

Mechanical properties and fracture characteristics of high carbon steel after equal channel angular pressing

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ABSTRACT

The ultra-microduplex structure was fabricated in a fully pearlitic Fe–0.8 wt% C steel after equal channel angular pressing (ECAP) at 923 K via the Bc route. The microstructures and mechanical properties, before and after deformation, were investigated using scanning electron microscopy and mini-tensile tests. The cementite lamellae are gradually spheroidized by increasing the number of ECAP passes. After four passes, the cementite lamellae are fully spheroidized. Microhardness and the ultimate tensile strength of pearlite increase with the strain, up to a peak value (after two passes) and then decrease significantly. The yield strength, elongation and percentage of reduction in area increase with the number of ECAP passes. The tensile fracture morphology changes gradually from brittle cleavage to typical ductile fracture after four passes.

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1. Introduction

The ultrafine grained (UFG) steel is an advanced high performance structure material, since it has high strength, excellent plastic deformation performance, and can be processed super-plastically at high strain rates and low temperatures. Grain refinement, which can synchronously enhance material's strength and toughness, is the main aim of research and development for the new generation steels. There are many techniques of grain refinement for carbon steels, associated with severe plastic deformation [1–4] and advanced thermo-mechanical processes [5–7]; however, most of the studies have focused on low or medium carbon steels. High carbon steels are widely used in industries. Therefore, from the application point of view, it is of great interest to synchronously obtain ultrafine microstructure and excellent mechanical properties for high carbon steels.

Furuhara and Maki, [8] reported that ultrafine ($\alpha+\theta$) microduplex structures (average sizes of ferrite grains (α) and cementite particles (θ) were 0.4 and 0.3 μm , respectively) were achieved by severe warm rolling of ($\alpha+\theta$) microstructure at 923 K for ultra-high-carbon steels (> 1.0 wt% C, such as SUJ2 Fe–1.4Cr–1.0C steel). Significant dynamic

recovery of α grain had happened after warm rolling, and the uneven microduplex structures caused the difference in mechanical properties. Meanwhile, the strain of warm rolling was very large, so higher requirements were needed for the rolling equipment. Wang et al. [9] also obtained the ($\alpha+\theta$) microduplex structures in a fully pearlitic 65Mn steel by ECAP via the C route at 923 K, and the effective strain was up to 5. The average sizes of the α grains were reaching the level of 0.3 μm , and the θ particle size distribution was bimodal. The grain size of the bulky θ particles was about 0.6 μm , and the average diameter of fully spheroidized θ particles was about 0.1 μm . However, the mechanical properties were not studied in detail in Ref. [9]. In the previous study [10], an ultrafine ($\alpha+\theta$) microduplex structure was fabricated by warm cross-wedge rolling (WCWR) at the surface layer of high carbon steel rod, where equiaxed α grain size was about 0.4 μm and the fully spheroidized θ particles were 0.1–0.2 μm in size. However, the spheroidization of the θ particles was non-uniform from the surface to the center. In the central region of the rod, original θ lamellae was fragmented, but the spheroidization was not complete. In the present study, the Fe–0.8 wt% C steel with a fully pearlitic structure was processed by equal channel angular pressing (ECAP) via the Bc route at 923 K. The underlying microstructure evolution mechanism was revealed and the mechanical properties were also systematically studied. The results discussed in this paper could provide some useful experimental support for the development and applications of ultrafine-grained high carbon steel materials.

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2. Materials and experimental procedure

2.1. Materials

The material used in this study was commercial high carbon steel (Fe–0.8 wt% C). To ensure the full evolution of pearlite, all specimens were vacuum annealed at 1273 K for 30 min and then placed into a salt bath furnace at 873 K for 30 min. This was followed by water cooling outside the furnace.

