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HIGH VERSATILITY OF NIOBIUM ALLOYED AHSS

The effect of processing parameters on the final microstructure and properties of advanced high strength CMnSiNb steel was investigated. Several processing strategies with various numbers of deformation steps and various cooling schedules were carried out, namely heat treatment without deformation, conventional quenching and TRIP steel processing with bainitic hold or continuous cooling. Obtained multiphase microstructures consisted of the mixture of ferrite, bainite, retained austenite and M-A constituent. They possessed ultimate tensile strength in the range of 780-970 MPa with high ductility $A_{5\ \mathrm{mm}}$ above 30%. Volume fraction of retained austenite was for all the samples around 13%. The only exception was reference quenched sample with the highest strength 1186 MPa, lowest ductility $A_{5\ \mathrm{mm}}$ = 20% and only 4% of retained austenite.

Keywords: TRIP steel, niobium, heat treatment, thermo-mechanical treatment

1. Introduction

With increasing concerns for vehicles safety, fuel consumption and carbon footprint, the automotive industry has been increasingly demanding materials, which would maintain good formability while improving tensile strength. The need to decrease the weight of the vehicles has prompted several decades of research and development of new grades of high strength steels. Several grades of advanced high strength steels have been based on the presence of strictly controlled amount and morphology of retained austenite. The first one were TRIP (transformation induced plasticity) steels. They possess high strength, good ductility, formability, and high ability to absorb impact energy. These properties results from their complex microstructure consisting of ferrite, bainite and retained austenite. Stabilization of retained austenite to room temperature is achieved by a special heat or thermo-mechanical treatment, which allows carbon enrichment of the remaining austenite before final cooling. Retained austenite can transform to martensite during cold plastic deformation. This effect can be used either to strengthen the final product by the last cold forming operation, or to absorb an impact energy during the crash in the case that retained austenite remains in the final product. Modern TRIP steels are low alloyed steels with typically 0.2-0.4% of carbon and 1-2% of silicon and manganese. Various alloying concepts of TRIP steels have been researched, for example complete or partial aluminium substitution of silicon, micro-alloying by niobium, copper, phosphorus or chromium alloying [1-4].

Niobium addition to a standard C-Mn-Si steel enables TRIP microstructure to be produced even at low cooling rates and without traditional bainitic hold [5], which makes this alloying concept very promising for commercial applications. In most of the works, the processing of TRIP steels with niobium is carried out either as cold rolling followed by intercritical annealing [6], or as thermo-mechanical processing with several deformation steps [7]. For thermo-mechanical treatment, the number of deformations, deformation temperatures and sizes are very important factors influencing resulting microstructures and properties. In this work, both heat and thermo-mechanical treatments were applied to an experimental C-Mn-Si-Nb steel to evaluate the effect of processing parameters on the final microstructure and properties.

The advantage of retained austenite to an enhancement of strength and ductility of steels has been recently successfully utilized also in advanced martensitic steels processed by quenching and partitioning heat [8] or thermo-mechanical treatments [9, 10].

2. Experimental program

2.1. Heat and thermo-mechanical processing

Several processing strategies were tested for CMnSiNb steel with 0.2%C, 1.5%Mn, 1.8%Si and 0.06%Nb (wt %). Several processing strategies, which typically produce TRIP microstructure, were designed (Table 1). To keep the processing

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cost efficient and environmental friendly, soaking was carried out for 100s at relatively low temperature of 900°C. Only in one case, the soaking temperature was increased to 1050°C to simulate typical heating conditions used in commercial rolling of low alloyed steels. For all processing methods, relatively high heating rate of 20°C/s was used.

The first processing consisted of a two-step heat treatment without deformation. However, most of the other strategies applied various numbers of deformation steps during the cooling from the soaking temperature to the bainitic hold temperature to evaluate the effect of the number of deformation steps on the final microstructure and properties. Either one, two or three individual compressive deformations or 20 incremental deformation steps were applied to the steel during the cooling from 900°C to 720°C. Individual deformations are commonly used to simulate real processing by various forming technologies, for example forging [11] or hot rolling [7]. The amount of each deformation step was equal to 10% of the actual length of the sample and all of them were compressive ones.

