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Microstructural and mechanical characteristics of EUROFER'97 processed by equal channel angular pressing

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Abstract: EUROFER'97 was processed by equal channel angular pressing (ECAP) at 823 K for a total of 4 or 8 passes, using a die angle of 105°, and its microstructure and tensile behavior in temperature range 568 873 K investigated. A single ECAP pass developed a deformation texture {1 1 0} ⟨0 1 1 ⟩ that was practically stable during subsequent ECAP passes. The materials processed by 1 or 2 passes exhibited a fine micro structure of recovered subgrains and tensile behavior very similar to that for the tempered material in the as received condition. The materials processed using 4 or 8 passes exhibited nearly equiaxial submicron grained structures with a high density of high angle grain boundaries. These materials became softer than the as received material at a testing temperature of ~823 K. EUROFER ECAP processed under the present conditions exhibited hardening ratio somewhat higher than that of the as received material.

1. Introduction

The reduced activation ferritic martensitic (RAFM) 9%CrWVTa steel EUROFER is being considered for structural applications in breeding blankets of ITER having an operational temperature in the range 523 823 K. High temperature strength, ductility, tough ness and microstructural stability are the critical properties required for any structural material for a breeding blanket. This has compelled a comprehensive characterization program of this material that still continues. The microstructural and mechanical characteristics of industrial batches of this steel named EURO FER'97, as well as its irradiation behavior, have been reported, see for instance [1 5] and references in Ref. [4]. The predictability and long term stability of tempered EUROFER'97 have been con firmed up to ~923 K [4,5].

The strength of structural materials can be increased by disloca tions, precipitation hardening and dispersion strengthening. How ever, all these strengthening mechanisms produce a deleterious effect on ductility and toughness that is more significant than the one induced by grain hardening. Then, grain refinement may be more effective for improving the mechanical properties com pared with other hardening mechanisms since it can also increases toughness [6]. Ultrafine grained (UFG) structures can be developed in metals by severe plastic deformation techniques (SPD). Among these techniques, equal channel angular pressing (ECAP) appears to be a method with the capability of producing submicron or even nanocrystalline structures, in bulk samples of steels [7]. These ECAP induced structures are more or less unstable depending on the processing conditions and the material characteristics. Thus, the application of ECAP to improve the mechanical properties would require the stabilization of the material structure. For instance, it has been reported that ECAP processing of steels at room temperature followed by annealing results in an increase of both tensile strength and toughness at room temperature compared with the non ECAP processed counterparts [8 10]. I n EUROFER'97, it has been reported that nano sized grain structures can be attained by multi step hydrostatic extrusion (HE), producing an impressive increase in the yield and tensile strength [11]. Nevertheless, this strengthening reduces ductility remarkably and decays with increasing temperature faster than the non extruded material does. Above 673 K, the decrease in the mechanical strength is accompanied by grain coarsening and a significant increase in ductility indicating that the nanocrystalline structure induced by HE in EUROFER'97 is unstable [11,12].

Bearing in mind these results, it is thought that warm ECAP under determined conditions of strain rate and die geometry might be more effective for improving the mechanical properties of steels than annealing after severe deformation by HE or ECAP at low tem perature. The aim of the present work is to explore the capability of warm ECAP to develop stable microstructures in tempered EUROFER that improve its mechanical behavior in the operational temperature range below 873 K, as well as provide information about the applications of UFG RAFM steels processed by ECAP in fusion devices.

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2. Experimental

2.1. Material

The starting material was EUROFER'97 produced by Böhler (Austria) with composition (mass%): 0.11%C, 8.7%Cr, 1%W, 0.10%Ta, 0.19%V, 0.44%Mn, 0.004%S, Fe for the balance (CIEMAT analyses). The as received conditions were normalizing at 1253 K for 27 min followed by tempering at 1033 K for 90 min.

