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Effect of austenitisation and tempering temperatures on mechanical properties of EUROFER 97 steel

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Abstract. In Europe EUROFER 97 has been recognised as reference steel for the nuclear costructions under high radiation density for first wall of a fast breeder reactors as well as in other high stressed primary structures such as the divertors, blanklet and vessels. Following to this a EUROFER 97 detailed knowledge of the microstructure evolution after thermo-mechanical processing is required. In this paper the effect of thermo-mechanical parameters on the mechanical behavior of EUROFER 97 has been investigated by hot rolling and heat treatment on pilot scale.

Results show a strong effect of reheating temperature before rolling on the material hardness, due to an increase of hardenability following the austenite grain growth. A poor effect of the hot reduction and of the following tempering temperature is detected in the total deformation-range investigated, 30 to 40%. A loss of impact energy is found coupled with the hardness increase.

The tensile properties values are strongly depending upon the tempering temperature and an increase of tensile yield stress (YS) and ultimate stress (UTS) have been recorded in tensile test carried on at T=550°C and T=650°C. In detail, as an exemplum: YS increase from about 400 MPa for standard EUROFER 97 [1] to about 550 MPa in samples adopting a tempering temperature of 720°C instead of the standard 760°C for EUROFER 97. Same trend of improvement has been recorded for UTS results comparison at testing temperature of T=650°C.

Keywords: EUROFER 97, microstructure, heat treatment

Introduction

The nuclear constructions have been facing with the problem of induced brittleness in structural materials exposed to the radiation damage. In Europe EUROFER 97 steel has been recognised as one of the best balance for radiation damage tolerance and mechanical properties [1] in nuclear costructions.

EUROFER 97 steel is adopted as reference steel for many structural part of the reactors, i.e.: wall of fast breeder reactors as well as in other high stressed primary structures such as the divertors, blanklet and vessels [2-4]. The main reason for this adoption is based on the EUROFER steel high mechanical properties at service temperatures coupled with the low or reduced activation (RAFM) characteristic under radiation, with the final result of low mechanical properties loss[1-5]. This material behavior has been reported in many literature studies and important initiatives are still ongoing [1, 5]. The reduced activation ferritic/martensitic steels differ from conventional Cr-Mo steels because of W presence instead of Mo. With this respect EUROFER 97 steel is essentially a low carbon steel with 9 Cr (% wt) with controlled Ta and V content that can have a important influence on resulting final mechanical properties especially for creep properties, [5-7].

This paper describes microstructure evolution correlated with Charpy-V notch impact test (CV-N) results and tensile tests results up to 650°C of the EUROFER 97 steel. These results are obtained modifying the tempering temperature, carried on at T=750°C and 720°C, with respect to the standard tempering condition for EUROFER 97 (tempering temperature at 760°C [1]).

EUROFER 97 reference chemical composition is reported in Table 1.

Table 1. EUROFER 97 chemical composition (wt, %)
(Ni, Mo, Cu, Nb, Al, B, Co: as low as possible (ALAP)

Fe	C	Mn	Cr	V	Ta	W	N	As+Sn+Sb+Zr
Balance	0.11	0.4	9.0	0.2	0.07	1.0-2.0	0.030	ALAP, 0,05

Moreover, other elements such us Mo, Nb, Ni, Cu and N, are maintained as low as possible. The irradiation tests carried on EUROFER 97 show that the resulting radioactivity levels over two orders of magnitude under those recorded for conventional Cr steels [8], with low affected mechanical and physical properties [9-10]. Low activation steels have a fully austenite structure when are austenitized in the temperature range from 850°C to 1200°C. Austenite phase transforms to martensite phase during air cooling or rapid cooling (quenching) to room temperature, and then steels are tempered to obtain a good combination of strength, ductility, and toughness. However, the use of these materials during long-time at high temperatures (thermal ageing) can produce microstructural changes (new precipitates, grain growth, segregation, etc.) which can significantly affect their mechanical properties (tensile, Charpy-V, fracture toughness, low cycle fatigue, etc.). For these reasons, an exhaustive knowledge of the metallurgical characteristics of these steels before and after thermal ageing is considered essential.