In the next step, a strategy with 20 incremental deformation steps was tested. This strategy simulate stress-strain states existing in materials processed by rolling mills or rotary spin extrusion with high accuracy and they enable higher total deformation to be applied to relatively small samples without the risk of cracking [12]. Each deformation step consisted of a tensile and compressive deformation. The compressive deformation was always slightly higher to achieve the change of the shape of the samples as well. The relative size of all the deformation steps was kept the same with regard to the actual size of the sample and the total logarithmic deformation was equal to 5.

Based on the results of previous experiments, 720°C was kept as the temperature of the last deformation and subsequent bainitic hold was performed at 425°C for 600s [13]. Average cooling rate around 14°C/s was used for the cooling from 900°C to 425°C.

Bainitic hold is a typical feature of TRIP steel processing. The last processing strategy was designed as a variant of a real rolling mill with continuous cooling and a higher soaking temperature of 1050°C. Bainitic hold was in this case replaced by a change of cooling rate from 16°C/s to 1°C/s at 425°C. Two compressive deformations were applied at 1050°C and 830°C.

Finally, a reference sample was quenched at 50°C/s from the prevailing soaking temperature of 900°C down to the room temperature. The first aim of this treatment was to check ferrite occurrence at this soaking temperature. The second was to obtain mechanical properties of the steel in as-quenched conditions.

Thermo-mechanical simulator was used to process all the samples (Fig. 1). The final microstructures were analysed using Zeiss Crossbeam Auriga and EVO 25 scanning electron microscopes with an EBSD detector and light microscopy with image analysis. The volume fraction of the retained austenite was determined by X-ray diffraction phase analysis using an AXS Bruker D8 Discover automatic powder diffractometer with a HI-STAR detector and Co lamp (λKa = 0.1790307 nm). A focusing polycapillary lens was used to achieve an X-ray spot with 0.5 mm diameter. The measurement was carried out in the central part of the samples and spectra were taken in the range of 2ϑ from 25° to 110°. The integrated intensities of (200) ferrite peak and (111), (002) and (022) austenite peaks were used for evaluation. In-house developed image analysis software was used to evaluate ferrite grains size by an intercept method. The mechanical properties were measured by tensile testing of flat mini-samples, with a gauge length of 5 mm.

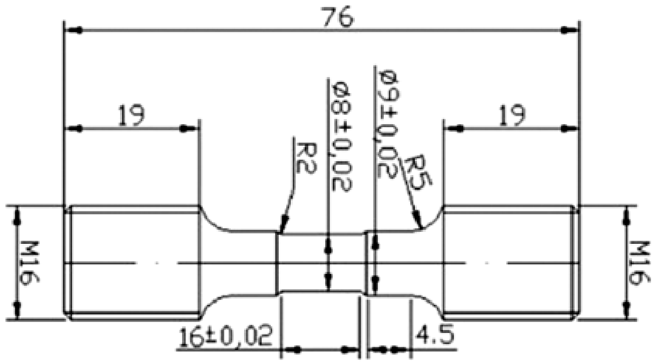

Fig. 1. Geometry of the sample for thermo-mechanical simulator

TABLE 1

Parameters of the treatment, Ferrite grain size, temperature of deformations (T_{def}), total logarithmic deformation (Φ), yield strength (*Re*), ultimate tensile strength (*Rm*), ductility ($A_{5\ mm}$), tensile strength and ductility product ($Rm \times A$), retained austenite volume fraction (*RA*)

Number of deformations	T_{def} [°C]	Φ [-]	*Re* [MPa]	*Rm* [MPa]	$A_{5\ mm}$ [%]	$Rm \times A$ [MPa%]	*RA* [%]	*F* grain size [mm]
Soaking at 900°C/100s, cooling to 425°C by 14°C/s, bainitc hold at 425°C/600s								
—	—	—	527	803	34	27302	14	4.8
1	720°C	0.1	583	829	31	25699	15	4.6
2	900, 720°C	0.21	654	859	34	29206	13	3.1
3	900, 870, 720	0.31	603	810	33	26730	12	4.4
20	900-720	5	440	783	34	26622	12	4.0
Soaking at 1050°C/100s, cooling to 425°C by 16°C/s, cooling to room temperature by 1°C/s								
2	1050, 830	0.21	403	978	30	29340	10	4.1
Reference sample: Soaking at 900°C/100s, quenching to room temperature by 50°C/s								
—	—	—	679	1186	20	23720	4	3.5

