Materials Science Forum
ISSN: 1662-9752, Vol. 1105, pp 47-52
doi:10.4028/p-t3IMA9

Submitted: 2023-05-10
Accepted: 2023-08-31
Online: 2023-11-29

Assessment of Mechanical Properties and Microstructure of EUROFER97 Steel after Thermo-Mechanical Treatments

Giulia Stornelli[1,a*], Andrea Di Schino[2,b], Roberto Montanari[1,c], Claudio Testani[3,d] and Alessandra Varone[1,e]

[1]Dipartimento di Ingegneria Industriale, Università di Roma "Tor Vergata", 00133 Rome, Italy.

[2]Dipartimento di Ingegneria, Università di Perugia, Via G. Duranti 93, 06125 Perugia, Italy.

[3]CALEF-ENEA CR-Casaccia, Via Anguillarese 301, S. Maria di Galeria, 00123 Rome, Italy

[a]giulia.stornelli@students.uniroma2.eu, [b]andrea.dischino@unipg.it, [c]roberto.montanari@uniroma2.it, [d]claudio.testani@consorziocalef.it, [e]alessandra.varone@uniroma2.it

Keywords: EUROFER97; RAFM steels; Thermo-Mechanical Treatments; Microstructure; Dislocation density; Recrystallization, Mechanical properties.

Abstract. EUROFER97 martensitic steel is recognized in EU as the reference material for the test blanket module in ITER reactor and for structural sections subject to high radiation doses in DEMO reactor. An extended experimental campaign has been carried out with the scope of improving strength without loss of ductility. The main idea behind the present study is to reach the goal through grain refinement achieved by cold rolling and heat treatments for inducing recrystallization of the work hardened structure. A combination of five cold rolling reduction ratios (CR) (20%, 40%, 50%, 60%, 80%) and eight heat treatments in the temperature range 400-750°C (steps of 50 °C) with soaking time of 1 hour has been examined to describe the evolution of microstructure and mechanical properties. The strength of deformed samples decreases as the heat treatment temperature increases and the change is more pronounced in the samples cold-rolled with higher CR ratios. The results showed that cold rolling with CR of 80% followed by a treatment at 650 °C produces a fully recrystallized structure with sub-micrometric grains which guarantees improved yield stress and hardness than standard EUROFER97 steel, with a comparable total elongation. In conclusion, this work demonstrated the feasibility to strengthen EUROFER97 without compromising its ductility.

Introduction

Reduced Activation Ferritic-Martensitic (RAFM) steels were developed as an alternative to traditional ferritic-martensitic steels for applications as structural materials in upcoming nuclear fusion reactors and Generation IV nuclear fission reactors [1,2]. EUROFER97 is the reference RAFM steel used in Europe for Test Blanket Modules (TBMs) in the ITER reactor [3,4] as well as a candidate for the first wall in DEMO reactor and in other reactor sections such as blanket, vessel and divertor [5,6]. The composition of EUROFER97 was developed to meet the requirements of reduced activation: in particular, some chemical elements, typically used in the commercial Cr-Mo ferritic-martensitic steels [7-11], were replaced with metallurgically equivalent elements, characterized by shorter radioactive decay times. More in detail, Mo was replaced with W and Nb with Ta and V. Similarly, the concentration of Ni, Cu, Al, Co and other impurity elements, with high long-term radioactivity, was kept as low as possible [12,13].
Plates of EUROFER97 steel are usually produced through a hot-rolling process followed by three heat treatment steps: (1) austenitization at 980 °C for 30 minutes, (2) air-cooling and (3) tempering at 760 °C for 90 minutes [1]. At the end of this process a tempered lath martensite microstructure is formed. Under irradiation EUROFER97 steel has good mechanical performances in the temperature range between 350 °C and 550 °C [14], and several studies have been performed with the aim to extend the operating temperature range. Accordingly, this implies the reduction of the lower operational temperature (below 350 °C) that depends on the embrittlement that the material undergoes

once irradiated at low temperatures, as well as on the excessive loss of toughness [7]. To reach the scope, a possible strategy consists in refining on a nanoscale the grain structure of EUROFER97.
In literature, the effect of different thermo-mechanical treatments on the irradiation resistance of EUROFER97 steel have been reported and, various strategies have been evaluated to refine its microstructure (e.g., [6,15–18]) with beneficial improvement on irradiation behavior. The goal of this work is to develop a thermo-mechanical treatment to refine the microstructure, and strengthen the material without causing a loss of ductility.

