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Effect of initial treatment on the structure and mechanical properties of medium carbon steel subjected to ECAP

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Abstract

The present work deals with grain refinement of medium carbon steel AISI 1045 (0.45 wt.% C) having different initial ferrite-pearlite microstructure resulted from thermal and thermomechanical (TM) treatment. The purpose of prior TM steel processing was to refine grains of ferrite phase and to modify coarse lamellae pearlite structure. The final grain refinement of steel structure was then accomplished during warm Equal Channel Angular Pressing (ECAP) deformation at 400 °C. Employment of this processing route, in dependence of the applied effective strain ε_{ef}, resulted in extensive deformation of ferrite grains and cementite lamellae fragmentation. When applying higher shear stress, the mixed structure of subgrains and ultrafine grains was formed within ferrite phase, regardless the initial steel modification. In pearlite grains modification of cementite lamellae due to shearing, bending, twisting and breaking was found efficient as straining increased. Processes of dynamic polygonization and recrystallization in deformed structure also contributed to submicrocrystalline grains structure formation in intensively deformed structure. Comparing results of coarse lamellae cementite spheroidization it was then more efficient when prior TM treatment of steel was introduced. The tensile deformation results confirmed the strength increase, however, deformation behaviour and strain hardening, generally for different initial structural conditions of steel, showed diversity across deformed bars.

Key words: carbon steel, microstructure, dissolving, TM treatment, microstructure, SPD, ECAP, mechanical properties

1. Introduction

Microstructural refinement of steels is usually achieved by alloying or by thermomechanical treatment, due to modification the phase transformation process. Recently, advancement of severe plastic deformation (SPD) techniques provided another efficient access for grain refinement of metals and alloys. Bulk materials fabrication with ultrafine grains has attracted a great deal of attention over the past two decades because of the materials enhanced strength properties [1].

In last years, ultrafine grained materials have attracted considerable research interest because they tend to possess high strength without sacrificing toughness and ductility. The great effort has been exerted in a number of research programs attempting to achieve an average ferrite grain size of 1–2 μm in low carbon steels via simple thermomechanical means of treatment. The role of grain size refinement in improving strength and toughness is well known. Fine grained structures in steels usually have been conventionally reached by recrystallization during their thermomechanical (TM) treatment. A lot of works on the grain refinement of ferrite structure employing different TM processing routes have been developed in last years and some of the results on this topic are published in [2–4]. However, recent investigations have shown that even with high levels of strain applied during rolling process of steel, it was

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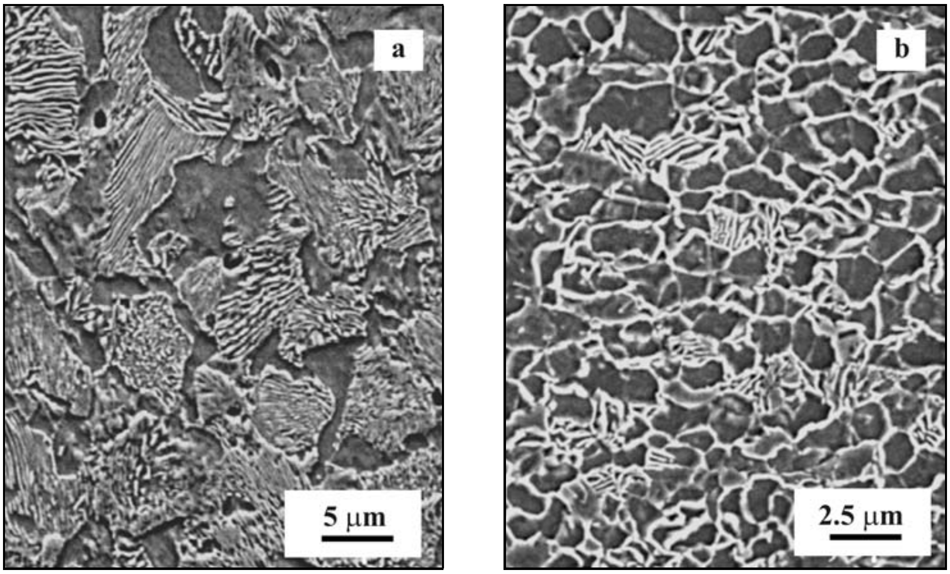

Fig. 1. Initial steel ferrite-pearlite structure resulting from: a) solution treatment, b) TM treatment (SEM mode).

difficult to refine the ferrite grain structures below 3 μm. It is supposed that high level of shear stress and high level of work piece undercooling can be the factors involved in effective ferrite grains refinement to 1 μm.