3. Results and discussion

3.1. Microstructure and properties

The highest average ferrite grain size was obtained after a two-step heat treatment without deformation, reaching 4.8 micrometres. Bainitic blocks of a similar size as ferrite grains were found in the microstructure and the distribution of phases and structural components was very uniform (Figs. 2,3). There was 14% of retained austenite in the microstructure. The tensile strength of this microstructure was 803 MPa and the ductility $A_{5\ \mathrm{mm}}$ reached 34%.

One deformation at 720°C resulted in a slight refinement of ferrite grains to 4.6 micrometres, however also in an increase of bainitic blocks size in the final microstructure and generally in a higher amount of lath bainite (Fig. 4). Bainite distribution was more heterogeneous than in the microstructure after pure heat treatment. The tensile strength increased to 829 MPa with the ductility $A_{5\ \mathrm{mm}}$ = 31% and 15% of retained austenite.

Two deformations carried out at 900°C and 720°C refined all phases in the final microstructure (Fig. 5). The final microstructure contained bainitic areas with predominantly globular morphology, with just an occasional occurrence of larger blocks of lath bainite. These blocks were of the same size as after one deformation; however, the laths inside of them were finer. The lowest ferrite grain size around 3 micrometres was achieved in this microstructure and a volume fraction of the retained austenite was very similar to the previous two cases, reaching