Materials and Methods

The nominal chemical composition for EUROFER97 steel is shown in Table 1 [1].

Table 1. Nominal chemical composition of EUROFER97 steel (wt. %) (Ni, Mo, Cu, Nb, Al, B, Co: as low as possible (ALAP), S = 20 ppm, P = 100 ppm, Fe to balance) [1].

Cr	C	Mn	V	W	Ta	As + Sn + Sb + Zr
9.00	0.11	0.40	0.20	1.07	0.07	< 0.05

EUROFER97 in standard condition was cold rolled and heat treated by combining five cold reduction (CR) ratios (20%, 40%, 50%, 60%, 80%) and eight heat treatments at different temperatures in the range 400-750 °C (steps of 50 °C) for 1 hour (see Figure 1). The heat treatment temperatures were chosen to operate in the ferritic field [16,19].

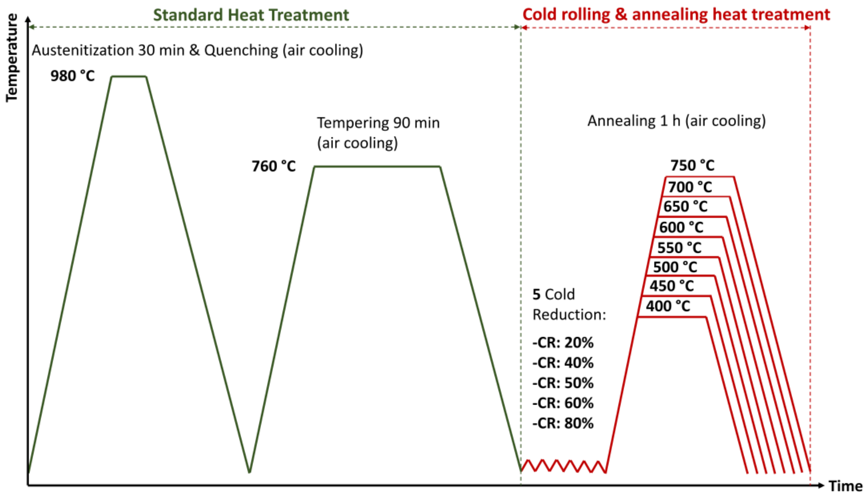


Fig 1. Scheme of the complete thermo-mechanical treatments.

The microstructure of the samples was examined by high-resolution electron scanning microscopy (FE-SEM- Zeiss, Gemini Supra 25, Jena, Germany) equipped with an EBSD detector (C Nano Oxford Instruments), using a 0.02 μm scanning step size and X-ray diffraction (XRD- PW 1729, Philips, Eindhoven, The Netherlands). SEM observations were carried out after mechanical polishing of the sample surface and etching by means of Vilella reagent. XRD patterns were collected in the 2θ angular range 15–60 ° by using the Mo-Kα radiation (λ = 0.070926 nm) with 2θ steps of 0.005 °and counting time per step of 4 s. After background subtraction the peak profiles were fitted by Lorentzian curves to eliminate the Kα2 component, then the full width at half maximum (FWHM) was corrected from the instrumental broadening to get the total broadening (β_T) that is the sum of two contributions due to the size (D) of coherently diffracting domains (β_D) and the micro-strains (β_ε) induced by dislocations:

$$\beta_T = \beta_D + \beta_\varepsilon = \frac{0.89\,\lambda}{D\cos\theta} + 2\varepsilon\tan\theta \tag{1}$$

where θ is the Bragg's angle of the XRD peak. The dislocation density ρ was then calculated through the Williamson–Smallman relationship:

$$\rho = \frac{\Xi\,\varepsilon^2}{Fb^2} \tag{2}$$

where Ξ = 16.1 is a constant, F ≈ 1 is a factor depending on the interaction of dislocations and b = 0.248 nm is the modulus of Burgers vector [20].
The mechanical properties were investigated through Vickers hardness tests (load of 5 kg) and tensile tests.

Results

The hardness values of the material after the examined thermo-mechanical treatments are reported in Table 2. Except for the sample deformed with CR: 20%, there is a critical treatment temperature above which hardness drops below the standard value: it is 650 °C for the samples deformed with CR of 60 % and 80 %, and 700 °C for lower CR (40% and 50%).

Table 2. Vickers hardness HV_5 after thermo-mechanical treatments. Green and red cells indicate the values above and below the reference one of standard EUROFER97 (yellow cell), respectively.