There have been attempts to develop TM processing to produce ultrafine grain microstructure through dynamic transformation during rolling process (SITR) [5]. It is supposed that high level of shear stress and high level of strip undercooling could be the factors involved in effective ferrite grains refinement to 1 μm. Great efforts have been exerted in a number of research programs attempting to achieve an average ferrite grain size of 1–2 μm in low carbon steels via simple TM process [6, 7].

As far as the issue of grain refinement of metallic materials is concerned, it is well known that technology of SPD of metallic materials is capable of producing ultrafine grained (UFG) materials with submicrometer or even nanometer grain size [8, 9]. Since ECAP and subsequently the other deformation technologies were introduced to refine structure of bulk metallic materials, many research groups devoted an effort not only to analyze the processing method details, but also investigated microstructural evolution and deformation to analyze ultrafine-grained materials.

Majority of early investigations with regard to SPD of metallic materials were focused on pure Al and Cu or their alloys. In last decade, continuously significant interest in structure refinement has shifted to the use of ECAP deformation in UFG processing of low carbon steels [10, 11]. This interest has been motivated in part by the fact that UFG low carbon steels can be used in many applications as structural materials, and in particular by ECAP capability to improve the strength of these steels without a need to change their chemical composition [12, 13]. It was then observed that the ultimate tensile strength (UTS) was increased with increased straining. On the other hand, the number of research works as to SPD of commercial medium carbon steels is still limited, probably because SPD processing is relatively difficult in steels with higher flow stress [14, 15]. To clarify the evolution of the deformation microstructures in medium carbon steels subjected to an effective strain ε_{ef} of 4 and higher, the warm or hot ECAP is recommended to provide the deformation required for the onset of dynamic recrystallization applying larger strain [16].

In the present study, at first the effect of initial structure modification of ferrite-pearlite microstructure of AISI 1045 steel (0.45 wt.% C) due to dissolving and TM treatment is described. Subsequently, the effect of initial structure modification in steel was correlated with development of ultrafine grain microstructure in condition of severe plastic deformation (ECAP) at increased temperature. Finally, resulting microstructure development was related to deformation behaviour and mechanical properties of steel.

2. Material and experimental procedure

To study an influence of initial structure characteristics of steel on the formation of ultrafine grain structure in condition of severe plastic deformation in medium carbon steels, different initial structural states of steel were prepared applying thermal and/or thermomechanical treatment. The experimental material for this study was commercial medium carbon steel grade AISI 1045 (Fe-0.45%C-0.23Si-0.63Mn-0.18Cr-0.43Al).

The mixture of ferrite and pearlite phases resulting from soaking treatment at 960 °C for 1 h and air cooling is presented in Fig. 1a. Large pearlite grains with size of ∼ 40 μm are lined by the finer ferrite grains (∼ 10 μm in size). In order to achieve prior ECAP deformation in steel, preliminary refined ferrite and pearlite structure, the TM processing took place at 900–700 °C with an aim to refine the coarse ferrite-pearlite structure. The resulting microstructure in the centre of deformed specimen is shown in Fig. 1b. The average grain size in deformed peg was then in the range of 5 μm.

For ECAP deformation experiment, the prior soaked and TM treated cylindrical bars of 9 mm in diameter and 50 mm in length were then machined. The warm ECAP pressing at 400 °C was performed and billets were subjected to $N = 4$, 5 and 6 passes, respectively. The ECAP channels had a round shape and the angle of channels intersection was $\phi = 120°$, yielding for each pass an effective strain $\varepsilon_{ef} = 0.67$. For ECAP experiment, the route Bc was chosen. The heating of bars before pressing was done inside pre-heated ECAP die until samples reached the pressing temperature of 400 °C.

The microstructural examination of thermally treated samples was carried out by scanning electron microscopy (SEM) and development of deformed microstructure in ECAP deformed bars was analyzed by transmission electron microscopy (TEM). Thin foils for TEM observation were sliced normal to the longitudinal axis of ECAPed billets.

Uniaxial tensile tests at room temperature were conducted using an Instron 5882 testing machine. Tensile specimens with gauge length of $l_0 = 20$ mm were tested at a constant crosshead displacement of 0.016 mm s^{-1} until failure. From received tensile data the engineering stress-strain curves were constructed for both initial structural states of steel.

