

EFFECT OF EQUAL-CHANNEL ANGULAR PRESSING ON CRACK RESISTANCE AND CORROSION DAMAGE OF ZIRCONIUM ALLOY Zr-2.5%Nb

ИССЛЕДОВАНИЕ ВЛИЯНИЯ РАВНОКАНАЛЬНОГО УГЛОВОГО ПРЕССОВАНИЯ НА ТРЕЩИНОСТОЙКОСТЬ И КОРРОЗИОННУЮ ПОВРЕЖДАЕМОСТЬ ЦИРКОНИЕВОГО СПЛАВА Zr-2.5%Nb

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Abstract: Testing method for static crack resistance has been developed and applied for thin sheet (sample thickness ~ 1mm) samples of zirconium alloy Zr-2.5%Nb. Linear parameter of fracture mechanics - stress intensity factor K_{Ic} has been determined. Alloy Zr-2.5%Nb after ECAP was tested for corrosion resistance in the physiological Ringer's solution for two years.

KEY WORDS: ZIRCONIUM ALLOY, BIOCOMPATIBLE MATERIALS, SEVERE PLASTIC DEFORMATION (SPD), EQUAL CHANNEL ANGULAR PRESSING (ECAP), CRACK RESISTANCE, CORROSION DAMAGE

1. Introduction

Fracture resistance is the important characteristic of any type of metallic implants used in the human body. Along with titanium alloys, zirconium alloys are also promising material for medicine, because they have good biocompatibility [1]. However, strength properties of zirconium alloys are insufficient, and in order to increase the strength with sufficient ductility zirconium alloys are subjected to severe plastic deformation (SPD), by means of equal channel angular pressing (ECAP) [2-5].

Currently, the actual problem is the evaluation of the corrosion damage of biocompatible zirconium material during the exploitation in the human body, as well as determination of critical stresses of biocompatible alloys that may appear in the implant.

2. Experimental procedure

Corrosion test and crack resistance test of flat sheet samples from zirconium alloy Zr-2.5%Nb before and after ECAP were conducted at room temperature.

The size of samples for corrosion test was 10×10×1 mm. Samples were incubated inside a glass bottle with Ringer's solution (composition per 200 ml of H₂O: NaCl - 8.6 g, KCl - 0.3 g, CaCl₂ - 0.25 g) within 730 days. These samples were placed in the glass bottle so that there were not contact between them to avoid a possible occurrence of "electrochemical couples". During the test, the glass bottle was hermetically closed to prevent the solution evaporation. Every 30 days, each sample was removed from the glass bottle using tweezers and was dried in air for 2 hours, then it was weighed on electronic scales GR-202.

Flat thin samples for static crack resistance test were cut on electrical-discharge machine Chmer GX360L from bars of zirconium alloy Zr-2.5%Nb after ECAP with a different number of passes (1-6). Then, hole with diameter of 1 mm was drilled and two thin transverse notches (2 mm in length) were made in the center of a flat sample (Fig. 1).

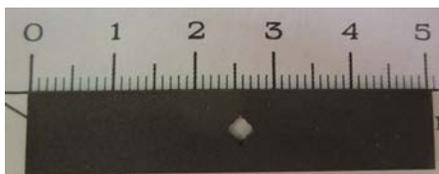


Fig. 1. Typical view of the sample for crack resistance testing

Uniaxial tensile tests of flat thin sheet specimens with the stress concentrators were carried on a universal test machine Instron 5569 using the acoustic emission (AE) method. The load of a crack initiation was determined from the combined strain curves and acoustic emission (Fig. 2).

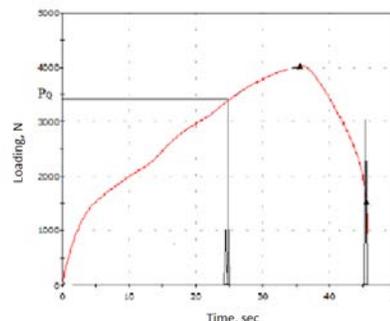


Fig. 2. Typical view of a combined strain curves and AE

Fracture surface of samples of zirconium alloy Zr-2.5% Nb in the initial state and after ECAP were investigated after the static crack resistance tests by scanning electron microscope (SEM) Hitachi S-800.

Also microstructure analysis of zirconium alloy Zr-2.5% Nb in the initial state and after ECAP was carried out by a transmission electron microscope (TEM) JEM 1400.

3. Results and discussion

Result of corrosion tests in Ringer's solution of the zirconium alloy Zr-2.5%Nb samples in initial state and after ECAP, showed that it was not revealed oxide film growth or shrinkage of the samples in the initial state and after ECAP.

Figure 3 shows structure of zirconium alloy Zr-2.5% Nb in the initial state and after ECAP.

