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Feasibility of in-vessel Bolts

Literature survey on structure-property stability of irradiation damage resistance alloys and assessment of candidate materials for pre-loaded bolts to be used in irradiated environment.

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1. INTRODUCTION

The design of the nuclear reactor components requests a deep knowledge of degradation mechanisms for the selected materials in the service conditions. Due to the very high energy (~14.06MeV) produced by deuterium-tritium fusion reaction in DEMO reactors, the materials in new fusion reactors experience a higher energy neutron. As the bolts have a structural function as attachment elements of in-vessel components, they are exposed to significant irradiation which may challenge the functionality of the structural materials.

High yield strength, thermal and irradiation creep resistance are required for the bolt materials to sustain high stresses without being plastically deformed, to minimise the size of attached units and to provide reliability of the attachment. In addition the bolts need to maintain its preload setting, with a limited yield loss due to thermal and irradiation creep.

This report presents the results of the literature survey on irradiation damage resistant alloys and their structure-property stability under irradiation. The literature analysis has been focused on the strength and creep resistance behaviour at different temperatures and irradiation damage levels in order to define candidate materials for bolts.

2. OBJECTIVE

The objective of the project was to carry out a literature survey of different materials that could potentially be used for preloaded bolts in the DEMO plasma chamber, highly resistant to irradiation damages in the range:

- 0.2-5 dpa,
- 150-350°C (DEMO IVC-typical temperatures)

The literature analysis aimed to define:

- which material(s) could be used and which ones are not suitable to be used for bolts in irradiated environment. A list of not suitable materials has provided with individual reason for their exclusion;
- for the suitable material(s) a prediction of their strength and creep resistance at different temperatures and irradiation damage levels.

The literature analysis has been focused on the following mechanisms of irradiation damage:

- thermal creep and irradiation creep coupled with swelling
- thermal and irradiation creep induced stress relaxation
- hardening and irradiation effect on yield strength
- microstructural evolution under irradiation

3. SURVEY OF IRRADIATION MECHANISMS AND DAMAGE OF MATERIALS

3.1 Irradiation-resistant materials

A literature review has been carried out to summarize the experiences from JET, ITER and fission power plants [1,2].

Generally bolts are made of austenitic stainless steels (XM-19, TP304, TP316, TP316L, Alloy 660) and Ni based alloys (NIM 80A, IN 625, IN 718). The martensitic steels and ODS (Oxide Dispersion Strengthened) ferritic martensitic steels have been investigated at laboratory level.

In *Table 1* and *Table 2* the chemical compositions for the irradiation–resistant alloys are given [3-6].

This report presents the results of the literature survey on the effect of irradiation on the strength and creep resistance behavior of irradiation resistant materials listed in Table 1 and Table 2.

Materials	Steel grade/ alloy	Chemical composition (mass%)											
		C	Si	Mn	Ni	Cr	Mo	Ti	Al	P	S	Fe	other requirements
austenitic steels	TP304	0.080	1.0	2.0	8.0-11.0	18.0-20.0				0.05	0.030	bal	
	XM-19	0.060	1.0	4.0-6.0	11.5-13.5	20.5-23.5	1.5-3.0			0.05	0.030	bal	0.10-0.30 V, 0.20-0.40 N, 0.10-0.30 Nb
	TP316	0.080	1.0	2.0	10.0-14.0	16.0-18.0	2.0-3.0			0.05	0.030	bal	
	TP316L	0.035	1.0	2.0	10.0-14.0	16.0-18.0	2.0-3.0			0.05	0.030	bal	
	660	0.080	1.0	2.00	24.0-27.0	13.5-16.0	1.0-1.5	1.90-2.35	0.35	0.04	0.030	bal	0.10-0.50 V
Ni based alloys	IN 718	0.045	0.35	0.35	50.0-55.0	17.0-21.0	2.8-3.3	0.80-1.15	0.40-0.60	0.01	0.010	bal	0.23 Cu, 1.0 Co, 0.0060 B
	IN 625	0.10	0.50	0.50	58.0	20.0-23.0	8.0-10.0	0.40	0.40	0.02	0.015	5.0	1.0 Co
	IN 800	0.10			30.0-35.0	19.0-23.0		0.15-0.60	0.15-0.60			bal	0.30-1.20 Al+Ti
	IN 80H	0.05-0.10			30.0-35.0	19.0-23.0		0.15-0.60	0.15-0.60			bal	0.30-1.20 Al+Ti
	IN 80HT	0.06-0.10			30.0-35.0	19.0-23.0		0.25-0.60	0.25-0.60			bal	0.85-1.20 Al+Ti
	NIM 80A	0.100	1.00	1.00	bal.	18.0-21.0		1.8-2.7	1.0-1.8		0.015	3.0	0.008 B, 2.0 Co, 0.15 Zr
	X750	0.080	0.50	1.00	bal.	14.0-17.0		2.25-2.75	0.4-1.0		0.010	5.0-9.0	0.7-1.2 Nb, 050Cu, 1.0 Co
	330	0.15	1.0-2.0	2.0	33.0-37.0	15.0-17.0					0.015	44.0	0.11 N

Table 1. Chemical compositions of irradiation-resistant austenitic steels and Ni based alloys.

Materials	Steel grade	Chemical composition (mass%)										
		C	Si	Mn	Ni	Cr	Mo	Ti	Al	P	S	other requirements
martensitic steels	P91	0.07-0.14	0.20-0.50	0.30-0.60	0.40	8.0-9.5	0.85-1.05	0.01		0.020	0.010	0.06-0.10 Nb, 0.18-0.25 V, 0.03-0.07 N
	F82H	0.090	0.07	0.10	0.001	7.87		0.004			0.001	1.98 W, 0.19 V, 0.030 Ta, 0.0002 B
	Eurofer 97	0.09-0.12	<0.05	0.20-0.60	<0.005	8.5-9.50		<0.01	<0.01	<0.005	0.005	1.0-1.2 W, 0.15-0.25 V, 0.13-0.14 Ta, 0.015-0.045 N, < 0.005 Mo, <0.01 Nb, <0.001 B
	HT9	0.210	0.21	0.50	0.51	11.95	1.03	<0.01	0.03	0.01	0.003	0.52 W, 0.33 V, 0.006 N
	EP 450	0.14	0.20	0.31	0.20	12.95	1.5					0.47 Nb, 0.22 V, 0.02 N, 0.004 B
	PH13-8Mo	0.050	0.1	0.10	7.5-8.5	12.25-13.25	2.0-2.5		0.90-1.35	0.01	0.008	N<0.01
martensitic ODS steels	12Y1	0.045	0.03	0.04	0.24	12.85	0.03	0.003	0.007	<0.001	0.002	0.007 V, 0.01 Cu, 0.004 B, 0.005 Co, <0.01 W, 0.03 Zr, 0.017 N, 0.15 O, 0.20 Y
	12YWT	0.050	0.18	0.60	0.27	12.58	0.02	0.35		0.019	0.005	0.002 V, 0.02 Cu, 0.02 Co, 2.44 W, 0.014 N, 0.16 O, 0.16 Y
	MA 956	0.030	0.050	0.06	0.11	21.17		0.33	5.77	0.008	0.005	0.029 N, 0.03 Co, 0.21 O, 0.38 Y, <0.05 Mo
	MA957	0.030	0.04	0.09	0.13	13.7	0.3	0.98	0.03	0.007	0.006	0.044 N, 0.0009, 0.21 O, 0.28 Y
	PM2000	0.01	0.04	0.11	0.01	18.92	0.01	0.45	5.10	0.002	0.002	0.01 Cu, 0.01 Co, 0.04 W,<0.01 Zr, 0.0028 N, 0.25 O, 0.37 Y

Table 2. Chemical compositions of irradiation-resistant martensitic steels and ODS steels.

3.2 Thermal creep and irradiation creep coupled with swelling

In the reactors materials experience different damage phenomena as thermal and irradion creep and swelling.

Irradiation creep is a time-dependent plastic deformation phenomenon and is mainly attributed to the supersaturation of point defects resulting from displacement damage. By altering the flux of point defects toward sinks, dislocation absorption, nucleation, climb and glide behaviors are affected by applied stresses, leading to a stress-induced microstructural evolution under irradiation. Since both irradiation creep and void swelling involve the redistribution of point defects during microstructural evolution, both the irradiation phenomena are coupled. In addition thermal creep is superimposed with increasing applied stress levels.

Thermal creep is of technical importance only at elevated temperatures, i.e. $T > T_M/2$, where T_M is the melting temperature. On the contrary irradiation creep can be an active mechanism even at low temperatures where most of structural reactor components operate. Since the irradiation creep deformation starts as soon as the material is exposed to the high energy particle irradiation, no incubation time as for swelling is observed [7].

Therefore in the temperature range of interest for this work [7], 150-350°C, under which bolts have to be used, thermal creep strain can be considered negligible in comparison with irradiation creep.

The relative contributions of thermal and irradiation creep to total strain have been measured using irradiation creep tests performed on IN 718 Ni alloy at a temperature of 300°C. Before the irradiation, the specimens were exposed to thermal creep conditions for 15-20 h in order to exhaust strain transients of thermal origin. The specimens were then irradiated with 17 MeV protons at a dose rate of 3.5-4.2 10^{-6} dpa/s. The maximum shear stresses ranged from 150-450 MPa. Stress and temperature values were equal to those imposed during irradiation [21].