In RAFM steels the desirable properties (low sensibility to radiation damage) are controlled by mean of the martensitic transformation thermal cycle design, and in particular are due the microstructure refinement (increase of the low and high angle boundaries) with clear advantages for applications in nuclear reactors [10]. The martensitic transformation occurs in steels by mean of a non-diffusional transformation when the material is cooled from above A_{c1} to a sufficiently lower temperature (M_s) with cooling rate higher than the “critical cooling-rate”: in these condition the transformation is lead from the energy decrease due to the metastable face-centered cubic (FCC) phase arrangement in the new stable body-centered cubic (BCC) phase [2]. The conventional EUROFER 97 thermal treatment consists in normalization at 980°C/30 minutes + temper at 760°C/90'/air-cooling, [1]. In this work the effect of thermo-mechanical treatment on the microstructure is analyzed, aimed to achieve higher tensile properties in order to evaluate its feasibility as possible structural material for fusion applications.

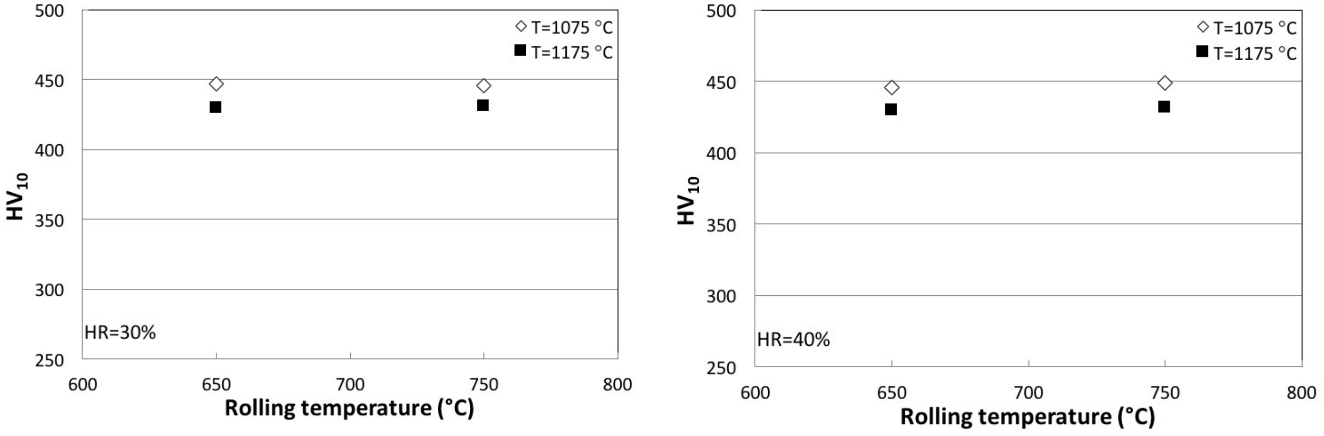
Experimental

Starting from a EUROFER 97 rolled plate with the steel chemical composition reported in Table 1, the effect of reheating temperatures (before hot rolling) and rolling temperatures is analyzed. The plate was hot rolled on a pilot scale adopting two different reheating temperatures (1075°C and 1175°C), together with two finish rolling temperatures (750°C and 650 °C) and two different total reductions (30% and 40%). The effect of tempering treatment after hot rolling is also analyzed (in the temperature range 720°C-760 °C). Hardness, Charpy-V impact tests at -20 °C and tensile tests have been carried out. The charpy-V notch have been carried on transverse full size specimens according to ASTM E23 and A263 standard. Microstructure is analyzed by light microscopy after Vilella etching.

The tensile tests have bene carried on using ASTM E21 standard at room temperature and two reference temperatures useful for a wide range of applications: T=550°C and T=650°C, Figure. 3.

Results

A limited effect is found following to the variation of rolling temperature, reheating temperature and reduction in the considered range (Figure 1).



