Review Article

Complex phase quantification methodology using electron manganese backscatter diffraction (EBSD) on low manganese high temperature processed steel (HTP) microalloyed steel

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Microalloyed steels are required to have good strength and toughness, properties that are mostly influenced by microstructure and phase distribution in the steel, obtained by controlling the thermomechanical processing. High temperature processing (HTP) Steels, with lower manganese content, has been recently developed in order to reduce segregation, but reduction of Mn decreases the yield strength. In order to understand the relationship between chemical composition, microstructure, mechanical properties and processing parameters, quantitative analysis of the final microstructure is required but identification and quantification of the ferrite microconstituents under optical microscopy or scanning electron microscopy is difficult due to their similarities. The present work presents a methodology for identification and quantification of complex microconstituents on a low manganese HTP steel, subjected to different hot deformation and cooling cycles, by means of electron backscatter diffraction (EBSD). The results showed that the EBSD methodology allowed to identify and quantify different microconstituents in a low manganese HTP steel with similar results as those obtained by point count methodology.

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1. Introduction

Microalloyed steels are required to have good strength and toughness [1,2]. These properties are mostly influenced by microstructure and their distribution in the steel, which are mainly obtained by controlling the thermomechanical processing [3,4]. For the Oil and Gas industries, some characteristics are necessary to enhance the performance of steel plate. In these steels, segregation control is required to avoid hydrogen embrittlement due to elongated manganese sulfide. To reduce banding susceptibility and central line segregation carbon and phosphor contents need to be lower than 0.06% and 0.015%, respectively. Lowering manganese content in steels also increases performance in hydrogen sulfide environment [5]. However, with lower manganese content, the yield strength is reduced due to the decrease in solid solution strengthen and increase in transformation temperature. Additions of chromium (0.20–0.65%) and niobium (0.065–0.095%) are usually done to reduce transformation temperature. Niobium also contributes to High Temperature Processing of austenite because niobium carbide solubility is reduced due to the low manganese content, favoring elevation of non-recovery temperature [6].

To understand the relationship between microstructure, mechanical properties and processing parameters, a
quantitative analysis of the final microstructure is required [7,8]. Improvements in fabrication routes and alloy design of steels have been important for the development of microalloyed steels, but also have become a problem for microstructure analysis because they have complex microconstituents, with the same crystallographic arrangement, but with different degrees of defects in their cells [9,10]. It is possible to obtain microstructures with different amounts of martensite/austenite (MA), polygonal ferrite (PF), quasi-polygonal ferrite (QF), acicular ferrite (AF) and bainite (B), for the same steel, when submitted to different thermomechanical processing [10,11].

Polygonal ferrite (PF) forms at high temperatures, low cooling rates, has equiaxial morphology and nucleates at austenite grain boundaries. The mechanism of transformation of PF is reconstructive, presents low dislocation density and does not presents substructures [12,13]. Quasi-polygonal ferrite (QF) forms during higher cooling rates than PF, which prevents carbon partitioning in austenite to ferrite during cooling, when the steel crosses the two-phase field, in high temperature without change in chemical composition. Quasi-polygonal grains are almost similar to PF, but QF grains are irregular and reveal substructures marks after chemical etching, contrary to PF grains [12].

According to Bhadreshiya et al. [14,15], acicular ferrite (AF) has intragranular nucleation, chaotic and irregular arrangement of their plates and a microstructure with high fraction of high angle grain boundaries (HAGBs) [14] associated with laths that grow in random directions, acting as a barrier for crack propagation [16,17]. It forms at intermediates temperatures under fast cooling rates, between polygonal ferrite and bainite formation, and has high dislocation density when compared with PF and QF. Acicular ferrite has become an interesting microconstituent for the microalloyed steels used in the Oil and Gas industry [18] because it increases mechanical strength, toughness [19,20] and hydrogen sulfide (H₂S) resistance [19,21] when compared to traditional ferrite-pearlite microstructure [21].