In Figure 1 the creep strain is plotted versus time for a specimen subjected to a constant maximum shear stress of 350 MPa, firstly under thermal, thereafter under irradiation conditions (dpa-rate = 3:5 10-6 (dpa/s)) at a temperature of 300°C.

At the end of the thermal creep period, the creep rate was almost zero and increased significantly as soon as the specimens were exposed to irradiation. At the beginning of the irradiation, a small strain transient was observed during which the creep rates decreased before reaching approximately constant values after a dose of 0.01 dpa.

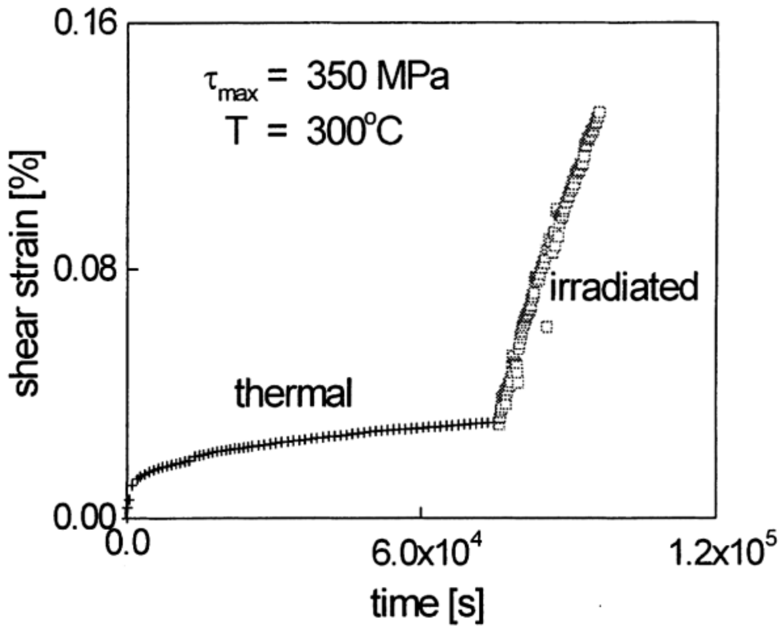

Figure 1. Creep under thermal and irradiation conditions of IN 718 Ni alloy at 300°C for a maximum shear stress of 350 MPa [21].

To assess the relative importance of thermal and irradiation creep, experiments were performed under He-implantation on ferritic martensitic steels for temperatures up to 500°C [29]. Irradiation creep does not show strong temperature dependence. For temperatures up to 500 °C creep and relaxation behavior of components are dominated by irradiation creep.

Irradiation creep behavior can be explained by the stress induced preferential absorption (SIPA) mechanism. Furthermore, at high stress level, irradiation creep behavior tends to change from the SIPA mechanism to a climb-glide mechanism [8-15].The irradiation creep strain rate, can be expressed in terms of stress, σ, as:

$$\dot{\varepsilon} = (B_0 + D\dot{S})d\dot{p}a\sigma^n \qquad (1)$$

where B_0 is the creep compliance coefficient in MPa^{-1} dpa^{-1}, D is the creep-swelling coupling coefficient, $\dot{S}$ is the volumetric swelling rate per dpa, and n is the stress exponent, often around 1 [10].

Irradiation creep behavior of different materials has been assessed using different facilities. For martensitic steels neutron irradiations showed negligible swelling up to doses in the 20 dpa range [16,17].

Irradiation creep-swelling behavior of modified 316 stainless steels, has been assessed using pressurized and open tubes experiment in FFTF/MOTA experiment (temperature 405÷670°C, exposed neutron dose 58÷206 dpa, hoop stress 0÷100 MPa) [18].

The creep and swelling strains as a function of neutron dose are shown in Figure 2.

The fitting curves are also shown and are used for deriving the instantaneous coupling coefficient D.

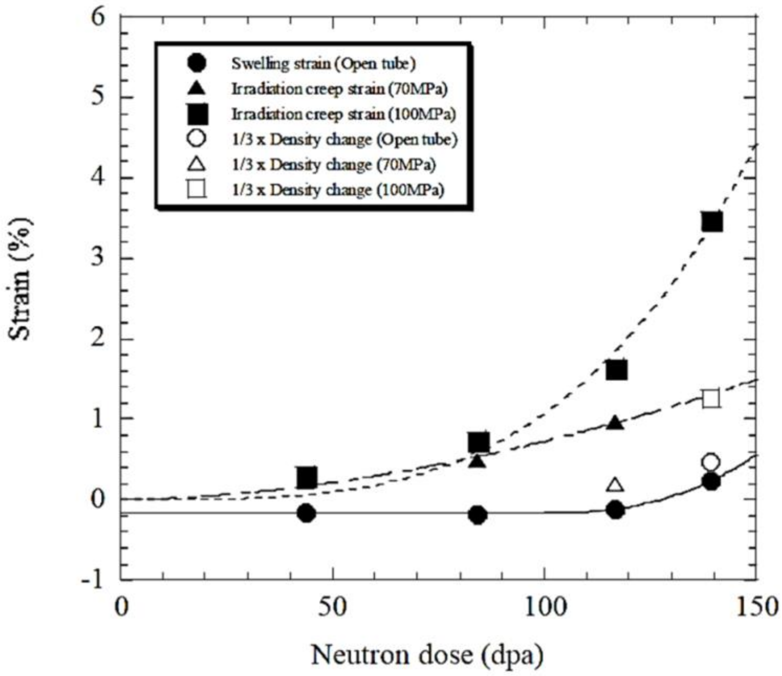

Figure 2. Creep-swelling interaction @ 405 °C in modified 316 stainless steel up to 200 dpa in n FFTF/MOTA [18].

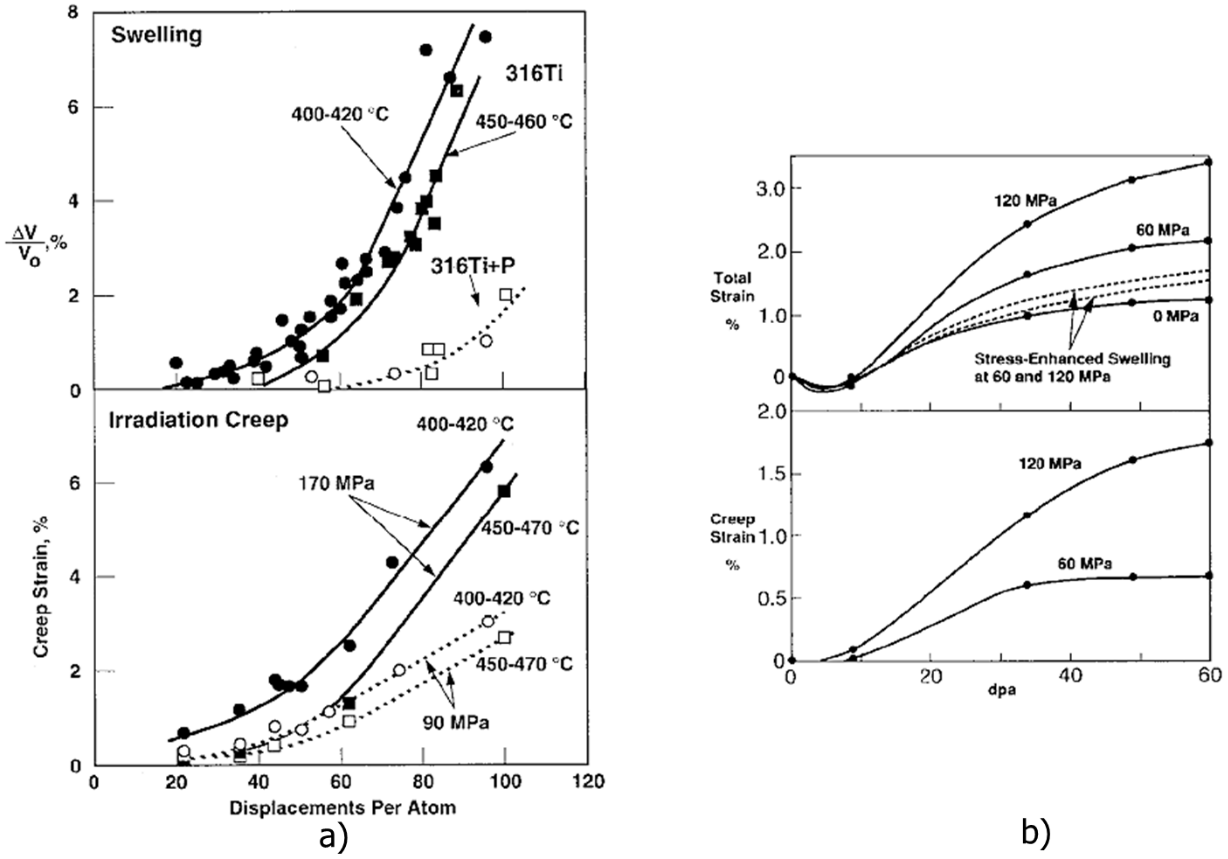

Figure 3. Swelling and creep strains observed in: a) two austenitic stainless steels irradiated as pressurized tubes in PHENIX; b) 330 Ni alloy pressurized tubes irradiated at 420°C [19].

