S. DOBATKIN, J. ZRNIK, I. MAMUZIC

ISSN 0543-5846
METABK 47(3) 181-186 (2008)
UDC – UDK 620.17:539.377:669.14=111

ULTRAFINE-GRAINED LOW CARBON STEELS BY SEVERE PLASTIC DEFORMATION

Received – Prispjelo: 2007-09-10
Accepted – Prihvaćeno: 2008-02-11
Original Scientific Paper – Izvorni znanstveni rad

The structure and properties of 0,14% C and 0,1% C - B low-carbon steels taken in two initial states, martensitic and ferritic-pearlitic, were studied after cold equal-channel angular (ECA) pressing. ECA pressing leads to the formation of only partially submicrocrystalline structure with a grain size of 150 – 300 nm, depending on the steel alloying and initial state. The finest structure with the elements of 190 nm in size is obtained in the 0,1% C - B steel microalloyed with boron. The strength of the 0,1% C - B steel after cold ECA pressing (R_m = 805-1235 MPa) meets the specifications of fasteners of the R80 - R120 strength grade. The strength of the deformed 0,14% C steel is close to the R80 strength grade.

Key words: low carbon steel, severe plastic deformation (SPD), equal channel angular pressing, submicrocrystalline structure, strength.

Ultrafino zrnati niskougljični čelici dobiveni intenzivnom plastičnom deformacijom. Struktura i svojstva niskougljičnih čelika sa 0,14%C i 0,1% C-B uzeta u dva početna stanja, martenzitnom i feritno-perlitnom, istraživani su poslije hladnog kutno kanalnog prešanja (KKP). KKP postupak dovodi do stvaranja parcijalne submikrokristalne strukture sa veličinom zrna 150-300nm, ovisno od vrste čelika i početnog stanja. Najfinija struktura sa veličinom zrna 190nm dobijena je za 0,1%C-B čelik (mikrolegiran borom). Vlačna čvrstoća ovog čelika poslije KKP-a (R_m=805-1235 MPa), uvrštava ovaj čelik u kvalitetnu skupinu R80 - R120. Za čelik sa 0,14%C dobijena vlačna čvrstoća ga uvrštava do R80.

Ključne riječi: nisko ugljični čelik, intenzivna plastična deformacija (IPD), kutno kanalno prešanje, submikrokristalna struktura, vlačna čvrstoća

INTRODUCTION

The methods of severe plastic deformation (SPD) allow one to obtain bulk samples with ultrafine-grained (nano- and submicrocrystalline) structure, which provides enhanced mechanical properties [1,2]. The equal-channel angular (ECA) pressing belonging to the most promising SPD methods was shown as the method improving the properties of materials, including low-carbon steels [3,4]. After cold ECA pressing one fails to obtain any developed submicrocrystalline structure, since the capabilities of the deforming equipment does not allow the fulfillment of more than 2-3 deformation cycles. However, even the obtained structure, which is partially submicrocrystalline and partially subgrained (cellular), provides a very high level of mechanical properties [5-8]. Warm ECA pressing also leads to the formation of the partially submicrocrystalline and partially recovered (polygonized) structure [9,10]. Such structure provides a high strength, but a low impact toughness. Hot ECA pressing due to its specific features leads the formation of a predominantly recovered (polygonized) structure with low-angle subgrain boundaries [9,10]. In this case, the strength decreases but remains sufficiently high, and the impact toughness substantially increases. The predominantly submicrocrystalline structure in the low-carbon steels can be obtained after cold ECA pressing and heating [8].

The purpose of this work was to study the possibility to obtain the submicrocrystalline structure in low-carbon steels after cold equal-channel angular (ECA) pressing and subsequent heating the 0,14% C and 0,1% C - B steels were selected as the candidate materials for the replacement of the steels with 0,3-0,4% C for high-strength fasteners (bolts, pin) (Table 1). An increase in the fraction of high-strength fasteners will reduce the consumption of materials in modern automobiles.

