Synergetic mechanism of high temperature radiation embrittlement of austenitic steels under long term neutron irradiation at high temperatures

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Abstract. The study results of fracture and embrittlement mechanisms are represented for austenitic steels of 18Cr-9Ni and 18Cr-10Ni-Ti grades after long term neutron irradiation at high temperatures. The effect of irradiation temperature, irradiation time and neutron dose is considered on the fracture strain and fracture mechanisms. On the basis of the obtained results and special tests a new mechanism of high temperature radiation embrittlement is proposed and justified as caused by the synergetic action of helium and thermal aging.

Key Words: Irradiated austenitic steel, Fracture strain, Fracture mechanism, High temperature radiation embrittlement

1. Introduction

Investigations of radiation embrittlement mechanisms and fracture properties for austenitic stainless steels undergone high temperature neutron irradiation were intensively carried out in the 1950s – 1980s. At that time research and power fast neutron reactors are known to be put into operation such as BOR-60 and BN reactors in Russia, Rapsodie and Phenix reactors in France, EBR reactors in USA and others. It should be mentioned that power fast reactor BN-600 is currently in operation more than 40 years.

Pioneer works were performed that reveal basic properties of high temperature radiation embrittlement concerning the fracture mechanisms and the mechanical properties [1-9]. At the same time it should be emphasized that most of these studies were performed for relatively short term irradiation either in FR or in various accelerators (ions, protons and electrons).

Helium accumulation on grain boundaries during irradiation was found to be the main mechanism of high temperature radiation embrittlement (HTRE) of austenitic steels. This mechanism allows the explanation of two typical features of HTRE: sharp decrease in plasticity at high test temperatures and appearance of intergranular fracture.

At present, investigations of HTRE are of more interest that is connected, first of all, with a necessity of the service life extension for BN-600 reactor, putting into operation of BN-800 reactor and designing of BN-1200 reactor.

At present a possibility has arisen to study the properties of austenitic steels after long term irradiation in power BN-600 reactor and research BOR-60 reactor due to scheduled removal of decommissioned components. On the basis of these studies results the design dependencies may be additionally verified that were proposed in “Standard for calculation of strength of basic components of FR with sodium coolant” (RD EO 1.1.2.09.0714). (Main developer of Standard is CRISM “Prometey”, and the basic principles of this Standard are presented in [10].) The performance properties database should be also filled up with the study results from the decommissioned components materials.

Since 2007 when the first version of this Standard was elaborated and approved and up to now a large complex of the studies of the decommissioned components materials was performed at CRISM "Prometey". These studies were performed for materials worked more than 10^5 hours and concern mainly the thermal aging effect [11-13].

The study results represented in the present paper are related to the joint effect of thermal aging and neutron irradiation on the properties of austenitic stainless steels. This joint effect is known to be typical for many structural components of FRs.

Thus, the present paper aims at analysis of the fracture and embrittlement mechanisms for austenitic stainless steels of 18Cr-9Ni and 18Cr-10Ni-Ti grades irradiated at high temperature during long time. This aim is achieved through (i) experimental determination of the fracture strain under tension over wide range of temperatures, (ii) investigation of the fracture mechanisms, and (iii) the analysis of the obtained results together with available data [1-9] and the results of additional special tests.

2. Materials and investigation methods

Materials used in the present investigation are austenitic stainless steels of 18Cr-9Ni and 18Cr-10Ni-Ti grades. These steels irradiated with low flux for long time at high temperatures are mainly under consideration. Irradiation conditions are as follows: irradiation temperature T_{irr}=500÷510°C, irradiation time t=111,676 and 123,456 hours and corresponding neutron fluence $F=1.8\cdot10^{21}$ and $2.5\cdot10^{21}$ n/sm^2 (E>0.1 MeV). Damage dose D corresponding to the indicated values of F is near 1 dpa. For comparison these steels in as-received condition were also tested. For experimental investigation the irradiated materials were taken from various fragments of decommissioned actuator of control and protection system of BN-600 reactor. These materials in initial (as-received) condition were taken from spare details of actuator being out of service.

For comparison these steels were also tested after relatively short term irradiation (t=3,696 hours, $F = 1.0\cdot10^{21}$ n/sm^2, D≈0.5 dpa) at T_{irr}≈550 °C. These materials were taken from material testing assembly of BN-600 reactor.

