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Structure and properties of nano-sized Eurofer 97 steel obtained by hydrostatic extrusion

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A B S T R A C T

The objective of the present work was to apply hydrostatic extrusion to Eurofer 97 steel – a fusion relevant material that can be used for structural application in ITER. Samples were hydrostatically extruded in a multi-step process with a total true strain of about 4. Microstructure observations via light and transmission electron microscopy revealed significant grain size refinement from about 400 nm to 90 nm and changes in carbide size and distribution. Microstructure changes resulted in substantial improvement in mechanical properties of the material (the ultimate tensile strength and yield stress increase from 691 MPa to 1769 MPa and 582 MPa to 1641 MPa, respectively) which were evaluated by hardness measurements and tensile tests.

1. Introduction

Eurofer 97 steel is a candidate material for structural application in ITER. It belongs to reduced activation ferritic–martensitic steels. Low activation is assured by the appropriate chemical composition. However, there are several important issues with regard to Eurofer 97 steel which can be addressed by appropriate microstructural characteristics [1,2]. These include: improvement of mechanical properties and a brittle-to-ductile transition temperature.

Microstructure of materials can be modified by hydrostatic extrusion (HE) processing. It was previously shown that HE processing leads to a significant grain refinement in a number of metallic materials [3,4]. HE also affects particle size, shape and distribution [5]. As a result, such materials exhibit very high strength combined with reasonable plasticity [6]. The aim of the present work is to determine the influence of HE on microstructure and mechanical properties of Eurofer 97 steel.

2. Material and experimental procedure

The material used in the present study was Eurofer 97 steel. In the as-received state it has a form of a rolled plate 26 mm in thickness. The samples of Eurofer 97 steel were hydrostatically extruded at room temperature in a multi-step process. The initial diameter of samples was 26 mm and the final one 3.28 mm, which corresponds to a true strain of ~4. (True strain is defined as $\varepsilon = 2\ln(d_i/d_f)$, where d_i is initial whereas d_f final diameter of extruded rods.). The extrusion parameters for individual extrusion steps are summarized in Table 1. The extrusion direction was parallel to the rolling direction. HE processes were carried out at the Institute of High Pressure Physics of Polish Academy of Sciences.

In order to determine the changes of mechanical properties during the consecutive steps of HE, microhardness was measured using ZWICK 3212-002 hardness testing machine using a load of 200 g. The mechanical properties of Eurofer 97 before and after HE were determined using an INSTRON 1115 testing machine. The tests were carried out at a strain rate of $4 \times 10^{-4}\ \mathrm{s}^{-1}$ on the samples of 2 mm in diameter and 20 mm in gauge length. Tensile tests were performed at −196, 20, 400, 550, 600 °C. Microstructures were observed using transmission electron microscope Jeol JEM 1200 and SEM Hitachi S-5500. The revealed microstructures were quantitatively described in terms of grain and particle size and distribution. To evaluate a grain and carbide size, their equivalent diameters d_2 (defined as the diameter of a circle which has an area equal to the surface area of a given grain) were measured.

3. Results

Fig. 1 shows microhardness changes as a function of true strain. Microhardness continuously increases with the increase of true strain and achieves the highest value (361 HV0.2) for the most deformed samples whereas the lowest value (229 HV0.2) was found for the as-received material.

Microstructure evolution during HE processing is strongly affected by plastic deformation which generates defects, e.g., dislocations. The higher the true strain the more defects are accumulated in the material leading to an increase of hardness. On the other hand, HE proceeds at high strain rate (~$10^2\ \mathrm{s}^{-1}$). In such a

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Table 1
Extrusion parameters for individual extrusion steps.

Step	Initial diameter (mm)	Final diameter (mm)	Reduction ratio	True strain per step	Accumulated true strain	Extrusion pressure (MPa)
1	26	19.93	1.70	0.53	0.53	623
2	19.93	16	1.55	0.44	0.97	616
3	16	12.95	1.53	0.42	1.39	704
4	12.95	10.96	1.40	0.34	1.73	669
5	10.96	8.93	1.51	0.41	2.14	872
6	8.93	7.47	1.43	0.35	2.49	804
7	7.47	6.46	1.34	0.29	2.78	798
8	6.46	5.56	1.35	0.30	3.08	935
9	5.56	4.85	1.31	0.28	3.36	798
10	4.85	4.23	1.31	0.27	3.63	963
11	4.23	3.89	1.18	0.17	3.80	809
12	3.89	3.62	1.15	0.14	3.94	633
13	3.62	3.28	1.22	0.20	4.14	1169