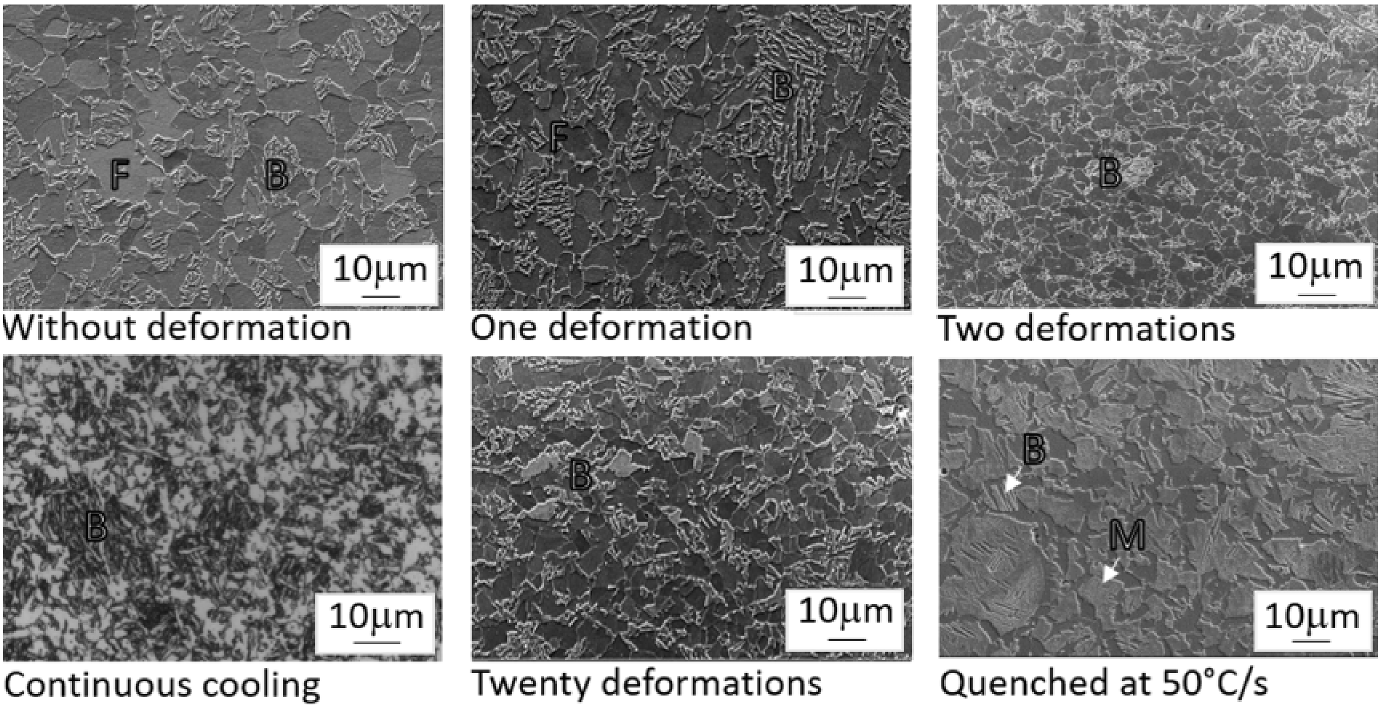

Fig. 2. Microstructure overview after various heat and thermo-mechanical treatments

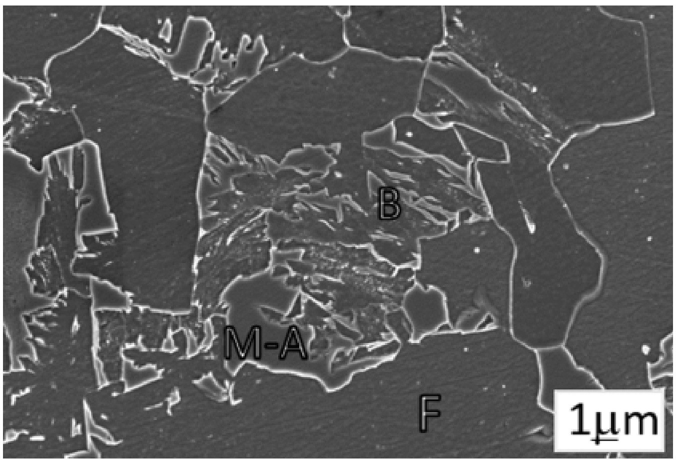

Fig. 3. Heat treatment without deformation, 900°C/100s, cooling at 14°C/s to 425°C/600s, coarse blocks of globular bainite, ferrite grains

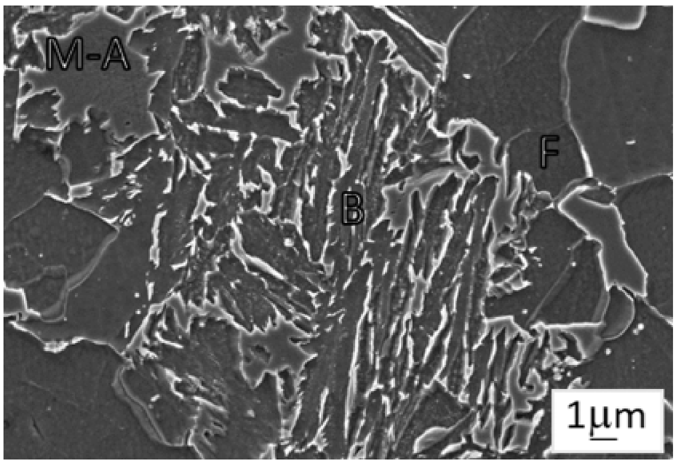

Fig. 4. Processing with 900°C/100s soaking, one deformation at 720°C, cooling at 14°C/s to 425°C/600s, coarse blocks of lath bainite in the microstructure

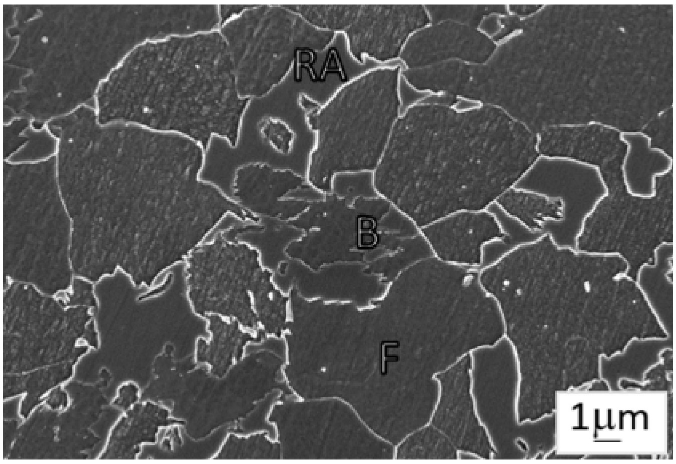

Fig. 5. Processing with 900°C/100s soaking, two deformations at 900°C and 720°C, cooling at 14°C/s to 425°C/600s, fine microstructure with globular bainite

13%. Ferrite refinement was therefore most probably responsible for another increase of the strength to 859 MPa combined with a high ductility $A_{5\ mm}$ of 34%.