2.2. Experimental methods

Billets in the as received condition with dimensions 12 × 12 × 65 mm^3 were ECAP processed at 823 K through a die with an inter section angle of 105°. The billet was rotated around its longitudinal axis before inserting in the die for the following ECAP pass, either 180° (route C) or +90° and 90° alternatively (route B_A). The micro structural characteristics, crystallographic texture and tensile properties of samples from billets processed by ECAP were studied. The billets were subjected to 1, 2 and 4 ECAP passes following route C, as well as 8 passes following route B_A. The microstructure was characterized by optical microscopy and transmission electron microscopy (TEM). Tensile tests in the temperature range 568 973 K at a constant crosshead rate of 0.1 mm/min were performed on flat tensile specimens with 20 mm gauge length × 3 mm width × 1 mm thickness cut parallel to the flow plane of the billets. Above room temperature, the tests were accomplished with the specimens in pure Ar flowing.

3. Results and discussion

Fig. 1 shows the ECAP processing effect on the crystallographic texture of tempered EUROFER'97. The material in the as received condition did not exhibit a preferential crystallographic orienta tion. After the first ECAP pass a texture, described by {1 1 0} ⟨0 1 1⟩ orientations, is developed albeit its intensity is relatively small. This texture is preserved after 2 ECAP passes and 4 passes via route C. This means that deformation induced by an even number of passes via route C, under the present processing condi tions, does not alter meaningfully the texture created by the first and following odd passes via route C, as theoretically expected under ideal conditions. This might be due to the fact that carbide precipitates contribute to stabilize the initial ECAP induced texture constraining the development of a new texture.

The effect of ECAP on the microstructure of tempered EUROFER is shown in Fig. 2. The microstructure of the as received samples exhibited traces of the prior austenite grains and martensite laths as Fig. 2a and b reveal. Furthermore, carbide particles were found homogeneously distributed in the matrix, as well as located at prior austenite grain boundaries and along the lath boundaries. After the first ECAP pass the microstructure exhibited large elon gated grains fragmented into subgrains, as expected. Fig. 2d shows that this fragmentation may be due to dislocation arrangement into walls and cells induced by the dynamic recovery mechanism during the ECAP process at 823 K. Note in Fig. 2d that subgrains 1 and 2 are separated by a dislocation wall, and subgrain 3 seems to have developed from a dislocation cell. After the fourth pass, the microstructure was no longer elongated, martensite laths were not observed and an equiaxed structure of submicron grains appeared, as shown in Fig. 2e and f, but still the number low angle grain boundaries was abundant. Eight passes via route B_A generated a more refined structure, with grain sizes less ~500 nm and a high number of high angle grain boundaries as Fig. 2h shows.

The tensile properties as a function of temperature are summa rized in Fig. 3. ECAP processing enhances both yield strength and ultimate tensile stress (UTS) up to temperatures around 773 K. Above this temperature, which is near the processing temperature, the material processed four times via route C or eight times via B_A became softer than the tempered material and the materials pro cessed by 1 pass, or 2 passes via route C. Fig. 4 shows the effect of ECAP on the temperature dependence of the hardening ratio (UTS/yield strength). In contrast to the expected result for steels, and other metals, processed by SPD, which do not exhibit signifi cant work hardening during tensile testing, EUROFER processed by ECAP does. The hardening ratio for ECAP processed EUROFER was somewhat enhanced compared with the as received EUROFER in the investigated temperature range, except for the materials ECAP processed by 8 passes via route B_A that had a lower hardening ratio at room temperature. It is should be noted that the steep increase in the hardening ratio for the materials processed by 4 or 8 passes is accompanied by an abrupt change in the temperature trend of their uniform and fracture elongations. However, tensile behavior of materials processed by 1 pass, or 2 passes via route C, is very similar to that for the as received material.

