Structure and low-cycle fatigue of steel AISI 316 after ECAP

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ABSTRACT

Purpose: Main aim of this paper is to describe the plastic deformation executed by ECAP on low cycle fatigue of steel AISI 316. Among others was attention fixed on mechanical properties after this treatment.

Design/methodology/approach: Experiments were planned and realised at the temperature ranging from room temperature up to 280 °C. After application of deformation the structure was investigated in dependence on accumulation of deformation and deformation temperature as well as abovementioned final properties.

Findings: Accumulated real (logarithmic) deformation varied from the value 2 to 8. Investigation of structure by electron microscopy was made with use of microscope JEOL JEM 2100. Mechanical properties were investigated by conventional tensile test and penetration test. Selected samples were subjected to low-cycle fatigue. Statistic evaluation of angular disorientation and of size of grains/sub-grains was also made with use of electron diffraction (EBSD) in combination with scanning electron microscope FEG SEM Philips.

Practical implications: The technology ECAP was applied on austenitic steel AISI 316. It was verification of ECAP application possibility on steel AISI 316 importantly for following applying on similar kinds of steel, because ECAP technology influence on fatigue properties was confirmed.

Originality/value: It can be predicted on the basis of obtained results that, contrary to low-cycle fatigue the ultra-fine grained material will manifest at fatigue load in the mode of constant amplitude of stress higher fatigue characteristics, particularly fatigue limit.

Keywords: Fatigue; Steel AISI 316; ECAP

1. Introduction

It is well known a positive influence of ECAP technology on final material properties namely non ferrous metals but steel as well. However not many works is focused on achieved fatigue properties after that treatment. This paper wants to contribute to knowledge distribution about austenitic stainless steel AISI 316 behaviour under ECAP.

2. Experimental

A series of samples made of austenitic stainless steel AISI 316 was processed by the ECAP technology. Basic chemical composition is given in the Table 1 and mechanical properties in the Table 2. The samples were manufactured with the following dimensions: φ 12 mm, length 60 mm. They were pushed through the ECAP matrix by 2 to 8 passes [1].
Matrix had channel diameter 12 mm and angle 105°. Pressure in the matrix varied around approx. 740 MPa. Temperature of extrusion varied from room temperature up to 280°C. After extrusion material was taken from the samples for metallographic testing and testing bars were manufactured for testing of mechanical properties. In order to expand the existing findings the testing bars were exposed to intensive magnetic field and impact of magnetic field on change of mechanical properties was investigated by tensile test. The sample was after eight passes subjected to structural analysis.

Ten samples were determined for investigation of influence of the ECAP technology on fatigue properties. Individual samples were subjected to different number of passes: 3 pieces had 4 passes, 4 pieces had 5 passes and 3 pieces had 6 passes. Test samples for testing of low-cycle fatigue had diameter of the measured part 5 mm and overall length 55 mm [2].

Table 1.
Basic chemical composition of the steel (%)  
| C  | Mn | Si | P  | S  | Cu | Ni | Cr | Mo  |
|----|----|----|----|----|----|----|----|-----|
| 0.03 | 1.64 | 0.18 | 0.011 | 0.007 | 0.06 | 12.5 | 17.6 | 2.4 |

Table 2.
Mechanical properties of the steel AISI 316 before ECAP (20°C)  
| Steel grade | E  | Rp0.2 | Rm | A  | Z  | KV | HB/HV |
|-------------|----|-------|----|----|----|----|--------|
| AISI 316    | 216 | 330   | 625 | 45 | -  | 90 | 210/- |

Amplitude of plastic deformation was the key factor for control of fatigue process. The following equation was used for the dependence \( \varepsilon_{ap} = \varepsilon_f (2N_j)^c \) [3,4]:

\[
\varepsilon_{ap} = \varepsilon_f (2N_j)^c
\]

where \( \varepsilon_f \) is coefficient of fatigue ductility, \( c \) is exponent of the service life curve.

### 3. Results and their analysis

#### 3.1. Structure

Structures were analysed from the viewpoint of the course of strengthening and restoring processes. Fig. 1 documents deformed sub-structure of the steel AISI 316 after ECAP deformation by 4 to 8. Metallic matrix contained sub-grains of uneven size. Size of sub-grains was in most cases smaller than 0.1 µm, only exceptionally some sub-grains/grains of the size of approx. 0.5 µm were observed.

Density of dislocations in metallic matrix was very high, presence of particles of precipitate was not found. In cases when neighbouring grains showed approximately identical diffraction contrast, it can be expected that angle of disorientation is only several degrees, while in case of significant changes of contrast rather high angular disorientation is probable. Fig. 2 documents a diffraction pattern, which was obtained from the area with diameter of approx. 1 µm. Occurrence of discontinuous circles and at the same time azimuthal blurring of diffraction traces evidences the fact that big amount of fine sub-grains/grains with more or less different crystallographic orientation was present in the investigated area.