Higher number of deformations resulted in a drop of tensile strength, even though the volume fraction of the retained austenite was still relatively high, reaching 12%. Three deformations applied at 900°C, 870°C and 720°C produced the microstructure with the tensile strength of 810 MPa and the ductility $A_{5\ mm}$ = 33%.

Twenty incremental deformation steps with a decreasing size of an applied deformation created more homogeneous distribution of the finer areas of lath bainite (Figs. 2,6). However, large complex laths of M-A constituent and bulk islands of M-A constituent with prevailing martensitic microstructure and retained austenite being only at the very edges, were also typical for this microstructure. There were still 12% of the retained austenite. This microstructure possessed a lower strength of only 783 MPa with the ductility $A_{5\ mm}$ = 34%. The lowest strength obtained by this processing was most probably caused by coarsening of M-A constituents, which could also influence the stability of the comprised retained austenite.

The effect of various numbers of deformation steps on texture of the final microstructures was not considered in this work. It was already demonstrated by Jirkova [14] for the same steel with 0.03% of niobium, that there was hardly any difference between the textures in ferrite and austenite obtained after thermo-mechanical treatment with 900°C soaking temperature and various numbers of deformation steps applied during the cooling from 900°C to 720°C. Based on the X-ray diffraction analysis, the same weak preferred orientation of <110> in ferrite and <200> in austenite were found in the microstructures after several treatments with the same thermal schedules and up to 20 deformation steps applied in the same temperature interval.

Thermo-mechanical processing of TRIP steels is typically done with a hold in a bainite transformation region. However, it has been demonstrated particularly for Nb alloyed steel that suitable microstructures with a sufficient volume fraction of retained austenite can be achieved also by the processing with continuous cooling. In a previous work, incremental deformation steps combined with a very slow cooling at a constant rate of 1°C/s were successfully utilized for the same CMnSiNb TRIP steel [5]. In recent work, two individual deformations were applied at the temperatures of 1050°C and 830°C and the cooling rate was changed at 425°C to allow quicker cooling at higher temperatures. The final microstructure consisted of a mixture of free ferrite, bainite and M-A constituent and the morphology was very similar as in the previous samples with bainitic hold (Fig. 7). Martensitic or M-A constituent laths or islands formed the edges of continuously cooled bainitic areas, showing a lower stability of the remaining austenite during the final cooling stage. Even a lower cooling rate of 1°C/s used for the cooling from 425°C to room temperature, did not offer enough time for partitioning of a sufficient carbon content into the remaining austenite to make it stable against martensitic transformation. However, there was still 10% of retained austenite detected by an X-ray diffraction and this microstructure possessed one of the best combinations of the mechanical properties of all the tested samples. It reached the highest strength of 978 MPa with a very good ductility A_{5mm} = 30%. In comparison, the microstructure after continuous cooling at a constant cooling rate of 1°C/s and 20 incremental deformation steps reached only the strength of 850 MPa with the ductility A_{5mm}=24% [5].

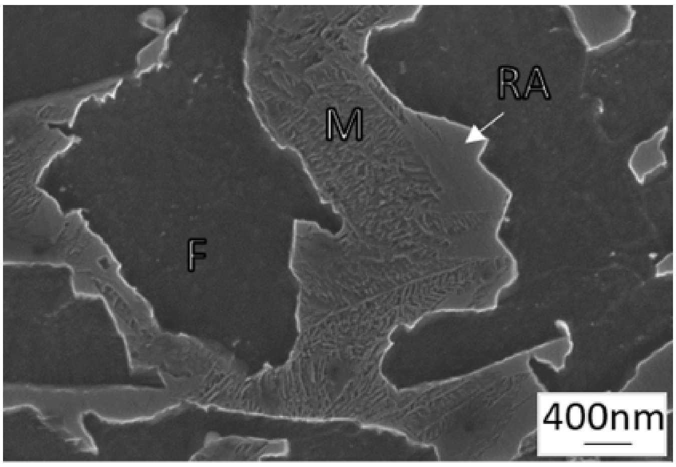

Fig. 6. Processing with 900°C/100s soaking, 20 incremental deformation steps at 900-720°C, cooling at 14°C/s to 425°C/600s, M-A constituent with complex shape

