

Application of mini-flat and cylindrical test specimens to extract hardening law and ductility of neutron irradiated Eurofer97

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Abstract

Tensile properties such as strength and ductility are essential for structural integrity assessment of critical components. In the context of nuclear applications, the flat tensile geometry as compared to the reference standard cylindrical geometry offers a number of advantages in terms of material use efficiency, ease of machining, best packing under irradiation and simplified remote handling of active samples. Accordingly, the interchangeability of data extracted from flat and cylindrical specimens is a key issue. Furthermore, this interchangeability must be demonstrated for irradiated samples. Many metallic materials show significant reduction or even a full lack of uniform elongation after neutron irradiation, with most of the strain hardening regime taking place during the post-necking stage. As the necking development depends on geometry, this raises questions on the validity of changing the test specimen geometry. Here, the interchangeability of mini-flat and cylindrical tensile samples after neutron irradiation is investigated by combining experimental and computational analysis. The investigated material is EUROFER97 steel irradiated at 300°C in the conditions relevant for the ITER fusion reactor. Finite element (FE) simulations are performed with a Gurson-type ductile fracture model parameterized based on the experimental tensile response. The hardening law extracted from mini-flat samples and applied to predict the stress-strain response of a cylindrical sample with 90% accuracy or better in terms of total elongation, reduction of area, and fracture strength as compared to the direct experimental data obtained with cylindrical tensile specimen geometry.

1. Introduction

Tensile properties are of the utmost importance in engineering design as they provide fundamental insights into material behaviour and strength under mechanical load. These properties, including yield strength, ultimate tensile strength, uniform elongation, true fracture strain and work hardening rate, are absolute pre-requisite for the structural analysis and design selection/optimization of the pressurized and load-bearing components. In the field of nuclear applications, understanding the material tensile behaviour is vital for assessing the design envisaged and off-design loads, for demonstration of structural integrity especially in the case of elastic-plastic analysis, and eventually for maintaining safety margins/rules. Accordingly, the determination of the relevant hardening laws for the plastic deformation of nuclear structural steels is needed to feed design codes such as ASME, RCC-MRx, R6, etc. For the future nuclear technology based on GEN IV and fusion, reduced activation ferritic martensitic (RAFM) steels are considered as perspective structural material candidates. So, efforts are made to populate the material handbook while putting emphasis on the modification of the properties due to the neutron irradiation.

EUROFER97 belongs to the family of RAFM steels and thanks to attractive mechanical properties and lack of long-term radiative decay becomes a preferred choice as the structural material for fusion reactors. In the near-term future, EUROFER97 will be applied for the Test Blanket Module (TBM) in ITER and is planned for DEMO as well. However, the utilization of EUROFER97 necessitates the completion of qualification tests, validation of design principles, and full integration into the RCC-MRx design code, which has been selected as the guideline for constructing ITER reactor (including its in-vessel components, operating during the nuclear phase). Particular emphasis is placed on the assessment of neutron irradiation effects as they play a significant role in the aging of in-vessel components. It is well-established that EUROFER97 steel experiences an irradiation-induced low-temperature hardening embrittlement (LTHE) phenomenon, leading to an increase of yield strength (YS) and ultimate tensile strength (UTS), an increase of ductile-to-brittle transition temperature (DBTT), and a loss of the total elongation when exposed to irradiation at temperatures ranging from 300 to 350 °C and doses as low as 0.1-15 dpa [2-4].

Considering the significant costs and limited volume in material test reactors, or even smaller volumes in dedicated fusion irradiation facilities (such as IFMIF and DONES), the European fusion programme has recognized the need to develop miniaturized specimen standards and protocols specifically tailored for fusion materials [5]. This research effort aims to enhance efficiency and cost-effectiveness in testing. The focus of the present study is to explore the utilization of mini-flat geometry as compared to a standard reference cylindrical geometry to derive the tensile properties, especially in the post-necking stage, which is particular to the irradiated materials. The usage of flat miniaturized samples has a number of important advantages over using cylindrical samples, namely: (i) better packing in small irradiation volumes; (ii) easier/faster fabrication by electric discharge machine (EDM), including re-usage of larger rectangular samples usually used for fracture toughness and impact testing; (iii) easier identification by labelling; (iv) easier manipulation and gripping systems; (v) possibility to apply hardness testing directly on the gauge section or in the cross-head sections; (vi) easier preparation of TEM samples, even after tensile testing. In the case of hard and brittle materials (i.e. tungsten), the fabrication of cylindrical samples can be essentially challenging or even impossible, and application of EDM remains the only viable option.

The geometry has an impact on the local stress distribution during the necking process [6], thereby influencing crucial material properties like total elongation and stress at fracture, which vary depending on the specific cross-sectional shape and material response to a localized plastic deformation. Accordingly, we investigate and establish the interchangeability of mini-flat and cylindrical geometry by combining experiments and recently proposed FEM-based procedure [1] which was developed also for EUROFER97. The samples were irradiated up to 0.5 dpa at irradiation temperature of 300 °C. The investigation was performed at room (RT) and irradiation temperature. Post-deformation microstructural examination was

performed to validate the prediction of the developed computational method and for the first time applied here for the neutron irradiated materials.

2. Material and methods

2.1 Experimental

2.1.1 Material

EUROFER97 is the European reference RAFM steel. The 4th batch of EUROFER97 with the ID number 79 was investigated in the present work in the frame of EUROfusion work package WPMAT before and after neutron irradiation [5]. The dimensions of the plate are 2345 x 1000 x 36mm³ after hot rolling. The heat treatments (heat number 988608) applied by the producer included normalization at 980°C for 45 minutes and tempering at 740°C with 18cm/min cycle time. The chemical composition of the EUROFER97 tested in this work is given in Table 1. The tensile properties in DEMO Material Property Handbook (MPH) which were determined from the first two batches of EUROFER97 will be used as the reference data. The basic mechanical properties of EUROFER97 can be found in [7], and the microstructure characterization was recently reported in [8].

