SEVERE WARM PLASTIC DEFORMATION OF CARBON STEEL

JOZEF ZRNÍK[1,2]*, JAROSLAV DRNEK[1], ZBYŠEK NOVÝ[1],
SERGEJ V. DOBATKIN[3,4], ONDŘEJ STEJSKAL[5]

[1] *Comtes FHT, Lobezská E981, 326 00 Pilsen, Czech Republic*
[2] *Technical University of Košice, Park Komenského 11, 040 01 Košice, Slovak Republic*
[3] *Moscow Institute of Steel and Alloys, Leninskiy pr. 4, 11936 Moscow, Russia*
[4] *Baikov Institute of Metallurgy and Materials Science, Russian Academy of Science, Leninskiy pr. 49, 11999 Moscow, Russia*
[5] *University of West Bohemia, Universitní 22, 306 14 Pilsen, Czech Republic*

Received 2 May 2005, accepted 27 July 2005

By application of thermomechanically controlled rolling and accelerated cooling, the ferrite grain refinement is limited to levels of $\sim 5\ \mu$m in steels. The strain assisted or strain induced transformation could be considered for the refining process. The present work, likewise, deals with grain refinement of medium carbon steel containing 0.45 wt pct carbon. The structure refinement was conducted in two steps. Preliminary refinement has been achieved due to multistep open die forging process which provided total strain of 3–4. Uniform and fine recrystallized ferrite structure with grain size of the order of 2 μm and with nest-like pearlite colonies was obtained. The further grain refinement was accomplished during warm Equal Channel Angular Pressing (ECAP) at 400 °C of preliminary thermomechanically processed specimens. The steel was subjected to two pressing passes. Employment of this processing route resulted in extensively elongated ferrite grains which were sufficiently refined. The submicrometer order dynamically recrystallized ferrite grains enclosed by subgrains of low angle boundaries were formed within the former elongated ferrite grains. The aim of the investigation was to find an optimized relationship between mechanical properties and microstructure developed in steel.

Key words: carbon steel, thermomechanical treatment, severe warm deformation, structure, mechanical properties

---

*corresponding author, e-mail: jzrnik@comtesfht.cz

1. Introduction

In the last years, ultrafine grained materials have attracted considerable research interest because they tend to possess high strength without sacrificing toughness and ductility. The role of grain size refinement in improving both strength and toughness is well known. Fine grained structures have been conventionally obtained by recrystallization during thermomechanical treatment of steels. Many studies on the refinement of ferrite grains employing different processing techniques and routes have been developed in the last years [1, 2, 3]. Recent investigations have shown that even with high levels of strain applied during rolling process of steels, it is difficult to refine the ferrite grains below 3 μm.

One of the current approaches is to adjust the thermomechanical processing parameters to achieve transformation during deformation [4, 5]. There have been attempts to develop thermomechanical processings to produce ultrafine microstructure through dynamic transformation during rolling process (SITR) [6]. However, the mechanism of grain refinement remains unclear. It is supposed that high level of shear stress and high level of undercooling are factors involved in effective ferrite grain refinement to 1 μm. There are now a number of research programs attempting to develop processes to achieve an average ferrite grain size of 1–2 μm in low carbon steels via simple thermomechanical means [7, 8].

It has been already well known that severe plastic deformation (SPD) of metallic materials is capable of producing ultrafine grained (UFG) materials with submicrometer or nanometer grain size [9, 10]. Since ECAP was introduced in the literature as an innovative technology of manufacturing bulk UFG metallic materials, many research groups worldwide have devoted effort to discover not only the processing characteristics but also the microstructural and mechanical characteristics of ultrafined materials.

Early investigations using ECAP processing were very often focused on pure aluminium and copper or their alloys. Very recently, significant interest has shifted to the use of ECAP in processing of UFG low carbon steels [11, 12]. This interest has been motivated in part by the fact that UFG low carbon steels can be used in many applications as structural materials [13, 14], and in part by ECAP capability to improve the strength of steels without a need to change their chemical composition. It was observed that the ultimate tensile strength (UTS) increased with increased number of passes. On the other hand, the number of research works as to SPD of commercial medium carbon steels is still limited [15, 16], probably because systematic SPD processing is relatively difficult in steels with higher flow stresses. To clarify the evolution of the deformation microstructures in medium carbon steels subjected to an effective strain of at least 4 and higher the warm or hot ECAP was used to provide the deformation required for the onset of dynamic recrystallization under larger strain [17].