Direct cooling from 900°C to room temperature at 50°C/s produced a mixed martensitic-bainitic microstructure with chains of proeutectoid ferrite at the prior austenite boundaries (Fig. 8). Around 20% of free ferrite were found in the microstructure by an image analysis. This microstructure reached the highest strength of 1186 MPa due to a large martensite volume fraction, however it was combined with the lowest ductility of 20% resulting from the lowest amount of retained austenite (4%) and ferrite. The reference sample demonstrated a relatively high

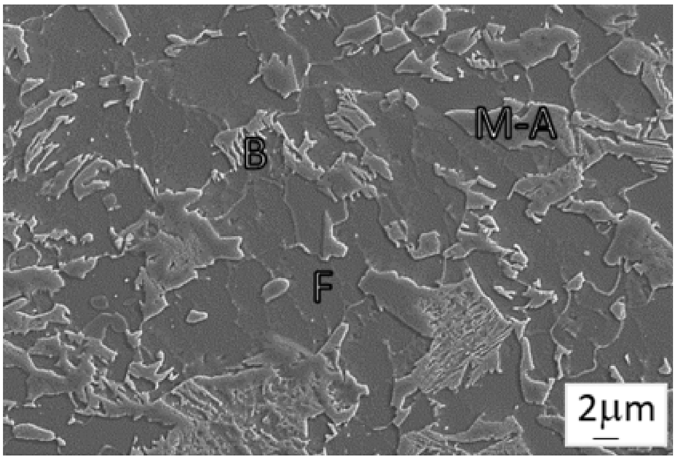

Fig. 7. Processing with continuous cooling from 1050°C with 2 deformations at 1050°C and 830°C, cooling rate 16°C/s to 425°C and 1°C/s to room temperature, bainitic blocks with large M-A constituent at the edges

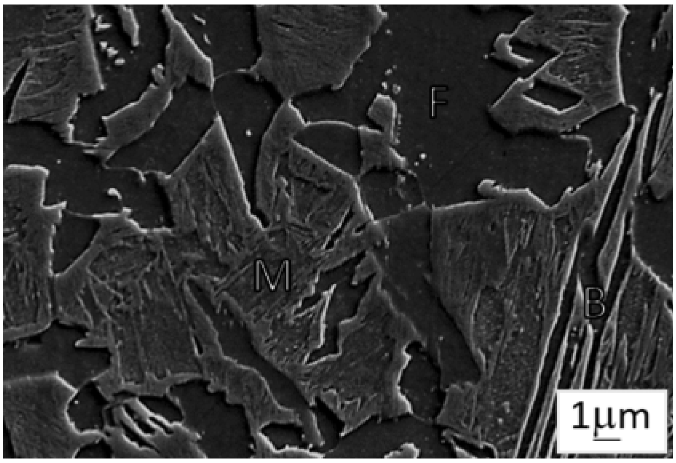

Fig. 8. Water quenching from 900°C/100s soaking at 50°C/s, large bainitic and martensitic blocks surrounded by ferrite grains

ductility of this steel even after a conventional quenching from an intercritical region. However, the stabilization of around 10% of retained austenite and exchange of most of the martensite for bainite improved the ductility by 50%, as its gradual transformation to martensite during tensile test postpones the onset of necking, thus prolonging the area of homogeneous plastic deformation.

