

The role of nickel in forming a structure providing increased service properties of reactor structural materials*

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Abstract

Nickel is an essential alloying element in steels used as structural materials in the most common nuclear power reactors of the VVER type. The paper considers reviews the results of structural studies of traditional and advanced materials of the vessels and internals of VVER-type reactors with high nickel contents in their compositions. It is shown that an increased nickel content (up to 5 wt.%) in the steels of VVER pressure vessels contributes to the formation of a more dispersed structure with a smaller size of substructural elements and an increased density of dislocations, as well as a higher volume density of carbide phases. The revealed features of the structure of the reactor pressure vessel steel with high nickel content have the prerequisites for improving the strength and viscoplastic properties due to the increased number of barriers both for the dislocation motion and brittle crack propagation. Using the example of materials for VVER internals, it is shown that the nickel content increased in them up to 25 wt.% contributes to an increase in the volume density of radiation defects (dislocation loops of various types) and radiation-induced phase precipitates (G-phase). As nickel increases from 10 to 25 wt.%, there is a tendency to reduce swelling, which contributes to less shape change of the components of the reactor vessel internals. At the same time, in the steel with the highest nickel content, the highest nickel content was found in the near-boundary regions of the matrix, which contributes to greater austenite stability and a lower probability of the formation of an embrittling α -phase. The data obtained in the work on the effect of nickel alloying on the steel structural phase state and service characteristics were used in the development of new materials for the vessels and internals of advanced reactors.

Keywords

RPV steels, RVI steels, nickel, structural characteristics, service properties, swelling, radiation resistance

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Introduction

Nickel is an essential alloying element in steels used as structural materials for the most common nuclear power reactors, such as materials for the reactor pressure vessels (RPVs) and reactor vessel internals (RVIs). Low-carbon steels of the 15Kh2MFA and 15Kh2NMFA types (is a Russian brand of structural alloy steel, where Kh = chromium (Cr), N = nickel, M = molybdenum (Mo), F = vanadium (V), A = impurity-free), which contain up to 1.5–2 wt.% Ni, are used as materials for the RPVs. Nickel is necessary to ensure hardenability and a whole range of strength and viscoplastic characteristics. A specific feature of these steels as materials with a body-centered cubic lattice is the presence of a ductile-brittle transition. Under the action of neutron irradiation and temperature, the structure is degraded, which manifests itself in a shift in the ductile-brittle transition interval and the critical brittleness temperature towards higher temperatures. At the same time, nickel is one of the elements, which, along with other elements, including impurities, determines the degree of structure degradation, which limits its content in reactor vessel steel. However, there are prerequisites indicating that nickel at a low content of elements such as Mn, Si, P does not adversely affect the degree of degradation of the structure.

Austenitic corrosion-resistant steels of the 08Kh18N10T type (is a Russian grade of corrosion- and heat-resistant austenitic steel, where Kh = chromium (Cr), N = nickel (Ni) and T = titanium (Ti)), which contain about 10 wt.% nickel, are used as materials for the RVIs. These steels, however, are characterized by a tendency to radiation swelling due to the formation of voids under the operating conditions of the elements of the RVIs, which ultimately can lead to an unacceptable decrease in crack resistance and strength characteristics (Kursevich et al. 2013; Gurovich et al. 2015).

Bur for the development of the nuclear power industry, it is necessary to create new reactors with a long service life, efficiency and safety. These properties are accompanied by increased requirements for structural materials due to increased radiation loads, operating temperatures and mechanical loads. One of the problems concerning the steels of the vessels of advanced nuclear reactors is the need to provide higher strength characteristics at an acceptable value of the critical brittleness temperature and its shift during the entire service life, which certainly requires an increased nickel content (Markov et al. 2016a). As such materials, consideration is given to steels with increased Ni content up to 4–5 wt.% and reduced content of elements that affect the degradation of the structure and properties of the RPV steel during operation. The nickel content in this concentration range is due to the fact that lower nickel content does not lead to a significant increase in the properties, whereas a higher nickel concentration causes instability of the structure (Markov et al. 2016a).