EXPERIMENTAL PROCEDURE

The 0.14% C and 0,1% C - B low-carbon steels (Table 1) in two initial states: ferritic-pearlitic (after normalization from temperature 920°C) and martensitic

S. Dobatkin, A.A.Baikov Institute of Metallurgy and Materials Science, Russian Academy of Sciences, Moscow, Russia, J.Zrnik Comtes FHT, Plzen, Czech Republic, I.Mamuzic, Faculty of Metallurgy, University of Zagreb, Sisak, Croatia

Table 1. Chemical composition of steel studied

Steels	Mass / %										
	C	Si	Mn	Cr	S	P	Mo	V	Nb	Ti	B
0,14%C	0,14	0,008	0,39	0,05	0,04	0,009	-	-	-	<0,003	0,0002
0,1%C-B	0,1	0,11	0,36	0,04	0,004	0,004	-	-	-	0,02	0,0029

(after water quenching from the same temperature) were used for the study. The ferrite grain size after normalization was ~ 17 μm in both steels, while the size of the former austenitic grain in the martensitic structure was 14 μm in the 0,14% C steel and 13 μm in the 0,1% C - B steel. The pearlite content did not exceed 5%.

The ECA pressing of the 0,14% C and 0,1% C - B steels at angles of 90° and 120° between the channels was performed at T = 20°C for the initially ferritic-pearlitic structure and at T = 300°C for the initially martensitic structure in the samples of 8 mm in diameter and 40 mm by route B_c, i.e., with a sequential rotation of the sample by 90° around its axis before the subsequent deformation cycle. The number of deformation cycles (passes) at an angle of 90° between the channels was N=2 ($\varepsilon \approx 2,3$) for both temperatures of ECA pressing, and, at an angle of 120° between the channels, this number was N = 3 ($\varepsilon \approx 2,0$) at T=20°C for the initially ferritic-pearlitic structure and N = 4 ($\varepsilon \approx 2,5$) at T=300°C for the initially martensitic structure. The number of passes corresponded to the maximum deformation without failure. After ECA pressing, the samples were heated to temperatures ranging between 400 and 700°C, held for 30 min, and quenched in water.

The structure examination was performed with an "Olympus PME 3" optical microscope and a JEM-100CX transmission electron microscope.

Microhardness was measured with an M-400-H "Leco" tester at a load of 50 g. The mechanical tests of the samples of 3 mm in diameter and 30 mm in gage length were performed with an INSTRON 1196 testing machine at a strain rate of 1,5 mm/min. Mechanical tensile tests at elevated temperatures were performed with a PV-3012M unit in a vacuum of 10^{-4} Pa on the samples of $7 \times 2.5 \times 0,6$ mm^3 in a gage base size. The initial tension rate was $5 \cdot 10^{-3}$ s^{-1}.

RESULTS AND DISCUSSION

The microhardness values of 0,14% C and 0,1% C - B steels in the initial undeformed ferritic-pearlitic and martensitic states are approximately identical, 1,4 and 1,9-2,0 GPa, respectively (Figure 1). After ECA pressing, microhardness substantially increases. This effect is higher for the initial martensitic state compared with the initial ferritic-pearlitic one, and for each state it is more pronounced in the 0,1% C - B steel microalloyed with boron (Figure1).

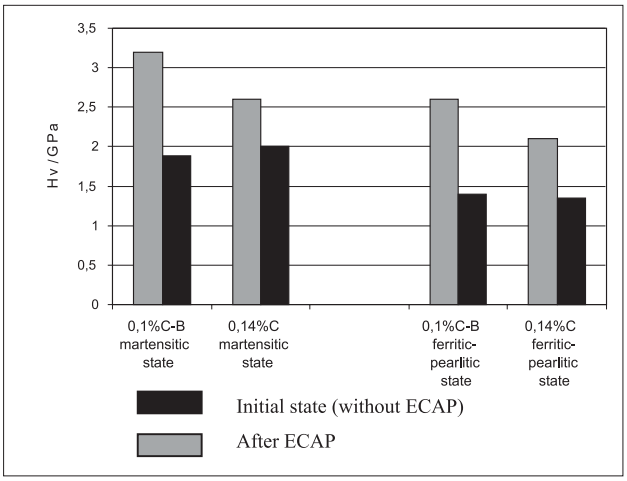

Figure 1. Microhardness of 0,14% C and 0,1% C-B steels with martensitic and ferritic-pearlitic initial states after cold ECA pressing.

