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EFFECT OF NEUTRON IRRADIATION ON MICROSTRUCTURE AND PROPERTIES OF AUSTENITIC AlSi 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 BÎR-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 submicrocrystalline (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-

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Table 1. Chemical composition of the material under investigation.

Element	C	Si	Mn	Cr	Ni	S	F	Cu	Co	Mo	W	Ti	V
Content,%	0.049	0.42	1.42	19.1	8.9	0.012	0.037	0.56	0.14	0.31	0.03	0.001	0.063

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 B_C [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

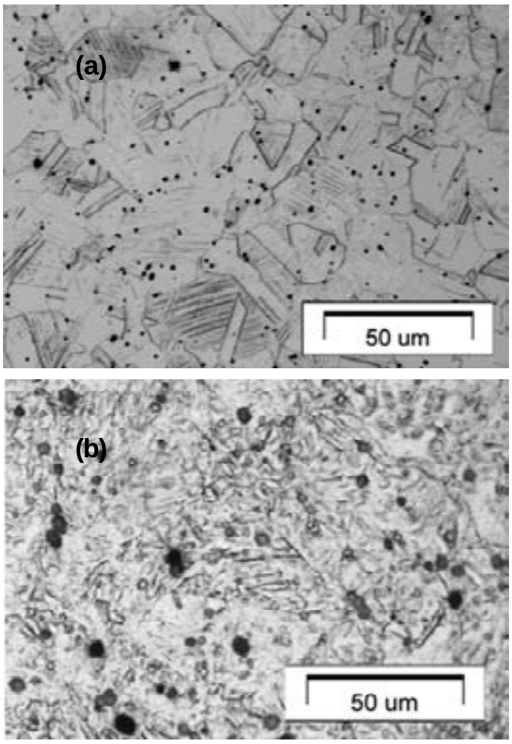


Fig. 1. The OM images of the as-received state (a) and the state after the ECAP (b).