For new RVI materials, it is necessary to provide less swelling rate and greater stability of the austenitic struc-

ture at an acceptable value of strength characteristics and cracking resistance (Kursevich et al. 2013). As a promising material, steels with 20–25 wt.% Ni are considered, since, according to estimates, this content is optimal in terms of stability of the austenitic structure and resistance to swelling (Kursevich et al. 2013). The nickel content of more than 25 wt.% has no prerequisites for a significant increase in the service characteristics. In addition, an increase in the nickel content above 25 wt.% in the steel can lead to intense swelling due to the production of a large amount of helium as a result of nuclear reactions on nickel under the conditions of a VVER reactor (Gurovich et al. 2015).

This paper summarizes the experimental results of structural studies of RPV and RVI materials with high content of Ni in advanced VVER reactors as well as the characteristics of the structure of materials that determine their service characteristics.

Materials and methods

RPV materials

The study was focused on the RPV material, which is close in composition to that of grade steel 15Kh2NMFA-A with reduced content of impurities and content of ~ 1.5 wt.% Ni, as well as a promising steel with content of up to 5 wt.% Ni (Markov et al. 2016b) with ultra-low content of Mn and impurity elements to provide increased strength characteristics and lower critical brittleness temperature, due to the steel structure already in the initial state.

RVI materials

The study included steels with 18 wt.% Cr, 10 wt.% Ni and Ti additions, as well as promising steels with a content of 20–25 wt.% Ni, which contained a slightly lower amount of Cr, but were alloyed with Mo to improve strength characteristics and reduce the level of segregation effects affecting intercrystalline corrosion (ICC) and stress corrosion cracking (SCC). Due to the fact that both Cr and Mo are ferrite-stabilizing elements, in order to maintain the stability of austenite when adding Mo, it is necessary to reduce the Cr content in accordance with the Schaeffler diagram.

To reveal the degradation features of the structure of austenitic steels with various nickel contents, we studied samples that were irradiated in the research fast reactor BOR-60 up to a irradiation dose of 29 dpa at a temperature of 425 °C. These conditions are due to the fact that at these values radiation swelling is already detected, and the main changes in the mechanical characteristics reach saturation. However, it should be noted that the swelling rate of RVI steels under VVER operating conditions may differ significantly from the swelling rate under irradiation conditions in fast reactors due to the difference in dose rates and helium production.

Research methods

The microstructure characteristics were determined with using the method of scanning electron microscopy (SEM) in the electron back-scattered diffraction (EBSD) mode. The second-phase precipitates, dislocation structures and radiation defects (dislocation loops) were investigated with using transmission electron microscopy (TEM) and SEM as well as scanning transmission electron microscopy (STEM) with applying a high-angle dark-field diffracted electron detector. The TEM method in the microdiffraction mode was used to identify carbide phases. The electron energy loss spectroscopy (EELS) methods were used for conducting elemental analysis of the precipitated phases, as well as obtaining grain boundary profiles and chemical elements distribution maps for the regions of interest in the samples. The radiation swelling of the samples was evaluated with using the TEM and SEM methods. The TEM investigations were performed on a FEI Titan 80–300 transmission electron microscope, and for the SEM investigations, a ZEISS Merlin scanning electron microscope was used.

Results and discussion

Investigation of reactor vessel steels with high Ni contents

One of the main factors that determine the strength and viscoplastic characteristics of metals is the effective grain size, which in steels with a tempered bainite structure (RPV steels belong to them) is not only the size of the primary austenite grain, but also that of substructural blocks separated by boundaries with a misorientation angle greater than 5–15° (Mohrbacher et al. 2018). Fig. 1 shows typical orientations distribution maps of substructural elements for the studied steels obtained by the SEM method in the EBSD mode.

Table 1 shows the corresponding dimensional characteristics of the substructural elements in RPV steels with various Ni contents.

