Structure and Properties of Materials after Pressing by the ECAP

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Abstract. The article presents results of investigation of structure and properties of low-carbon steel grade P2-04BCh after application of Equal Channel Angular Pressing (ECAP) at the temperature of approx. 290°C. The ECAP method leads to significant improvement of strength of investigated material. Investigation of structure was made by combination of TEM and FEG SEM together with EBSD. It was proven that the ECAP method enables obtaining of ultra fine-grained ferritic structure formed by re-crystallised grains with very low dislocation density and a small fraction of spheroidised carbides, which occurred usually at the ferritic grain boundaries. It was established with use of the EBSD technique that after 8 passes through the ECAP die the sub-grains with misorientation angles smaller than 10° formed less than 20% of the final structure. Average size of ferrite grains with high-angle boundaries after 8 passes was approx. 0.32μm.

Introduction

Enhancement of strength properties of polycrystalline metallic materials with preservation of sufficient toughness can be achieved by refining of grains [1, 2]. Dependence between the grain size and the level of yield strength is described by the Petch–Hall relation :

$$\sigma_y = \sigma_0 + kd^{-1/2} \tag{1}$$

where σ_y is flow stress, σ_0 and k are constants, d is grain size. This relation can be used in extensive interval of grain sizes, up to several dozens of nanometres [3]. Search of possibilities of efficient refining of structure of technical materials lead to important modification of technology of thermo-mechanical treatment, which enable obtaining grain size at the level of several micrometres. The most efficient processes are the following: deformation induced ferritic transformation, dynamic re-crystallisation of austenite during hot deformation with subsequent $\gamma \rightarrow \alpha$ transformation, hot rolling at inter-critical interval of temperatures and dynamic re-crystallisation of ferrite after large hot deformation [3].

Further refining of grain size requires, however, application of extreme value of plastic deformation of material. During last two decennia many methods were developed that enable achievement of severe plastic deformation. Important place among them holds the equal channel angular pressing (ECAP) [4-6]. Principle of this method consists in severe deformation of massive samples realised by shear without change of cross section. The sample is pressed through a die, in which two channels intersect, forming an angle usually of 90°. Pressing is made either at room or at increased temperature. Equivalent deformation can achieve the value of 10 or even higher. The most critical for development of microstructure and resulting properties of samples is above all number of passes and selection of deformation route (manner of turning of the sample after each pass). It was established from the analysis of shear characteristics at various deformation routes that turning of the sample by 90° was optimal. Many works, dealing with optimisation of the laboratory ECAP

equipment, were published. Promising modifications for production of ultra fine-grained massive semi-products in industrial practice have appeared [3,4,6,7].

The ECAP method makes it possible to obtain the grain size of several hundreds of nanometres [8-11]. Materials with sub-micron size of sub-grains/grains (d=0.1-1 μm) are usually classified as ultra fine-grained materials [3]. The ECAP method was so far unsuccessful at attempts of obtaining nanometric materials, i.e. materials with grain size under 0.1μm. Characterisation of the fraction of sub-grains formed by recovery and grains separated by high-angle boundaries, which are formed by re-crystallisation [11], is very important for understanding the mechanisms of materials structure evolution at application of methods of extreme plastic deformation. Definition of difference between sub-grains and grains is not rigid. The values of 10-15° [1, 2] are usually given as a critical misorientation angle. It is known that grains separated by high-angle boundaries have generally much more important influence on the level of mechanical properties than sub-grains divided by low-angle boundaries [2]. In the area of grain size under approx. 0.3μm the classical mechanism of plastic deformation by dislocation sliding is replaced by other mechanisms. The most important causes of this phenomenon comprise increasing surface of grain boundaries per unit of volume of material, decrease of dislocation density inside the grains with grain size under 0.1μm, and localisation of deformation into shear bands. Important problems connected with development of ultra fine-grained materials include in the first place lower level of plasticity, non-homogeneity of structure across cross section of the pressed blanks and thermal stability of ultra fine-grained structure at higher temperatures [3].

