Effect of ECAP on structural, mechanical and functional characteristics of the austenitic Cr-Ni-Ti steels

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Abstract. In this study, equal-channel angular pressing (ECAP) of austenitic Cr-Ni-Ti stainless steel was carried out. Effect of ECAP on the evolution of the microstructure and mechanical and service properties of the Cr-Ni-Ti steel was investigated. The microstructure in the completely austenitic state processed by 6 passes of ECAP at 200 °C was obtained and examined by optical and transmission electron microscopy. Mechanical properties were determined by the Vickers micro-hardness measurements and tensile testing. Fracture toughness was evaluated and tribological properties of Cr-Ni-Ti stainless steel processed by ECAP were studied. It was revealed that submicrocrystalline structure with an average size of structural elements of 100 nm produced by ECAP significantly increased the strength properties of Cr-Ni-Ti stainless steel than that in the quenched state. After ECAP the ultimate tensile strength increased by 1.7 times, the fatigue limit by 1.5 times than that in the initial state. It was revealed that Cr-Ni-Ti steel after ECAP demonstrates 2.9 times higher fracture toughness than that in the quenched state. It is established that ECAP does not significantly affect the coefficient of friction and mass wear.

1. Introduction

Austenitic Cr-Ni-Ti stainless steels have many advantages, such as high strength, corrosion-resistance and the possibility of adjusting structure due to phase transformations. They already widely used in many industrial applications up to critical components of nuclear reactors and in medicine. However, the serious disadvantage of these steels is the low yield strength, which can be significantly increased by refinement of the structure to the submicrocrystalline state by applying the methods of severe plastic deformation (SPD). The possibility of obtaining a submicrocrystalline structure in austenitic stainless steels by the method of equal-channel angular pressing (ECAP) is reliably established in many studies [1, 2]. In particular, it has been shown a significant improvement in the mechanical properties of stainless steels after ECAP [2]. In [2] the yield strength of austenitic stainless steels increased up to 1300 MPa after ECAP, with effectively improved ductility after ECAP and heating. After four passes of ECAP oriented structure of the austenitic stainless steel was observed [3] with the high density of dislocation and different microstructural features formed both by sliding and deformation twinning mechanism. Obtained ultrafine grained microstructure with high density of twinning in SUS 316L steel [4] after four passes of ECAP at 280 °C shows the yield strength of 1063 MPa, the ultimate
tensile strength of 1099 MPa, and the elongation to failure of 15%. The authors of the study [5] show that the combination of the high strength (1600 ± 30 MPa) with the endurable ductility about 17% can be achieved by ECAP at the temperature up to 250 °C.

Cr-Ni-Ti steel is a promising material for creating by SPD in it an UFG structure due to a high initial ductility. SPD at room temperature of such steel causes the martensitic transformation. The fraction of strain-induced martensite increases with the increasing ECAP passes and leads to increase in strength but to decrease in corrosion resistance. The aim of this study was investigation of the mechanical and service properties of the Cr-Ni-Ti stainless steel in the completely austenitic state obtained by ECAP at 200 °C as this temperature is higher than that for formation of deformation martensite.

2. Experimental

For this study we choose the austenitic stainless steel with the chemical composition shown in table 1. This steel in the hot-rolled state was quenched at 1050 °C for 1 hour with water cooling. We made the billets, that are 20 mm in diameter and 90 mm in length. They were processed by six passes of ECAP at temperature 200 °C using a die with channels intersecting at 120° via route Bc. It means that the billet was rotated by 90° after each pass (figure 1) [6]. The equivalent plastic strain applied to the samples per pass equals 0.9 (shear strain γ = 1.5) [7, 8].

| Elements | C  | Cr  | Ni  | Ti  | Cu  | Si  | Mn  | S, P | Fe  |
|----------|----|-----|-----|-----|-----|-----|-----|-----|-----|
| Amount (wt.%) | 0.07 | 17.3 | 9.2 | 0.7 | 0.2 | 0.6 | 1.4 | 0.003 | balance |

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

The microstructure analysis was conducted with Olympus PME 3 optical microscope and a JEM-2100 transmission electron microscope operated at 200 kV. The samples for the metallographic analysis were electrolytically etched in HNO₃. XRD analysis was performed by a DRON 4.07 diffractometer using Co Kα radiation at 40 kV and 30mA.

Specimens for the tensile and fatigue testing with a 5.75 mm gage length and the cross-section 1 mm × 2 mm were shaped from the ECAP billets by spark erosion, mechanically grounded and electropolished to minimize the errors.