CR / Temperatures	20 %	40 %	50 %	60 %	80 %	Standard EUROFER97
N.T.	228.1±1.2	257.4±3.3	266.1±3.2	275.2±1.8	298.9±1.1	201.6 ± 1.9
400 °C	227.6±1.9	249.8±2.9	258.1±2.5	269.5±0.8	290.4±1.6	
450 °C	223.8±1.9	243.5±2.1	257.6±2.5	263.9±0.3	290.8±1.9	
500 °C	222.8±0.5	241.4±1.4	252.1±2.1	261.2±1.2	284.6±0.7	
550 °C	220.5±1.3	240.1±2.5	249.2±0.3	259.0±0.9	280.4±2.5	
600 °C	220.6±1.2	233.8±1.9	242.4±0.1	249.5±0.7	253.1±0.9	
650 °C	212.0±1.9	224.6±0.9	232.7±0.4	238.9±2.7	231.3±1.1	
700 °C	211.1±1.2	221.5±0.1	217.6±2.4	156.7±0.9	153.1±0.6	
750 °C	203.4±0.7	130.0±0.5	131.2±0.2	129.1±0.7	137.1±0.9	

Since EUROFER97 deformed with CR of 80% exhibits the greatest hardness, some samples heat-treated at temperatures ranging from 400 °C to 650 °C (green cells in Table 2) were submitted to tensile tests (see Table 3). As expected, ultimate tensile stress (UTS) and yield stress (YS) decrease with increasing treatment temperature, while the total elongation (A%) exhibits the opposite trend. In comparison to the values of standard EUROFER97 (yellow cells in Table 3) UTS and YS of samples heat treated at temperatures up to 600 °C are always higher but their total elongation A% is lower. The treatment at 650 °C (green cells in Table 3) guarantees the best compromise: YS and UTS values result significantly increased with respect to those of standard EUROFER97 of 12% and 5%, respectively, with a comparable total elongation of about 22%.

Table 3. Ultimate tensile strength (UTS), yield strength (YS) and elongation (A%) of EUROFER97 steel with 80% cold reduction and heat treatment at 400°C, 500°C, 600°C, and 650°C for 1 hour. The values of standard EUROFER97 steel [21] are reported for comparison. The values highlighted in green indicate the best thermo-mechanical treatment condition.

	YS (MPa)	**UTS (MPa)**	**A (%)**
Standard EUROFER97	575 [21]	680 [21]	22.1 [21]
CR: 80% heat treated at 400 °C	908	932	10.3
CR: 80% heat treated at 500 °C	847	878	12.2
CR: 80% heat treated at 600 °C	731	760	15.6
CR: 80% heat treated at 650 °C	641	689	22.3

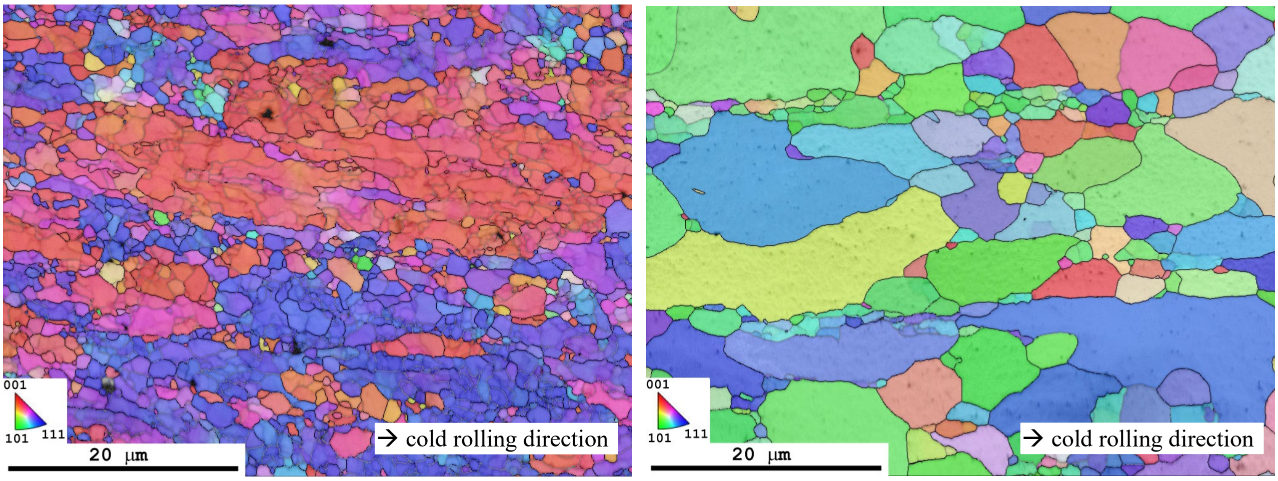
Table 4 reports microstructural information obtained from XRD, i.e. the mean grain size D and dislocation density ρ. The increase of treatment temperature leads to a gradual reduction of dislocation density, which results from the formation of new grains during recrystallization. Similarly, the average grain size follows a comparable trend up to 650 °C because the heavily deformed matrix of the steel has a large number of preferred nucleation sites for new grains, however, above 650 °C, grain growth occurs leading to a progressive increase of the average size.