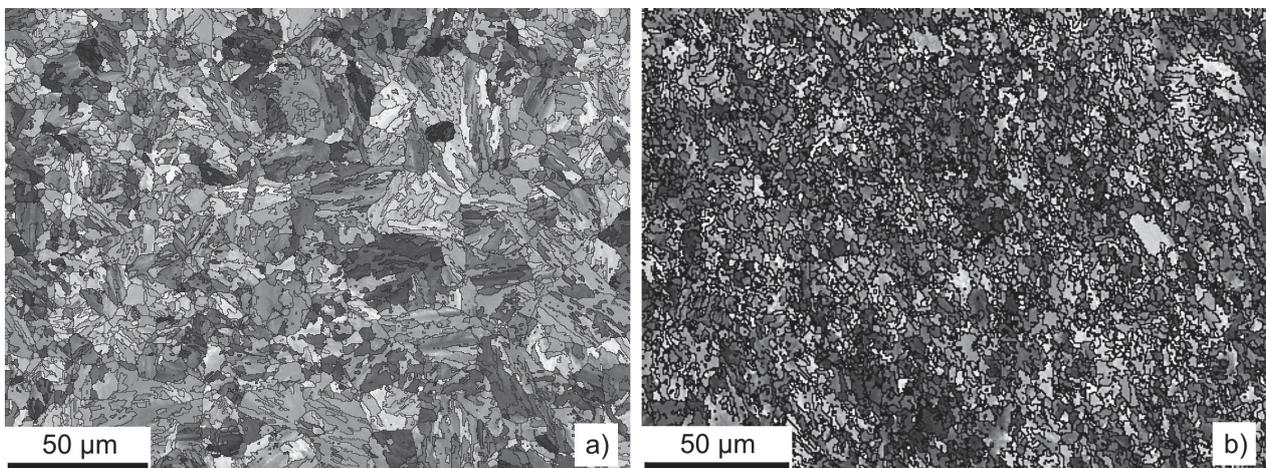


Figure 1. Orientation distribution (EBSD) maps of the substructural elements separated by high-angle boundaries in the RPV steels with Ni contents of 1.5 wt.% (a) and 5.2 wt.% (b).

Table 1. Characteristics of the microstructure of RPV steels with various Ni contents

Ni content, wt. %	Block width, μm		Block length, μm	
	Average	Range	Average	Range
1.5	2.1 \pm 0.5	0.5–9.0	5.0 \pm 0.5	1.0–15.0
5.2	1.0 \pm 0.6	0.3–5.0	2.3 \pm 0.7	0.4–14.0

The data given in the table show that, for steel with high nickel content, a half size of the substructural elements was obtained, which, apparently, is associated with the presence of a more dispersed bainitic (martensitic) component in the structure. The increased dispersion of the structure is probably associated with the fact that Ni is an austenite-stabilizing element and significantly reduces the temperatures of the onset of bainitic and martensitic transformations (Gulyaev and Gulyaev (2011)). A more dispersed subgrain structure of steel with high Ni content due to a greater number of barriers to the brittle crack propagation and dislocation motion is one of the factors influencing the ensuring both higher strength characteristics and a lower critical brittleness temperature (Qiu et al. 2020). At the same time, despite the fact that the ductile fracture stress also increases due to the increased yield strength, a decrease in the effective subgrain size leads to a more significant increase in the brittle fracture stress, which contributes to a lower ductile-brittle transition temperature (Morris et al. 2001).

It is known that, in addition to substructural hardening, the strength characteristics of metals are affected by the density of dislocations (Qiu et al. 2020), as well as large and small carbide precipitates, which are the main strengthening phases in steels of this class in the initial state (Qiu et al. 2020), acting as barriers to the dislocation motion. In general, these steels contain small carbides of MeC and Me₂C type based on V, Mo and Cr, large carbides of Me₃C, Me₇C₃, Me₂₃C₆ based on Fe and Cr and MeC type carbides based on Nb. Fig. 2 shows typical STEM images of identified large carbide precipitates. Table 2 summarizes the characteristics of the dislocation structure and carbide phases in RPV steels with various Ni contents.

