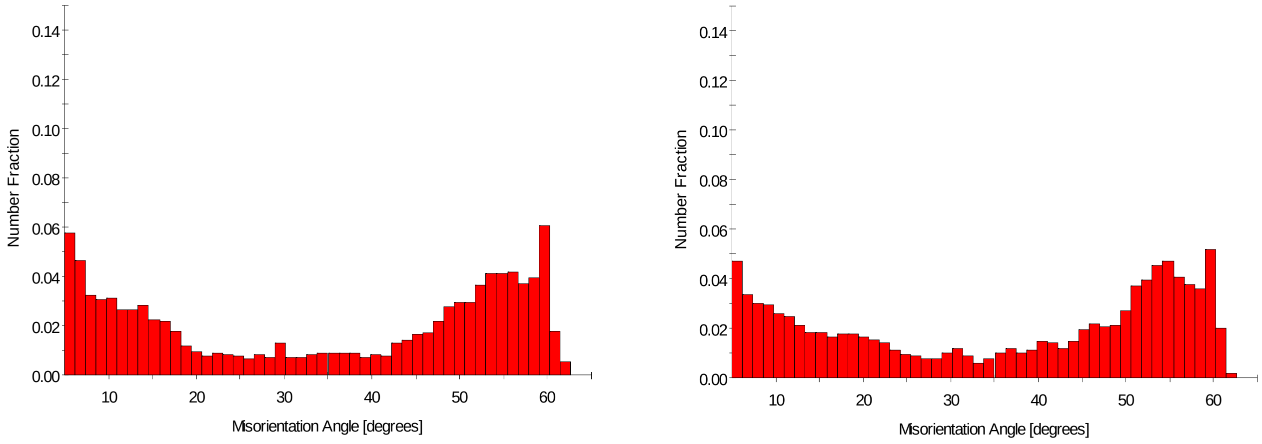

Figure 12: Grain boundary misorientation distribution of heavy plate material (left) and of plate material rolled at the SZMF (right) measured at S=0.5

6. Conclusions

The microstructure of high strength heavy plates for large diameter pipe line application has been described using electron diffraction methods exceeding the possibilities of light optical microscopy. It was shown that the microstructural consequences of different processing parameters on the one hand and their influence on the mechanical properties on the other hand could be described in detail applying mathematical analyses on EBSD datasets.
Furthermore it was shown that the accelerated cooling parameters influence the mechanical properties and at the same time the microstructure. A correlation was identified between cooling stop temperature, yield strength, tensile strength and average cell size. The kernel average misorientation was found to increase with decreasing cooling stop temperature and increasing strength. Due to the high sensitivity of the EBSD method, even the variation of cooling rates through the wall thickness of industrial material was detected.
Comparison of laboratory rolled material with industrially produced material showed that similar misorientation distributions were obtained, i.e. the microstructure of material rolled in the laboratory is similar to that obtained on the production scale.

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