In the present study, the evolution of ultrafine ferrite-pearlite microstructure during thermomechanical processing and subsequent warm severe deformation imposed by ECAP of conventional medium carbon steel AISI 1045 type was investigated. Thermomechanical treatment conducted by multistep open die press forging and followed by the ECAP pressing was carried out at the temperature of 400 ℃. It is the purpose of this article to analyse the results that revealed the underlying relationship between microstructure and mechanical properties.

2. Materials and experimental procedure

The chemical composition of the commercial steel grade (in weight pct) is stated in Table 1. Experimental steel was received as hot rolled bars with ferrite--pearlite structure and grain size of several tens of micrometers as shown in Fig. 1a. The ferrite-pearlite structure represented the initial state for evaluation of material properties. By appropriate thermomechanical treatment, the refining of the fairly coarse structure is possible and more suitable combination of strength, toughness and ductility can be obtained without additional alloying.

Prior to ECAP pressing, the conventional thermomechanical processing has been designed to achieve preliminary refinement of the ferrite grains and break down the coarse grains of pearlite. Cylindrical specimens, with initial diameter of 18 mm and length of 40 mm, which were used for experiment were cut off from rods, machined to form of peg, and subjected to compressive deformation. The initial sample design is documented in Fig. 1c. The repetitive deformation has been performed continuously without reheating in the recrystallized, non-recrystallized and intercritical $\alpha+\gamma$ temperature region and was expected to result in structure modification. The specimens were soaked at temperature of 900 ℃ which corresponds to starting forming temperature of specimen. To develop high and uniform strain, the repeated axial compressions of specimens between flat dies of hydraulic press were executed. Among successive deformation reductions the specimen was rotated about its axis until final shape of specimen was obtained, Fig. 1d. Scanning electron micrograph of the microstructure in the centre of the specimen cross-section is shown in Fig. 1b. The average ferrite and pearlite grain size was measured at areas of various compressive strains from near the surface to the center of the specimen. The microstructure within the specimen was not perfectly homogeneous either in cross-section or along its length. The details of thermomechanical procedure are described in Table 2. Finish forging temperature was measured by means of an

Table 1. Chemical composition of the experimental steel

Element	C	Mn	Si	P	S	Cr	Al	N
[wt pct]	0.457	0.63	0.23	0.010	0.033	0.18	0.043	0.004