Majority of the works published until now dealt with application of the ECAP method on pure metals, while much less attention was given to investigation of commercial steels [10]. Our article summarises the results obtained at investigation of severe plastic deformation by the ECAP method on strength characteristics and structure of a low-carbon steel P2-04BCh. Detailed investigation of structure evolution was made not only with use of Transmission Electron Microscopy (TEM), but also by Scanning Electron Microscopy (SEM) in combination with Electron Backscattered Diffraction (EBSD) [12], which enables characterisation of misorientation angles of individual crystallites on the surface of metallographic sections. If the Field Emission Gun (FEG) is used, it is possible to obtain at present the spatial resolution of approx. 0.1μm.

Experimental material and technique

Investigation was made with use of a commercial low-carbon steel grade P2-04BCh. Table 1 gives its chemical composition.

Table 1 Chemical composition of the steel P2-04BCh, weight %

C	Mn	Si	Cr	Mo	Ti	B
0.034	0.67	0.23	0.10	0.017	0.001	0.002

The supplied material was in the state after free cooling from the rolling temperature. Cylindrical samples of dimensions ϕ12x60mm were manufactured from this initial material. The angle between the channels of the used ECAP die was 105°. This design made it possible to reduce deformation resistance and it ensured good filling of the die edges [13]. The samples were before the pressing re-heated in the furnace to the temperature of approx. 320°C, temperature of the ECAP die was approx. 290°C. Deformation route B_c was applied (turning of the sample after each pass by 90° in the same direction), moreover the front end of the sample was replaced by the rear end of the sample. This deformation route is considered generally as the quickest manner of achievement of homogenous

structure formed by equiaxed grains [3]. The maximum number of realised passes through the ECAP die was 16.

Samples for tensile test were prepared from individual deformed samples. This test was made at room temperature. For the purposes of structural analysis sections were made perpendicularly to the longitudinal axis of the samples after 4 (equivalent deformation ε=3.5) and 8 (ε=7.1) passes through the ECAP die. Final polishing of the samples for the SEM analysis was made with use of colloidal solution of SiO_2 with granularity 0.05μm. Crystal orientation maps (COM), study on misorientation angles of individual sub-grains/grains and statistic evaluation of grain size was made by the apparatus Sirion 200 FEG SEM equipped with the HKL Technology Channel 5 EBSD system. Thin foils for the TEM were prepared perpendicularly to the longitudinal axis of the samples from the approximately ¼ of the diameter of initial samples. The foils were prepared by electrolytic polishing in the solution containing 5% of $HClO_4$ and 95% of CH_3COOH at the room temperature and voltage of 60V. The TEM investigation was performed on the microscope JEOL JEM 2100 equipped with the PGT EDX analyser.

Experimental results and discussion

Microstructure and mechanical properties of steel in the initial state:
Results of the tensile test of the supplied material at the room temperature are given in the Table 2.

Table 2 Results of tensile test of the steel P2-04BCh

Yield stress [MPa]	Tensile strength [MPa]	Elongation [%]	Reduction of area [%]
281	355	31.5	72.5

Microstructure of steel was formed by equiaxed grains of ferrite, which were discontinuously decorated by carbidic particles, see the Fig. 1. Small islands of decomposed ferritic-carbidic component were present in a very small quantity at the boundaries of ferritic grains. Small precipitates were observed also inside ferritic grains. Average size of ferritic grains was approx. 35μm.