For example, after ECA pressing, the microhardness values of the 0,14% C steel in the initially martensitic state and of the 0,1% C - B steel in the initially ferritic-pearlitic state are the same: 2,6 GPa. The maximum strengthening, up to 3,2 GPa, was observed after ECA pressing of the 0,1% C - B steel in the initially martensitic state.

The transmission electron-microscopic (TEM) examination showed that the cold ECA pressing of the 0,14% C steel samples with the ferritic-pearlitic structure at an angle of 90^0 between the channels and at N = 2 leads to the formation of the subgrained, predominantly cellular structure characterized by a high imperfection of subgrain boundaries and a high density of free dislocations (Figure 2a). There are both the regions of oriented structure and the regions of relatively equiaxed structure elements including isolated grains with high-angle grain boundaries. The normalized 0,1% C - B steel after ECA pressing has a similar structure, which differs by a smaller density of free dislocations, more perfect and thinner subgrain (cell) boundaries, more pronounced structure orientation, and a smaller size of structure elements, 230 nm against 285 nm typical of the 0,14% C steel (Figure 2c and Table 2).

The structure observed after ECA pressing in both steels taken in the initial martensitic state is more perfect, less oriented, and more dispersed than that considered above (Figures 2 b,d and Table 2). The presence of substantially larger number of grains with high-angle boundaries in the sample is confirmed by ring-like electron diffraction pattern with a large number of reflections and a

