MICROSTRUCTURE REFINEMENT IN MARTENSITIC CREEP RESISTANT STEEL VIA SEVERE PLASTIC DEFORMATION

Petr KRAL[1*], Jiri DVORAK[1], Vaclav SKLENICKA[1], Yoichi TAKIZAWAA[2,3,4], Takahiro MASUDA[2,3], Zenji HORITA[2,3], Kveta KUCHAROVA[1], Marie KVAPILOVA[1], Marie SVOBODOVA[5]

[1]Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Brno, Czech Republic, EU
[2]Department of Materials Science and Engineering, Faculty of Engineering, Kyushu University, Fukuoka, Japan
[3]WPI, International Institute for Carbon-Neutral Energy Research (WPI-I2CNER), Kyushu University, Fukuoka, Japan
[4]Technology Department, Nagano Forging Co., Ltd, Nagan, Japan
[5]UJP PRAHA a.s., Prague, Czech Republic, EU

Abstract

In this study, the effect of severe plastic deformation on grain size refinement and mechanical properties of a coarse-grained steel was investigated. The experimental material used in the present investigation was advanced tungsten modified 9 %Cr P92 steel. The coarse-grained state of P92 steel was deformed at room temperature by high-pressure sliding or high-pressure torsion. The microstructure was investigated using Tescan Lyra 3 scanning electron microscope equipped with an electron-back scatter unit. Severe plastic deformation at room temperature led to grain size reduction down to sub-microscopic level. The grain size and hardness exhibited significant changes to the equivalent strain of about 10, and slight changes between the equivalent strains of 10 - 20. Equivalent plastic strains higher than 20 only led to insignificant changes in the mean grain size and hardness. The creep behaviour of the ultrafine-grained state exhibited the minimum creep rate of about two orders of magnitude greater when compared to the coarse-grained P92 steel.

Keywords: Steel, ultra-fine grained materials, creep, electron back scatter diffraction

1. INTRODUCTION

Tempered martensitic 9-12 % chromium steels are widely used for high-temperature components of steam power plants. The creep resistance of these steels is particularly given by the microstructure containing free dislocations and low energy boundaries, such as low angle grain boundaries and martensitic lath boundaries pinned by precipitates [1,2]. Their microstructure stability is significantly reduced during long-term operation by recovery of tempered martensite, coarsening and/or dissolving of primary precipitates and precipitation of new secondary phases.

Bulk ultra-fine grained and nanostructured materials can advantageously be produced by the methods of severe plastic deformation (SPD) [3-6]. The most attractive method is high pressure torsion (HPT) refining the structure of metallic materials to the ultra-fine (<1 μm) or even nano (<100 nm) scale [7-10]. The grain size of HPT-processed materials is so small that new low-angle grain boundaries are not formed in the grain interior. Thus the grain boundaries limit the mean free dislocation path and the dislocations easily recover at high-angle boundaries. Recent works [11,12] showed that grain size reduction to the microscopic level and high number of high-angle grain boundaries (HAGB) increase the contribution of boundary-mediated processes to the total creep deformation. Also, creep resistance was found to be reduced by high density of mobile dislocations in P91 steel after ageing for 1 week at 973 K and subsequent deformation by 1 ECAP pass at an elevated temperature [13]. The aim of this work is to study the influence of the application of severe plastic deformation on microstructure and creep behaviour of ultrafine-grained P92 steel.

2. EXPERIMENTAL MATERIAL AND PROCEDURES

The used experimental material was advanced tungsten modified 9 %Cr P92 steel. The chemical composition and heat treatment of the as-received state are given elsewhere [14]. Discs of 30 mm diameter and 1.1 mm thickness and sheets with dimensions of 10x100x1.1 mm^3 were cut from as-received P92 steel. Further, the discs were processed by 1 rotation HPT at room temperature under the pressure of 6 GPa and rotation speed of 0.1 mm per second. The sheets were processed by high-pressure sliding (HPS) at room temperature with the sliding distance of 15 mm under the pressure of 4 GPa. The value of von Mises equivalent strain during HPT is proportional to the radius of the disc according to the following equation [5]:

$$\varepsilon_{eq} = 2\pi r N/\sqrt{3}t \qquad (1)$$

where r is the distance from the disc centre, N is the number of turns and t is the thickness of disc. The value of von Mises equivalent strain during HPS process was estimated using the following equation [15]:

$$\varepsilon_{eq} = x/\sqrt{3}t \qquad (2)$$

where x is the sliding distance of plunger with respect to anvil and t is the thickness of the sample.

