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Creep behavior of EUROFER97–3 steel during neutron irradiation at 325 °C and 550 °C to 7–24 dpa

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ABSTRACT

Irradiation experiments on pressurized creep specimens of EUROFER97–3 steel were conducted using two heat treatment conditions, EUROFER97–3_1100/700 and EUROFER97–3_980/780, in the BOR-60 reactor at temperatures of 325 °C and 550 °C, reaching damage doses of 18–24 dpa. During three reactor shutdowns, intermediate unloading and outer diameter measurements of the creep specimens were performed at three different dose intervals. The results showed that, at both irradiation temperatures, the effective strain and steady-state creep rate of EUROFER97–3_1100/700 specimens were lower than those of EUROFER97–3_980/780. At 550 °C, both the effective strain and the steady-state creep rate were higher compared to 325 °C for both heat treatments. The irradiation creep modulus of EUROFER97–3 was found to be slightly higher than values reported in the literature for irradiated ferritic steels under comparable irradiation temperatures, effective stresses, and damage doses. However, in this study, the modulus exhibited a decreasing trend at damage doses exceeding 20 dpa.

1. Introduction

The EUROFER97, a reduced activation ferritic/martensitic (RAFM) steel, is expected to be a primary structural material for the DEMO blanket [1–3]. The EUROFER97 steel is the successful result of the development of low-activation alloys in Europe, which began in 1986. This steel exhibits an excellent combination of strength and plasticity, along with low activation under irradiation for all included alloying elements. The first industrial batch of EUROFER97 steel, funded by the European Commission, was produced in 1999. This paper uses the third batch of EUROFER97, produced under the Fusion for Energy (F4E) program, referred to as EUROFER97–3.

In alignment with the DEMO R&D program [4,5], critical properties of EUROFER97–3 steel—essential for both the DEMO starter and second (advanced) blankets—are evaluated through neutron irradiation tests conducted in the BOR-60 reactor, followed by post-irradiation examinations (PIEs). The BOR-60 research reactor offers high fast neutron fluxes, allowing damage doses of 20 dpa and 40–50 dpa to be achieved for the starter and second blankets, respectively, within reasonable irradiation periods of 2 and 4 calendar years [6]. These doses (20 dpa and 40–50 dpa) represent the end-of-service doses for the starter and second blankets in DEMO, with 330 °C and 550 °C being the upper temperature limits for water- and helium-cooled blanket designs. Fracture mechanics (FM) data and tensile properties are fundamental values for design rules, determining the operating temperature limits. There is also missing data concerning creep, FM, and low-cycle fatigue (LCF) properties, which will be obtained for the first time during the current irradiation campaign in the BOR-60 reactor.

The objective of this study is to investigate the creep properties of EUROFER97–3 steel using pressurized tube specimens irradiated at temperatures of 325 °C and 550 °C, with damage doses ranging from 6.8 to 24.1 dpa.

2. Experimental

2.1. Materials

The EUROFER97–3 plate, with dimensions of 1015 × 532 × 33.3

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mm, was manufactured by Saarschmiede Freiformschmiede GmbH following these primary production steps:

- melting in a vacuum induction furnace;
- remelting using a vacuum arc remelting furnace;
- forging of a 700 × 150 mm billet;
- cutting off scrap material from the top and bottom ends of the slab after forging. This scrap material was utilized for product analysis;
- rolling the plate at a trio rolling mill (thickness limit: ≥19 mm);
- conducting a quality heat treatment.

Specimens were fabricated from the final plate in two batches, differing by their heat treatment states (see Table 1).

The chemical composition of the EUROFER97–3 steel is shown in Table 2. It was measured at KIT (third column in Table 2) using mainly ICP-OES (iCAP 7600 DUO, Thermo-Fisher-Scientific) device which includes the Echelle grating optical system combined with a dual-segmented SCD detector. It allows to measure of almost all elements except gases with concentrations range from less than 1 µg/g to 100 wt. % in solids. To measure the concentrations of C, N, O gases, the Oxygen-Nitrogen Analyzer (TC600, LECO), the Carbon-sulfur analyzer (CS 600, LECO) and the G8 Galileo, Bruker AXS devices with analysis range from 0.0001 to 100 wt. % were used.

Table 2
Chemical composition of the EUROFER97–3 steel in wt. %.

Element	Delivery status	Measurement at KIT
Cr	8.50–9.50	9.00 ± 0.19
C	0.090–0.120	0.102 ± 0.009
N	0.015–0.045	0.0430 ± 0.0074
O	0.01	0.0004 ± 0.0001
B	0.002	0.0004 ± 0.0001
Al	0.01	0.0015 ± 0.0003
Si	0.05	0.0184 ± 0.0008
P	0.005	0.0017 ± 0.0003
Ti	0.02	0.00018 ± 0.00002
V	0.15–0.25	0.2117 ± 0.0044
Mn	0.20–0.60	0.529 ± 0.009
Fe	Not defined	87.5 ± 2.6
Co	0.01	0.0020 ± 0.0003
Ni	0.01	0.0099 ± 0.0005
Cu	0.01	0.0015 ± 0.0002
Nb	0.005	<0.0004
Mo	0.005	0.0021 ± 0.0003
Ta	0.10–0.14	0.125
W	1.00–1.20	1.152 ± 0.040

2.2. Creep specimens

Rod stocks were cut from the heat-treated plates, drilled, and processed to fabricate tubes with dimensions Ø5 × 0.3 mm and 28 mm in length. These tubes, along with end-caps, were then delivered from KIT to RIAR for the production of gas-filled pressurized creep specimens.

The creep properties of EUROFER97–3 steel under reactor conditions were evaluated using gas-filled creep specimens (Fig. 1). Each specimen (see the design in Fig. 1a, which shows two mutually perpendicular orientations) consists of a cladding tube with dimensions Ø5 × 0.3 mm and a length of 28 mm (2), a bottom plug (3), and an upper tip (1), inserted at both ends. The components are welded to the cladding using annular perimeter joints created via tungsten inert gas (TIG) welding in a high-purity argon atmosphere.

