Texture and mechanical properties of EUROFER 97 steel processed by ECAP

M. Eddahbi a,*, M.A. Monge a, T. Leguey a, P. Fernández b, R. Pareja a

* Departamento de Física, Universidad Carlos III, 28911 Leganés, Madrid, Spain
b CIEMAT, Avda. Complutense 22, 28040 Madrid, Spain

Abstract: The EUROFER 97 steel was processed by equal channel angular pressing (ECAP) at 550 °C for four passes via route C. The starting material consisted of ferrite–martensite dual phase composed by small subgrains of about 0.5 µm and low angle boundaries less than 5°. The volume fraction of second phase particles was around 10 vol.%, besides a texture formed by several fibers orientations belonging to the zone axes (1 1 0), (1 1 1) and (1 1 2). Increasing ECAP deformation, this microstructure became into equiaxed grain structures of less than 1 µm, and the misorientation between contiguous grains increased. This refine-ment of the microstructure was accompanied by the development of a new texture described by a family of fiber orientations related by rotations around axes (1 1 0) and (1 1 1). Tensile tests have revealed that an ECAP treatment at 550 °C for two passes could significantly strengthen the tempered material still maintaining good ductility.

Keywords: RAFM steels EUROFER 97, ECAP processing, Texture, Microtexture, Mechanical properties

1. Introduction

Due to its mechanical properties, long-term stability and predictability up to ~650 °C, the reduced activation ferritic–martensitic EUROFER 97 steel is being considered for structural applications in the breeding blankets of ITER, and for the demonstration fusion reactor DEMO [1,2]. The strength of this steel can be enhanced by precipitation hardening, and strengthening induced by dislocations, particle dispersion or grain boundaries. However, precipitation hardening and strengthening by dislocations or particles dispersion can reduce dramatically ductility and toughness compared with grain refinement [3].

Ultafine grained structures can be developed in metals by severe plastic deformation techniques (SPD), among which ECAP is a successful method to develop submicron microstructures in bulk steel [4–9]. The aim of the present work has been to explore the capability of warm ECAP for developing a stable grain structure and texture in tempered EUROFER 97 that can enhance its mechanical behavior in its operational temperature range, i.e. below 600 °C.

2. Experimental procedure

The material used for this study was produced by Böhler (Austria) with composition (wt.%): 0.11%C, 8.7%Cr, 1%W, 0.1%Ta, 0.19%V, 0.44%Mn, 0.004%S, with the balance Fe (EUROFER 97). The as-received plates were normalized at 980 °C for 27 min followed by tempering at 760 °C for 90 min. 12 mm × 12 mm × 65 mm billets of the as-tempered material were ECAP processed at 550 °C with a velocity of 10 mm min⁻¹ through a die with an intersection angle of 105°. The billets were subjected to one, two and four ECAP passes. They were rotated 180° around its longitudinal axis before inserting in the die for the subsequent ECAP pass, what is referred to as route C.

Samples for microstructural studies and tensile tests were machined from the billets, polished with alumina and etched by solution of 5 g FeCl₃ + 6 ml HCl + 100 ml H₂O. The microstructure was characterized by optical microscopy (OM), Scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

Room temperature tensile tests were performed on flat tensile specimens at a constant crosshead rate of 0.04 mm min⁻¹ (initial strain rate of about 3.3 × 10⁻⁵ s⁻¹). The specimens with a gauge length of 20 mm, a width of 3 mm, and a thickness of 1 mm were cut parallel to the flow plane of the billets as shown in Fig. 1.

Texture measurements were accomplished by measurement of pole figures using the Schulz reflection method, in a Siemens diffractometer equipped with a DS000 goniometer. The measurements were performed through the range of azimuthal angles χ between 0° and 75° in the step mode with increments of Δχ = ΔΦ = 5°. A textureless standard sample of annealed pure iron was used for defocusing correction. Quantitative three-dimensional orientation distribution functions (ODFs) were obtained using a Siemens software [10]. These were represented by iso-intensity lines in equidistant sections, Δϕ₁ = 10°, through the Euler space defined by ϕ₁, Φ and ϕ₂ angles. For the representation

* Corresponding author. Tel.: +34 91 624 8734; fax: +34 91 624 8749.
E-mail address: meddahbi@fis.uc3m.es (M. Eddahbi).
of the ODFs, a monoclinic symmetry was used resulting in a good concordance between the measured ODFs and the calculated ones for all ECAP deformed samples.