Hardness measurement HV30 was carried out to get initial structural values of the steel, and then an evaluation of the ECAP straining effect in dependence on ε_{ef} strain was carried out. The hardness data were related to structural characteristics, mechanical properties, and to deformation behaviour of steel in the condition of mechanical tensile testing.

3. Experimental results and discussion

3.1. Initial steel microstructure characteristics

It is generally known that medium carbon steel, when thermally treated under annealing condition, consists of ferrite and lamellae pearlite constituents. Deformation behaviour and properties of carbon steel, having higher carbon content, then significantly depend on morphology, distribution and volume fraction of phases present in steel. In this work, two different procedures, specifically solutioning and/or thermomechanical treatment of steel, were carried out with an aim to prepare different initial ferrite-pearlite microstructure characteristics of steel, with regard to morphology and distribution of ferrite and pearlite constituents. Subsequently, the effect of structure modification was verified with regard to ultrafine grain structure formation in condition of severe plastic deformation (ECAP) and from an aspect of deformation behaviour modification of steel.

The steel microstructure resulting from dissolving treatment at 900 °C and air cooling was then the mixture of lamellae pearlite and equiaxed ferrite grains. The average size of pearlite grains was about 50 μm. The pearlite grains were lined by finer ferrite grains, Fig. 1a. The volume fraction of pearlite in structure was ∼ 80 vol.% and the rest was ferrite phase. The mean linear intercept size of larger and smaller ferrite grains is ∼ 2 and ∼ 5 μm, respectively.

In order to modify the coarse initial steel ferrite-pearlite structure the TM treatment of steel bars was conducted and resulting structure refining was then apparent, Fig. 1b. Two different morphologies of ferrite grains were present in microstructure. The first one, fine equiaxed grains, which resulted from the transformation of deformed austenite with size of ∼ 2 μm, and the second one, the elongated grains of already transformed ferrite, which was deformed when passing through intercritical $\alpha + \gamma$ region. These ferrite grains were then larger with size of about 5 μm. In the central part of specimens, the pearlite grains were comparable to that of ferrite grains and their distribution was uniform there. Some spheroidized cementite rods were found scattered along ferrite grains boundaries. Towards the specimens edge the size of pearlite colonies increased and microstructure heterogeneity, as regards pearlite grains size and their distribution, was increased.

3.2. Solution treatment and ECAP deformation

Initial microstructure of steel prior ECAP processing was modified applying solution treatment at 900 °C for 1 h and air cooling. Thermally treated steel bars were then deformed in an ECAP die. Performing ECAP pressing the individual steel bars were then experienced $N = 4$, 5 and 6 passes through the die. Finishing ECAP deformation, the corresponding effective strain in dependence on number of passes was $\varepsilon_{ef} =$ 2.7, 3.4 and 4, respectively, for individual samples. The microstructural characteristics of deformed ferrite and pearlite structures, experienced $N = 4$ and 6 ECAP passes, are presented in Fig. 2. Performing $N = 4$ passes ($\varepsilon_{ef} = 2.7$), the resulting deformed structure was found heterogeneous and diverse along deformed bar. The equiaxed ferrite grains, resulting from