It's shown that the average grain size in the initial state is 3.3±0.5 μm (12 hours annealing) and 2.5±0.1 μm (6 hours annealing), but the average grain size after ECAP is 300±65 nm (1 pass) and 250±50 nm (6 passes).

The severe plastic deformation by ECAP leads to the formation of partially oriented grain, subgrain and submicrocrystalline structure.

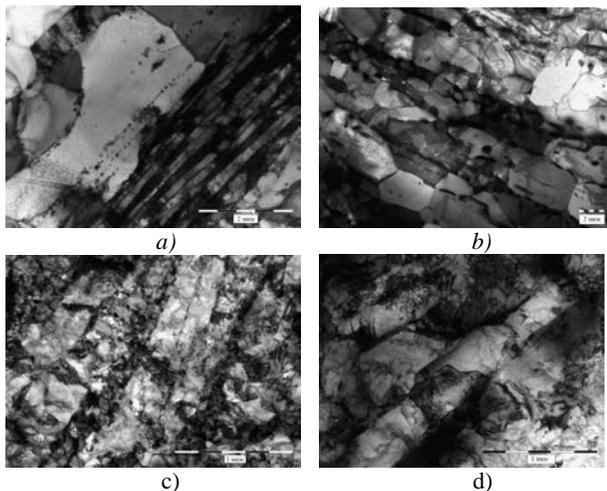


Fig. 3. The microstructure of zirconium alloy Zr-2.5%Nb in the initial state: a - annealed for 12 hours at 580 °C, b - annealed for 6 hours at 580 °C; and after ECAP: c - single pass, d - 6 passes (TEM)

The test results of flat thin samples of zirconium alloy Zr-2.5% Nb showed that the highest crack resistance observed in samples in the initial state after annealing for 6 hours at 580 °C ($K_{Ic} = 29.4 \pm 1.8 \text{ MPa} \cdot \text{m}^{1/2}$) and the lower crack resistance observed in samples after ECAP in a single pass ($K_{Ic} = 14.8 \pm 1.5 \text{ MPa} \cdot \text{m}^{1/2}$).

The fracture surfaces of zirconium alloy Zr-2.5% Nb samples in the initial state and after ECAP after the static crack resistance tests are shown at Fig. 4.

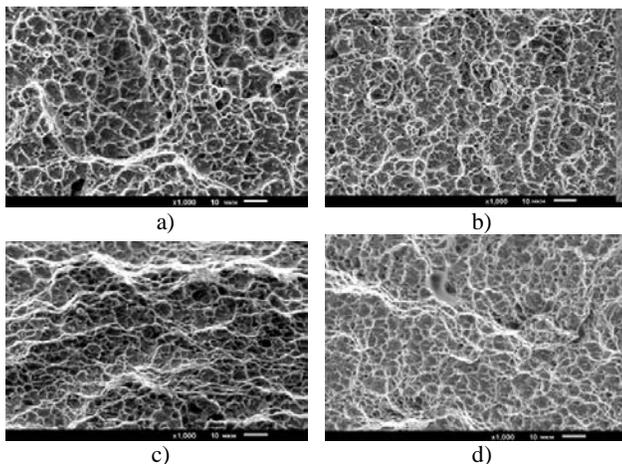


Fig. 4. The fracture surface of zirconium alloy Zr-2.5% samples in the initial state: a - annealed for 12 hours at 580 °C, b - annealed for 6 hours at 580 °C; and after ECAP: c - single pass, d - 6 passes (SEM)

Fractography analysis showed that all the samples were fracture by ductile fracture mechanism. The average size of dimples for samples in the initial state amounted $5.3 \pm 0.5 \mu\text{m}$ (annealed for 12 hours at 580 °C) and $4.5 \pm 0.5 \mu\text{m}$ (annealed for 6 hours at 580 °C) but the average size of dimples after ECAP were $2.8 \pm 0.3 \mu\text{m}$ (single pass) and $2.6 \pm 0.4 \mu\text{m}$ (6 passes).

4. Conclusion

Prolonged corrosion tests in Ringer's solution of the samples of zirconium alloy Zr-2.5%Nb in the initial state and after ECAP were performed for two years with a periodic sample weighting. According to the test results no significant gain or loss of the sample weight was revealed in the initial state and after ECAP.

Quantitative comparative values of crack resistance of the Zr-2.5%Nb alloy were measured after ECAP and annealed states (after 6 and 12 hours of annealing at 580 °C). The crack resistance of samples from zirconium alloy Zr-2.5% Nb in the initial state after annealing for 6 hours at 580 °C was in two times higher compared to the samples after ECAP in a single pass.

Metallographic and fractographic analysis showed that the main factor influencing on the crack resistance of zirconium alloy Zr-2.5%Nb after various modes of thermomechanical treatment is the presence of subgrain structure and recrystallized grain size before and after ECAP.

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5. Literature

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