Following the welding of the annular joints, the specimens undergo heat treatment in a sealed metal capsule filled with high-purity helium. The heat treatment parameters are as follows:

- heating temperature T_1=720 °C;
- heating rate t_1 =10 °C/mint;
- second heating temperature T_2= 750 °C;
- second heating rate t_2 =1 °C/min;
- cooling: oven-cooled.

A final view of the EUROFER97–3 creep specimen after fabrication and heat treatment is shown in Fig. 1b The marking on the bottom plug was applied using a thermogravimetric method.

The internal argon pressure at room temperature (P_{25}), and irradiation temperatures ($P_{325/550}$), hoop stress ($\sigma_{H325/550}$), and von Mises effective stress ($\sigma_{E325/550}$) in the creep specimens placed in the IR1b and IR3 rigs are presented in Table 3.

In selecting the internal argon pressure for filling the creep specimens at room temperature, the gas pressure was recalculated to achieve the desired hoop stress. The selection process was guided by the yield strength of EUROFER97–3 steel under the two heat treatment conditions at the irradiation temperatures. Specifically, the maximum pressure was chosen to correspond to 0.5–0.8 of the estimated yield strength. Other gas pressure values were evenly distributed between the maximum and minimum of them. For calculations of pressure at the irradiation temperatures ($P_{325/550}$), the following correlation was used:

$$P_{325/550} = P_{25}T_{25}/T_{325/550} \tag{1}$$

where P_{25} is the argon pressure in MPa at room temperature;

T_{25} = 25 °C is the room temperature;

$T_{325/550}$ is the irradiation temperature of 325 °C or 550 °C.

For calculation of the hoop stress $\sigma_{H325/550}$ at irradiation temperatures of 325 °C or 550 °C, the correlation was used as follows:

$$\sigma_{H325/550} = P_{325/550} \times d_{inn}/2t, \tag{2}$$

where $P_{325/550}$, MPa is the argon pressure at irradiation temperatures of 325 °C or 550 °C; d_{inn}= 4.4 mm is the inner-wall diameter of the creep specimen;

t = 3 mm is the tube wall thickness.

To aid in comparing creep data obtained from the pressurized tube specimens such as in this study with creep data obtained from standard creep tests, the von Mises effective stress $\sigma_{E325/550}$ is used [7,8]:

$$\sigma_{F25/550} = \sqrt{3} \times \sigma_{H325/550}\Big/2, \tag{3}$$

where $\sigma_{H325/550}$ is the hoop stress at irradiation temperatures of 325 °C or 550 °C.

Table 3 shows the argon pressure and stress parameters which were used at the study of the creep specimen behavior under irradiation and calculated using the correlations (1–3).

The relative creep strain ε at this study is obtained by the measurements of outer-wall diameters d of the creep pressurized specimens as follows:

$$\varepsilon = \Delta d/d_0 = ((d_{ir} - d_0)\,/\,d_0) \times 100\%, \tag{4}$$

where d_0, mm is the diameter of the creep specimen before irradiation; d_{irr}, mm is the diameter of the creep specimen after irradiation.

The outer-wall strain ε is converted into a mid-wall strain ε_M using a

Table 1
Heat treatment of the EUROFER97–3 creep and swelling specimens in two states.

State/Grade	Austenitization	Quenching	Tempering	Final cooling
1/EUROFER97–3_1100/700	1100 °C, 0.5 h	air	700 °C, 2 h	air
2/EUROFEF97–3_980/780	980 °C, 0.5 h	air	780 °C, 2 h	air

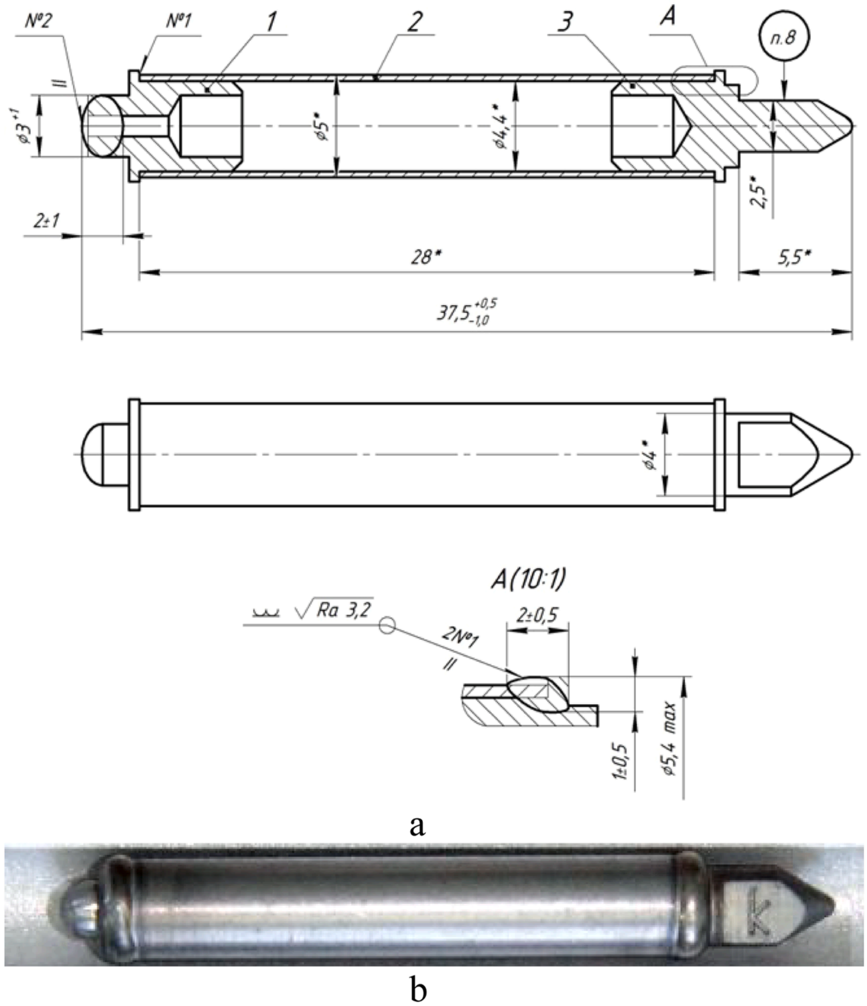


Fig. 1. Creep pressurized specimen of EUROFER97–3 steel.