In Figure 3 is shown that the swelling and irradiation creep behavior of 316 stainless steels and 330 nickel-base exhibit a similar dependence from the dose, starting from dose rate greater than 20 dpa [19].

The previous experimental data have shown that for low radiation damage, 0.2-5 dpa, under which bolts have to be used, swelling was determined to be negligible for irradiation – resistant austenitic steels, Ni alloys and martensitic steels (Figure 3).

Equation 1 becomes [24,32]:

$$\dot{\varepsilon} = B_0 d\dot{p}a\sigma \qquad (2)$$

According to equation (2), the irradiation creep rate is proportional to the applied stress and dose rate, depending on the material irradiation creep compliance coefficient B_0. The lower is irradiation creep compliance B_0 of material, the higher is its structure-property stability under irradiation.

The irradiation creep compliance coefficient of stainless steel, IN 718 nickel base alloy, ferritic/martensitic and ferritic/martensitic/ODS steels has been assessed using different facilities.

For austenitic stainless steels, there is a large irradiation creep data base which indicates that, in the absence of swelling, two separate creep regimes exist, described as transient (primary) creep and steady state (secondary) creep. Primary irradiation creep is characterized by an initially high creep rate that decreases with time under irradiation. The creep rate, after a dose of about 0.5-1 dpa, approaches a steady state value that may be much smaller than the initial value. Therefore, the magnitude of irradiation creep compliance under dose level lower than 1 dpa for the transient regime is higher than that of the steady state regime [20, 25].

Steady-state irradiation creep compliance B_0 under operating temperature 300÷500 °C alloy is order of ~10^{-6} $MPa^{-1}dpa^{-1}$ as shown in *Figure 4* [18]. Similar values of creep constant B_0 for Inconel 718 have been determined in irradiation creep tests at 300°C [22].

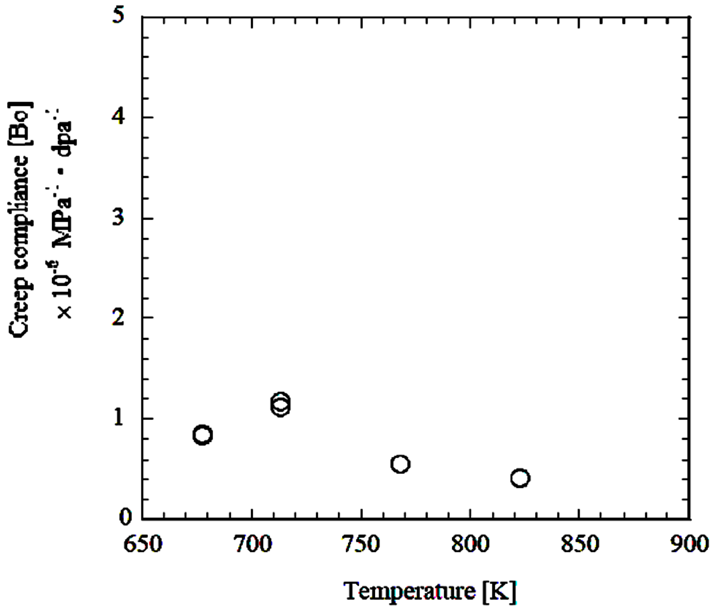

Figure 4. The creep compliance B_0 in modified 316 stainless steel under damage 58-200 dpa as a function of irradiation temperature derived from FFTF/MOTA irradiation [18].

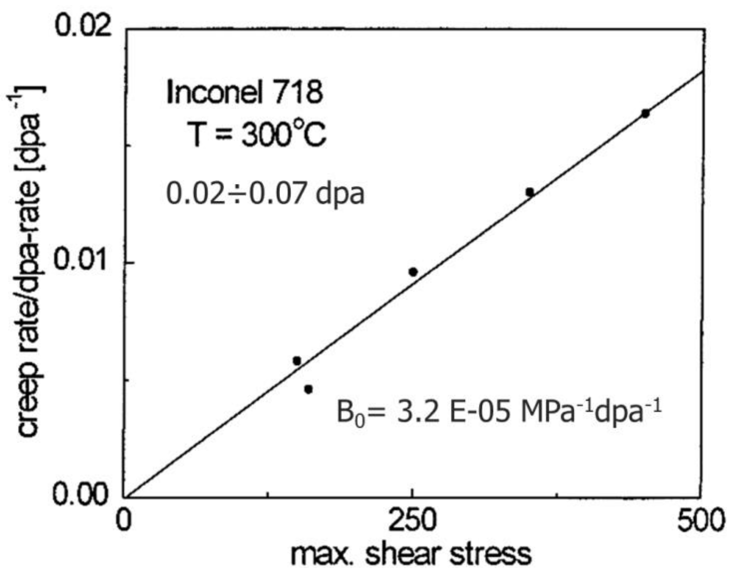

Figure 5. Stress dependence of the irradiation creep rate for Ni alloy IN 718 under irradiation damage 0.02÷0.07 dpa and operating temperature 300°C [21].

Irradiation creep compliance, B_0, in the transient regime has been studied for IN718 Ni alloy. In *Figure 5* the quantity creep rate divided by dpa rate is plotted versus the applied maximum shear stress, where the creep rates correspond to the mean slope of the irradiation creep curves in the dose range 0.02-0.07 dpa at the operating temperature of 300°C [21]. Figure 5 shows that the creep rate is a linear function of stress in the stress range of 150-450 MPa with an irradiation creep compliance B_0= 3.2 E-05 Mpa^{-1} dpa^{-1}. Irradiation creep compliance B_0 of both austenitic stainless steels and Ni base alloys for dose rate less than 1 dpa, transient irradiation creep, is in order of 10^{-5} MPa^{-1} dpa^{-1} [20,21].

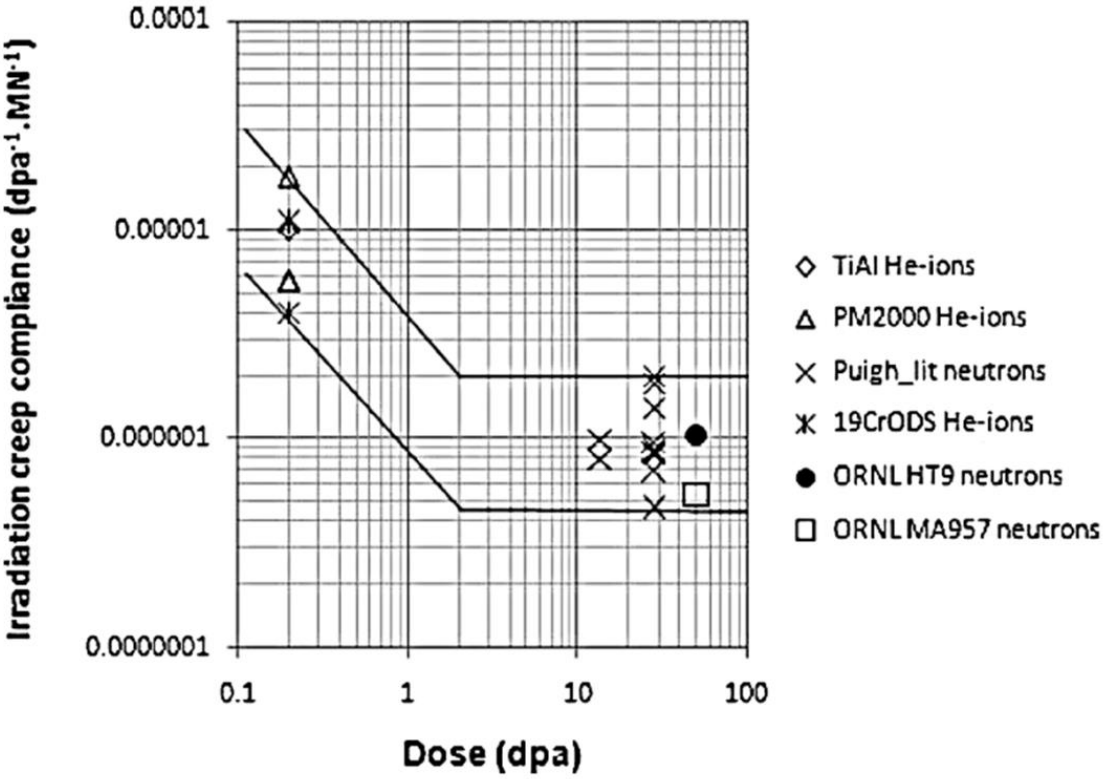

Figure 6. Irradiation creep compliance for ferritic martensitic steels in a temperature range from 300 to 600 °C. The two lines represent the scatterband of the data. The inflection points at 2 dpa were chosen based on a similar plot for austenitic steels [24].

Determination of irradiation creep compliances, B_0, from published results on neutron irradiated pressurized tubes of different ferritic and ferritic–martensitic steels [29, 30] revealed the values measured for a total dose of 0.2 dpa were about ten times higher than results of creep experiments under neutron irradiation with total dose higher than 10 dpa. This indicates a sharp drop of B_0 at total dose between 0.1 and 2 dpa as for austenitic stainless steels.