18Cr-9Ni steel was tested also after long term irradiation (t=132,000 hours) with two neutron fluences $F = 2\cdot10^{21}$ and $44\cdot10^{21}$ n/sm^2 (D ≈ 1 and 22 dpa) at lower temperature (T_{irr}≈370 °C). (It should be noted that the irradiation time is the same, and different neutron doses are caused by different fluxes.) This material was taken from simulator-assembly of BN-600 reactor.

Thus the above irradiation conditions allow one to analyze the effect of irradiation time t, irradiation temperature T_{irr} and dose D on the fracture strain and the fracture modes.

In the present study the fracture strain ε_f has been determined under uniaxial tension of standard round bars with a gauge diameter of 3 mm and gauge length of 15 mm. The fracture strain ε_f is calculated with equation $\varepsilon_f = -ln(1 - \psi)$ where ψ is the relative area reduction at rupture taken in absolute units.

It is appropriate to mention here that in Standard RD EO 1.1.2.09.0714 the fracture strain ε_f determined under tension is used as one of the main performance properties of materials that are necessary for lifetime and structural integrity assessment of BN-600 reactor components.

The mechanical tests were carried out with Schenk PSB-100 test machine in "hot" laboratory at CRISM "Prometey". Uniaxial tension tests were performed with the strain rate of 10^{-3} c^{-1} over temperature range from T=20°C to 600°C. To measure the neck diameters of ruptured round bars a television-computer device was used that provides neck images in several projections and, hence, sufficient exact measurement of average neck diameter.

To study the fracture mechanisms the fracture surfaces were examined with SEM "Hitachi TM 3000" and "Phenom" that are located in "hot" cell when using remote device.

3. The results of experimental research

The average values of the fracture strain ε_f for the investigated steels in various conditions are given in Table 1. (The left column provides conditional numbers of the investigated materials for further references to be more comfortable.)

TABLE 1. THE FRACTURE STRAIN ε_f FOR THE INVESTIGATED STEELS OF 18Cr-10Ni-Ti and 18Cr-9Ni GRADES AFTER VARIOUS IRRADIATION HISTORIES

nn	Test temperature, T °C									
	20	200	250	300	350	400	450	500	550	600
1	18Cr-10Ni-Ti steel, irradiated * ($F=1.8\cdot10^{21}$ n/sm^2, $T_{irr}\approx500^oC$, t=111,676 h.)									
	0.99	-	-	-	0.87	-	-	0.53	0.38	0.31
2	18Cr-10Ni-Ti steel, irradiated ($F=2.5\cdot10^{21}$ n/sm^2, $T_{irr}\approx500°C$, t = 123,456 h.)									
	1.20	-	-	-	-	-	0.94	-	0.48	0.24
3	18Cr-9Ni steel, irradiated ($F=2.5\cdot10^{21}$ n/sm^2, $T_{irr}\approx500°C$, t = 123,456 ч.)									
	1.14	-	-	-	0.84	-	-	-	0.51	0.41
4	18Cr-10Ni-Ti steel, irradiated ($F=1.0\cdot10^{21}$ n/sm^2, $T_{irr}\approx525\div585°C$, t = 3,696 h.)									
	1.17	-	0.99	-	0.89	-	-	0.97	-	1.02
5	18Cr-9Ni steel, irradiated ($F=1.0\cdot10^{21}$ n/sm^2, $T_{irr}\approx525\div585°C$, t = 3,696 h.)									
	1.24	-	0.99	-	0.93	-	-	0.97	-	1.00
6	18Cr-9Ni steel, irradiated ($F=2\cdot10^{21}$ n/sm^2 ($D\approx1$ dpa), $T_{irr}\approx370^oC$, $t\approx132{,}000$ h.)									
	-	1.27	-	1.24	-	1.11	-	-	-	0.86
7	18Cr-9Ni steel, irradiated ($F=44\cdot10^{22}$ n/sm^2 ($D\approx22$ dpa), $T_{irr}\approx370^oC$, $t\approx132{,}000$ h.)									
	0.89	0.78	-	0.56	-	0.49	-	0.36	-	0.24
* the data were published in [14].										