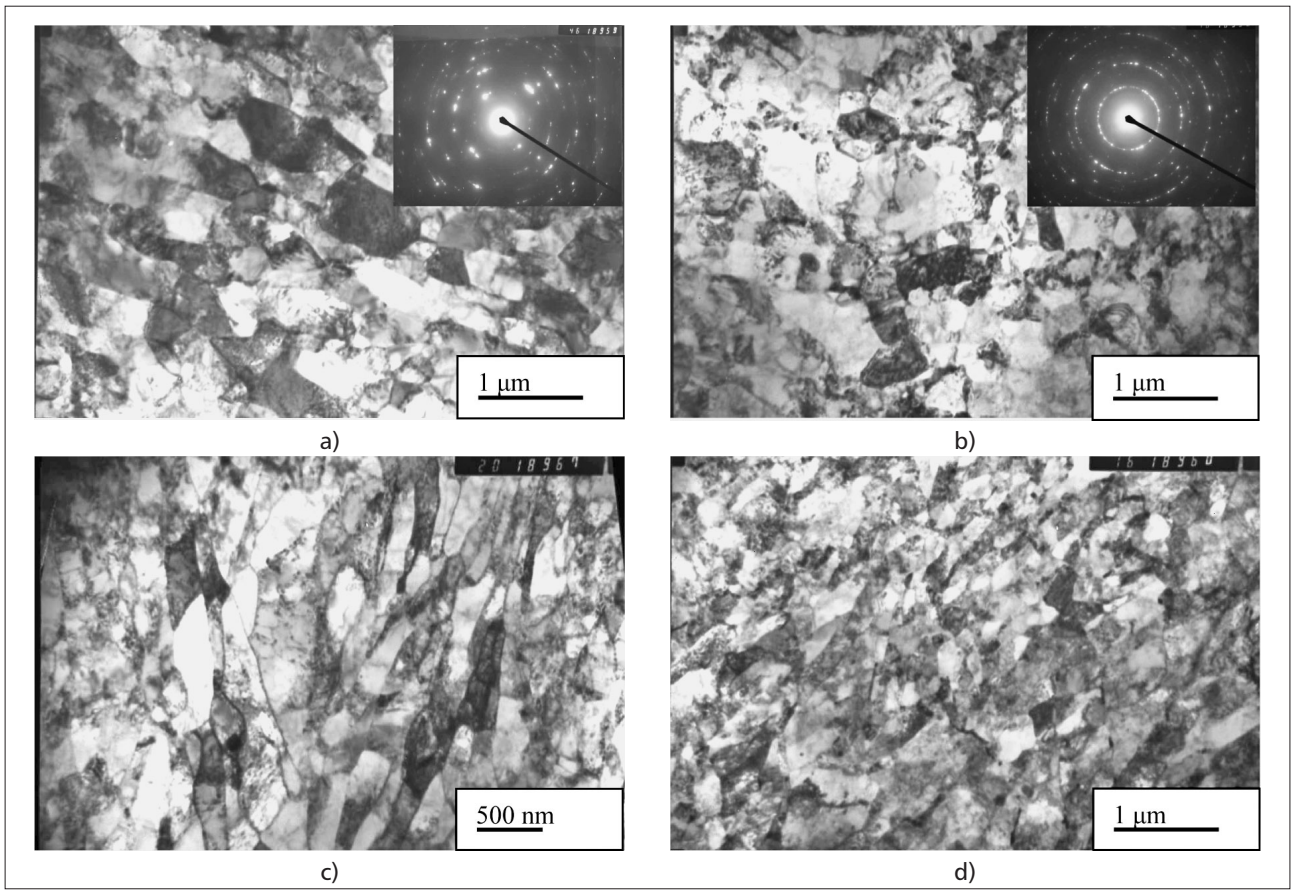

Figure 2. Structure of 0,14% C (a,b) and 0,1% C-B(c,d) steels with martensitic(b,d) and ferritic-pearlitic (a,c) initial states after ECA pressing.

TEM contrast at the grain boundaries (Figures 2b,d). The average size of structure elements after ECA pressing in the initially martensitic state is smaller in the 0,1% C - B steel compared with the 0,14% C steel, 190 nm versus 245 nm, respectively (Table 2 and Figures 2 b, d). The smaller size of structure elements after ECA pressing in the steels with the initially martensitic structure can be explained by a substantially higher initial dislocation density, by the presence of fine carbides and carbonitrides, and by higher deforming loads applied upon ECA pressing, i.e., by higher pressure. The smaller size of structure elements in the 0,1% C - B steel compared with the 0,14% C steel is caused by the presence of boron. Boron is added for the improvement in hardenability, since fasteners are usually used after quenching and high-temperature tempering. In this case, boron should be present in the solid solution rather than in borides. For this reason, small additions of Ti and Al are introduced into the steel for biding nitrogen and carbon into carbonitrides (Table 1) [11]. Thus, boron in solid solution decelerates the diffusion and decreases the stacking fault energy. This, in turn, decreases the size of structure elements. Fine carbonitrides act in the same manner.

Table 2. **Structure element sizes of the 0,14%C and 0,1%C-B low-carbon steels studied after ECAP and heating / nm**

N°	Steel	0,14%C		0,1%C-B	
Initial structure Treatment		ferritic -pearlitic	martensitic	ferritic -pearlitic	martensitic
1	ECAP	285	245	230	190
2	ECAP+ heating 500°C, (30min.)	=5000	=2000	200-750	200-450

Thus, only partially submicrocrystalline structure with a grain size of 150 – 300 nm, depending on the alloying and the initial state, can be obtained immediately after ECA pressing in both steels taken in both initial states. Along with the grained structure, the subgrained and cellular structures, including oriented one, are observed. To obtain more perfect submicrocrystalline structure, one should perform additional heat treatment.

Upon heating, we determined the conditions providing a uniform submicrocrystalline (grain size <1000 nm) grained structure. A decrease in microhardness upon heating in the absence of any new grains detectable by optical microscopic examination was used as the criterion. The decrease in microhardness can be caused by the recovery of grain boundaries, by decreasing density of free dislocations, and by increasing fraction of submicrocrystalline structure.

The 0,14% C and 0,1% C - B steels after ECA pressing were heated to temperatures ranging between 400