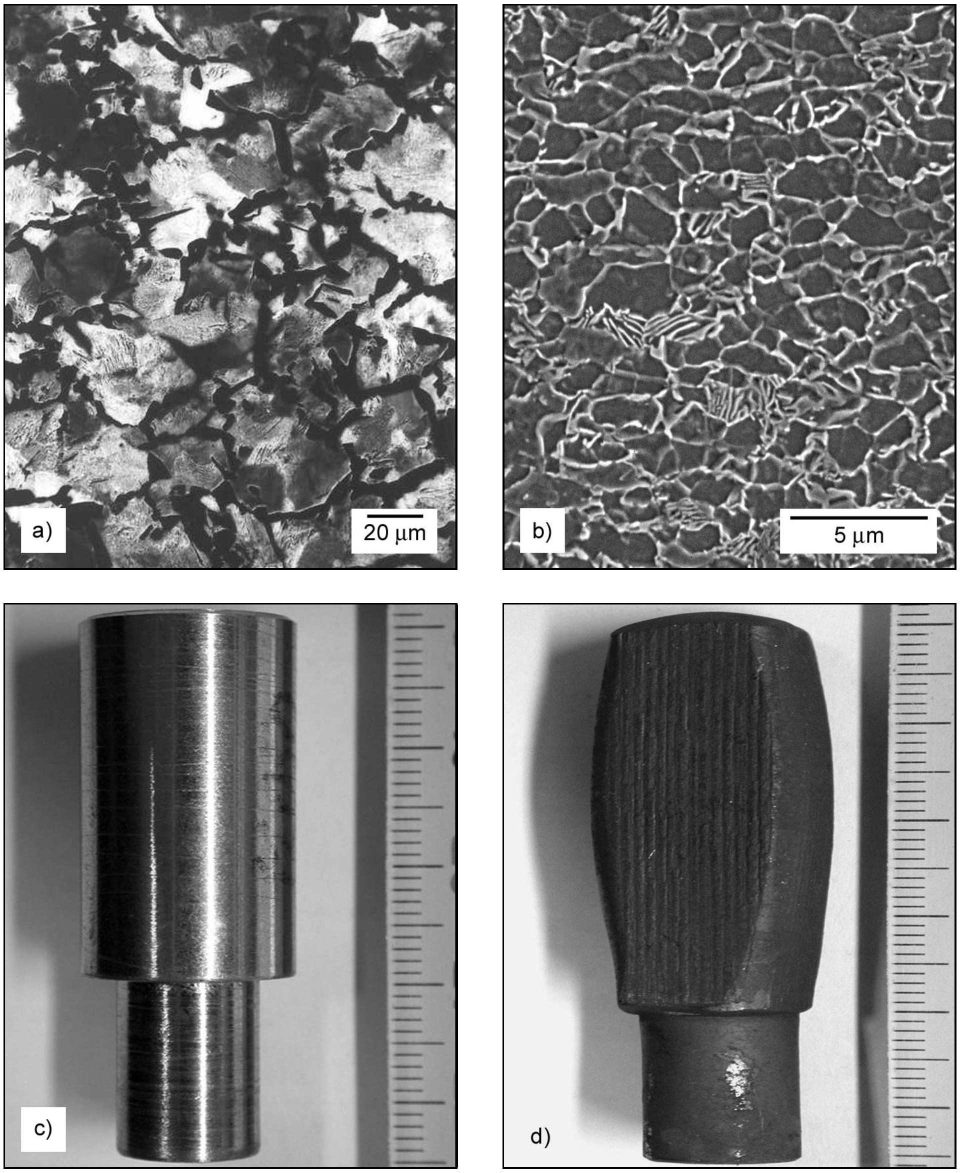

Fig. 1. SEM micrographs of ferrite-pearlite structure of the as-received (a) and thermomechanically processed medium carbon steel (b). Design of workpiece for TM processing (c) and shaped workpiece (d).

optical spectrometer. After finishing the last reduction the specimen temperature was about 700 ℃.

Table 2. Open die thermomechanical process parameters

Reduction step	1	2	3	4	5	6
Specimen rotation	0°	90°	45°	90°	45°	90°
Thickness upon rotation [mm]	9	11	8	10	7	11
Deformation temperature region	T_{rec}	T_{rec}	T_{nonerec}	T_{nonerec}	T_{nonerec}	$T_{(\alpha+\gamma)}$

Description of this sophisticated forging process has been attempted with aid of numerical simulation [18]. FEM software package has been used for analysis of stress, strain and temperature fields. According to the results obtained the effective strain (independent of loading cycle) in central area of forged specimen of 3 to 4 was computed. This region had a diameter of about 5 mm and well corresponded to uniform microstructure region in the specimen.

After machining, the cylindrical samples of 9 mm diameter and 50 mm in length from the thermomechanically treated specimens were subjected to severe plastic deformation using ECAP at temperature of 400 ℃. The ECAP was performed with channel intersection angle of 90 deg in two cycles ($N = 2$). The specimen heating for 300 s prior to pressing was done inside the pre-heated die until it reached the pressing temperature. A 250 tonne hydraulic press was employed for extrusion and pressing rate of 16 $\mathrm{mm \cdot s^{-1}}$ was used. The temperature of die was maintained in range of ± 1 ℃. During the pressing operation, the sample was rotated by 90 deg in the same direction between consecutive passes through the die. This procedure is generally termed processing route $\mathrm{B_c}$ and it was selected because it leads most rapidly to a homogeneous microstructure of equiaxed grains separated by high angle grain boundaries [19, 20].

The microstructure of processed samples was examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The sample was sliced normal to the longitudinal axis of ECAP pressed billets. A 3% Nital solution was used to etch the samples for SEM examination. The thin foils for TEM observation were prepared first by manual polishing and then by electrolytic polishing technique in mixture of perchloric acid (10 pct) and acetic acid (90 pct) at an applied potential of 12 V at room temperature. The SEM and TEM micrographs were obtained by using a JEOL JSM 6380 SEM operating at 10 kV and JEOL JEM 2000 FX TEM operating at 200 kV.

Tensile testing at ambient temperature was performed using Zwick universal testing machine equipped with Multisens extensometer. Tensile specimens were cut off from the as-pressed billets with gauge length of 20 mm and gauge sections lying parallel to the pressing direction of billet. Tensile tests at a constant cross-head velocity of 0.016 mm/s until failure were carried out. The engineering stress-strain curves were constructed.

3. Experimental results and discussion

3.1 Microstructure prior to ECAP

SEM micrographs of microstructure in hot rolled bars exhibit an equiaxed pearlite grains with maximum size of approximately 40 μm which are surrounded by ferrite network. The as-received steel consists of approximately 80 vol.% of pearlite (bright contrast) and the reminder is ferrite (dark contrast), Fig. 1a. The mean linear intercept size of larger and smaller ferrite grains was ~ 2 and ~ 5 μm, respectively.