Bainite forms at fast cooling rates, preventing pearlite formation, but not so fast to form martensite. Its morphology is lenticular or lath like in shaves, connected by low angle grain boundaries. The ferritic laths usually have cementite particles between or inside them [15]. Bainite has high defects densities, increasing the strength but decreasing ductility due to the presence of low angles grain boundaries (LAGBs) [11].

As stated above, the mechanical properties of steels, depend of the amount and distribution of the microconstituents obtained after thermomechanical processing. In order to identify and quantify theses microconstituents, the point counting methodology has been used, based on microconstituents appearance under optical microscopy (OM) or scanning electron microscopy (SEM) [17,22]. Identification and quantification of these microconstituents by MO or SEM may be a problem due to the similarities between them [7,9]. Color etching as Le Pera or Klemm’s, together with OM, has been used to identify different microconstituents in transformation induced plasticity (TRIP) steels, but has not been able to identify all the microconstituents because some of them have thin plates which cannot be resolved by OM [23]. Also, most of these etches depend on the carbon content and, in some cases, bainite may appear white or brown depending of the carbon content and/or densities of defects [7,22]. Furthermore, point counting methodology requires analysis of a large number of images in order to obtain a 95% of reliability [24], which usually demands a lot of work and time [3].

Electron backscatter diffraction (EBSD) has recently been used to identify and quantify microstructures in steels [1,3,7,9–11,25]. This technique allows to calculate grain/sub grains sizes, texture analysis [10], misorientation of grain boundary [26,27] and phase identification based on maps of crystallographic orientation (differences at the structure or defects of cells) [10,28]. Ryde [25] used band slope (BS) to separate polygonal ferrite, bainite in ferrite and martensite and quantify then in low carbon bainitic steel. Band slope is an EBSD technique based in Kikuchi pattern, where the slope of intensity changes between background of pattern and band. BS is described as the sharpness of the band edges and allows to separate peaks of different phases. It is very used to separate martensite from ferrite due color contrast because the martensite appears darker when compared with ferrite. This approach may be used together with other techniques as grain size to refine results. Feng et al. [29] used image quality to separate and quantify polygonal ferrite from martensite on a TRIP steel, obtaining reasonable results. This technique is based in pattern quality and can be used because phases with high dislocation densities degrade the diffraction pattern, generating images with poor quality. Zaefferer et al. [7] identified and quantified bainite from ferrite by using the confidence index, which is based in the same principle of image quality. All parameters cited allowed to identify and quantify steels with simple microstructure, but these techniques are sensitive to sample preparation and contamination. To minimize this problem, new methodologies based in grains characteristics like aspect ratio, grain internal misorientation and grain area has been developed, minimizing dependence of sample preparation.

A recent method proposed by Zhao et al. [3] allowed to identify and quantify PF, AF and bainitic ferrite (BF) on a deformed microalloyed steel using parameters based on the grain characteristics, internal misorientation, aspect ratio and fraction of HAGBs for each microconstituent. Sherestha et al. [11] also created a methodology to quantify complex ferritic microconstituents (PF, AF and B) in three deformed steels (Nb free, 0.4% Nb, and 0.8% Nb, in wt%) by using EBSD. Their methodology was based in grain units adopting criterions as aspect ratio, grain internal misorientation, grain boundary characteristics and grain area were used to quantify the microconstituents. The authors compared their results using EBSD with point counting and found a maximum difference in phase quantification of 6%.

In the present work, a methodology for phase quantification using EBSD on non-deformed and deformed samples was developed and applied on an HTP microalloyed steel, with low manganese content, subjected to different deformation and cooling cycles. The identification and quantification of microconstituents was performed based on the grains characteristics and, the results were compared with those obtained by point counting. To understand the relationship of low manganese in HTP steel, a phase quantification methodology was
developed to evaluate microconstituents formed and their volume fraction.

