Prior thermo-mechanical processing to modify structure and properties of severely deformed low carbon steel

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Abstract. The article focuses on the severe plastic deformation (SPD) of low carbon steel AISI 1010 performed at increased temperature. The grain refinement of ferrite structure is monitored and described with respect to different initial steel structure modified by thermal and thermo mechanical (TM) treatment (TM) prior severe plastic deformation. The refinement of coarse initial ferrite structure with grain size in range of 30 – 50 µm resulted from solutioning was conducted then in two steps. Preliminary structure refinement has been achieved due to multistep open die forging process and quite uniform ferrite structure with grain size of the order of µm was obtained. The further grain refinement steel structure was then accomplished during warm Equal Channel Angular Pressing (ECAP φ = 120°) at 300°C, introducing different strain in range of \( \varepsilon_{\text{eff}} = 2.6 - 4 \). The change of microstructure in dependence of the effective strain was evaluated by SEM and TEM study of thin foils. The high straining of steel resulted in extensive deformation of ferrite grains and formation of mixture of submicron grains structure in banded deformed structure with dense dislocation network and subgrains. The dynamic polygonization process, due to increased ECAP temperature, modified the sub-microcrystalline structure formation. There was only indistinctive difference observed in structure refinement when considering different initial structure of steel. The tensile behaviour was characterized by strength increase followed by softening. None work hardening phenomenon appeared at tensile deformation of deformed bars.

1. Introduction
Ultrafine grained materials obtained by severe deformation are receiving increasing attention in the material science community in the last years. The term “ultrafine grain structure” is referring to nanostructure with grain size of less than 100 nm and sub-microcrystalline structure with grain size between 100 and 1000 nm. The fabrication of bulk materials with ultrafine grain sizes has attracted great deal of attention over the last decades because of the materials’ enhanced strength properties [1-3]. In recent years, it has become a worldwide effort to develop a manufacturing process to obtain ultrafine grain structure in steels. Currently, there are two main approaches for refining ferrite grains down to the ultrafine grain range in bulk steels. While the first group comprises advanced thermo-mechanical processes [4-7], the approach of the second group employs various sever plastic deformation techniques, including ECAP [1,8], high pressure torsion (HPT) [9], accumulative roll bonding (ARB) [10,11], constrained groove pressing (CGP) [12], to refine the structure by introducing the large plastic strain into bulk material. The use of ECAP for commercial steels has been studied elsewhere. There were studies dealing with the use of ECAP for low carbon steels as well. However, with cold ECAP a low carbon steels can be only pressed by two or three passes with channel intersection of 90° and then surface defects following the shearing planes appear. The two to four passes realized currently with cold ECAP are insufficient and the achievable strain level is insufficient to produce a completely refined grain structure [13]. To form stable ultrafine grain structure in metals and metallic alloys the ECAP deformation should be carried out at the temperature corresponding to the temperature of cold working. In the present study, the modification of ferrite microstructure due to thermo-mechanical processing is described. Subsequently the effect of structure modification on development of...
ultrafine grain microstructure resulting from warm SPD was investigated and is assessed comparing with UFG structures resulted at SPD of conventionally treated AISI 1010 steel. The underlying relationship between microstructure and mechanical properties of the experimental steel is reviewed.

2. Material and experimental procedure
In this work, the commercial low carbon steel AISI 1010 was used. The chemical composition of the carbon steel in wt % is as follow: 0.1 C, 0.42 Mn, 0.18 Si, 0.024 P, 0.018 S. Prior to ECAP pressing a conventional solutioning treatment of billets was carried out at the temperature of 920°C foe 1 hour, followed by air cooling. The resulted microstructure, documented by SEM after solution treatment is presented in Fig. 1. The cylindrical billets with initial with \( l = 50 \text{ mm} \) and \( \phi = 9 \text{ mm} \) were cut off the for ECAP deformation experiment.