3.2. Effect of niobium

It has been long known that the effect of niobium on the final microstructure and mechanical properties depends on its state [15-18]. Niobium can be present in a steel either dissolved in a solid solution or precipitated in niobium carbides (NbC) or more complex niobium carbo-nitrides (Nb(C,N)). The ratio of dissolved and precipitated niobium is mainly determined by soaking conditions, namely by a heating rate, soaking temperature and soaking hold. During thermo-mechanical treatment, dissolved niobium can participate in strain induced precipitation, which can have significant effect on recrystallization processes in the steel and subsequent phase transformations. A soaking temperature of at least 1200°C is generally required to dissolve a major fraction of primary NbC in low alloyed C-Mn-Si-Nb steels [17]. Relatively low heating temperature of 900°C with 100s hold combined with a higher heating rate of 20°C/s would keep Nb diffusivity and Nb content in solid solution low for most of the processing methods used in this work. Hong et al. [17] reported that 94% of niobium carbides was still undissolved after 2 min hold at 900°C and no change in NbC state was found after 60s hold at this soaking temperature by Hausmann et al. [18]. It can be therefore assumed that the amount, size and morphology of NbC precipitates did not play significant role in variations of the final microstructures and mechanical properties obtained for the samples soaked at 900°C, as the precipitates remained in all samples in their original pre-processing state.

However, partial NbC dissolution could have occurred at a soaking temperature of 1050°C, where around 10% of primary niobium carbides could be dissolved according to [17]. This treatment with continuous cooling resulted in the highest strength of 972 MPa. It should be also noted that a similar treatment with the same soaking conditions and deformation temperature interval, but with slower cooling rate of 1°C/s, produced in previous work markedly lower strength and ductility [5]. High strength obtained in this work for the sample soaked at 1050°C should therefore not be seen primarily as a result of partial NbC dissolution at a higher soaking temperature and possible strain induced precipitation during the following deformations. Good combination of mechanical properties should be in this case attributed rather to the synergic effect of a more complex final microstructure with bainitic areas strengthened by adjacent M-A constituent islands distributed homogenously in a soft ferritic matrix.

3.3. EBSD

As the volume fraction of the retained austenite was nearly the same for all the processing, EBSD analysis was performed at two samples with the most different treatments. The first one was after the heat treatment without deformation (Fig. 9) and the second one after thermo-mechanical processing with 20 deformation steps (Fig. 10). In both microstructures, retained austenite was detected in the form of the bainitic laths and fine bulk islands. Inverse pole figures were calculated for both samples in all three directions x,y and z. For both samples, retained austenite laths in neighbouring bainitic blocks had the same orientation, which happened also to be similar to the orientation of surrounding grains of proeutectoid ferrite. It is interesting to note that the orientation of bainitic ferrite laths is quite different. Bulk islands of retained austenite lining the boundary of free ferrite grain (Fig. 9) had different orientation from austenitic laths of nearest bainitic areas.

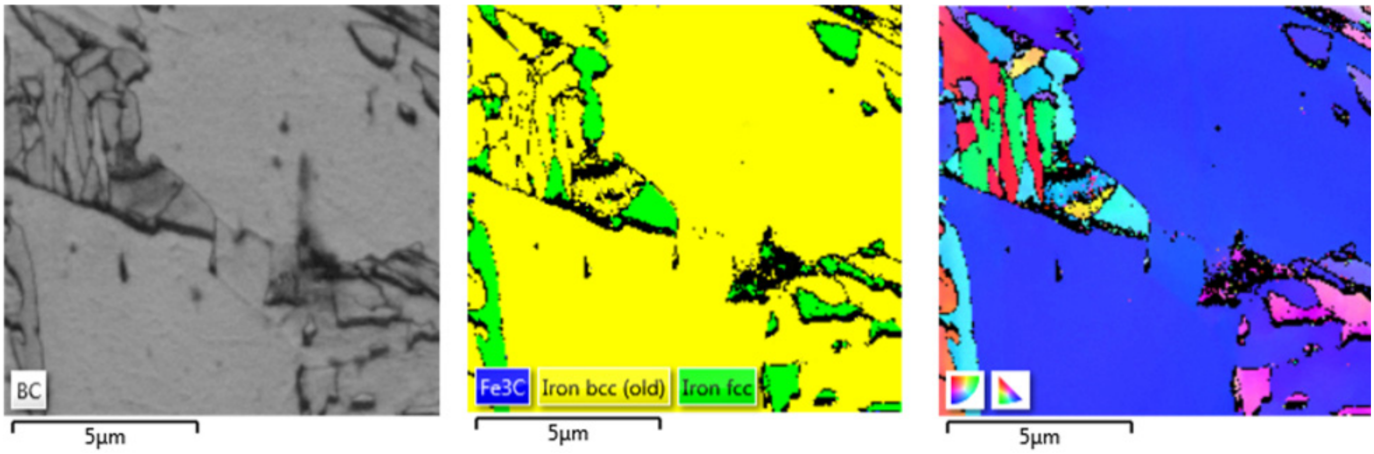

Fig. 9. Heat treatment: Band contrast, phase map, IPF Z map, several different orientations of retained austenite, the laths within the same bainitic block had the same orientation