2.2. Experimental procedure

The ECAP work was done in Central Iron and Steel Research Institute for Structural Materials. The samples used for ECAP were machined into cylindrical bars, 49 mm in length and 8.3 mm in diameter. Repetitive pressing was feasible, as the sample's cross-section remained unchanged. The ECAP experiments were conducted using a split die with two channels intersecting at inner angle of 120° and outer angle of 30°. Therefore, the strain per path was 0.62 [11]. Up to four passes of ECAP were conducted on the samples at 923 K using the Bc route method, in which the specimen was rotated 90° along the longitudinal axis between the passes. It is known that the Bc route method is effective in obtaining a homogeneous microstructure [12]. Before extrusion, the die was preheated to 573 K and the samples were heated up to the deformation temperature and held for 20 min. The samples were pressed at a pressing speed of ~2 mm/s to prevent surface cracking. The time from taking out the samples from the furnace to finishing 1 per pass of ECAP was less than 30 s. The specimen and the die were both coated with graphite and MoS₂ for lubrication, before being placed in the entrance channel at the testing temperature.

The samples used for the metallographic investigation were cut from transverse cross-sections by wire-electrode cutting method, before and after ECAP, and were then subjected to several successive steps of grinding and polishing. After that, the samples were etched in a 4 vol% nitric acid solution, and scanning electron microscopy (SEM, Hitachi-S4300) was used for microstructure analysis. Mini-tensile test specimens with a gage length of 6 mm and cross-sections of 1 × 2 mm² were sectioned along the longitudinal axes. The mini-tensile test was conducted on Gleeble 3500 thermo-mechanical simulator, with a chuck moving velocity of 0.2 mm/min. The morphologies of the fracture surfaces were observed using JSM-5610LV SEM. Microhardness was measured using MH-3 Vickers microhardness tester with 200 g normal load and 10 s holding time on the as-polished regions. An average microhardness value was determined based on 5 indentation measurements.

3. Results and discussion

3.1. Microstructure before and after ECAP

Fig. 1 shows the SEM micrographs before and after different passes of ECAP. The initial microstructure of the as-received Fe–0.8 wt% C steel is shown in Fig. 1a. It can be seen that the initial microstructure was fully pearlite. After one pass, the cementite lamellae shears in a regular way, as seen in Fig. 1b, the cementite plates remain parallel to each other, but they are in a discrete form, indicating the occurrence of the cementite lamella breakage during ECAP. In addition, the discrete cementite lamella are severely bent and kinked, and the occurrence of spheroidization in a small portion of cementite can be also found. The spheroidized cementite particles remain at the initial cementite lamellar domain. The present observation reveals that

cementite possesses a considerable capability for plastic deformation under the deformation mode induced by the present ECAP. Fig. 1c and d are the SEM micrographs of the sample after two and three ECAP passes, respectively. After two passes, the cementite lamellae are fully fractured and then spheroidized. The shape of the cementite is a short bar, or an ellipse, as seen in Fig. 1c. With further increase in strain, in Fig. 1d, spheroidization of the cementite lamellae is increased and most of the cementite lamellae are transformed to particles. After four passes, as seen in Fig. 1e, the microstructure of pearlite consists of nearly spheroidized cementite particles, with only a few lamellae still present. This indicates that the cementite lamellae are fully spheroidized. The driving force for spheroidization is the reduction of the interfacial surface area between ferrite and cementite that occurs when the structure transforms from lamellar to spheroidal [13]. Meanwhile, with further ECAP, the plastic deformation is accelerated [14,15] and the driving force for spheroidization increases. Therefore, the cementite lamellae are gradually spheroidized by increasing the number of ECAP passes. In addition, the effect of the deformation temperature is also significant. Wetscher et al. [16] deformed a fully pearlitic R260 steel rail by ECAP, at room temperature. After three passes, the lamellae spacing decreased significantly, but globular cementite was not found. This was most likely an effect of the elevated 923 K temperature, during which this ECAP experiment was conducted. The high deformation temperature increases iron and carbon atoms diffusion, and thus promotes cementite spheroidization. Consequently, the cementite lamellae are fully spheroidized. The microstructure of ECAP ultrafine-grained high carbon steel was investigated and the corresponding grain refinement mechanism initiated by ECAP was explored in the previous work [17].