Table 1. Chemical composition of EUROFER97 batch 4 (wt.%)

C	Si	P	S	Ni	Cr	Mo	V	Ta	W	Ti	Cu	Nb	Al	B	Co
0.09	0.019	0.002	0.003	0.01	8.6	0.002	0.2	0.12	1	0.001	0.005	0.001	0.0001	0.0009	0.03

2.1.2 Neutron irradiation

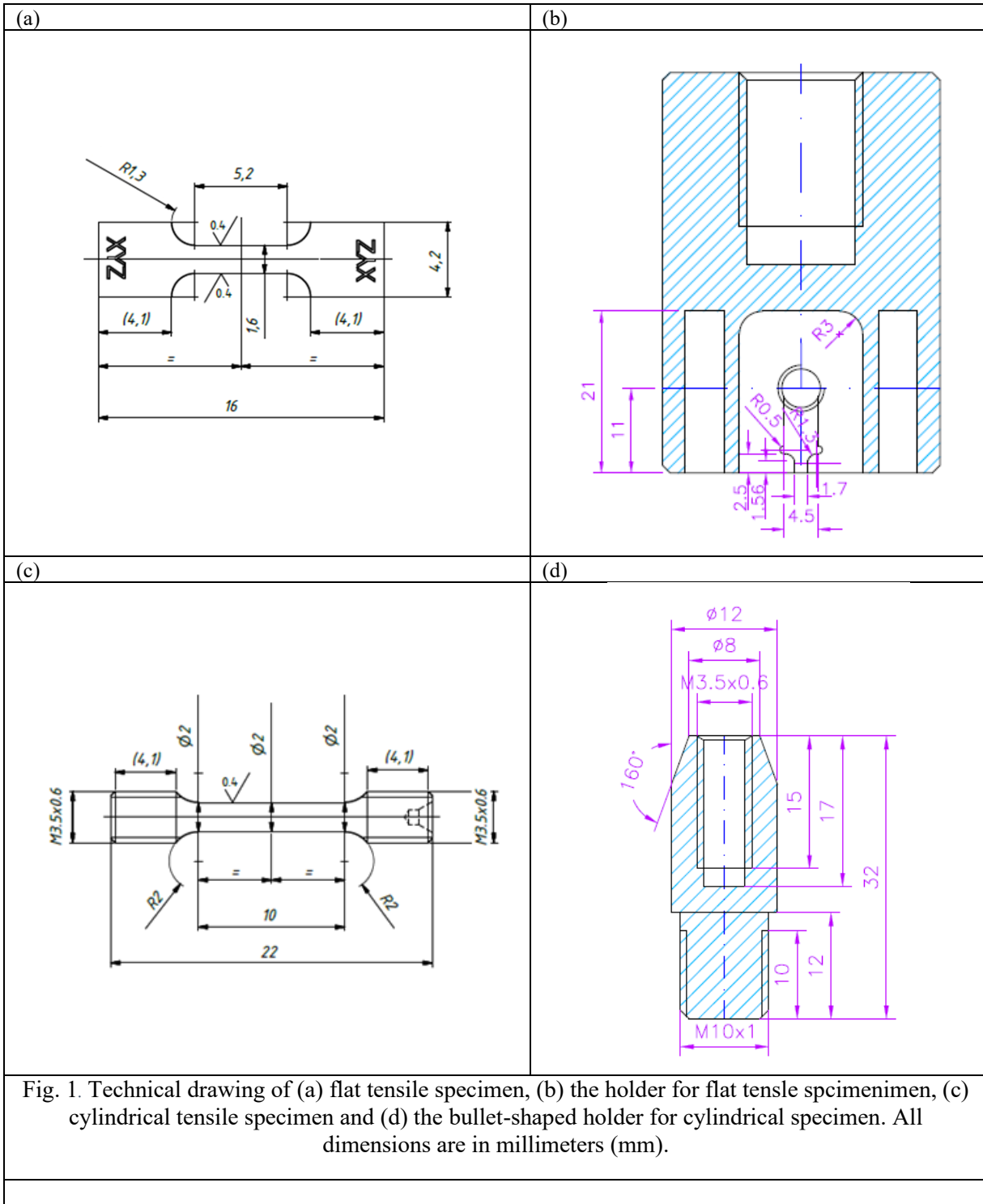
Neutron irradiation was performed in the Belgian Material Test Reactor 2 (BR2) at SCK CEN. The irradiation capsules were made of stainless steel with 1.35 mm thick walls and filled with helium to avoid the oxidation as well as to obtain necessary thermal balance to achieve the desired irradiation temperature. The capsules filled with samples were placed inside the fuel element in the position close to central channel of the reactor and in a mid-plane where fast neutron ($E > 0.1$ MeV) flux was equal to 7×10^{14} n/cm²/s at a nominal power of 60 MW. The thermal and neutronics calculations were performed in order to achieve a desired $T_{irr} = 300$ °C by tuning the gas pressure and the gap. The irradiation was carried out for one reactor cycles, and the dose reached is 0.5 dpa according to MCNPX 2.7.0 [9, 10]. For more details about the neutron irradiation conditions, one may refer to earlier similar studies [11-15].

2.1.3 Uniaxial tension testing

In this study, two types of miniaturized tensile samples were tested and investigated: flat dog bone with 1×1.6 mm² cross-section, and the reference cylindrical geometry with a gauge diameter of 2 mm. Both of them were cut along the rolling direction of the plate (L direction). The technical drawings of the specimen and the corresponding holders are shown in Fig. 1.

The uniaxial tensile tests were performed using INSTRON model 1362 for non-irradiated specimens and INSTRON model 8501 for irradiated specimens. The air furnace can heat up to 600 °C. The tests were performed at RT and irradiation temperature (300°C) under a strain rate of 2.78×10^{-4} /s for both geometries. All the tensile tests were performed following ASTM E8/E8M guidelines to generate the tensile properties

of the studied material.



2.2 FEM calculation procedure

The commercial software ANSYS Mechanical 2022 R2.02 was used for the FEM simulations. The mechanical behaviour of EUROFER97 was modelled assuming isotropic elasticity and a porous elastoplastic response represented by the Gurson-Tvergaard-Needleman (GTN) constitutive law [16]. The

use of a damage model makes the extraction of the hardening law of the dense material more accurate than if using a plasticity model without damage, as it allow decoupling damage softening effect from the strain hardening. This effect is not very strong but not totally negligible, e.g. [17, 18]. The GTN model is a built-in function in ANSYS workbench which accounts for the nucleation, growth, and coalescence of voids when evaluating the evolution of the yield locus, allowing to simulate, in addition to the elastoplastic response the fracture of any structure of test specimen (as long as the mechanism of damage and failure is the appropriate one). The material parameters entering the GTN model consist in the (isotropic) hardening law and the descriptors of the initial state of damage (essentially initial porosity), of the void nucleation law and of the porosity at coalescence.