The $\varepsilon_f(T)$ values given in Table 1 (nn.1÷3) are represented in Fig. 1 together with the data for initial condition. As seen from this figure, for the investigated steels in initial condition, the fracture strain ε_f decreases slightly with temperature that is typical for BCC steels when fracture occurs on classic mechanism of transcrystalline ductile fracture [15]. At the same time, for the investigated steels after long term irradiation at high temperature, the test results have shown sufficiently large decrease in the fracture strain ε_f over temperature range $T>450^oC$ and weak decrease for $T\leq450^oC$.

These features of the mechanical behavior are in agreement with the SEM study results of the fracture surfaces. SEM photos of the fracture surfaces are shown in Fig. 2. The obtained results of the fracture surfaces examination may be summarized as follows. Fracture of unirradiated specimens over the whole investigated temperature range is classic transcrystalline ductile fracture by the mechanism of nucleation, growth and coalescence of voids for that so-called dimple fracture surface is typical.

Fracture of irradiated specimens tested at $T\leq450^oC$ also occurs by the classic transcrystalline ductile fracture mechanism. For irradiated specimens tested at $T\geq500^oC$ the mixed fracture surface is observed (Fig. 2) which consists of dimple zone and intercrystalline fracture surface. A share of intercrystalline fracture surface is near 30% of the fracture surface for 18Cr-10Ni-Ti steel and reaches 90% for 18Cr-9Ni steel. (These estimations are given for the central part of the ruptured specimens, so-called cup, where fracture is initiated for tensile specimens.)

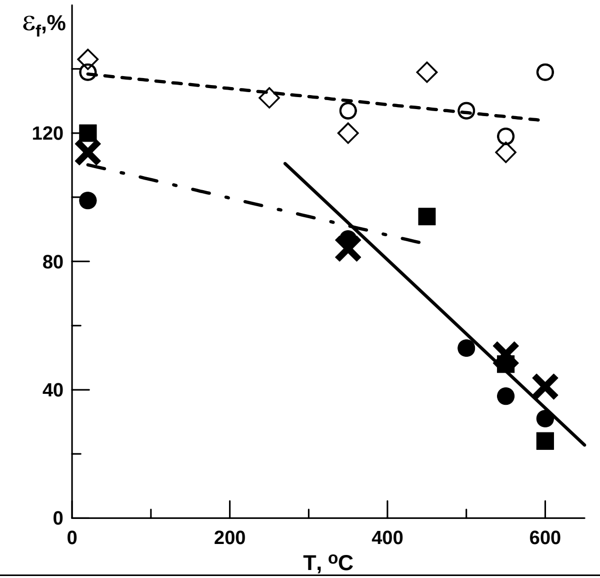


FIG. 1. Fracture strain for 18Cr-9Ni and 18Cr-10Ni-Ti steels in initial condition and after long term irradiation at high temperature: lines are the whole data set treatment with the least square method;

○ - *18Cr-10Ni-Ti, initial;*

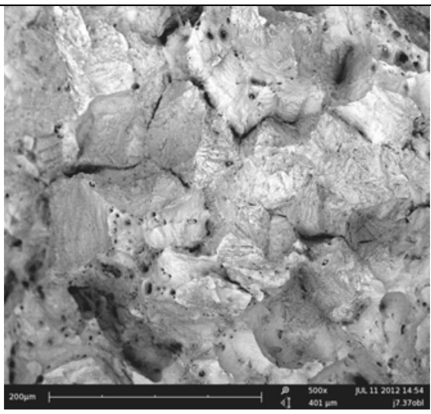
◇ - *18Cr-9Ni, initial;*

● - *18Cr-10Ni-Ti, irr. (Table 1, n. 1);*

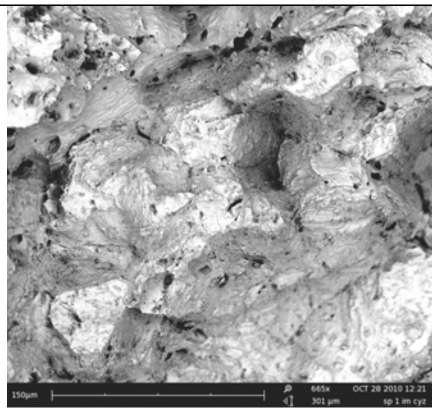
■ - *18Cr-10Ni-Ti, irr. (Table 1, n. 2);*

✖ - *18Cr-9Ni, irr. (Table 1, n. 3).*

FIG. 1. Fracture strain for 18Cr-9Ni and 18Cr-10Ni-Ti steels in initial condition and after long term irradiation at high temperature: lines are the data treatment with the least square method.