condition, the process has adiabatic character. As a result, although the process was conducted at room temperature, the temperature rise takes place in the material due to the conversion of plastic work into heat. The temperature rise during consecutive extrusion steps was calculated from the following equation (the calculations assume that all plastic work is transformed into heat):

$$Q_v = c\rho\Delta T$$

where Q_v is the amount of heat per unit volume (Q_v is the equal to the value of extrusion pressure which is registered during processing), c is the specific heat, ρ is the density, ΔT is the temperature rise. The results of these calculations are presented in Fig. 2. Temperature rise varies from 170 to 350 °C depending on processing step. The oscillations of temperature rise for individual extrusion steps are due to the variation in extrusion pressure which in turn depends on cross-section reduction ratio and hardening behaviour of a material. The temperature rise favors defects rearrangement and new grain formation which leads to effective grain refinement in Eurofer 97 steel during HE processing. It should be noted that excessive influence of heat and grain growth is avoided thanks to very fast processing.

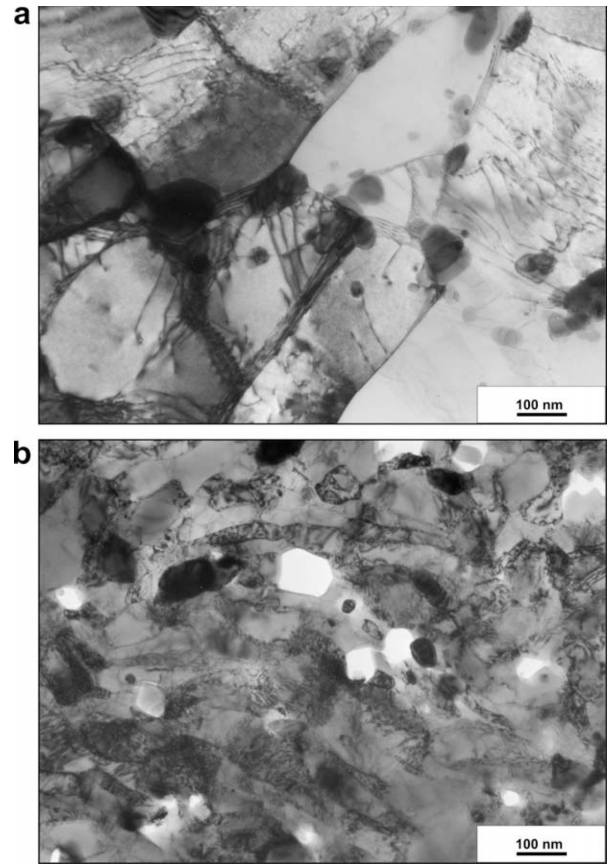

Fig. 3. Microstructure of Eurofer 97 steel before (a) and after (b) hydrostatic extrusion.

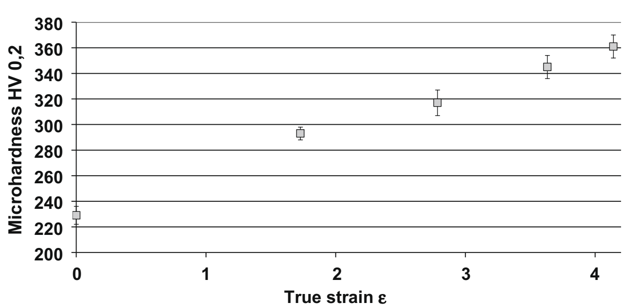

Fig. 1. Microhardness as a function of the true strain.

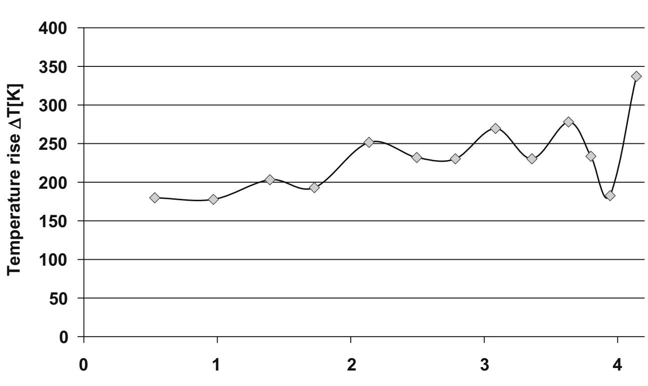

Fig. 2. The temperature rise during individual extrusion steps.

