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Assessment of segregation kinetics in water-moderated reactors pressure vessel steels under long-term operation



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ABSTRACT

In reactor pressure vessel (RPV) bcc-lattice steels temper embrittlement is developed under the influence of both operating temperature of ~300 °C and neutron irradiation. Segregation processes in the grain boundaries (GB) begin to play a special role in the assessment of the safe operation of the RPV in case of its lifetime extension up to 60 years or more. The most reliable information on the RPV material condition can be obtained by investigating the surveillance specimens (SS) that are exposed to operational factors simultaneously with the RPV itself. In this paper the GB composition in the specimens with different thermal exposure time at the RPV operating temperature as well as irradiated by fast neutrons $(E \ge 0.5 \text{ MeV})$ to different fluences $(20-71) \cdot 10^{22} \text{ m}^{-2}$ was studied by means of Auger electron spectroscopy (AES) including both impurity and main alloying elements content. The data obtained allowed to trace the trend of the operating temperature and radiation-stimulated diffusion influence on the overall segregants level in GB. The revealed differences in the concentration levels of GB segregants in different steels, are due to the different chemical composition of the steels and also due to different grain boundary segregation levels in initial (unexposed) state. The data were used to estimate the RPV steels working capacity for 60 years. The estimation was carried out using both the well-known Langmuir-McLean model and the one specially developed for RPV steels, which takes into account the structure and phase composition of VVER-1000 RPV steels, as well as the long-term influence of operational factors. © 2016 Elsevier B.V. All rights reserved.

1. Introduction

Long-term operation of nuclear power plants (NPP) under irradiation and thermal exposure leads to the degradation of RPV bcc-lattice steel properties due to the both phase composition changes and formation of grain boundary and intergranular segregations. This reduces the resistance of materials to brittle fracture, which is determined by the shift of ductile-brittle transition temperature (DBTT) in impact tests or fracture toughness curve shift to higher temperatures. An increase of DBTT limits the RPVs safe operation time, which is particularly important when extending the RPV lifetime up to 60 years or more.

The main radiation embrittlement mechanisms are the hardening one (due to formation of radiation defects and radiationinduced precipitates) and non-hardening one (associated with the formation of grain boundary and intragranular impurities segregation). The role of the hardening mechanism (yield strength increase) is well understood, while the development of segregation processes and their effect on the RPV steels working capacity have not been given enough attention yet. The RPV lifetime extension trend both in Russia and abroad requires a more careful study of segregation processes. These processes affect both the assessment of the RPV steel current state, and their working capacity prediction for the extended period. The development of segregation processes in the RPV materials can be traced by the change in the fraction of brittle intergranular fracture, which correlates with the phosphorus concentration in the grain boundary [1,2]. However, the most accurate picture of the processes occurring in the GB under the influence of operational factors one can form on the basis of



Abbreviations: RPV, reactor pressure vessel; WM, weld metal; BM, base metal; AES, Auger electron spectroscopy; APT, atom probe tomography; SS, surveillance specimens.

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Auger studies. It should be noted that VVER-1000 RPV steels are characterized by some segregants content in GB in the initial state due to the cooling rate in the temperature range of the maximum temper embrittlement development during the standard heat treatment [3,4].

The current world decision of RPV lifetime extension raises a question of the possible modernization of RPV steels radiation embrittlement prediction and clarification of the grain boundary segregation contribution into the total model of RPV steel properties degradation (both for irradiated and non-irradiated parts). Understanding the ongoing processes in RPV steels is of particular importance when making decisions whether to extend the NPP safe operation period or carry out the restoring measures to recover initial RPV steel properties, (such as recovery annealing). In this paper the database of grain boundaries elemental composition in RPV steels surveillance specimens (SS) after long-term exposure within both irradiated and non-irradiated parts of the reactor was analyzed. The changes in the grain boundary segregation level for an extended period were also assessed using the Langmuir-McLean model [5] and the one specially developed for RPV steels that takes into account its phase composition and microstructure [6].

2. Experimentals

In this paper a comprehensive analysis of the available temperature and radiation sets of SS for both VVER-1000 base metal (BM) and weld metal (WM) was conducted. It should be noted that the specimens for studies were selected not only from different SS sets, but also specimens from the same set, irradiated with different fluxes to different fluences that is due to garlands form of the SS loading scheme inside the RPV.

The chemical composition of the studied RPV materials is presented in Table 1.