A b

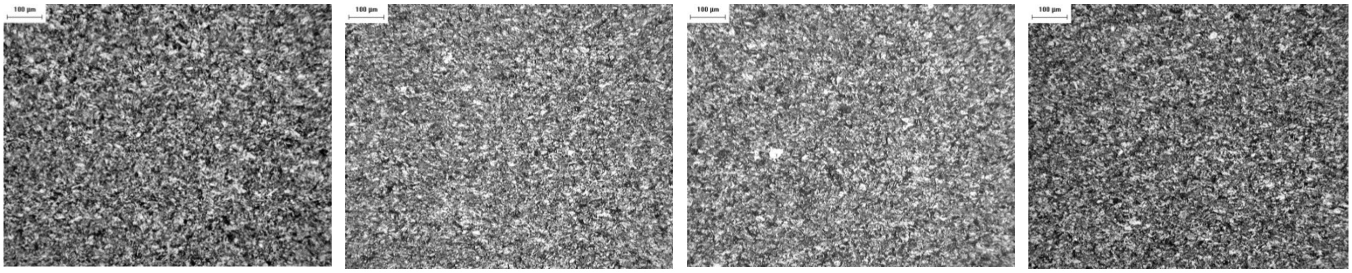
Figure 1. Effect of thermo-mechanical parameters on EUROFER 97 hardness (a: 30% hot reduction, b: 40% hot reduction)

The effect of tempering following the hot rolling as a function of thermo-mechanical parameters is reported in Table 2.

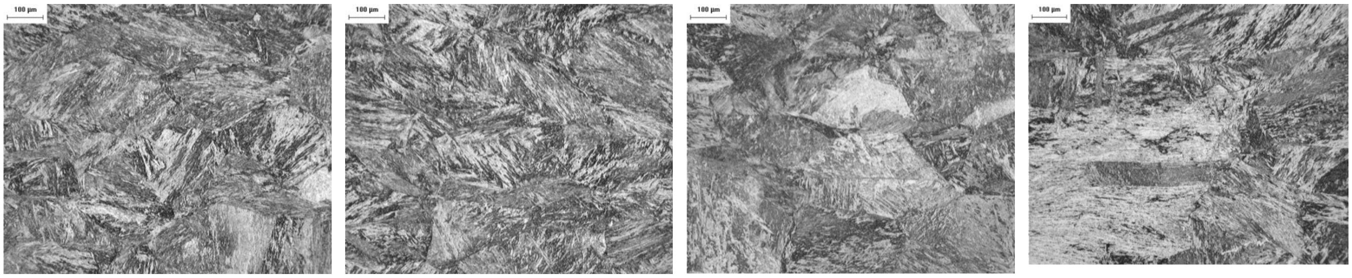
Table 2. Effect of tempering after hot rolling

Specimen n.	Reheating T (°C)	Rolling T (°C)	Hot reduction, (%)	Tempering T (°C)	
				720	760
				HV_{10}	HV_{10}
1	1075	750	30	278	225
2			40	267	225
3		650	30	271	228
4			40	270	234
5	1175	750	30	284	251
6			40	290	246
7		650	30	298	254
8			40	306	259

Results show that higher hardness values are found after re-heating at higher temperature (1175°C). This is due to an improvement of hardenability following an increase of austenite grain size. In Figure 2 the microstructure evolution is reported for specimens 1-8 after tempering at T=720 °C. Results show a clear effect of reheating temperature on austenite grain growth. The same effect is independent and effective also in the case of specimens after tempering at T=760 °C.



