EFFECT OF NEUTRON IRRADIATION ON MICROSTRUCTURE AND PROPERTIES OF AUSTENITIC AISi 321 STEEL, SUBJECTED TO EQUAL-CHANNEL ANGULAR PRESSING

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Abstract. This paper presents the results of comparative analysis for the samples of austenitic AISI 321 (18Cr-9Ni) steel in the as-received state and the state after the equal channel angular pressing (the ECAP-state) before and after the reactor irradiation on fast neutrons BIR-60 up to maximum damage dose 5.3 dpa at 350 °C. The results point out the mixed fragmentary character of the structure with a large non-homogeneity degree. The equiaxial fragments with the size of 300-400 nm have been observed along with the elongated grains with the size up to a few micrometers. The tensile testing of the irradiated steel in the ECAP-state has been conducted in the range of temperatures from 20 to 650 °C. It has been demonstrated that mechanical characteristics of the irradiated steel in the ECAP-state do not concede, or, they are even superior to the properties of an initial (coarse-grained) material.

1. INTRODUCTION

New opportunities in application of conventional reactor materials are being revealed in the result of developing and using the severe plastic deformation (SPD) methods. The SPD methods of high pressure torsion and equal channel angular pressing (ECAP) [1] provide obtaining submicrorystalline (SMC) structures with the grain size from hundreds to tens of nanometers [2]. The high dislocation density, the occurrence of non-equilibrium low-angle and high-angle grain boundaries provide the grain-boundary and substructural strengthening, and change the complex of physical and mechanical properties of metals and alloys, subjected to the SPD. There is a crucial possibility to reduce the development of such phenomena as irradiation embrittlement, irradiation-induced swelling, stress-corrosion cracking initiated by irradiation, etc., due to the increase of sink concentration in SMC-structures in real reactor constructions on fast and slow neutrons.

The experimental data about the peculiarities of mechanical property changes of stainless steels with SMC structure, irradiated by neutrons in the range of operational parameters of critical components of radiation zone and construction of the exploited and projected atomic electric stations (AES) do not exist.

The goal of the present paper is to investigate the alterations of microstructure and mechanical properties in austenitic AISi 321 steel in a struc-
tural state before and after the ECAP, irradiated by neutrons. Austenitic stainless AISI 321 steel is the basic material applied during the fabrication of internals in water-cooled reactors.

2. MATERIALS AND TECHNIQUES OF THE INVESTIGATION

The research of microstructure and mechanical properties has been carried out on austenitic AISI 321 steel in two states: the as-received state, the state after the ECAP.

The chemical composition (Table 1) has been determined by the method of atomic-emission spectrometric analysis with inductively coupled plasma (AES ICP). The carbon content has been determined by the automatic coulometric titration method.

The ECAP has been conducted on the rods with the diameter 10 mm and the length 60 mm at temperature 400 °C along the route Bc [1]. The rods have been pressed in a special die through two channels with similar cross sections, which intersected at the angle of 120°.

To study the fine structure of non-irradiated steel in the as-received state and after the ECAP, the method of optical metallography (OM) and the method of electrons back scattered diffraction (EBSD) have been used. The last one has been realized with the help of high-resolution field-effect emission scanning microscope SUPRA55VP, equipped by energy-dispersive X-ray spectrometer [3]. The equipment has provided the possibility of determining the grain boundaries with low and high misorientation angles in the material structure. The result of the experiment has been presented as diffraction maps, characterizing the misorientation spectrum of grain boundaries due to the following criterion: more than 15°, more than 2°, as well as the phase attribute of certain structure areas in FCC- or BCC – lattice.

The irradiation of the samples in the reactor natrium media at temperature ~350 °C for ~10 months has been carried out in the reactor on fast neutrons BOR-60 in a dismountable experimental device. The maximum damage dose on the samples has been 5.3 dpa.

The samples for tensile tests (in the form of “dumbbell”, Fig. 1) with the length of the working section 15 mm, the diameter 3.0 mm have been fabricated out of the rods after the ECAP. For comparison, the samples from the as-received state have been fabricated too.