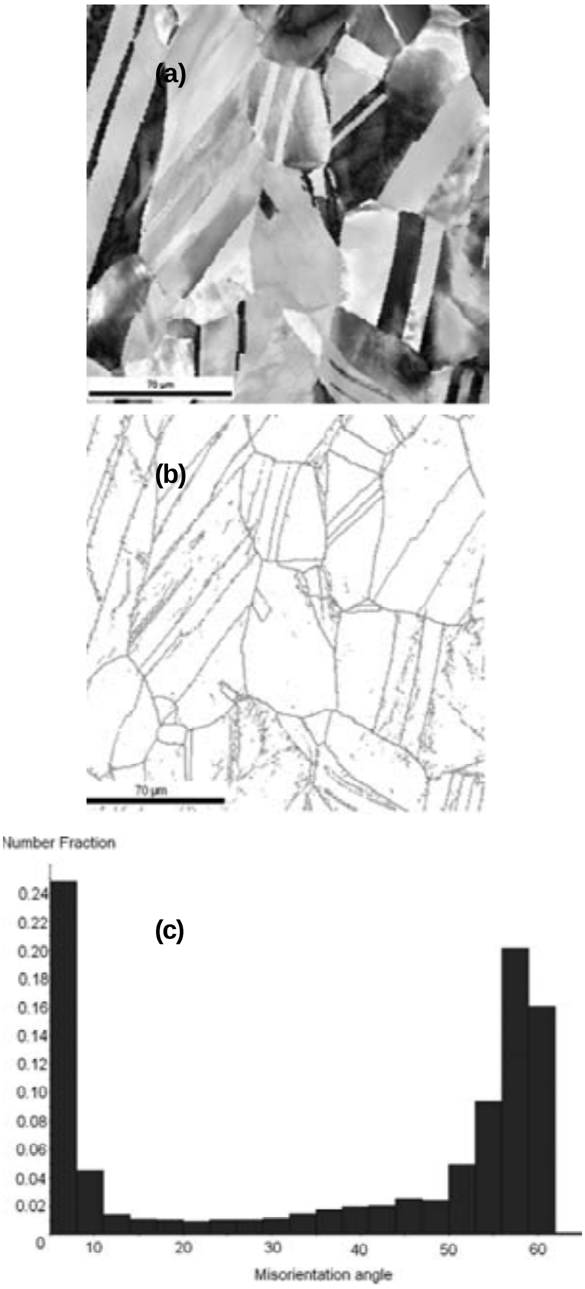


Fig. 2. Orientation diffraction map for the steel in an as-received condition.

– submicrocrystalline structure (SMC)) is characteristic for the sample structure after the ECAP (Fig. 1b).

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

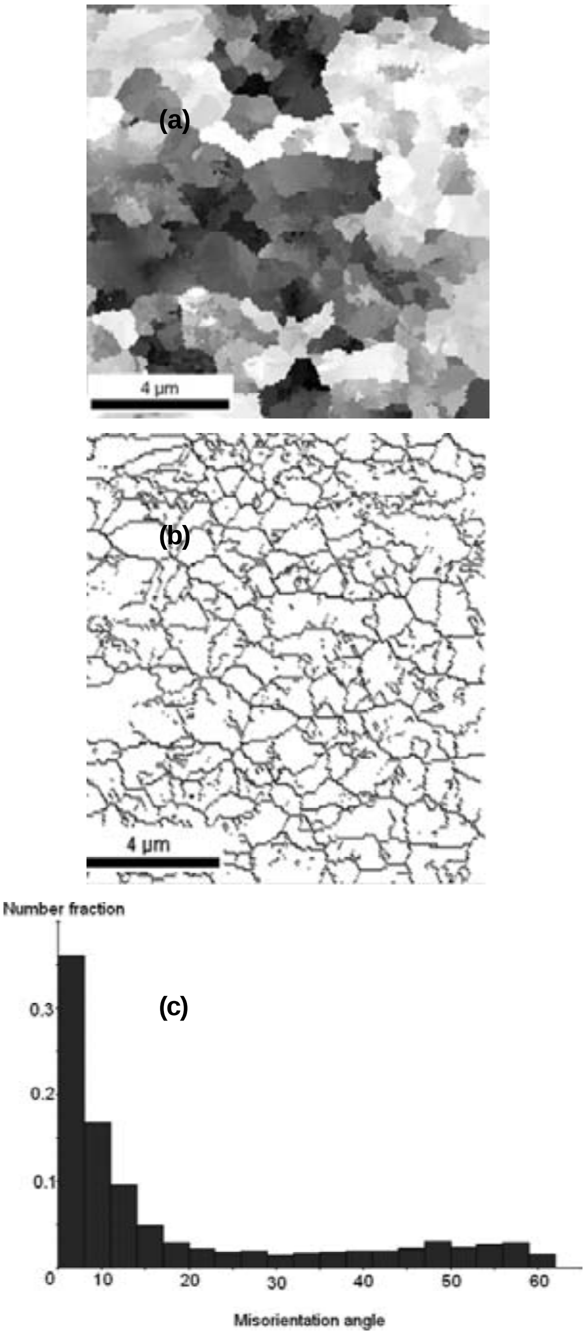
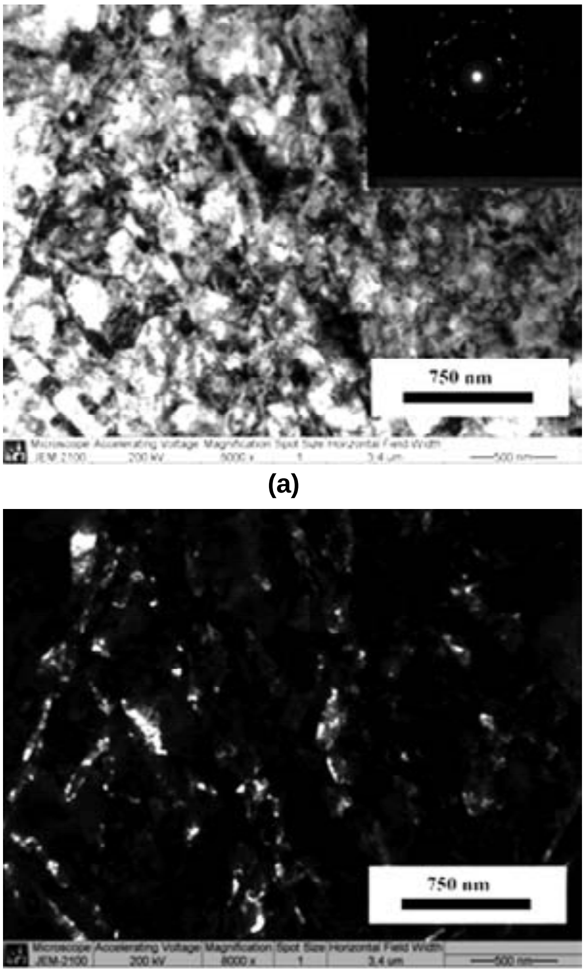


