Ultrafine grained structure development in steel with different initial structure by severe plastic deformation

Zrnik Jozef[1,2], Dobatkin V. Sergey[3] ,Raab George[4], Fujda Martin[2], Kraus Libor[1*]

Summary

The present work deals with grain refinement of medium carbon steel, having different initial microstructure, modified by either thermal and/or thermomechanical treatment (TM) prior severe plastic deformation. In case of TM treated steel, structure refinement was conducted in two steps. Preliminary structure refinement has been achieved due to multistep open die forging process which provided total strain of 3. Uniform and fine recrystallized ferrite structure with grain size of the order of 2-5 μm and with nest-like pearlite colonies was obtained. The further grain refinement of steel samples having different initial structure was accomplished during warm Equal Channel Angular Pressing (ECAP) at 400℃. The micro structure development was analyzed in dependence of effective strain introduced (ε_{ef} ~ 2.5 - 4). Employment of this processing route resulted in extensive deformation of ferrite grains where mixture of subgrains and ultrafine grain was found regardless the preliminary treatment of steel. The straining and moderate ECAP temperature caused the partial cementite lamellae fragmentation and spheroidization as straining increased. The cementite lamellae spheroidization was more extensive in TM treated steel samples. The tensile behavior was characterized by strength increase for both structural steel states; however the work hardening behavior was modified in steel where preliminary TM treatment was introduced to modified coarse ferrite-pearlite structure.

Keywords: steel, ECAP, microstructure, mechanical properties.

First TMS-ABM International Materials Congress
Held in conjunction with the 65th Annual Congress of ABM and the 18th IFHTSE Congress
July 26-30, 2010
International Rio Hotel, Rio de Janeiro, Brazil
Symposium: Mechanical Properties of Materials with Emphasis on Grain-Size Effects

[1]ComtesFHT, Inc., Dobrany, Czech Republic, - professor
[2]Technical University of Kosice, Slovak Republic, - professor
[3]Baikov Institute of Metallurgy and Materials Science , RAS, Moscow, Russia, - professor
[4]Institute of Physics of Advanced Materials, Ufa, Russia, - research fellow
[1*] ComtesFHT, Inc., Dobrany, Czech Republic, - director

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. Microstructural refinement of steel is usually achieved by alloying and/or thermomechanical treatments accompanies various types of phase transformation. Recently, advancement of severe plastic deformation (SPD) techniques provide another efficient access for grain refinement of metals and alloys. The fabrication of bulk materials with ultrafine grain sizes has attracted a great deal of attention over the past two decades because of the materials' enhanced properties [1, 2]. In recent years a worldwide effort in manufacturing process to obtain ultrafine grain structures in steels is persiting.

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 [3, 4]. 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 [5,6]. This interest has been motivated in part by the fact that UFG low carbon steels can be used in many applications as structural materials [7,8], 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 [9,10], 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 [11].

In the present study, the modification of ferrite-pearlite microstructure due to thermomechanical processing is described. Subsequently the effect of structure modification on development of ultrafine grain microstructure resulting from warm SPD was investigated and assessed with UFG structure resulted at SPD of conventionally treated AISI 1045 steel. The purpose of this article is to review the microstructures and revealed the underlying relationship between microstructure and mechanical properties of the steel.

Experimental procedures

Material and testing conditions. The medium carbon steel grade AISI 1045 was used for experimental. The chemical composition of steel was as follows: Fe-0.45C-0.23Si-0.63Mn-0.18Cr-0.043Al (in wt pct). The ferrite-pearlite microstructure, represented the initial state, with grain size of about 50 µm resulting from soaking treatment at the temperature of 960℃ is shown in Fig. 1a. By appropriate thermomechanical (TM) treatment, the refining of the coarse structure is possible and more suitable combination of strength, toughness and ductility can be obtained without additional alloying. Consequently, in order to achieve, prior ECAP pressing, preliminary refinement in complex ferrite-pearlite structure the thermomechanical