The mechanical tensile tests in the range of temperatures from the room one up to 650 °C have been carried out on a remote multi-purpose testing machine 1794U (with the electro-mechanical loading system) at rate of motion of the active grip 1 mm/min. The standard characteristics have been calculated from the tensile diagrams: the flow limit (during the plastic deformation 0.2%), the tensile stress, the uniform and total relative elongation.

3. RESULTS

3.1. Microstructure

The microstructure in the as-received condition is presented by polygonal grains of austenite with the average size 40 μm (further – coarse-grained (CG), Fig. 1a) and is characterized by the high number of twins. The evident grain refinement (less than 1 μm

![Fig. 1. The OM images of the as-received state (a) and the state after the ECAP (b).](image-url)
Effect of neutron irradiation on microstructure and properties of austenitic AlSi 321 steel...

In Fig. 2 there is a view of the investigated steel structure (the as-received condition), obtained by the method of EBSD. The average grain size is 40 μm. Inside the big grains there are quite a lot of twins. There are a lot of low-angle boundaries (about 30%), and the high-angle grain boundaries can also be observed here. The twin boundaries (the annealing twins) can be observed inside the grains.

Fig. 3 shows the EBSD image of structure for the ECAP state. In the material structure (Fig. 3) the boundaries of macro-grains with the misorientation angle more than 15°, sub-boundaries with the misorientation angle more than 2° are well resolved. The brightness nuance in certain surface areas can be explained by the existence of local intragranular (intracrystalline) misorientations with the angle less than 2°. The development of submicrocrystalline structure is characteristic for material in the ECAP condition. This fact is followed by the destruction of high-angle boundaries and

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**Fig. 2.** Orientation diffraction map for the steel in an as-received condition.

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**Fig. 3.** The EBSD image of structure for the ECAP state with high-angle grain boundaries and low-angle sub-boundaries. The profile of the local grain misorientations.
development of the new low-angle ones. Thus, the number of low-angle boundaries is increasing up to 60% in general. The analysis of the phase composition has shown an inconsiderable amount of $\alpha$ phase.

The TEM has proved the fragmentation of the steel structure in the process of the ECAP. The equiaxial crystals with the size of 300-400 nm and the elongated grains with the size up to a few μm have been observed here (Fig. 4). The EBSD pattern (Fig. 4) approves the grain fragmentation (one can see the singular ring-shaped reflexes).

The initial state is characterized by high microhardness values (3200 MPa). The ECAP has brought to the increase of microhardness values up to 4100 MPa in a longitudinal section and 4000 MPa in the cross sections of the sample. Annealing during 4 hours at temperature 350 °C for the check of the ECAP-state thermostability has demonstrated the conservation of microhardness values at the same level. This fact is in a good agreement with the results obtained in work [4].

3.2. Short-term mechanical tensile tests

The results of the tensile tests are reduced in Table 2 and Figs. 5 and 6.

One of the significant peculiarities for the non-irradiated states is considerable hardening due to the ECAP (the increase $\sigma_{0.2}$ at 20 and 350 °C is ~200 and ~270 MPa, respectively), the loss of ability to deformation hardening and increase of the attitude to plastic deformation localization in the neck in comparison with the initial CG state. It is expressed in a less degree at $T = 20$ °C and quite drastically at $T = 350$ °C.

The increase of the flow limit up to ~380 MPa for the as-received state and up to ~300 MPa for the ECAP-state in comparison with the corresponding
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Table 2. Mechanical properties of the samples of AISI 321 steel (in the as-received state and the ECAP-state) before and after the irradiation at $T_{\text{irrad}} = 350 \, ^\circ\text{C}$.