It is known that impurity atoms accumulate in the space between grains, and the thickness of the segregation zone doesn't exceed 3–5 interatomic distances [5,7]. Studies of the chemical composition of GB segregation were performed by quantitative Auger analysis, providing locality of ~10 nm and analytical sensitivity sufficient for analyzing the chemical composition of the solids to a depth of several nm from the surface, which is comparable to the GB segregation zone size [8]. Thus, the main characteristics of AES method allow a local study of the both chemical composition and elements distribution on the brittle intergranular fracture surface in RPV steels.

The measurements were performed using the PHI-700 scanning Auger Nanoprobe on the circular notched cylindrical specimens (3.2 mm diameter and 18 mm heigh). In order to obtain a clean surface and reduce the adsorbed atoms influence on the measurement results the specimens were cooled by the liquid nitrogen and fractured in a special attachment inside the UHV (ultra-high vacuum) chamber, at a pressure of $\leq 7 \cdot 10^{-10}$ torr. This sample preparation method allowed to study clean fracture surfaces for an average time of ~200 min prior to the significant effect of impurities adsorption from the vacuum, such as hydrocarbon, water, oxygen and CO_x molecules.

The fracture surface studies were carried out with 10 keV primary electron beam energy and 5 nA target current. This mode was chosen as the optimal one, since, on the one hand, it provided sufficient resolution to identify GB regions on brittle fracture surfaces, and, on the other hand, there was no carbon concentration increase during the fracture surface measurements. To perform accurate measurements in a scanning electron microscope mode GB regions of brittle fracture free of cleavages or nonmetallic inclusions were selected.

Additional GB studies by atomic probe tomography (APT) were carried out. For this purpose the specimens containing grain boundary were prepared by means of focused ion beam (FIB) lift-out method using FEI Helios Nanolab 600 machine. APT studies were carried out using the Cameca LEAP-4000 HR atom probe in voltage pulse mode. The data analysis was performed using the Cameca's IVAS 3.6.8 software.

For quantitative analysis of the measurement results, two methods were used: a simplified, expressing the phosphorus content in GB in monolayer fractions [9], as well as a method allowing to measure the atomic concentration of all the detected elements in GB. The latter is the extension of standard PHI-Ulvac quantitative Auger analysis method [10], but adapted for the GB segregation analysis.

The Auger spectra were processed by means of PHI-Ulvac MultiPack software. The obtained spectra were smoothed, differentiated and identified by reference spectra presented in Ref. [10]. The Auger lines intensity was evaluated by the peak amplitude first derivative ("peak-to-peak") [9].

According to the simplified method, the phosphorus content C_P in GB fracture surface was expressed in phosphorus monolayer fractions [9]:

$$C_P = K_{PM} \frac{I_P}{I_M} \tag{1}$$

where: I_P , I_M – phosphorus (P) and metal (M) peak intensities. The matrix coefficient for the P element adsorbed on the matrix boundary is determined from the following formula:

$$K_{PM} = 2 \left[\frac{\lambda_P(E_P) \cos \theta}{a_P} \right] \left[\frac{1 + r_P(E_P)}{1 + r_M(E_P)} \right] \frac{I_M^{\infty}}{I_P^{\infty}}$$
(2)

where $\lambda_P(E_P)$ – the mean free path of an Auger electrons of energy E_P in the matrix; a_P , r_P and r_M – terms describing the backscattering, θ – emission angle with respect to the sample surface normal, I_M^{∞} and I_M^{∞} are pure elements signal intensities specified in Ref. [10].

The relative atomic concentration of each detected element was determined by the standard PHI-Ulvac Auger spectroscopy method used and adapted in NRC "Kurchatov Institute" for the most complete and informative analysis of brittle intergranular fracture surfaces of RPV steels. This method was certified in accordance with the All-Union State Standard. It is based on differential Auger spectra analysis, wherein only those Auger lines, which are not distorted by the other elements lines superposition are selected.

Table 1	l
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Chemical composition of the studied RPV materials (in mass %)

Material	Steel grade	Composition, mass %								
		С	Ni	Р	Cu	S	Mn	Si	Cr	Мо
BM-1, BM-2 WM-1, WM-2, WM-3 BM-3	15Kh2NMFAA Sv-10KhGNMAA Research reactor material	0.15-0.18 0.04-0.09 0.25	1.00-1.35 1.10-1.89 1.07	0.006-0.010 0.002-0.014 0.018	0.04-0.12 0.02-0.08 0.10	0.009-0.013 0.005-0.015 0.040	0.38–0.54 0.53–1.10 0.47	0.21-0.33 0.25-0.36 0.31	1.82–2.23 1.41–1.89 3.30	0.51–0.59 0.56–0.68 0.37

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