After thermomechanical treatment of specimens, the structure refining is apparent. Two different morphologies of ferrite grains were found in microstructure. First, the fine equiaxed grains which resulted from the transformation of deformed austenite with size of ~ 2 μm, and second, the elongated grains of already transformed ferrite, which experienced deformation in intercritical $\alpha + \gamma$ region and have the size of about 5 μm. In the central part of specimen, the pearlite grain size was comparable to that of ferrite grains and their distribution was uniform. Some spheroidized cementite rods were accidentally scattered along ferrite grain boundaries already. Towards the specimen edges the size of pearlite colonies increases and microstructure inhomogeneity, as regards pearlite grain size and its distribution, increases as well, Fig. 2.

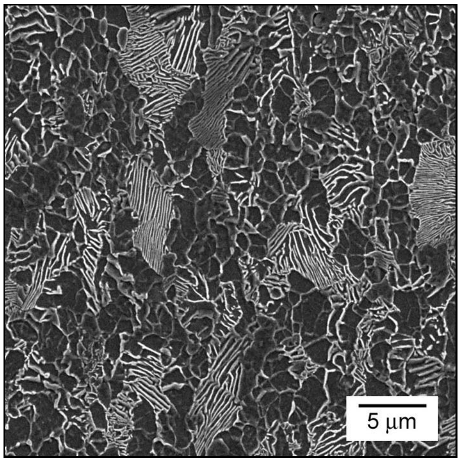

Fig. 2. SEM micrograph of structure in TM processed workpiece close to outer surface.

3.2 Microstructure characteristics of as-ECAPed specimens

The use of repetitive pressing in ECAP die provides an opportunity to develop different microstructures by rotating the samples between consecutive passes. Deformation characteristics for chosen processing route have been analysed in detail in sections normal to specimen axis. The SEM micrograph of the as-pressed steel provides evidence of effective ECAP straining, Fig. 3. It is clearly shown that after a single pass the initial equiaxed ferrite grains of the thermomechanically treated specimens became elongated on normal plane while pearlite grains, although partially crushed, preserve still lamellar morphology. The pearlite volume fraction does not seem to be changed. Only some fragmentation of cementite lamellae in pearlite

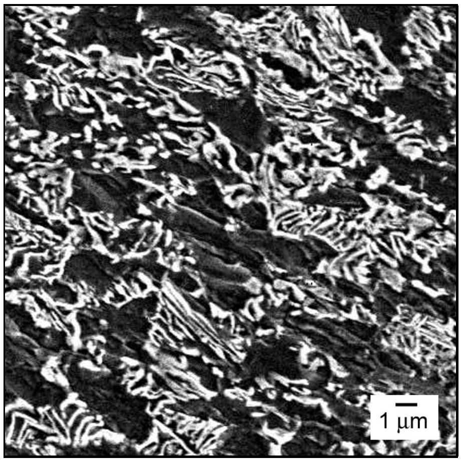

Fig. 3. SEM micrographs showing the pearlite morphology fabricated in ECAP pressing after one pass.

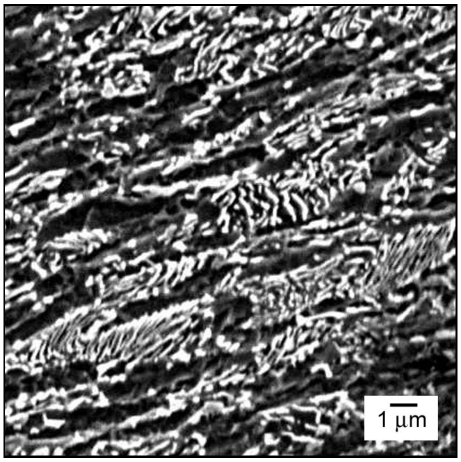

Fig. 4. SEM micrograph showing the effect of ECAP on pearlite deformation behaviour in specimen subjected to two passes.

Fig. 5. TEM micrograph of as-pressed microstructure of array of deformed ferrite consisting of elongated grains produced by one pass.