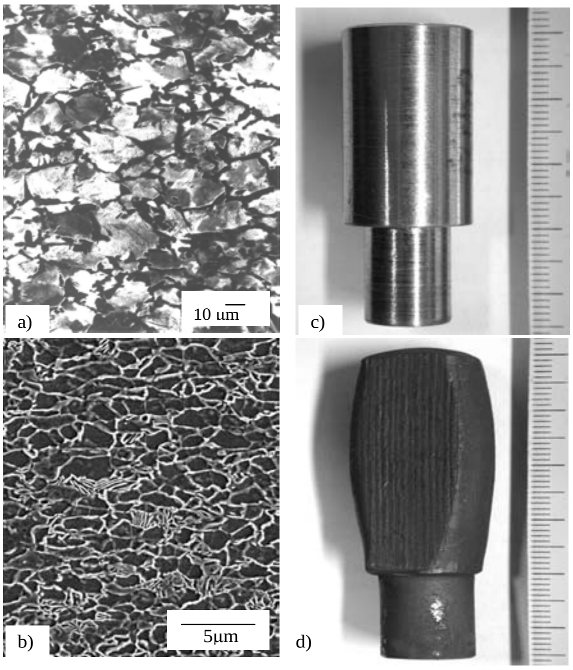

Fig 1. Ferrite - pearlite structure of the soaked (a) and thermomecanically processed medium carbon steel (b). The workpieces for TM processing (c) and after shaping (d).

processing has been designed. Cylindrical specimens in form of pegs, with initial diameter of 18 mm and length of 40 mm, were subjected to compressive deformation are documented in Fig. 1c. The repetitive deformation, after soaking at 900℃, has been performed continuously without further reheating in the recrystallized, non-recrystallized and intercritical α+γ temperature region and was expected to result in structure modification. To develop high and uniform strain across the peg, the repeated axial compressions of the peg 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 resulting microstructure in the centre of the specimen cross section is shown in Fig. 1b. The average ferrite and pearlite grain size measured at areas of various compressive strains from near the surface to the center of the specimen was below 5 µm. The last reduction of the specimen was at temperature of about 700℃.

Description of this sophisticated forging process has been attempted with aid of numerical simulation. 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 the effective strain 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.

From the soaked and TM treated pegs the cylindrical billets of 9 mm in diameter and 50 mm in length were machined for the ECAP experiment. The angle of intersections of the two channels was of φ = 120° in ECAP die. The warm ECAP pressing at 400℃ was performed and billets were su bjected to number of passes, N = 4, 5, 6 respectively. ECA pressing for each pass yielded an effective strain of ε_{ef} = 0,67. In both ECAP experimental the route Bc was chosen. The sample was rotated 90° around its longitudinal axis between each pass, in the same direction. The heating of sample for prior to pressing was done inside the pre-heated die until sample reached the pressing temperature of 400℃.

The microstructural examination of thermally treated and ECAP samples was carried out by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Thin foils for TEM observation were sliced normal to the longitudinal axis of ECAP pressed billets. The SEM and TEM microstructures were obtained by using JEOL JSM 6380 SEM operating at 10 kV and JEOL JEM 200FX TEM operating at 200 kV.

Tensile test were carried out using Zwick universal testing machine equipped with Multisens extensometer. Tensile specimens with gauge length of lo= 20 mm were tested at a constant cross-head speed of 0.016 mm/s until failure. The engineering stress-strain curves were constructed.

Experimental results and discussion

Microstructure of initial treated steel. The microstructure after solutioning treatment of bars exhibits an equiaxed pearlite grains with maximum size of approximately 50 µm which are surrounded by ferrite network. The structure 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 conducting the thermomechanical treatment of specimens, structure refining is apparent, Fig. 1b. 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 α+γ 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, increased as well.

