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# Mechanical properties of Eurofer 97 in Pb–16Li and irradiation effect

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#### Abstract

To be used in a fusion reactor, structural materials, and in particular steels, has to be selected and optimised in their composition to achieve a reduction in the long-term radioactive waste. A reduction in the long-term radioactive inventory could be reached substituting elements like molybdenum, niobium and nickel with other ones like tantalum and tungsten which have the same functions as alloying elements and, if irradiated, do not produce long lived radioisotopes. The martensitic steel belonging to the family of 8–9% Cr Eurofer 97 is considered the reference structural steel for fusion application. However, only few information are available about its mechanical properties in the liquid eutectic alloy Pb–16%Li. Particularly, the problem of liquid metal embrittlement (LME) has not been studied in detail and the effect of neutron irradiation on LME has not been investigated at all so far. This work presents the results obtained irradiating tensile specimens of Eurofer 97 up to 5.9 dpa in lead lithium. Tensile tests of samples have been performed out of pile in the same alloy at the same temperature at which irradiation was carried out. © 2008 Elsevier B.V. All rights reserved.

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# 1. Introduction

It is well known that reduced activation martensitic steels are of particular interest in the field of R&D for fusion oriented materials. As a matter of fact, in order to fully exploit the advantage connected to both fuel and reaction product in D–T fusion reactions which do not possess a long-term radioactivity, it is important to optimise the structural materials towards a reduced long-term radioactive inventory. This is possible substituting elements like molybdenum, niobium and nickel with other ones like tantalum and tungsten which have the same functions as alloying elements and, if irradiated, do not produce long lived radioisotopes.

However, only little information are available about the effect of long-term irradiation on the mechanical proper-

ties, especially in presence of lead lithium which may cause liquid metal embrittlement (LME). LME and its dependence on neutron irradiation are of basic importance because under certain operative conditions the consequences on structural materials can be dramatic. It is well known that the main phenomena associated to LME are a high crack propagation rate and a drastic reduction of the plastic strain at rupture and a modification of stress– strain curve, especially in the plastic region [1]. This means that, under irradiation, a catastrophic ductile to brittle transition can occur in operation.

Literature results [2,3] referred to martensitic steels tested in presence of Pb–16Li demonstrated that LME is strongly dependent on the hardness of the material.

However, the different theories so far developed do not seem to describe in a satisfactory way the basic mechanisms related to LME even without irradiation. As a consequence, reliable data on LME, especially under neutron irradiation, are necessary and have to be obtained through a well defined experimental campaign.

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Investigations of reactor irradiation influence on LME of steel Eurofer 97 have been carried out for samples irradiated in the fast reactor BOR-60. The samples were placed in ampoules filled with the eutectic alloy Pb–16Li. Irradiated and tested.

Irradiation temperature was 310–340 °C and damaging dose was 0.2–5.9 dpa depending on disposition throughout the height of the core region. Tensile tests of samples have been performed out of pile in the same alloy at the same temperature at which irradiation was carried out. Mechanical properties of irradiated samples are here presented.

# 2. Experimental

## 2.1. The irradiation assembly

## 2.1.1. Irradiation ampoule

The irradiation ampoule is presented in Fig. 1. in which claddings and specimen before assembly are presented. The ampoule consists of the cladding (1) and lower (2) and upper (3) end-plugs. From the inner side the lower endplug has a screwed hole. The outer side of this plug can be maintained in the grip of the loading mechanism. Final elements are fastened to the sample by means of fingers (4). After assembly the ampoule is filled with Pb-16Li alloy, completely covering the specimen. Thus, after opening, and maintaining the irradiated ampoule in the tensile test machine, the specimen can be tested being in contact with the alloy in which it was immersed during irradiation. In Fig. 2 the specimen geometry is reported. Specimens are realized in Eurofer 97, the reference European low activation martensitic steel, produced by Boehler Edelstahl GMBH. The material was delivered normalized and tempered, and machine worked as received. Each ampoule contains one specimen.

# 2.1.2. Material testing assembly for irradiation

The design of the material testing assembly used for irradiation is presented in Fig. 3. It consists of outer (4) and inner (5) hexagonal wrappers, lower (2) and upper (11) adaptors, a tail (1) and a removable head (12). Heat insulation of the inner part of the testing assembly from sur-



Fig. 2. Specimen geometry.

rounding fuel assemblies is provided by filling up the gap between the outer (4) and inner (5) hexagonal wrappers with argon. The tail (1) has lateral holes to let the coolant flows into the assembly from the high-pressure chamber of the BOR-60 reactor. The lower suspension has two irradiation ampoules (3) fixed to it at level 0. The center of level 0 specimens is 427 mm below the core central plane. The upper suspension contains eight ampoules 7-10 at levels 1, 2, 3, and 4 (2 ampoules at each level). At every level a fluence monitor is embedded in suspension. In the upper part of the testing assembly two temperature monitors are maintained. The fluence monitor consists of 3-mm diameter quartz ampoule with 93 Nb and 54 Fe isotopes inside. The ampoule is covered with aluminium foil. About 1.7 mm diameter inner hole of the ampoule is filled with cubic lattice SiC powder. The temperature monitor ampoules are also covered with aluminium foil.

In order to determine the samples irradiation temperature as well as the rate of coolant flow through the material



Fig. 1. Ampoule details and specimen before assembly.



Fig. 3. Irradiation assembly.

testing assembly thermal hydraulics calculations were performed.

#### 2.1.3. Tensile test load-inducing mechanism

A special tensile test load-inducing mechanism has been designed and fabricated as a structural part of the in-cell test facility, to be operated on irradiation ampoules. The load-inducing mechanism is designed as an electromechanical drive, which provides a tensile test with a desired rate of specimen elongation.

