STRENGTH AND SERVICE PROPERTIES OF STAINLESS Cr-Ni-Ti STEEL AFTER EQUAL CHANNEL ANGULAR PRESSING IN THE TEMPERATURE RANGE 200-400°C

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Abstract: *The research paper presents the improvement on the mechanical and service properties of the austenitic stainless 0.07% C-17% Cr-9% Ni-0.7%-Ti steel in the fully austenitic state obtained by equal-channel angular pressing (ECAP) at the temperatures of 200 °C and 400 °C. Subgrain oriented structure in the Cr-Ni-Ti steel after ECAP significantly enhances the strength characteristics of steel at satisfactory plasticity. The fatigue limit of steel after ECAP at T = 200 ° C is higher than that after ECAP at T = 400 ° C, increasing in comparison with the initial state by 2.2 and 1.7 times, respectively. It was revealed that severe plastic deformation by ECAP increases the friction coefficient of the material by the 1.1 and 1.7 times at T = 200 ° C and 400 ° C, respectively. Despite this, the wear rate after ECAP at T = 200 ° C and 400 ° C decreases by 7 and 40 times, respectively, compared to initial state.*

Keywords: austenitic stainless steel, equal-channel angular pressing (ECAP), ultra-fine grained structure, austenite, martensite, strength, fatigue, wear

1. Introduction

Austenitic stainless steels are one of the most widely used materials because of their high corrosion resistance and good formability. However, they exhibit a relatively low yield strength and poor tribological properties. Nowadays a lot of attention is paid to increasing the strength of stainless steel, using a variety of methods and approaches. The refinement of the structure is one of the effective approaches for increasing the strength. It can be produced by equal-channel angular pressing (ECAP) [1, 2]. Several researches have shown a significant improvement in the mechanical properties of stainless steels after ECAP [3 - 5]. For instance, the yield strength (YS) of stainless steels increased from 198 to 1100 MPa and 1300 MPa after 4 and 8 ECAP-passes, respectively [5]. At the same time, decrease of plastic properties after ECAP can be effectively improved by heat treatment [5]. In the study [6] was observed structure of the two samples of steels 316L and 316L(N) after four ECAP-passes with elongated grains and the high density of dislocation. Different microstructural features were revealed in different areas of the samples. They were formed both by sliding and twinning deformation mechanism. The increase of deformation twins density with an increase of the number of passes was shown in study [6]. Greger et al. [7] obtained SUS 316L steel after four ECAP-passes at 280 °C with twinning in the ultrafine grained microstructure, which the yield strength (YS) and the ultimate tensile strength (UTS) were 106.3 and 109.9 MPa, respectively, with the elongation to failure (EL) of 15%. The authors of the study [8] show that the combination of the high strength (to 1600 ± 30 MPa) with the endurable ductility (EL=17%) can be achieved by ECAP in the temperature range of 0 - 250 ° C.

The structure formation of the austenitic stainless steel 316L during high pressure torsion (HPT) in the temperature range from -196 ° C to 720 ° C was investigated in research [9]. It was found that the structure formation at different deformation temperatures similar to the evolution of the microstructure of other materials with the low stacking fault energy: the dominant mechanism of deformation at high temperature of deformation (T_{def}> 450 ° C) is the slip of dislocation, while the typical mechanism for medium deformation temperature (450 ° C> T_{def}> 20 ° C) is mechanical twinning. The mechanical twinning at low temperatures of deformation (20 ° C> T_{def}> -196°C) was replaced by deformation induced martensitic transformation γ (fcc) $\rightarrow$ ε (hcp) mechanism.

Steels of 316L - type contain metastable austenite, which can be transformed to martensite of deformation at temperature below M*d*. The sizeable fraction of deformation martensite increases with the applied load and leads to substantial increase in strength but decrease of corrosion resistance. The purpose of this work was to study the mechanical and service properties of the Cr-Ni-Ti steel in the fully austenitic state obtained by ECAP.

2. Materials and experiment

The chemical composition of the stainless Cr-Ni-Ti steel used in this study is shown in Table 1. The material in as-received condition is austenitized at 1050 °C for 1 hour and water quenched. ECAP was carried out on the billets 20 mm in diameter and 80 mm in length using a die with channels intersecting at 120° via route Bc, when the billet rotated by 90° between the passes [10]. The samples were processed by six ECAP - passes at temperatures 200 °C and 400 °C. The equivalent plastic strain applied to the billet per pass for the defined die geometry equals 0.9 (shear strain γ = 1.5) [11, 12].

