LOW-CYCLE FATIGUE OF STEEL AISI 316 AFTER ECAP

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Abstract

Main aim of this paper is to describe the plastic deformation executed by ECAP during low cycle fatigue of steel AISI 316. Among others, main attention was fixed on mechanical properties after this treatment. Experiments were planned and realised at temperature ranging from room temperature up to 280 °C. After deformation the structure was investigated to evaluate accumulation of deformation, deformation temperature as well as above-mentioned final properties. Accumulated true (logarithmic) deformation varied from the value 2 to 8. Structures were investigated via scanning electron microscope JEOL JEM 2100. Mechanical properties were evaluated by conventional tensile test and penetration test. Selected samples were subjected to low-cycle fatigue. Statistic evaluation of angular disorientation and grain/sub-grain size was also carried out with use of electron diffraction (EBSD) in combination with scanning electron microscope FEG SEM Philips. The ECAP was applied on austenitic steel AISI 316.

Keywords: Mechanical properties, low cycle fatigue, AISI 316, ECAP

1. INTRODUCTION

Stainless steel is a widely used structural material in various fields such as nuclear power plants or petrochemical industry. Steel AISI 316 is different from other stainless steel grades because of reduced content of C (0.03 wt. %) and the addition of Mo (2.5 wt. %). The addition of Mo is known to improve both corrosion resistance and hot deformation behaviour [1]. Moreover, Mo in solid solution acts as a favourable element in reducing dislocation mobility. It is related to the stacking fault energy of the material and the magnitude of controls that ease the cross-slip [2]. Deformation twins are expected to form easily in steel AISI 316 with low SFE, and play an important role in determining the subsequent microstructure during deformation process instead of cross-slip [3]. Coincidence site lattice boundaries, especially twin boundaries have low grain boundary energy compared to random boundaries. It is generally accepted that low energy grain boundaries, as contrasted with high energy boundaries, are highly resistant to grain boundary deterioration. A report suggested that the microstructure of steel AISI 316 was stable in spite of the prolonged annealing at 700 °C [4]. The grain size and the fractions of the twin boundaries were similar for all annealing conditions. From above investigations, it is deduced that once the microstructure is controlled to form high density of twin boundaries, the chemical, mechanical properties of material could be more enhanced than the material with random boundaries. Therefore, the way to generate high density of twin boundaries, their stability and effects on mechanical properties becomes great concern.

2. EXPERIMENT DESCRIPTION

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.**

Table 1 Basic chemical composition of the steel AISI 316 in wt. %

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 (GPa)	$R_{p0.2}$ (MPa)	R_m (MPa)	A (%)	Z (%)	KV (J)	HB (-)
AISI 316	216	330	625	45	-	90	21

The samples were manufactured with the following dimensions: ϕ 12 mm, length 60 mm. They were pushed through the ECAP matrix 2 to 8 times passes [5].

Matrix had channel diameter 12 mm and angle of 105°. Pressure in the matrix was kept on 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 evaluation of mechanical properties. In order to expand the existing findings the testing bars were exposed to intense magnetic field and impact of magnetic field on mechanical properties was investigated by tensile test. The sample was subjected to structural analysis after eight passes through the ECAP 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.

Amplitude of plastic deformation was the key factor for control of fatigue process. The following equation was used for the dependence $\varepsilon_{ap} - N_f$ [6] :

$$\varepsilon_{ap} = \varepsilon_f \cdot (2 \cdot N_f)^c \qquad (1)$$

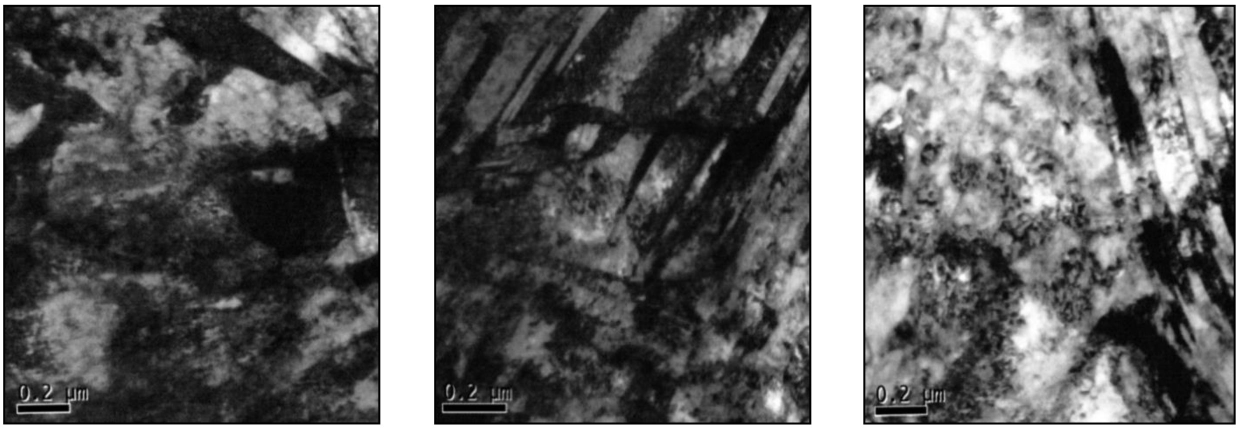
where ε_f is coefficient of fatigue ductility, c is exponent of the service life curve.