2.2.1 FEM model

The axisymmetric FEM models were built according to the dimensions of tested samples for both geometries as shown in Fig. 2. An extremely small geometrical defect (1 μm) was introduced in the sample center for both geometries as illustrated in Fig. 3 to ensure the necking takes place in the middle section. The mesh near the necking region was refined to the size of 0.015 mm to have at least 53 elements over the width or diameter for both cylindrical geometry and flat geometry. Mesh sensitivity studies were performed for both geometries by increasing or decreasing the mesh size by 50%, and a marginal difference (lower than 5 %) on the predicted engineering stress-strain curves was found in both cases.

Given that this work addresses two different specimen geometries, it is important to investigate the difference in terms of the key characteristic of the necking process which is the evolution and magnitude of the stress triaxiality in the minimum cross-section, e.g. [19]. Indeed, the stress triaxiality will directly impact the void growth and coalescence rate and set the value of the fracture strain in terms of total elongation or true fracture strain. The level of the stress triaxiality is known to decrease as the strain hardening capacity increases due to shallower neck profiles. This adds to the fact that a larger strain hardening exponent delays the onset of necking hence delays the stage at which the stress triaxiality starts departing from the value of 1/3. These subtle non-linear effects cannot be captured with closed-form models but require FE simulations in a finite strain setting. The effect of sample's shape on the distribution of the stress triaxiality between two geometries studied in this work was already studied in [1] in the non-irradiated state..

Secondly, it is important to recognize that the investigated material has very fine microstructure, with the principal structural element being the lath-type constituent, which has a dimension of the order of 300-500 nm only. This implies that the surface roughness of the samples is even larger than the size of the lath. Accordingly, the flat-surface curvature in the flat and cylindrical samples is highly unlikely to affect the response of these specimens. Therefore, no attention to this aspect when setting up and analyzing the results of the FEM tool.

(a)

(b)



Fig. 2. Schematics of the axisymmetric model for (a) cylindrical tensile geometry and (b) flat tensile geometry with mesh.

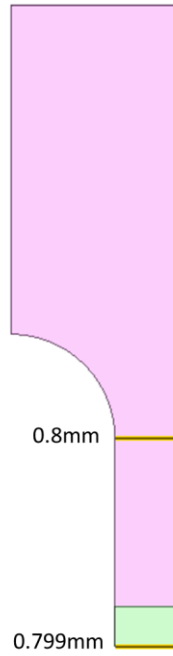


Fig. 3. Schematics of the geometric defect

2.2.1 Hardening law

The hardening law of a material expresses the variation of the current yield stress with the accumulated plastic strain due to strain hardening and sets the isotropic expansion of the yield surface during plastic deformation. This information can be determined in principle based on any mechanical test out of which effective stress versus accumulated plastic strain response can be extracted. The uniaxial tensile test is the most common geometry providing this behaviour up to the ultimate tensile strength (UTS) point (the onset of diffuse necking) by means of

$$\sigma_{true} = \sigma_{eng}(1 + \varepsilon_{eng}), \quad (1)$$

$$\varepsilon_{true} = \ln(1 + \varepsilon_{eng}), \quad (2)$$

where σ_{true} , ε_{true} indicate the true axial stress and corresponding true total strain, respectively, and σ_{eng} , ε_{eng} indicate the engineering stress and strain, respectively. The effective stress is equal to the true stress and the true accumulated plastic strain is equal to the true strain minus the elastic strain. The.

After the onset of necking, the hardening law can no longer be derived directly from the force-displacement record due to the inhomogeneous deformation across the sample gauge section. Accordingly, the local information about the neck geometry and section reduction is needed to deduce the true effective stress – true accumulated plastic strain response beyond the point of necking. Various approaches have been developed, such as the Bridgman correction, initially developed for cylindrical samples, to extract hardening data from the post-necking deformation region of the force-displacement curve [20]. This method necessitates information about both the evolution of the neck section's radius (R) and the radius (a) of the neck curvature. However, the extraction of reliable measurements is challenging due to the random positioning of the neck on a smooth specimen. Thus, Le Roy proposed an empirical relationship between a and R , incorporating the minimum cross-section radius (a) and the necking curvature radius (R), along with the average equivalent strain in the minimum cross-section for cylindrical samples [21]. For the flat geometries, several researchers have proposed modified equations based on the Bridgman correction [6, 22, 23]. However, uncontrolled uncertainties remain regarding the non-uniform flow process occurring from the initiation of diffuse necking to the onset of localized necking, and regarding the approximate treatment of the plasticity problem associated to the Bridgman model.

Digital image correlation (DIC) was employed to capture the true stress-strain curve after the onset of necking [24-26]. However, the applicability of DIC is limited due to certain constraints and the absence of standardized test conditions. Additionally, challenges arise when employing DIC at elevated temperatures, also as achieving the desired speckle pattern on miniaturized samples proves to be a non-trivial task. The application of DIC tools on irradiated samples with high activity is extremely challenging due radioactive environment inside the hot cells damaging the DIC equipment. Finally, the approximations associated to the Bridgman type corrections remain and should be challenged.

The true stress-strain response of EUROFER97 was reported in [27] using both the DIC method and the Bridgman correction technique. The investigation revealed that the post-necking behaviour exhibited a quasi-linear trend, extending up to significant strains prior to fracture. And from our previous study [1], we have proven that a good agreement in reproducing engineering stress-strain curve from FEM compared to experimental data can be achieved under assumption of a linear increase of hardening law from UTS to fracture point.

The fracture initiation point can be obtained as

$$\sigma_{true f} = \frac{F_f}{A_f}, \quad (3)$$

$$\varepsilon_{true f} = \ln\left(\frac{A_0}{A_f}\right), \quad (4)$$

where $\sigma_{true f}$, $\varepsilon_{true f}$ indicate true axial fracture stress and true fracture strain, respectively. F_f is the load at the moment of cracking initiation usually appearing as a sharp change of the slope with brutal drop of the force afterwards. A_0 , A_f are the cross-section area of the thinnest gauge part in the undeformed configuration and after the fracture, respectively.

The slope that can be drawn from the true stress at necking and the true stress at fracture provides a first estimate of the slope of the hardening law that can be used for the first iteration of the inverse FE procedure to extract a precise hardening law. Note that indeed the true axial stress at fracture is not equal to the effective stress at fracture (hence not equal to the yield stress at fracture) as, during necking, transverse stresses develop. The slope of the post-necking linear extrapolation is then iteratively changed until the FE simulation perfectly reproduces the engineering stress-strain curve. This methodology was used for neutron irradiated specimens. For more details, we refer the reader to [1].