The above results indicate that the microstructure developed by ECAP at 823 K with up to 2 passes via route C preserves the tensile behavior with temperature for EUROFER'97 up to 873 K, while slightly improving the work hardening rate, and strength at low temperature. This suggests that this microstructure may remain stable up to about 873 K, in addition to be somewhat refined. On the contrary, the softening observed for the materials processed by 4 or 8 passes suggests that the corresponding microstructures are unstable above 823 K. This softening can be attributed to dislo cation recovery, and partial recrystallization and grain coarsening, during the tensile tests. These phenomena would be more probable and have a greater effect in these materials because of the very high dislocation densities accumulated after 4 or 8 ECAP. The TEM observations revealed that a very high density of tangled dislocations trapped at grain boundaries appeared in samples processed by 4 and 8 passes while the samples processed by 1 and 2 passes exhibited subgrain boundaries free of this type of

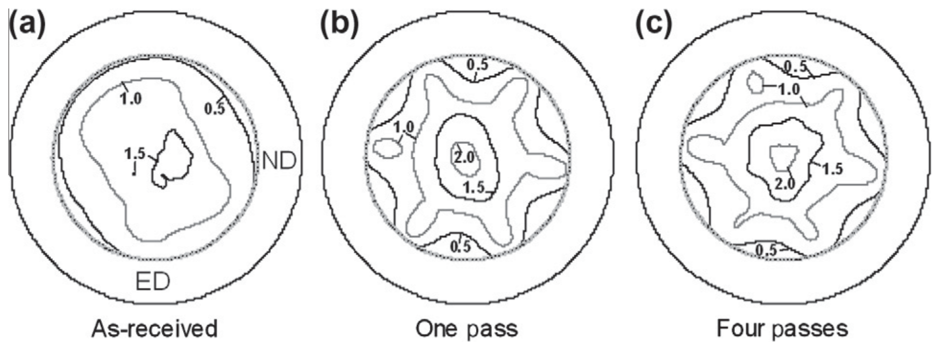

Fig. 1. Measured pole figures (1 1 0) for tempered EUROFER'97: (a) as-received, and ECAP processed, (b) 1 pass and (c) 4 passes.