Grain orientation maps of the material in the as-tempered condition and after ECAP deformation were obtained from the corresponding electron backscattering diffraction (EBSD) patterns recorded using a HKL Nordlys EBSD system interfaced with a field emission gun scanning electron microscope FEG-SEM JEOL JSM-6500F equipped with an energy dispersive spectrometer (EDS). The applied accelerating voltage and the working distance were 25 kV and 15–20 mm, respectively. The EBSD scans using step sizes of 0.14–0.18 μm were performed on 54 μm × 43 μm areas in central region of the specimen surfaces parallel to the flow plane. These surfaces were carefully polished. The EBSD data were analyzed using the in-house software package CHANNEL 5. The OIM images applying the usual coloring scheme for the Euler angles were obtained using the external software package MTEX MatLab Toolbars for quantitative texture analysis [11]. Grain boundaries with misorientation between the contiguous grains of $\theta_{\text{mis}} \leq 10^\circ$ have been defined as low angle grain boundaries (LABs), or sub-grain boundaries, and the ones with $\theta_{\text{mis}} > 10^\circ$ referred to as high angle grain boundaries (HABs).

3. Results and discussion

3.1. Microstructure

Fig. 2 shows the effect of the ECAP processing on the EUROFER 97. The microstructure of the material in the as-tempered condition exhibits laths substructure as Fig. 2(a) and (b) reveal. Both the OM observations and the SEM analyses revealed that this microstructure contains small carbide particles as Fig. 2(b) shows. Most of these particles were found at the prior-austenite boundaries and along the lath sub-boundaries. Two types of carbides were identified: Cr-rich $\text{M}_2\text{C}_3$ with sizes of less than ~0.2 μm, and fine Ta- and V-rich MX carbides. The volume fraction of second phase particles was estimated of about 10% using SME and EBSD measurements.
After the first ECAP pass the microstructure was reoriented and shear strained as shown in Fig. 2(c) and (d) [12]. After four passes, Fig. 2(e) and (f), the microstructure turned out near equiaxed. In addition, the SEM images show that ECAP deformation did not induce agglomeration of second phase par-ticles.

### 3.2. EBSD observations

Fig. 3 shows the effect of four ECAP passes on the grain structure. The indexation of the EBSD patterns also showed that the samples exhibit a dual phase ferrite–martensite microstructure. The OIM images and the distributions of grain sizes and misorientation reveal that ECAP deformation under the present conditions induces grain refinement and misorientation increment in the ferrite phase. Moreover, ECAP deformation transformed martensite into ferrite. The martensite fraction of \( \sim 27 \) vol.% for the EUROFER 97 in the as-tempered condition was lowered to 5 vol.% after four ECAP passes.

The as-tempered state consisted of fine subgrains confined in coarse grains of up to 7 \( \mu \text{m} \) in size as shown in Fig. 3(a) and (b). Most of the subgrains are of about 0.5 \( \mu \text{m} \) in size. The misorientation histograms corresponding to both ferrite and martensite phase in as-tempered state revealed the presence of high den-sity of LABs of less than 5° and low density of HABs as shown in Fig. 3(c). After four ECAP passes the subgrain size is less than 1 \( \mu \text{m} \) and the misorientation angle in the ferrite phase increases, Fig. 3(f).
3.3. TEM observations