B_0 is plotted in *Figure 6*, as a function of irradiation damage doses for different ferritic and ferritic-martensitic steels and ODS steels (average dispersoid diameters 25 and 2.2 nm) [24]. Assuming a transient stage up to 2 dpa, also quite similar values between conventional ferritic/martensitic steels and ODS steels can be evaluated. These results seem to demonstrate that the presence of dispersoids does not have a significant influence on the irradiation creep behavior. Steady-state irradiation creep compliance, B_0, is about 0.5 x 10^{-6} $MPa^{-1}dpa^{-1}$. This relatively low creep compliance is consistent with the fact that the irradiation creep rate of martensitic alloys is significantly lower than austenitic stainless steels [15].

Irradiation creep compliance, B_0, assessed for different irradiation resistance materials under different range of operating temperature and irradiation damage, are summarized in Table 3 and Table 4.

Materials	Steel grade/alloy	Irradiation: operating temperature and damage		Irradiation creep compliance
		T °C	dose dpa	B_0 (10^{-6} $MPa^{-1}dpa^{-1}$)
austenitic steels	316	300 430 400 405 440 495 550	0.02-0.3 <0.2 0.1-0.23 182 79 58 117	11 7 21.7 0.9 1.2 0.6 0.4
Ni based alloys	IN 718	300	0.02-0.07 0.1-1.0	32 1.2

Table 3. Irradiation creep compliance of irradiation-resistance austenitic steels and Ni based alloys [18,21,22, 25].

Materials	Steel grade/alloy	Irradiation: operating temperature and damage		Irradiation creep compliance
		T °C	dose dpa	B_0 (10^{-6} $MPa^{-1}dpa^{-1}$)
martensitic steels	P91	500 500 500	0.8 1.2 1.5	3.1 4.8 3.3
	F82H	300 500	5 5	2 8
	HT9	300-400 400 330 540-565	50 50-165 19 10-20	1.0 0.95-1.9 0.5 0.3
	EP 450	310 320 330 390 400 410 480 500	61 81 20 60 45 20 60 45	0.75-1.1 0.2-0.5 0.5-0.8 0.75-1.2 0.2-0.7 0.6-0.75 0.25-0.5 0.3-0.75
	PH13-8Mo	288-310	1-4	0.45*
ODS steels	MA957	300-400 400	50 50-165	0.5 0.25-0.60
	PM2000	300 400 500	up to 0.2	5.7 5.7 18

Table 4. Irradiation creep compliance of irradiation-resistance martensitic steels [15,16, 24,28, 39,30].
** Assessed value using data of Figure 9.*

3.3 Thermal and irradiation creep induced stress relaxation

A high level of pre-loading is required for the bolts in mechanical joints and the initial pre-load should be maintained possibly throughout the operating time. The bolts may sustain high stresses without being plastically deformed.

A contribution of thermal creep to the pre-load relaxation can be excluded, since thermal creep is negligible at temperature lower than 450°C for the bolts material. The extent of thermal expansion, which may relax the initial pre-load, can be also considered negligible in comparison to irradiation creep for operating temperature 150÷300 °C [21,22].

It is known that creep is a temperature–time driven phenomenon. However, the material susceptibility to creep can be influenced by irradiation. The irradiation creep phenomenon is the permanent deformation under constant loading. The total strain ε under creep conditions is composed of two components, an elastic component ε_e, which is recoverable, and the creep component ε_c, which is irrecoverable. If the total strain is kept constant, as in the case of bolts, the stress, σ, can change only if elastic strain is converted into plastic strain. So, $d\sigma=-Ed\varepsilon_c$, where $d\sigma$ is the stress relaxation which corresponds to the creep strain $d\varepsilon_c$ and E Young Modulus. According to equation (2), the creep rate is proportional to the applied stress and dpa-rate, with irradiation creep compliance B_0. Therefore, it follows:

$$-d\sigma = EB_0 d\dot{p}a\sigma \qquad (3)$$

such that the stress relaxation as a function of irradiation dose can be written as [22]:

$$\sigma = \sigma_0 \exp\left(-EB_0 dpa_T\right) \qquad (4)$$

where dpa_T corresponds to the total dose.

1.1.1 Irradiation creep induced stress relaxation

Irradiation creep is a powerful stress relaxation mechanism, which affects the stress strain condition in the bolts and the surrounding materials. Although much information is available for the measurements of the steady-state irradiation creep compliance, B_0, only few examinations have been performed on irradiation creep stress-relaxation. These data are very limited and restrained to dose damage less than 1.0 dpa or in the field of operating temperature in the range 300÷500°C.

Stress relaxation curves have been assessed for limited type of materials on the base of the irradiation creep curves. The irradiation creep curves can be converted into relaxation curves, analytically, since there is a linear relationship between stress and irradiation creep rate.

In order to estimate the magnitude of irradiation induced stress relaxation, proton irradiation creep tests have been carried out on IN 718 Ni alloy at a temperature of 300°C for stresses which ranged from 27 up to 83% of the minimum yield stress at the test temperature [22]. According equation (4) two stress relaxation curves are plotted in Figure 7. Two values for the irradiation creep constant are assumed: k_1=1.2 10^{-11} (dpa^{-1} Pa^{-1}), as derived from the experimental data, and k_2=0.410^{-11} (dpa^{-1} Pa^{-1}) taken as a lower limit assuming that the creep rate may drop when the dose is increased. A stress relaxation of 10% may be reached after a dose of 0.1 dpa.

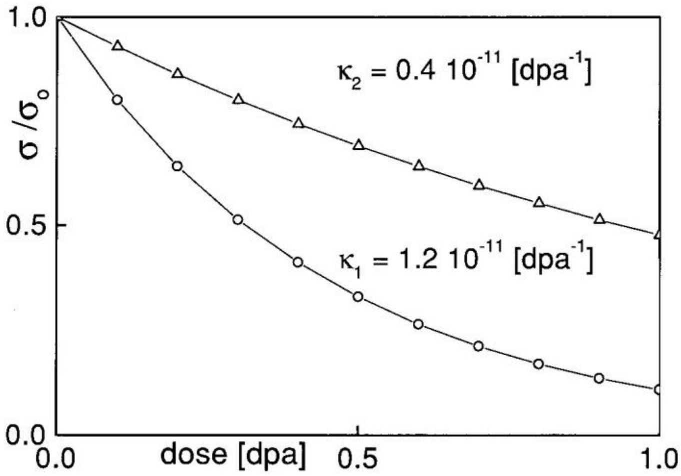

Figure 7. Stress relaxation of IN718 Ni alloy @ 300°C calculated by assuming two irradiation creep constants k_1 and k_2. k_1 is derived from the present data. k_2 is taken as a lower limit [22].

Figure 8 shows the measured stress relaxation, normalized to the initial applied stress, for neutron irradiated Inconel X750 springs [26,27]. Nearly complete relaxation of the initially applied stress on the springs occurred after an irradiation dose of ~20 dpa at ~400 °C.

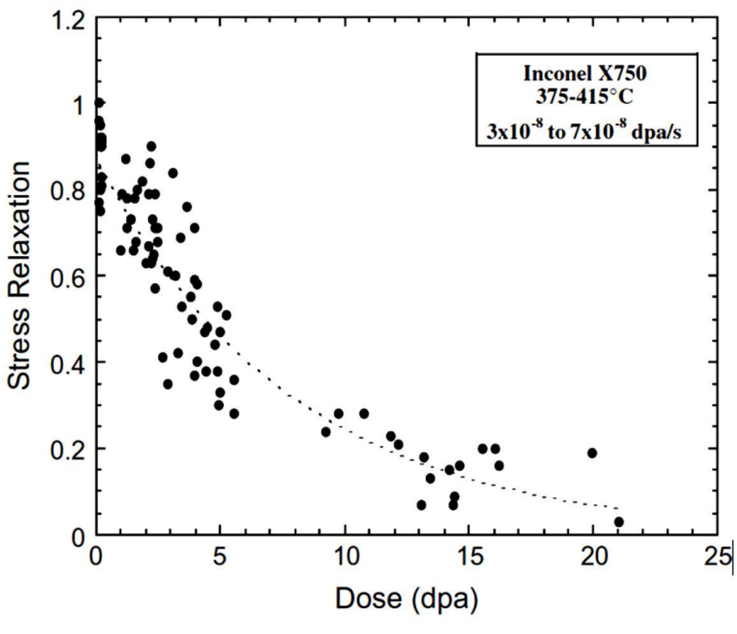

Figure 8. Stress relaxation for Inconel X750 springs irradiated in the EBR-II fast fission reactor [27].

Figure 9 shows the stress relaxation results for the strained bolts and the bent strips of Alloy 625+, PH13-8Mo and Eurofer97 together with the power law fits to the experimental results [28].