Fig. 3. The EBSD image of structure for the ECAP state with high-angle grain boundaries and low-angle sub-boundaries. The profile of the local grain misorientations.



(a)

(b)

Fig. 4. The TEM images of fine structure after the ECAP: a) the light field and electron-diffraction pattern; b) the dark field pattern.

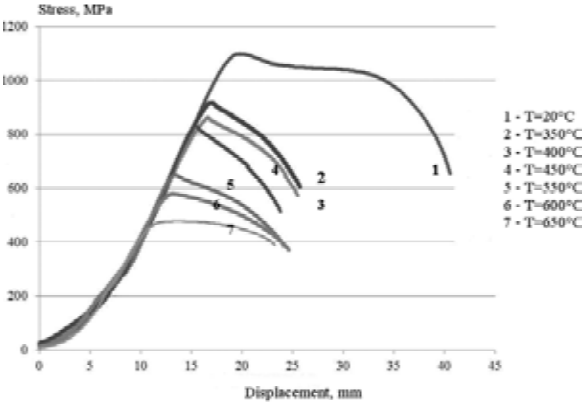


Fig. 5. The tensile deformation diagrams of the as-received state irradiated at 350°C and deformed at the different temperatures.

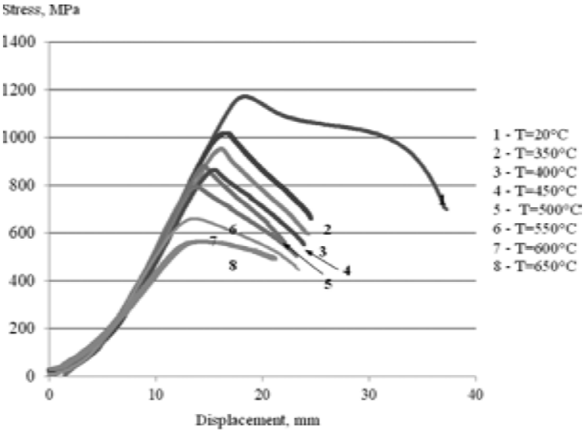


Fig. 6. The tensile deformation diagrams of the ECAP-state irradiated at 350 °C and deformed at different temperatures.

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 α – 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 ìm 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 microhardeness 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

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_{irrad} = 350 °C.