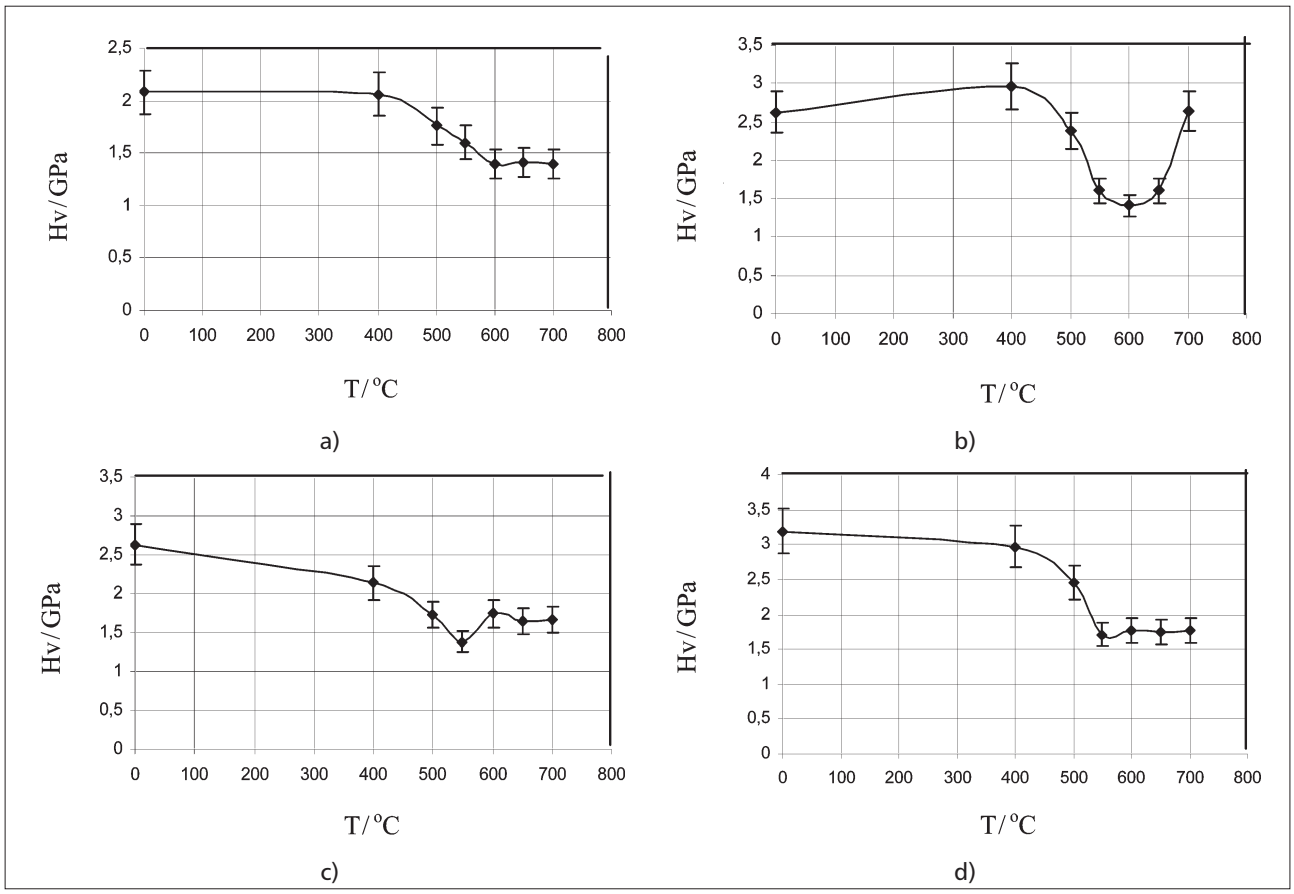

Figure 3. Microhardness of 0,14% C (a,b) and 0,1% C-B (c,d) steels with martensitic(b,d) and ferritic-pearlitic(a,c) initial states during heating at 400-700°C (holding time 30 min.) after ECA pressing.

and 700°C. The microhardness little changes after heating to 400°C and is retained for the quenched and normalized steels at levels of 2,8 GPa and 2,1 GPa, respectively, for the 0,14% C steel and at levels 3,1 GPa and 2,2 GPa, respectively, for the 0,1% C - B steel (Figure3).

After heating to 500°C, we observed a decrease in microhardness for the quenched and normalized steels approximately to 2,4 and 1,7 GPa, respectively, for the 0,14% C and to 2,5 and 1,5 GPa, respectively, for the 0,1% C - B steel due to the nucleation and growth of new grains (Figure 3). After decrease in microhardness, its value remains at levels of approximately 1,5 and 1,7 GPa for the 0,14% C and 0,1% C - B steels, respectively.

After heating to 700°C, the microhardness of the 0,14% C steel initially taken in the quenched state abruptly increases to approximately 2,65 GPa due to the phase transformation including the formation of austenite, which is transformed to bainite upon subsequent water quenching (Figure 3b).