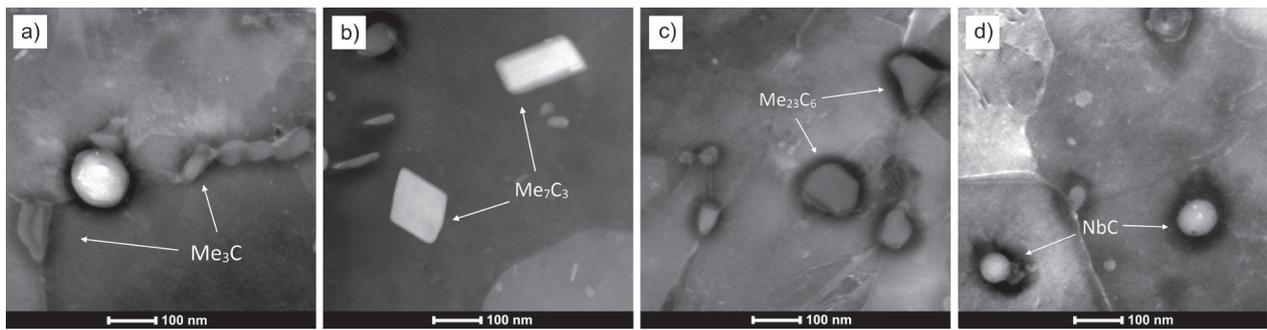


Figure 2. Typical STEM images of large carbide precipitates found in the examined RPV steels.

Table 2. Characteristics of the dislocation structure and carbide phases in RPV steels with various Ni contents

Ni content, wt. %	Dislocation density, 10^{14} m^{-2}	Small carbides		Large carbides	
		Size, nm	Density, 10^{21} m^{-3}	Size, nm	Density, 10^{19} m^{-3}
1.5	2.0–4.0	11.8±0.8	1.3±0.2	80±20	2.7±0.7
5.2	6.0–8.0	4.6±0.3	30±5	60±10	6.7±1.2

The data presented in Table 2 show that the steel with high nickel content is characterized by a significantly higher density of dislocations and carbide precipitates, in particular, small carbides, which, apparently, is one of the factors that makes it possible to obtain higher strength characteristics at an increased nickel content in the RPV steel due to dislocation and dispersion strengthening, respectively (Qiu et al. 2020).

The revealed structure features are closely related to each other. As noted, the steel with higher Ni content is characterized by a higher structure dispersity. Meanwhile, it is known that structure dispersity is associated with its defectiveness (Garcia-Mateo et al. 2009). Thus, as the transformation temperature decreases during cooling from the austenite region, shearing transformation mechanisms begin to predominate, and diffusion processes manifest themselves to a lesser extent, which leads to an increase in the dislocation density (Garcia-Mateo et al. 2009).

In addition, an increase of the nickel content contributes to a decrease in the solubility of carbon in α -Fe, which leads to a greater supersaturation of the solid solution with carbon after relatively rapid cooling from the austenitic region and subsequent intensification of the carbides formation during steel tempering (Michaud et al. 2007). In this case, a higher density of the carbide precipitates, for which dislocations are one of the preferred places for nucleation, contributes to the stabilization of the dislocation structure, which also has a positive effect on the strength characteristics (Thuvander et al. 2021).

Thus, it can be concluded that an increased Ni content in RPV steels contributes to the formation of a structure that has the prerequisites for ensuring high strength characteristics at a low value of the ductile-brittle transition temperature.

