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MECHANICAL AND SERVICE PROPERTIES OF LOW CARBON STEELS PROCESSED BY SEVERE PLASTIC DEFORMATION

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The structure and properties of the 0,09 %C-Mn-Si-Nb-V-Ti, 0,1 %C-Mn-V-Ti and 0,09 %C-Mo-V-Nb low-carbon steels were studied after cold equal-channel angular pressing (ECAP). ECAP leads to the formation of partially submicrocrystalline structure with a grain size of 150 – 300 nm. The submicrocrystalline 0,09 %C-Mn-Si-Nb-V-Ti steel compared with the normalized steel is characterized by R_e higher more than by a factor of 2 and by the impact toughness higher by a factor of 3,5 at a test temperature of -40 °C. The plasticity in this case is somewhat lower. The high-strength state of the submicrocrystalline 0,1 %C-Mn-V-Ti and 0,09 %C-Mo-V-Nb steels after ECAP is retained up to a test temperature of 500 °C. The strength properties at 600 °C (i.e. the fire resistance) of these steels are higher by 20-25 % as compared to those of the undeformed steels. The strength of the 0,09 %C-Mo-V-Nb steel at 600 °C is substantially higher than that of the 0,1 %C-Mn-V-Ti steel.

Key words: severe plastic deformation (SPD), ECAP, low carbon steel, submicrocrystalline structure,mechanical properties.

Mehanička i uporabna svojstva niskougljičnih čelika poslije intenzivne plastične deformacije. Istražena je struktura i svojstva niskougljičnog čelika 0,09 %C-Mn-Si-Nb-V-Ti, 0,1 %C-MN-V-Ti i 0,09 %C-Mo-V-Nb poslije hladnog kanalno kutnog prešanja (KKP). KKP dovodi do ustroja submikrokristalne strukture s veličinom zrna 150-300 nm. U submikrokristalnom čeliku 0,09 % C-Mn-Si-Nb-V-Ti se povećala više od dva puta, a udarna žilavost pri temperaturi ispitivanja -40°C više od tri i pol puta usporedbom s normaliziranom stanju tog čelika. Plastičnost se pri tome nešto smanjuje. Visoka čvrstoća submikrokristalnog čelika 0,1 %C-Mn-V-Ti i 0,09 %C-Mo-V-Nb zadržava se do temperature ispitivanja 500 °C. Vatrootpornost ovih čelika pri 600°C 20-25% je više nego u nedeformiranom stanju. Čvrstoća čelika 0,09 % C-Mo-V-Nb pri 600 °C je znatno viša nego za čelika 0,1 %C-Mn-V-Ti.

Ključne riječi: intenzivna plastična deformacija (JPD), kutnokanalno prešanje (KKP), niskougljični čelik, submikrokristalna struktura, mehanička svojstva

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 pressing (ECAP) 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 ECAP 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 ECAP 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 ECAP 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 ECAP and heating [8].

The purposes of this work were: 1. to study the possibility to obtain the submicrocrystalline structure in low-carbon steels after cold ECAP pressing and subsequent heating; 2. to estimate the mechanical and service properties of submicrocrystalline (SMC) low-carbon steels including cold resistance (impact toughness at temperatures down to -40 °C) and fire resistance (strength at 600 °C).

For increasing strength properties (including the fireproof properties) at elevated temperatures, it is necessary

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Table 1. **Chemical composition of steel studied**

Steels	Mass / %										
	C	Si	Mn	Cr	S	P	Mo	V	Nb	Ti	B
0,09 %C-Mn-Si-V-Nb-Ti	0,09	0,62	1,3	0,1	0,008	0,012	–	0,08	0,01	0,021	–
0,1 %C-Mn-V-Ti	0,1	–	1,12	–	–	–	–	0,08	–	0,07	–
0,09 %C-Mo-V-Nb	0,09	0,32	0,63	0,076	0,004	0,007	0,08	0,06	0,03	0,026	–

to retain the ultrafine grains during heating. This seems to be possible due to the microalloying of the low carbon steel with the elements, which form fine thermally stable precipitates of carbonitride phases for the stabilization of the grain structure. The 0,1 %C-Mn-V-Ti steel with carbonitride strengthening, and the micro-alloyed molybdenum-containing 0,09 %C-Mo-V-Nb steel now in use as fireproof were studied in this work in submicrocrystalline state (Table 1).

The 0,09 %C-Mn-Si steel is usually used as structural steel in Northern climate. In this work this steel was micro-alloyed with V, Nb and Ti for additional grain refinement (Table 1).

EXPERIMENTAL PROCEDURE

The 0,09 %C-Mn-Si-Nb-V-Ti low-carbon steel (Table 1) was taken in ferritic-pearlitic initial state after normalization from temperature 920 °C. The ECAP of the samples of 20 mm in diameter and 80 mm in length was performed by cold deformation at T =20 and 300 °C for two passes at an angle of 90° between the channels by route B_c.