2.2.2 Gurson-Tvergaard-Needleman (GTN) model

Ductile fracture in a metal is generally the result of process of nucleation, growth, and coalescence of microvoids, e.g. [28-30]. Some micro-voids are present in the material due to the manufacturing steps while more voids are nucleated during deformation mostly through the decohesion and/or fracture of second phase particles, see e.g. [31, 32]. Gurson proposed a yield function considering an ideal-plastic Mises material with an isolated spherical void in 1977 [33] and was later modified and introduced new adjustment parameters by Tvergaard and Needleman [34]. Furthermore, based on Chu and Needleman's empirical law for void nucleation [35], Tvergaard and Needleman supplemented the Gurson model with a phenomenological law of the loss of load-carrying capacity at a realistic void volume fraction [16]. The modified yield function is commonly called Gurson –Tvergaard–Needleman (GTN) model. This model has been successfully applied to simulate deformation-induced damage in many ductile metallic materials, including EUROFER97 steel. The yield surface of the GTN model is given by:

$$\Phi = \left[\frac{\sigma_{eq}}{\bar{\sigma}} \right]^2 + 2q_1 f^* \cosh \left[\frac{3q_2 \sigma_m}{2\bar{\sigma}} \right] - 1 - (q_1 f^*)^2, \quad (5)$$

where σ_{eq} , $\bar{\sigma}$, and σ_m are the von Mises equivalent stress, the flow stress of the matrix material, and the hydrostatic pressure, respectively; f^* is the effective void volume fraction (VVF) proposed by Tvergaard and Needleman. This phenomenological function is defined as:

$$f^* = \begin{cases} f, & f \leq f_c, \\ f_c + \frac{f_u - f_c}{f_f - f_c} (f - f_c), & f_c < f < f_f, \end{cases} \quad (6)$$

where f is the true VVF, f_c corresponding to the onset of void coalescence, f_f is the threshold for the moment at which microvoid coalescence ends resulting in full loss of load-bearing capacity. The evolution of the volume fraction f consists of two terms: the nucleation of new voids and the growth of existing voids, as:

$$\dot{f} = \dot{f}_{nucleation} + \dot{f}_{growth}, \quad (7)$$

where $\dot{f}_{nucleation}$ is the rate of the void nucleation with respect to the average effective plastic strain rate of the matrix material $\dot{\epsilon}_{eq}^{pl}$ was proposed by Chu and Needleman, in the following form

$$\dot{f}_{nucleation} = A \dot{\epsilon}_{eq}^{pl}, \quad (8)$$

the parameter A is a Gaussian distribution as defined as

$$A(\bar{\epsilon}) = \frac{f_n}{S_n \sqrt{2\pi}} \exp \left[-\frac{1}{2} \left(\frac{\bar{\epsilon} - \epsilon_N}{S_n} \right)^2 \right], \quad (9)$$

where f_n is the total nucleated VVF, $\bar{\epsilon}$ is the equivalent plastic strain of matrix material, and ϵ_N and S_n are the mean value of the Gaussian distribution of the nucleation strain and standard deviation, respectively.

$\dot{f}_{growth}$ is the rate of the growth of existing void with respect to the trace of the macroscopic plastic strain rate tensor $\dot{\epsilon}_{kk}^p$, which writes

$$\dot{f}_{growth} = (1 - f) \dot{\epsilon}_{kk}^p. \quad (10)$$

Finally, the relationship between the plastic dissipation within the matrix material and the plastic work rate of the voided material proposed by Gurson in his original paper is used together with the hardening law:

$$\bar{\sigma} \dot{\epsilon}_y^p (1 - f) = \sigma_{ij} \dot{\epsilon}_{ij}^p . \quad (11)$$

where $\dot{\epsilon}_y^p$ is the accumulated plastic strain rate of the matrix material, and $\bar{\sigma}(\epsilon_y^p)$ the hardening law that is searched in this study.

3. Results

3.1 Experimental data on reference and irradiated tensile samples

Fig. 4(a) and Fig. 4(b) show the experimental engineering stress – strain (S-S) curves for non-irradiated and irradiated tensile specimens for both geometries. The flat geometry gives a total elongation larger by 5% and 2.5% as compared to the cylindrical geometry for both non-irradiated and irradiated specimens, respectively. The LTBE phenomenon is well pronounced for both geometries, and after the neutron irradiation there is an evident increase in YS, UTS and decrease in TE. For the flat specimens, the irradiation leads to an increase of the yield strength by 25% at RT and 18% at 300°C while the total elongation is reduced by 29% at RT and 22% at 300°C. For cylindrical specimens, the yield strength increased by 18% at RT and 20% at 300°C while the total elongation was reduced by 14% at RT and 13% at 300°C after irradiation. Although the application of flat tensile sample leads to larger total elongation, the absolute value of this “overestimation” reduces after the irradiation. However, no premature necking occurs yet in the studied irradiation condition, apparently a higher irradiation dose (or lower irradiation temperature) is required for that. Based on these tensile curves and visual inspection of the broken samples applied to deduce the reduction of area (which is a much better and scientifically relevant indicator than the total elongation) the deduction of the hardening law was performed following the methodology described in the previous section.

(a)

(b)

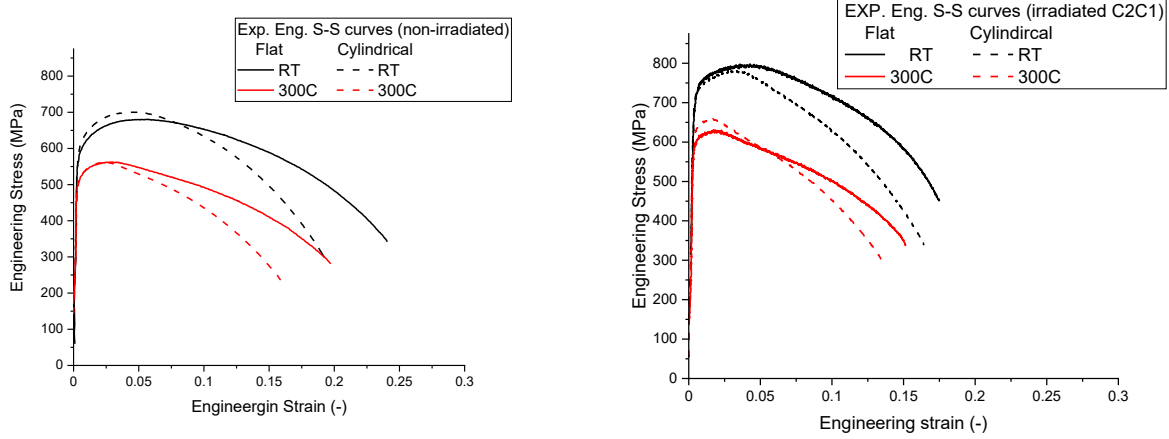


Fig. 4. Engineering S-S curves of flat and cylindrical specimens in (a) non-irradiated state [1] (b) irradiated state.