Also, the TEM images shown in Fig. 4 illustrate the ECAP effect on the microstructure of the EUROFER 97. After the first and second pass, the microstructure turned out highly elongated and shear strained as shown in Fig. 4(a) and (c). The microstructure after a single pass exhibited a low dislocation density and small subgrains, or grain domains, as Fig. 4(a) and (b) reveal. These domains appear to form by the development of a structure of dislocations walls (indicated by arrows) that can transform into sub-boundaries and boundaries during further deformation via dislocation motion and accumulation into the sub-boundaries, i.e., by extended recovery, also called continuous recrystallization [13]. The sub-boundaries are pinned by small carbide particles retaining the microstructure on the succeeding passes. EBSD analysis and TEM observations indicate that the mechanism of continuous recrystallization appears to be the main responsible for the grain refinement induced by ECAP in EUROFER 97. Nevertheless, the formation of new subgrains is also observed to occur at precipitate particles associated to sub-boundaries or grain triple junctions as shown in Fig. 4(b). In particular, the size of the subgrains nucleated at the triple junctions appear to be less than 100 nm, i.e. much smaller than the subgrains produced by the mechanism of extended recovery. Due to the considerable volume fraction of precipitate particles present in this steel, the mechanism of subgrain nucleation at particles associated to sub-boundaries, or triple junctions, could significantly contribute to the development of a homogeneous ultrafine-grained structure. It should be emphasized that the mechanism of subgrain formation apparently induced by particles associated to sub-boundaries should be different from the particle
stimulated nucleation (PSN) mechanism based on the deformation zones around the second phase particle [14,15]. This mechanism is characteristic of some alloys containing hard particles and requires a critical particle size of \( \sim 1-2 \, \mu \text{m} \), below which a subgrain cannot develop and become into a recrystallization nucleus [14]. Instead, it is suggested that ECAP deformation in EUROFER 97 contributes to activate new slip systems and creates a homogenous distribution of strain in the surrounding region of a small carbide particle that favors the dislocation rearrangement into wall and sub-boundaries, i.e. giving rise to subgrains.

After two ECAP passes the samples exhibited a microstructure composed of elongated grains containing equiaxed substructure

Fig. 5. The ODFs for EUROFER 97, represented through \( \phi_1 \)-sections (\( \Delta \phi_1 = 10^\circ \)) and the spatial arrangement of the fibers in the Euler space, showing the effect of ECAP deformation. a) and b) as-tempered material; c) and d) after a single pass; e) and f) after two passes and g) and h) after four passes.
as shown in Fig. 4(c) and (d). The corresponding OIM images obtained from a sample area of 2322 μm² reveal enhancement of the microstructure fragmentation and a shift of the misorientation distribution toward high θmax values although the LABs are still the major fraction of boundaries present in the ECAP deformed samples. On the contrary, the equiaxed substructure had a high dislocation density, and boundaries that exhibited a thick and blunt image. After four passes, the elongated structure is no longer visible and the microstructure is roughly equiaxed as shown in Fig. 4(e) and (f). This result is in contrast to that obtained for an IF steel, where the lamellar structure is still retained after four passes via route C [16].

The morphology of the substructure developed by ECAP will depend on the initial grain orientation with respect to the shear plane imposed in the ECAP die. This substructure would control the rearrangement of dislocations into dislocation walls contributing to increase the angular misorientation across the cell walls or sub-boundaries. The as-tempered material consists of laths substructure randomly distributed in different orientations into the volume material (Fig. 2(a) and Fig. 3(a)), which would imply different ECAP induced substructures depending on their initial orientation respect to the shear plane. ECAP deformation activates dislocation gliding on the slip planes giving rise to an elongated lamellar substructure on these planes. After an ECAP pass, this elongated substructure tends to align almost parallel to the shear plane, thus should conserve the initial lamellar substructure on ECAP deformation, although they become more elongated and its spacing might be reduced. On the contrary, if the lamellar substructure, or the active slip planes, forms a high angle with shear plane, a new substructure would be developed, so that ECAP deformation refines the substructure of these grains. After several ECAP passes, the lamellar structure would be fragmented resulting in a more equiaxed and refined substructure with higher misorientation θmax across the subgrain boundaries as Fig. 3(f) shows. The effectiveness of the ECAP treatment for refining the grain structure will depend on the change of strain path induced by the ECAP routes.