Tensile testing was carried out by INSTRON 3380 machine with a load capacity of 100 kN. The high-cycle fatigue tests were performed under repeated tension conditions using a servo-hydraulic machine ElectroPulsTM 3000 with a loading capacity of 100 kN that was operated at testing frequency of 30 Hz and a stress ratio R = 0.1.
Compact disk shaped specimens with the dimensions about ø20×3.5 mm were used to estimate the fracture toughness in accordance with ASTM E399. The standard technique was modified to take into account the small size of the sample - the crack opening displacement sensor was mounted on the stem of the fatigue machine. During the tests, the software compensation of compliance was performed by subtracting its values obtained for the sample without a notch. A preliminary fatigue crack at the notch tip was generated with a decreasing stress intensity factor in order to prevent the formation of plastic deformation zone at the crack tip that affects the fracture toughness value.

Tribological tests were carried out by UMT-3MT tribometer at applied loads of 80N and 160N in the air at room temperature with estimation of friction coefficient, wear mass loss and acoustic emission (AE) amplitude of the samples of Cr-Ni-Ti steel after ECAP and quenching. Dry sliding wear tests were performed in condition of reciprocating pin-on-flat wear with a speed of 3 mm/s. AISI 3Cr12 steel pins (220 HB) were employed as the counter face with a diameter of 10 mm.

3. Results and Discussion

The microstructure of the Cr-Ni-Ti steel in the quenched state had an average grain size of about 20-30 μm (figure 2a). After ECAP, metallographically revealed extended initial granular structure (figure 2b). No structure was etched inside grains.

![Figure 2](image-url)

**Figure 2.** Light micrograph of structure of Cr – Ni-Ti steel (a) quenched state (b) state after ECAP at 200°C, n=6

TEM analysis shows that the intergranular structure after ECAP is oriented with an average size of structural element of 10⁴±3 nm. It can be shear bands (figure 3a), disperse deformation twins (figure 3b), oriented subgrains and grains.

The reflection diffusion in the ring electron diffraction pattern indicates mainly low-angle boundaries. The refinement of the structure proceeds through the formation of shear bands with a high dislocation density inside and along the boundaries. Inside the shear bands, the formation of a cellular structure proceeds by forming cross bulkhead due to dislocation sub-boundaries and high density of disperse deformation twins of 28 ± 3 nm in thickness (figure 3b). In addition, the formation of the grain structure from the subgrains with high dislocation density occurs by sliding (figure 3 c, d). Banded contrast confirms that grains with size about 60 nm with high-angle boundaries are formed.

The XRD analysis revealed the completely austenitic state of the stainless Cr-Ni-Ti steel after ECAP at T = 200 °C at 6 passes (figure 4).

The thermal stability of the Cr- Ni – Ti steel after ECAP was studied by the determination of microhardness after annealing in the temperature range between 100 and 850 °C (figure 5).
Figure 3. TEM images of the microstructure of stainless Cr-Ni-Ti steel after ECAP at \( T = 200^\circ C \); (a-c) bright field, (d) dark field.

A slump in microhardness above 650 ° C can be explained by increase of the grains size.

Figure 4. X-ray diffractions of the Cr-Ni-Ti steel produced by ECAP at \( T = 200^\circ C \).

Figure 5. The microhardness vs annealing temperature of the Cr-Ni-Ti steel after ECAP at \( T = 200^\circ C \).

The strength characteristics (\( \sigma_{YS}, \sigma_{UTS} \)) are significantly increased by severe plastic deformation after ECAP of the austenitic Cr-Ni-Ti stainless steel, but at the same time it leads to some reduction in
ductility (table 2, figure 6). The fatigue strength is considered to be an important criterion to estimate stability of the structural state of the steel and its workability under cyclic loads. Figure 7 shows the curves of the high-cycle fatigue tests of the stainless steel samples in the initial state and after ECAP at 200 °C. The high-cycle fatigue curves show that fatigue strength of the steel at $10^7$ cycles after ECAP at 200 °C is higher than that in the initial state (table 2, figure 7). It should be noted that the samples after ECAP demonstrate the results for the ratio of the fatigue limit to the ultimate tensile strength ($\sigma_f/\sigma_{UTS}$), indicating the working capacity of the material under cyclic loading similar to the initial state (table 2).

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 deformation.

**Table 2.** The mechanical properties of the Cr-Ni-Ti steel in the initial state and after ECAP at $T=200^\circ$C.

| State | $\sigma_{UTS}$, MPa | $\sigma_{YS}$, MPa | $\delta$, % | $\sigma_f$, MPa | $\sigma_f/\sigma_{UTS}$ | $K_Q$, MPa$\cdot\sqrt{m}$ |
|-------|-------------------|-------------------|---------|----------------|---------------------|---------------------|
| Initial state | 605 | 313 | 46 | 425 | 0.70 | 23.1 |
| ECAP at $T=200^\circ$C, n=6 | 1047 | 1038 | 21 | 656 | 0.62 | 67.4 |

One of the important for the strength of materials parameters is the fracture toughness. The reason is in defects and discontinuities of different origin in real structures, which can turn into cracks in the process of operation. For materials with an ultrafine-grained structure, an evaluation of this characteristic is also an actual problem [10, 11].