TEM observations (Fig. 3) revealed that during HE processing the grain size was significantly reduced from 400 to 85 nm. HE also leads to a more uniform grain size distribution which was quantified by the coefficient of variation, CV (defined as a ratio of the standard deviation to the mean grain size). The microstructural parameters such as mean value of equivalent diameter, variation coefficient and shape factor (defined as the ratio of a maximum to equivalent diameter) before and after HE are summarized in Table 2.

HE induces also changes in carbides size and distribution (Fig. 4). The average size of carbides decreases from 111 to 75 nm. In the initial state, the carbide size was nowhere near the grain size whereas in the final state it is only a little smaller than the grain size. HE also makes carbide spatial distribution more uniform what can be quantitatively described by SKIZZ tessellation

Table 2
Statistical parameters of grains in HE processed Eurofer 97 steel.

State of Eurofer 97 steel	$E(d_2)$ (nm)	$SD(d_2)$ (nm)	$CV(d_2)$	$E(d_{max}/d_2)$
As-received	400	206	0.52	1.56
HE processed	86	31	0.36	1.59

E, mean value; SD, standard deviation; CV, coefficient of variation.

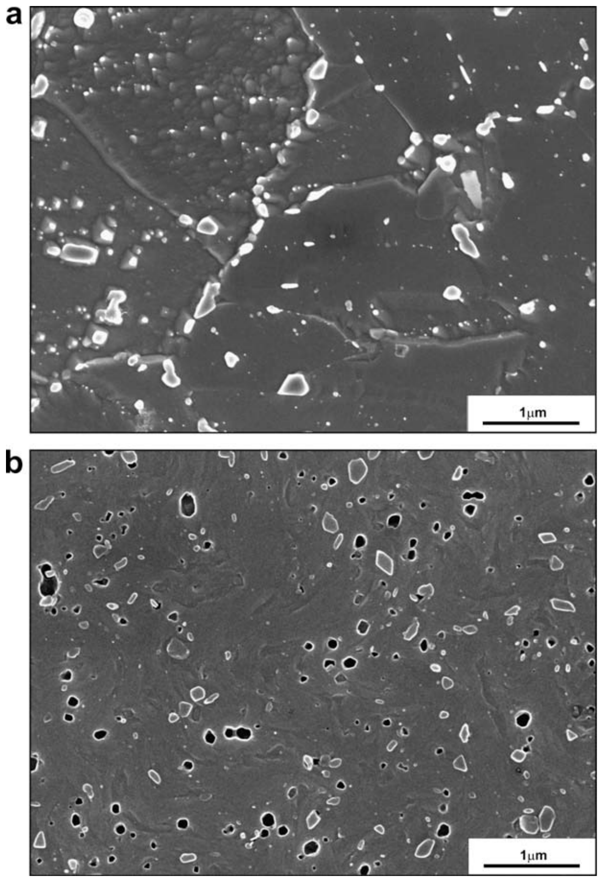

Fig. 4. Distribution of carbides in Eurofer 97 steel before (a) and after (b) hydrostatic extrusion.

method. In this method, Voronoi cells surrounding each particle are generated. The variation coefficient of Voronoi cells area, CV (Av) describes the uniformity of spatial distribution of elements. In the case of Eurofer 97 steel processed by HE, the value of CV (Av) decreases from 0.76 to 0.58. This proves that particle spatial distribution becomes more uniform.

Mechanical properties of Eurofer 97 steel before and after HE processing were compared in a wide range of temperatures as shown in Fig. 5. It is clearly visible that the ultimate tensile strength and the yield stress are lower in the whole range of temperatures for as-received samples than for HE processed ones. The ultimate tensile strength for the former samples changes almost linearly from 313 MPa at 600 °C to 1177 MPa at −196 °C, whereas the yield stress changes from 300 MPa at 600 °C to 1160 MPa at −196 °C. At the same time, the ultimate tensile strength for the latter samples increases from 355 MPa at 600 °C to 2365 MPa at −196 °C, whereas the yield stress increases from 334 MPa at 600 °C to 2197 MPa at −196 °C. It should also be noted that at low temperature (up to 400 °C) the mechanical strength of HE processed material is nearly two times higher than for as-received samples. For high temperature (above 400 °C) the mechanical strength of both materials is similar.