(a)



(b)

FIG. 2. Fracture surfaces of tensile round bars from 18Cr-9Ni (a) and 18Cr-10Ni-Ti (b) steels in irradiated conditions (T_{irr}≈500°C, t ≈ 120,000 h, D ≈ 1 dpa) tested at T= 600°C.

It may be seen from Fig. 2 that intercrystalline fracture occurs by brittle and ductile rupture of grain boundaries surfaces. Appearance of intercrystalline fracture correlates with decreasing the fracture strain.

Experimental data in Table 1 (n. 4) show that short term irradiation, as distinct from long term irradiation, does not result in large decrease in ε_f at the test temperature T>450^{o}C, and very slight decrease of ε_f(T) is only observed that is typical for transcrystalline ductile fracture [15]. The performed SEM studies of the fracture surfaces confirm this finding: for all these specimens the dimple fracture surfaces are observed.

The test results represented for 18Cr-9Ni steel irradiated for long time (t≈132,000 h.) at lower temperature T_{irr}=370^{o}C (see Table 1. nn. 6 and 7) show not large decrease of ε_f(T) for D≈1 dpa and large decrease of ε_f at high test temperature for D≈22 dpa. The SEM examination results shown in Fig. 3 correlate with these data. Fracture of specimens irradiated with D≈1 dpa occurs on classic transcrystalline ductile fracture mechanism over the whole temperature range including high temperatures (see Fig. 3a).

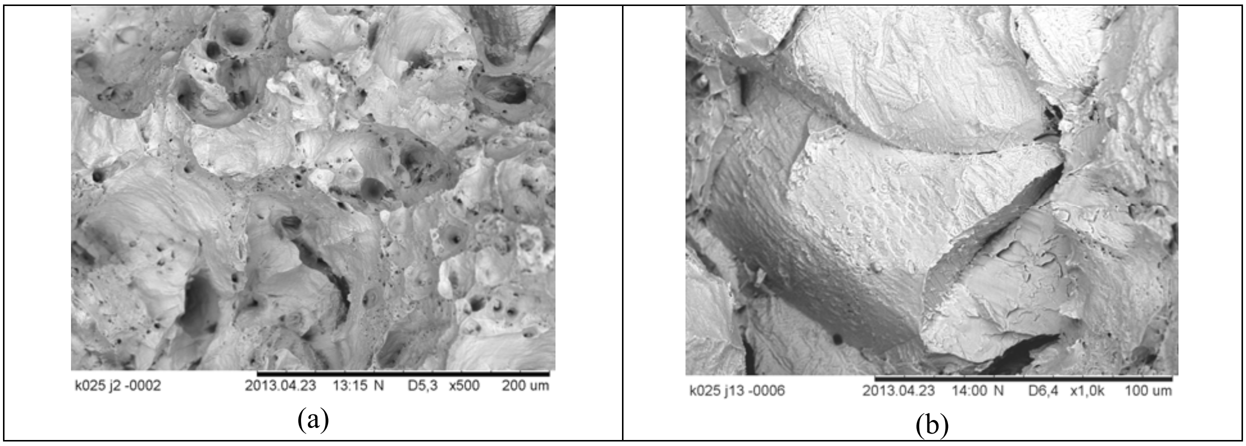


FIG. 3. Fracture surfaces of tensile round bars from 18Cr-9Ni steel after neutron irradiation at T_{irr}≈370°C for t ≈ 130,000 h. with D ≈ 1 dpa(a) and D ≈ 22 dpa (b) tested at T= 600 ℃.

At the same time for specimens irradiated with D≈22 dpa the mixed fracture mode is observed at test temperature T≥500°C (see Fig. 3b): the fracture surface consists of dimple zone and intercrystalline brittle fracture facets.

4. Discussion of embrittlement mechanism

The test results have shown that for the investigated steels after long term irradiation (t≈120,000 h.) at high temperature (T_{irr}≈500°C) the fracture strain ε_f decreases significantly over test temperature range T>450°C (see Fig. 1) and the intercrystalline fracture mode is observed (see Fig. 2).