3. RESULTS AND THEIR ANALYSIS

3.1. Structure

Structures were analysed from the viewpoint of the course of strengthening and restoring processes. **Figure 1** documents deformed sub-structure of the steel AISI 316 after ECAP deformation by 4 to 8 passes through the die. 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 precipitate particles was not found. In cases where 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. **Figure 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. Austenitic matrix often contained deformation bands, which were formed during the ECAP deformation, see **Figures 1** and **Figure 2**. Deformation bands in austenitic steels can be formed by irregularly overlapping tiered errors, deformation twins or ε-martensite. These deformation bands are formed along octahedral planes {111} γ 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} *γ proves frequent occurrence of crystallographic defects in these formations [7].



a) deformation by 4 passes   b) deformation by 6 passes   c) deformation by 8 passes

Figure 1 Structure of steel AISI 316 after ECAP

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} γ, see **Figure 1b** and **Figure 1c**. Points of intersection of deformation bands generally represent preferential points for formation of particles of α'-martensite. However, electron diffraction analysis did not confirm occurrence of α'-martensite in these areas [8]. Occurrence of α'-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 taken 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.

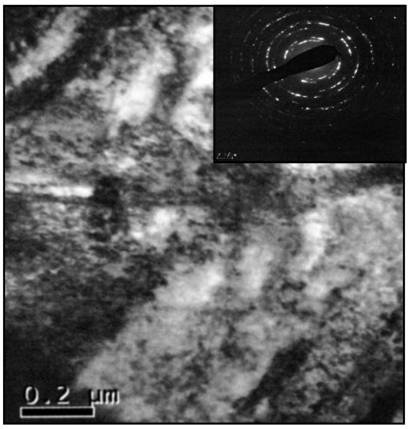


Figure 2 Diffraction pattern and structure of steel AISI 316 after ECAP, deformation by 8 passes

3.2. Mechanical properties

Samples after ECAP with number of passes (4, 5, 6) were used for investigation of influence of the ECAP process 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. Effect of magnetic field on change of mechanical properties was investigated from tensile test results as seen in **Table 3.**

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

Designation	E (MPa)	$R_{p0.2}$ (MPa)	R_m (MPa)	Z (%)
1	190,837	271.3	586.2	80.6
2	200,717	280.6	586.2	80.6
Magnetic field	1,188,276	283.1	588.5	81.5
Magnetic field	2,201,817	278.4	587.6	80.6
Magnetic field	3,195,199	282.1	592.0	80.8

Results of tensile tests showed 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 poly-crystalline materials.

Mechanical properties change in dependence on numbers of passes, strength properties ($R_{p0.2}$ and R_m) distinctly increase and plastic properties described by narrowing almost do not change (see **Table 4**).

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.

Table 4 Change of mechanical properties of steel AISI 316 after the 2nd to 5th pass

Number of ECAP passes	$R_{p0.2}$ (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

Increase of strength properties in dependence on grain size d (mm) is determined by the Hall-Petch relation [9]:

$$\sigma_y = \sigma_o + k_y \cdot d^{1/2} \tag{2}$$

where σ_y (MPa) is the yield stress, σ_0 (MPa) is the internal stress, k_y is 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_y = 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 cyclic deformation strengthening were prepared from extruded samples after 2 to 6 ECAP passes. Apart from 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 and 4 samples after 5 passes) and 12 samples with initial structure.