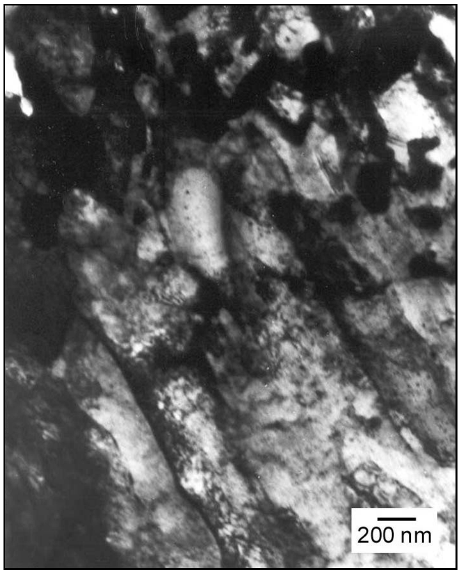

Fig. 6. TEM micrograph of dense dislocation network induced by one pass in elongated ferrite grains.

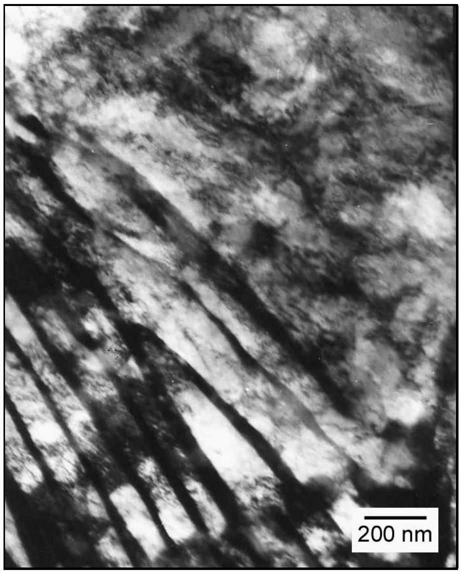

Fig. 7. TEM micrograph showing the lamellar morphology of pearlite grain dislocation density in pearlite-ferrite and ferrite grains after one pass.

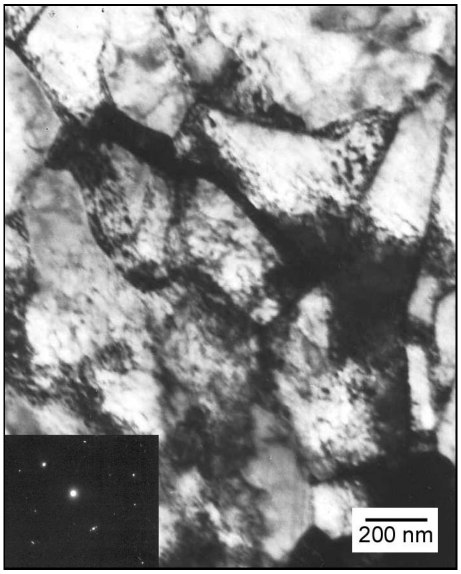

Fig. 8. TEM micrograph showing submicrocrystalline grains forming in ferrite during warm ECAP deformation.

colonies seemingly increases the volume fraction of ferrite in structure as compared with initial structure. On the other side, the elongated ferrite grains undergo severe shear deformation.

After the second pass the structure is shown in Fig. 4, and elongated grains of both phases are stretched in one direction with the length of pearlite grains and ferrite aggregates up to 10 μm. The former elongated grains are deformed more severely and are sheared into several shorter segments. The ferrite grains are sufficiently refined to render the grain boundaries indistinguishable under SEM analysis. This analysis also failed to observe the subgrains. The higher degree of deformation caused the majority of pearlite to disintegrate to larger extent after second pass but in some of the former larger grains the lamellar morphology of cementite is still preserved. The SEM analysis showed that ferrite-pearlite structure subjected only to two ECAP passes, and prior to thermomechanically processed, is uniformly deformed over the cross-section. Both ferrite aggregates and pearlite grains on the normal plane of specimen with respect to longitudinal axis of billet are oriented in one direction.

The microstructure of specimens subjected to warm ECAP was further investigated on their normal planes by TEM. Electron microscopy provided opportunity

Fig. 9. TEM micrograph showing the substructure in ECAPed specimen subjected to two passes.