The optical microscopic analysis revealed that the grains can be metallographically observed by etching only after heating to temperatures above 550°C. With increasing temperature from 550 to 700°C, the grain size in both steels increases from 7 to 12 μm. For the TEM structure examination, we selected the heat-treatment regime including heating to 500°C and holding for 30 min.

The degree of structure orientation of the 0,14% C and 0,1% C - B steels taken in both initial states and subjected to ECA pressing decreases after heating to 500°C (Figure 4). The fraction and size of grains in the samples increase (Table 2 and Figure 4). At the general equiaxed character of the structure elements after heating, the regions of oriented structure are retained in all cases. The structure is characterized by the grain nonuniformity. The maximum nonuniformity was observed in the 0,14% C steel with the initially ferritic-pearlitic structure. The grain size varies from 200 nm to 5 μm with the retention of the regions of oriented subgrained structure. In the 0,14% C steel with the initially martensitic structure, the grain variation is somewhat less 200 – 2000 nm. The structure of the 0,1% C - B steel after ECA pressing and heating is finer and more uniform than that of the 0,14% C steel under the same conditions. This effect is more pronounced in the case of the initial martensitic structure (Figure 4d). However, even in the case where the grain size generally varies between 200 and 450 nm, we observe also isolated grains of micron size. The high uniformity of the 0,1% C - B steel initially taken in the quenched state and then subjected to ECA pressing and heating is explained by a higher uniformity of the initial martensitic structure and by the presence of fine carbides and, especially, carbonitrides, which retard the grain growth upon heating.

Thus, the TEM examination of both steels after ECA pressing and heating to 500°C showed the formation of