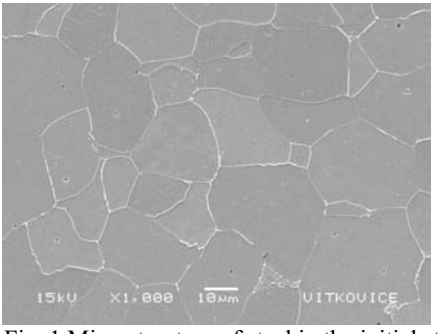

Fig. 1 Microstructure of steel in the initial state

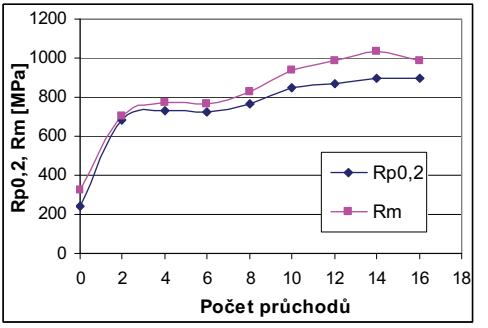

Fig. 2 Results of tensile tests of material deformed in the ECAP die

Microstructure and mechanical properties of steel after application of ECAP

Severe plastic deformation of the investigated steel in the ECAP die lead to significant enhancement of strength properties. Obtained results are shown in the Figure 2. The biggest increase in strength properties was found after the first two passes. Next passes resulted only in very gradual

enhancement of strength parameters. After 16 passes even slight decrease of strength properties was already observed.

Microstructure of steel after 4 ECAP passes was non-homogeneous, original ferritic grains were largely deformed. Deformed ferritic grains forming elongated bands are clearly visible in the Figure 3. Substructure was present inside ferritic grains, distribution of carbidic particles remained unchanged. Slip lines were observed in some ferritic grains, see the arrow in the Figure 4.

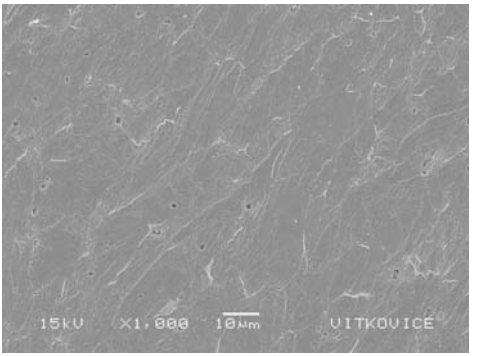

Fig. 3 Microstructure after 4 ECAP passes

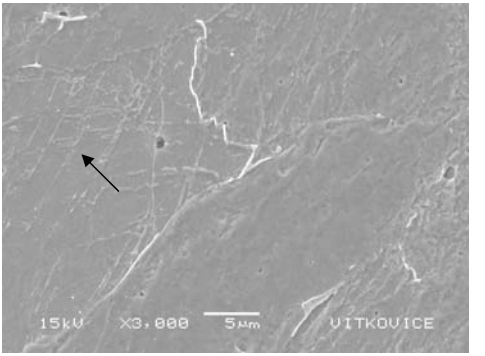

Fig. 4 Microstructure after 4 ECAP passes

The TEM analysis proved that original equiaxed ferritic grains were replaced by stretched sub-grains/grains of variable size. Sub-grains/grains formed usually elongated parallel bands, see the Figure 5. Pronounced local differences of diffraction contrast indicated that misorientation angles between individual sub-grains/grains were highly variable. Density of dislocations inside individual stretched ferritic sub-grains/grains was usually comparatively high, or arrangement of dislocations into dislocation walls was observed. Small grains with well defined boundaries and low density of dislocations were observed locally. It can be therefore assumed that formation of fine-grained structure was influenced not only by mechanisms of fragmentation of deformed grains, but also by re-crystallisation processes.