Available data on high temperature plasticity of austenitic chromium-nickel steels irradiated in fast reactors [2, 3, 5, 6, 8] confirm in whole the obtained results. Difference mainly concerns the test temperature at which sharp decrease in plasticity begins to be observed.

It is appropriate to mention that an appearance of intercrystalline fracture with increase of test temperature is not trivial phenomenon for austenitic steels under tension with standard strain rate. Intercrystalline fracture may be expected for metal with low cohesive strength of grain boundaries when test temperature decreases as low test temperature results in large flow stress and hence in microdiscontinuities initiation and propagation on weak grain boundaries [11].

Sharp decrease of the fracture strain and appearance of intercrystalline fracture over elevated test temperature range are features of high temperature radiation embrittlement (HTRE). The main mechanism of HTRE is known to be helium accumulation mainly on grain boundaries [1-8]. When test temperature increases the diffusive mobility of helium also increases that results in growth of helium bubbles on grain boundaries and increase of pressure in bubbles. Therefore microdiscontinuities are easily initiated on grain boundaries.

Let's consider a possibility of helium mechanism for the above case.

4.1. Role of helium mechanism of HTRE for the investigated steels

The helium mechanism is stable for thermal treatment of a material and remains even at annealing temperature [16]. It means that if helium is the main reason of HTRE for the considered case its effect should be observed after annealing.

To verify the helium mechanism for the investigated steels after long term irradiation at high temperature the following test was performed. Irradiated samples were annealed at T=1,050°C during t=0.5 h. This thermal treatment regime was taken for radiation defects to be annealed and for helium accumulated under neutron irradiation to be conserved [16]. Then tensile specimens were machined and tested.

The obtained results for 18Cr-10Ni-Ti steel are shown in Fig. 4 together with the results for initial and irradiated conditions. As seen from this figure, after annealing the fracture strain increases up to the ε_f values for initial condition. The fracture surfaces of the tested specimens were examined with SEM, and intercrystalline fracture was not revealed. These results allow one to conclude that the reasons of HTRE for the investigated steels cannot be explained only with helium mechanism.

This conclusion may be confirmed also when comparing the helium concentration C_{He} for austenitic steels from various irradiated components of BN-600 FR. According to the data obtained at JSC "OKBM Afrikantov" the helium concentrations for the investigated steels taken from different components are as follows. For steel of control and protection actuator (T_{irr}≈500°C, t≈120,000 h., D≈1 dpa) C_{He}≈0.5 ppm; for steel of simulator-assembly (T_{irr}≈370°C, t≈130,000 h.) C_{He}≈0.5÷2.0 ppm at D≈1 dpa and C_{He}≈7÷14 ppm at D≈22 dpa.

As seen the helium concentration for steels for which HTRE is revealed is less than for steel from simulator-assembly irradiated with D≈1 dpa for which HTRE is not revealed. Only significant increase of the helium concentration (at D≈22 dpa) results in HTRE. It means that He is not the only reason although its concentration promotes HTRE.

Thus, a question arises whether other reasons may promote HTRE together with helium or may work instead of helium. The test results represented in Table 1 may help to answer this question. These results allow the analysis of the irradiation time and irradiation temperature effect on fracture for the investigated steels. The ε_f values in Table 1 (nn.4 and 5) after short term irradiation (t≈4,000 h.) show that there is no sharp decrease of ε_f at high test temperatures as distinct from long term irradiation (nn. 1, 2 and 3) with the close dose. The same conclusion may be drawn for steel irradiated at T_{irr}≈370°C for long time with D≈1 dpa (see Table 1 n. 6). SEM examination has not revealed intercrystalline fracture for steels after irradiation histories in Table 1, nn. 4, 5 and 6.

These findings show that the irradiation time and irradiation temperature are important factors of HTRE for the investigated steels. In other words, HTRE depends to a great extent on thermoactivated processes under irradiation. Most known thermoactivated process is thermal aging. Therefore a role of thermal aging was studied and the results are represented hereafter.