Table 1: *Chemical composition of the stainless Cr-Ni-Ti steel*

Elements	C	Cr	Ni	Cu	Ti	Si	Mn	S,P
Amount [wt%]	0.07	17.3	9.2	0.2	0.7	0.6	1.4	0.003

The tensile and fatigue plate-type specimens with a 5.75 mm gage length and the cross-section 1 mm × 2 mm were shaped by spark erosion from the ECAP billets. The specimens were mechanically polished using SiC grit papers and diamond paste and electrolytically polished in a solution containing 100 g of chromic anhydride and 850 ml of orthophosphoric acid at 20 °C and 15 V for 5 min.

Static tensile tests were performed using an INSTRON 3380 tensile testing machine with a load capacity of 100 kN. The high-cycle fatigue (HCF) tests were carried out under repeated tension conditions on an ElectroPulsTM 3000 servo-hydraulic machine with a load capacity of 100 kN operate at 30 Hz testing frequency and a stress ratio R = 0.1.

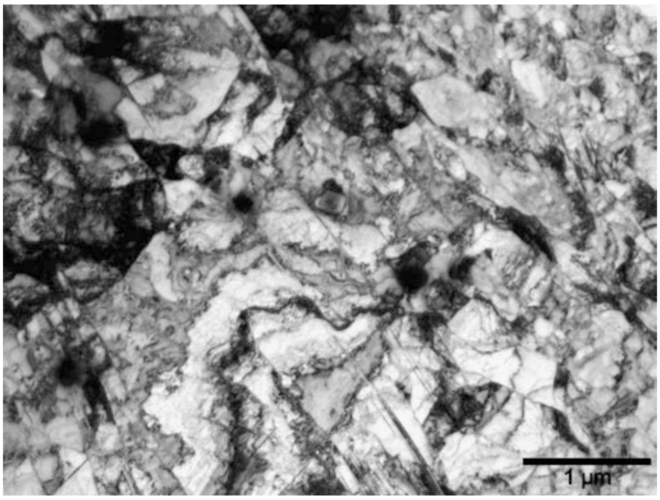
The microstructure was investigated using an Olympus PME 3 optical microscope and a JEM- 1400 transmission electron microscope operated at 120 kV in longitudinal direction. The samples for the metallographic analysis were electrolytically etched in aquafortis at 20 °C and 3V. An X-Ray diffraction (XRD) analysis was carried out on a DRON 4.07 diffractometer.

Dry-sliding tribological tests of the stainless Cr-Ni-Ti steel were carried out on multipurpose CETR UMT-3MO "pin on-disc" wear tester in the air at room temperature. AISI52100 steel disk (60 HRC hardness) of 100 mm in diameter and 10 mm in thickness were employed as a counterface. The stainless steel pins were 5 mm in diameter and 10 mm in thickness. Steady-state wear rates were calculated by weight loss measurements. Friction coefficient was also measured during the wear, which increased at the beginning of the test and reached a steady-state value.

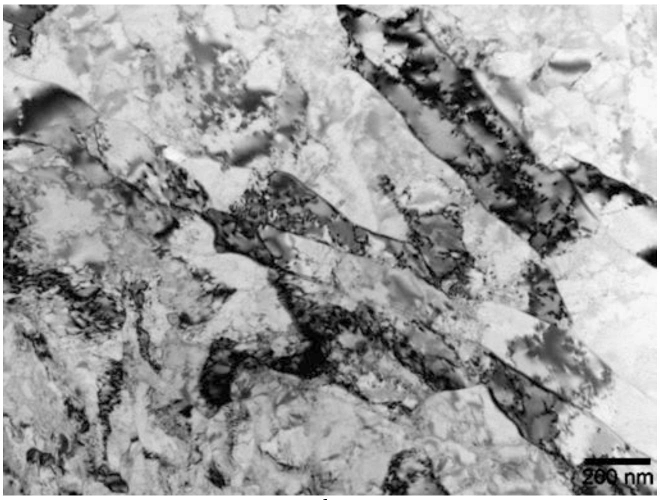
Taking into account the possibility of the biomedical applications in orthopedic surgery, and that the peak normal stress of joints is in the range of 0.5-5.0 MPa [13] pin-on-disk wear tests were performed in air under load of 10 N, which corresponds to the nominal contact pressure in the range of peak normal stress of joints. A constant slow sliding velocity of 0.1 m/s was selected to suppress flash heating at the contacting surfaces.