3.4. Texture

Fig. 5 shows the effect of ECAP deformation via route C on the texture of EUROFER 97 material. The ODFs are represented in the Euler space through φ1-sections with φ1 = 0° – 180°, for the material in the as-tempered condition (Fig. 5(a)), and ECAP processed for one pass (Fig. 5(c)), two passes (Fig. 5(e)) and four passes via route C (Fig. 5(g)). The corresponding spatial arrangements of the ODFs are represented in Fig. 5(b), (d), (f) and (h) showing the positions of the fibers in the Euler space. For more details, Fig. 6 plots the orientation intensity, f(θ), as a function of θ for the ECAP deformed materials. In the present work, the Miller index (hkl) represents an orientation that has an (h k l) parallel to the flow plane of the billet, i.e. plan normal to ND in Fig. 1, and an (u v w) direction parallel to the longitudinal axis of the billet, i.e. PD in Fig. 1. As observed in Fig. 5(a) and (b), the as-tempered material exhibits numerous weak fibers. This texture results from a combination of severe rolling and cross rolling underwent by the material before being tempered. Each fiber is an ensemble of orientations related to each other by rotations around the (110), (111) and (112) axes. It is worth noticing that these axes are either parallel to the ND or located at an azimuth angle.

After the first ECAP pass some weak fibers disappear and new ones form, compare Fig. 5(b) and (d). However, the stronger fiber components observed in the material ECAP deformed for a single pass, marked as S1, S2, S3, S4, S5 and S6 remain after four ECAP passes, although their intensity varies along the skeleton line as shown in Fig. 6(a)–(c). The maximum intensities for these fibers correspond to {110}{122}, {110}{111} and {111}{123} orientations. The weak fibers, labeled W1, W2 and W3 can be described by {100}{1 1 0}, {0 1 3}{3 4 1} and {1 0 3}{4 3 1} orientations. The present ECAP induced texture in the steel EUROFER 97 contrast with the fiber texture {111} and {100} and the fibers {110}{u v w} and {h k l}{111} reported for low carbon and interstitial-free (IF) steels EUROFER deformed at room temperature [9,16]. The mis-orientations could be attributed to the differences in the ECAP parameters as well as to the high content of carbide particles in EUROFER 97.

The crystallographic texture developed during the first ECAP pass is retained after the succeeding passes as shown in Fig. 5(c) and (g), albeit the distribution of orientations along the fibers changes. After the second ECAP pass the intensity of the main fibers decreases and the weak fibers vanish or even disappear as occurred for the W1, W2 and W3-fibers, compare Fig. 6(a) and (b). Then, the second pass does not reverse the first pass texture to the original one of the billet as the approach assuming simple shear deformation at the intersection plane of the die channels predicts for ECAP processing of bcc materials [17,18]. After the fourth ECAP pass, the intensities of two main fibers increase respect to the second pass, and the weak fibers, which are removed or reduced by the
second pass, reappear. These results indicate that shear deformation induced by the first pass, or by an odd-numbered pass, is not reversed by the subsequent pass to the strain state of the billet at the beginning or after a previous even-numbered route C pass. In fact, it has been reported for IF steel that the real ECAP processing conditions alter the cyclic changes of the texture predicted by the simple shear model [16,19]. The inhomogeneous strain across the ECAP processed billet, the complex microstructure, the die geometry and the interaction with neighboring grains appear to constraint the capability of a route C pass to reverse shear deformation induced by the previous ECAP pass. However, the cyclic tendency of the texture for going back to the initial one can be still maintained for an IF steel, in particular if the die angle is changed from 90° to 120° [17,18]. In the present work, the cyclic reversion of the texture by ECAP via route C neither appears to be partially accomplished even though a die with θ = 105° is used. The results obtained for tempered EUROFER 97 ECAP processed under the present conditions reveal that shear deformation induced by a pass is practically irreversible against a subsequent route C pass. This irreversibility that is much stronger than the reported for IF steel in Refs. [17,18] may be attributed to the complex substructure and high concentration of carbide particles present in the tempered EUROFER 97 steel.