State of the Material	$T_{def.}$°C	σ_{UTS} ,MPa	$\sigma_{0,2}$, MPa	δ_{un}%	δ_{o},%	ψ,%	State of the Material
As-received condition	20	766	673	22.9	34.8	90	Initial
	350	556	497	7.9	18.0		
ECAP	20	917	869	2.0	25.2		
	350	778	766	0.8	11.5		
As-received condition	20	1130	1048	1.6	23.3	72	After irradiation
	350	933	907	0.6	8.9	57	
	400	862	839	0.5	8.9	51	
	450	831	827	0.3	8.2	52	
	550	649	637	0.4	9.5	63	
	600	579	567	0.6	10.3	60	
	650	477	453	2.2	9.6	55	
ECAP	20	1208	1178	0.9	16.2	77	
	350	997	954	0.6	7.3	58	
	400	951	943	0.5	8.8	57	
	450	878	874	0.4	8.1	56	
	500	864	825	0.7	8.8	60	
	550	791	746	0.7	9.0	54	
	600	658	631	1.0	9.5	59	
	650	567	542	1.4	9.3	53	

non-irradiated states has been observed at T = 20 °C (Fig. 5). At T = 350 °C the increase of the flow limit after the irradiation for the as-received state has been ~400 MPa and ~200 MPa for the ECAP state. For both states the value of homogeneous elongation, as it can be seen from Table 2, is decreasing up to the testing temperatures 600-650 °C. The values of homogeneous and total relative elongation for both states of the irradiated steel at T = 350 °C almost have no difference.

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 650 °C (Fig. 6). The total relative elongation of both states after the irradiation in the range of the testing temperatures 450÷650 °C is equal (Table 2).

Some increase of the homogeneous relative elongation found out in the irradiated samples of the ECAP-state at T = 350-600 °C (Fig. 7) is the consequence of the processes of recovery and partial annealing of the irradiation defect complexes, developed at T_{irrad}~350 °C. It is a common knowledge [5] that in the range of temperatures 350-550 °C in strained materials the processes of irradiation creep and relaxation of the strains are the most effective ones (this refers to residual stresses, as the result of severe plastic deformation). After the beginning of plastic flow, the irradiated steel in both states does not reveal capabilities to deformation strengthening. The applied stress decreases sharply, the process of plastic deformation localization starts here, the decay can also be observed at general deformation from 3.5% to 9.0% (Table 2). The minimum value of the homogeneous relative elongation can be observed at T_{def}=450 °C (Fig. 9).

After deformation at testing temperature more than 350 °C the negative hardening coefficient differs by the less rate of descent and higher stability for the irradiated ECAP samples with the deformation increase (compare the diagrams for corresponding testing temperatures in Figs. 7 and 8). The more descent of it after achieving about half a value from the relative elongation (e.g., the diagram for T_{def} = 350 °C) is characteristic for the as-received state at the same time. This peculiarity, in our opinion, points out quite an evident breakdown to the crack propagation in the ECAP-state.

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

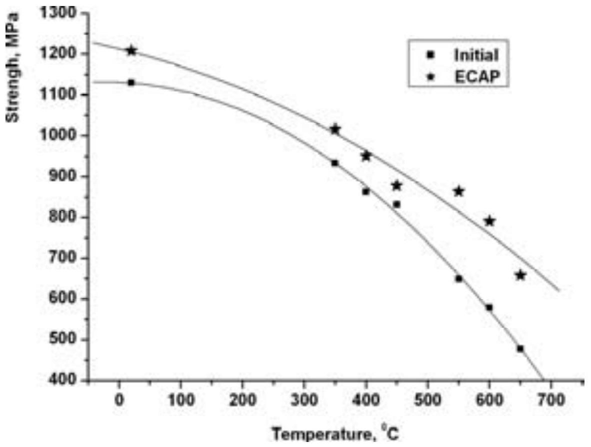


Fig. 7. The dependency of the flow limit from the testing temperature after irradiation.

dots"), defect Frank loops <111>, voids and dislocations in the structure. So, the high density (independent from the irradiation temperature) of the defect clusters (the so-called "black dots") about 2 nm in diameter is revealed in the microstructure of austenitic stainless steels, irradiated in the range of temperatures up to 250 °C. At temperature higher than 250 °C the density of the "black dots" is dipping down (Fig. 9).