3. Results and discussion

The microstructure of the Cr-Ni-Ti steel after the heat treatment reveals an average grain size of about 20-30 μm. TEM analysis of the stainless Cr-Ni-Ti steel after ECAP at T = 200 ° C showed the formation of nano- and submicrocrystalline structure with dispersed deformation twins (Figure 1a, b). The presence of predominately high angle boundaries, ie, grain structure was defined by point reflections of electron diffraction ring and banded contrast at the boundaries.



a



b

Fig. 1 *TEM images of the microstructure of stainlessCr-Ni-Ti steel after ECAP at T=200°C (a,b), and after ECAP at T=400°C (c,d)*

The grain-subgrain structure with a size of the structural elements of 100-300 nm after ECAP at T = 400 °C was formed. The grain-subgrain structure is partially oriented. The separated grains with high angle boundaries are formed too, that can be defined by banded contrast.

The XRD analysis revealed the presence of the completely austenitic phase in the stainless Cr-Ni-Ti steel after ECAP at T = 200 ° C and 400 ° C at 6 passes.

The thermal stability of the stainless Cr-Ni-Ti steel after ECAP by changes of the microhardness depending on the annealing temperature in the range of 20 - 800 ° C was studied. Figure 2 shows the dependence of microhardness of stainless steel after ECAP from the annealing temperature. A sharp drop in microhardness above 650 ° C was observed. It can be explained by a significant increase of the grains size.

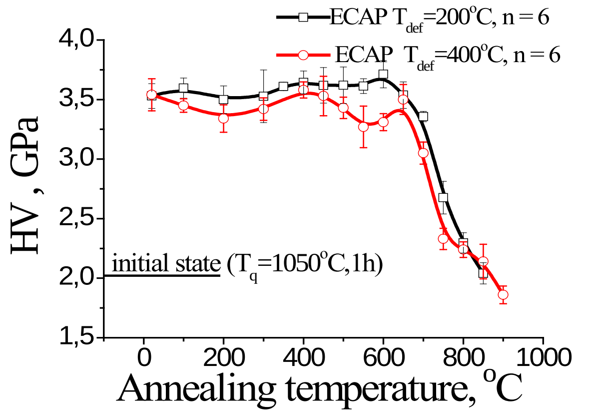


Fig. 2 *The microhardness vs annealing temperature of the Cr-Ni-Ti steel after ECAP at T=200°C and 400°C*

Severe plastic deformation by ECAP of the austenitic stainless Cr-Ni-Ti steel significantly increases the strength characteristics (YS, UTS) and at the same time leads to some reduction in ductility (Fig. 3). The level of ductility and strength of the samples after ECAP at temperatures 200 and 400 ° C is practically identical. It was noticed that the increased density of dislocations and twins during ECAP at 200°C is compensated then by a decrease in the size of the structural elements at 400 ° C. As a result both samples have nearly the same strength.

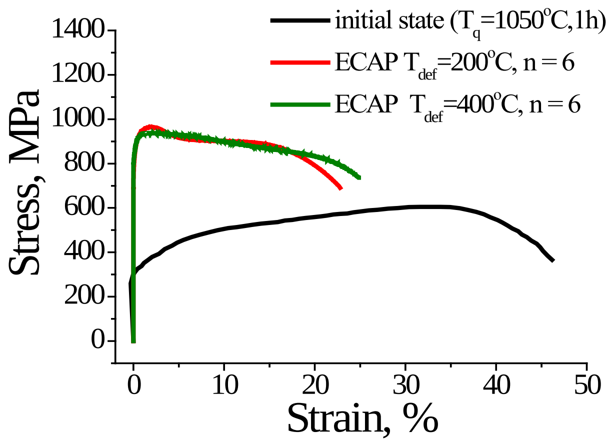


Fig. 3 *The mechanical properties of the Cr-Ni-Ti steel in the initial state, after ECAP at T=200°C and T=400°C*

The fatigue strength is an important criteria for evaluating stability of the structural state of the material and its workability under cyclic loads. Figure 4 shows the curves of the high-cycle fatigue tests of the stainless steel samples in the initial state and after ECAP at 200 °C and 400 °C. The high-cycle fatigue curves show that fatigue strength of the steel at 10^7 cycles after ECAP at 200 ° C (600 MPa) is higher than that after ECAP at 400 ° C (475 MPa) i.e. it increases, compared with the initial state (275 MPa).

The reason for increasing the fatigue strength should be sought not only in the initial structure, but also in the structural and phase transformations that take place during the cyclic tests.

