Microstructure evolution and strength response of ultrafine grain medium carbon steel processed by high pressure torsion

Jozef Zrnik1,2, Reinhard Pippan3,4, Stephan Scheriau1,4
1 COMTES FHT Ltd, Prumyslova 995, 33441 Dobrany, Czech Republic
2 West Bohemian University, Universitni 22, 301 01 Plzen, Czech republic
3 Erich Schmid Institute of Materials Science, Austrian Academy of Science, Jahnstrasse 12, A-8700 Leoben, Austria
4 Christian Doppler Laboratory for Local Analysis Deformation and Fracture, Jahnstrasse 12, A-8700, Leoben, Austria

jzrnik@comtesfht.cz

Abstract. High pressure torsion (HPT) deformation method at increased temperature of 400°C with varying strain was applied to refine microstructure of the medium carbon steel (AISI 1045). To investigate the deformation behaviour of the ferrite-pearlite two phase structure the different shear deformation and constant high hydrostatic pressure of 7 GPa were applied. The shear stress evolution during deformation and measurement of the torque were recorded and related to structure development and mechanical strength. In order to characterize microstructure development the transmission electron microscopy (TEM) was accomplished. By execution of the first turn the grain refinement was observed at deformed disc periphery. In the centre of the disc, no matter what strain was introduced, the ferrite grain structure showed moderately deformed features and pearlite colonies were preserved. With further equivalent strain increase, equilibrium between the refinement of coarse phases and new grains restoration processes led to saturation of the grain refinement process. Upon tensile properties testing, the yield strength and ultimate strength increased with increasing equivalent strain ($\varepsilon_{eq}$) and only short region of strain hardening period prior failure appeared, regardless the strain applied. A small drop in the hardness across the disc was measured after execution of N= 4 and 6 turns, which may be related to formation of fine grain structure and structure recovery in disc centre.

1. Introduction
During the recent decade, bulk nanostructered materials produced by severe plastic deformation (SPD) have been investigated intensively. The production of fine grained materials by SPD, leads to a large number of investigations focusing on the substructure development and related mechanical properties. It has been already well known that SPD of metallic materials, involving processes such as equal angular pressing (ECAP), accumulative roll bonding (ARB) and high pressure torsion (HPT) is capable of producing ultrafine grained (UFG) materials with submicrometer or nanometer grain size [1,2]. The wide range of results available for various materials deformed by HPT confirmed a saturation in microstructural refinement and the strength and hardness as strain increases [3, 4]. The results point out that there are differences in the development of the microstructure between the pure and multiphase steels [5,6].

In this work two phase medium carbon steel has been deformed at increased temperature of 400°C
by high pressure torsion to different strain (executing different numbers of turns) at hydrostatic pressure of 7 GPa. The underlying relationship between microstructure and mechanical properties was analyzed with respect to the local axial position of analyses on the deformed disc.

2. Material and experimental procedure
A commercial medium carbon steel (Fe-0.45%C, 0.42%Mn, 0.23%Si 0.18%Cr, 0.043%Al in wt. %) was supplied in form of rod with a diameter of 20 mm. Prior HPT deformation the steel was annealed for 1.5 h at temperature of 850°C followed by air cooling in order to obtain uniform normalized structure. The mixture of quite coarse ferrite-pearlite structure was obtained as showed in SEM micrograph in Fig. 1. Following the annealing treatment, the size of pearlite colonies was of ~30 µm and of ~15 µm was size of ferrite grains.

The discs of 8 mm in diameter and ~0.95 mm in thickness have been cut off from the rod were deformed by torsion up to 6 turns at temperature of 400°C and at a pressure of 7 GPa. The equivalent von Mises strain \( \varepsilon_{eq} \) as a function of the number of turns \( n \) was calculated according to the relation: 

\[
\varepsilon_{eq} = \frac{2\pi nr}{t \sqrt{3}}.
\]

The size of effective strain conducting N-1, 2, 4, and 6 turns was \( \varepsilon_{eq} \sim 15, 30, 60 \) and 90. The changes in mechanical properties in dependence on straining (number of turns) were determined by microhardness measurement across the deformed disc with a load of 300 g using Vickers microhardness tester, by tensile test using subsize tensile pieces cut off at discs periphery (in radial direction) and by in-situ measurement of the torque. The reliable and accurate possibility to measure the torque during deformation allowed to quickly to determine changes in mechanical strength (shear stress \( \tau \)) without applying other method afterwards.

The transmission electron microscopy (TEM) was used to evaluate the microstructural evolution with respect to disc site and the number of turns corresponding to shear stress. The TEM micrographs were obtained by using JEOL JEM 2000FX operating at 200 kV. The purpose of different site selection was to evaluate an effect of shear strain magnitude across the disc (at peripheral and axial position of disc) on ultrafine grain microstructure development.

3. Results and discussion
Investigating material comprised of the coarse ferrite-pearlitic steel AISI 1045, as shown in Fig.1. Refining coarse steel structure to nanostructure size the application of large strain, usually with an equivalent strain \( \varepsilon_{eq} \) more than 10, is required. Electron microscopy analysis of thin prepared from
deformed discs exposed to different strain revealed formation of various structure characteristics in dependence of shear strain applied and localisation.