3.2. Mechanical properties

The engineering stress–strain curves from the mini-tensile test of the high carbon steel samples before and after different ECAP passes at room temperature are shown in Fig. 2. After three and four ECAP passes, the stress–strain curves exhibit the obvious yielding point and the discontinuous yielding phenomena, to some extent compared with that of the original lamellar pearlite structure specimen. According to the polycrystalline yield theory, the obvious yield point attributes to the dislocation pile-up on the ferrite grain boundaries, and dislocations activation in ferrite grains. However, the original lamellar pearlite, ferrite, is divided into brittle and hard lamellar cementite. Without multi-directional free slip-deformation ferrite does not have the ability for cooperation–deformation. Obstacles effect of the cementite lamellae on deformation is beyond the grain boundaries. Thus, pearlite structures are unable to offer high plasticity. Correspondingly, there is no obvious yield point in the stress–strain curve. According to Lin et al. [18], as long as granular carbide occurs in the grain boundary region, avoiding closed pearlite lamellae, the obvious yield point occurs, which validates the results of this paper. As indicated by Hayashi et al. [19], the dispersed cementite particles can significantly affect the plasticity of refined-grain ferrite steels. The smaller the cementite particles, the more uniform the plasticity is. Song et al. [20,21] also showed that the dispersed fine cementite particles can effectively improve the ability of work hardening.

Fig. 3 shows the mechanical properties of pearlite before and after different ECAP passes. It can be seen that the microhardness and the ultimate tensile strength (UTS) of pearlite increase with strain, up to a peak value (two passes), and then decrease significantly, but after four passes, the values slightly increase. This is due to the joint action of work hardening, spheroidization of the cementite lamellae and grain refinement. The microhardness and