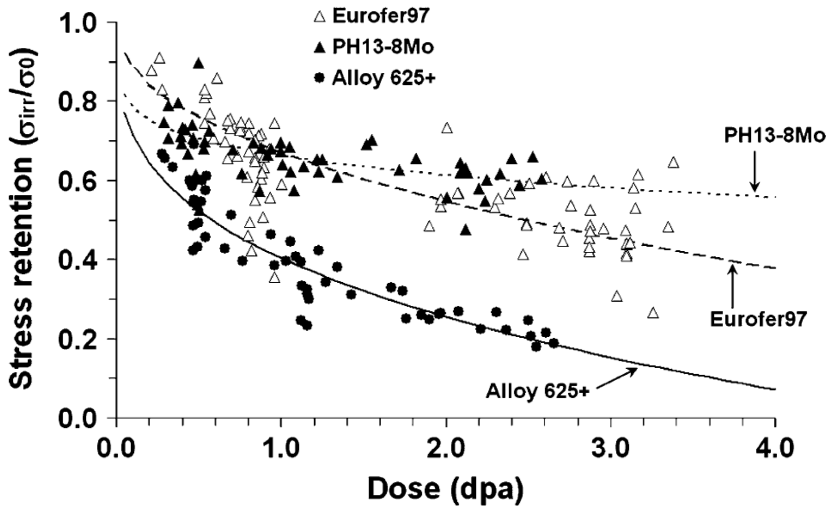

Figure 9. Stress retention (σ/σ_0) as a function of irradiation dose (dpa) for different materials (Eurofer97, Alloy 625+ and PH13-8Mo). Lines are the least-square power law fitting curves [28].

It can be seen that the stress relaxation of Alloy 625+ under irradiation is very large. After an irradiation dose of 2.7 dpa only 20% of the original pre-stress is retained. In spite of the large scatter in the results, the stress relaxation behavior of PH13-8Mo and Eurofer97 is better than that of alloy 625+. However, faster stress relaxation occurs in Eurofer97 at the later stage compared to the PH13-8Mo alloy; care should be taken in the analyses of the results as the scatter for Eurofer97 is large and no data for PH13-8Mo above 2.5 dpa are available. After an irradiation dose of 2.7 dpa 42–47% of the original pre-stress is retained in Eurofer97.

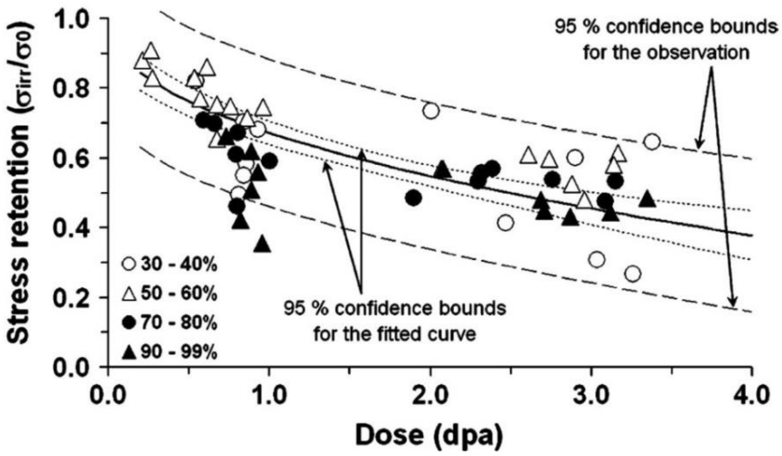

Figure 10. Stress retention (σ/σ_0) as a function of irradiation dose (dpa) for bolt specimens of Eurofer 97 martensitic steel. Different pre-stress levels are shown using different symbols: open circles 30–40%, open triangles 50–60%, solid circles 70–80% and solid triangle 90–99% of the yield strength at 300°C. The solid black line is a least-square power law fit to the experimental results [28].

In order to study the effect of the pre-stress level on the stress relaxation behavior, different pre-stress levels from 30% to 90% of the yield strength at 300 °C were applied on Eurofer 97 [28]. Figure 10 shows the dependence of the stress retention (σ/σ_0) on the pre-stress level. As expected no significant influence of the pre-stressed level is observed on the stress relaxation [28].

In-situ irradiation creep under He-implantation for dose damage level up to 0.2 dpa, was performed on several ferritic steels, for a temperature range 300÷500°C [29]. Relaxation curves for thermal and irradiation creep, calculated for a temperature of 500 °C, are shown in Figure 11.

The curves have been calculated using experimental irradiation creep compliance B_0 measured at a stress level of 150 MPa for a relevant time period of 60 years. The dpa-levels accumulated over the whole period cover the range 0.1–10 dpa. It can be seen that even for 1 dpa, irradiation creep becomes the only important mechanism under the present irradiation conditions.

For temperatures up to 500° C creep and relaxation behavior of components is dominated by irradiation creep. The comparison to P91 data shows that no difference between PM2000 and T91 exists for 10 dpa.

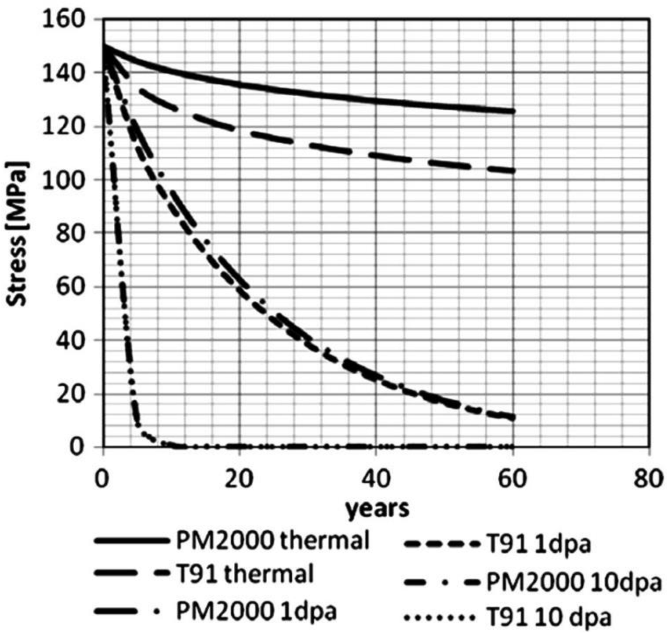

Figure 11. Calculated stress relaxation of 150 MPa over 60 years at 500 °C for martensitic steels [29].

Stress relaxation normalized to the initial applied stress assessed for several irradiation resistant materials, under different range of operating temperature and irradiation damage, are summarized in Table 5.

Materials	Steel grade/alloy	Irradiation		irradiation induced relaxation	
		T °C	dose dpa	stress relaxation σ/σ_0	pre-stress loss %
Ni based alloys	IN 718	315	1.0	0.42	58
	X750	375-415	1.0 4.0 21.0	0.80 0.40 0.05	20 60 95
	IN 625	288-310	1.0 4.0	0.40 0.075	60 92.5
martensitic steels	P91	500	1.0 10.0	0.68 0.13	32 87
	Eurofer 97	288-310	1.0 4.0	0.70 0.40	30 60
	PH 13- 8Mo	288-310	1.0 4.0	0.70 0.55	30 45
martensitic ODS steels	PM2000	500	1.0 10.0	0.66 0.13	34 87

Table 5. Stress relaxation of irradiation-resistant materials [18, 21, 22, 25].

3.4 Irradiation hardening effect on tensile properties and fracture toughness

Irradiation hardening at low and intermediate temperatures is due the production of high densities of nanoscale defect clusters and dislocation loops, which serve as obstacles to dislocation motion. The production of nanoscale defect clusters and dislocation loops results from the displacement damage. Therefore the degree of hardening is expected to increase with irradiation dose even if it can saturate when defect overlapping starts to occur [15,27].

This hardening is generally associated to a reduction of tensile elongation and fracture toughness. The radiation hardening and the elongation and fracture toughness reduction, typically emerge at damage levels above 0.1 dpa. They are generally more pronounced for homologous irradiation temperatures below $0.35T_M$, where TM is the absolute melting temperature [27].

Figure 12 shows an example of the effect of moderate neutron damage on the engineering stress–strain curve for an austenitic stainless steel and a 8– 9% Cr-tempered martensitic steel at 250 °C.

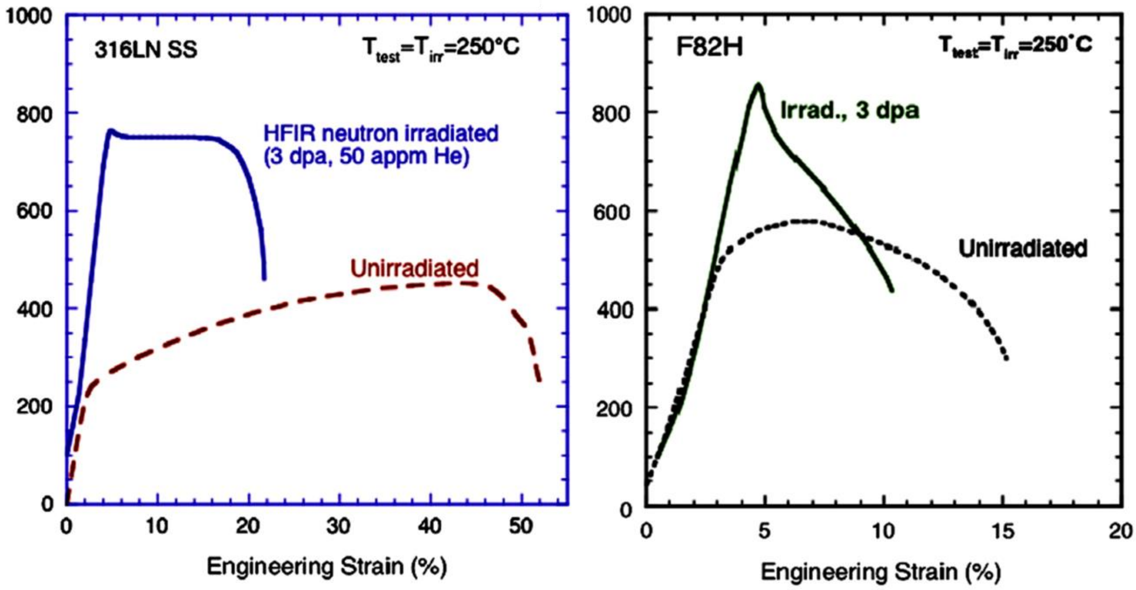

Figure 12. Effect of 3 dpa neutron irradiation on the engineering stress–strain curves for solution annealed 316LN austenitic steel and F82H-tempered martensitic steel at 250 °C ([27].