Microstructure of solutioned and ECAP deformed steel. The SEM microstructures of steel after ECA pressing, experienced N = 4 and N = 6 passes are presented in Fig. 2 and that corresponds to ε_{ef} = 2,7 and 4 respectively. At ECAP channel angle of 120° the structure deformation was found heterogeneous across the deformed bar. It is clearly shown that after N= 4 passes the initial equiaxed ferrite grains of the solutionized specimens remain their coarse size and, although partially crushed, preserve lamellar morphology. The cementite lamellae within the grains are partially distorted and curved, (Fig. 2a). With increasing straining, executing N = 6 passes, the pearlite grains are stretched to a great extend in direction of shear stress (Fig. 2b). The ferrite grains are severely deformed and stretched out among the deformed pearlite grains. The higher degree of deformation caused the majority of pearlite to disintegrate to larger extent after sixth pass but in some of the former larger grains the lamellar morphology of cementite is still preserved. Both ferrite aggregates and deformed pearlite grains with respect to longitudinal axis of billet are oriented in one direction.

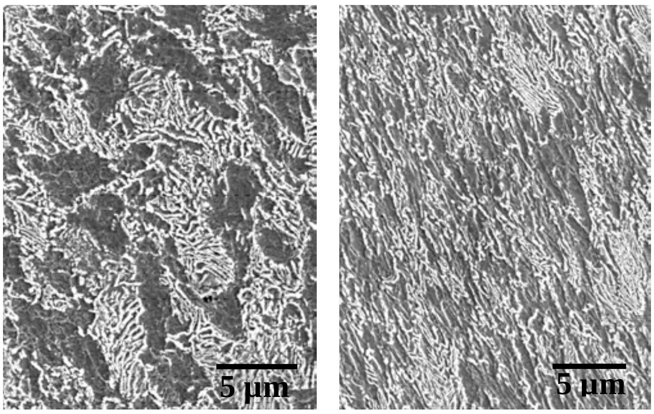

Fig. 2. Deformed solutioned ferrite-pearlite grains after ECAP experienced of N=4 and N=6 passes.

The substructure of deformed billets subjected to warm ECAP at temperature of 400℃ was investigated by TEM on plane parallel with billet axis. The deformed mikrostructure after a N = 4 passes through the die is shown in Fig. 3. In deformed ferrite grains the parallel bands are formed in elongated grains, Fig. 3a. The formation of dislocation cell sub-structure and sub-grain structure dominates in

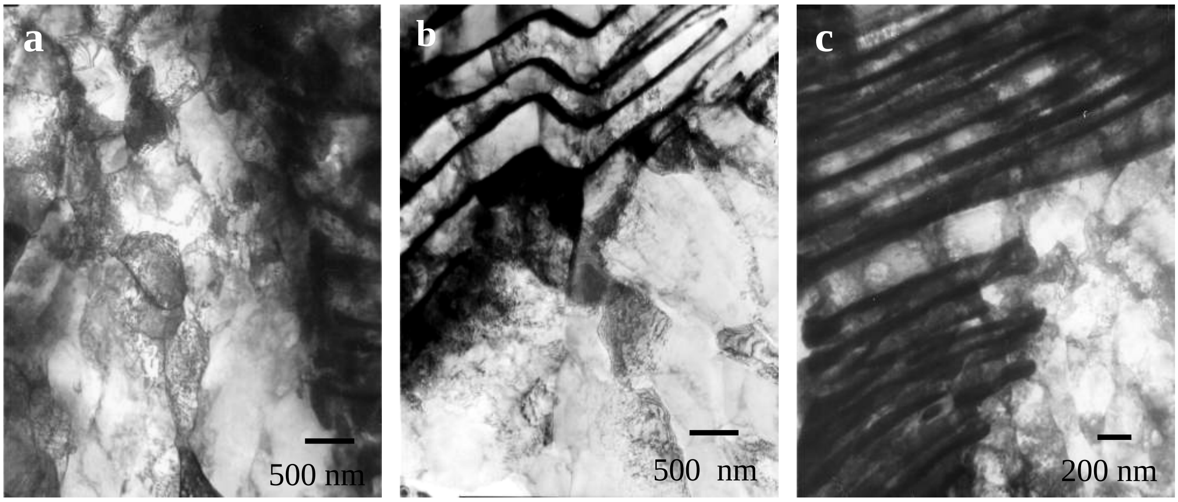

Fig. 3. Deformed microstructure after different ECAP straining:a) N=4; b) N=6; c) N=5.