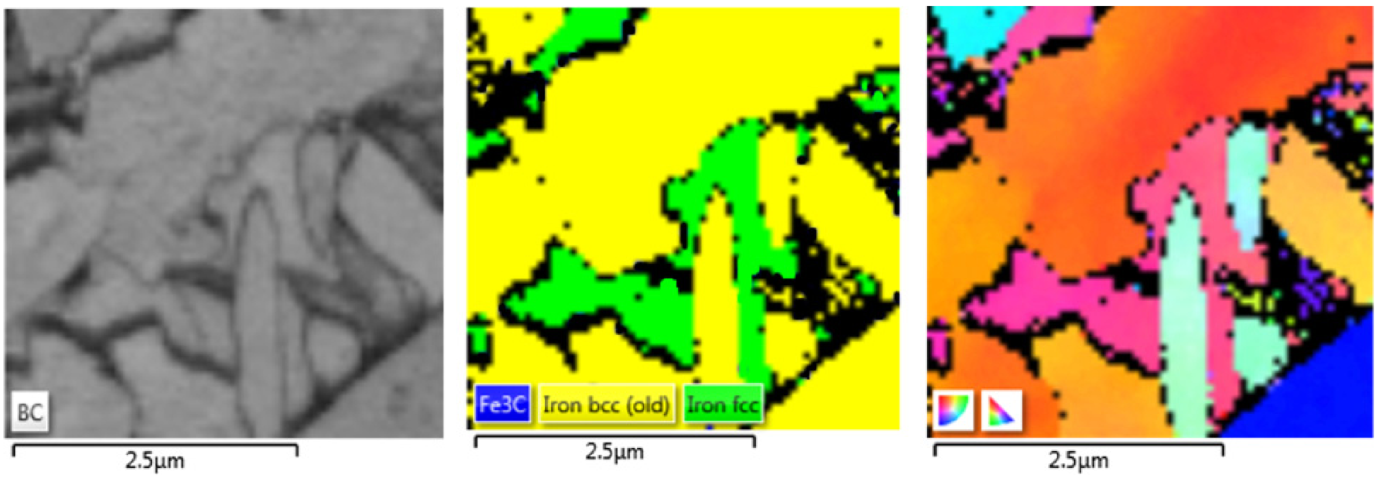

Fig. 10. Processing with 20 deformation steps: Band contrast, phase map, IPF Z map – areas marked as retained austenite had the same orientation

4. Conclusions

High versatility of niobium alloyed high strength steel was demonstrated in this work. The steel possessed good strength of 1186 MPa and ductility $A_{5\ \mathrm{mm}}$ = 20% even after simple quenching from an intercritical region. These properties were however further improved by dedicated heat and thermo-mechanical treatments.

Two step heat treatment, or thermo-mechanical processing with either several individual deformations, or with a set of 20 incremental deformation steps can be carried out on C-Mn-Si-Nb steel to produce TRIP microstructure with the strength around 850 MPa and the ductility over 30%. The microstructure with 10%-14% of retained austenite can be obtained even after thermo-mechanical processing with continuous cooling and by heat treatment alone, without any deformation. Due to the low sensitivity of final mechanical properties to the change of processing parameters, this steel has high potential for a wide range of commercial applications.

The best combinations of strength and ductility, accompanied by high energy absorption during deformation, expressed by strength and ductility products ($Rm \times A$) around 30 000 MPa%, were obtained for two different thermo-mechanical processing. The first one was carried out with 900°C soaking temperature and two deformations during the cooling to a bainitic hold, while the second processing used 1050°C soaking temperature followed also by two deformations but with continuous cooling schedule. Continuous cooling resulted in the highest strength of nearly 980 MPa with very good ductility of 30%, while the processing with bainitic hold possessed higher ductility of 34% with tensile strength of 859 MPa.

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