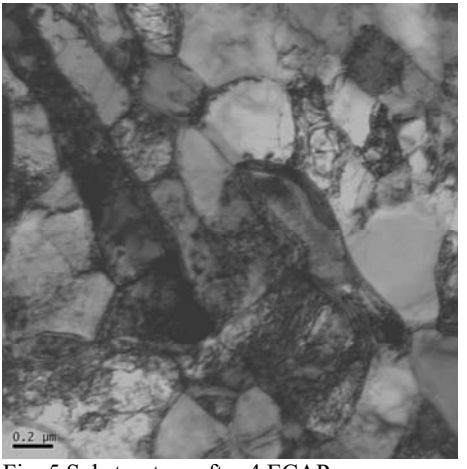

Fig. 5 Substructure after 4 ECAP passes

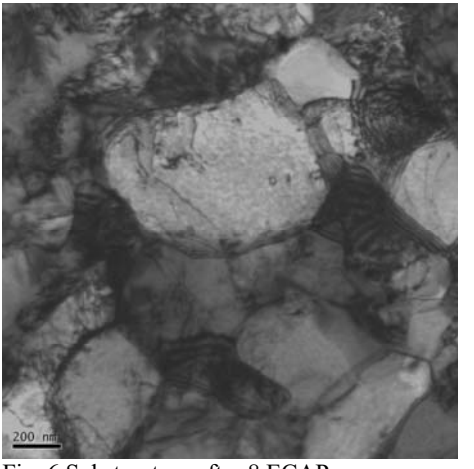

Fig. 6 Substructure after 8 ECAP passes

It was established at the TEM analysis of the sample after 8 ECAP passes, that increase of number of passes resulted in improvement of uniformity and fineness of grains of resulting structure. This is result of synergic effect of the applied temperature of pressing, total real deformation and latent heat generated by the severe plastic deformation. Diffraction contrast of some adjacent sub-grains/grains was very similar, while in other cases it was very different. This indicates that structure is formed by a mixture of sub-grains with very low misorientation angles and also grains separated by high-angle boundaries. Density of dislocations inside sub-grains/grains was mostly very low, boundaries of sub-grains/grains were well defined. Majority of sub-grains/grains was equiaxed, however, in some areas significantly stretched sub-grains/grains were observed. Results of the TEM analysis confirm, that re-crystallisation processes influenced significantly formation of the ultra fine-grained ferritic structure.

Size of some sub-grains/grains was smaller than 0.1μm, size of other ones was bigger than 0.5μm. Globular particles of carbides were present at boundaries of some ferritic grains. It can be assumed that these carbidic particles have a positive effect on stabilisation of ultra fine-grained ferritic structure against coarsening. Typical examples of sub-structure of the sample after 8 ECAP passes are shown in the Figures 6 - 8.