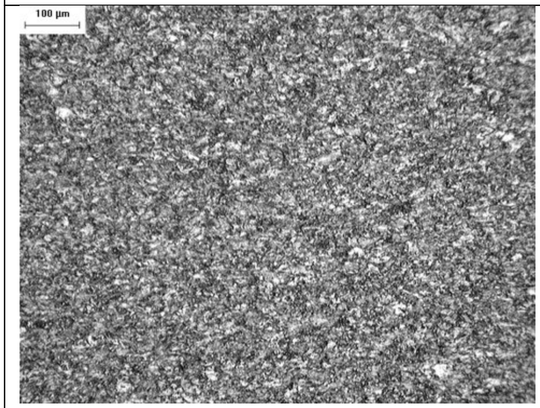
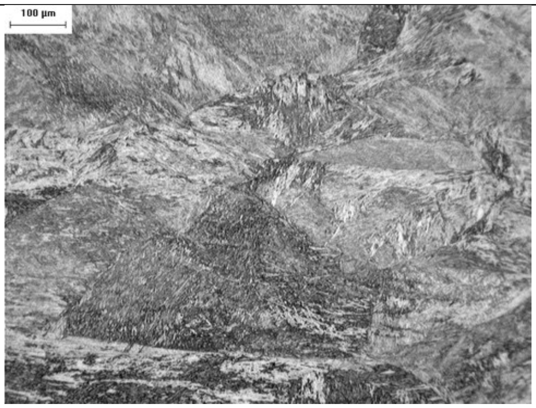
Specimen n.1 Specimen n.2 Specimen n.3 Specimen n.4



Specimen n.5 Specimen n.6 Specimen n.7 Specimen n.8

Figure 2. Microstructure evolution of EUROFER 97 after thermo-mechanical processing according to Table 2, all markers show 100 microns.

Table 3: Effect of reheating temperature on Charpy-V notch toughness

<table>
<tr><td colspan="2">Reheating T=1075 °C</td><td colspan="2">Reheating T=1175 °C</td></tr>
<tr><td colspan="2">Specimen 2</td><td colspan="2">Specimen 8</td></tr>
<tr><td colspan="2"></td><td colspan="2"></td></tr>
<tr><td colspan="2">HV$_{10}$=267</td><td colspan="2">HV$_{10}$=306</td></tr>
<tr><td colspan="2">CVN - full size specimen
test temperature = -20°C</td><td colspan="2">CVN - full size specimen
test temperature = -20°C</td></tr>
<tr><td>mean Value (J) (three tests)</td><td>Dispersion (J)</td><td>Mean Value (J) (three tests)</td><td>Dispersion (J)</td></tr>
<tr><td>63</td><td>+/- 15</td><td>9</td><td>+/- 2</td></tr>
<tr><td colspan="2">Fracture appearance=100% ductile</td><td colspan="2">Fracture appearance=100% brittle</td></tr>
</table>

At the same time larger austenitic grain size (due to higher austenitization temperature) leads to a dramatic decrease of impact toughness behavior. The effect of austenite grain size growth following an increase of reheating temperature on CVN is reported in Table 3.

Moreover, tensile tests have been carried on at T=550°C and 650°C. In order to point out any macro-difference in tensile properties two samples (specimen 2 and 8) obtained with different process condition, but same hot reduction, have been selected, Table 2.
The tensile results carried on at room temperature and T=550°C and T=650°C are reported in Table 4 and Figure 3. A summary of experimental tensile tests superimposed for comparison with the EUROFER 97 standard tensile properties obtained from the literature, [1] are shown in figure 3.