Constant load tensile creep tests were conducted at 873 K and 150 MPa. The testing was performed in protective argon atmosphere using flat specimens with the gauge length of 10 mm and cross section of 3 x 1 mm^2. The centre of the gauge length of tensile HPT-processed specimen was situated 7.5 mm from the central part of the disc. The UFG creep specimen was thus manufactured from the disc region with the equivalent strain of about 20-30. The gauge length of the HPS tensile specimen was taken from the central part of the sheet. The equivalent strain of the HPS specimen was about 7.9.

Microstructure investigations were performed using scanning electron microscope Tescan Lyra 3 equipped with NordlysNano EBSD detector operating at the accelerating voltage of 20 kV with specimen tilted at 70°. The electron back scatter diffraction (EBSD) analyses of UFG P92 steel were performed in the metallographic section perpendicular to the rotation axis of the disc and in the section parallel to the anvil. The step size of 25 nm for the EBSD maps was selected with respect to the inter-boundary spacing. The EBSD data were analyzed using HKL Channel 5 software developed by Oxford Instruments. The high-angle grain boundaries (HAGBs) were characterized as boundaries with misorientation angle $\theta > 15°$ and low-angle grain boundaries (LAGBs) as boundaries with $\theta < 15°$. Standard intercepts counting of LAGBs and HAGBs were carried out along tests lines. The value of the inter-boundary spacing was determined as the arithmetic average of the results in two perpendicular directions (horizontal, vertical).

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1. Microstructure after severe plastic deformation

Figure 1 shows the microstructure of P92 after 1 HPT revolution at room temperature in various positions from the center of the HPT disc. The microstructure acquired ~ 0.5 mm from the centre of HPT disc contained about 55 % of HAGBs (**Figures 1a and 1b**). The equivalent strain of about 1.6 caused reduction of the mean grain size down to 0.57 μm. However the microstructure was not homogeneous and contained the mixture of fine and large grains exceeding 1 μm. The further increase in plastic strain up to 23 led to additional reduction of grain size down to about 0.15 μm. The microstructure acquired ~ 7 mm from the centre of HPT disc exhibited nearly random distribution of misorientation angles (**Figures 1c and 1d**). The results demonstrate that the microstructure homogeneity increases with increasing value of the plastic strain occurring during HPT. Nevertheless, the microstructure was not fully homogeneous even after ε_{eq}=23 (r = 7 mm).