3.2 FEM results with GTN model (flat geometry)

As mentioned in 2.2.1, the hardening law was extracted from a uniaxial tensile test following a three-steps procedure. The first step simply consists of the determination of the first part of the hardening law from the yield point to the UTS as it is (while only extracting the elastic strain from the total strain). The second step is to connect the UTS to the fracture point through a linear evolution. The third step consists of fine tuning the linear post necking evolution to get a perfect match with the experimental engineering response. Table 2 provides the accumulated plastic strain versus yield stress pointwise values of the extracted and fine-tuned hardening laws from the irradiated flat samples, that can then be used to simulate other test configurations. Fig. 5 shows the comparison of the engineering S-S curves from FEM (dashed lines) with experimental data (solid lines) demonstrating good accuracy of the fit after step 3.

The use of the GTN model is key to predict cracking initiation when changing geometry and thus the stress triaxiality. It has been found that the values of the parameters of the GTN model extracted at RT can also be used up to 450°C providing a good agreement with experimental data, as shown in our previous study [1]. Chakraborty et al. have reported that the GTN parameters are unaffected by irradiation for EUROFER97 up to 1.5 dpa [36]. Hence, most of the GTN parameters extracted from the non-irradiated tests, indicated as “Cold” in Table 3, are also applied to simulate the response of the irradiated material. However, ε_N and S_N differ from the non-irradiated values. These parameters can be obtained from experiments directly as proposed by Pan et al. [37]. Fig. 6(a) shows the comparison of FEM calculations with the GTN model (dashed lines) with experimental data (solid lines). The total elongation is overestimated for both test temperatures. However, the change of initial void volume fraction after neutron irradiation was not accounted for in [36]. Neutron irradiation at 300 °C is known to induce voids and so we increased the initial void fraction from 0% to 0.1%, as shown in Table 3, which is close to the experimental observation of EUROFER97 by Coppola et al. under similar irradiation condition as studied here [38] (note also that such small porosity do not affect the overall stress strain response and thus not affect the extraction of the hardening law). Furthermore, Pareige et.al. reported the density and the size of the solute-vacancy-rich clusters observed in Fe-9%Cr steel under ion irradiation ($T_{irr} = 300C$, 0.5 dpa) and neutron irradiation ($T_{irr} = 300C$, 0.6 dpa) [39], which are $2.8 \pm 0.6 \times 10^{23} m^{-3}$, $1.5 \pm 0.3 nm$ and $2.4 \pm 0.5 \times 10^{23} m^{-3}$, $1.6 \pm 0.2 nm$, respectively. These clusters may act as precursors of the damage, thereby strengthening the hypothesis that a fraction of 0.1% of irradiation-induced defects may occur. Other GTN parameters were kept unchanged. Fig. 6(b) shows the FEM results (dashed lines) after setting the initial

void volume fraction to 0.1%, showing that fracture occurs earlier and now matches much better the experimental data. Fig. 7(a) and Fig. 7(b) demonstrate a good agreement in the comparison of the area after fracture in flat samples between FEM with the 0.1% initial void fraction (red line) and experimental data at RT and 300°C, respectively. Detailed comparison of total elongation, reduction of area and fracture strength from experiments and FEM using different sets of GTN parameters are summarized in Table 4.

Table 2. The pointwise hardening response extracted from irradiated flat geometry at different test temperatures using the inverse identification procedure

RT													
Accumulated Plastic Strain	0	0.008	0.016	0.024	0.032	0.040	0.048	0.052	0.055	0.105	0.505	1.005	1.525
Yield Stress	733	781	799	812	825	834	842	846	849	860	941	1043	1149
300°C													
Accumulated Plastic Strain	0	0.002	0.004	0.006	0.008	0.010	0.012	0.014	0.2	0.4	0.6	0.8	1.040
Yield Stress	595	614	622	626	630	634	637	641	661	682	703	724	750

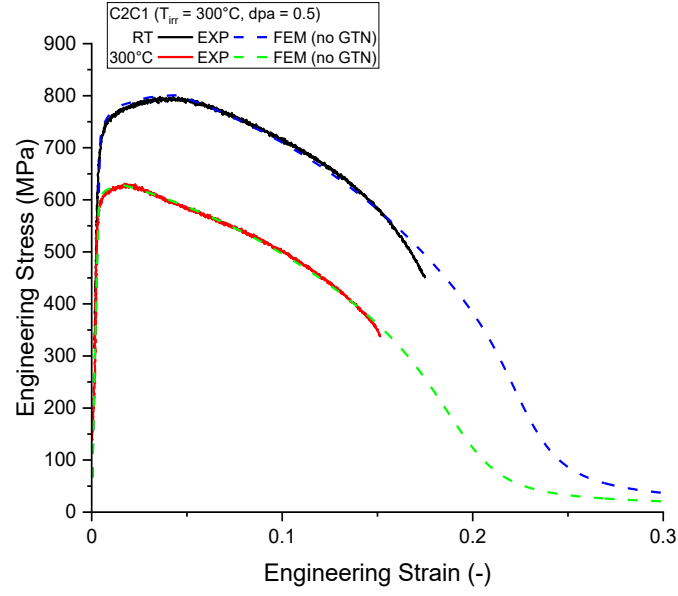
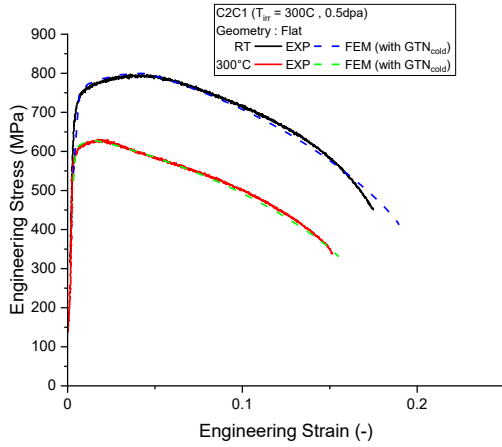


Fig. 5. The comparison of the experimental data and FEM results using extracted hardening laws