![Fig. 1. Microstructure of solutioned steel.](image)

![Fig. 2. Resulted microstructure of TM treated steel.](image)

By appropriate thermo-mechanical (TM) treatment the refining of the coarse ferrite structure is possible and more suitable combination of the strength, toughness and ductility can be obtained without additional alloying. Accordingly, in order to achieve prior ECAP pressing advanced preliminary grain refinement in solutioned steel, the TM processing has been performed as follows. After prior steel soaking at 900°C steel rods in form of pegs, with initial diameter of 18 mm and length of 40 mm were the first subjected to compressive deformation [1]. The repetitive axial pressing deformation of the peg between flat dies of hydraulic press performed continuously, without further inter reheating, through the recrystallized, non-recrystallized and inter-critical \( \gamma-\alpha \) temperature region, was expected to result in prior ferrite structure refinement. The last reduction of the specimen was then carried out at temperature of about 700°C. The peg specimen between successive deformation reductions was rotated about its axis until the final shape of specimen was obtained. The resulting SEM microstructure in the centre of the peg is shown in Fig. 2. The average ferrite grain size measured at areas of various compressive strains across the peg, from the peg surface to its centre, was less than 5 \( \mu \text{m} \).

The ECAP pressing was performed at temperature of 300°C. The angle of channels intersection (\( \phi \)) in ECAP die was equal to 120°. The ECAP die used for deformation experiment was heated to pressing temperature and held on for 30 minutes to have uniform temperature. The samples was heated for 300 s prior to pressing, which was done inside the pre-heated die until samples reached the pressing temperature. Each billet was then pressed to four, five and six passes through the die. The effective strain corresponding to one pass was \( \varepsilon = 0.67 \). Performing six passes the corresponding total strain \( \varepsilon_{\text{tot}} \) was equal 4. The billet was rotated between the consecutive passes about its longitudinal axis by 90° in the same direction. The procedure is generally referred to as the processing route Bc and it was selected to deform bars because it enables the formation of homogenously deformed microstructure in billet. It was not considered that the stress generated in sample after each
pass would be recovered (static recovery) due to repeated reheating of the each billet inside the die prior the next pass will be carried out.

The microstructural examination of samples experienced thermal and/or TM treatment and ECAP deformation was conducted by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Thin foils for TEM analyses were sliced normal to the longitudinal axis of ECAP press billets. The SEM and TEM microstructures were obtained by using SEM JEOL JSM 6380 operating at 10 kV and JEOL JEM 200FX TEM operating at 200 kV. Selected area electron diffraction microscopy (SAED) was then used to investigate the ultrafine grain transformation process in dependence the strain introduced.

Vicker hardness and tensile tests were carried out using MTS universal testing machine equipped with Multisens extensometer. Tensile specimens with gauge length of $l_0 = 30$ mm were tested at a constant cross-head speed of 0.016 mm/s until failure. The engineering stress-strain curves were constructed.

3. Experimental results and discussion

3.1. Microstructure of solutioned and ECAP steel

The ferrite structure morphology when applied steel solutioning at 920°C was found uniform across the billet, Fig. 1. Scarcely, the cementite particles were precipitated along grain boundaries. The ferrite grains mean size was in range of 30 – 50 µm. Microstructural characteristics of prior solutioned and prior TM treated steel exposed to different ECAP straining, for the chosen processing route Bc, were analyzed in details on sections normal to longitudinal axis of deformed billet. Light and SEM micrographs of the as-pressed steel samples provided evidences of effective steel straining, either prior in coarse and/or in prior refined (TM) ferrite structure. Representative micrographs of deformed solutioned and TM prior treated steel which were taken on the X plane (perpendicular to deformed bar axis) are shown in Fig. 3 and Fig. 4. Each microstructure represents the effect of different strain (number of executed passes) the steel was exposed. Deformation characteristics, regarding the initial structure condition and applied strain, provided evidences on deformation heterogeneity distribution in deformed samples, regardless the applied ECAP straining. In prior coarse steel initial microstructure, introduced straining, no matter what was the level,
was not sufficient to deform steel structure uniformly across the billet, as could be seen in light and Fig. 3. Deformation localization and slip banding of ferrite grains was increasing with increasing straining, i.e. with increasing ECAP passes. Deformation bands appeared more frequent and were distributed in more dense manner as straining increases. In the steel bars, which were prior refined by applying TM treatment, applying straining resulted in more homogenized deformation structure, however deformation heterogeneity was detectable as well, however in less extent. In deformed structure it was possible to find differently exposed areas where due to strain non-homogeneity the grain refining was efficient regardless the higher strain level was increased. In deformed bands where shearing effect was evident the grain refinement appeared to be effective and fragmentation of deformed grains due to effective straining is evident, Fig. 4, (N6). These areas in deformed structure however suggest that transformation process for ultrafine grain structure tends to be more efficient when prior ECAP deformation the TM process was introduced to pre-refined microstructure. Performing N-5 and N-6 passes deformation resulted in more dense banding and within these bands the dense dislocation network and dislocation cell structure provide good conditions for more effective structure refining. Not regularly, but inside the deformation bands, the fragmentation of extended grains, as results of dislocation slipping process, may support the formation of new small refined grains. The received results on severe deformation of low carbon steel suggest, that using the die with channels angle of $\varphi = 120$, the higher straining is needed to prepare homogeneous deformed structure and substructure in steel. The TM preliminary treatment of steel by only small deal contributes to deformation homogeneity and structure refinement at deformation.