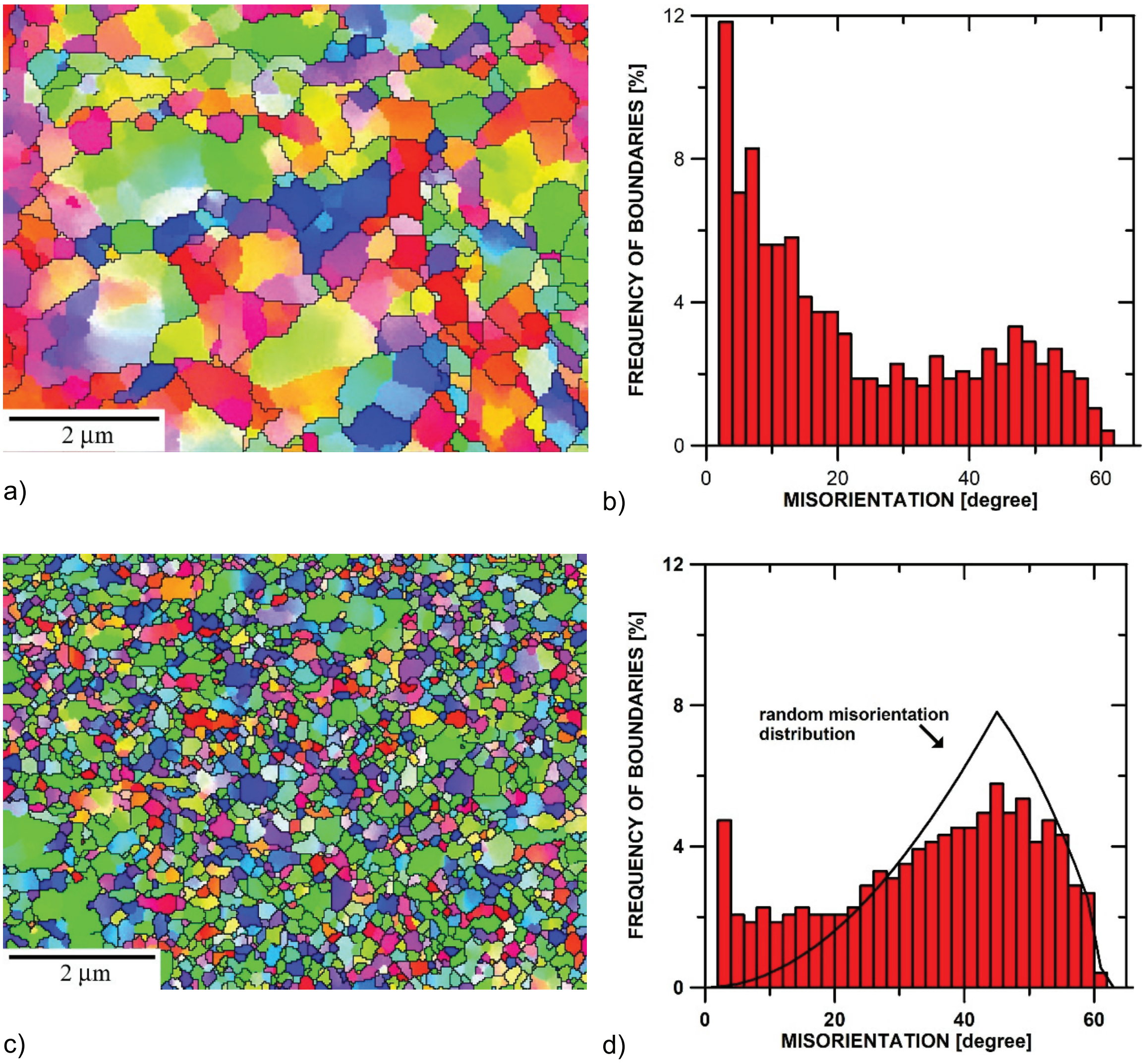

Figure 1 Microstructure and misorientation distribution of P92 steel after HPT deformation a, b) r = 0.5 mm (ε_{eq}~1.6), c, d) r = 7 mm (ε_{eq}~23)

Figure 2 shows the microstructure of the specimen processed by 1 HPS pass taken from the rear (**Figures 2a and 2b**) and middle (**Figures 2c and 2d**) parts of the sheet. Microstructure in the middle part of the sheet contained grains with the mean size of about 0.3 μm. In the microstructure about ~ 77 % of HAGBs was estimated. EBSD analyses revealed that the microstructure of the HPS-processed sheet was not fully homogeneous. The microstructure observed in the edges was different in comparison with the microstructure situated in the middle part.

The different microstructure at the edge can be the consequence of the material flow - excess material at the front part and lack of material at the rear part [15]. The results demonstrate that plastic strain of about 1.6 led to the formation of ultra-fine grained microstructure with high number of HAGBs. It means that the boundaries of tempered martensite in the as-received state with the preferential misorientations near Σ 3 (111/60°) [16] were replaced by random HAGBs. The further increase in plastic strain led to the formation of more or less homogeneous structure with almost random grain misorientation distribution. Similar microstructures have been found after large strains in regions of microstructure refinement saturation [17].