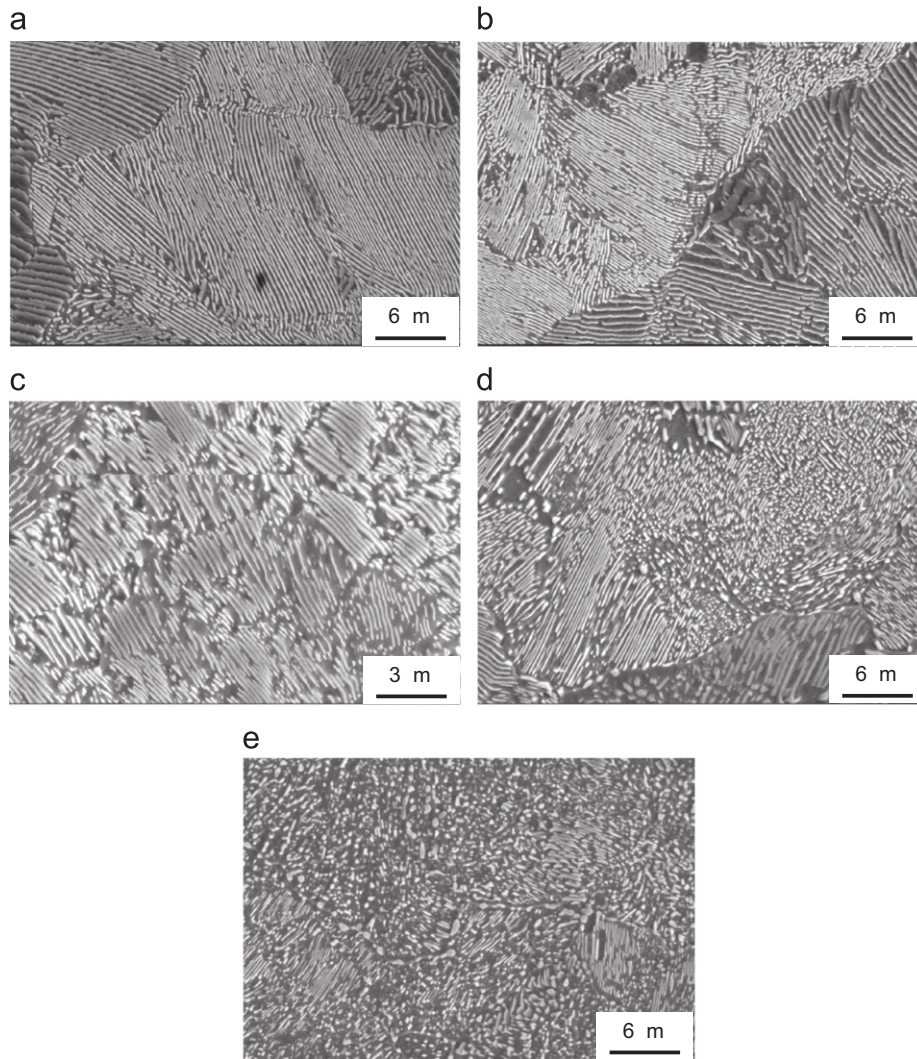


Fig. 1. SEM images of microstructure before and after different passes of ECAP: (a) pearlite, (b) 1 pass, (c) 2 pass, (d) 3 pass and (e) 4 pass.

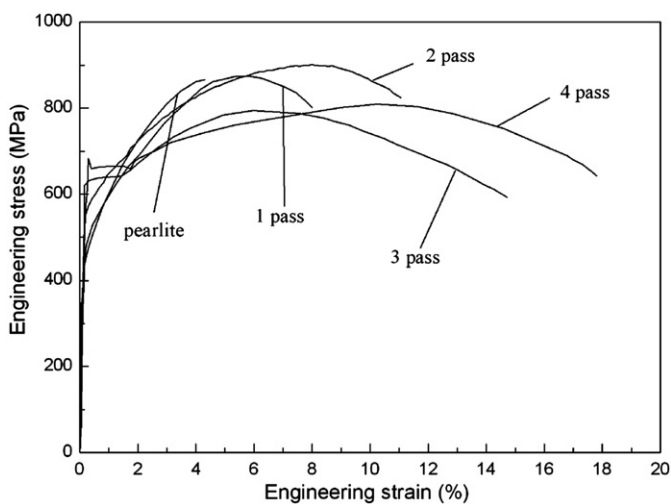


Fig. 2. Engineering stress–strain curves of the mini-tensile samples before and after ECAP.

UTS increase due to the work hardening of ECAP; however, recrystallization of the ferrite and spheroidization of the cementite lamellae during warm deformation decrease the hardness. At the initial stage of deformation, the work hardening effect is prior to the

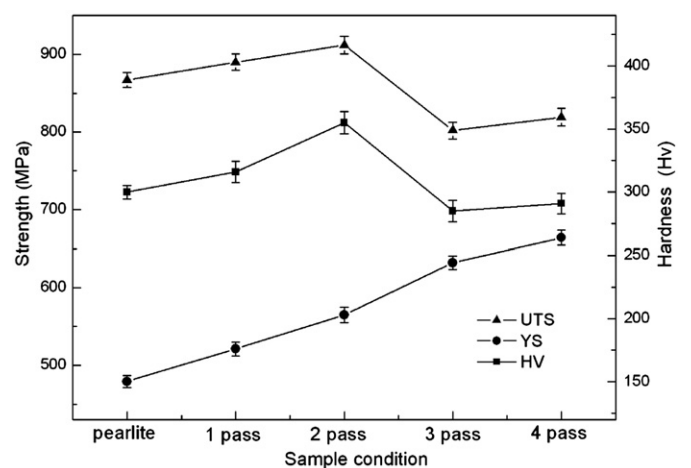


Fig. 3. Mechanical properties (HV, UTS and YS) of pearlite before and after ECAP.

combined effect of recrystallization and spheroidization, which causes the microhardness and UTS increase. Thus, after two ECAP passes, peak values are reached, 355 HV microhardness and 912 MPa UTS. With further straining, the rate of work hardening decreases, and most dislocations disappeared due to dynamic recrystallization. Meanwhile, most of the cementite lamellae are spheroidized, thus,

the value of the microhardness and UTS decreased quite a bit. After four ECAP passes, the grain is refined, so the values of the microhardness and UTS increase to 291 HV and 819 MPa respectively,