Both materials exhibit significant radiation-induced increase of yield and ultimate tensile stress, large reductions of elongation (particularly uniform elongation), associated to a decrease of strain hardening. In addition to the decreased elongation, neutron irradiation at low temperature also generally produces a decrease in fracture toughness.

Figure 13 summarizes some of the fracture toughness data for AISI 304 and 316 austenitic stainless steels after irradiation at LWR-relevant conditions in the range 250–350 °C [27]. The fracture toughness decreases rapidly with increasing irradiation dose, and approaches a value near 50 MPa $m^{1/2}$ after 5–10 dpa.

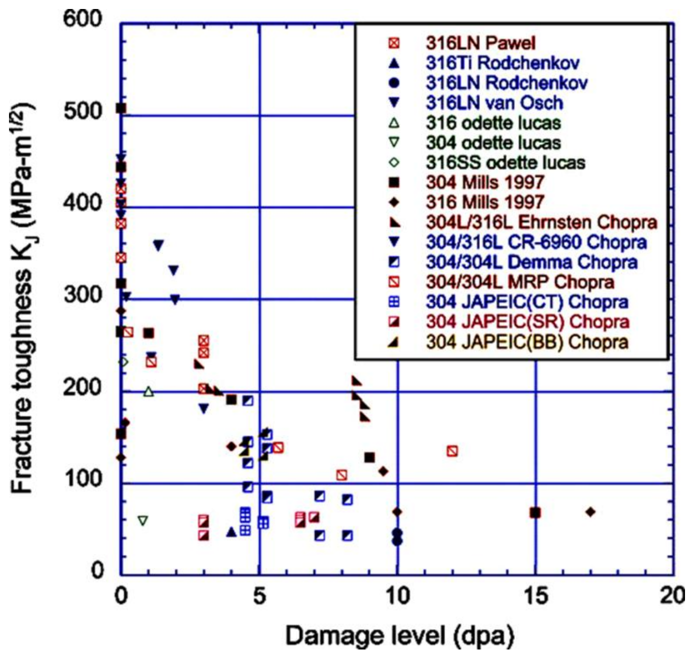

Figure 13. Fracture toughness of Types 304 and 316 austenitic stainless steels at 250–350°C [27].

Low-temperature irradiation - induced embrittlement is recognized to be one of the key issues for ferritic/martensitic steels. It has been shown that the ductile-brittle transition temperature (DBTT) increases rapidly with irradiation at temperatures below 0.3 T_M [15]. DBTT shift in RAFM steels often becomes quite large [15,31].

Some tensile test results of HT-9 obtained from temperature irradiations range of 90-400°C are plotted as a function of dose in Figure 14 . Despite a large scatter, the dataset clearly shows that yield strength increases sharply at low doses but saturates around 10 dpa.

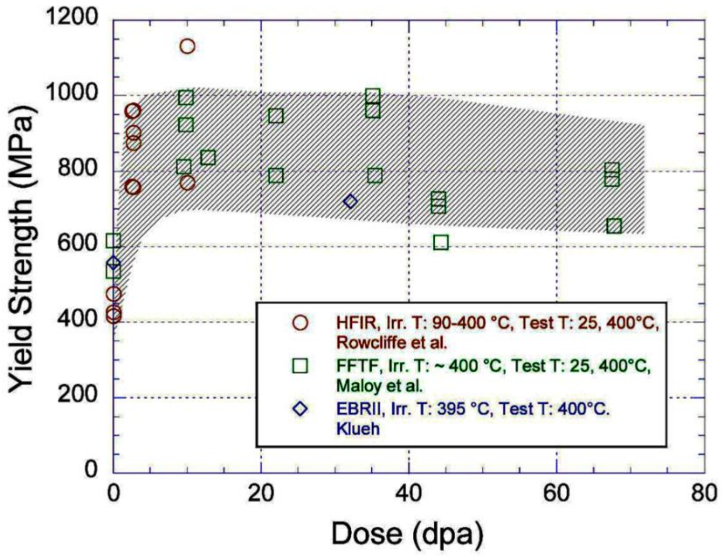

Figure 14. Dose Dependence of Yield Strength for HT-9 Irradiated at HFIR and FFTF below 400°C [15].

The effects of irradiation hardening on the tensile properties depend on irradiation temperature as shown in Figure 15. At low-temperature irradiations, yield strength becomes almost double of the unirradiated value. As the irradiation temperature increases, above 400°C, softening can occur and promotes a decrease of yield strength.

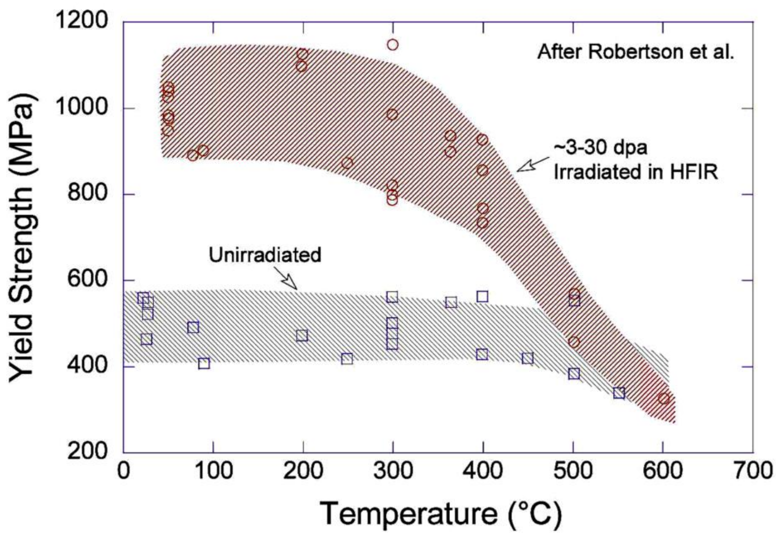

Figure 15. Temperature Dependence of Yield Strength of HT-9 irradiated to ~3-30 dpa [15].

Dose dependence of the tensile test results for F82H irradiated at 200–500 °C is shown in Figure 16 [31]. Below 400 °C yield stress increases linearly with the logarithm of the displacement damage levels up for values up to 10 dpa. Elongations decreased with dose; at temperatures of 400 and 500 °C, the change caused by irradiation was rather small.

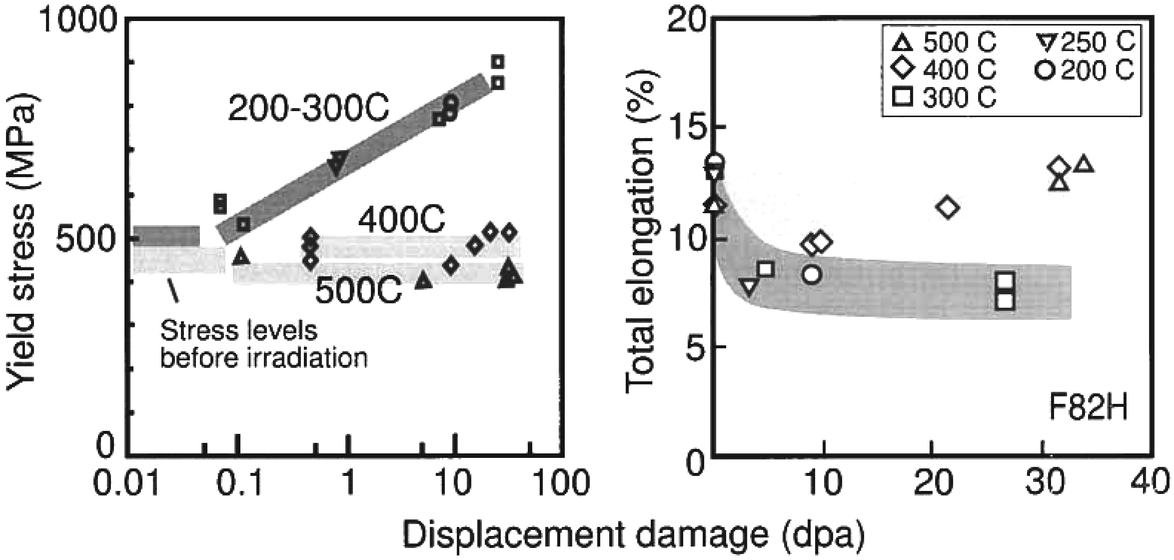

Figure 16. Dose dependence of (a) yield stress and (b) total elongation of IEA-F82H irradiated in HFIR to about 30 dpa [31]

The DBTT for several ferritic/ martensitic steels after irradiation at 300-400°C is plotted in Figure 17, as functions of dose [33]: the DBTT shift tends to saturate with dose. RAF/Ms exhibited smaller shift compared to the results of non-reduced activation alloy (MANET I/ II).