![Fig. 1. Structure of steel AISI 316 after ECAP. Logarithmic deformation: 4 (a), 6 (b) and 8 (c)](image)

![Fig. 2. Diffraction pattern and structure of steel AISI 316 after ECAP. (Log. deformation: 8)](image)

Austenitic matrix often contained deformation bands, which were formed during the ECAP deformation, see Figures 1 and 2b. Deformation bands in austenitic steels can be formed by irregularly overlapping tiered errors, deformation twins or \( \alpha \)-martensite. These deformation bands are formed along octahedral planes \{111\}\(\gamma\) of austenitic matrix. It was proved with use of electron microscopy that in majority of cases these are deformation twins, nevertheless, presence of distinct stretching of reflections intensity ("streaking") in directions \{111\}\(\gamma\) proves frequent occurrence of crystallographic defects in these formations [5,6]. Width of deformation bands was very variable. In some areas intersecting systems of deformation bands occurred, which were formed at several planes of the type \{111\}\(\gamma\), see Figures 2b and 2c. Points of intersection of deformation bands generally represent preferential points for formation of particles of \( \alpha' \)-martensite. However, electron diffraction analysis did not confirm occurrence of \( \alpha' \)-martensite in these areas [7-9]. Occurrence of \( \alpha' \)-martensite in investigated sample was not confirmed even by X-ray diffraction analysis. Deflection (deformation) of deformation bands was in many cases quite distinctly visible in pictures in light field. This evidences the fact that deformation bands formed during the ECAP deformation were further deformed during next passes. Sub-grains with high density of dislocations were usually aligned along deformation bands.

#### 3.2. Mechanical properties

Samples after ECAP with number of passes (4, 5, 6) were used for investigation of influence of the ECAP technology on fatigue properties of the steel AISI 316 with special focus on the
area of low-cycle fatigue. In order to expand the existing findings the testing bars were exposed to intensive magnetic field and impact of magnetic field on change of mechanical properties was investigated by tensile test. Results of tensile testing, which were used for investigation of impact of intensive magnetic field on mechanical properties are given in the Table 3.

Table 3. Influence of magnetic field on mechanical properties of the steel AISI 316

| Designation        | $E$ [MPa] | Rp0.2 [MPa] | Rm [MPa] | $Z$ [%] |
|--------------------|-----------|-------------|----------|---------|
| 1                  | 190837    | 271.3       | 586.2    | 80.6    |
| 2                  | 200717    | 280.6       | 586.2    | 80.6    |
| Magnetic field     | 1188276   | 283.1       | 588.5    | 81.5    |
| Magnetic field     | 2201817   | 278.4       | 587.6    | 80.6    |
| Magnetic field     | 3195199   | 282.1       | 592.0    | 80.8    |

It follows from results of tensile tests that influence of magnetic field on mechanical properties determined by tensile test was not confirmed in investigated material. Minor differences in individual mechanical properties can be attributed to the scatter of mechanical properties within the frame of polycrystalline materials.

Mechanical properties change in dependence on numbers of passes, strength properties (Rp0.2 and Rm) distinctly increase, plastic properties described by narrowing almost do not change (Table 4). Intensity of increase in Rp0.2 and Rm is shown in the Fig. 3.

Micro-structural condition for increase of strength properties in investigated steel is fine grain and its stability. Several methods for grain refining and limitation of its growth are known at present – phase transformations, re-crystallisation, big plastic deformations (deformation of alloys with duplex structure, distribution of phases in duplex alloys, dispersion segregated particles), etc. Selection of methods of grain refining and slowing of its growth is in individual cases given by state and properties of structure [10].

Increase of strength properties in dependence on grain size is determined by the Hall-Petch relation:

$$\sigma_s = \sigma_0 + k_\gamma d^{-1/2},$$

(2)

where $\sigma_s$ = the particle friction stress, and it is the yield stress for the limit $d \to \infty$, $k_\gamma$ = the slope of the line and it is known as the dislocation locking parameter, which represents the relative hardening contribution due to grain boundaries.

For ordinary grade the following values are usually given $\sigma_0 = 70 - 104$ MPa and $k_\gamma = 18.1$ MPa mm$^{1/2}$.