Table 3
The internal argon pressure P_{25} at room temperature (25 °C) and the argon pressure $P_{325/550}$, the hoop stress $\sigma_{H/325/550}$, the von Mises effective stress $\sigma_{E325/550}$ at irradiation temperatures of 325 °C and 550 °C in EUROFER97–3 creep specimens.

State/T_{irr}, °C	P_{25}, MPa	$P_{325/550}$, MPa	$\sigma_{H/325/550}$, MPa	$\sigma_{E325/550}$, MPa
1/325	20	40	293	254
	15	30	220	191
	10	20	147	127
	5	10	73	63
2/325	16	32	237	205
	13	26	191	165
	10	20	147	127
	5	10	73	63
1/550	16	44	323	280
	13	36	264	229
	10	28	205	178
	5	14	103	89
2/550	8	22	161	139
	6	17	125	108
	4	11	81	70
	2	5.5	40	35

conversation factor of the order of 1.1 which is constant for outer-wall strains up to 10 % [8,9]. Then, the mid-wall strain ε_M can be converted to an effective plastic strain ε_E [8–10] as follows:

$$\varepsilon E = 2 \times \varepsilon M_M \Big/ \sqrt{3} \tag{5}$$

where ε_M, % is the relative mid-wall strain.

Despite the fact that internal pressure and wall thickness of the creep specimens decrease with time, the stresses in the tube wall remain constant for uniform strains as large as 10 %. This is different to a typical uniaxial creep test in which the load remains constant, and the stress increases as the cross-sectional area of the specimen decreases [8].

2.3. Swelling specimens

The swelling behavior of the EUROFER97–3 steel was studied using cylindrical specimens with sizes of Ø5 × 20 mm. The density of the swelling specimens before and after irradiation was evaluated using a density measurement unit of the AX analytical balance Mettler Toledo, Switzerland.

The density was measured as follows:

- the dry weight of the specimens was determined by weighing in air (with an accuracy of 0.1 mg);
- the weight of a reference of known volume was measured to determine the density of the working fluid (carbon tetrachloride) before starting to measure the swelling specimens;
- the weight of each specimen and the density reference in the working fluid was measured in the same way as weighing in air (the

working fluid temperature was controlled according to the thermocouple readings);
- the value of the specimen density ρ was calculated according to the formula:

$$\rho = \rho_{lq} \times m_{dr} / (m_{dr} - m_{lq}), \quad (6)$$

where ρ_{lq} is the density of the liquid used, g/cm^3; m_{dr} is the dry weight of a specimen, g; m_{lq} is the weight of a specimen in the liquid, g.

For each swelling specimen, 5 dry weight measurements and 5 liquid weight measurements were performed. The accuracy of the swelling measurements is ± 0.01 %. Before the density measurements, the irradiated swelling specimens were washed in a 95 % ethyl alcohol-water solution.

2.4. Irradiation

Irradiation of the gas filled creep specimens of EUROFER97–3 steel of two heat treatments was conducted in the IR1b and IR3 rigs in the BOR-60 fast reactor with a sodium coolant. The creep specimens were washed with liquid sodium during irradiation. Both irradiation temperature and neutron fluence (see Table 4) of each rig have been determined by the neutronic and thermohydraulic calculations. The accuracy of the irradiation temperature calculation is ± 10 %, and for neutron flux is ± 20 %. The damage dose calculation was performed according to the current international Norgett-Robinson-Torrens (NRT) standard model [11,12] for the rigs and reactor channels.

To verify the IR-3 (T_{irr}= 550 °C) calculations, a short-term methodical experiment was performed before the first cycle of the long-term irradiation, using a thermal probe, in the D-23 cell of the BOR-60 reactor core. Specimen dummies were placed in the IR suspension. According to the short-term methodical experiment results, a cell for long-term irradiation was selected by keeping a close neutron flux.

The irradiation temperatures and the neutron fluences on the creep specimens were controlled not only by calculations but also experimentally using fusible monitors and neutron activation detectors. For this purpose, a set of fusible monitors and neutron activation detectors were installed in the IRs and replaced with each removal of the creep specimens for interim measurements from the rigs. The investigations of the fusible monitors and neutron activation detectors showed a good agreement between the obtained results and the calculated data for the irradiation temperatures and neutron fluences.

2.4. Research methods

The diameters of the creep specimens were measured on an automated laser measuring system, which includes:

- laser micrometer Mitutoyo series 544 LSM-902 with a digital indication device with accuracy ± 1 μm:
- the specimen installation unit;
- the unit to move a specimen to be measured;
- the unit to rotate a specimen at a given angle.

The software of the measuring system allows for automated measurements, displaying data in real time and recording and storing measurement results in both ASCII text and in Excel formats. Before measuring the irradiated specimens, the caliber was measured. The measured caliber diameter was within the allowable error of ± 1 μm. Before measurements, the irradiated specimens were washed in a 95 % ethyl alcohol-water solution.

Before irradiation, the creep specimens' diameters were measured by an automatic laser measuring system PROFILAB.X40 with accuracy ± 1 μm.

The creep specimens were measured in five positions along the length: in the center and at distances of ± 5 and ± 10 mm from the specimen center, in two mutually perpendicular directions. After preliminary processing of the measurement results at five positions along the length of the specimen, the results at the specimen center were selected for subsequent analysis since they showed the maximum strain values.

Table 4
Irradiation parameters of the EUROFER97–3 creep specimens where T_{irr} is the irradiation temperature, F is the neutron fluence, D_{max} is the maximum damage dose for this irradiation temperature.

Rig	T_{irr}, °C	F, × 10^{26} m^{-2}, $E > 0.1$ MeV	D_{max}, dpa
IR1	325–342	4.10 ± 0.22	18.3 ± 1.0
IR3	519–568	5.28 ± 0.14	24.1 ± 0.7

3. Results

3.1. Swelling

The results of the swelling measurements for the EUROFER97–3 specimens are presented in Tables 5 and 6 for IR1 and IR3 rigs, respectively. These results indicate that the density of the swelling specimens after irradiation either remains unchanged or changes insignificantly with increasing damage dose. This suggests that the swelling of EUROFER97–3 steel is negligible under the given irradiation parameters [13].