<table>
<thead>
<tr>
<th>State of the Material</th>
<th>$T_{\text{act}} , ^\circ\text{C}$</th>
<th>$\sigma_{\text{UTS}}, \text{MPa}$</th>
<th>$\sigma_{0.2}, \text{MPa}$</th>
<th>$\delta_{\text{as}}$, %</th>
<th>$\delta_{\text{tr}}$, %</th>
<th>$\psi$, %</th>
<th>State of the Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-received condition</td>
<td>20</td>
<td>766</td>
<td>673</td>
<td>22.9</td>
<td>34.8</td>
<td>90</td>
<td>Initial</td>
</tr>
<tr>
<td></td>
<td>350</td>
<td>556</td>
<td>497</td>
<td>7.9</td>
<td>18.0</td>
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<tr>
<td>ECAP</td>
<td>20</td>
<td>917</td>
<td>869</td>
<td>2.0</td>
<td>25.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>350</td>
<td>778</td>
<td>766</td>
<td>0.8</td>
<td>11.5</td>
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<tr>
<td>As-received condition</td>
<td>20</td>
<td>1130</td>
<td>1048</td>
<td>1.6</td>
<td>23.3</td>
<td>72</td>
<td>After irradiation</td>
</tr>
<tr>
<td></td>
<td>350</td>
<td>933</td>
<td>907</td>
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<td>8.9</td>
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<td></td>
<td>400</td>
<td>862</td>
<td>839</td>
<td>0.5</td>
<td>8.9</td>
<td>51</td>
<td></td>
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<tr>
<td></td>
<td>450</td>
<td>831</td>
<td>827</td>
<td>0.3</td>
<td>8.2</td>
<td>52</td>
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<td></td>
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<td>649</td>
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<td>567</td>
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<td>10.3</td>
<td>60</td>
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</tr>
<tr>
<td></td>
<td>650</td>
<td>477</td>
<td>453</td>
<td>2.2</td>
<td>9.6</td>
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<tr>
<td>ECAP</td>
<td>20</td>
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<td>0.9</td>
<td>16.2</td>
<td>77</td>
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<tr>
<td></td>
<td>350</td>
<td>997</td>
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<td>7.3</td>
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<td>951</td>
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<tr>
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<tr>
<td></td>
<td>650</td>
<td>567</td>
<td>542</td>
<td>1.4</td>
<td>9.3</td>
<td>53</td>
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</tr>
</tbody>
</table>

The experimental results testify about an increased thermal stability of the ECAP-state after the irradiation. The higher irradiation hardening, found out in the case of the ECAP-state in comparison with the as-received state, preserves up to the temperature 600-650 °C. The values of homogeneous and total relative elongation for both states of the irradiated steel at $T = 350 \, ^\circ\text{C}$ almost have no difference.

The alterations of the flow limit and ductility after the neutron irradiation are determined by the microstructure changes, which have been studied comprehensively in austenitic stainless steels [6,7] and are connected with the development of such components of radiation effect as defect clusters (*black...

Fig. 7. The dependency of the flow limit from the testing temperature after irradiation.

Fig. 8. The homogeneous elongation dependency from the testing temperature after irradiation.

Fig. 9. Temperature dependency of density of various components of irradiation defects in the structure of austenitic stainless steel, irradiated in reactors with combined and fast neutron spectrum, replotted from [8].

4. CONCLUSIONS

The research of the neutron irradiation effect on the microstructure and the short-term mechanical properties of AISI 321 steel in the CG state and the state
after the ECAP, before and after the neutron irradiation at temperature ~350 °C in the reactor BOR-60 with the maximum damage dose 5.3 dpa, has been carried out. The analysis of microstructure and phase composition of steel in the as-received state and the ECAP-state, carried out by the scanning electron microscopy method with the X-ray microanalysis and EBSD method has revealed: a wide spread of grain values from tens of microns to the hundreds of nanometers, the increase of the average misorientation of grain boundaries due to the ECAP approximately twofold, the thermal stability (up to 650 °C) of irradiation hardening in the ECAP state after the neutron irradiation.

It is necessary to continue the work about irradiation and after-reactor research. It is impotent to investigate the changes of physical, mechanical and structural properties of austenitic steels in the ECAP-state due to irradiation. It will allow learning the peculiarities of irradiation damage of SMC-structures and develop of recommendations for their application in reactor technologies.

REFERENCES