The 0,1 %C-Mn-V-Ti and 0,09 %C-Mo-V-Nb low-carbon steels (Table 1) was investigated in two initial states, ferritic-pearlitic (after hot forging) and martensitic (after quenching). The 0,09 %C-Mo-V-Nb steel was quenched from 950 °C (after holding for 30 min), while the 0,1 %C-Mn-V-Ti steel was quenched from 1180 °C. ECAP of the both steels for both initial states was performed at an angle of 120° between the channels on the samples of ∅10×60 mm in size at T=300 °C for N=6 passes by route B_c.

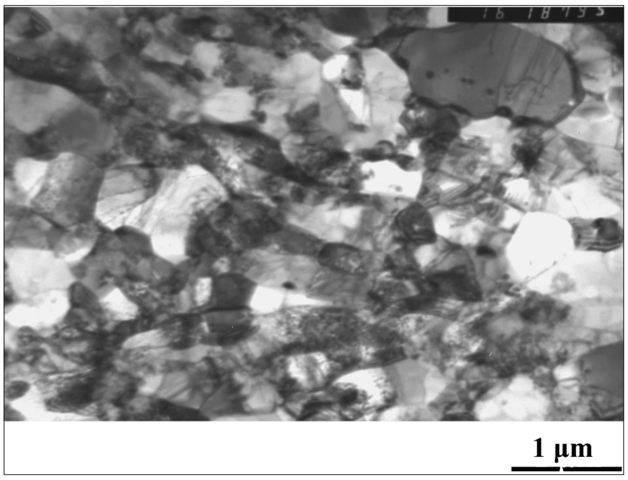

Figure 1. Structure of 0,09 %C-Mn-Si-Nb-V-Ti after ECAP and heating at 500 °C (holding time 5 h).

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 7×2,5×0,6 mm^3 in a gage base size. The initial tension rate was 5×10^{-3} s^{-1}.

RESULTS AND DISCUSSION

1. Cold resistance of the 0,09 %C-Mn-Si-Nb-V-Ti low carbon steel after ECAP and heating.

The ECAP of the 0,09 %C-Mn-Si-Nb-V-Ti low-carbon steel both at T=20 °C and T=300 °C leads to the orientation of grains along the sample axis at a certain angle to it. The electron-microscopic examination revealed two structure types: a cellular structure with a high dislocation density at cell boundaries and an oriented subgrained structure with a high density of free dislocations. Such structure is far from perfection and is not energetically profitable. For the formation of the equilibrium structure with a grain size at the submicron level, the samples after ECAP were heated to 400-750 °C and held for different durations. The study of the dependences of microhardness and grain size on temperature and holding time revealed their similarity for both ECAP routes at 20 and 300 °C.

The criterion of the possible formation of submicrocrystalline structure was taken as a decrease in microhardness without the appearance of metallographically detectable new grains. For this reason, we performed electron-microscopic examination of the samples after ECAP by both routes and holding at 500 °C for 5 h and at 550 °C for 30 min.

Holding at 500°C causes the formation of predominantly submicrocrystalline structure with a grain size of 200-400 nm in the samples deformed by both routes (Figure 1).

The dislocation density inside the grains is relatively low. A high-angle misorientation at grain boundaries was indicated by their banded contrast.

Holding at 550 °C for 0,5 h led to the formation of two structure types: a submicrocrystalline structure with a grain size of 200-400 nm and a grained structure with a grain size of 2-5 μm at the stage of secondary recrystallization. The average grain size after ECAP at 300 °C and holding at 550 °C (0,5 h) is somewhat higher.

Thus, the posed problem of the formation of the submicrocrystalline structure in the 0,09 %C-Mn-Si-Nb--V-Ti steel can be solved by ECAP at a temperature of 20 - 300 °C at N=2 and holding at 500 °C for 5 h.

The mechanical properties of the 0,09 %C-Mn-Si-Nb-V-Ti steel after ECAP differently change relative to those of the initial normalized steel: the yield strength increases almost by a factor of 3, the impact toughness decreases almost by a factor of two, and the elongation values become very low (Table 2).

Table 2. **Mechanical properties of 0,09 %C-Mn-Si-Nb-V-Ti after ECAP and heating at 500 °C (holding time 5 h).**

State	R_m / MPa	R_e / MPa	A / %	Z / %	KCV / MJ/m^2	
					+20 °C	-40 °C
Initial State (920 °C, air cooling)	505	340	16	-	0,22	0,13
ECAP (T=200 °C, N=2, φ=90 °C)	980	980	5,0	-	0,13	0,06
ECAP + heating 500 °C, 5 h	785	775	5,3	63,1	0,80	0,69

Holding at 500 °C for 5 h increases plasticity compared to that observed directly after ECAP and increases the impact toughness, especially at a test temperature of -40 °C (by a factor of 11,5). In this case, the strength value remains sufficiently high.