3.2. Creep

Fig. 2 shows the dependence of the effective strain ε_E of the EUROFER97–3 steel creep specimens, differing of the heat treatment and the irradiation temperature, on increasing damage dose D. For all combinations of heat treatment and irradiation temperature factors, there is an increase in the strain with increasing dose. Also, for all combinations of these factors, one can see a tendency of increasing strain ε_E with increasing effective stress σ_E. Significant differences are visible in the behavior of the creep specimens with different heat treatments and at different irradiation temperatures. At both irradiation temperatures, a greater strain of EUROFER97–3_980/780 compared to EUROFER97–3_1100/700 is clearly visible. If to compare each of two states of the EUROFER97–3 steel irradiated at two different temperatures, it is obvious that the strain is higher at 550 °C compared to the 325 °C irradiation temperature.

Fig. 3 shows the steady state creep rate ε' of the EUROFER97–3 specimens versus the effective stress σ_E. This graph shows the averaging of the relative strain for two specimens of each effective stress. It is evident that the creep rate is very low for two states of EUROFER97–3 steel and both irradiation temperatures. However, for both irradiation temperatures the creep rate is comparatively higher for state 2 of the steel to state 1. It should also be noted that the creep rate is

Table 5
Density ρ of EUROFER97–3 swelling specimens in IR1 in g/cm^3 where ρ_0, ρ_1, ρ_2, ρ_3 are the density values in non-irradiated state, and after irradiation at 325 °C to damage doses of 6.8, 12.7, 18.3 dpa, respectively.

Grade	ρ_0	ρ_1	ρ_2	ρ_3
EUROFER97–3_1100/700	7.83	7.83	7.84	7.83
	7.83	7.83	7.84	7.84
	7.83	7.83	7.84	7.84
	7.83	7.83	7.84	7.84
EUROFER97–3_980/780	7.83	7.82	7.83	7.83
	7.83	7.83	7.83	7.83
	7.83	7.83	7.84	7.84
	7.83	7.83	7.84	7.83

Table 6
Density ρ of EUROFER97–3 swelling specimens in IR3 in g/cm^3 where ρ_0, ρ_1, ρ_2, ρ_3 are the density values in non-irradiated state, and after irradiation at 550 °C to damage doses of 8.1, 16.1, 24.1 dpa, respectively.

Grade	ρ_0	ρ_1	ρ_2	ρ_3
EUROFER97–3_1100/700	7.83	7.83	7.84	7.83
	7.84	7.83	7.84	7.83
EUROFER97–3_980/780	7.83	7.82	7.83	7.82
	7.83	7.83	7.84	7.83

comparatively higher for the higher irradiation temperature of 550 °C to 325 °C. An important fact is also the tendency for the creep rate to increase with increasing effective stress, which is different for each of the two irradiation temperatures. At 325 °C the creep rate tends to slow down with increasing stress while at 550 °C the creep rate slightly accelerates with increasing stress. It should also be noted that after increasing the dose from 6.8 to 12.7 dpa for 325 °C and from 8.1 to 16.1 dpa for 550 °C, a general decrease in the creep rate occurs for all stresses, but a further increase in the dose to 18.3 dpa for 325 °C and 24.1 dpa for 550 °C does not lead to a further general decrease in the creep rate, that is, a saturation effect is observed in the tendency to decrease the creep rate with increasing dose.

Fig. 4 shows the stress-normalized creep strain ε_E/σ_E on damage dose D for different effective stresses ε_E in the EUROFER97–3 pressurized specimens. This ratio increases with increasing dose, while a general decrease in the value of this ratio with increasing effective stress in the shell is clearly observed. It should also be noted that state 1 has a smaller absolute value of the ratio compared to state 2. Also, for both states the ratio is significantly higher for the higher irradiation temperature of 550 °C compared to 325 °C.

4. Discussion

According to [14–17], the stress-normalized creep strain ε_E/σ_E is expressed by the following correlation:

$$\varepsilon_E / \sigma E = \mathrm{B} \times \mathrm{D}, \tag{7}$$

where D, dpa is the damage dose;

B, (MPa × dpa)$^{-1}$ is the irradiation creep modulus.

Therefore, the irradiation creep modulus B can be expressed from (7) as follows:

$$B = \varepsilon E / \sigma E / D, \tag{8}$$

Knowledge of B depending on the effective strain and stress in the specimen shell and irradiation parameters (temperature and damage dose) allows one to quantitatively evaluate the ability of the specimen material to exhibit the creep effect under given conditions and compare it with other structural materials. It should be noted that for ferritic steels, swelling under the irradiation parameters considered in the work is a small value, even, for example, the swelling rate was estimated as 0.012 %/dpa for the HT9 and 9Cr-1Mo ferritic steels irradiated to 208 dpa at 400 °C [18–20], therefore, in the following, the swelling effect will not be taken into account, since the considered dose of ~20 dpa is too low for its noticeable manifestation.

4.1. Irradiation temperature of 325 °C

It is known that at 325 °C thermal creep in EUROFER97 and other ferritic steels does not occur [20], while under irradiation conditions the irradiation creep occurs [19–21]. In the present work (see Fig. 2a,b), the effect of radiation creep is also clearly manifested.