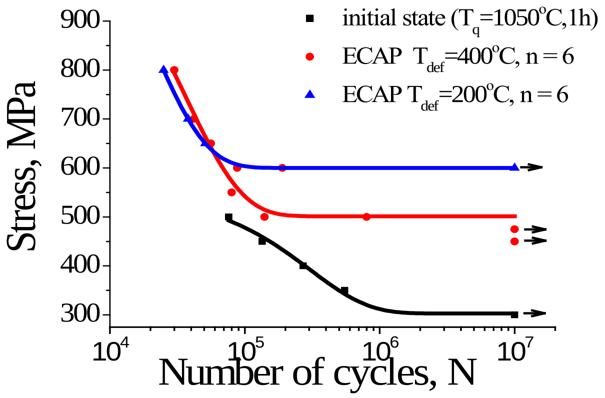


Fig. 4 *The high-cycle fatigue tests of the Cr-Ni-Ti steel in the initial state, after ECAP at T=200°C and 400°C*

Tribological tests revealed that severe plastic deformation by ECAP increases the coefficient of friction of the austenitic stainless Cr-Ni-Ti steel at T = 200 ° C and 400 ° C by the 1.1 and 1.7 times, respectively (Figure 5a). In this case, the wear rate after ECAP at T = 200 ° C and 400 ° C decreases by the 7 and 40 times, respectively, compared with the initial state (Figure 5 b).

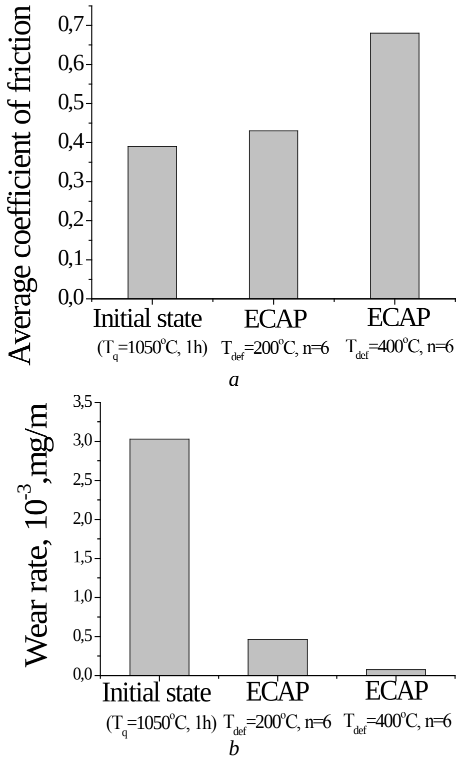


Fig. *5 The friction coefficient (a) and the wear rates of Cr-Ni-Ti steel in the initial state, after ECAP at T=200°C and 400°C*

The austenitic stainless Cr-Ni-Ti steel after ECAP at T = 400 ° C shows the maximum wear resistance, which changes the mechanism of frictional effects, and, in turn, affects the reduction of mass wear rate.

The research of friction and wear processes in steels with different structures and levels of strength in [14-17] identified common patterns of improvement of this process.

Reasons for changes in the wear mechanism of Cr-Ni-Ti steel after ECAP at T = 400 ° C should be looked in a more closely study of the structure and phase composition of the surface layers of samples subjected to friction.

4. Conclusions

1. It was revealed that predominantly oriented subgrain structure with the 100-350 nm size of the structural elements and enhanced density of dislocation and 5-10 nm in thick deformation twins in austenite is formed in the samples during ECAP at 200 ° C. The grain-subgrain structure with the average grain size about 100 nm, the lower dislocation density and deformation twins is formed after ECAP at 400 ° C.

2. Subgrain oriented structure in the Cr-Ni-Ti steel significantly enhances the strength characteristics of steel with satisfactory plasticity after ECAP at T = 200 ° C: UTS = 966 MPa and EL = 23%, compared with initial state: UTS = 601 MPa, EL = 48%. ECAP at T = 400 ° C leads to a certain reduction of strength properties (UTS = 937 MPa) compared with the ECAP at 200 ° C and to slight increase in ductility (EL = 25%).

3. The fatigue strength of steel after ECAP at T = 200 ° C (600 MPa) is higher than that after ECAP at T = 400 ° C (475 MPa), increasing in comparison with the initial state by 2.2 times and 1.7 times, respectively.

4. It was revealed that severe plastic deformation by ECAP increases the friction coefficient of the material by the 1.1 and 1.7 times at T = 200 ° C and 400 ° C, respectively. Despite this, the wear rate after ECAP at T = 200 ° C and 400 ° C decreases by 7 and 40 times, respectively, compared to initial state.

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