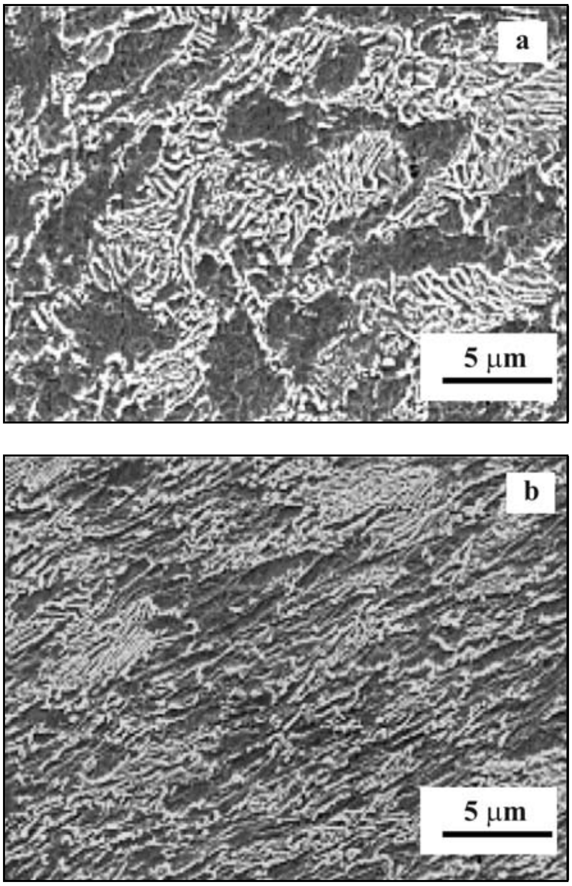

Fig. 2. ECAP deformed ferrite-pearlite microstructures experience different straining: a) $N = 4$ and b) $N = 6$ passes. Initial solution treatment (SEM mode).

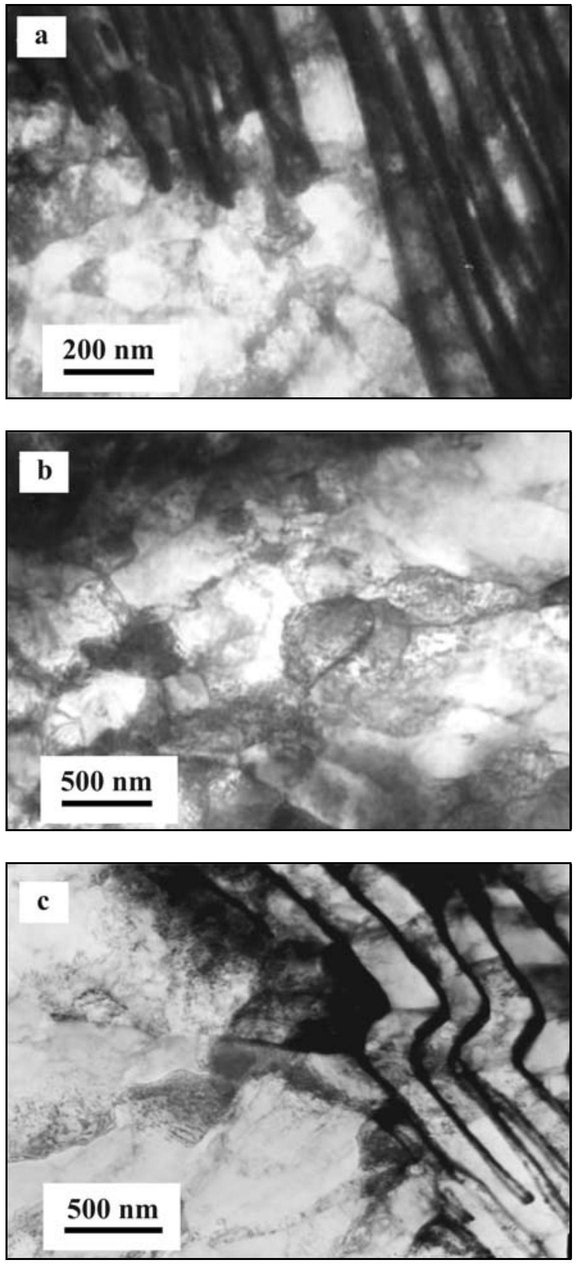

Fig. 3. ECAP deformed microstructures of steel experience different straining: a) $N = 4$, b) $N = 5$, c) $N = 6$ passes. Initial solution treatment (TEM mode).

solution treatment, were found only partly deformed. The cementite lamellae were curved, bowed and only slightly distorted, preserving lamellar morphology, as shown in Fig. 2a. Executing $N = 6$ passes, the ferrite and pearlite grains were substantially stretched in shear direction, as shown in Fig. 2b. More effective straining caused larger extent lamellae pearlite breakage, however, locally in some pearlite grains the partially modified lamellae morphology was still preserved.

The effect of straining on deformed microstructure formation was also investigated using TEM of thin foils on the plane parallel with billet longitudinal axis. The deformed ferrite and pearlite structures, which experienced different straining performing $N =$ 4, 5 and 6 passes, are presented in Fig. 3. Formation of ultrafine grain structure in initially coarse ferrite and pearlite grains was only of low efficiency when experiencing the lowest effective strain ($\varepsilon_{\text{ef}} = 2.7$, $N = 4$). Dense dislocation network and subgrain structure were preferentially formed within ferrite grains, Fig. 3a. The cementite lamellae morphology was predominant and in majority of grains preserved. The lamellar pearlite morphology and crushed cementite lamellae were also found in case when executing a larger effective straining was applied ($\varepsilon_{\text{ef}} = 3.4$, $N = 5$). Due to higher level of straining in formerly deformed ferrite grains, the new grains of submicron size appeared sporadically, Fig. 5. Fine grains of submicron size in deformed ferrite grains together with shear bands in cementite lamellae areas were found more frequently as strain was increased to $\varepsilon_{\text{ef}} = 4$, which corresponds to execution of $N = 6$ passes, Fig. 3c. As records show in pearlite grains the shear bands de-