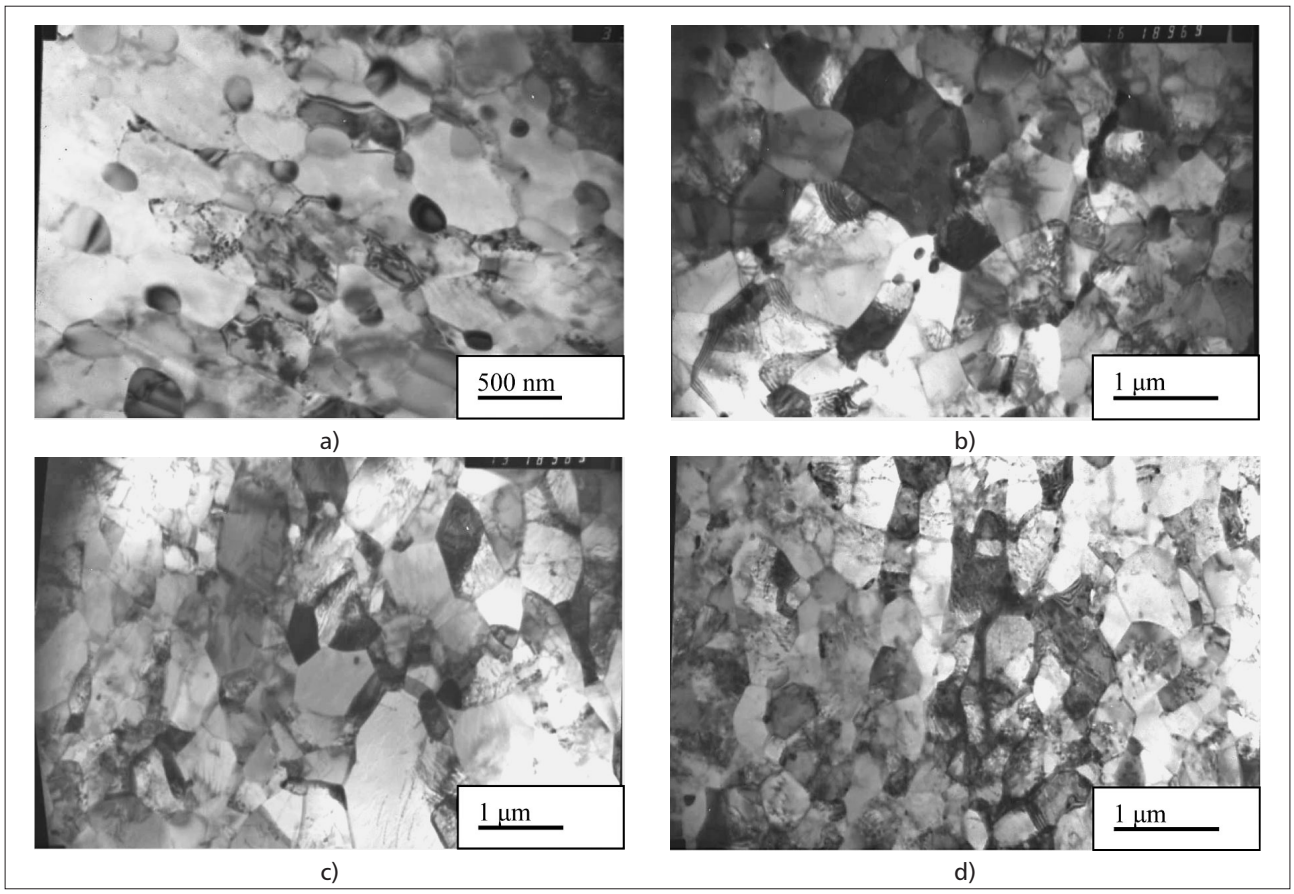

Figure 4. Structure of 0,14%C (a,b) and 0,1% C-B (c,d) steels with ferritic-pearlitic (a,c) and martensitic (b,d) initial states after ECA pressing and heating at 500°C (holding time 30 min.).

heterogeneous submicrocrystalline structure with a grain size ranging from 200 nm to 5 μm and the regions of oriented subgrained structure. The grain size and structure nonuniformity are smaller in the 0,1% C - B steel compared with the 0,14% C steel and, in both steels, they are smaller in the samples with the initially martensitic structure compared with the ferritic-pearlitic one. It seems that a more uniform grained structure can be obtained by heating to lower temperatures such as 400-450°C and longer holdings.

After ECA pressing at an angle of 90° between the channels, the strength characteristics of the steels substantially increase. For example, the strength of the deformed 0,14% C steel increases more than by a factor of two relative to the normalized 0,14% C (R_m = 335 MPa) (Table 3).

After ECA pressing, the strength of the steels initially taken both in the ferritic-pearlitic and in the martensitic states changes in accordance with the structure dispersion and special features considered above (Tables 2 and 3) and is higher for the initially martensitic state in both steels and in the 0,1% C - B steel compared with the 0,14% C steel for both initial states. Note the following special features:

1. The difference in the strength characteristics for both initial states is smaller in the 0,14% C steel compared with the 0,1% C - B steel.

Table 3. **Mechanical properties of the 0,14%C and 0,1%C-B low-carbon steels after ECAP**

N°	Steel	Initial state	Regime of ECAP	R_e / MPa	R_m / MPa	A / %	Z / %
1	0,14%C	normalized	T=20°C; *N=2; *φ=90°	770	780	7	39
2		quenched	T=300°C; N=2; φ=90°	780	795	13	56
3	0,1%C-B	normalized	T=20°C; N=2; φ=90°	760	805	8	55
4		quenched	T=300°C; N=2; φ=90°	840	910	11	71
5		normalized	T=20°C; N=4; φ=120°	825	830	8	19
6		quenched	T=300°C; N=3; φ=120°	1090	1235	7	13

* - N – number of passes
** - φ – angle of channels intersection

2. The plasticity characteristics (both relative elongation and reduction in area) of both steels after ECA pressing are higher for the initial martensitic state.

3. The R_e/R_m ratio being high for both steels is smaller for the 0,1% C - B steel.

The mechanical properties of the 0,1% C - B steel were determined also after ECA pressing at an angle of 120° between the channels. In this case, we reached a higher total degree of deformation than at an angle of 90° and, therefore, obtained more developed structure with a larger fraction of grains with high-angle boundaries. This is indirectly confirmed by higher strength characteristics of the steels after ECA pressing at an angle of 120° between the channels (Table 3). Such increase in strength compared with that observed after ECA at an angle of 90° between the channels is higher for the 0,1% C - B steel with the initially martensitic structure. The R_m value reaches 1235 MPa. The obtained strength characteristics of the 0,1% C - B steel for both cases of ECA pressing ensure the level of the properties specified for the fasteners of R80 - R120 (or 8,8.-12,9 according to the Russian standard) strength grades. The strength characteristics of the 0,14% C steel after ECA pressing are close to those of the R80 strength grade. For this reason, we assume that the 0,1% C - B steel after ECA pressing belongs to the high-strength steel grade and can be recommended for the manufacture of bolts and pins in automotive industry.