Although HE has caused a considerable increase in strength, the plasticity of the material is reduced. The total elongation of as-received samples ranges between 17.50% (at 600 °C) and 9.00% (at 400 °C). The uniform elongation reaches the highest values at the lowest measured temperature 8% and decreases with the increase of the temperature to the value of 0.53% at 600 °C. The total elongation of HE samples is almost stable up to 400 °C when achieves 4.75%. At 600 °C is almost ten times higher and reaches 41.63%. Contrary to the total elongation, the uniform elongation changes in the range of 1.15% to 1.85% at 400 °C. It is important to notice that at 400 °C the HE processed sample has higher strength and the same plasticity as in as-received state.

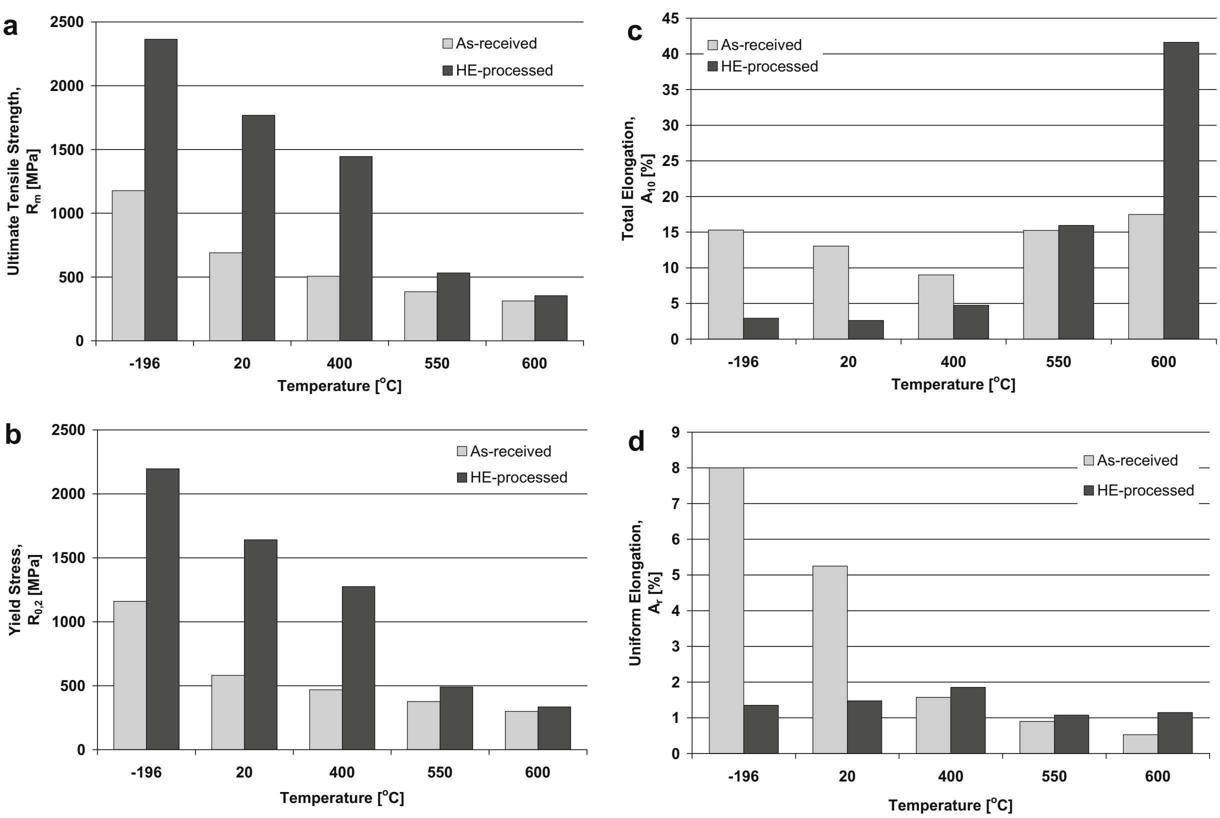

Fig. 5. Mechanical properties of Eurofer steel as a function of test temperature: ultimate tensile stress R_m (a), yield stress $R_{0.2}$ (b), total elongation A_{10} (c) and uniform elongation A_r (d).

4. Conclusions and future plans

The results obtained in the present work clearly show that Eurofer 97 steel can be processed by HE to improve its mechanical properties. Such changes in mechanical properties are due to grain refinement to the nanoscale. Further research, however, is needed to optimize the balance between mechanical strength and ductility of HE processed materials.

Acknowledgements

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