2. Material and experimental procedure

2.1. Material

The material studied in this work was a microalloyed steel with high niobium and low manganese content, proposed by Gray et al. [5,30]. The chemical composition of the steel is presented in Table 1.

2.2. Deformation and cooling cycles of the samples

Dilatometry tests without deformation were performed in order to simulate different continuously cooling of the HTP microalloyed steel and to generate microstructures with different volume fraction of microconstituents. Samples (4 mm of diameter and 10 mm of length) were austenitized at 950 °C for 2 min and continuously cooled to room temperature at 10, 20 and 50 °C/s, in a dilatometer (Bähr type 805 A/D) under helium atmosphere.

Torsion test was also performed in order to generate microstructures with different volume fraction of microconstituents, due to different deformation during processing, and to compare these microstructures to those obtained only by continuous cooling. Two schedules of torsion tests were performed in a GLEEBLE 3800. These schedules represent rolling of heavy plates with 25.6 mm, starting from sketches with 50 and 100 mm. The schedule of deformation from 50 to 25.6 mm was called 50% of deformation below \( T_{nr} \) and the schedule of deformation from 100 to 25.6 mm was called 75% of deformation below \( T_{nr} \). Deformation was performed below non-recrystallization temperature (\( T_{nr} \)) (samples were not deformed after austenite-ferrite transformation) and after the last pass, the samples were continuous cooled at 20 °C/s to 560 °C and immediately cooled at 0.3 °C/s to room temperature with air. Deformation of the 50% below \( T_{nr} \) sample started at 926 °C and finished at 894 °C, while for the 75% below \( T_{nr} \) sample, deformation started at 913 °C and finished at 896 °C. Samples of 10 mm diameter and 20 mm length were cut off at the center of the torsion tests samples for microstructure analysis at the surface.

2.3. Scanning electron microscopy (SEM)

For SEM analysis the samples were cut, mounted, mechanically ground by using sandpapers from 220 to 1500 grade, polished with a 3 and 1 μm diamond suspension and chemically etched with Nital 5%. The analyses were performed using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS)/EBSD-field emission gun (FEG) model JEM 7100 FLV – JEOL.

2.4. Electron backscatter diffraction (EBSD) measurements

For EBSD analysis, samples were prepared similarly than those for SEM analysis with additional polishing in colloidal silica for 30 min. The EBSD analysis were performed in the same SEM/FESEM microscopy mentioned above, equipped with an EBSD detector, model Nordlys-Max, from Oxford Instruments. The SEM was operating at 20 kV, the samples were tilted 70° in relation of electron beam and the step size used was 0.3 μm. After EBSD scanning, the results were analyzed using the Tango application of Oxford Instruments Channel 5 software. Criterions of internal misorientation of grains, aspect ratio and grain area were used to create parameters to identify and quantify the different ferrite microconstituents.

2.5. Phase quantification using EBSD

2.5.1. Grain detection and noise reduction

Noise reduction was used to remove and correct points that could not be indexed [31].

Grain detection is defined as misorientation between pixels. If this misorientation is higher than certain value, these pixels are considered as grain boundary.

In order to decide which misorientation to use in the present work, both misorientations of 5° and 15° were studied in order to select the one that could separate correctly different microconstituents that otherwise may be considered as one [3].

Initially MA and/or P was identified, followed by PF/QF, AF and B. The criterion of internal misorientation of grains and aspect ratio were used to identify and quantify PF/QF in all conditions. The same procedure was performed for deformed samples, with the difference that the grain area criterion was used to separate and quantify AF from B. The identification and quantification were performed as follows.

2.5.2. Identification and quantification of MA and/or P-EBSD

Pearlite and MA was classified by using the Band Slope (BS) criterion, which is related to diffraction pattern of samples and has been used to identify martensite, bainite and ferrite in some steels [9,22,25]. BS allows to separate phases according to color contrast, dark (MA and/or P) and bright (ferrite).