Fig. 5 shows the irradiation creep modulus B behavior of EUROFER97–3 on increasing damage dose D under irradiation at 325 °C to a damage dose of about 20 dpa. There is a significant difference in the value of B between EUROFER97–3_1100/700 and EUROFER97–3_980/

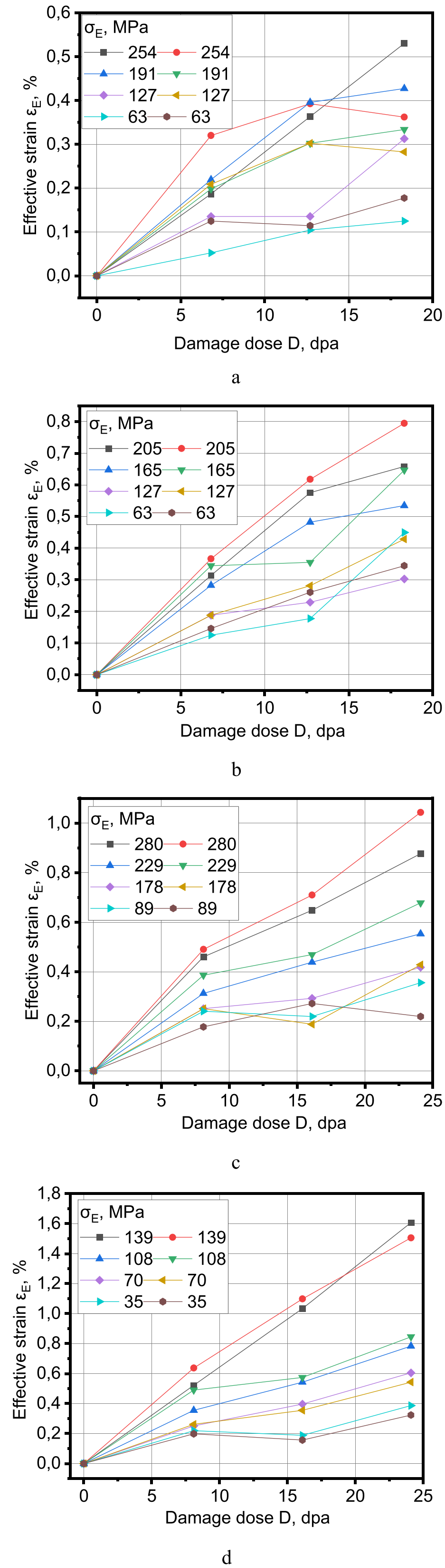
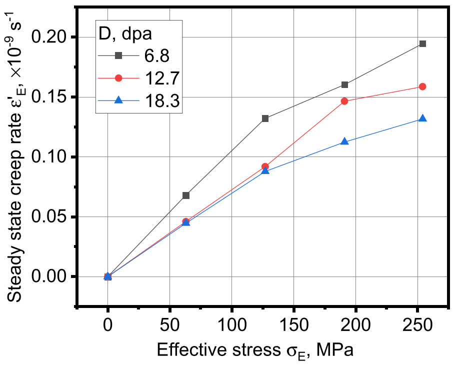


Fig. 2. Effective plastic strain ε_E for creep specimens EUROFER97–3 as a function of damage dose D:.
a) T_{irr}=325 °C, EUROFEF97–3_1100/700;b) T_{irr}=325 °C, EUROFER97–3_980/780;c) T_{irr}=550 °C, EUROFEF97–3_1100/700;d) T_{irr}=550 °C, EUROFEF97–3_980/780



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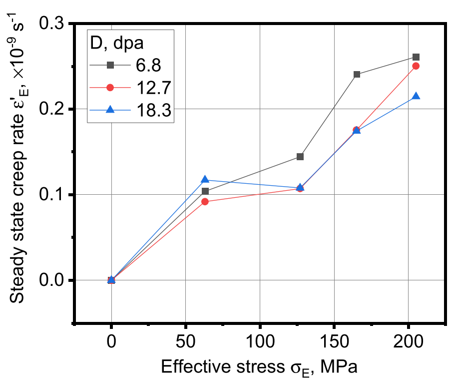

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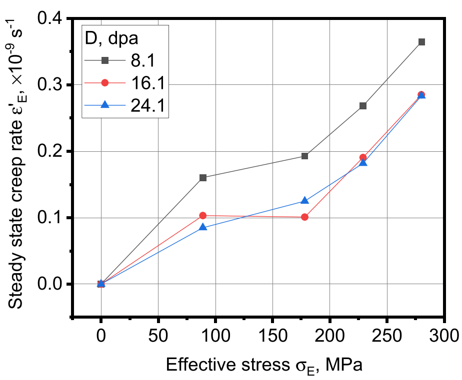

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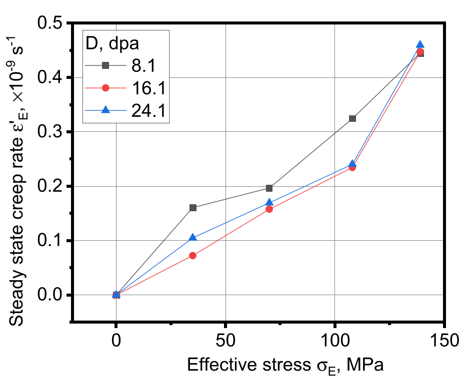


d

Fig. 3. Steady state creep rate ε' of creep specimens EUROFER97–3 as a function of effective stress σ_E:.
a) T_{irr}=325 °C, EUROFER97–3_1100/700;b) T_{irr}=325 °C, EUROFER97–3_980/780;c) T_{irr}=550 °C, EUROFER97–3_1100/700;d) T_{irr}=550 °C, EUROFER97–3_980/780

780. In general, state 1 has lower B compared to state 2. A tendency towards a decrease in B with increasing damage dose is also visible for both heat treatments of EUROFER97–3 steel excluding the lowest effective stress of 63 MPa where an unregular behavior of B is observed.

4.2. Irradiation temperature of 550 °C

Fig. 6 shows the irradiation creep modulus B behavior of EUROFER97–3 on increasing damage dose D under irradiation at 550 °C to a damage dose about 25 dpa. The difference in the B value between the two heat treatments of EUROFER97–3 is significantly more pronounced: despite the significantly lower value of the applied effective stresses, B for EUROFER97–3_980/780 (Fig. 6b) significantly exceeds B for EUROFER97–3_1100/700 (Fig. 6a). The tendency for B to stabilize in the dose range from 15 to 25 dpa is also more pronounced, with a significant decrease from the beginning of irradiation to a dose of 15 dpa. At an irradiation temperature of 550 °C, both thermal and radiation creep components contribute to the effective strain so, B is comparatively higher than at an irradiation temperature of 325 °C, with comparable effective stresses. It is also noteworthy that B decreases over the entire dose range with a decrease in the applied effective stress, in contrast to an irradiation temperature of 325 °C, where such a clear correlation is not observed.