deformed ferrite when effective strain was of ε_{ef} = 2,7. Appropriate to this in deformed ferrite was found the nuclei of new grains having high angle boundaries. Increasing number of passes to N = 6, (ε_{ef} = 4), in the deformed ferrite grains the submicron grains with high angle boundaries were more frequently observed, as documented in Fig. 3b. Colonies of lamellar pearlite, which survived in structure together with areas of crushed cementite particles were found often in deformed structure, when straining was increased, Fig. 3c. In the next place, colonies of lamellar pearlite, next to deformed ferrite with new ultrafine grains, were of frequent appearance there. As strain increased the cementite lamellae fragmentation and spheroidization in pearlite grains became a mechanism to disintegrate the cementite lamellae. This destructive process was also not only due to effective shearing but also was supported by increased temperature.

Microstructure of TM processed and ECAP deformed steel. Executing TM processing prior ECAP straining the refining of the microstructure was observed at SEM analysis and two different morphologies of ferrite were found in deformed microstructure. The fine equiaxed grains which resulted from the transformation of deformed austenite, and the elongated grains of already transformed ferrite, which experienced deformation in intercritical α+γ 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 lamellae were accidentally scattered in deformed fine grained ferrite structure. The refined deformed microstructure experienced different straining

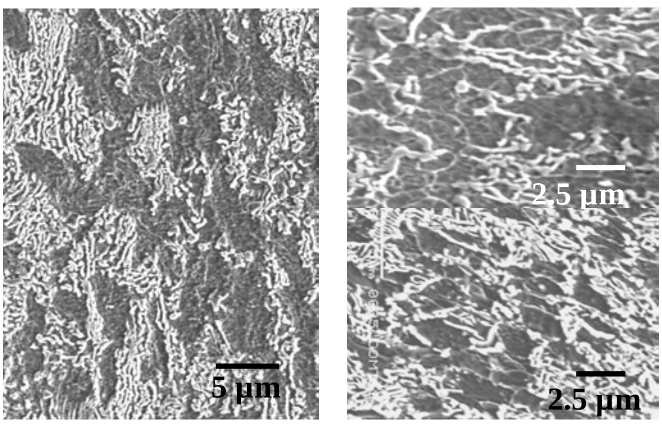

Fig. 4. Prior TM treated ferrite-pearlite structure experienced ECAP of N=4 and N=6 passes.

corresponding to N = 4 and N = 6 is presented in Fig. 4.

The TEM deformed micro- structures of steel after ECAP at 400°C are presented in Fig. 5. Conducted ECAP with channel angle of 120° the deformed structure was found heterogeneous across the billet. The areas of severe deformation where fragmentation of cementite and dislocation network in ferrite are evident and there are next to polygonized structure within the deformed ferrite grains. Investigating the substructure changes, also cementite lamellae spheroidization was apparent due increased temperature of deformation. Dislocation substructure in ferrite grains was modified upon dynamic polygonization, however the low angle boundaries are still in ferrite grains. Submicrocrystalline structure is formed within ferrite grains. As ECAP straining increases (N = 6) the progress in dynamic polygonization proceeded and formation of submicron size grains can be observed in ferrite and also between residue of cementite lamellae. This observation on substructure development indicates that progress in formation of more homogeneous submicrograined structure was retarded in medium carbon steel due to insufficient straining of specimen resulting from six passes using ECAP die with angle of 120°.