2.5.3. Identification and quantification of PF/QF

Polygonal and quasi-polygonal ferrite were considered as one microconstituent due their similarities in transformation mechanisms, morphology and defect densities. The first criterion to identify and separate PF/QF from AF/B was the grain internal misorientation. By using Grain Orientation Spread (GOS), it was possible to calculate the mean deviation of misorientation between each pixel of the grain and the mean
misorientation of the grain [32], which is related to defect density inside a grain. GOS was also used by Zhu et al. [22] associated with other parameters to identify and quantify ferritic microconstituents. PF/QF present low defects when compared with acicular ferrite and bainite, reflecting on a lower GOS [3]. GOS graphics were plotted for all samples in the same condition (deformed and non-deformed) and compared. The results showed that the curves for non-deformed conditions were similar, but also differed from the curves obtained for the deformed conditions, which is why a unique value of GOS was chosen for each condition (non-deformed and deformed samples).

After using GOS, a second criterion (aspect ratio) was used to refine the results and to avoid that some grains of acicular ferrite or bainite, with GOS bigger than the determined value were wrongly classified as PF/QF due to a unique value of GOS adopted for each condition. Aspect ratio (ratio between length and width of a grain) is associated with morphology of grains and, due to differences in morphology between PF/QF and AF, these phases can be distinguished by their difference of aspect ratios. PF/QF are equiaxial [12] and have low aspect ratio [11] while AF has higher aspect ratio due to the lath morphology [14]. Wu [33] studied the aspect ratio of AF in three dimensions and his results showed that AF have aspect ratio higher than PF/QF with values near 10. Bhadeshia [12] found that AF has aspect ratio higher than 3.

2.5.4. Identification and quantification of AF and B

Finally, the remaining microconstituents in the microstructure were identified as AF in non-deformed samples, by exclusion, after quantify MA, P e PF and QF, the remain microconstituent in microstructure were AF. For deformed samples, it was necessary to use another criterion to separate AF from B.

The quantification of B was obtained by exclusion after quantify MA and/or P, PF/QF and AF for deformed samples.

2.6. Point counting methodology

Point count quantification method was used in this work to corroborate the results obtained by EBSD. It was performed according ASTM E 562-02 [24], using a grid with 100 points and a maximum with 10% were adopted to standard. Analysis was performed in forty micrographs with 960 µm² each. Point counting methodology and EBSD measurements were performed in different regions of sample.

3. Result and discussion

3.1. Scanning electron microscopy (SEM)

Fig. 1(a, b) shows the microstructure of the sample cooled at 10 °C/s. Most of the grains are PF/QF and it is possible to observe some AF grains randomly distributed in microstructure. Pearlite and MA were randomly observed in this condition. With the increase of cooling rate to 20 °C/s, Fig. 1(c, d), the predominant microstructure observed was still PF/QF but it was possible to observe more AF grains. The pearlite became finer and less frequently, while MA grains becomes smaller and appear more frequently. In these cooling rates pearlite did not develop in form of lamellar structure due to fast cooling which decreases time to form organized lamellar structure [15]. The sample cooled at 50 °C/s, Fig. 1(e, f), presented the highest volume fraction of AF from all for the non-deformed samples, but the predominant microstructure was still PF/QF. Pearlite was not observed in this condition and MA was observed more frequently and was well distributed throughout microstructure.

In 50% of deformation below Tₚ₉ sample, Fig. 1(g, h), the microstructure presented a higher volume fraction of PF/QF. This micrograph showed some acicular ferrite grains with bainite. The microstructure of the sample 75% of deformation

![Fig. 1 - SEM images of HTP steel continuous cooled at (a) 10 °C/s, (b) 20 °C/s and (c) 50 °C/s in non-deformed condition. (d) 50% of deformation below Tₚ₉ and (e) 75% of deformation below Tₚ₉. P, polygonal ferrite; AF, acicular ferrite; B, bainite.](image-url)
below \( T_{nr} \) presented higher volume fraction of acicular ferrite, Fig. 1(i, j), when compared with the 50% of deformation below \( T_{nr} \) deformed sample, as expected. This image shows some PF/QF grains surrounded by acicular ferrite grains. Bainite was observed in this sample. Also, it was observed that the increase in deformation increase slightly the bainite volume fraction. Zhao et al. [34,35] also observed that an increase in deformation, favors AF formation. Deformation acts as driving force for AF nucleation, inserting defects inside the austenite grain, which are pancaked when deformed below \( T_{nr} \).