4.3. Features for both 325 °C and 550 °C irradiation temperatures

It is necessary to note the limited number of experimental results on radiation creep of ferritic steels and, in particular, for EUROFER97 steel. In [22], F82H IEA heat (8Cr-2WVTa) and JLF-1 (9Cr-2WVTa) steels were studied using helium-pressurized creep tubes irradiated at temperatures of 300 °C and 500 °C to a dose of 5 dpa. The effective strain for both steels at 300 °C does not exceed 0.2–0.35 % at an effective stress of 350 MPa, and 0.5–1 % at 500 °C for 130–140 MPa, which correlates well with the results of the present work (see Fig. 2). In [23], pressurized tubes of 9Cr1Mo-(EM10), EUROFER and 9Cr2WTaV steels were irradiated to determine the deformation due to irradiation-creep at 300 °C with doses ranging from 19.3 to 63 dpa. These ferritic steels show nearly the same average value of the irradiation creep modulus, which is $B = (0.7 \pm 0.1) \times 10^{-6}$ (dpa × MPa)$^{-1}$. This value of B is somewhat lower than the results obtained in our study at an irradiation temperature of 325 °C (see Fig. 5), however, in [23], the irradiation temperature is lower and the damage dose is significantly higher. Additionally, in our results, a tendency for B decrease with increasing dose takes place. In [8,16,17,19,24,25], there are values of the irradiation creep modulus for ferritic steels such as $(0.5–0.8) \times 10^{-6}$ (dpa × MPa)$^{-1}$ which are lower than the values obtained in this study ($(0.6–7) \times 10^{-6}$ (dpa × MPa)$^{-1}$). However, increasing the irradiation dose should result in a decrease in the modulus.

4.4. Microstructure evolution

It was found by Duerrschnabel [26] before irradiation that the microstructure of EUROFER97–3 can be characterized by an average grain size of 5–6 μm and containing the $M_{23}C_6$ phase with the average particle sizes of 137–152 nm located mainly on grain sizes as well as the VN (29–35 nm) and TaC (45–52 nm) phases located in the grain bodies, and with the number density of all types of precipitates in the range of 10^{19}–10^{20} particles per m^2. Structural studies of the irradiated EUROFER97–3 steel will be conducted and published later. Presumably, the effect of irradiation will consist of the evolution of the phase structure of steel, leading to a change in the effective creep rate at given effective stresses and, accordingly, to evolution of the irradiation creep modulus.

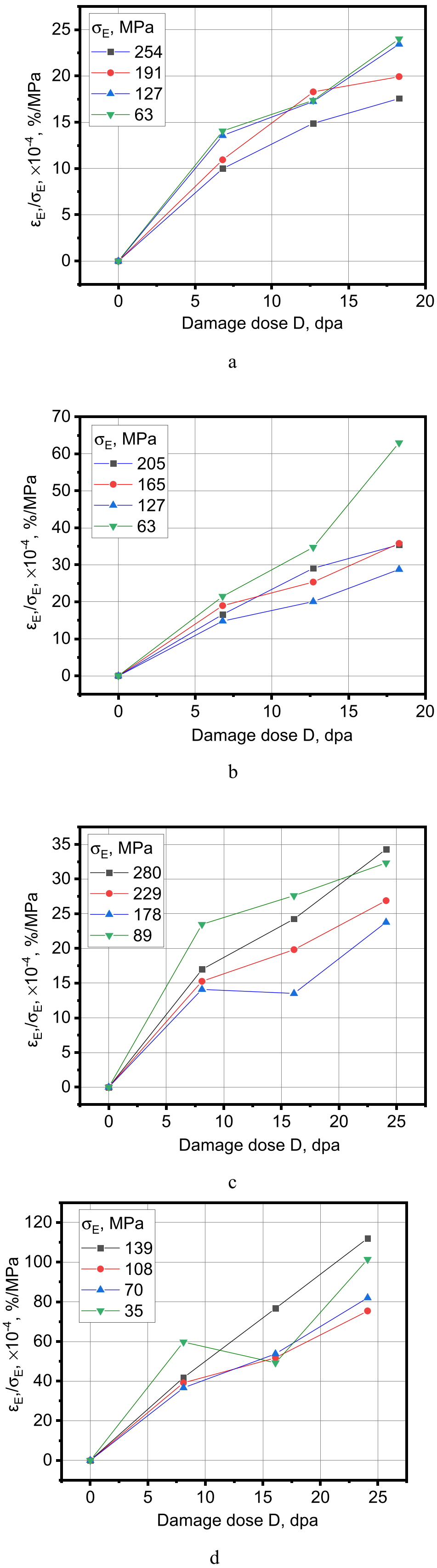


Fig. 4. Dependence of ε_E/σ_E on damage dose D for creep specimens EUROFER97–3:.
a) T_{irr}=325 °C, EUROFER97–3_1100/700;b) T_{irr}=325 °C, EUROFER97–3_980/780;c) T_{irr}=550 °C, EUROFER97–3_1100/700;d) T_{irr}=550 °C, EUROFER97–3_980/780

Klimenkov [27] investigated the microstructure of the EUROFER97 steel after annealing at 450, 500, 550, 600, 650 °C for 120,000 h (≈14 years). The long-term annealing resulted in significant evolution of the microstructure, which can be approximately comparable with the effect of irradiation at the corresponding temperatures. It was found that there is a modification of existing phases and the formation of new phases. In particular, the size of $M_{23}C_6$ precipitates increases significantly at 650 °C. At lower temperatures the size of $M_{23}C_6$ particles moderately increases (at 600 °C) or even slightly decreases (<600 °C). The composition of these precipitates changes only slightly. The Cr content in the $M_{23}C_6$ precipitates increases with increasing annealing temperature from 67 to 75 %. The V content in (Ta,V)C and (V,Ta)N particles increases slightly with annealing at 650 °C, while their composition does not show significant changes at lower temperatures. The formation of a modified Z-phase (CrVN) was detected at 550 °C and 600 °C. The size of the Z-phase particles can reach up to 1 μm. At 600 °C, the formation of Z-phase was observed after a few thousand hours. Formation of intermetallic WFe_2 Laves phase was observed at 500 °C and 550 °C. At annealing times of more than 120 kh, the size of the Laves phase particles can reach up to 1 μm.