Furthermore, the present texture analyses give rise to issues related to deformation mechanism responsible for the evolution of the microstructure during severe plastic deformation of EUROFER 97. Actually, it was found that the orientations developed during a single ECAP pass are connected by rotations in the range 10°–40° around the same axes found for the material in the as-tempered condition, i.e. around ⟨110⟩ and ⟨111⟩ axes. Nevertheless, these axes are now located between the PD and TD directions. Similar results are found in the material deformed for two and four ECAP passes. To illustrate these rotations qualitatively, Fig. 7 shows the localization of ND of the orientations in the cubic stereographic projection. The differences in the ND distribution with increasing ECAP deformation, and keeping the rotation axes ⟨110⟩ and ⟨111⟩, are clearly evident. For one ECAP pass, the S1-fiber orientations are connected through 35° rotation around [1 11] and those corresponding to the S0-fiber through rotation of 20° and 10° around [0 1 1] and [1 0 1]. However, both fibers are closely joined together into rotations around ⟨110⟩ axes after two passes. Furthermore, the S1-fiber described by a rotation of ~30° around [1 0 1] transforms into the S11 and S12-fibers as shown in Fig. 7(a) and (b). The S11-fiber main-tains the rotation axis [1 0 1] whereas the rotation axis in S12-fiber is parallel to the ED. These results indicate that route C can produce significant changes on the distribution of orientations along the main fibers during ECAP deformation of EUROFER 97.

3.5. Tensile properties

Tensile test true stress–true strain curves are depicted in Fig. 8. The tensile strength of the samples ECAP deformed for one pass and two passes via route C increased noticeably in comparison with that for the as-tempered material. The yield stress σy, ultimate tensile strength (UTS) and the uniform elongation (δu) for the as-tempered material are 549 MPa, 685 MPa and 9.4%, respectively. After two passes δu decreased to about 7.5% whilst σy and UTS increased to 638 MPa and 800 MPa, respectively. However, ECAP deformation for four passes resulted in a decrease in the mechanical properties compared to two passes (σy = 602 MPa, UTS = 746 MPa and δu = 4%).

It has been reported that route C beyond two passes is ineffective for reducing the grain size in an IF steel ECAP processed at room temperature [17]. The strength decrease in EUROFER 97 deformed for four passes under the present ECAP conditions cannot be attributed to a limited capability for the grain refinement beyond the second pass via route C because grain refinement was still achieved during the fourth pass as the TEM observations showed. Even assuming stabilization in the grain refinement beyond two ECAP passes, this cannot give account for the decrease in strength and ductility by itself. Nevertheless, the heterogeneous substructures developed in elongated and equiaxed subgrains could influence the mechanical behavior of the material. In fact, the microstructure after two passes is a combination of lamellar and equiaxed substructures, which evolved into an equiaxed substructure after four passes. Both substructures contribute to strain hardening at room temperature for a given strain rate, although hardening should be higher in the regions with equiaxed substruc-
The EUROFER 97 steel was processed by ECAP at 550 °C for up to four passes via route C, and the effects on the grain structure, crystallographic texture and tensile properties were investigated. The main results are as follows:

1) The initial microstructure of the material in the as-tempered condition consisted of a ferrite-martensite dual phase microstructure (volume fraction of martensite was 27 vol.%). The microstructure contains fine subgrains of about 0.5 μm in size and LABs less than 5° and second phase particles around 10 vol.%. The texture was composed by several weak fibers with their orientations into the zone axes (1 1 0), (1 1 1) and (1 1 2).

2) The first ECAP pass produced a highly elongated and shear strained structure with fragmented grains. After two ECAP passes a grain microstructure composed of lamellar and equiaxed substructure was observed. With further passes the fragmentation of the microstructure progressed giving rise to a practically equiaxed substructure after four passes.

3) The evolution of the microstructure upon ECAP deformation was accompanied by the development of a new texture described by a family of fibers retaining the zone axes (1 1 0) and (1 1 1) after the successive passes. The martensite phase changes to ferrite and the fraction of LABs decreases with ECAP deformation. The martensite fraction lowered to ~5 vol.% after four ECAP passes.

4) The yield strength and tensile strength increased significantly after deformation for two ECAP passes but decreased after four passes. The enhanced mechanical behavior of the ECAP deformed steel EUROFER 97 could be attributed to the formation, refinement and texture evolution of the lamellar substructure induced by ECAP deformation.

Acknowledgements

This work has been supported by Madrid Community through the project TECHNOFUSION (S2009/ENE-1679) and Spanish Ministry of Science and Innovation (Contract ENE2008-06403-C06-04). The authors thank the microscopy laboratory of CENIM-CSIC for EBSD measurements.

References