Table 3. GTN parameters used in this studied

set	q ₁	q ₂	q ₃	f ₀	f _N	ε _N		S _N		f _c	f _F
Cold	1.5	1	2.25	0	0.00055	RT	0.041	RT	0.014	0.18	0.22
						300°C	0.019	300°C	0.006		
0.1% initial void fraction	1.5	1	2.25	0.001	0.00055	RT	0.041	RT	0.014	0.18	0.22
						300°C	0.019	300°C	0.006		

(a)



(b)

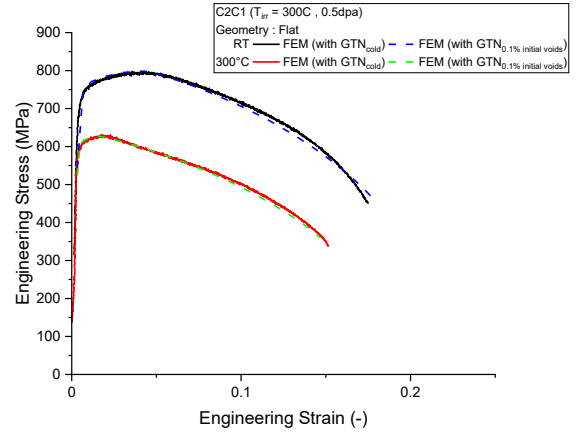


Fig. 6. The comparison of the experimental data and (a) FEM results with GTN model obtained from

cold sample, (b) FEM results with increased initial void volume fraction (0.1%)

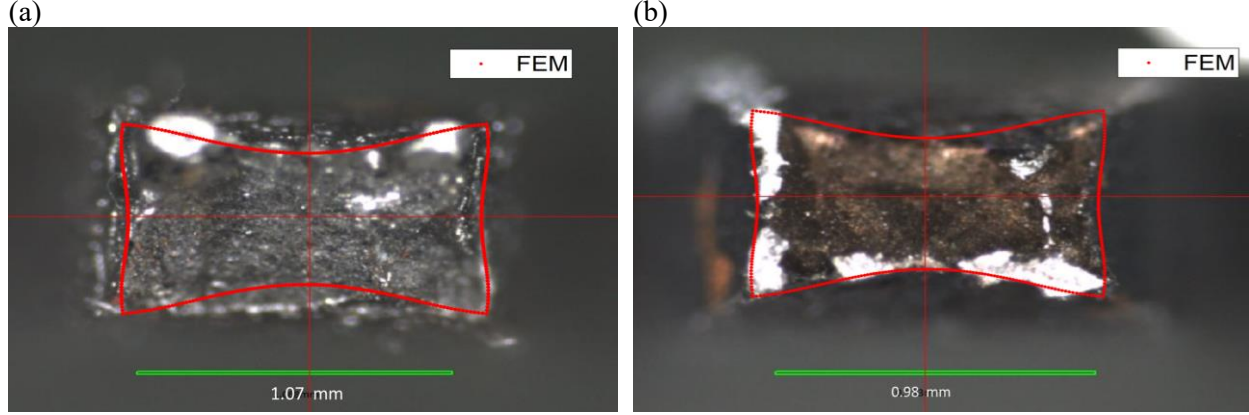


Fig. 7. Comparison of the simulated (red line) and experimental fracture area with the scale bar (green line) at (a) RT and (b) 300°C

Table 4. Comparison of total elongation (TE), reduction of area (RA), fracture strength (FS) and prediction accuracy (PA) for the flat geometry as obtained by FEM and experiment.

GTN parameter set	Temp. (°C)	TE (%)			RA (%)			FS (MPa)		
		EXP	FEM	PA	EXP	FEM	PA	EXP	FEM	PA
Cold	RT	17.6	19	92.0%	65.6	69.6	93.9%	1320.7	1356.3	97.3%
	300°C	15.1	15.5	97.4%	62.3	65.2	95.3%	906.5	950.1	95.2%
0.1% initial void fraction	RT	17.6	17.9	98.3%	65.6	64.8	98.8%	1320.7	1290.2	97.7%
	300°C	15.1	14.7	97.4%	62.3	60.7	97.5%	906.5	915.1	99.1%

3.3 FEM simulation of cylindrical geometry using parameters extracted from flat geometry

The hardening law and GTN parameters with 0.1% void fraction as extracted from the irradiated flat samples at different test temperatures were used to simulate the uniaxial test performed with the cylindrical geometry. Fig. 8 shows the comparison of the engineering S-S curves from FEM (dashed) to experimental ones (solid). The predicted results such as UTS, UE and TE exhibit small deviation from the experimental data. This deviation may be attributed to the accuracy of the proposed method as well as to a slightly different irradiation condition. Indeed, Fig. 4(b) shows that the YS and work hardening slightly differ for flat and cylindrical samples. Although both set of samples were irradiated in the same capsule, the local temperature distribution during the irradiation may have slightly varied (because of the different shape of the sample holders). Fig. 9(a) and Fig. 9(b) demonstrate a good agreement in the comparison of the area after fracture in cylindrical samples between FEM with the 0.1% initial void fraction (red line)

and experimental data at RT and 300°C, respectively. Detailed comparison of TE, RA and FS obtained from FEM to experimental data are reported in Table 5: the prediction accuracy for each extracted property exceeds 90%.

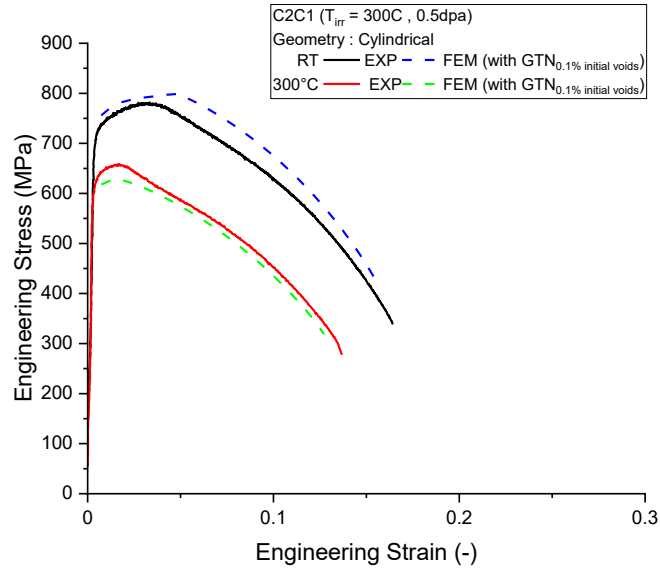


Fig. 8 Engineering S-S curve of cylindrical geometry from FEM compared to experimental curves by using the constitutive law extracted from flat specimen