Table 4. Mean grain size D and dislocation density ρ of EUROFER97 cold rolled with CR of 80% and heat treated at temperatures in the range 400-750 °C.

Heat treatment temperature (°C)	**Mean grain size D (nm)**	**Dislocation density ρ (cm^{-2})**
Not treated	400	1.20×10^{12}
400	380	5.00×10^{11}
450	330	2.90×10^{11}
500	275	2.70×10^{11}
550	260	2.70×10^{11}
600	250	1.30×10^{10}
650	210	9.80×10^{8}
700	218	3.90×10^{8}
750	350	3.10×10^{8}

The grain size trend vs. treatment temperature is confirmed by the EBSD maps displayed in Figure 2 (a-b). After the treatment at 650 °C (Figure 2 a) the steel has an extremely fine microstructure of equiaxed grains whereas the mean grain size is larger after heating at 750 °C and abnormal grain growth is also observed (Figure 2 b). Some grains, aligned along the rolling direction, display a size of about 30-40 µm while that of neighbour ones is ~ 350 nm. The increase of mean grain size and abnormal grain growth lead to the degradation of mechanical properties reported in Tables 2 and 3, below the values of standard EUROFER97.

In conclusion, the ideal microstructure consists of equiaxed grains with small and homogeneous size, therefore the best mechanical properties are achieved at the completion of recrystallization before the onset of grain growth. The thermo-mechanical treatment with CR of 80% and heating at 650 °C produces the desired microstructure with grains of ~ 200 nm and leads to better mechanical properties than those of standard EUROFER97 with the same ductility.



(a) (b)

Fig. 2. EBSD maps of EUROFER97 deformed with CR of 80 % and heat treated for 1 hour at 650 °C (a) and 750 °C (b).

Conclusions

Forty groups of EUROFER97 samples were prepared by combining cold rolling with five different CR (20%, 40%, 50%, 60%, 80%) and heat treatments at eight different temperatures ranging from 400-750 °C for 1 hour. The results can be summarized as follows.

1- Recrystallization occurs in cold rolled and heat-treated samples, with kinetics dependent on the treatment temperature and cold rolling ratio.
2- Hardness decreases with increasing heat treatment temperature, and this effect is more pronounced in samples with higher cold rolling ratios.
3- Equiaxed grains of sub-micrometric size were observed in samples with a cold rolling ratio of 80% at 650°C, indicating the completion of primary recrystallization.
4- Abnormal grain growth was observed, for the same CR: 80%, at higher treatment temperatures (750 °C).
5- Compared to standard EUROFER97, the treatment at 650°C increases YS and UTS of about 12% and 5%, respectively, while total elongation remains almost the same (~22%).

This study demonstrated the possibility of strengthening EUROFER97 steel without compromising its ductility, which is a promising finding for nuclear fusion applications.

References

[1] M. Rieth, M. Schirra, A. Falkenstein, P. Graf, S. Heger, H. Kempe, R. Lindau, H. Zimmermann, EUROFER 97 Tensile, Charpy, Creep and Structural Tests. Report FZKA6911, Eurofusion programme, 2003.

[2] K.D. Zilnyk, V.B. Oliveira, H.R.Z. Sandim, A. Möslang, D. Raabe, Martensitic transformation in Eurofer-97 and ODS-Eurofer steels: A comparative study, Journal of Nuclear Materials, 462 (2015) 360–367.

[3] J. Hoffmann, M. Rieth, M. Klimenkov, S. Baumgärtner, Improvement of EUROFER's mechanical properties by optimized chemical compositions and thermo-mechanical treatments, Nuclear Materials and Energy, 16 (2018) 88–94.

[4] G. Stornelli, M. Rallini, C. Testani, R. Montanari, A. Di Schino, G. Stornelli, R. Montanari, C. Testani, A. Di Schino, Effect of thermo-mechanical treatment on EUROFER 97 steel for nuclear fusion application, La Metallurgia Italiana, 112(10) (2020) 34–44.