The principle scheme, which illustrates the load-inducing mechanism action, is shown in Fig. 4. The mechanism consists of

- (1) the electric motor 1
- (2) the force-increasing gearbox 2 transmitting rotation to the worm screw 3
- (3) the worm wheel 4, forming a worm-gear with the screw 3, tightly connected with the tubular nut 5, which is forming a motion screw pair with the screw 6.

The tensile load gauge 9 is a special device, which contains several springy elements with tensiometric resistors



Fig. 4. The load-inducing mechanism.

connected to them. The clamp system 10 is intended for joining the active draw to upper element of the specimen's assembly (the position 7 in figure), which is designed as an extension plug of a tested specimen upper head. Data are acquired and processed with an electronic system.

## 2.1.4. Specimens irradiation

According to neutron-physical calculations, the neutron fluence for the specimens of level from 0 to 4 reached the dose 0.26; 4.0; 5.5; 5.9 and 5.3 dpa, respectively. Irradiation parameters of the specimens, presented in Table 1, were stable during irradiation period.

The temperature distribution along the material test assembly was in the range 310-335 °C. During the experiment an average power of about 51 MW was determined. In the same time, the real inlet temperature of the reactor coolant was equal to calculated one, 310 °C.

# 2.1.5. Post irradiation tests

After irradiation the material testing assembly was extracted from the reactor.

Table 1									
Irradiation	parameters	in	channel	E41	of	BOR	60	reactor	

Ampoule level number	Distance from the core central plane (mm)	dpa	Temperature (°C)		
4	109	5.3	332		
3	3	5.9	331		
2	-103	5.5	330		
1	-209	4.0	323		
0	-652	0.26	311		

Some standard procedures, such as washing of the assembly from sodium coolant and decontamination, were performed. The assembly was opened and the inner suspension with irradiated specimens was transported to the material test hot laboratory.

The low strain tensile tests of irradiated samples were carried out in liquid metal environment. Tests have been performed at the calculated irradiation temperature of samples. The strain rate was  $1 \times 10^{-4} \text{ s}^{-1}$ .

Calculation of mechanical characteristics of irradiated samples were carried out according to the standard procedure with test diagrams. All calculations were performed for the mean diameter of the sample effective part.

## 3. Results and discussion

In Table 2 are summarised the results in terms of yield stress, ultimate stress and uniform elongation.

An increase in the Yield and Ultimate stress (YS and UTS) is observed passing from 0.26 to 4.4 dpa, with a slight decrease to higher doses (Fig. 5). The plastic properties level of irradiated samples remained sufficiently high: 9.7-14.0% for the total elongation and 0.7-1.1% for the uniform elongation (Fig. 6). Literature results referred to in gas irradiated martensitic steels generally agreed on the fact that the YS and UTS values vary not monotonically with dose with a very rapid increase up to 10 dpa. These assumption is also confirmed in [4]. Even the minimum value of uniform elongation is generally reached for doses lower than 10 dpa, than remaining constant. In the present experiment the uniform elongation is constant for doses higher than 4.4 dpa, while no clear effects appears on the total elongation. The increase in the tensile strength is not in direct relation with the decrease in the total elongation. In any case an increase in the Yield strength is accompanied by a decrease in the total elongation. This phenomenon is well evidenced in the Fig. 7.

Comparing the YS at 0.26 dpa with the one presented in [5] at 300 °C and 0.35 dpa the value is almost the same. This result seems to confirm that the liquid metal embrittlement effect (LME) is not additive to the irradiation effect. Even considering the YS at 6 dpa extrapolated

Table 2 Experimental results of mechanical characteristics



Fig. 5. YS and UTS as a function of irradiation dose.



Fig. 6. Uniform and total elongation as a function of the irradiation dose.

using the curves in [4] at the same temperature the value is similar to the one obtained in this experiment. This is in good agreement with the results presented in [2] and referred to unirradiated F82H steel. In that case it was assessed that no LME was present in the temperature range 250–400 °C in case of tempered fully martensitic state of the steel. Experiments on unirradiated Eurofer presented in [3], performed at 480 °C in lead lithium, confirmed a similar behaviour of this material in terms of LME.

Level	Ampoule	Sample	Dose (dpa)	In pile T (°C)	$\sigma_{\rm R}$ (MPa)	$\sigma_{0.2}$ (MPa)	Total $\varepsilon$ (%)	Uniform $\varepsilon$ (%)
4	9	18	5.3	330	690	662	14.0	1.1
	8	17			700	677	13.0	0.9
3	7	10	5.9	330	715	680	13.3	0.9
	6	9			737	697	11.6	0.7
2	5	8	5.0	330	712	678	11.8	0.8
	4	7			720	658	10.8	1.1
1	3	6	4.4	323	743	726	9.7	0.8
	1	4			730	707	10.4	0.8
0	1	19	0.26	315	600	559	16.0	1.9
	2	5			606	548	15.9	1.9



Fig. 7. Yeld strength versus total elongation.

## 4. Remarks

Eurofer 97 tensile specimens were irradiated and tested in lead lithium at about 330 °C. The obtained results in terms of YS and UTS are aligned to the ones available in literature. This testifies that irradiation in the liquid alloy has not most likely resulted in liquid-metal embrittlement (LME) of the steel, and radiation hardening made the main contribution to mechanical characteristics change. This is in good agreement with out of pile experiments of LME performed on martensitic steels with tempered fully martensitic state. A larger PIE campaign, including SEM analysis, will be in any case necessary to exclude the LME effect. However, to fully understand the effect of lead lithium it could be useful to explore the behavior of the material at higher doses and different temperatures.

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