Thus, the submicrocrystalline 0,09 %C-Mn-Si-Nb-V--Ti steel compared with the normalized steel is characterized by R_e higher more than by a factor of 2 and by the impact toughness higher by a factor of 3,5 at a test temperature of -40 °C. The plasticity in this case is somewhat lower.

2. Elevated-temperature mechanical properties of the 0,1%C-Mn-V-Ti and 0,09%C-Mo-V-Nb steels after ECAP.

The ECAP was performed using the 0,1 %C-Mn-V-Ti steel in two initial states: ferritic-pearlitic and martensitic. Cold ECAP at 300 °C for N=6 by route B_c for both initial states leads to the formation of the mixed (cellular and subgrain) structures with isolated grains of submicron size. There are regions of oriented and equiaxed structures. The size of structural elements is 150-350 nm. For the initial martensitic state, the size of structural elements after ECAP is smaller than for the initial ferritic-pearlitic state.

With increasing test temperature, the yield strength and ultimate tensile strength are observed to monotonically decrease with simultaneously increasing plasticity of the steel (Figure 2).

After ECAP, the initially martensitic 0,1%C-Mn-V-Ti steel compared to the initially ferritic-pearlitic steel exhibits its higher strength properties in a test temperature range of 20…500 °C. At T_{test} = 600 °C, the yield strength R_e decreases to 150 MPa for both initial states.

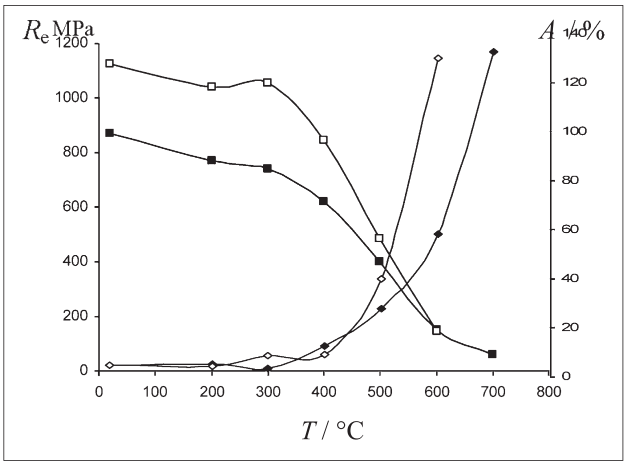

Figure 2. Temperature dependence of the mechanical properties of the 0,1%C-Mn-V-Ti steel after ECAP: (□,◇) initial martensitic state; (■,◆) initial ferritic-pearlitic state.

The ratio of yield strength values at temperatures 20 and 600 °C, $R_e^{600}/R_e^{20} \geq 0,5$ can be used as the criterion of fire resistance. The steel, especially in the initial martensitic state, is substantially softened at a test temperature of 600 °C (holding time of 0,5 h). For the high-strength state after ECA pressing, R_e^{600}/R_e^{20} = 0,13, and for the initially undeformed state, R_e^{600}/R_e^{20} = 0,23. At a test temperature of 500°C, R_e^{500}/R_e^{20} = 0.45 and 0,8, respectively. Thus, the 0,1%C-Mn-V-Ti steel after ECAP retains a high-strength state up to a test temperature of 500 °C. At T_{test} = 600 °C, the steel exhibits a substantial weakening.

For the study of the possibility to increase the fire resistance at the expense of SMC structure, it was decided to investigate the 0,09 %C-Mo-V-Nb steel, which belongs to the most fireproof steels. The ECAP (300 °C, N = 6, the angle between the channels is 120°) of the 0,09% C-Mo-V-Nb steel with the initial martensitic structure leads to formation of the structure similar to that obtained in the 0,1%C-Mn-V-Ti steel after ECAP (Figure 3).

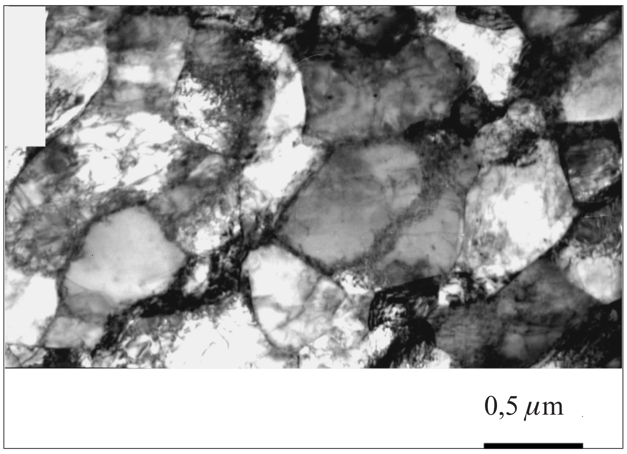

Figure 3. TEM image of the SMC structure of the 0,09 %C-Mo-V-Nb steel after ECAP.