For samples non-deformed and continuously cooled at 10 and 20 °C/s, it was not possible to separate of MA and P. Individually, P may be identified using Kernel Average Misorientation (KAM), that plot a local image showing orientation gradients between pixels [35,36], but in this work, KAM were not able to detect gradients between pixels in HTP steel because P colonies are smaller (approximately 1 µm) [36]. Takahashi et al. [36,37] studied KAM of pearlite and obtained reasonable results, in their researches, the grain size of colonies was bigger than 10 µm.

3.2. Phase quantification

It is possible to observe in Fig. 2(b) that two grains are considered as one grain when the misorientation of 15° is used, while in Fig. 2(a) when a misorientation of 5° is considered, 2 grains are defined as different grains.

Grain detection is still controversial, with some authors using low misorientation angles (between 2° and 5°) for grain definition, while others use 15°. Isasti et al. [38] used 4° or 15° based in steel chemical composition, cooling rate, austenite grain size and retained strain to predict microstructural refinement in different steels. Olsolo et al. [39], affirmed that low angle boundaries, 4°, is a factor that control yield strength and tensile strength, due to low angle boundaries oppose dislocation movements while, high angle boundaries, 15°, act as a barrier for cleavage fracture. In their study, the authors analyzed the influence of cooling rate and chemical composition in microstructural evolution by means grain size 4° and 15° grain boundaries definitions. They showed that microstructural refining is more efficient in low cooling rates and in recrystallized austenite microstructure. Isasti et al. [40] adopted grain angle definition of 4° and 15° to evaluate microstructural evolution and homogeneity in continuously cooled steels and showed that this is an effective tool to quantify microstructural effect in strength and toughness.

By using high angle values to define the grain, it may occur that two grains with different microconstituents are considered as one grain. Zaefferer et al. [7] used a misorientation of 5° in their work to define a grain in order to separate ferritic and bainitic phases on an annealed TRIP steel. Kang et al. [9] also used 5° to separate ferrite from martensite and the authors reported reasonable results, concluding misorientation between 5° and 10° is acceptable. Furthermore, Zhao et al. [3,11] used a misorientation of 5° because it allowed them to separate PF/QF from AF and BF.

For non-deformed samples the difference in phase quantification between misorientation of 5° and 15° was 9%, for sample continuously cooled at 10 °C/s, followed by samples continuously cooled at 20 and 50 °C/s with a difference of 4%. For deformed samples, the maximum difference between phase quantification using misorientation of 5° and 15° was 4% for all conditions. It was then decided to use a misorientation of 5° for grain detection for the present work.

The distribution curve of GOS, shown in Fig. 3, has an asymmetric shape which means than more than one microconstituent are influencing the curve shape. A Gaussian deconvolution of peaks was then performed by using the analysis software Origin. The GOS values for PF/QF separation found were 0.4 for non-deformed samples (Fig. 3a) and 1.25 for deformed samples (Fig. 3b).

The aspect ratio was obtained by deconvolution of the peaks in the distribution curve of aspect ratio, as shown in Fig. 4. By plotting the distribution curve of aspect ratio, it was possible to determine aspect ratio value of PF/QF. The distribution curves of aspect ratio, Fig. 4, have an asymmetric curve, so, more than one microconstituent is influencing the curve shape. The values found for non-deformed and deformed samples were 2, according Fig. 4(a, b). Aspect ratio may not be used to separate AF and B from PF/QF, due B presents scattered aspect ratio depending of arrangement.
Fig. 3 – Distribution curve of grain orientation spread (GOS) for HTP steel (a) continuously cooled at 10 °C/s and (b) 75% of deformation below Tnr.