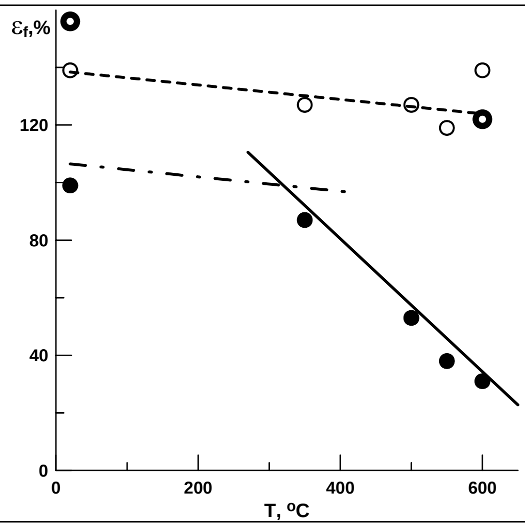


FIG. 4. Comparison of fracture strain for 18Cr-10Ni-Ti steel in initial condition, after long term irradiation at high temperature and after annealing: the lines are the same as in Fig. 1, i.e. constructed for the whole data set, and the dots are as follows:

○ - *initial*
● - *irradiated (Table 1. n. 1)*
◉ - *irradiated (Table 1. n. 1) + annealing (t=0.5 h. at T=1,050°C)*

4.2. Role of thermal aging in HTRE for the investigated steels

Thermal aging of the considered steels is known to result in segregation of various phases [11, 17], mainly, carbides of $Me_{23}C_6$ type in a grain and on grain boundaries that results in decrease of the cohesive strength of grain boundaries.

To assess the thermal aging effect on the fracture strain and fracture mode the following tests were performed as applied to 18Cr-10Ni-Ti steel. Samples from this steel in initial condition were aged at T=700°C for t=6,500 h. This thermal aging is equivalent to aging at T=550°C for t=280,000 h. (This estimation is based on the Holomon equation proposed in [17].) Then tensile specimens were machined and tested at T=20 and 600°C. The fracture surfaces of the tested specimens were examined with SEM.

The obtained ε_f values are shown in Fig. 5 where they are compared with the results for initial and irradiated conditions. These and SEM results revealed neither sharp decrease of the fracture strain nor appearance of intercrystalline fracture for the steel after thermal aging.

These results have shown that aging results in some decrease of ε_f as compared with initial condition that is in agreement with the test results for 18Cr-9Ni steel after long term service aging at T≈510°C for t≈130,000 h. [11]. It has been found in [11] that thermal aging results in significant segregation of various phases in a grain and on grain boundaries, nevertheless, intercrystalline fracture is never observed over elevated test temperature range.

Thus, it may be concluded from this study that sharp decrease of the fracture strain and appearance of intercrystalline fracture for the materials irradiated at high temperature during long time cannot be explained only by the thermal aging effect.

4.3. Synergetic mechanism of HTRE

The obtained results allow one to propose a new mechanism of HTRE for the investigated steels that is caused by the synergetic action of helium and thermal aging. Thermal aging results in decrease of the cohesive strength of grain boundaries which is sufficiently high for these steels in initial condition. (The latter follows from the fact that no intercrystalline fracture is observed for initial condition.) Accumulated under irradiation helium creates additional stresses on grain boundaries due to disjoining pressure that increases when the test temperature grows. As a result, for metal with weakened grain boundaries intercrystalline fracture becomes possible even if the helium concentration is not large.

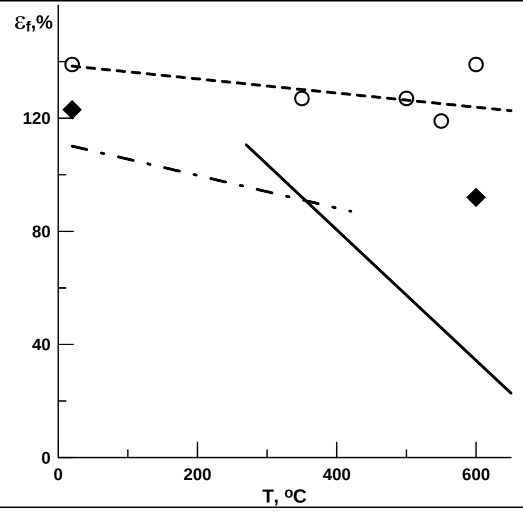


FIG. 5. Comparison of fracture strain for 18Cr-10Ni-Ti steel in initial condition, after long term irradiation at high temperature and aged condition: the lines are the same as in Fig. 1, i.e. constructed for the whole data set, and the dots are as follows:
○ - *initial;*
◆ - *thermal aging.*

It may be mentioned here that the microstructural phases due to thermal aging differ from the phases arising under long term neutron irradiation at high temperature [7, 9]. Neutron irradiation is known to accelerate diffusion processes and create additional centers for segregation such as radiation-induced dislocation loops. As a result, additional phases, for example, G-phase may be revealed in austenitic steels irradiated in FR [9].