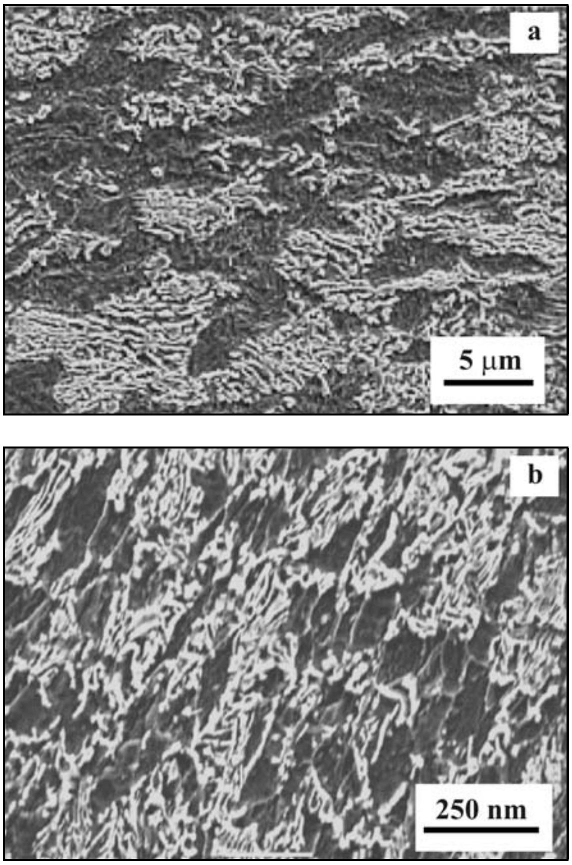

Fig. 4. Resulted ferrite-pearlite structure of prior TM treated steel experienced different ECAP straining: a) $N = 4$ and b) $N = 6$ passes (SEM mode).

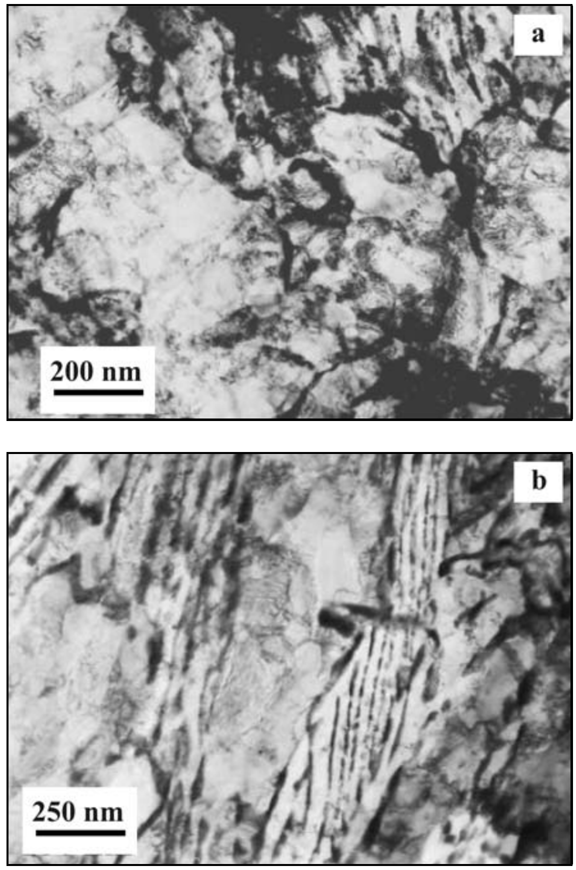

Fig. 5. ECAP deformed microstructure experienced different straining: a) $N = 4$ and b) $N = 6$ passes (TEM mode).

forming cementite lamellae were found locally as well.