The concentration of the defect Frank loops is increasing along with the temperature growth in a low-temperature area up to 300 °C**.** Quite a sharp decrease of density of loops occurs at temperature, separating the low-temperature and the high-temperature areas (Fig. 8), beginning from T_{irrad} ~350 °C and the damage dose more than 5-10 dpa [6]. In the result the transition from the structure with prevalence Frank loops in it to the structure with voids predomination, emissions and dislocation network at T_{irrad} higher than 400 °C is observed here. It should be noted that it is characteristic for both the austenitic and the cold-deformed materials [7].

The temperature dependency of the flow limit of steel in the ECAP-state (in comparison with the as-received state) is presented in Fig. 7. It is evident that the increase of the flow limit after the irradiation reaches its maximum near the irradiation temperature ~350 °C and correlates with the maximum density of the Frank loops (Fig. 9). This fact, on the one hand, is in agreement with the results of work [8]. On the other hand, the increase of the flow limit, caused by irradiation in the ECAP-state of steel, exceeds such values for austenitic steels of this type, irradiated by the comparable doses in fast-neutron and thermal-neutron reactors [9]. This fact should be involved for explaining a higher irradiation hardening of the ECAP state by the development, not just existence of defect clusters, defect loops, voids, dislocations and their emissions in the structure. The important role is played by the processes of grain fragmentation (the alteration of the angles of grain misorientation at the stage of the ECAP realization).

In connection with the peculiarities of irradiation damage and evolution of the ECAP structure at small damage doses in a high-temperature area (from 350 °C to 700 °C) it is too early to make any final conclusions because of the absence of any structural experimental data about it.

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

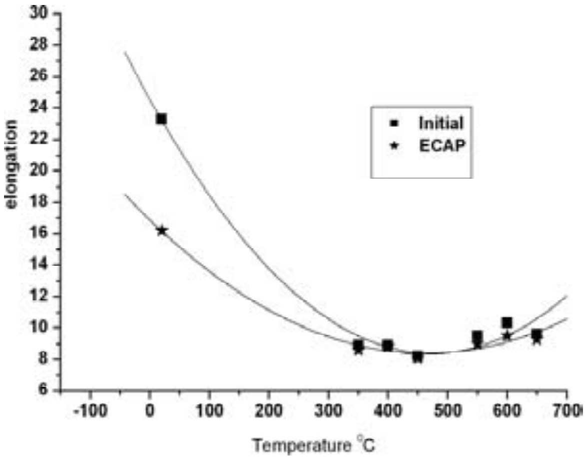


Fig. 8. The homogeneous elongation dependency from the testing temperature after irradiation.

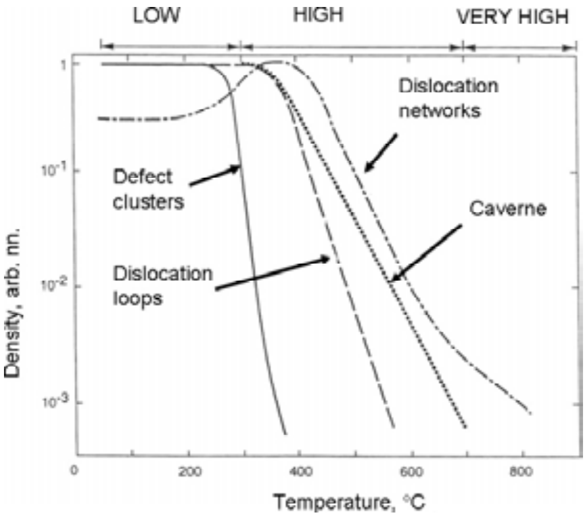


Fig. 9. Temperature dependency of density of various components of irradiation defects in the structure of austenitic stainless steel, irradiated in reactors with combined and fast neutron spectrum, replotted from [8].

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.

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