The results of special tests represented above (see Figs. 4 and 5) have shown that two factors of the proposed mechanism, helium and aging, work jointly, and none of them is a sole reason of HTRE of austenitic steels of 18Cr-10Ni-Ti and 18Cr-9Ni grades after long term neutron irradiation at high temperatures.

The proposed synergetic mechanism of HTRE allows the explanation of all the results represented above. Moreover, this mechanism appears to explain many contradictory results concerning HTRE, for example, represented in [7]. The results in [7] have shown that for nickel a sharp decrease in plasticity occurs after high temperature neutron irradiation and does not occur after helium implantation in spite of very high helium concentration. A reason may be just in insufficiency of the only helium presence in metal with high cohesive grain boundary strength.

For steel with low cohesive strength of grain boundaries, as for the investigated steels after long term neutron irradiation at high temperatures, intercrystalline fracture may happen also over low test temperature range. The helium effect is excluded for low test temperatures, and intercrystalline fracture happens due to high yielding stresses.

This effect may clearly be demonstrated by impact strength tests that allow one to estimate materials for their sensitivity to thermal aging and, in particular, to intercrystalline fracture [11]. For this study, V-notched Charpy specimens with sizes 5x5x27.5 mm from the investigated steels in various conditions were tested at low temperatures.

The test results have shown the following. The average KCV values at test temperature T=-100^{o}C are as follows: for 18Cr-10Ni-Ti and 18Cr-9Ni steels in irradiated condition (T_{irr}≈500°C, t≈120,000 h., D≈1 dpa) KCV=48 J/cm^2 and 27 J/cm^2 respectively. For 18Cr-10Ni-Ti steel in aged condition (T=700^{o}C, t=2,000 h.) KCV=36 J/cm^2; and for 18Cr-9Ni steel in aged condition (T≈500^{o}C, t≈130,000 h. and t≈195,000 h.) KCV≈29 J/cm^2. These close values for irradiated and aged conditions mean that thermal aging processes occur intensively under long term irradiation of the investigated steels.

The SEM examination of the ruptured Charpy specimens has shown intercrystalline fracture both for irradiated and aged conditions. As illustrative example, in Fig. 6 the intercrystalline brittle fracture surfaces are shown for 18Cr-9Ni steel in aged and irradiated conditions. As seen from Fig. 6, for steel in irradiated and aged conditions very rough uneven relief is a specific feature of intercrystalline fracture facets. Classic intercrystalline brittle fracture is known to be characterized by more even intercrystalline surfaces [7].

5. Conclusions

1. The experimental research results have been represented for mechanisms of fracture and embrittlement of austenitic steels of 18Cr-9Ni and 18Cr-10Ni-Ti grades after long term neutron irradiation at high temperatures.

2. It has been found in the present research that long term neutron irradiation at high temperature (T_{irr}≈500^{o}C, t≈120,000 h., D≈1 dpa) results in significant decrease of the fracture strain for uniaxial tensile specimens tested at T>450^{o}C. This decrease is accompanied by the transition from transcrystalline ductile fracture to intercrystalline quasi-brittle fracture.