3.3. Prior steel TM treatment and ECAP processing

In order to refine an initial ferrite-pearlite structure in medium carbon steel, a preliminary structure refinement was carried out applying thermomechanical processing. It was then expected that the execution of TM processing of steel prior ECAP deformation and support of spontaneous recrystallization process at higher temperature will result in more effective austenite grain refining and subsequently, the ferrite and pearlite structure characteristics in time of repetitive ECAP deformation process will be refined and modified.

When executing TM multiaxial step pressing of steel bar prior ECAP, it resulted in advance ferrite grains refinement and pearlite (cementite lamellae) morphology modification. Applying repetitive pressing deformation it also influenced, due to temperature gradient and strain distribution heterogeneity across deformed peg, the ferrite grain size heterogeneity and cementite lamellae morphology modification (lamellae fracturing). It was then expected that development and formation of ultrafine grain structure, when ECAP process was effective, would then be subsequently modified to some extent, due to preliminary initial structure modification resulting from prior TM steel treatment.

As structure results showed, formation of ultrafine grain structure was preferential in former ferrite grains, and the critical factor for structure refinement was the level of applied effective strain ε_{ef}. As concerns ferrite grains refinement and pearlite lamellar morphology modification, only small contribution from prior steel microstructure modification due to TM treatment execution was observed. The deformed microstructure, which resulted from different ECAP straining of steel, related to different number of passes through the die channel ($N = 4$ and $N = 6$ passes), is presented in Fig. 4a,b. The microstructure changes resulted from TM treatment applied prior ECAP provided only small contribution as to structure modification, especially lamellar pearlite morphology modification, and also on strengthening effect and deformation behaviour of experimental steel.

In order to characterize deformed microstructure

T a b l e 1. Mechanical properties of steel

Steel state	$R_p0.2$ (MPa)	R_m (MPa)	A (%)	Z (%)	HV30
Steel solution	402	726	18	21	253
TM treatment	574	748	26	49	265
Steel solution + ECAP N4	892	981	11.8	43	320
Steel solution + ECAP N5	900	992	10.6	34	328
Steel solution + ECAP N6	949	1023	8.5	21	379
TM treatment + ECAP N4	1087	1155	6.7	43	306
TM treatment + ECAP N5	1065	1127	9.1	47	310
TM treatment + ECAP N6	1042	1119	11.4	55	312

of steel resulting from different ECAP straining at 400 ℃ in details, the TEM micrographs are presented in Fig. 5a,b. After conduction ECAP deformation with channel angle of 120°, the deformed structure was found heterogeneous across the billet, regardless the strain applied, corresponding to $N = 4$ and $N = 6$ passes. The areas of severe deformation, where cementite fragmentation and formation of dislocation network in ferrite are evident, were found next to polygonized microstructure due to the deformation of extended ferrite grains. Investigating the deformation substructure, also a progress in cementite lamellae spheroidization due to increased temperature of deformation is apparent as well. The dislocation substructure in deformed ferrite grains was modified upon effective dynamic polygonization process. However, next to this refined grains sites, the low angle boundaries are still apparent in ferrite grains. Submicrocrystalline structure is formed within ferrite grains as well. As ECAP straining increases reaching $\varepsilon_{ef} = 4$, ($N = 6$), the progress in dynamic polygonization proceeded and formation of submicron size grains, having high angle boundaries, was observed in ferrite grains and also between fractured residues of cementite lamellae. This observation on substructure development indicates that the progress in formation of more UF grains was less effective due to insufficient straining of specimen resulting from six passes using ECAP die with an angle of 120°.

3.4. Mechanical properties of prior dissolved steel

Mechanical properties of experimental steel subjected to thermal and thermomechanical treatment prior SPD which were evaluated in condition of tensile deformation and by hardness measurement HV30 are summarized in Table 1. The tensile deformation records for initial structure states of steel modified by dissolution and TM treatment were carried out at room temperature and are shown in Fig. 6. In case of the initial dissolving of steel there is a distinctive period of work hardening resulting in quite large elongation to failure. In the same figure, the deformation curve corresponding to TM treated steel, which results in grains refinement and structure homogenization, shows slight work hardening course and results in shorter deformation course to failure. The records of deformation behaviour also confirmed that additional TM treatment slightly improved the strength level of steel, but modified deformation hardening behaviour due to refinement of ferrite grain size and cementite lamellae modification.