Fig. 4 – Distribution curve of aspect ratio for steel HTP (a) continuously cooled at 10 °C/s in non-deformed condition and (b) 75% of deformation below Tnr.

Fig. 5 – Grain area graphic for steel HTP 50% of deformation below Tnr, to separate phases.

and depend of quantity and arrangement of laths inside B grains [3].

Fig. 5 shows a curve of grain area as function of the area fraction were two different regions are observed. These regions are related to transformation mechanisms of AF and B. AF laths development in packets that grow in randomly directions in short range and form small packs of high-density laths [14,35]. Bainite forms laths that nucleate in grain boundaries and may cross entire grain (long directions of growing), when compared with AF that nucleate inside a grain [14], forming grains with long laths and low density of laths per area. This characteristic generates distinct profile when the grain area graphic is plotted (bimodal distribution), allowing to identify and quantify AF and B. Transition between each condition, downward to upward changing in overall trend, show a valley point (the smaller point between transition of high area fraction with low grain area to a region that area fraction grow back with high areas), which allows to separate AF from B. Below this point only AF grains are observed. The valley point was observed at 75 μm², which means that for smaller areas, the microconstituents were identified as AF.

Tables 2 and 3 show aspect ratio, grain internal misorientation (GOS) and grain size values used to identify and

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<th>Table 2 – Criteria for identification and quantification of PF/QF, AF and B in non-deformed condition.</th>
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<th>Table 3 – Criteria for identification and quantification of PF/QF, AF and B in deformed condition.</th>
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quantify microconstituents in non-deformed and deformed condition.

Table 4 shows constituent volume fraction difference of HTP steel using EBSD and point count methodology. The results obtained by point counting showed that the maximum difference between both methods was 5%, according Table 5, phase quantification by EBSD allowed to identify and quantify successfully different constituents. Table 5 shows the results obtained for phase quantification by using the point counting methodology for the samples continuously cooled at 10 and 20 °C/s in non-deformed condition and for the sample 50% of deformation below Tnr.

The criteria used in this work were selected with care to avoid errors, observing the quality of data in EBSD scan to obtain reasonable results, that allowed identify and quantify complex phase in HTP steel. It is important to state that criteria for separation will vary for different types of steels and processing parameters (deformed and non-deformed samples), for this works the values are suitable. For others conditions and steels, others criteria and values may be used to obtain reasonable results.

Zhao [41] showed that in HTP API X-80, with 0.045C-1.43Mn-0.14Si-0.09Nb-0.21Cr-0.12Ni-0.21Cu-0.01Ti (wt.%), non-deformed, subjected to continuous cooling of 20 °C/s, the predominant microstructure was bainite (80%). Comparing results obtained by Zhao [41] with the results of this research for non-deformed samples, continuous cooled at 20 °C/s, it was possible to determine that manganese favors bainite formation in non-deformed samples. At 20 °C/s, in non-deformed condition, the predominant microstructure (76%) in low manganese HTP was PF/QF.

4. Conclusions

The results of the present work suggested that the methodology proposed allowed to identify and quantify complex microconstituents in low manganese HTP steel in deformed (50% below Tnr and 75% below Tnr, in austenite region) and non-deformed conditions. The results were similar than those obtained by the point count methodology.

Grain boundaries characteristics allowed identification and quantification of the different ferritic microconstituents in a low manganese HTP steel in deformed (50% below Tnr and 75% below Tnr, in austenite region) and non-deformed conditions.

The results suggested also that manganese may favor bainite formation in non-deformed samples, but further test and analysis should be performed.

**Conflicts of interest**

The authors declare no conflicts of interest.

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