The issue of radiation and thermal resistance of RPV steel with high Ni content requires additional discussion. It is known that the embrittlement of RPV steels occurs as a result of the impact of two mechanisms: hardening

and non-hardening ones (Kuleshova et al. 2017). Each of these mechanisms, as well as the impact of both of them, leads to a shift in the ductile-brittle transition to higher temperatures. Ni takes an active part in both mechanisms (Kuleshova et al. 2019). In the hardening mechanism it stimulates the formation of a larger amount of precipitates, in particular, based on nickel (Ni-Si-Mn), whereas in the non-hardening mechanism, acting together with phosphorus, it intensifies grain-boundary segregation. Accordingly, an increased Ni content in RPV steel should lead to a significant increase in the embrittlement rate during operation. However, in this case, there are several factors, the consideration of which can make it possible to level the possible negative consequences of the increased Ni content in RPV steels.

First, it is known that Ni and Mn synergistically affect the process of precipitate formation (Kuleshova et al. 2021). For example, it was shown in Stofanek et al. 1993 that steels with ultra-low Mn content at relatively high Ni content did not show a more intense embrittlement rate. In addition, a higher expected operating temperature of the pressure vessels of advanced reactors of about 400 °C, at which no formation of radiation-induced precipitates and complexes of radiation defects was detected for traditional RPV steels, can additionally contribute to a lower manifestation of radiation hardening and embrittlement, respectively (Kuleshova et al. 2017). As for the non-hardening mechanism associated with the accumulation of grain-boundary segregation of phosphorus, it can be useful in this case to provide higher purity during steelmaking, in particular, to reduce the content of harmful impurities, including phosphorus. Thus, the combined action of these factors suggests that there are prerequisites for compensating for possible negative effects under the influence of irradiation from an increased Ni content in RPV steel in the presence of a positive effect on the strength characteristics.

Investigation of austenitic steels with high Ni contents

As already noted, the main issue for RVI steels is to ensure less swelling and greater stability of the austenitic structure at an acceptable value of strength characteristics and crack resistance.

In this regard, the nature of the change in the characteristics of radiation defects (dislocation loops of various

types) and radiation-induced phases in steels with various Ni contents is of particular interest.

Fig. 3 shows a typical dislocation structure with radiation defects in a RVI steel sample after irradiation. Table 3 presents the characteristics of radiation defects corresponding to steels with various Ni contents.

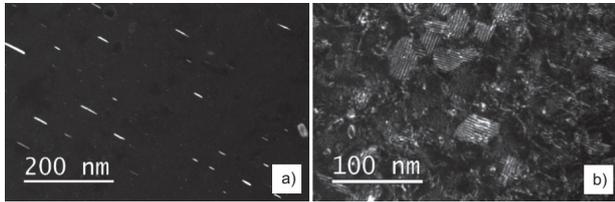


Figure 3. A typical dislocation structure with radiation defects in a RVI steel sample after irradiation: **a)** dark-field images of Frank's dislocation loops; **b)** blackdots-type defects.

Table 3. Characteristics of radiation defects in RVI steels with various Ni contents

Ni content, wt. %	Frank loops		Black dots	
	Size, nm	Density, 10 ²¹ m ⁻³	Size, nm	Density, 10 ²¹ m ⁻³
10	19±2	9±3	4.0±0.2	3.3±0.7
20	16±2	15±4	3.0±0.2	9±3
25	13±2	10±6	2.7±0.2	9±3

The data presented in Table 3 show that an increase in the nickel concentration leads to an increase in the total density of radiation defects, which can be interpreted in terms of the stacking fault energy, which increases together with the nickel content in the steel (Li 2009). An increase in the stacking fault energy correspondingly leads to the stabilization of radiation defects, i.e., dislocation loops of various types. In addition, the stacking fault energy correlates with the resistance of austenitic steels to stress corrosion cracking under irradiation, which is associated with a lower localization of plastic deformation (Li 2009).

As for radiation-induced phases, the formation of G- and γ-phases based on Ni, Si, Ti and containing other alloying elements is possible in austenitic steels under irradiation. These phases, along with radiation defects, also provide input into radiation hardening (Gurovich et al. 2015). Moreover, due to the presence of Ni in these phases, their formation can lead to depletion of the matrix near precipitates with alloying elements (Ni, Cr), that are

important for providing a high complex of service characteristics (Kursevich et al. 2013).