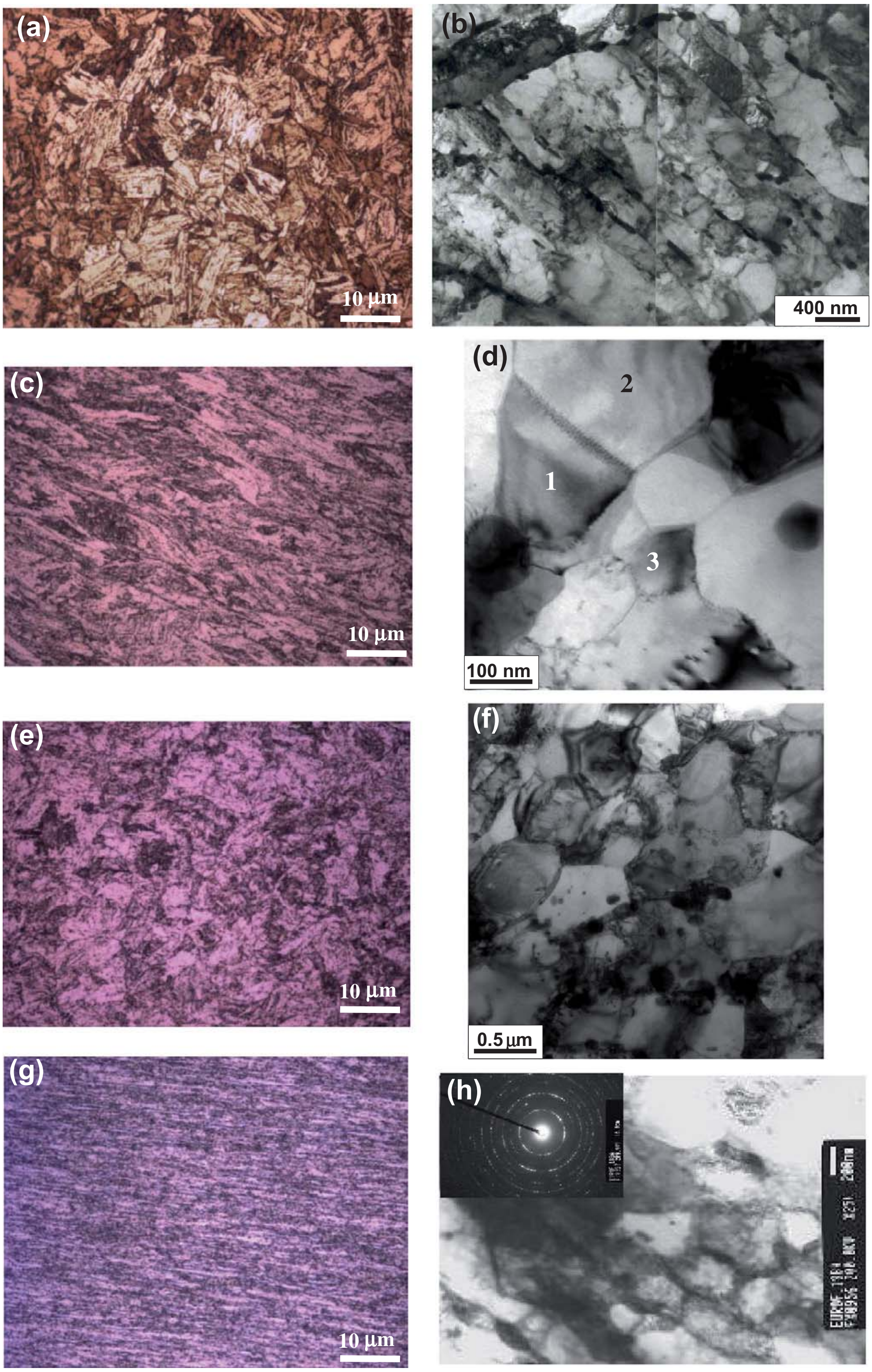

Fig. 2. Optical and TEM images of the microstructure of tempered EUROFER'97: (a and b) as-received, and ECAP processed; (c and d) 1 pass; (e and f) 4 passes via route C; (g and h) 8 passes via route B_A. The magnification is the same for all the optical images.

dislocations. The high angle grain boundaries developed after 4 or 8 ECAP passes, i.e. geometrically necessary boundaries (GNBs), can trap dislocations induced by deformation, but the subgrain bound aries developed from mutual dislocation trapping after 1 or 2 passes, i.e. incidental dislocation boundaries (IDBs), are permeable to moving dislocations [13].

4. Conclusions

A single ECAP pass at 823 K induces a {1 1 0} ⟨0 1 1⟩ texture that was not altered by subsequent 823 K ECAP passes via route C. EUROFER'97 processed by 1 or 2 ECAP passes presented a recov ered fine microstructure that is stable, in terms of mechanical