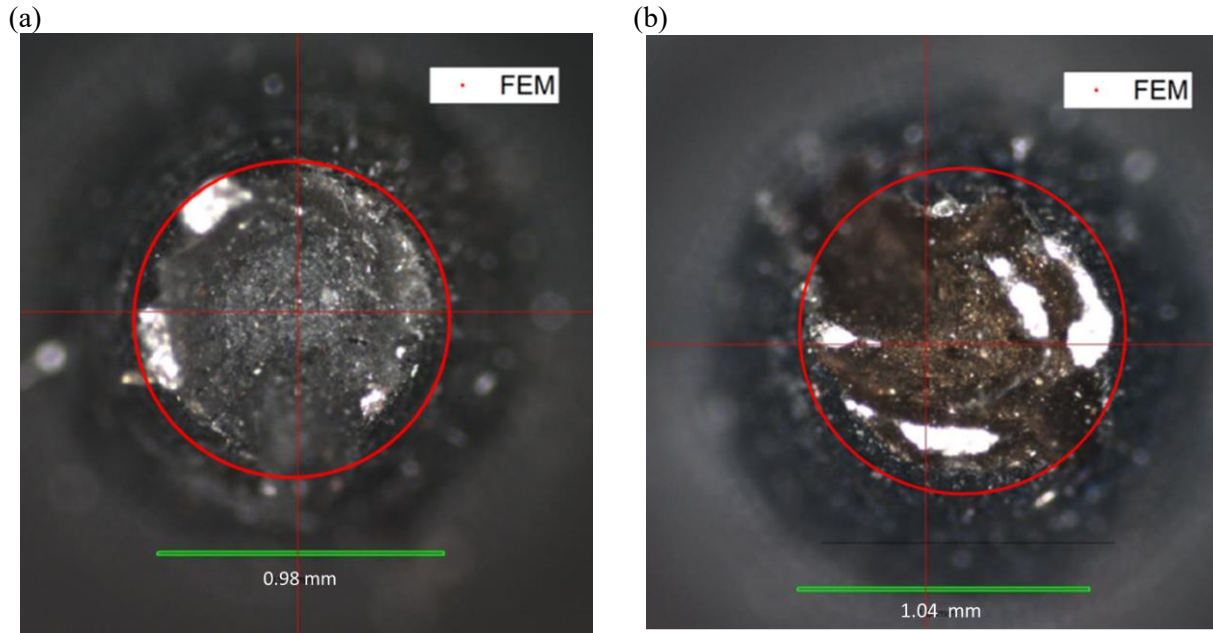


Fig. 9. Comparison of the simulated (red line) and experimental fracture area with the scale bar (green line) at (a) RT and (b) 300°C

Table 5. Comparison of total elongation (TE), reduction of area (RA), fracture strength (FS) and prediction accuracy (PA) in cylindrical geometry as obtained by FEM and experiment.

GTN parameter set	Temp. (°C)	TE (%)			RA (%)			FS (MPa)		
		EXP	FEM	PA	EXP	FEM	PA	EXP	FEM	PA
0.1% initial void fraction	RT	16.2	15.6	96.2%	75.9	69.4	91.4%	1354.5	1356.2	99.9%
	300°C	13.5	12.7	94.1%	71.1	66.8	94.0%	960.6	960.9	100%

4. Discussion

The currently available DEMO Material Property Handbook (MPH) [40] incorporates data on the irradiation effects acquired from EUROFER97 Batch 1 and Batch 2. However, neither of these batches is presently intended for the weldment studies or the fabrication of mock-up TBM sub-components. To close the gap, it is crucial to conduct a thorough characterization of the irradiation effects in EUROFER97 Batch 3 and Batch 4, especially considering their exposure to neutron irradiation at operational temperature.

Fig. 10 (a) and Fig. 10(b) shows the tensile properties of EUROFER97 batch 4 tested at SCK CEN compared to the data for EUROFER97 batch 1 and 2 from MPH for non-irradiated state and irradiated state, respectively. For non-irradiated state, EUROFER97 batch 4 demonstrates a better performance in terms of higher YS, higher UTS while remaining almost the same UE, TE and RA. Furthermore, EUROFER97 batch 4 also demonstrates a better resistance to neutron irradiation in terms of lower increase of YS and UTS compared to irradiated data for EUROFER97 batch 1 and 2 as shown in 10(b), yet having a slightly higher TE. Note that the UE of EUROFER97 batch 1 and 2 is currently not included in the MPH due to the difficulties of measuring very small deformations. It is a well-known phenomenon that the UE of EUROFER97 irradiated around 300 °C is reduced already at dose as low as 0.04-0.1 dpa and saturates to the value lower than 0.4-0.5% at higher doses of 15-20 dpa [41, 42]. However, in this study, the UE of the irradiated samples remains more or less the same as non-irradiated samples. Apparently, the saturation of hardening might not be reached in the present experiments due to an insufficiently high dose.

(a)

(b)

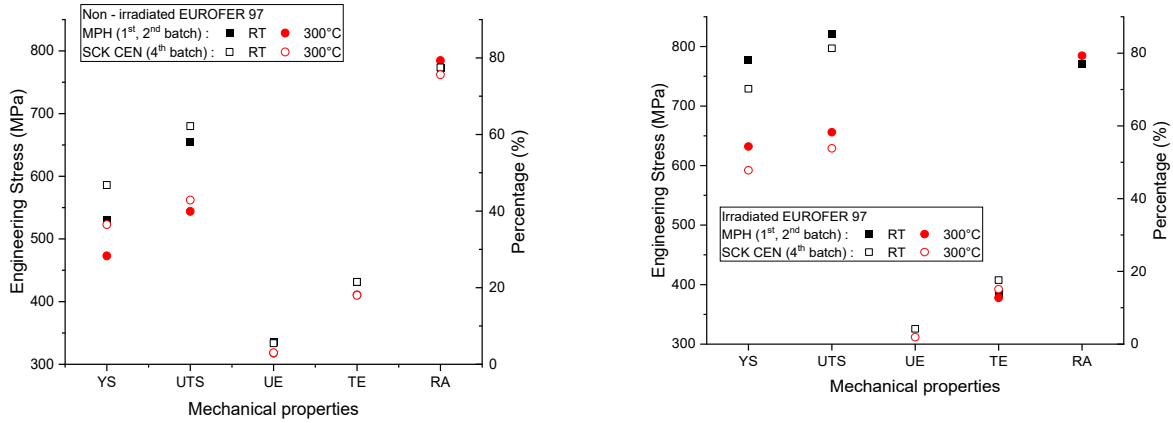


Fig. 10. The comparison of the tensile properties between the material handbook (reference materials) and new batch of EUROFER97 in (a) non-irradiated state and (b) irradiated state.