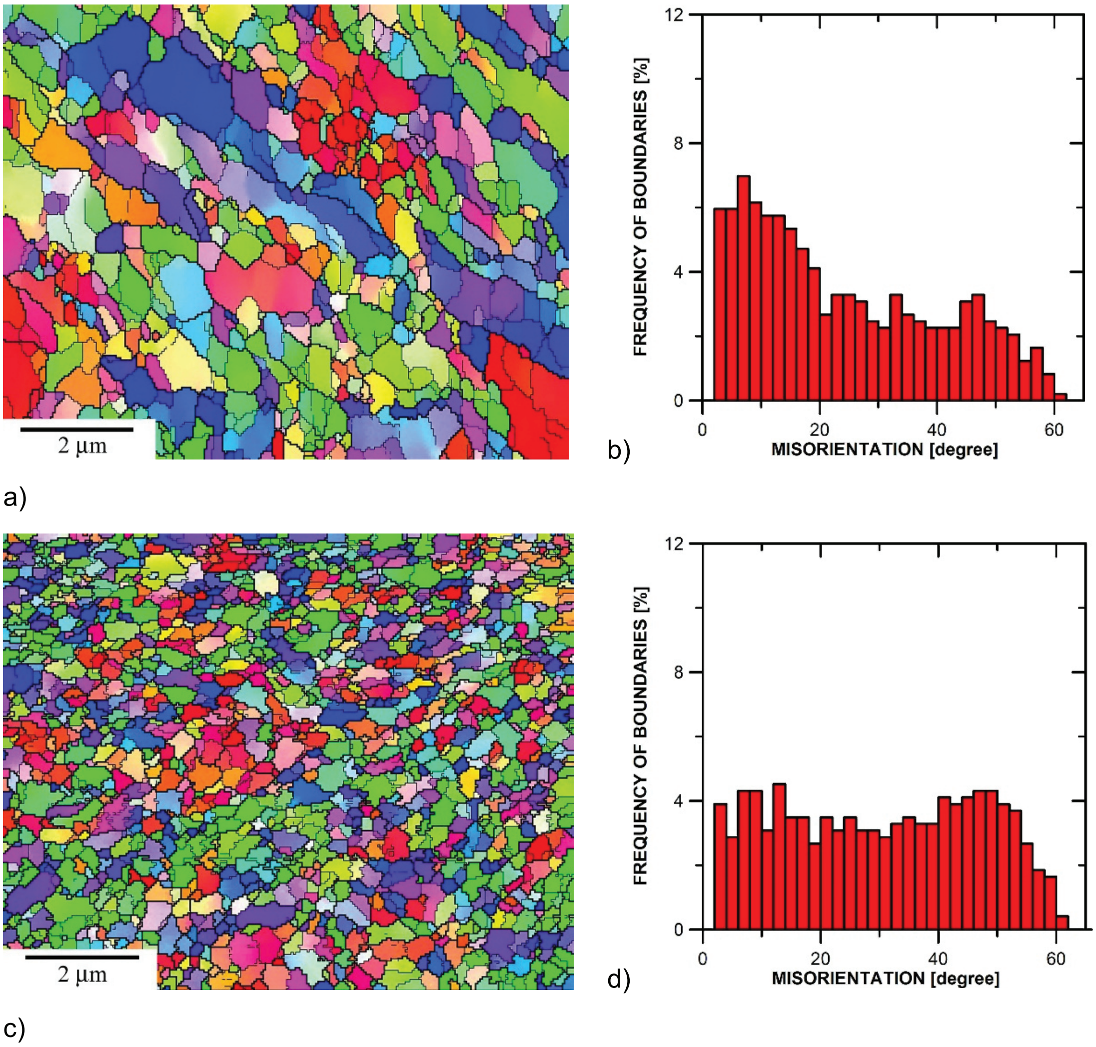

Figure 2 Microstructure and misorientation distribution of P92 steel after HPS deformation a,b) rear part, c,b) middle part (ε_{eq}~7.9)

3.2. Mechanical properties of P92 steel after severe plastic deformation

Figure 3 shows the dependence of average hardness on equivalent strain measured after HPT and HPS. The results show that hardness changes significantly to the strain of about 10. The further increase in plastic strain to 10 - 20 only caused slight additional increase in hardness. Equivalent plastic strains higher than 20 led to insignificant changes in hardness. The results also show hardness saturation at large strains. Thus, present observation is consistent with the results published in other works [17,18]. The microstructure refinement saturation often begins at strains between 10 and 30. It means that the grain size and HAGBs frequency do not change with further deformation. The comparison of creep curves acquired for as-received state of P92 steel and specimens processed by HPT and HPS are shown in **Figure 3b**. Inspection of **Figure 3b** revealed that the minimum creep rate becomes faster and the time to fracture becomes shorter with increasing plastic strain introduced into the microstructure during HPT and HPS. The faster creep rate observed in HPT and HPS processed specimens is related to the grain refinement and can be explained by boundary-mediated processes such as enhanced storage and recovery of dislocations at HAGBs and grain-boundary sliding [11,12]. The slight differences in creep behaviour found in HPT and HPS processed specimens could be caused by larger grains found in HPS processed specimen. Thus the lower frequency of HAGBs measured in HPS processed microstructure is available for boundary-mediated processes.