3.2. TEM microstructure of solutioned and ECAP steel

Microstructure analysis by SEM provided the evidence on applied effective strain, which was not sufficient to deform structure uniformly across the deformed steel bar, regardless the initial structure was either coarse and/or refined by preliminary TM treatment. In order to receive more details on ultrafine grain structure formation in deformed steel the structure
transformation was also investigated by TEM of thin foils. It was then possible to specify the
mechanism which could govern the structure transformation in severely deformed steel.
The development of deformed substructure in steel with different initial microstructure and
subjected to different straining is shown as collection of substructures in Fig. 5. The
substructure developed in deformed billets subjected to warm ECAP at temperature of 400°C
was investigated on plane parallel with billet longitudinal axis and substructure analysis
provided the evidence on efficiency of deformation process for structure refinement in the
same steel having different initial microstructure. These representative microstructures show
the progress in ultrafine grained as straining increases.

Comparing effect of prior structure refinement of steel by TM processing no substantial
difference in microstructure formation was observed. At both initial structures, either coarse
or refined by TEM, the process. The deformation bands, which are formed in structure,
consist of the elongated ferrite grains where dense dislocation network and dislocation cell
structure is build up, as can be seen in Fig. 5 a,e. Considering the local straining
heterogeneity, the microstructural evolution and structure development depends on the
selected position of analysis. The local substructure modification across the sample varied
locally. At both ECAP steel samples with increased straining (N=5 and N=6) dislocation
activities can be related to progress of polygonization and nucleation of new subgrains within
the elongated ferrite grains, as shown in Fig. 5 b,c,f. The specific fringe contrast along some
subgrains boundaries and appearance of small grains free of dislocations can be attributed to
in-situ dynamic recovery and polygonization process, and probably recrystallization as well.

The more developed small equiaxed grains with high angle grain boundaries and with less
dislocation inside are shown for both steel states subjected to \(N=6\) passes, Fig. 5 d,g,h. More frequently was this ultrafine grain found in steel, which was treated by TM treatment prior ECAP deformation. The SAED net patterns indicated an increase in the reasonable portion of boundaries having high angles of misorientation. This result can be due the fourfold effect, involving grain refinement, the ECAP pressing temperature, strain introduced and latent heat generated at SPD, acted as an effective driving force for dynamic recrystallization process. The progress in ultrafine grained structure formation was more evident in deformed steel samples, which were preliminary TM treated prior ECAP processing. More effective structure refinement after ECAP can be due the structure refinement by preliminary TM processing of steel. It is probably caused by cumulative straining resulted from both processes - TM and ECAP of steel and reaching higher driving force for dynamic recovery and recrystallization process in deformed structure.