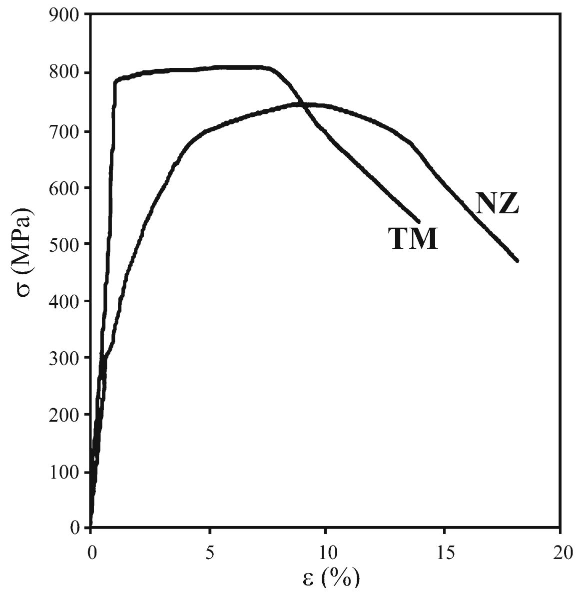

Fig. 6. Stress-strain dependences for steel experienced soaking (NZ) and TM treatment.

When to relate structural characteristics and mechanical properties of the steel having modified structural states, which resulted from applied prior dissolving and then applying additional TM treatment, the hardness values were in good conformity with recorded course of deformation behaviour of steel. The results showed the good agreement (Table 1) with the change of structural state of steel resulting from the applied thermal and thermomechanical treatment of medium carbon steel.

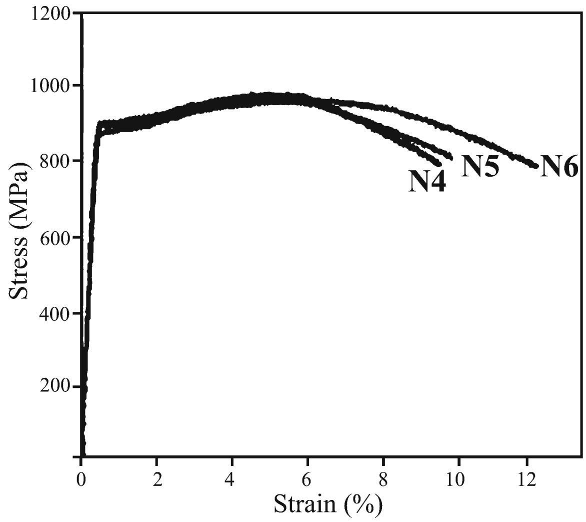

Fig. 7. Stress-strain dependences for steel experienced soaking and ECAP deformation exposed to different straining, $N = 4, 5, 6$ passes.

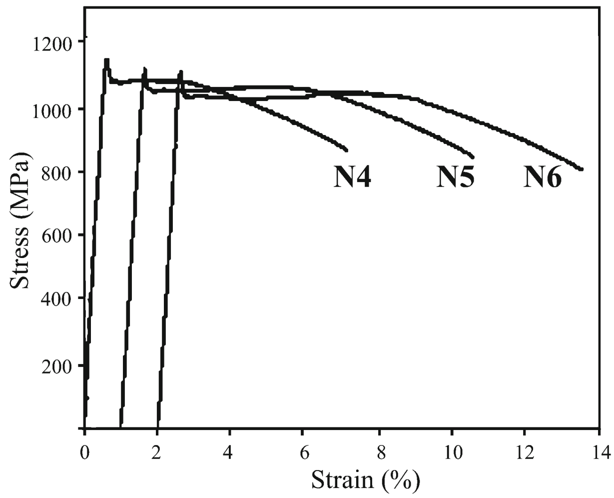

Fig. 8. Stress-strain dependences for steel experienced TM treatment prior ECAP deformation and exposed to different straining, $N = 4, 5, 6$ passes.