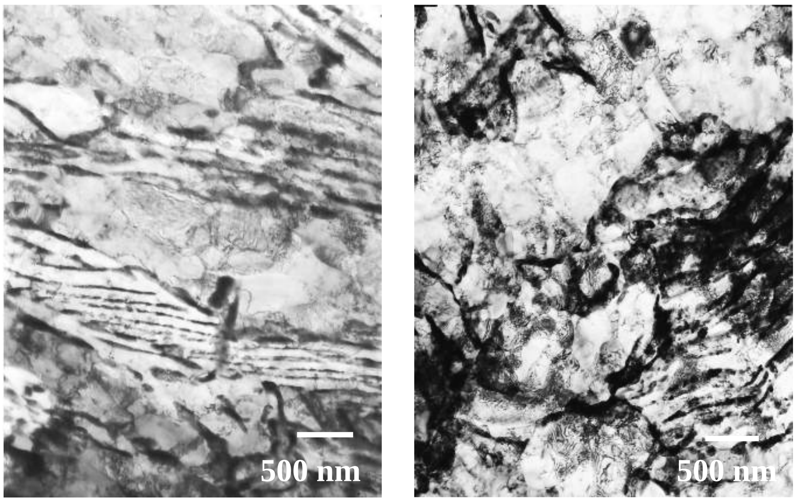

Fig. 5. TEM microstructure of ferrite-pearlite resulted after ECAP at 400°C: N=4.

Mechanical properties of solutioned and ECAP deformed steel. The mechanical properties of experimental steel, which was subjected to thermal and thermomechanical treatment prior SPD were measured by tensile test and hardness measurement. The results of tensile testing perform at room temperature are shown in Fig. 6. In case of the initial annealed steel condition there is an extensive period of work hardening and large elongation to failure. The deformation curve corresponding to TM treated steel shows slight work hardening effect and shorter deformation to failure.

The deformation behaviour of soaked and ECAP steel specimens is very similar for all three specimen, which have experienced different deformation. There is, after reaching the yield stress, section of slight hardening, which is extending as straining is increased. On the other side the strength value is of the same level for all specimens, which can be incurred by explain by quite large fraction of preserved lamellar pearlite. The contribution of this well preserved pearlite is inexpressive as regards to increase plasticity and/or to strength. On the other side, to balance this pearlite drawback, the contribution of ferrite UF grains either to strength or ductility is probably due to small volume fraction also slight, when compared with steel, which was TM treated.

Mechanical properties of TM processed and ECAP deformed steel. As concerns the deformation behaviour of the medium carbon steel which was TM processed and subjected to ECAP the tensile tests records are shown in Fig. 6c. For all specimens the deformation behaviour is very similar and after discontinuous yielding (sharp stress drop reaching the yield stress) there is a region of "creep-like"behaviour where no work hardening is observed. The section is extended as straining increases. Due to this sharp drop of stress the ultimate tensile strength is lower than "upper" yield stress. The sharp drop of plastic behaviour of this steel is noticeable different from that of the low carbon steel. However, this phenomenon can