CONCLUSIONS

1. Cold ECA pressing of the 0.14% C and 0.1% C - B steels initially taken in two states, normalized and quenched, leads to the formation of partially submicrocrystalline structure of 150 – 300 nm in the size of structure elements, depending on the alloying and the initial state. The deformed structure is finer for the initially martensitic structure compared with the ferritic-pearlitic one and in the 0,1% C - B steel compared with the 0,14% C steel.

2. Heating to 500°C of the 0,14% C and 0,1% C - B steels after ECA pressing leads to the formation of nonuniform submicrocrystalline structure of 200 nm - 5 μm in grain size, in which the regions of the oriented subgrained structure are retained. The grain size and structure nonuniformity are smaller in the 0.1% C - B steel compared with the 0,14% C steel and for the initially martensitic structure compared with the ferritic-pearlitic one. A fine and uniform structure of 200-450 nm in the size of structure elements is obtained in the initially martensitic samples of the 0,1% C - B steel microalloyed with boron.

3. The structure of the 0,1% C - B steel after cold ECA pressing provides a high strength (R_m = 805-1235 MPa) and a good plasticity (A = 7 - 11%), which meet the specifications of the fasteners of the R80- R120 strength grade. The strength of the deformed 0,14% C steel is close to the R80 strength grade.

Acknowledgements – The work was supported by the RFBR (project no.07-03-00342) and by the Russian Ministry of Education and Science (contracts nos. 02.527.11.9019 and 02.513.11.3197).

REFERENCES

[1] T.C. Lowe, R.Z. Valiev (Eds), Investigations and Applications of Severe Plastic Deformation, Kluwer Academic Publishing, Dordrecht, The Netherlands, 2000, p.395.

[2] R.Z. Valiev, I.V.Alexandrov, Nanostructured Materials Obtained by Severe Plastic Deformation. Logos, Moscow, Russia, 2000, p. 272. (in Russian).

[3] V.M. Segal, V.I. Reznikov, A.E. Drobyshevskiy, V.I. Kopylov, D.A. Pavlik, Metalls 1 (1981) 115-123 (in Russian).

[4] S.V. Dobatkin, in: T.C. Lowe, R.Z. Valiev (Eds.), Investigations and Applications of Severe Plastic Deformation, NATO Science Series, Kluwer Academic Publishers, Netherlands, v.3/80, 2000, p.13-22.

[5] Y.Fukuda, K.Oh-ishi, Z.Horita, T.G.Langdon, Acta Materialia 50 (2002) 1359-1368.

[6] D.H. Shin, K.-T. Park, in: M.Zehetbauer, R.Z.Valiev (Eds.), Nanomaterials by Severe Plastic Deformation, J.Wiley VCH Weinheim, Germany, 2003, pp.616-622.

[7] J.Wang, C.Xu, Y.Wang et al, in: M.Zehetbauer, R.Z.Valiev (Eds.), Nanomaterials by Severe Plastic Deformation, J.Wiley VCH Weinheim, Germany, 2003, pp.829-834.

[8] S.M.L. Sastry, S.V. Dobatkin, S.V. Sidorova, Metals 2 (2004) 28-35 (in Russian).

[9] S.V. Dobatkin, P.D. Odessky, R.Pippan et al, in: M.Zehetbauer, R.Z.Valiev (Eds.), Nanomaterials by Severe Plastic Deformation, J.Wiley VCH Weinheim, Germany, 2003, pp.804-809.

[10] S.V. Dobatkin, P.D. Odessky, R. Pippan et al, Metals 1 (2004) 110-119 (in Russian).

[11] M.V.Bobylev, E.G.Koroleva, P.A.Shtannikov, MiTOM 5 (2005) 51-55 (in Russian).

Note: The responsible translator for Englisch Language is the Author S. Dobatkin.