### 3.3 Mechanical properties of steel

The mechanical properties of experimental steel, which was subjected to thermal and thermo-mechanical treatment prior SPD were measured by tensile test and hardness measurement. The results of tensile testing performed at room temperature are shown in Fig. 6 a,b, which represents the initial solutioned structure, solutioning and ECAP and TM and ECAP treatment. In case of the initial annealed steel condition there is an extensive period of work hardening and large elongation to failure. The deformation curve corresponding to TM treated steel shows slight work hardening effect and shorter deformation to failure. The mechanical properties data for all structural states are stated in Table 1 and Table 2.

| state | E [GPa] | \(R_p_{0.2}\) [MPa] | \(R_m\) [MPa] | \(A_{\text{tot}}\) [%] | \(Z\) HV30 [%] |
|-------|---------|----------------|---------------|----------------|----------|
| SOL   |         | 245            | 303           | 37             | 81       | 101 |
| N4    | 200     | 813            | 819           | 10.4           | 63       | 264 |
| N5    | 191     | 763            | 768           | 9              | 60       | 260 |
| N6    | 208     | 851            | 857           | 9              | 60       | 268 |

| state | E [GPa] | \(R_p_{0.2}\) [MPa] | \(R_m\) [MPa] | \(A_{\text{tot}}\) [%] | \(Z\) HV30 [%] |
|-------|---------|----------------|---------------|----------------|----------|
| TM    | 216     | 357            | 370           | 31             | 76       | 109 |
| N4    | 224     | 811            | 819           | 10.8           | 58       | 320 |
| N5    | 213     | 767            | 778           | 9.5            | 60       | 260 |
| N6    | 210     | 824            | 833           | 10             | 54       | 379 |

The deformation behaviour of soaked and ECAP steel specimens is very similar for both initial steel structural states and/or three specimen, which have experienced different deformation. There is, after reaching the yield stress, section of slight hardening increase, which is not modified as the straining is increased. On the other side the strength value is of the same level for all specimens, which can be incurred by explanation of large deformation strengthening of ferrite component, as could be seen in deformed ferrite steel structures. It seems curious that variation in prior steel thermal treatment did not change deformation behaviour of steel when ECAP-ed.
4. Experimental results evaluation and discussion
When evaluating the progress in ultrafine grain structure development and mechanical with respect to strain introduced a conclusion can be expressed that there was not achieved a significant difference observed what initial state of structure in steel was prepared. Microstructural evolution process was only slightly modified as to the formation of deformed structure advancement with a certain level of strain introduced. TEM substructure analysis however provided the evidence that formation of ultrafine grained structure was a step forward when compared with initial coarse steel structure deformation process. Deformation heterogeneity, which was observed higher in coarse grain structure, restrained consequently the recovery and polygonization process in deformed structure. As the result of this postponement the formation ultrafine grains with was subsequently delayed and smaller fraction of them was found in mixed microstructure deformed to various strain. The contribution of this fact was detected also by small decrease of the strength value for TM treated steel where the more advanced volume fraction of ultrafine grains in microstructure cause slight drop in ultimate strength of steel. The level of deformation strengthening was still higher in coarse structure. Nevertheless the progress of structure refinement influence only in small extent the mechanical behaviour of different steel states.
When final evaluation of deformation experiment of low carbon steel AISI 1010 it is important to state out that resulted strength values of experimental steel modified by intensive channel deformation reached the value, which was more then two times higher when comparing with values resulting from industrial heat treatment of low carbon steel (see Table 1). The drawback of this fact is the lack of further plastic deformation ability of severely deformed steel as results of ductility evidence.

Conclusions
Low carbon steel AISI 10 was subjected to severe plastic deformation using Equal Channel Angular Pressing process. The aim of this search was to explore the formation of ultrafine grain structure evolution in dependence of the initial steel structure modification. The analyses on microstructure results provided clear evidence that the modification of as-received microstructure and the deformation conditions applied were only partially effective in formation of homogeneous ultrafine grain structure of adequate volume fraction in the steel. Permanent banded microstructure containing cell and subgrain substructure dominated for applied straining and initial structure condition of steel.
Deformation conditions, dynamic recovery and polygonization process contributed to final structure refinement and/or to further modification of the refined ferrite microstructure. Permanent banded microstructure with cells and subgrains was dominated in deformed steel regardless the level of applied strain.

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