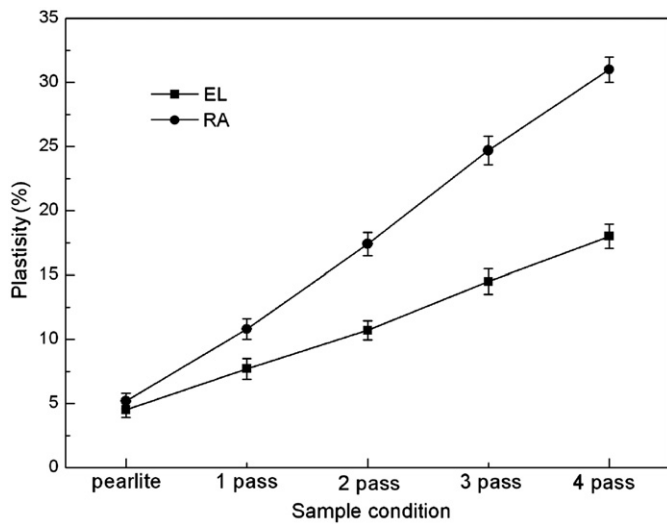


Fig. 4. Mechanical properties (EL and RA) of pearlite before and after ECAP.

which are slightly below the original lamellar pearlite (300 HV and 867 MPa). Generally speaking, for the same compositions, the plasticity of granular pearlite is better, but its strength/hardness is lower than that of lamellar pearlite [22]. In addition, as can be seen in Fig. 3 the yield strength (YS) increases with the number of ECAP passes, along with the ratio of yield strength to tensile strength. The ratio of yield strength to tensile strength of the original lamellar pearlite is 0.55. Nevertheless, the ratio of yield strength to tensile strength after one, two, three and four ECAP passes is 0.57, 0.62, 0.79 and 0.81, respectively. This difference in the ratio of yield strength to tensile strength can be attributed to different microstructures after different number of ECAP passes. Similar changes in the mechanical strength due to hot deformation are reported in other studies for eutectoid steels, see for example [23].

The elongation (EL) and percentage reduction in area (RA) of pearlite before and after different ECAP passes are shown in Fig. 4, where a similar trend is observed. Both the EL and RA are obviously increased by ECAP. After four passes EL/RA are the highest, 18% and 31% respectively, markedly higher than those of the lamellar pearlite. The above phenomena can be explained by the microstructure distribution. Cementite after four ECAP passes has been spheroidized completely and the spheroidized cementite particles are distributed uniformly in the recrystallized ferrite matrix. However, cementite after one to three passes is only partially spheroidized, with poor microstructure uniformity