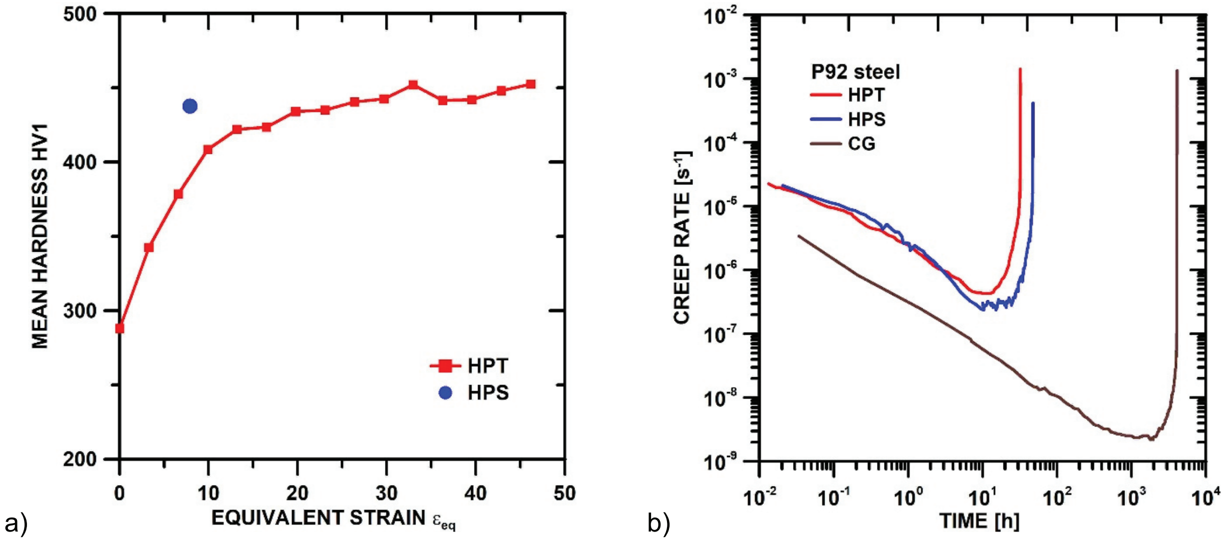

Figure 3 Mechanical properties of P92 steel processed by HPT and HPS deformation a) hardness at RT, b) creep behaviour at 873 K

4. CONCLUSIONS

Microstructure investigations showed that HPT and HPS at room temperature reduced grain size down to submicrometer level. P92 steel processed by HPT and HPS exhibited higher hardness at room temperature and faster minimum creep rate in comparison with coarse-grained P92 state. It is suggested that the change of mechanical properties in UFG steel is influenced by HAGBs. They strengthen UFG state at room temperature serving as the obstacles to dislocation glide but may soften the UFG material through boundary mediated processes such as enhanced storage and recovery of dislocations at HAGBs or grain-boundary sliding at elevated temperatures.

ACKNOWLEDGEMENTS

We acknowledge the financial support provided by the Czech Science Foundation under the project no. 16-09518S. This work was also supported by a Grant-in-Aid from the MEXT, Japan, for Scientific Research (S) (No.26220909). The HPT process was carried out in the International Research Center on Giant Straining for Advanced Materials (IRC-GSAM) at Kyushu University, Japan.

REFERENCES

[1] ABE, F., KERN,T-U., Viswanathan, R. *Creep-resistant steels*, Woodhead Publishing: Sawston, Cambridge, UK, 2008.

[2] ORLOVA, A., BURSIK, J., KUCHAROVA, K., SKLENICKA, V. Microstructural development during high temperature creep of 9 % Cr steel. *Mater. Sci. Eng. A*, 1998, vol. 245, pp. 39-48, https://doi.org/10.1016/S0921-5093(97)00708-9.

[3] LUKÁČ, P., KOCICH, R. GREGER, M., PADALKA, O., SZARAZ, Z. Microstructure of AZ31 and AZ61 Mg alloys prepared by rolling and ECAP. *Kovove Materialy*, 2007, vol. 45, pp. 115-120.

[4] KOCICH, R., GREGER, M., MACHÁČKOVÁ, A. Finite element investigation of influence of selected factors on ECAP process. *Metal 2010: 19th international metallurgical and materials conference*, Roznov pod Radhostem, Czech Republic, 2010, pp. 166-171.

[5] VALIEV, R.Z., ISLAMGALIEV, R.K., ALEXANDROV, I.V. Bulk nanostructured materials from severe plastic materials. *Prog. Mater. Sci.*, 2000, vol. 45, pp. 103-189, https://doi.org/10.1016/S0079-6425(99)00007-9.