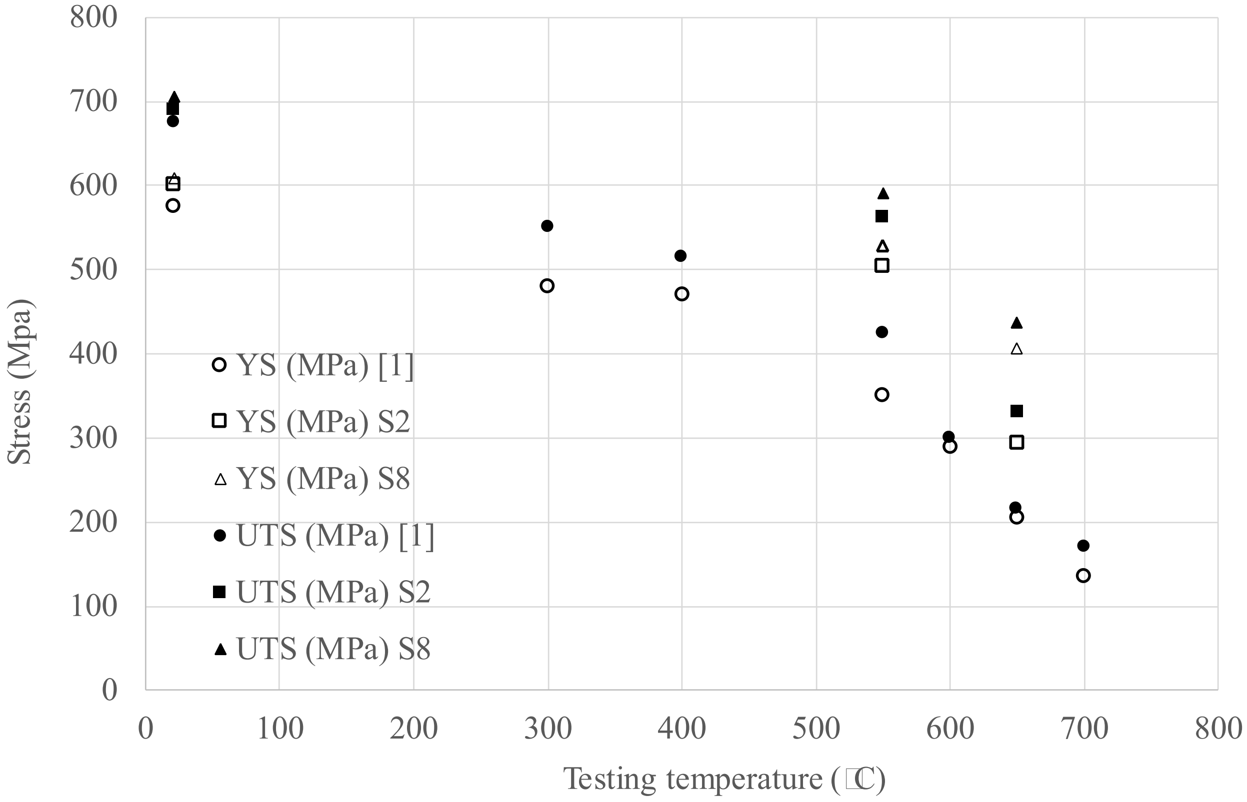


Figure 3: EUROFER 97 tensile properties comparison: experimental vs literature data [1], (void bullets represent YS, bold bullets represent UTS)

Table 4: EUROFER 97: correlation between process condition, hardness and tensile tests

Process					**Tensile tests**								
					Room T			T=550°C			T=650°C		
S	**T_{RH}**	**R_{TR}**	**T_R**	**T_T**	**YS**	**UTS**	**A**	**YS**	**UTS**	**A**	**YS**	**UTS**	**A**
#	°C	(%)	°C	°C	MPa	MPa	(%)	MPa	MPa	(%)	MPa	MPa	(%)
2	1075	40	750	720	602	691	21	504	528	22	294	331	24
8	1175	40	650	720	608	706	20	407	438	18	563	591	18

S: specimen; T_{RH}: Reheating T; R_{TR}: rolling thickness reduction; T_R: Rolling T; $T_{T:}$ tempering T

Conclusions

The effect of thermo-mechanical parameters on the mechanical behavior of EUROFER 97 has been investigated by hot rolling and tempering heat treatment on pilot scale. Results show a strong effect of reheating temperature before rolling on the material hardness, due to an increase of hardenability following the austenite grain growth. A poor effect of the hot reduction and of the following tempering temperature is detected in the total thickness reduction range: 30-40%. A dramatic loss of CV-N impact energy is found coupled with the hardness increase when the reheating temperature from 1075°C is increased up to 1175°C, Table 3. All the results obtained with a tempering temperature lowered at T=720°C show important Charpy V-notch impact energy and tensile strength improvements with respect to the standard EUROFER 97 literature best results. In particular the samples rolled at T=750°C and tempered at T=720°C maintain an interesting mechanical behavior: enough CV-N toughness at T= -20°C and at least 20% of tensile strength properties increase at T=650°C, Table 3 and Figure 3.

In conclusion EUROFER 97 is a high sensitive material to the thermo-mechanical process and thermal post process cycle to be carefully controlled in order to avoid any potential negative result on final properties.

Acknowledgement

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