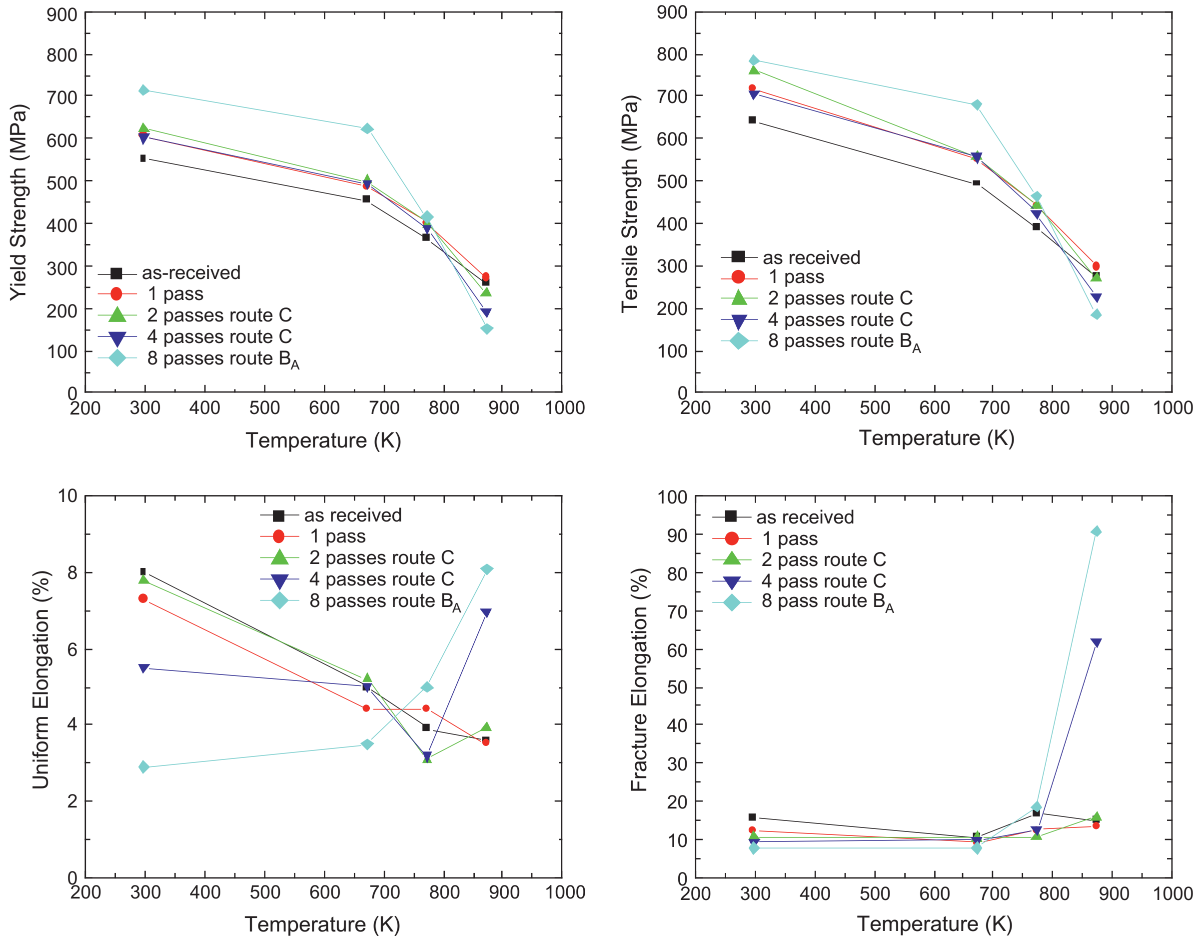

Fig. 3. Effect of ECAP processing on the tensile properties of tempered EUROFER'97.

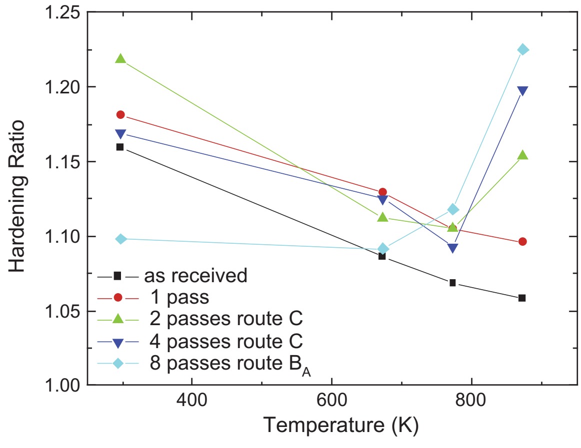

Fig. 4. Effect of temperature and ECAP processing on the hardening ratio for tempered EUROFER'97.

behavior, up to somewhat above the processing temperature. However, when it is subjected to 4 or 8 passes its microstructure appeared to be unrecovered, at least in part, and unstable just above the processing temperature.

The origin of the unusual tensile behavior of the EUROFER steel processed by warm ECAP compared with the one reported for low carbon steels, also warm ECAP processed [8,14], should be investi gated in relation to the processing parameters, alloying elements, content and distribution of dispersed particles, grain refinement and microstructure stability.

Acknowledgements

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