The banded and equiaxed subgrain and cellular structures with isolated grains of submicron size are observed in the steel. The average size of structure elements is 0,3 μm.

The steel in the initial state after quenching and high-temperature tempering demonstrates high strength and plasticity at 20 °C (Table 3). With increasing temperature, the strength properties of the steel monotonically decrease with the retention of high plasticity.

Table 3. **Mechanical properties of the 0,09 %C-Mo-V-Nb steel**

State	T_{test} / °C	R_e / MPa	R_m / MPa	A /%
Initial state (water quenching from 920 °C - 0,5 h and subsequent tempering at 670 °C for 1 h)	20	550*	621	22,3
	200	484	558	18,5
	300	452	566	17,8
	400	431	565	19,3
	500	405	456	14,3
	600	253	282	18,8
Initial state + ECAP	20	904	934	4,8
	200	767	843	8,1
	300	723	811	7,9
	400	694	752	7,1
	500	540	575	9,3
	600	295	362	21,4

* - the value corresponds to the true yield strength YS_T (yield plateau);

The ECAP significantly (more than by a factor of 1,5) increases the room-temperature strength properties of the 0,09 %C-Mo-V-Nb steel (Table 3). The plasticity decreases, but remains sufficient for the technological application. The above-mentioned relationship of the strength properties is retained in the investigated material with increasing temperature to 400 °C and then abruptly decreases with simultaneous increase in plasticity. Thus, the ECAP of the 0,09 %C-Mo-V-Nb steel substantially increases the strength properties in a wide range of deformation temperatures.

The criterion of fire resistance R_e^{600}/R_e^{20} for the 0,09 %C-Mo-V-Nb steel is equal to 0,33 in the case, where R_e^{20} corresponds to the state after ECAP, and 0,54 in the case where this value corresponds to the yield strength of the undeformed state after quenching and high-temperature tempering. The corresponding values for 500 °C, R_e^{500}/R_e^{20}, are 0,6 and 0,98, respectively. This means that the yield strength of the 0,09 %C-Mo-V-Nb steel at T_{test} = 500°C after ECAP is equal to the yield strength of this steel at T_{test} = 20 °C after quenching and high-temeprature tempering. Note that the strength characteristics upon tension at a temperature of 600 °C are higher than those exhibited by the 0,1 %C-Mn-V-Ti steel. The 0,09 %C-Mo-V-Nb steel with the SMC structure compared to the structure realized after quenching and high-temperature tempering is characterized by the yield strength higher by 17 % and the ultimate tensile stress to failure higher by 28 % at T_{test} = 600 °C.

Thus, the high-strength state of the 0,1 %C-Mn-V-Ti and 0,09 %C-Mo-V-Nb steels after ECAP is retained up to a test temperature of 500 °C. The strength properties at 600 °C of the 0,1 %C-Mn-V-Ti and 0,09 %C-Mo-V-Nb steels with the SMC structure obtained by ECAP are higher by 20-25 % as compared to those of the undeformed steels. The strength of the 0,09 %C-Mo-V-Nb steel at 600 °C is substantially higher than that of the 0,1 %C-Mn-V-Ti steel. At T_{test} = 600 °C, the strength characteristics (i.e. the fire resistance) of the 0,09 %C-Mo-V-Nb steel are satisfactory both before and after ECAP.

CONCLUSIONS

1. The submicrocrystalline 0,09 %C-Mn-Si-Nb-V-Ti low-carbon steel after ECAP and heating at 500 °C (5 hours) compared with the normalized steel is characterized by R_e higher more than by a factor of 2 and by the impact toughness higher by a factor of 3,5 at a test temperature of -40 °C.
2. The high-strength state of the 0,1 %C-Mn-V-Ti and 0,09 %C-Mo-V-Nb steels after ECAP pressing is retained up to a tensile-test temperature of 500 °C. The strength properties at 600 °C of the 0,1 %C-Mn-V-Ti and 0,09 %C-Mo-V-Nb steels with the SMC structure obtained by ECAP are higher by 20-25 % as compared to those of the undeformed steels. The strength of the 0,09 %C-Mo-V-Nb steel at 600 °C is substantially higher than that of the 0,1 %C-Mn-V-Ti steel.

Acknowledgements

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Note: The responsible translator for English Language is S. Dobatkin.