[5] X. Chen, A. Bhattacharya, M.A. Sokolov, L.N. Clowers, Y. Yamamoto, T. Graening, K.D. Linton, Y. Katoh, M. Rieth, Mechanical properties and microstructure characterization of Eurofer97 steel variants in EUROfusion program, Fusion Engineering and Design, 146 (2019) 2227–2232.

[6] G. Stornelli, R. Montanari, C. Testani, L. Pilloni, G. Napoli, O. Di Pietro, A. Di Schino, Microstructure refinement effect on EUROFER 97 steel for nuclear fusion application, Materials Science Forum, 1016 MSF (2021) 1392–1397.

[7] S.J. Zinkle, A. Möslang, Evaluation of irradiation facility options for fusion materials research and development, in: Fusion Engineering and Design, 2013: pp. 472–482.

[8] A. Di Schino, C. Testani, L. Pilloni, Effect of thermo-mechanical parameters on the mechanical properties of Eurofer97 steel for nuclear applications, Open Engineering, 8 (2018) 349–353.

[9] G. Stornelli, M. Gaggiotti, D.M. Gattia, R. Schmidt, M. Sgambetterra, A. Tselikova, G. Zucca, A. Di Schino, Vanadium alloying in s355 structural steel: effect on residual austenite formation in welded joints heat affected zone, Acta Metallurgica Slovaca, 28(3) (2022) 127–132.

[10] G. Stornelli, A. Tselikova, D. Mirabile Gattia, M. Mortello, R. Schmidt, M. Sgambetterra, C. Testani, G. Zucca, A. Di Schino, Influence of Vanadium micro-alloying on the microstructure of structural high strength steels welded joints, Materials, 16(7) (2023) 2897

[11] A. Di Schino, M. Gaggiotti, D. Mirabile Gattia, R. Schmidt, M. Sgambetterra, G. Stornelli, C. Testani, A. Tselikova, G. Zucca, Vanadium micro-alloying effect on heat affected zone microstructure in welded joints for structural applications, La Metallurgia Italiana, 113 (2022) 8–14.

[12] A.A.F. Tavassoli, E. Diegele, R. Lindau, N. Luzginova, H. Tanigawa, Current status and recent research achievements in ferritic/martensitic steels, Journal of Nuclear Materials, 455 (2014) 269–276.

[13] K. Mergia, N. Boukos, Structural, thermal, electrical and magnetic properties of Eurofer 97 steel, Journal of Nuclear Materials, 373 (2008) 1–8. https://doi.org/10.1016/j.jnucmat.2007.03.267.

[14] R. Coppola, M. Klimenkov, Dose dependence of micro-voids distributions in low-temperature neutron irradiated Eurofer97 steel, Metals (Basel), 9 (2019) 1–12.

[15] C. Cristalli, L. Pilloni, O. Tassa, L. Bozzetto, Mechanical properties of several newly produced RAFM steels with Tungsten content in the range of 2 wt%, Nuclear Materials and Energy, 25 (2020) 100793.

[16] V.B. Oliveira, H.R.Z. Sandim, D. Raabe, Abnormal grain growth in Eurofer-97 steel in the ferrite phase field, Journal of Nuclear Materials, 485 (2017) 23–38.

[17] L. Pilloni, C. Cristalli, O. Tassa, I. Salvatori, S. Storai, Grain size reduction strategies on Eurofer, Nuclear Materials and Energy, 17 (2018) 129–136.

[18] G. Stornelli, A. Di Schino, S. Mancini, R. Montanari, C. Testani, A. Varone, Grain refinement and improved mechanical properties of eurofer97 by thermo-mechanical treatments, Applied Sciences (Switzerland), 11 (2021) 10598.

[19] S. Mancini, L. Langellotto, P.E. Di Nunzio, C. Zitelli, A. Di Schino, Defect reduction and quality optimization by modeling plastic deformation and metallurgical evolution in ferritic stainless steels, Metals (Basel), 10(2) (2020) 186.

[20] R.E.; Reed-Hill, R. Abbaschian, L. Abbaschian, Physical Metallurgy Principles, 1973.

[21] E. Materna-Morris, H.C. Schneider, A. Möslang, Tensile behavior of RAFM alloys after neutron irradiation of up to 16.3 dpa between 250 and 450 °C, Journal of Nuclear Materials, 455 (2014) 728–734.