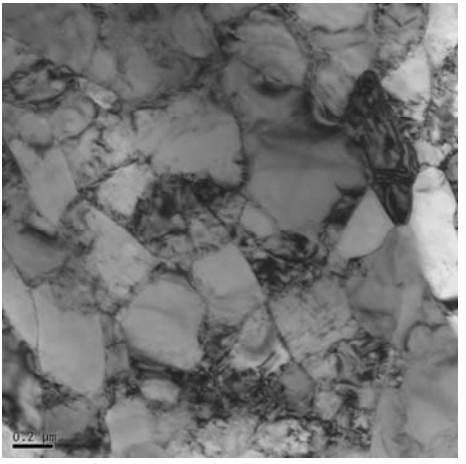

Fig. 7 Substructure after 8 ECAP passes

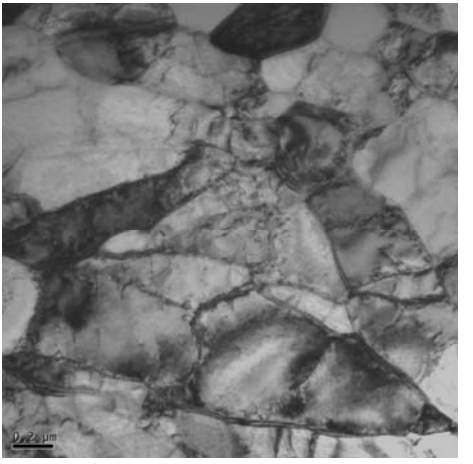

Fig. 8 Substructure after 8 ECAP passes

Understanding of mechanism of formation of ferritic grains in deformed samples, as well as objective assessment of the size of grains with high-angle boundaries, requires information about misorientation angles of individual sub-grains/grains. Ideal experimental technique for obtaining these data is at present the FEG SEM in combination with the EBSD [12]. This technique enables determination of crystallographic orientation (Miller indices of the direction perpendicular to the sample surface) at any place on the sample surface on the basis of the Kikuchi lines, formed by diffraction of originally non-coherently scattered electrons right below the surface of heavily inclined sample. Mapping of crystallographic orientation of the sample surface can be done with a minimum step of 0.1μm.

EBSD results obtained on the sample after 8 ECAP passes were processed in the form of crystal orientation maps (COM), where the areas of various orientation on the sample surface are discriminated by different colouring. The obtained results were further processed by computer as follows:

- in the areas, where the misorientation angle of adjacent pixels was greater than 2°, the boundaries were plotted. In this manner boundaries of sub-grains were visualised, as well as boundaries of grains separated by high-angle boundaries.
- in order to differentiate between the sub-grains and grains the boundaries of grains were plotted only in the areas, where misorientation of adjacent pixels exceeded 10°.
- grain boundaries were plotted in the areas, where misorientation of adjacent pixels exceeded 20°.

Map of crystal orientations (COM), shown in the Figure 9, documents a large quantity of differently oriented sub-grains/grains in the investigated area. The Figure 10 shows the boundaries generated in the areas, where the misorientation angle between the adjacent pixels was at least 2°. If we define sub-grains as areas with the maximum misorientation angle of 10°, it is possible to discern the sub-grains from the grains with high-angle boundaries by comparing the Figures 10 and 11. It was found that in some cases the misorientation angle of part of perimeter of one grain corresponded to a sub-grain, and the rest of the perimeter was a boundary with high-angle misorientation. The Figure 12 documents a distribution of grains in the investigated area with the misorientation angles exceeding 20°.

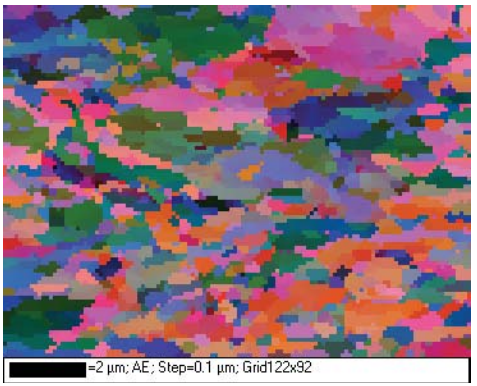

Fig. 9 Crystal orientation map, 8 ECAP passes

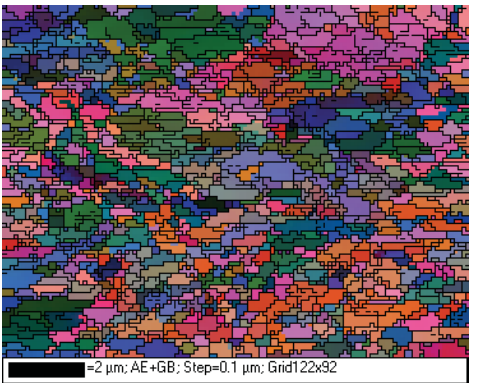

Fig. 10 Boundaries of sub-grains/grains with misorientation angles greater than 2º, sample after 8 ECAP passes

Results of statistic processing of misorientation angles of sub-grains and grains in the sample after 8 ECAP passes are shown in the Figure 13. It is obvious that sub-grains with the misorientation angle under 10° formed only approx. 15% of all ferritic grains. This confirms the fact that majority of ferritic grains was formed by the mechanism of re-crystallisation. No preferential occurrence of special boundaries was observed in the area of high-angle boundaries, such as e.g. twin boundaries. The biggest share of sub-grains corresponded to the misorientation angles up to 4°.
In conformity with data from literature it can be presumed that influence of sub-grains on the level of mechanical properties of investigated steel is lower than in case of the grains separated by high-angle boundaries [2]. Important in this connection is the fact that majority of ultra fine-grained ferritic grains in the structure was separated by high-angle boundaries.