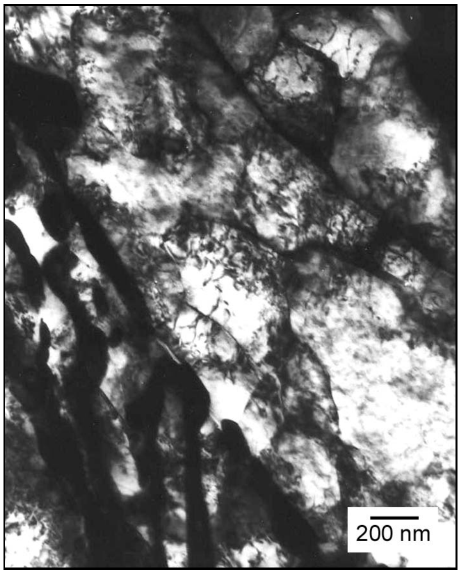

Fig. 10. TEM micrograph of subgrains appearance in deformed ferrite grain.

to analyse the changes in structure, which happened during ECAP pressing on submicron level. The microstructure after a single pass through the die is shown in Fig. 5. The ferrite microstructure mainly consisted of parallel bands of elongated grains having a width of ~ 0.4 μm. In the interior of these elongated grains, there is a well-developed dislocation structure or dislocation cells are present as documented in Fig. 6. In some regions the areas of granular structure are present as well what can display a section of ferrite bands in parallel alignment with specimen axis. Due to deformation heterogeneity across the transverse section of specimen the structure diversity was observed. Colonies of lamellar pearlite survived in structure, as shown in Fig. 7, together with areas of crushed cementite particles (Fig. 6), which are often present in structure. TEM analysis also revealed the fact that probably in more heavily deformed bands of ferrite regions the formation of submicrocrystalline structure occurred there. Figure 8 shows a TEM micrograph and corresponding selected area diffraction (SAD) pattern of submicrocrystalline grains. Both equiaxed and rather elongated grains with high angle grain boundaries are present there. Submicrocrystalline grains are formed as well in ferrite structure in areas of crushed cementite. The size of these submicrocrystalline grains varies in range of 0.2–0.5 μm.

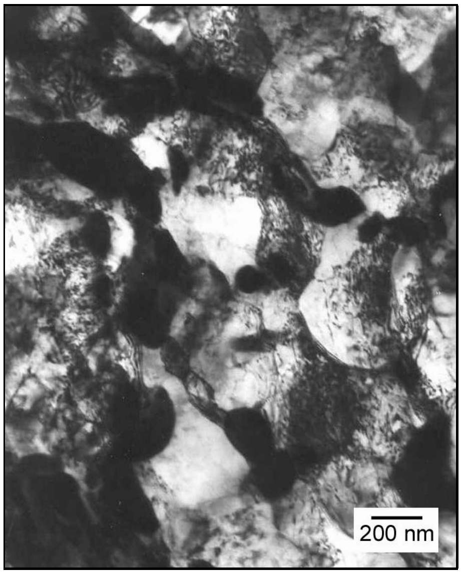

Fig. 11. TEM micrograph of subcrystalline grains developed during ECAP second pass in area of disintegrated cementite.

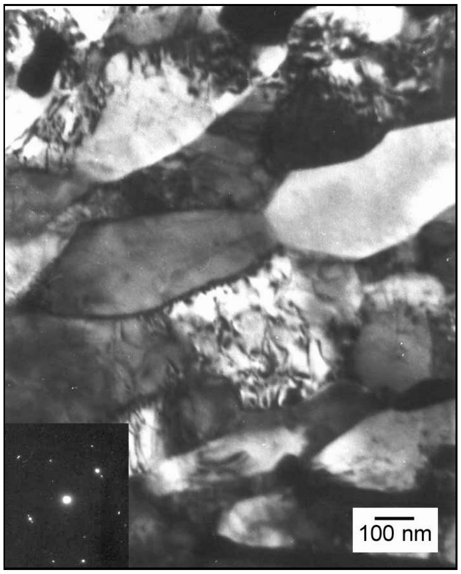

Fig. 12. TEM micrograph of submicrocrystalline recrystallized ferrite structure.

Second pass through the die, following a rotation by 90 deg between passes, leads to microstructure presented in Fig. 9. Substructure characteristics depend on the local position of searching. In elongated ferrite grains partly polygonized substructure can be observed in Fig. 10 where individual low angle boundaries separate the subgrains. The discernible dislocations inside subgrains are forming the dislocation network. These subgrains can act as nuclei at formation of submicrocrystalline structure. The formation of more advanced equiaxed grains can be seen in Fig. 11 with distinction that segments of boundaries display the characteristic fringe contrast. This fringe contrast can be accepted as a fact that in this area submicrocrystalline structure is already present. Average size of equiaxed grains is $\sim 0.4\ \mu\text{m}$ and they have been found in areas where fragmentation and spheroidization of cementite is observed. It can be proposed that prior thermomechanical grain refinement followed by warm ECAP pressing resulted in local high straining where onset of dynamic recrystallization must have contributed to forming of nanocrystalline structure already in time of second ECAP pass. The presence of polygonal fully recrystallized grains in structure, as can be seen in Fig. 12, is real evidence of this process. However, the residual dislocation segments are present within recrystallized grains.