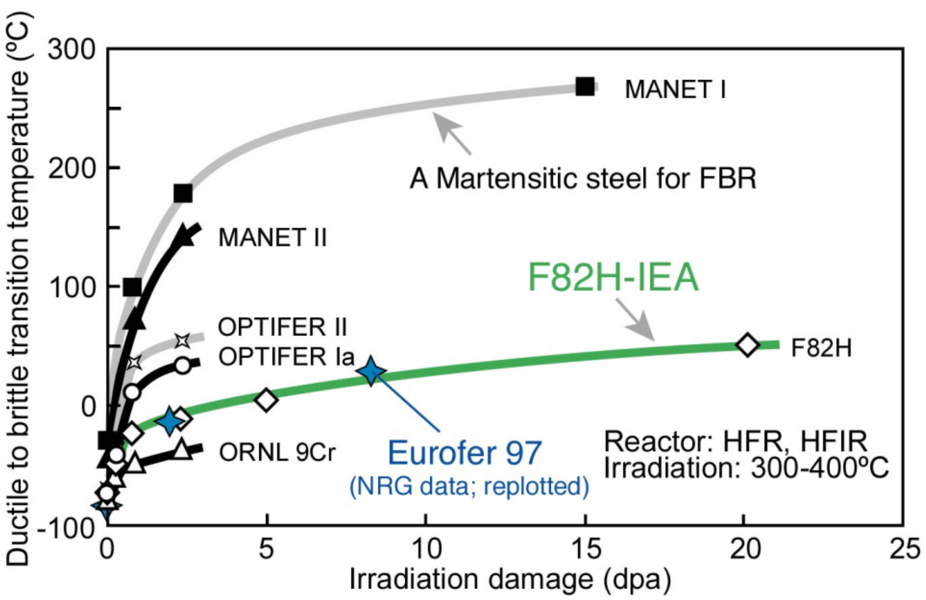

Figure 17. Damage level dependence of DBTT-shift of several martensitic steels (Optifer, F82H and ORNL 9Cr) [33].

Tensile properties of IN 718 Ni alloy change slightly under neutron irradiation up to 0.5 dpa: the limited increase of strength and the light ductility decrease will not impact to the component structural integrity and lifetime [22].

3.5 Microstructural evolution under irradiation

Microstructural evolution under irradiation at intermediate temperature is mainly due to solute segregation and associated precipitation kinetics.

Radiation-induced segregation of alloy elements at grain boundaries in austenitic alloys is well explained by the inverse Kirkendall mechanism [34]. Grain boundary Cr depletion and Ni enrichment are widely observed in austenitic Fe-Cr-Ni systems after neutron irradiation.

Figure 18 summarizes the several phases that can be formed in a single-phase austenitic stainless steel due to the localized radiation-induced solute segregation processes during neutron irradiation [27, 35]. Partially shaded data points at temperatures <400 °C denote the presence of γ' phase. Solid data points are for G and related phases and for an unidentified phase. Initial investigations indicated that radiation-induced precipitation was limited to temperatures above 400 °C, but recent long-term experiments have observed radiation-induced precipitation in austenitic stainless steel for temperatures as low as 300 °C [35].

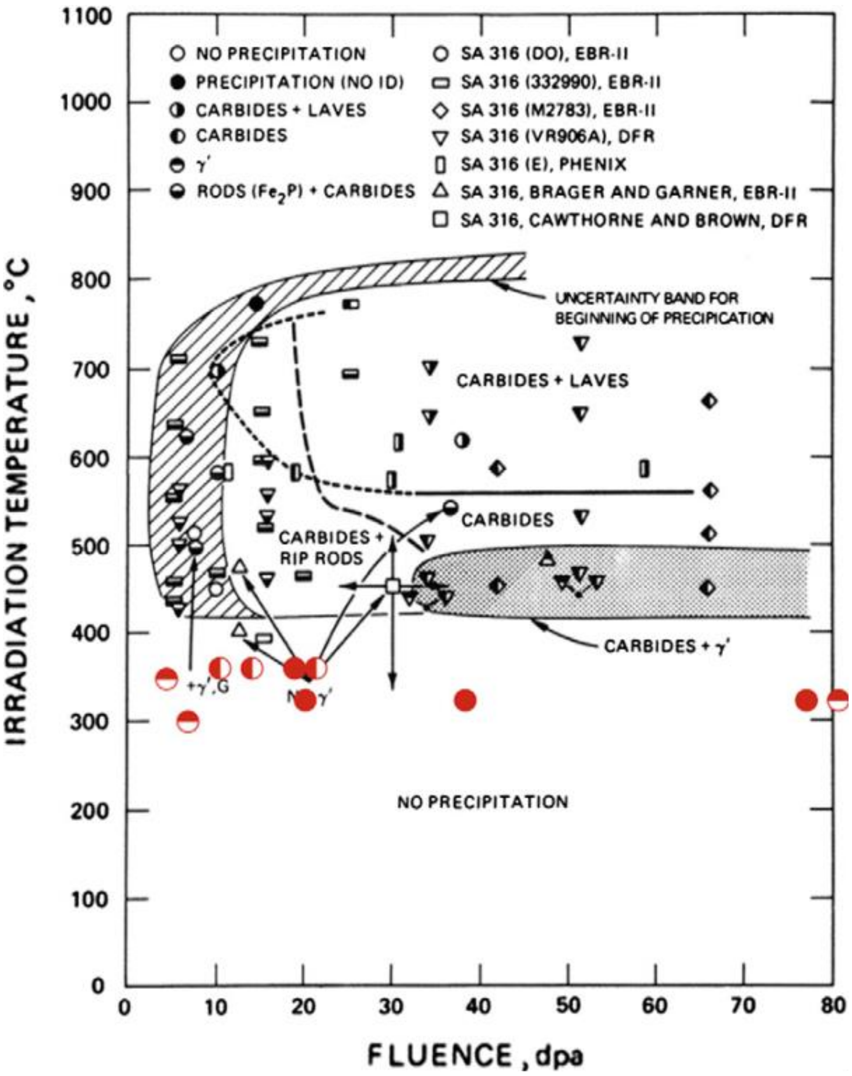

Figure 18. Precipitate phases observed in Type 316 austenitic stainless steel after neutron irradiation [27, 35]

Radiation-induced segregations play a significant role in the formation and stability of irradiation-induced phases in martensitic steels [36].

The most stable carbide in unirradiated high-Cr steels is $M_{23}C_6$ which usually forms at prior austenite grain and martensite lath boundaries. Small amount of MC, M_2X and η-carbide (M_6C) can also be found in some high-Cr steels. Laves and chi (χ) phases may develop by prolonged thermal aging at elevated temperatures.

After irradiation, α′, G-phases, η and χ phases have been reported in HT- 9 martensitic steel in a low-dose (~7 dpa) HFIR irradiation at 400 °C. Cr-rich α′ and η were identified along with the dislocation loops in HT-9. The authors suggested that dislocation loops provided sites of Cr segregation and therefore played an important role in the precipitation development in martensitic steels. It is believed that the formation of α′ is due to radiation-enhanced spinoidal decomposition of Fe-Cr alloys and η precipitation is attributed to radiation induced-segregation of Ni and Si.

In another HFIR irradiation, it was found that the original precipitate structure in HT-9 was considerably coarsened at 500°C, and the $M_{23}C_6$ was replaced by irradiated-produced η phase. Fine MC precipitates evolved and coarsened during irradiation at 300 and 500°C. Occasionally, fine G-phase particles were also reported.

Precipitate microstructure in HT-9 was also studied in a Phenix irradiation experiment up to 110 dpa below 530°C. Apparently, the original $M_{23}C_6$ and MC were not affected by irradiation in this study. Again, new fine precipitates (η-carbide) were induced by the irradiation. These precipitates were uniformly distributed at low temperature (420°C) and narrow depleted zones could be seen along lath and grain boundaries. At 460°C, the precipitates coarsened and depleted zones widen and larger particles were produced along dislocation lines [15].

Table 6 summarizes the numerous radiation-induced phases that can be induced in HT9 martensitic steel.

Irradiation Facility	Precipitate	Temperature (°C)	Dose (dpa)
FFTF	α’, χ	420	35
	η	407	47
	α’, G	380–440	20–155
Phenix	α’	400–530	30–116
	η	419	79
EBR-II	α’, χ, G	400, 425	25–60
HFIR	α’, η	400	7.4
	G	300, 400, 500	10–12, 38
14 MeV Ni	α’, χ	300–600	200

Table 6. Main Radiation-induced Precipitates in HT-9 martensitic steel [15].

4. ASSESSMENT OF CANDIDATE MATERIALS FOR PRE-LOADED BOLTS TO BE USED IN IRRADIATED ENVIROMENT

4.1 Materials Ranking

On the basis of degradation and damage mechanisms in irradiated environment described in the literature survey, an analysis of property-structure stability of different materials has been performed, in order to define suitable materials to be used for bolts at the target temperature and dose:

- 150-350°C (DEMO IVC-typical temperatures),
- 0.2-5 dpa.

The most critical requirement for bolts is the maintenance of their pre-load setting to assure a limited loss due to thermal and irradiation creep. A high level of pre-load is required for the bolts in mechanical joints and the initial pre-load should be maintained possibly throughout the operating time.