### 3.3. Tests of low-cycle fatigue

Testing specimens for determination of the Manson–Coffin curve and curve of deformation strengthening were prepared from extruded samples after 2 to 6 ECAP passes. Apart form extruded samples the initial state was tested as well. The aim was to determine influence of number of ECAP passes on shape and position of the Manson-Coffin curve and curve of deformation strengthening. Altogether 10 samples were processed after application of the ECAP technology (3 samples after 4 passes and 6 samples after 4 and 4 samples after 5 passes) and 12 samples with initial structure [11].

| Number of ECAP passes | Rp$_{0.2}$ [MPa] | R$_m$ [MPa] | E [MPa] | A [%] | Z [%] |
|-----------------------|------------------|-------------|---------|-------|-------|
| Initial state         | 330              | 590         | 190 000 | 60    | -     |
| 2                     | 899              | 916         | 179 215 | 22    | 68    |
| 3                     | 970              | 998         | 180 125 | 15    | 60    |
| 4                     | 1 063            | 1 099       | 179 819 | 15    | 60    |
| 5                     | 1 103            | 1 140       | 182 028 | 15    | 60    |

Fig. 3. Influence of number of passes on strength properties of steel AISI 316

Test of low-cycle fatigue were performed according to the standard ASTM E 606 at laboratory temperature on servo-hydraulic testing equipment MTS 100 kN by „hard“ method of load in alternate traction – pressure. During these tests a constant amplitude of total deformation $\varepsilon_{ac}$ was preserved. Tests of low-cycle fatigue were realised at constant rate of total deformation $\varepsilon_{ac} = 4 \times 10^{-3}$ s$^{-1}$. Longitudinal deformation of testing specimens was read by the sensor MTS 632-42C-11 with the basis 12 mm.

During loading of individual testing specimens hysteresis curves were read and recorded (dependence stress – deformation), from which after rupture of individual testing specimens the level of elastic ($\varepsilon_{el}$) and plastic deformation ($\varepsilon_{pl}$) for $N_i/2$ was evaluated.

After completion of each test the number of cycles till rupture $N_i$ was recorded and from hysteresis curve for approximately $N = N_i/2$ for the chosen amplitude of total deformation $\varepsilon_{ac}$ there were deducted amplitude of plastic deformation $\varepsilon_{pl}$ and amplitude of elastic deformation $\varepsilon_{el}$ and amplitude of stress $\sigma_{ac}$. Curves of service life expressed in the form were plotted from experimental data:

$$\varepsilon_{ac} = \varepsilon_{el} + \varepsilon_{pl} = \frac{\sigma_{ac}^f}{E} (N_f)^b + \varepsilon_f (N_f)^c$$

(3)

Cyclic curves stress-deformation were also determined for complex assessment of response of steel after the ECAP to alternating plastic deformation in traction – pressure:

$$\sigma_{ac} = k \varepsilon_{ap}^n$$

(4)

Manson-Coffin curves of service life were plotted from the obtained values, as well cyclic curve of deformation strengthening. These values characterise deformation behaviour of material for prevailing time of its fatigue service life and they are therefore material characteristics. Results of individual test of
low-cycle fatigue were processed in a form of graphic diagrams (Figures 4 to 6).

![Graph of low-cycle fatigue](image)

**Fig. 4. Curves of low-cycle fatigue ε_pl-N_f**

![Graph of low-cycle fatigue](image)

**Fig. 5. Curves of low-cycle fatigue ε_pl-N_f**

![Graph of dependence between amplitude of stress σ_k - ε_pl](image)

**Fig. 6. Curves of dependence between the amplitude of stress σ_k - ε_pl**

4. **Conclusions**

The following findings were obtained on the basis of experimental works:

- Mechanical properties of the steel AISI 316 were determined by miniaturised tensile test, as well as by penetration test, selected samples were subjected to verification analysis of their chemical composition. Basic mechanical properties of the steel AISI 316 were determined in dependence on number of passes. Series of experiments was also realised in order to verify influence of intensive magnetic field on structure and mechanical properties of this steel.

Fatigue behaviour of the steel AISI 316 was investigated after application of various number of passes through the ECAP tool, structural stability was preserved, however, fatigue service life in the area of timed fatigue strength decreased after application of ECAP.

It follows from these results that materials with ultra-fine grain after intensive plastic deformation by the ECAP technology show at fatigue loading in the mode of constant amplitude of deformation (low-cycle fatigue) shorter fatigue service life in comparison with initial state. Nevertheless, it is possible to regard as highly positive the fact, that ultra-fine grained structure shows comparatively good mechanical stability after fatigue test, which is given by the fact that grains in structure are so small, that they prevent forming of dislocation structure. This fact conforms to the findings published in the work, where it was observed and verified on Cu. It can be predicted on the basis of obtained results that, contrary to low-cycle fatigue the ultra-fine grained material will manifest at fatigue load in the mode of constant amplitude of stress (high-cycle fatigue) higher fatigue characteristics, particularly fatigue limit. Confirmation of this presumption requires, however, realisation of additional experimental works aimed at the area of high-cycle fatigue of investigated material AISI 316 and detailed investigation with use of electron microscopy of possible structural changes in material after tests of high-cycle fatigue.

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