For correct determination of the fracture toughness $K_{IC}$ it is necessary that the both sample size $B$ and the crack length $a$ exceeds the calculated value according to:

$$B \text{ and } a > 2.5 \cdot (K_Q \cdot \sigma_{YS})^2$$

here $K_Q$ - calculated value of stress intensity factor, $\sigma_{YS}$ – yield strength. This study showed that the condition for the valid estimation $K_{IC}$ is not satisfied due to the small specimen size. Therefore, comparing the fracture toughness of specimens with in initial state and after ECAP were performed by using the $K_Q$ value. It was found that Cr-Ni-Ti steel after ECAP has higher fracture toughness (2.9 times) as compared with the quenched state (table 2).

**Figure 6.** The mechanical properties of the Cr-Ni-Ti steel in the initial state, after ECAP at $T=200^\circ$C.

**Figure 7.** The high-cycle fatigue tests of the Cr-Ni-Ti steel in the initial state, after ECAP at $T=200^\circ$C.
The influence of the ECAP on the tribological characteristics, such as mass wear and friction coefficient as well as parameters of acoustic emission registered in the course of friction was studied.

Figure 8 shows a graph of the mass wear of the studied samples. At low times (t < 200 min) of frictional loading, the mass wear for these samples is approximately the same, regardless of the contact pressure. At longer times (t > 200 min), an increase in the frictional load to 160N leads to a slight increase in mass wear. From the obtained results it follows that the ECAP does not affect mass wear of studied steel.

Figure 8. Mass wear of Cr-Ni-Ti steel at loads of 80N and 160N: 1-ECAP, 80N; 2-Initial, 80N; 3-ECAP, 160N; 4-Initial, 160N.

Figure 9. The time dependences of the coefficient of friction of Cr-Ni-Ti steel at loads of 80N and 160N: 1-ECAP, 80N; 2-Initial, 80N; 3-ECAP, 160N; 4-Initial, 160N.

The graphs of the friction coefficient versus the wear time are shown in figure 9. At the initial stage at load of 80 N, the coefficient of friction in the sample after ECAP is greater than in the initial state. At load of 160 N, this difference is significantly smaller and increases substantially with prolonged friction. At that, the friction coefficient in the quenched sample is greater than in the sample after ECAP.

Measurements of the acoustic emission amplitude \( U_{AE} \) revealed the following. In the sample after the ECAP at a friction load of 80 N at the initial stage the \( U_{AE} \) values changes periodically and increases with increasing wear time (figure 10). In the sample after quenching, the periodicity of the AE signals were also observed, but their amplitudes were much lower, due to the greater plasticity of the steel in the initial state.

Figure 10. Dependences of AE amplitudes \( U_{AE} \) on wear time in samples after ECAP and after quenching at loads of 80N and 160N.
The periodicity of acoustic emission signals at a load of 160N, similar to that observed in tests with a load of 80N, was not detected.

The observed periodicity of AE signals at 80N can be related to processes of hardening and softening within the process zone and separation of the fragments of the surface layer of the sample during the friction tests. As the friction time increases, the friction paths harden, which probably leads to a change in the periodicity of the acoustic emission signals. Thus, for \( t > 104 \) s, the period of AE signals has increased by 3 times.

An increase in the contact pressure up to 160N leads to an increase in the amplitude of the AE signals in the sample after quenching to a greater extent than in the specimen after ECAP. The reason is probably a larger plasticity of the material after quenching. It should be noted that changes in the coefficient of friction \( k \) and acoustic emission signals \( U_{AE} \) are correlated. The observed changes in the friction coefficient and the AE amplitudes require further structural study of the process zone at different stages of testing and at various loads.

4. Conclusions
During this research the effect of severe plastic deformation is not only in a significant increase of strength properties with satisfactory plasticity, but also in simultaneous increase in service properties, such as fatigue properties and fracture toughness was established.
1. After ECAP at 200 °C it was revealed that predominantly oriented subgrain structure with the average size of the structural elements about 104 nm and high density of dislocation and deformation twins about 28 nm in thick in austenite is formed.
2. Obtained structure of the Cr-Ni-Ti steel significantly enhances the strength properties after ECAP at \( T = 200 \) °C \((\sigma_{UTS} = 1047 \text{ MPa})\) compared with quenched state \((\sigma_{UTS} = 605 \text{ MPa})\).
3. The fatigue limit of austenitic stainless steel after ECAP at \( T = 200 \) °C is higher than that in the quenched state by 1.5 times.
4. It was revealed that the ultrafine grained Cr-Ni-Ti steel demonstrates by 2.9 times higher fracture toughness \( K_I \) than that in the quenched state.
5. It was established that ECAP does not significantly affect the coefficient of friction and mass wear.
6. The amplitude of the acoustic emission signals increases with increasing wear time, and maximum growth occurred with testing samples in the initial state at the load of 160N.
7. The observed periodic character of the acoustic emission signals at the load of 80N can be explained by strengthening-softening processes within the friction paths of the samples.

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