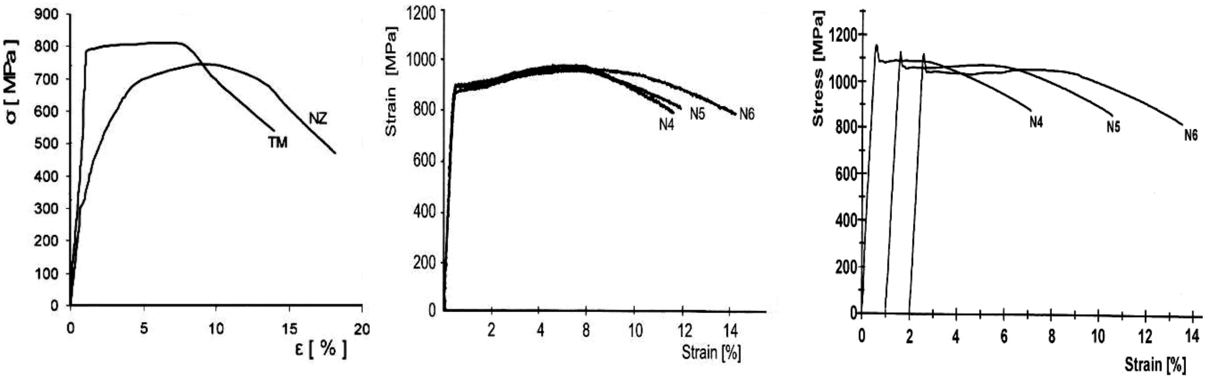

Fig. 6. Stress – strain curves for steel processed for different structural condition: a) initial soaking treatment (NZ) and thermomechanical treatment (TM); b) soaking treatment and ECAP; c) thermomechanical treatment and ECAP deformation.

be a result of cementite particle dissolution in ferrite and carbon atom saturation in lattice. The appearance of flat region on deformation curve can be attributed to a balance of strengthening effect, caused by newly arising increasing portion of fine new submicrocrystalline grains and on the other side by the more advanced dznamic recovery and dynamic recrystallization. These processes, as microstructure results confirmed, actually could participate in structure transformation process and contributed to deformation behaviour.

Conclusions

Microstructural evolution in time of warm ECAP was studied in medium carbon steel AISI 1045 with different initial microstructure resulted from thermal and TM treatment conducted to modify the initial structure prior SP deformation process. The major results can be summarised as follows:

1. Warm ECAP coarse initial structure of steel leads to formation of deformed microstructure. In dependence of the effective strain applied the ferrite substructure consisted of dislocation cell structure and subgrains. The lamellae pearlite fragmentation was nonproductive. With increased straining the more effective transformation of deformed structure to subgrain structure of ferrite was successful.

2. Microstructural observation of ECAP medium carbon steel subjected to preliminary TM treatment and grain size refinement did not show substantial structural changes in structure transformation in comparison with steel that was not TM treated.

3. Applying higher straining at increased temperature polygonized submicro-crystalline structure of high angle grain boundaries in large extent was form.

Formation of UF grain polygonized microstructure recovered the plastic deformation ability with less work hardening effect.

Acknowledgement

This paper contains results of investigation conducted as part of the MSM2631691901 project funded by the Ministry of Education of the Czech Republic.

Literature

[1] V.M. Segal: Mater. Sci. Eng. A, 197 (1995), p. 157.

[2] R.Z. Valiev, R.K. Islamgaliev, I.V. Alexandrov: Progr. Mater. Sci., 45 (2000), p. 103.

[3] V.M. Segal, V.I. Reznikov, A.E. Drobyshevskiy, V.I. Kopylov: Russ. Metall., 1 (1981), p. 99.

[4] R.Z. Valiev, A.V. Korznikov, R.R. Mulyukov: Mater. Sci. Eng., Vol.A. 168 (1993), p. 141.

[5] D.H. Shin, I. Kim, J. Kim, K.T. Park: Acta Mater., 48 (2000), p. 2247.

[6] K.T. Park, Y.S. Kim, D.H. Shin: Metall. and Mat. Trans. A, 32 (2001), 9, p. 2373.

[7] S.V. Dobatkin: In: Investigation and Application of Severe Plastic Deformation, edited by T.C. Lowe, R.Z. Valiev, Dodrecht, Kluwer (2000), p. 13.

[8] Y. Fukuda, Z. Ohishi, Z. Horita, T.G. Langdon: Acta Mater. Trans., 50 (2002), p. 1359.

[9] R.Z. Valiev, Y.V. Ivanisenko, E.F. Rauch, B. Baudelet: Acta. Mater., 44 (1996) p. 4705.

[10] N. Tsuji, Y. Ito, Y. Saito, Y. Minamino: Scripta Mater., 47 (2002), p. 893.

[11] S.M.L. Sastry, S.V. Dobatkin, V. Sidorova: Russian Metalls (Metally), 2 (2004), p. 129.