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Texture and mechanical properties of EUROFER 97 steel processed by ECAP

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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 $\langle 110 \rangle$, $\langle 111 \rangle$ and $\langle 112 \rangle$. Increasing ECAP deformation, this microstructure became into equiaxed grain structures of less than 1 μm , and the misorientation between contiguous grains increased. This refinement of the microstructure was accompanied by the development of a new texture described by a family of fiber orientations related by rotations around axes $\langle 110 \rangle$ and $\langle 111 \rangle$. 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 $\sim 650^\circ\text{C}$, 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].

Ultrafine 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 $\times$ 12 mm $\times$ 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 $3.3 \times 10^{-5} \text{ s}^{-1}$). 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 D5000 goniometer. The measurements were performed through the range of azimuthal angles χ between 0° and 75° in the step mode with increments of $\Delta\chi = \Delta\Phi = 5^\circ$. 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, $\Delta\varphi_1 = 10^\circ$, through the Euler space defined by φ_1 , Φ and φ_2 angles. For the representation

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Fig. 1. Sketches of (a) the ECAP die used, and (b) samples machined from the ECAP processed billets for tensile tests.

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 $\mu\text{m} \times 43 \mu\text{m}$ areas in cen-

tral 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 M_{23}C_6 with sizes of less than $\sim 0.2 \mu\text{m}$, 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.

Fig. 2. OM and SEM micrographs of EUROFER 97. (a) and (b) microstructures in the as-tempered condition showing martensite laths, carbide particles and prior-austenite boundaries decorated with carbide particles. Microstructure after (c) and (d) one pass ECAP deformation, and (e) and (f) four ECAP passes. The pressing direction (PD) is horizontal.

Fig. 3. ECAP deformation effect on the grain structure of the EUROFER 97. (a) and (d) OIM images for as-tempered material and ECAP deformed for four passes, respectively. (b) and (e) grain size distribution, and (c) and (f) misorientation distribution for the material in the as-tempered condition and ECAP deformed for four passes, respectively.

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 particles.

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 ~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 μm in size as shown in Fig. 3(a) and (b). Most of the subgrains are of about 0.5 μm in size. The misorientation histograms corresponding to both ferrite and martensite phase in as-tempered state revealed the presence of high density 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 μm and the misorientation angle in the ferrite phase increases, Fig. 3(f).

Fig. 4. TEM micrographs showing the effect of the ECAP deformation on the microstructure of as-tempered EUROFER 97. (a) Elongated substructure and (b) details showing the nucleation of subgrains at a grain triple junction for the material ECAP deformed for one pass; the arrows indicate sub-boundaries or dislocation walls. Microstructure in a sample processed for two passes showing: (c) lamellar and (d) equiaxed grain structure. (e) and (f) images showing equiaxed grains with high density of dislocations in a sample ECAP deformed for four passes.

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 dis-location 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

Fig. 5. The ODFs for EUROFER 97, represented through ϕ_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.

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 $\sim 1\text{--}2\ \mu\text{m}$, 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

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 $\theta = 105^\circ$ 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.

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 σ_y , ultimate tensile strength (UTS) and the uniform elongation (ε_u) for the as-tempered material are 549 MPa, 685 MPa and 9.4%, respectively. After two passes ε_u decreased to about 7.5% whilst σ_y 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 ($\sigma_y = 602$ MPa, UTS = 746 MPa and $\varepsilon_u = 4\%$).

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 observa-

Fig. 7. Traces of the ND to the fiber orientations in the stereographic projection for EUROFER 97 ECAP deformed via route C for: a) a single pass, b) two passes and c) four passes. Each fiber is marked by its corresponding symbol.

tions 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 substructures.

Fig. 8. Effect of ECAP deformation via route C on the tensile properties of as-tempered EUROFER 97.

tures. Then, the expected flow stress increase in these zones should be relieved by assuming propagation of deformation into the lamellar substructure preventing the localization of high stresses in the equiaxed substructures. This stress release mechanism could be responsible for the improvement of tensile properties in the material ECAP deformed for one or two passes. So, it appears that the mechanical behavior of the ECAP deformed steel EUROFER 97 could be explained in terms of a combination of lamellar and equiaxed substructures where the morphology, refinement, fraction and orientation of the lamellar substructure would determine the strength and ductility. Further assessment is needed for a comprehensible understanding of the strengthening mechanisms competing in EUROFER 97 taking into account dislocation density, volume fraction of martensite phase and carbide particles.

Also, in the material ECAP deformed for two passes it should be noted that the $\langle 110 \rangle$ directions are found at $\sim 30^\circ$ – 40° from the ED of the billet in the S_5 and S_6 -fibers and at $\sim 30^\circ$ in the s_{11} -fiber. This means that many more subgrains in this case are favorably oriented to be plastically deformed in comparison with the materials deformed for one or four ECAP passes, which accounts for the better tensile properties in the steel EUROFER 97 deformed for two ECAP passes.

According to the results obtained in EUROFER 97 ECAP processed via route C, it is expected that a selected combination of routes and annealing treatments should produce a nanostructured microstructure with enhanced mechanical properties.

4. Conclusions

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 $0.5\ \mu\text{m}$ 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 $\langle 110 \rangle$, $\langle 111 \rangle$ and $\langle 112 \rangle$.

- 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 $\langle 110 \rangle$ and $\langle 111 \rangle$ after the successive passes. The martensite phase changes to ferrite and the fraction of LABs decreases with ECAP deformation. The martensite fraction lowered to $\sim 5\ \text{vol.}\%$ 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.

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