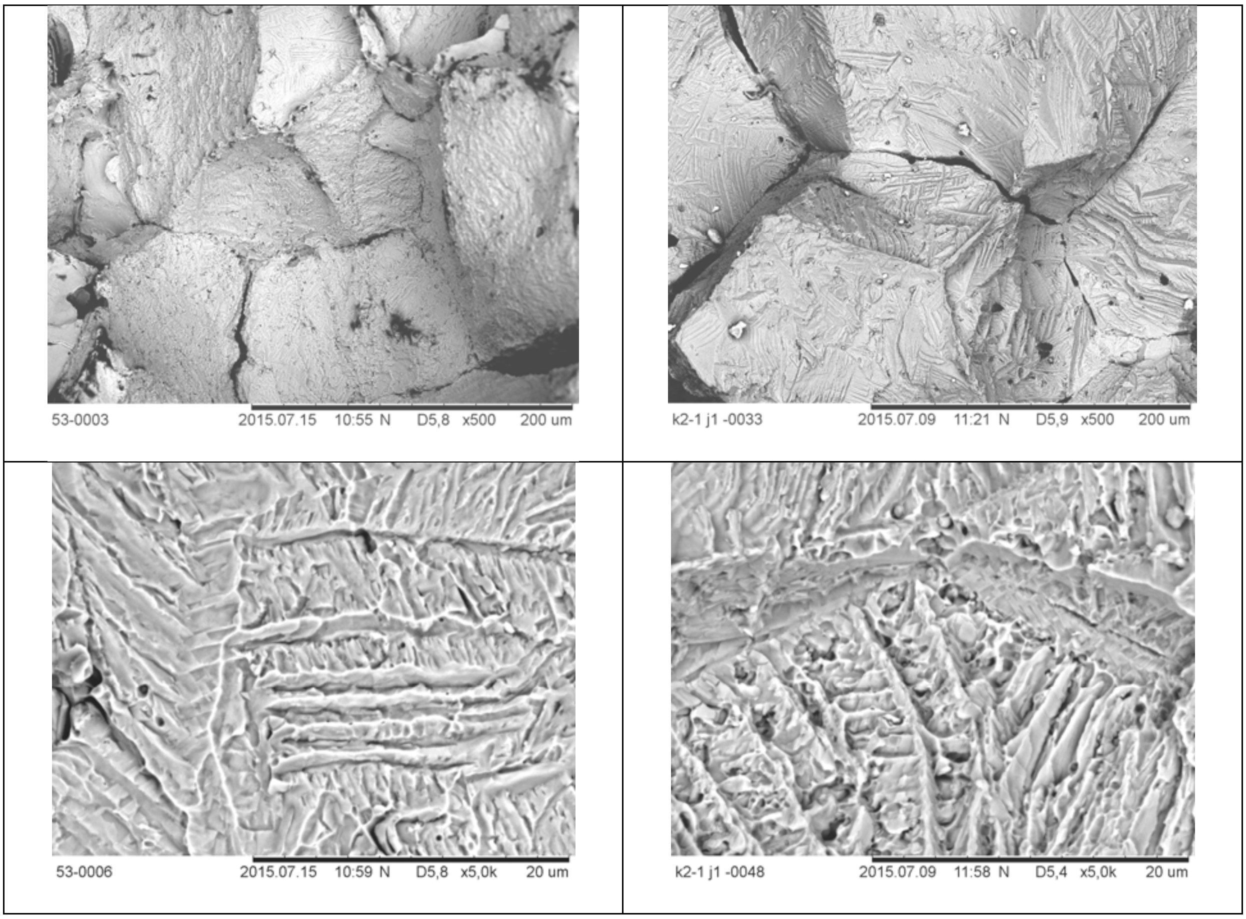


FIG. 6. Intercrystalline brittle fracture: 18Cr-9Ni steel in aged condition ($T \approx 500$ °C, $t \approx 130{,}000$ h) (left) and irradiated condition ($T_{irr} \approx 500$ °C, $t \approx 120{,}000$ h.) (right), impact strength tests, $T=-100^{o}C$.

3. Possible reasons of high temperature radiation embrittlement have been analyzed. It has been shown that the known mechanism of high temperature radiation embrittlement (HTRE) connected with accumulation and growth of helium bubbles on grain boundaries is not the only reason of this embrittlement for the investigated steels and irradiation condition.

4. It has been revealed in the present study that HTRE is caused by synergetic action of two factors - helium and thermal aging. Thermal aging results in formation of various phases on grain boundaries and, hence, in decrease of grain boundary strength. Helium diffusion at high test temperatures stimulates accumulation and growth of helium bubbles on weakened grain boundaries. Thus, thermal aging promotes the helium brittleness development. However, separately neither thermal aging nor helium bubbles results in high temperature radiation embrittlement for the investigated steels and irradiation condition.

Acknowledgments

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