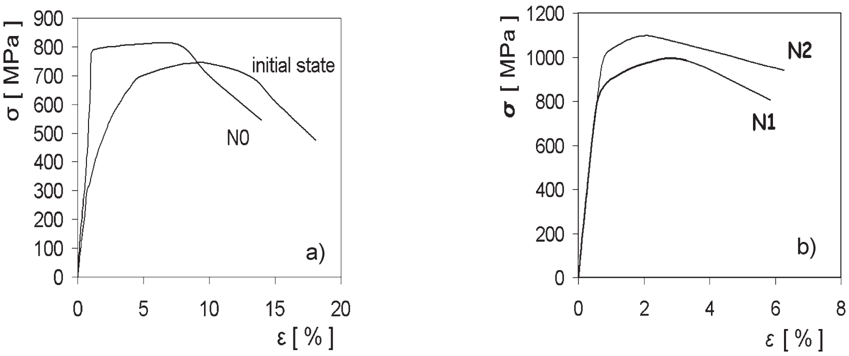

Fig. 13. Tensile properties in engineering stress-strain curves at ambient temperature: (a) initial state; N0 – TM processed steel; (b) ECAP processed specimens, N1 – one pass; N2 – two passes.

3.3 Tensile properties

The tensile behaviour of all experimental material was evaluated at room temperature and results in form of engineering stress as a function of engineering strain dependence are documented in Fig. 13. The records of tensile behaviour of as-received steel and specimens thermomechanically processed are presented in Fig. 13a. The proof stress corresponding to initial coarse grained ferrite-pearlite structure at strain of 0.2 % is 398 MPa. There is an extensive region for work hardening after yielding and quite large elongation to failure.

The plastic behaviour of the thermomechanically processed specimens is particularly different as presented in the same Fig. 13a. From strain-stress curve it is clearly seen that yield stress is more than twice higher as compared to initial state, reaching the value of 792 MPa. Larger uniform elongation to failure of these specimens with higher strength is attributed to a presence of very fine ferrite grains and modified pearlite structure, which resulted from thermomechanical processing. The received structure conditions are available for uniform deformation prior to specimen necking. On the other side, nearly micron size ferrite grains most probably control the strengthening process and due to negligible strengthening the ultimate strength is only a bit higher than yield stress, and reaches the value of 805 MPa.

In order to clarify the effect of grain refinement on strength, the tensile properties of warm ECAPed steel are examined, as shown in Fig. 13b. There is a significant effect of extrusion on the strength. The plastic behaviour of the ECAPed steel is particularly different from those of the as-received and thermomechanically processed specimens. Already after one pass, the ECAP specimens exhibit considerable increase of tensile strength than that of thermomechanically processed specimens. The ultimate tensile strength increases to over 985 MPa and ECAP specimen exhibits period of strain hardening and then a continuous drop in the stress-strain curve with accompanying necking.

After two passes, the ultimate tensile strength increases to 1090 MPa. In plastic region there is observed also a short section of strain hardening prior continuous geometrical softening sets in. It is worth noting that presence of a short strengthening period can be attributed to existence of submicrocrystalline grains with high angle grain boundaries which were observed inside ferrite areas (Fig. 12), and which are most probably the result of dynamic recovery and/or dynamic recrystallization actually running in the most severely deformed ferrite region.

4. Conlusions

1. Thermomechanical processing of as-received steel carried out prior to ECAP successfully refined the ferrite and pearlite grains. Intensive austenite straining and its successive transformation in intercritical temperature region resulted in two different morphologies of ferrite grains and nest-like pearlite grains. The pearlite structure heterogeneity with respect to size of pearlite colonies was observed towards the edge of deformed workpiece.

2. Execute one ECAP cycle at increased temperature, the ferrite grains became elongated and underwent severe deformation. Dense dislocation network and dislocation cells regularly appear across normal section of specimen. In some areas of severely deformed ferrite equiaxed submicrocrystalline grains were found. SAD analysis indicated the high angle boundaries separated the grains and they are products of dynamic recrystallization process. In ferrite within survived lamellar cementite high dislocation density is present.

3. Upon the second warm ECAP pass deformation, heterogeneity within the cross section was still apparent, however the ferrite grain banding was more advanced. Microstructural analysis revealed the appearance of refined microcrystalline grains in elongated ferrite and in areas with crushed pearlite. The subgrained structure present in banded ferrite grains indicates that grain refinement after second pass may result from mechanical fragmentation due to severe straining and parallel refinement mechanism associated with dynamic recrystallization process. After second warm pass, well developed submicrocrystalline equiaxed structure was present, regardless that some pearlite grains still possess lamellar structure.