Neutron irradiation of EUROFER97–3 steel at temperatures of 325 °C and 550 °C can lead not only to modification of the phase structure existing before irradiation and formation of radiation-induced new phases, but also to formation of radiation defects, such as dislocation loops, voids and bubbles [28–30]. Only direct TEM examination of irradiated EUROFER97–3 specimens will allow to establish the nature of the real evolution of the microstructure to find an explanation for the obtained results on creep behavior of this steel presented in this study.

5. Conclusions

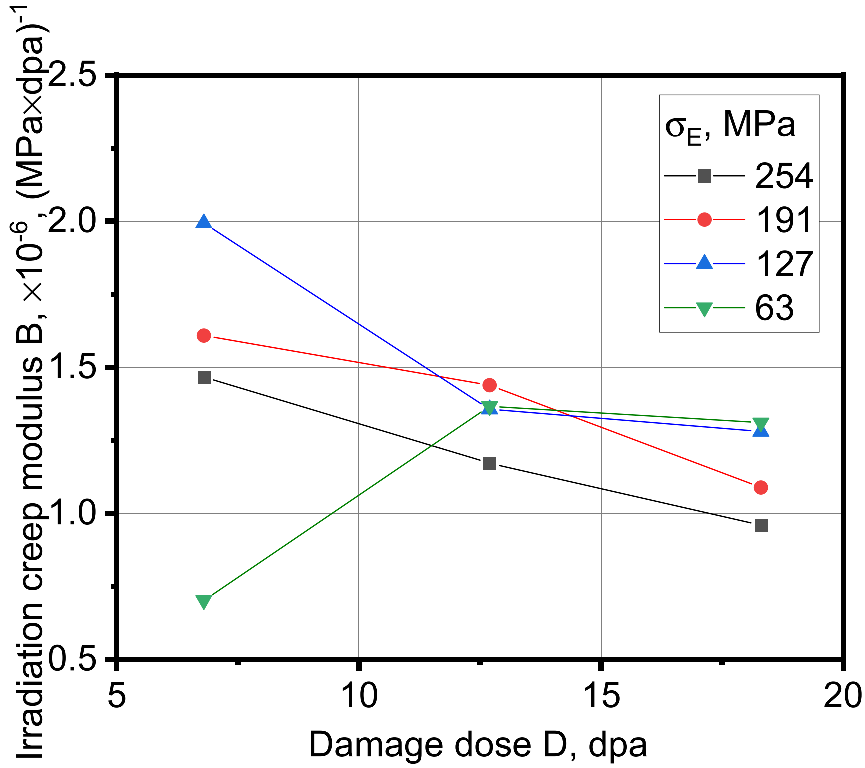
The study of creep behavior was conducted on pressurized creep specimens of EUROFER93–7 steel with two heat treatments during irradiation in BOR-60 reactor at 325 °C and 550 °C to damage doses of 6.8 to 24.1 dpa.

If to compare the behavior of EUROFER93–7 steel after each of the two heat treatments and at different irradiation temperatures, the following features can be noted:

- lower effective strain and steady-state creep rates at both irradiation temperatures for specimens of EUROFER97–3_1100/700 heat treatment compared to EUROFER97–3_980/780;
- for an irradiation temperature of 550°C, the effective strain and the steady-state creep rate are higher compared to an irradiation temperature of 325°C for both heat treatments of the EUROFER97–3 steel;
- the obtained irradiation creep modulus is slightly higher than the values of the modules from literature on irradiated ferritic steels for comparable irradiation temperatures and effective stresses. At high damage doses, however, the modulus measured and calculated in the work tends to decrease with an increasing dose of more than 20 dpa.

CRediT authorship contribution statement

Vladimir Chakin: Writing – review & editing, Writing – original draft, Supervision, Methodology, Investigation, Conceptualization. **Carsten Bonnekoh:** Writing – review & editing, Methodology, Investigation, Formal analysis. **Rainer Ziegler:** Writing – review & editing, Investigation. **Ramil Gaisin:** Writing – review & editing, Visualization, Methodology, Investigation. **Michael Duerrschnabel:** Writing – review & editing, Methodology, Investigation. **Michael Klimenkov:** Writing – review & editing, Investigation. **Michael Rieth:** Writing – review &