A contribution of thermal creep to the pre-load relaxation can be excluded, since thermal creep is negligible at temperature lower than 450°C for the bolts material. The extent of thermal expansion, which may relax the initial pre-load, can be also considered negligible in comparison to irradiation creep, that represents the most important relaxation mechanism for operating conditions.

Stress decreases exponentially as a function of irradiation dose with irradiation creep compliance B_0. The higher is the irradiation creep compliance the higher is the amount of initial stress decrease.

A proposal for a ranking of bolt materials in irradiated environment is based on the combined information of stress relaxation and irradiation creep compliance data.

Irradiation-creep compliance

A comparison of irradiation creep compliance B_0 values for different irradiation resistance materials has been plotted in Figure 19 on the base of data reported in the Table 3 and Table 4.

The higher is the irradiation creep compliance the lower is the irradiation creep resistance of the material. Materials with the lowest irradiation creep compliance, highlighted in the Figure 19 by red stars, can be considered suitable for pre-loaded bolts in irradiated environments. For this materials B_0 is lower than 10^{-6}MPa^{-1}dpa^{-1}: this value can be assumed as target to assure a high irradiation creep resistance.

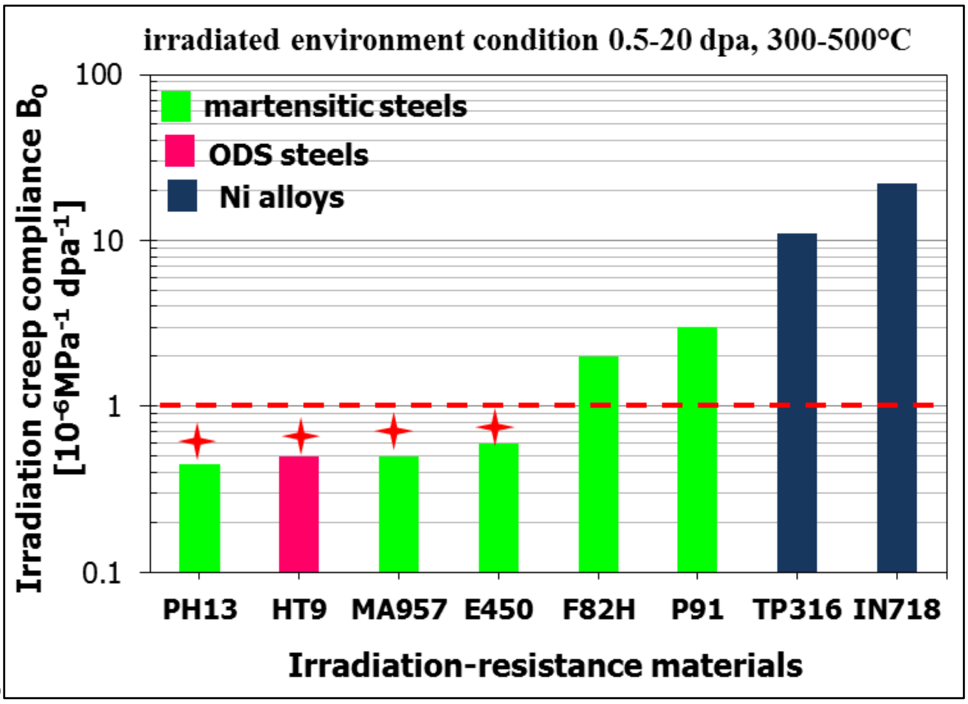

Figure 19. Comparison of B_0 coefficients for different irradiation resistant materials at 0.5-20 dpa, 300-500°C.

Irradiation-induced stress relaxation

A comparison of pre-stress loss for different irradiation resistance materials has been plotted in Figure 20 on the basis of data reported in the Table 5.

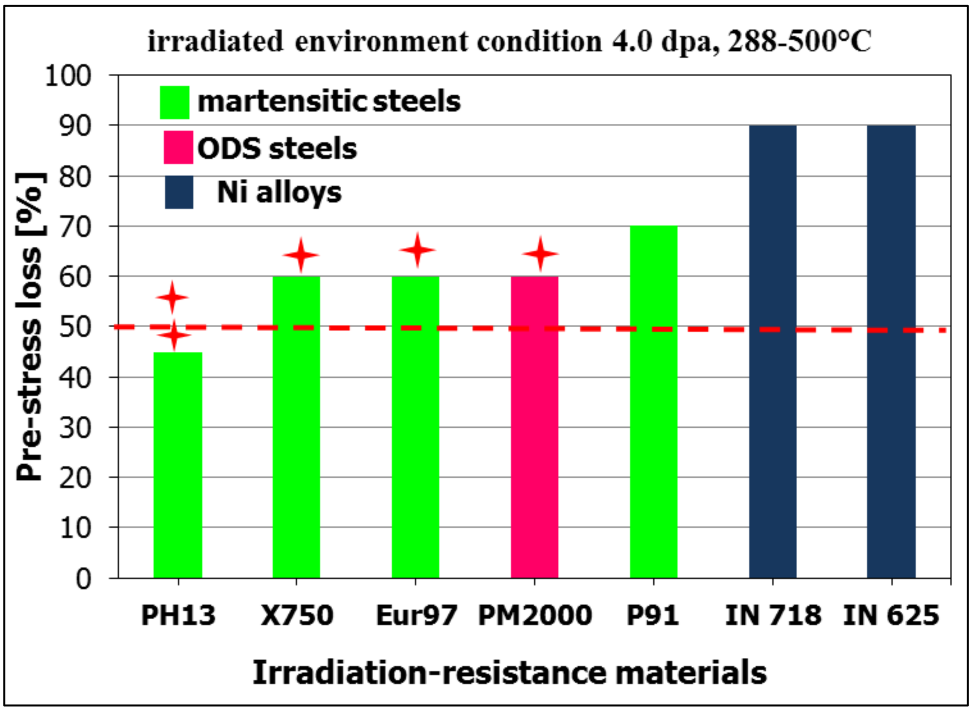

Figure 20. Comparison of pre-stress loss of different irradiation resistant materials at 4.0 dpa, 288-500°C.

The lower is the press-stress loss the better is the maintenance of the initial pre-load. Materials with the lowest pre-stress loss, highlighted in the Figure 20 by red stars, can be considered suitable for pre-loaded bolts in irradiated environments. For this materials the pre-stress loss is lower than 50%: this value can be assumed as target to assure a limited pre-load loss.

PH13-8 Mo martensitic steel, showed as double 4 point red star in the Figure 20, is the most promising material to be used for bolts in irradiated environment. PH13-8 Mo is a martensitic precipitation age-hardening stainless steel with nominal composition 12.25-13.25 Cr, 7.5-8.5 Ni, 2.0-2.5 Mo, 0.9-1.35 Al (Table 2).

Irradiation-embrittlement

Irradiation resistance materials have been classified as:

- limited irradiation embrittlement materials: nickel based alloys and austenitic steel
- high irradiation embrittlement materials: martensitic steels ODS martensitic steels

Irradiation induced-embrittlement increases yield and ultimate tensile strength, reduces elongation and fracture toughness, whose effect on the performance of pre-loaded bolts in irradiated environments is not clearly assessed. This phenomenon remains an open issue for the selection of bolt materials.

Irradiation - induced / modified precipitation

As for irradiation embrittlement literature survey shows a lack of experimental data on the effects of irradiation on precipitation kinetics in irradiated environment condition at the target values 0.2-5 dpa, 150-350°C.

4. CONCLUSIONS

Irradiation creep represents the most important stress relaxation mechanism, which affects the stress/strain behaviour of pre-loaded bolts and, consequently, of the surrounding materials in irradiated environment condition 0.2-5 dpa, 150-350°C.

A ranking of suitable materials for pre-loaded bolts in irradiated environment is proposed, based on the combined information on stress relaxation and irradiation creep behaviour.

In terms of irradiation creep behavior, martensitic steels are the most suitable materials for pre-loaded bolts in irradiated environment, as they have irradiation creep compliances lower than austenitic stainless steels and Ni based alloys.

Similar values of irradiation creep compliance have been reported in literature for conventional martensitic steels and ODS martensitic steels. These results seem to demonstrate that the presence of dispersoids does not have a significant influence on the irradiation creep behavior.

Ni based alloys are not suitable materials for pre-loaded bolts in irradiated environment, since they exhibit the highest irradiation creep compliance.

In terms of stress relaxation behavior, martensitic steels are also the most suitable materials for pre-loaded bolts in irradiated environment, as they have stress relaxation lower than austenitic stainless steels and Ni based alloys.
Martensitic-ODS steels stress relaxation behavior is quite similar to that of martensitic steels. These results seem to demonstrate that, also in this case, the presence of dispersoids does not have benefic effects on a reduction of stress –relaxation.
Not suitable materials for pre-loaded bolts in irradiated environment are Ni based alloys as they exhibit the highest pre-stress loss.
The most promising material to be used for pre-loaded bolts is PH13-8 Mo martensitic precipitation age-hardening stainless steel, 12.25-13.25 Cr, 7.5-8.5 Ni, 2.0-2.5 Mo , 0.9-1.35 Al, as it exhibits the lowest pre–stress loss.

On the basis of the above considerations, PH13-8 Mo martensitic steel can be considered the best candidate material to be used for pre-loaded bolts in irradiated environment.

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