Fig. 4 shows a typical dark-field TEM image using the G-phase as an example and the corresponding element distribution maps obtained by energy-filtered TEM. Table 4 presents the characteristics of the G-phase corresponding to various steels.

Table 4. Characteristics of the G-phase in RVI steels with various Ni contents

Ni content, wt. %	Size, nm	Density, 10 ²¹ m ⁻³	Volume fraction, %
10	13±4	8±1	0.9
20	12±6	15±3	1.4
25	20±5	14±3	6.9

The characteristics of the G-phase presented in Table 4 show that in the steel with 25% nickel, the largest volume fraction of the G-phase is observed, which, apparently, is due to a large amount of nickel as a “building material”. Accordingly, the phase is more likely to be formed due to the local excess of nickel concentration.

It is clear that a large volume fraction of radiation-induced phases and radiation defects contributes to radiation hardening, which adversely affects the cracking resistance due to a decrease in strain hardening. However, as shown in Gurovich et al. 2015, radiation hardening (i.e., rising the yield strength) may have a less significant effect on cracking resistance even at its high values compared to swelling, which can contribute to a drop in cracking resistance to zero values (Margolin et al. 2009). In addition to a decrease in cracking resistance, swelling leads to a change in the shape of the RVI elements, which can impair their working performance. In this regard, it is of interest to compare the swelling of steels with various nickel contents as a result of irradiation under different conditions.

Fig. 5 shows a typical TEM image of voids under underfocusing and overfocusing conditions in a RVI steel sample after irradiation. Table 5 presents the swelling characteristics corresponding to steels with various Ni contents. It should be noted that, for these steels, the voids density and, accordingly, the swelling rate are extremely inhomogeneous over the cross section of the samples.

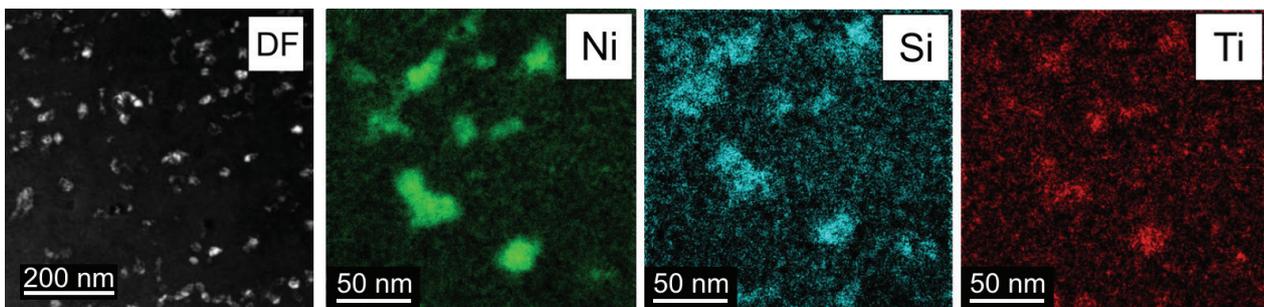


Figure 4. Typical dark-field TEM (DF) image of a G-phase and corresponding element distribution maps (Ni, Si, and Ti) obtained by energy-filtered TEM.

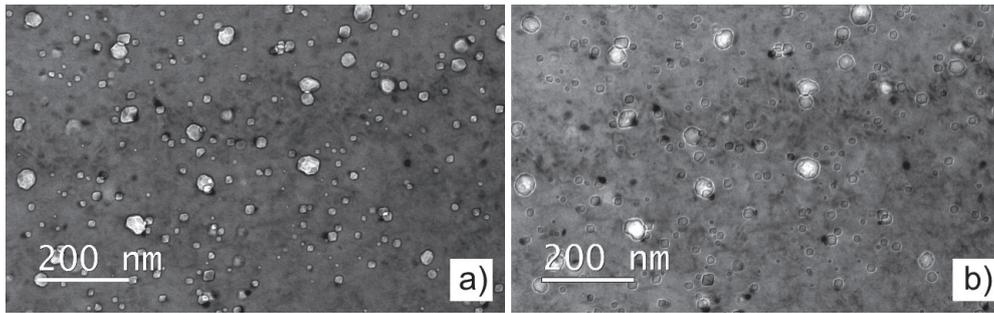


Figure 5. Typical TEM image of voids under underfocusing (a) and overfocusing (b) condition in a RVI steel sample after irradiation in the BOR-60 reactor at 425 °C to a irradiation dose of 29 dpa.