[6] KOCICH, R., KUNČICKÁ, L., KRÁL, P., MACHÁČKOVÁ, A. Sub-structure and mechanical properties of twist channel angular pressed aluminium. *Mater. Character.*, 2016, vol. 119, pp. 75-83,

[7] FURUKAWA, M., HORITA, Z., NEMOTO, M., LANGDON, T.G. The use of severe plastic deformation for microstructural control. *Mater. Sci. Eng. A,* 2002, vol. 324, pp. 82-89, https://doi.org/10.1016/S0921-5093(01)01288-6.

[8] KUNČICKÁ, L., KOCICH, R. Comprehensive characterisation of a newly developed Mg-Dy-Al-Zn-Zr alloy structure. *Metals*, 2018, vol. 8. pp. 73. https://doi.org/10.3390/met8010073.

[9] TAKIZAWA, Y., KAJITA, T., KRAL, P., MASUDA, T., WATANABE, K., YUMOTO, M., OTAGIRI, Y., SKLENICKA, V., HORITA, Z. Superplasticity of Inconel 718 after processing by high-pressure sliding (HPS). *Mater. Sci. Eng. A*, 2017, vol. 682, pp. 603-612, http://dx.doi.org/10.1016/j.msea.2016.11.081.

[10] KUNČICKÁ, L., LOWE, T.C., DAVIS, C.F., KOCICH, R., POHLUDKA, M. Synthesis of an Al/Al2O3 composite by severe plastic deformation. *Mater. Sci. Eng. A*, 2015, vol. 646, pp. 234-241. doi:10.1016/j.msea.2015.08.075.

[11] BLUM, W., LI, Y.J., ZHANG, Y., WANG, J.T. Deformation resistance in the transition from coarse-grained to ultrafine-grained Cu by severe plastic deformation up to 24 passes of ECAP. *Mater. Sci. Eng. A*, 2011, vol. 528, pp. 8621-8627, https://doi.org/10.1016/j.msea.2011.08.010.

[12] KRAL, P., DVORAK, J., BLUM, W., KUDRYAVTSEV, E., ZHEREBTSOV, S., SALISHCHEV, G., KVAPILOVA, M., SKLENICKA, V. Creep study of mechanisms involved in low-temperature superplasticity of UFG Ti-6Al-4V processed by SPD. *Mater. Character.,* 2016, vol. 116, pp. 84-90, http://dx.doi.org/10.1016/j.matchar.2016.04.007.

[13] KOSTKA, A., TAK, T-G., EGGELER, G. On the effect of equal-channel angular pressing on creep of tempered martensite ferritic steels. *Mater. Sci. Eng. A*, 2008, vol. 481-482, pp. 723-726, https://doi.org/10.1016/j.msea. 2007.02.154.

[14] KRAL, P., DVORAK, J., SKLENICKA, V., MASUDA, T., HORITA, Z., KUCHAROVA, K., KVAPILOVA, M., SVOBODOVA, M. The Effect of Ultrafine-Grained Microstructure on Creep Behaviour of 9 % Cr Steel. Materials 11, 2018, Issue 5, No. 787, pp. 117-118, doi:10.3390/ma11050787.

[15] FUJIOKA, T., HORITA, Z., Development of High-Pressure Sliding Process for Microstructural Refinement of Rectangular Metallic Sheets. *Materials Transactions*, 2009, vol. 50, No. 4, pp. 930 - 933.

[16] SKLENICKA, V., KUCHAROVA, K., KRAL, P., KVAPILOVA, M., SVOBODOVA, M., CMAKAL, J. The effect of hot bending and thermal ageing on creep and microstructure evolution in thick-walled P92 steel pipe. *Mater. Sci. Eng. A,* 2015, vol. 644, pp. 297-309.

[17] PIPPAN, R., SCHERIAU, S., TAYLOR, A., HAFOK, M., HOHENWARTER, A., BACHMAIER, A. Saturation of Fragmentation during Severe Plastic Deformation. *Annu. Rev. Mater. Res.*, 2010, vol. 40, pp. 319-343.

[18] PIPPAN, R., WETSCHER, F., HAFOK, M., VORHAUER, A., SABIROV, I. The Limits of Refinement by Severe Plastic Deformation. Adv. Eng. Mater., 2006, vol. 8, No. 11, pp. 1046-1056.