Fig. 11 shows the hardening laws as extracted from non-/irradiated flat tensile samples at different test temperatures. The pronounced irradiation hardening can be seen in the increased yield stress which is due to the presence of the microstructural defects (voids and dislocation loops) introduced by neutron irradiation which act as obstacles to dislocation motion. The hardening rate is reduced by the irradiation. Such observation was also reported by Liu et.al. who performed the true stress-strain curve extraction from ion irradiated RAFM steel by various methods [43]. The reduction of the work hardening slope from UTS to the fracture point may imply that the necking localized deformation exhibits weaker resistance in the irradiated material as compared to the non-irradiated one. This weaker resistance might be related to the formation of clear channels and subsequent localized heterogeneous deformation, as was found in the case of pure Fe and Cu [44, 45]. The mechanism of the clear channel formation is attributed to the absorption of the dislocation loops by moving dislocations [46, 47], which leads to (i) reduction of the obstacle density thus reducing the potential for increase of actual flow stress and (ii) to the formation of defect-free bands where the plastic deformation localizes causing the emergence of stress-concentration at the end of channels (channels are expected to be ended at grain boundaries) or at the intersection of the channels. Given that in the present irradiation conditions the presence of dislocation loops is expected [48, 49], the observed reduction of the work hardening can be indeed explained by the formation of the deformation channels.

In addition to the pure plastic deformation, the irradiation defects may influence the physical mechanisms of fracture/cracking behavior that could be associated with e.g. grain boundary embrittlement due to the segregation/precipitation or bubble formation, which would require the modification of the FEM model.

However, in the present study we did not consider grain boundary or interface debonding features. Indeed, an adequate investigation of fracture and crack propagation must involve fracture toughness and fatigue crack growth tests to define the interface debonding stress in a statistically meaningful manner. These tests on neutron irradiated material are not available, whereas in the case of tensile tests, the fracture occurs shortly after the critical crack is nucleated. This is why tensile test is not well suited for the determination of the full set of GTN parameters. In this work, the focus is therefore made on the post-necking deformation but not on the fracture initiation. We have used the GTN parameters defined in earlier study for Eurofer97 and demonstrated: (i) good prediction of the fracture point for non-irradiated samples; (ii) that the prediction of the fracture point of the 0.5 dpa irradiated sample can be explained by an increase of

the initial void volume fraction of 0.1%. In fact, previous TEM studies made on < 3 dpa irradiated Eurofer97 at 60-300 °C temperature range did not reveal the TEM-resolvable voids. However, more sensitive techniques such as small angle spectroscopy [50] actually determined the presence of voids and confirm that the size of those voids is below 2 nm, which is why these are not resolvable in TEM. The void fraction reported at 2.7 dpa is about 0.3% which is in the range of here applied value of the void fraction. We also would like to highlight that at 0.5 dpa $T_{irr}=300$ °C, the damage level is still too low to cause any significant chemical modifications such as segregation or precipitation. At least, up to our best knowledge, such microstructural modifications were never reported for such moderate irradiation dose as 0.5 dpa.

However, the detailed microstructural analysis with the application of transmission electron microscopy and void-sensitive techniques such as positron annihilation spectroscopy or small angle neutron scattering in the uniform elongated and necked region would for sure help to better substantiate the selection of the FEM model and its parameterization.

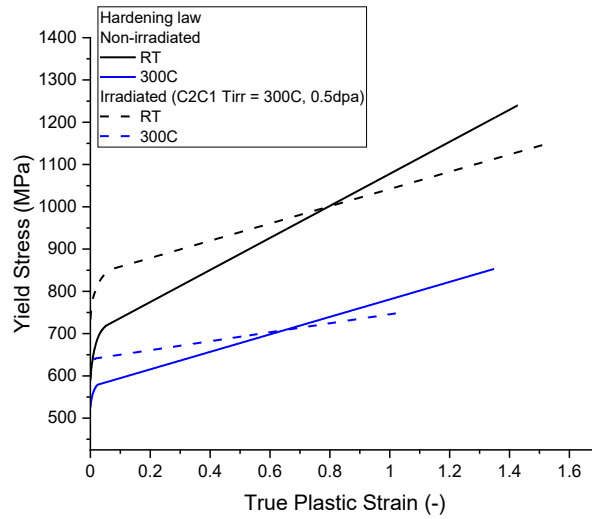


Fig. 11. The comparison of the hardening laws extracted from non-irradiated and irradiated samples at different test temperatures.

5. Conclusions

In this work, the recently proposed method of extracting the true stress–strain curve is applied to the flat tensile sample after neutron irradiation. Furthermore, the GTN damage model is used to capture fracture strain with the adjusted parameters to achieve a good agreement between FEM and experimental tensile data, in term of the elongation and area of reduction. The GTN model was successfully applied for the flat samples at two different test temperatures. The final goal of this study is to assess the accuracy and conservatism of applying the constitutive laws extracted from irradiated flat samples to simulate the tensile response including post-necking deformation of the cylindrical sample geometry. The accuracy of the prediction was then validated using the experimental data.

Based on the obtained results and discussion, the following statements can be made on the interchangeability of flat and cylindrical tensile samples for the derivation of the post-necking properties after neutron irradiation:

- 1) The proposed simplified method of extracting hardening law can also be used for irradiated tensile samples offering good accuracy.
- 2) The most important post-necking tensile characteristics such as total elongation, reduction of area and fracture stress can be reproduced by FEM with an accuracy above 90% compared to experimental data by using the modified set of cold GTN parameters for irradiated flat samples at different test temperatures. All GTN parameters derived from non-irradiated samples were used as is to reproduce the fracture of the neutron irradiated samples except the three parameters which were modified, namely: the initial void volume fraction f_0 was increased from 0 to 0.1% and ε_N and S_N were modified according to the previous study done for irradiated EUROFER97.
- 3) The predicted post-necking tensile characteristics for the cylindrical samples are in a good agreement with experimental data, offering 90% or better prediction accuracy.

The methodology for the transfer of the tensile characteristics by testing a flat sample and accurately predicting the response of cylindrical sample has been demonstrated for neutron irradiated samples up to the damage dose of 0.5 dpa. The next step is to investigate the applicability of this method up to 3-5 dpa for EUROFER97 and copper-based alloys, which is a relevant irradiation dose for structural materials for ITER and DEMO components.

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