Table 5. Characteristics of swelling in RVI steels with various Ni contents

Ni content, wt. %	Swelling, % (TEM)	Swelling at a dose of 90–110 dpa, % (Margolin et al. 2019)
10	1.3±0.3	15–20
20	1.2±0.2	10–15
25	1.0±0.2	5–10

The characteristics of the G-phase presented in Table 5 show that, in general, taking into account the data obtained by the TEM method, as the nickel content increases, there is a tendency to reduce swelling, and the most pronounced effects are achieved at higher doses (Margolin et al. 2019). At the same time, for the steel with 25% nickel, the lowest swelling value was obtained, which indicates a lower swelling rate of this steel under the considered conditions. It should also be noted that the observed small difference in the swelling values for steels with various nickel contents is characteristic of the conditions of the irradiation performed (Garner 1994). The positive effect of nickel on swelling resistance is associated with a decrease in the rate of vacancy voids nucleation, in particular due to the complexes formed by nickel and interstitial atoms, which are the site for vacancies recombination (Kursevich et al. 2013).

Another factor that can lead to a significant degradation of the structure is radiation-induced segregation (RIS), since the depletion of near-boundary regions as a result of boundary enrichment can lead to conditions under which the γ - α transformation and the manifestation of radiation embrittlement inherent in the α -phase become possible

(Kursevich et al. 2013). In addition, RIS contributes to the depletion of the boundaries in chromium, which is fraught with a decrease in the corrosion resistance of the boundaries as well as the manifestation of ICC and SCC (Kursevich et al. 2013). It should be noted that the formation of the α -phase was not revealed in the investigated steels even in the steel with 10 wt. % Ni, which, apparently, is associated with a low irradiation dose.

Fig. 6 shows typical chemical elements distributions near the grain boundaries obtained by the STEM method in the EELS mode in RVI steels with various Ni contents. Table 6 presents the corresponding characteristics of RIS.

Table 6. Characteristics of grain boundary RIS in RVI steels with various Ni contents

Ni content, wt. %	Δ Ni, at. %	Δ Cr, at. %
10	+18	-8
20	+26	-8
25	+36	-9

The given elements distributions show that the level of nickel depletion of the matrix decreases as the nickel content in the steel increases, despite the increased enrichment of the boundaries with nickel. From this point of view, for the steel with 25% Ni, due to the least nickel depletion of the matrix, the formation of the α -phase and the corresponding embrittlement at higher irradiation doses are the least likely. The lower concentration of chromium observed for this steel near the grain boundaries requires separate investigations and tests aimed at identifying its susceptibility