3.5. Mechanical properties of prior dissolved and ECAP processed steel

The mechanical properties of experimental steel subjected to thermal treatment (soaking) prior severe plastic deformation were evaluated by hardness measurement (HV30) and by tensile test, and are presented in Table 1, as well. Deformation behaviour of soaked and ECAP steel specimens is very similar for all three initial states of deformed specimens. Regardless the different ECAP straining (ε_{ef}) resulting from deformation condition there is only a small difference in effective stress reaching ε_{ef} value. The tensile deformation results received for all structure states of steel exposed to different ECAP straining ($N = 4$, 5 and 6 passes) carried out at room temperature are shown in Fig. 7. There is, after reaching the yield stress, a section of strengthening behaviour (hardening course) similar for all three deformation states, which is extending slightly as straining is increased. However, on the other side the strength values are of the same level for all specimens, regardless the different effective strain level introduced. This is probably incurred by quite large fraction of lamellar pearlite morphology preserved in structure and then by its pronounced modification due to lamellae breaking as effective straining ε_{ef} is increasing. However, generally contribution of this preserved lamellae and modification (breaking) of cementite lamellae characteristics with respect to straining behaviour seems to have an inexpressive effect, as regards strain and ductility modification of steel. The reason is probably due to cementite lamellae morphology preservation in strongly deformed structure, regardless the level of introduced straining (Fig. 2b). Considering ferrite grains modification and contribution in dependence on straining effect (deformation behaviour records), there was not observed difference in strengthening behaviour of steel samples as ε_{ef} was increasing due to increased number of deformation passes. The section of uniform deformation and strengthening effect (deformation course) are similar as regards stress value and straining for individual sample states. Also values of maximum stress, reached for different straining, are recorded at the same level. From the resulted deformation records it is then evident that no change in deformation behaviour of steel was recorded as straining of steel samples increased. One of the reason for such deformation behaviour can be then behind contribution from carbon solution strengthening.

3.6. Mechanical properties of prior TM processed and ECAP processed steel

To describe the deformation behaviour of the medium carbon steel, which was TM processed prior to the ECAP, the tensile test records are shown in Fig. 8 and stated in Table 1. For all subjected samples with different straining, the records of deformation behaviour are very similar. In deformation records after discontinuous yielding (sharp stress drop on deformation records reaching the yield stress), there is a region of "creep-like" deformation behaviour, where period of work hardening is not appearing on deformation records. The section is then extended as straining, i.e., the value of ε_{ef} increases. Due to this sharp drop of stress the ultimate tensile strength is lower than the "upper" yield stress. The sharp drop of plastic behaviour of steel is noticeably different from that appearing at deformation behaviour of the low carbon steel, where the effect of ageing (in carbon atmosphere) can modify the process of yielding. However, this phenomenon can be developed due to Cottrell interaction (carbon atoms – dislocations) as a result of cementite particle dissolution in ferrite during severe straining and carbon atoms saturation in lattice due severe straining in steel as well. The appearance of flat region on the deformation curve can be then attributed to a balance of strengthening effect, caused by newly formed and by increased portion of fine submicrocrystalline grains and on the other side by more effective progress of dynamic recovery and recrystallization. These processes, as microstructure results confirmed, actually could contribute and participate in structure transformation process and contributed to steel deformation behaviour.

4. Summary and conclusions

Microstructure evolution in time of warm ECAP was studied in medium carbon steel AISI 1045 with different initial microstructure due to application of the thermal and TM treatment prior to deformation process. The major experimental results are summarized as follows:

– The warm ECAP of coarse initially dissolved structure of experimental steel led to formation of deformed microstructure in dependence on the effective shear strain applied. The lamellae pearlite fragmentation was non-productive and resulted in heterogeneous distribution of fractured pieces of cementite lamellae in ferrite matrix ($N = 4$). However, as straining increased ($N = 5$ and 6), the more effective transformation of deformed structure to fine subgrained and ultrafine grained mixture in initial ferrite grains was successfully realized.

– Microstructural observation of ECAP processed medium carbon steel subjected to preliminary TM treatment did not show substantial structural changes, with respect to grain refinement, in comparison with steel that was subjected to initial soaking treatment only. The grains refining contribution was evident as regards the pearlite lamellae modification (breaking, cementite fragments dissolution) and in formation of ultrafine grains in large extent in ferrite when applying higher straining of $\varepsilon_{\mathrm{ef}} = 4$.

– Microstructure analyses results of medium carbon steel preliminary modified by TM treatment and subjected to ECAP did not show substantial structural changes with regard to grain refinement in comparison with steel that was preliminary treated in soaking condition. The contribution was evident as regards the pearlite lamellae modification and formation of ultrafine grains in large extent in severely deformed ferrite. Applying higher strain $\varepsilon_{\mathrm{ef}} = 4$ at increased temperature, the polygonized submicrocrystalline structure with high angle boundaries was formed in large extent. Formation of UF grain polygonized microstructure recovered the plastic deformation ability of steel to some extent, however, with indistinctive work hardening period.

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