3.1. Microstructure
In order to compare the size of torsion straining at different position of deformed disc, Fig. 2 shows the microstructure observed in steel after exposure to the first turn at temperature of 400°C. Finishing the first turn ($\varepsilon_{eq} \sim 15$) the heterogeneity in structure development was still evident across the disc. At disc periphery alignment of smeared (partially dissolved) lamellae in pearlite next to the area of banded subgrain-like structure is documented in Fig. 2a,b. In the centre of the disc the lamellae remained, but some dissolution of the cementite lamellae, can be observed with smeared-like features, as presented in Fig. 2c. Conducting 4 and 6 turns ($\varepsilon_{eq} \sim 60$ and $\sim 90$), the dual phase structure was significantly modified with respect to analysis site as presented in Fig. 3. Regardless the strain size, successfully refined structure with small grains (grain size about of $\sim 200$ nm) having random high angle orientation was found in disc edge area as presented in Fig. 3a. Selected area diffraction method (SAED) confirmed high angle disorientation of small grains. In the centre of the disc the structure has moderately deformed features. In ferrite grains tangled dislocations are present, and pearlite colonies were only slightly deformed and/or locally fragmented, Fig. 3b,c. From the TEM micrographs cementite lamellae thinning in central disc area is evident in structure. This fact is attributed to partial dissolution of cementite during shearing and results in decreasing of the lamellae thickness.

3.2. Mechanical properties
In order to characterize the changes in mechanical properties due to the large shear deformation the various methods were applied. Vickers microhardness and tensile properties were evaluated at room temperature. The possibility to measure the torque in-situ during deformation provided a method to determine mechanical strength. The torque value includes contribution from disc deformation inside the cavity and contribution from outside the cavity where material is impressed btw anvils (burr) [5]. Microhardness evaluation after different straining (N turns) was convenient choice to evaluate the mechanical strength and is often applied [6,7]. In present work the method was mainly used to determine the strength change across the disc (at disc periphery and the centre) after application of different strain $\varepsilon_{eq}$ and compared with hardness of the initial steel. The measured Vickers hardness data (HV30) for different number of turns are stated in Tab.1. The results show that some decrease in hardness appeared across disc, performing the 4th turn. In deformed microstructure analyzed by TEM of thin foils there were not observed any specific structure diversities, that could match the results, and
then returning again to previous higher hardness values after 6 turns.

To obtain mechanical strength values, the specimens for tensile tests were machined out of HPT samples [8]. Two specimens (see in fig. 4) were cut off from the deformed disc. Tensile behaviour was evaluated at room temperature and records in form of load and sample elongation are documented in Fig. 4. The strength was increasing with increasing strain (turn numbers) and maximum load value obtained was higher than 1700 N. The development of the ultimate tensile strength is similar to the in-situ measured torque. The records of in-situ measured torque and shear stress (τ) for turn’s number of N1 and N6 are presented in Fig. 5. The possible explanation could be that the onset of steady state for both values coincidences with saturation in the decrease of the grain structure size and microhardness values. The strain necessary to reach this state deformation depends on the materials structural state.

4. Summary and conclusions

High pressure torsion method at increased temperature of 400°C was applied to refine microstructure in AISI 1045 steel. In dependence of strain applied there are markedly differences in the development of the microstructure as regards the site of analysis. Grain refinement and cementite lamellae dissolution was observed after the first turn (εeq ~ 15) at disc periphery. In disc centre, the deformed structure of ferrite with dislocation tangles had moderately deformed features of coarse cementite lamellae in pearlite grains. At higher strain applied cementite lamellae are aligned, thinner and fragmented in dependence of the strain introduced. The effective strain increase, set an equilibrium between the fragmentation cementite phase and new fine grains restoration processes and led to saturation of the grain refinement process. Upon tensile properties, the yield strength and ultimate strength increased with increasing Εeq. A small decrease in the hardness across the disc was measured across the disc after execution of 4 turns, which may be related to formation of fine grain structure in the disc and dislocation structure recovery at the disc centre.

Acknowledgement: The support from the Erich-Schmid-Institute of Materials Science of the Academy of Sciences in Leoben, Austria and form the Ministry of Education of the Czech Republic (MSM 2621691901) is greatly appreciated.

References

[1] V.M. Segal, V.I. Reznikov, A.E. Drobyshevski, Russian Metallography, 1, 1981, 99.
[2] R.Z. Valiev, A.V. Korznikov, R.R. Mulyukov, Mater. Sci. Eng. A 168 1993, 99.
[3] F. Wetscher, A. Worhauer, R. Stock, R. Pippan, Mater. Sci. Eng. A 387-389 2004, 809.
[4] N.Q. Chinh, G. Horvath, Y. Horita, T.G. Langdon, Acta Mater. 52 2004, 3555.
[5] F. Wetscher, Doctoral thesis, Leoben 2006, Austria.
[6] M. Richert, Q. Liu, N. Hasen, Mater. Sci. Eng. A 260 1999, 275.
[7] R.K. Islamgaliev, W. Buchgraber, Y.R. Kolobov, N.M. Amirkanov, A.V. Sergueva, K.V. Ivanov, G.P. Grabovetskaya, Mater. Sci. Eng A 319-321 2000, 872.
[8] A. Vorhauer, R. Pipan, Scripta Mater. 51 2004, 921

Fig. 4. Tensile records of after different HPT straining.
Fig. 5. Comparison of the in-situ torque and shear stress curves for N1 and N6