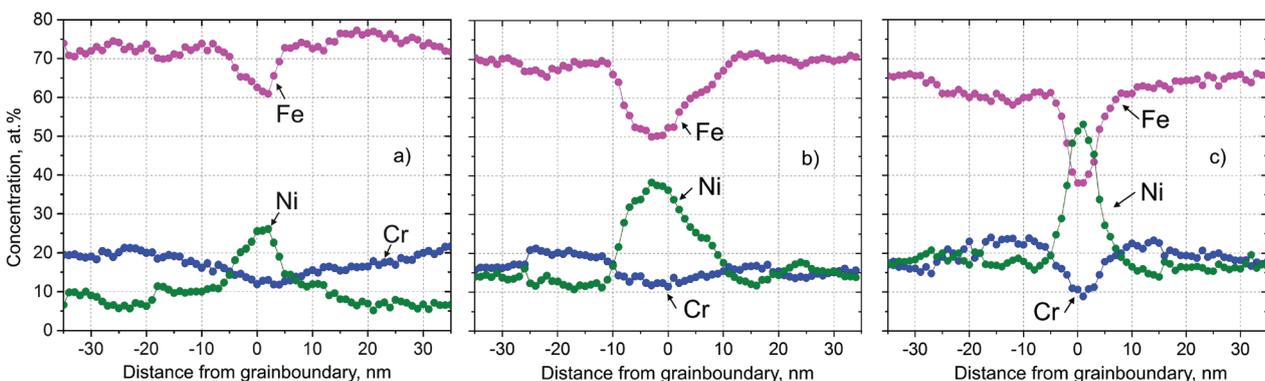


Figure 6. Typical chemical elements distributions near the grain boundaries, obtained by the STEM method in the EELS mode in RVI steels with various Ni contents: 10 (a), 20 (b) and 25 wt.% (c).

to SCC, in particular, as a result of ICC. However, there are prerequisites that allow us to assume that there are no tendencies for a more intense manifestation of SCC in the case of high-nickel steel under the operating conditions of the internals of advanced reactors. This, first of all, concerns the increased content of Mo and Ni near the grain boundaries, which have a positive effect on the resistance of steel to oxidation and SCC (Feron and Staehle 2016; Young 2016) as well as the features of the stress-strain state due to lower swelling effects (Margolin et al. 2009).

Conclusion

The authors carried out structural investigations of RPV and RVI steels with typical and increased nickel contents to identify the role and influence of this element on their structural features and service characteristics.

Structure investigations of the RPV steels with various nickel contents revealed the following:

1. The RPV steels with higher nickel content are characterized by a higher microstructure dispersity associated with lower temperatures of phase and structural transformations. This, as a result, contributes to an increase in strength characteristics and a decrease in the ductile-brittle transition temperature due to the increased number of barriers to the dislocations motion and the propagation of brittle transcrystalline cracks. The increased strength characteristics are also achieved due to a higher density of dislocations and carbide precipitates, which is also indirectly related to the effect of an increased nickel content on the structure dispersity and the solubility of carbon in α -Fe.
2. The increased rate of radiation embrittlement of steel due to the increased nickel content in RPV steels can be compensated by deep purification from impurities and higher operating temperatures, at which the rate of embrittlement of RPV steels is significantly reduced.

The studies of the structure of the RVI steels with various nickel contents showed that:

- the steels with high nickel content are characterized by higher densities of radiation defects, which is probably due to the high stacking fault energy, which contributes to the stabilization of radiation defects, i.e. dislocation loops of various types, which, although it can lead to radiation hardening and a corresponding decrease in cracking resistance, but not to critical values that limit the possibility of using the steels;
- at the considered doses in the steels, as nickel increases in the range of 10, 20, and 25%, there is a tendency to reduce swelling, which contributes to less deformations and tensile stresses;
- in the steel with 25% nickel, the highest values of enrichment of boundaries in nickel and depletion in chromium were obtained, but, at the same time, in the near-boundary regions of the matrix, the nickel content is the highest, which contributes to greater austenite stability and a lower probability of an embrittling α -phase formation, whereas depletion of boundaries in chromium requires further research and tests.

As a result, we can conclude that nickel is one of the few elements that make it possible to improve the service characteristics of the structural materials for the pressure vessels and internals of VVER-type reactors. Possible negative consequences from the increased nickel content in terms of radiation resistance are either compensated by additional measures or may be completely insignificant due to the operational factors. Due to this, we can consider RPV and RVI materials with increased Ni contents up to 5 and 25 wt %, respectively, as promising ones.

The data obtained in this work on the effect of nickel alloying on the steel structural-phase state and service characteristics were used in the development of new steels for RPVs with ~5 wt.% Ni (Markov et al. 2016b) and for RVIs with ~25 wt.% Ni (Margolin et al. 2019) in advanced reactors.

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