Test of low-cycle fatigue was performed according to the standard ASTM E 606 at laboratory temperature on servo-hydraulic testing equipment MTS 100 kN by strain control mode testing. During these tests a constant amplitude of total strain ε_{ac} was preserved. Tests of low-cycle fatigue were realised at constant strain rate $4 \cdot 10^{-3}$ s^{-1}. Longitudinal deformation of testing specimens was read by the sensor MTS 632-42C-11 with the gage length 12 mm.

During loading of individual testing specimens, hysteresis curves were read and recorded (dependence stress – strain), from which after rupture of individual testing specimens the level of elastic (ε_{ael}) and plastic deformation (ε_{apl}) for N_f / 2 was evaluated.

After completion of each test the number of cycles till rupture N_f was recorded and from hysteresis curve for approximately $N = N_f$ / 2 for the chosen amplitude of total deformation ε_{ac} there were deducted amplitude of plastic deformation ε_{apl}, amplitude of elastic deformation ε_{ael} and amplitude of stress σ_a. Curves of service life expressed in the form were plotted from experimental data [10]:

$$\varepsilon_{ac} = \varepsilon_{ael} + \varepsilon_{apl} = \frac{\sigma'_f}{E}\cdot\left(N_f\right)^b + \varepsilon_f\cdot\left(N_f\right)^c \qquad (3)$$

Cyclic stress-strain curves were also determined for complex assessment of response of steel after the ECAP to alternating plastic deformation in traction – pressure [11]:

$$\sigma_a = k\cdot\varepsilon_{apl}^n \qquad (4)$$

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 (see **Figure 3**, **Figure 4** and **Figure 5**).

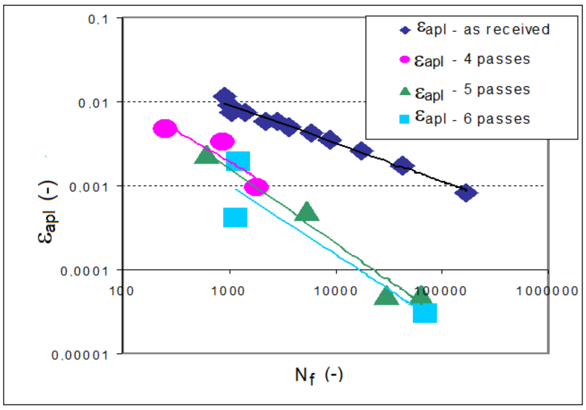


Figure 3 Dependence of plastic portion of deformation amplitude on number of cycles

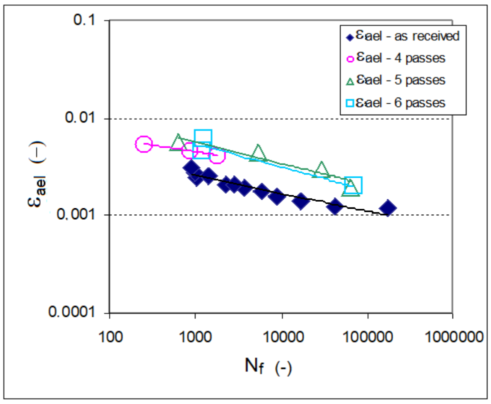


Figure 4 Dependence of elastic portion of deformation amplitude on number of cycles

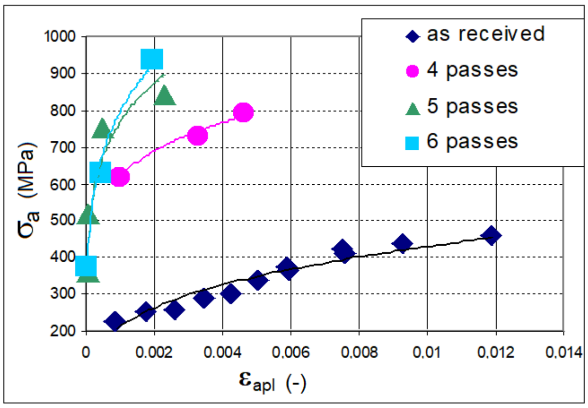


Figure 5 Cyclic stress-strain curves

4. CONCLUSION

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 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 were 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 die, 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 size after intensive plastic deformation by the ECAP technology at fatigue loading in the mode of constant amplitude of deformation (low-cycle fatigue) show shorter fatigue service life in comparison with initial state. Nevertheless, it is possible to regard as highly positive 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 confirms findings published in the work, where it was observed and verified on Cu [5]. It can be predicted on the basis of obtained results that, contrary to low-cycle fatigue the ultra-fine grained material at fatigue load in the mode of constant amplitude of stress (high-cycle fatigue) will manifest 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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