4. The tensile behaviour of ECAPed specimens is noticeably different from that of thermomechanically processed steel. Fine grained structure prior to ECAP and ECAP itself contributed significantly to yield stress and ultimate tensile stress. The strain hardening before continuous softening can be attributed to process of dynamic recrystallization which yields subcrystalline structure and influences deformation behaviour of both ECAPed specimens.

Acknowledgements

This work was supported by the Ministry of Education, Youth and Sports of the Czech Republic in frame of Research Proposal the contract No. MSM 2631691901.

REFERENCES

[1] SMIRNOVA, N. A.—LEVIT, V. I.—KUZNETSOV, V. P.—DEGTARYEV, M. V.: Fizika Metalov i Metalov., *62*, 1968, p. 566.
[2] VALIEV, R. Z.—KORZNIKOV, A. V.—MULYUKOV, R. R.: Mater. Sci. Eng., *A 168*, 1993, p. 141.
[3] SEGAL, V. M.—KORZNIKOV, A. V.—KOPYLOV, V. I.: Process of plastic structure formation in metals. Science and Engineering Publ. House, Minsk, Belorus 1994 (in Russian).
[4] DE HODGSON, P.—HICKSON, M. R.—GIBBS, R. K.: Mater. Sci. Forum, *63–72*, 1998, p. 284.
[5] SHANG, C.—YANG, S.—YUAN, Y.—HE, X.: In: HSLA Steels' 2000. Eds.: Liu, G., Wang, F. Beijing, Metallurgical Industry Press 2000, p. 304.
[6] BELADI, H.—KELLY, G. L.—SHOHOUHI, A.—DE HODGSON, P.: Mater. Sci. and Eng., *A 371*, 2004, p. 374.
[7] YANG, Z. M.—ZHAO, Z.—WANG, R. Z.—CHENG, Y. M.: Acta Metall. Sinica, *8*, 2000, p. 818.
[8] HURLEY, P. J.—MUDDLE, B. C.—DE HODGSON, P.: Materials Science & Technology, *16*, 2000, p. 1376.
[9] SEGAL, V. M.—REZNIKOV, V. I.—DROBYSHEVSKIY, A. E.—KOPYLOV, V. I.: Russ. Metall., *1*, 1981, p. 99.
[10] VALIEV, R. Z.—KORZNIKOV, A. V.—MULYUKOV, R. R.: Mater. Sci. Eng., *A 168*, 1993, p. 141.
[11] SHIN, D. H.—KIM, I.—KIM, J.—PARK, K.-T.: Acta Mater., *48,* 2000, p. 2247.
[12] PARK, K.-T.—KIM, Y. S.—SHIN, D. H.: Metall. and Mat. Trans., *32A*, 2001, p. 2373.
[13] DOBATKIN, S. V.: In: Investigation and Application of Severe Plastic Deformation. Eds.: Lowe, T. C., Valiev, R. Z. Dodrecht, Kluwer 2000, p. 13.
[14] FUKUDA, Y.—OHISHI, Z.—HORITA, Z.—LANGDON, T. G.: Acta Mater. Trans., *50*, 2002, p. 1359.
[15] VALIEV, R. Z.—IVANISENKO, Y. V.—RAUCH, E. F.—BAUDELET, B.: Acta Mater., *44,* 1996, p. 4705.
[16] TSUJI, N.—ITO, Y.—SAITO, Y.—MINAMINO, Y.: Scripta Mater., *47,* 2002, p. 893.
[17] SASTRY, S. M. L.—DOBATKIN, S. V.—SIDOROVA, V.: Russian Metalls (Metally), *2*, 2004, p. 129.
[18] DRNEK, J.—NOVÝ, Z.—FIŠER, P.—ŠAŠKOVÁ, J.—HORŇÁK, P.: In: Forming 2004. Eds.: Polák, K., Schindler, I., Hadasik, E. STU Bratislava 2004, p. 49.
[19] FURAKAWA, M.—IWAHASHI, Y.—HORITA, Z.—NEMOTO, M.—LANGDON, T. G.: Mater. Sci. Eng., *A 257*, 1998, p. 328.
[20] OH-SHI, K.—HORITA, Z.—FURAKAWA, M.—NEMOTO, M.—LANGDON, T. G.: Metall. Mater. Trans., *A 29*, 1998, p. 2011.