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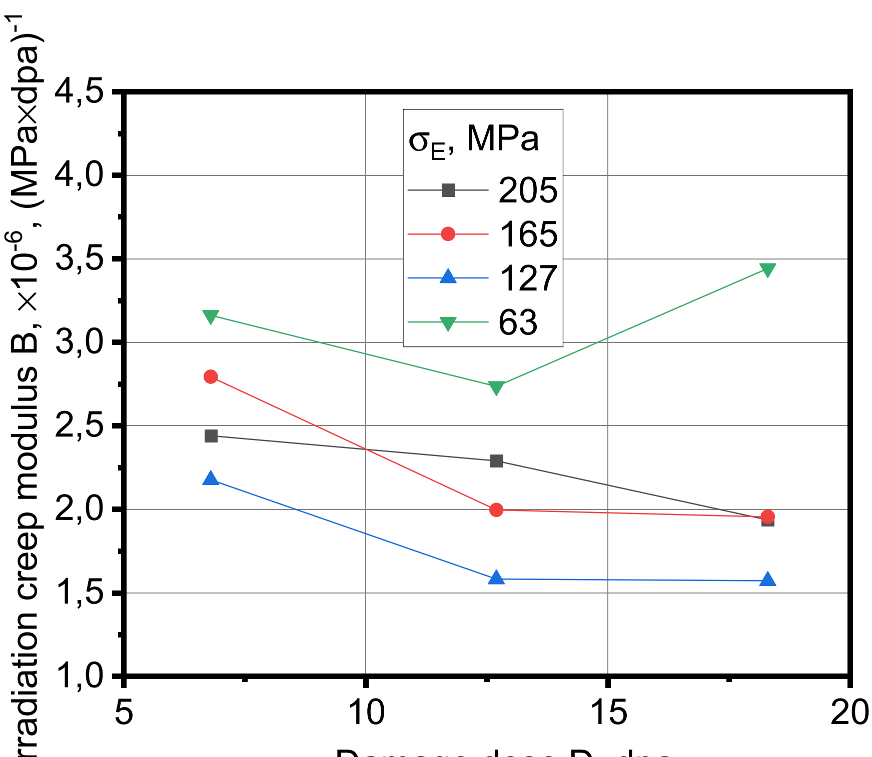
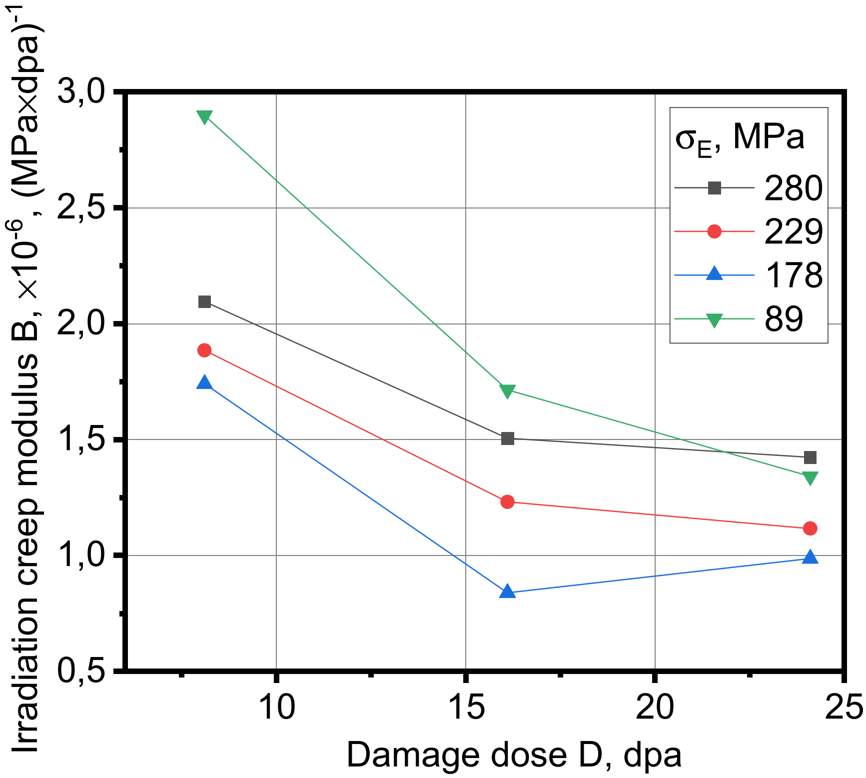


b

Fig. 5. Irradiation creep modulus B of creep specimens EUROFER97–3 after irradiation at T_{irr}=325 °C as a function of damage dose D:. a) EUROFER97–3_1100/700; b) EUROFER97–3_980/780



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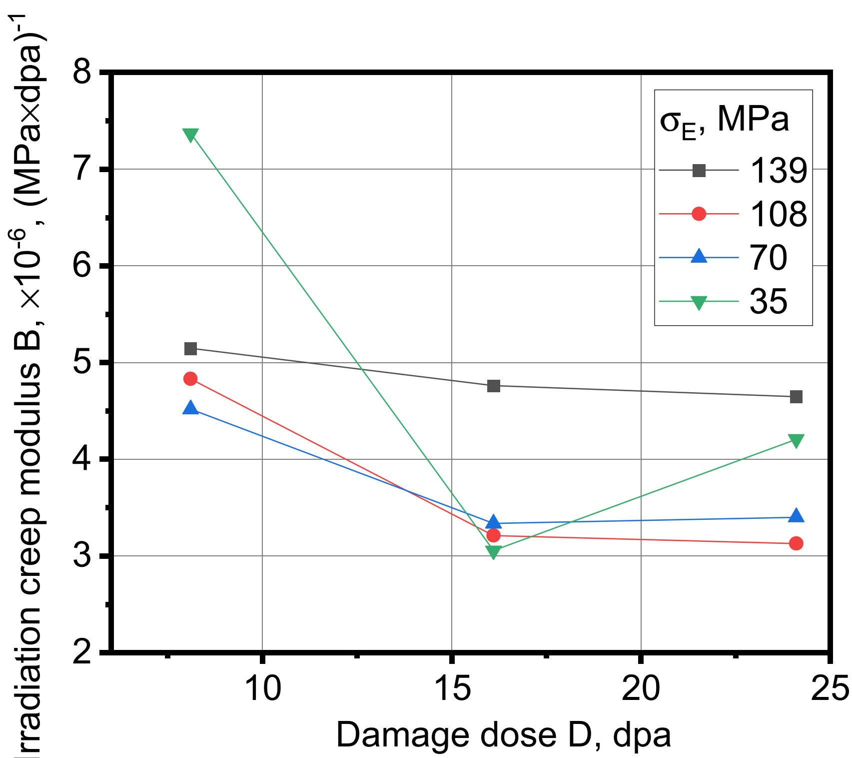

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Fig. 6. Irradiation creep modulus B of creep specimens EUROFER97–3 after irradiation at 550 °C as a function of damage dose D:. a) EUROFER97–3_1100/700;b) EUROFER97–3_980/780

editing, Resources, Project administration, Methodology, Funding acquisition, Conceptualization. **Bronislava Gorr:** Writing – review & editing, Project administration, Funding acquisition, Conceptualization. **Mychailo Toloczko:** Writing – review & editing, Methodology, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Vladimir Chakin reports article publishing charges was provided by Karlsruhe Institute of Technology. Vladimir Chakin reports a relationship with Karlsruhe Institute of Technology that includes: employment. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

No data was used for the research described in the article.

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