Histogram of size distribution (equivalent diameter) of the grains with high-angle boundaries is shown in the Figure 14. After 8 ECAP passes approximately 25% of all grains was in the lowest size class (0.1 – 0.15µm). On the other hand size of some ferritic grains was bigger than 1µm. It is obvious from results of the TEM analysis, that many grains were smaller than the smallest usable step at the EBSD analysis (0.1µm). Average equivalent diameter of grains with the misorientation

angle greater than 10° was 0.32±0.20μm. This is only a roughly result, since grains with the size smaller than 0.1 μm could not be included into this analysis.

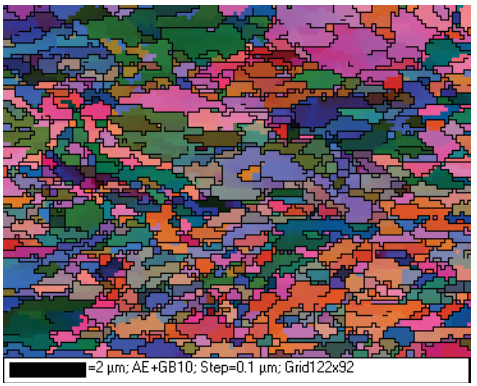

Fig. 11 Boundaries of grains with misorientation angles greater than 10º, 8 ECAP passes

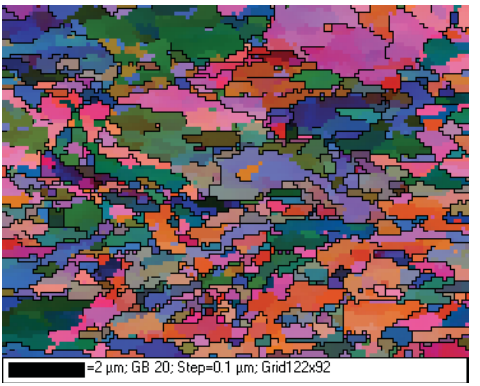

Fig. 12 Boundaries of grains with misorientation angles greater than 20º, 8 ECAP passes

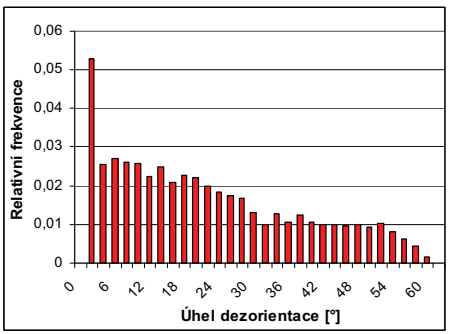

Fig. 13 Distribution of misorientation angles of angle sub-grain and grain boundaries, 8 passes

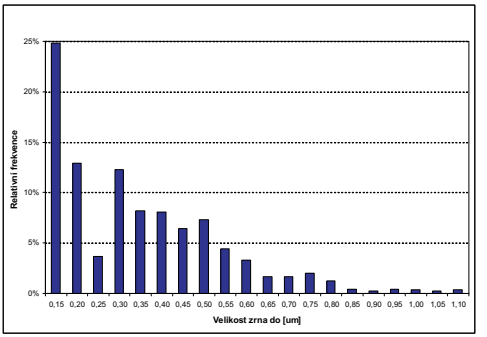

Fig. 14 Size distribution of grains with high-boundaries, 8 ECAP passes

Summary

The results obtained at the analysis of influence of severe plastic deformation by the ECAP method on structure and properties of the low-carbon steel grade P2-04BCh can be summarised as follows:

- Deformation of investigated steel by the ECAP method at the temperature of approx. 290°C lead to important improvement of strength properties. The biggest increase in strength was found after the first two passes through the ECAP die.
- Deformation occurred during 8 ECAP passes lead to formation of ultra fine-grained ferritic structure with a small fraction of globular carbidic particles, which were usually present at the boundaries of ferritic grains. Density of dislocations inside ferritic grains was very low. Majority of ferritic grains was formed by the mechanism of re-crystallisation of deformed metallic matrix.
- Sub-grains with an angle of misorientation under 10° formed after 8 ECAP passes only approximately 15% of all ferritic grains.
- Average size of ferritic grains with high-angle boundaries after 8 ECAP passes was 0.32±0.20μm. However, the analysis could not include the grains, the size of which was

smaller than 0.1μm. In comparison with the as received state the grain size was refined by two orders.

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References

[1] H. Gleiter: Deformation of Polycrystals: Mechanisms and Microstructures, ed. N. Hansen et al., Riso National Laboratory 1981, 15.

[2] W.D. Callister, Jr.: Materials Science and Engineering – an Introduction, John Wiley & Sons, Inc., 2000, ISBN 0-471-32013-7.

[3] J. Zrník, L. Kraus, T. Prnka a K. Šperlink: Příprava ultrajemnozrnných a nanokrystalických materiálů extrémní plastickou deformací a jejich vlastnosti, IV. Řada, Evropská strategie výrobních procesů [Preparation of ultra fine-grained and nano-crystalline materials by severe plastic deformation and their properties, Series IV., European strategy of production processes], ČSNMT, 2007, ISBN 978-80-7329-153-2.

[4] R.Z. Valiev: Some Trends in SPD Processing for Fabrication of Bulk Nanostructured Materials, Mat. Sci. Forum, 503-504, 2006, 3.

[5] R.Z. Valiev, I.V. Islamgaliev a I.V. Alexandrov: Bulk Nanostructured Materials from Severe Plastic Deformation, Prog. Mater. Sci., 45, 2000, 103.

[6] Y. Zhu, T. Lowe: Observation and Issues on Mechanisms of Grain Refinement during ECAP Process, Mat. Sci. Eng., A29, 2000, 46.

[7] Y. Iwahashi, J. Wang, Z. Horita, M. Nemoto a G. Langdon: Principle of Equal Channel Angular Pressing of Ultra Fine Grained Materials, Scripta Materialia, 35, 1996, 143.

[8] K.J. Kurzydlowski: Microstructural refinement and properties of metals processed by severe plastic deformation, Bulletin of the Polish Academy of Sciences, Technical Sciences, Vol. 52, No. 4, 2004, 301.

[9] P.B. Pragnell, J.R. Bowen, A. Gholinia: The Formation of Submicron and Nanocrystalline Grain Structures by Severe Deformation, Proc. 22nd. Riso Int. Symp. Scince of Metastable and Naniocrystalline Alloys – Structure, Properties and Modelling, ed. By A.R. Dinesen et al., Riso National Laboratory, Roskilde 2001, 105.

[10] J. Zrník, I. Mamuzič, S.V.Dobatkin, Z. Stejskal, L. Kraus: Low carbon Steel Processed by Channel Angular Warm Pressing, Metalurgia, Vpl. 46, 2007, 1, 21.

[11] R. Song, D. Ponge a D. Raabe: Grain boundary characterization and grain size measurement in an ultrafine grained steel, Max-Planck Institut fur Eisenforschung GmbH, www.mpie.de/1081, 16.12. 2005.

[12] www.ebsd.com

[13] M. Greger: Závěrečná zpráva o řešení projektu Výzkum a využití nanotechnologií a výroby nanostrukturních materiálů s vysokými pevnostními vlastnostmi pro moderní konstrukce [Final Report of the project Research and use of nano-technologies and production of nano-structural materials with high-strength properties for advanced structures, Project No. : FI-IM/033 VŠB-TU Ostrava, 2007.