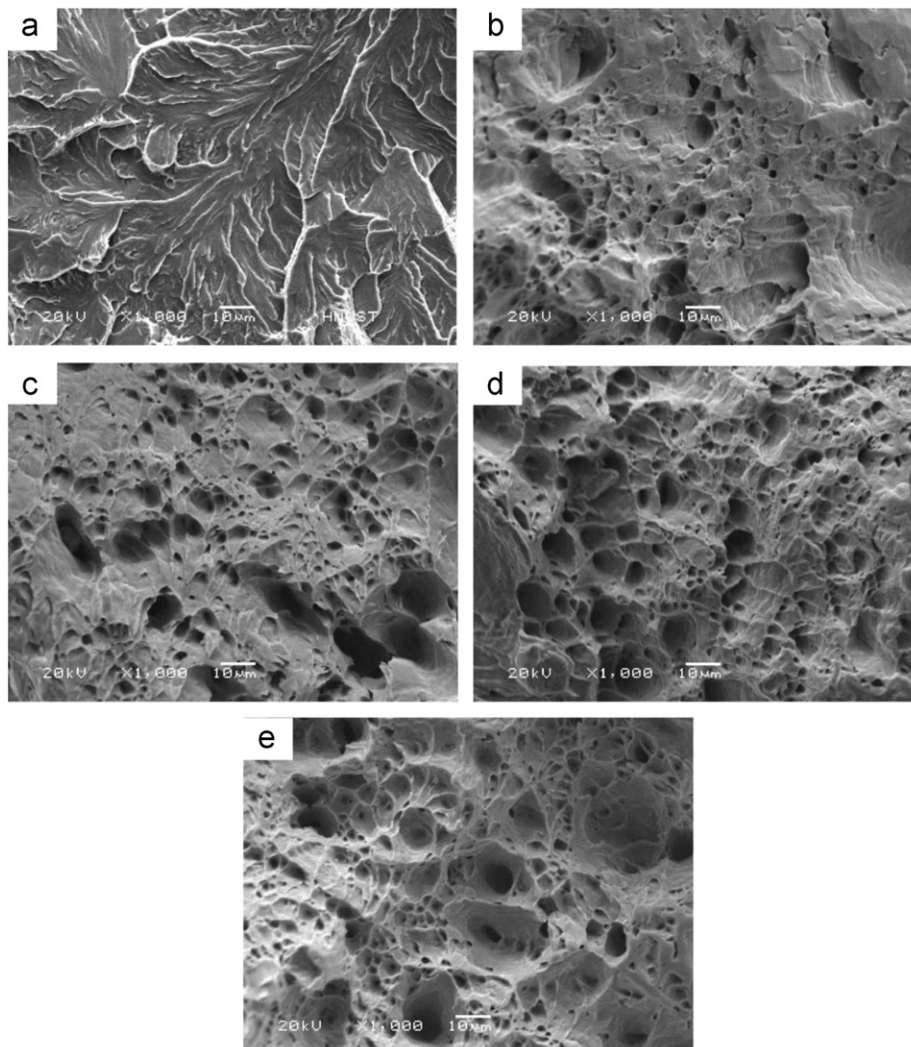


Fig. 5. Fracture surface morphology of the mini-tensile samples: (a) pearlite, (b) 1 pass, (c) 2 pass, (d) 3 pass and (e) 4 pass.

(e.g. a small amount of lamellar pearlite still present in the microstructure). It can be concluded that the spheroidization of cementite and the recrystallization of ferrite have a remarkable effect on the EL and RA of ECAP samples.

The fracture surface morphologies of pearlite before and after different number of ECAP passes are shown in Fig. 5. Fig. 5a shows the fracture surface of the original lamellar pearlite structure, which exhibits a typical river-like pattern, and is the cleavage fracture in the present work. After one pass, some small dimples can be found in Fig. 5b, indicating the occurrence of the breakage and spheroidization of the cementite lamellae. Fig. 5c and d are the fracture surface morphologies of the sample after two and three ECAP passes, respectively. With higher strain, the number and the depth of the dimples increases, suggesting higher degree of cementite lamella spheroidization and improved plasticity. After four passes, the microstructure changes from lamellae to equiaxed grains. The fracture surface morphology of the ultra-microduplex structure can be seen in Fig. 5e, which is a typical ductile fracture. The fracture surface of the sample is smooth, and many dimples with different sizes and depths can be found. The average size of few large and deep dimples is about 8 μm , while a large number of small and shallow dimples is distributed uniformly, and the average size is about 1 μm , as seen in Fig. 5e.

4. Conclusions

- (1) The ultra-microduplex structure was fabricated in a fully pearlitic Fe–0.8 wt% C steel after four ECAP passes at 923 K via the Bc route. The cementite lamellae are gradually spheroidized by increasing the number of ECAP passes. After four passes, the cementite lamellae are fully spheroidized.
- (2) Microhardness and the ultimate tensile strength of pearlite increase with the strain up to a peak value (after two passes), and then decrease significantly, but with further ECAP, the value slightly increases. After four ECAP passes, the microhardness and the ultimate tensile strength increase to 291 HV and 819 MPa, respectively. The yield strength and the corresponding ratio of yield strength to tensile strength increase with the number of ECAP passes. After four passes, the elongation and percentage reduction in area are 18% and

31%, respectively, markedly higher than those of the lamellar pearlite.

- (3) With increasing number of ECAP passes, the tensile fracture morphology gradually changes from brittle cleavage to typical ductile fracture (after four passes).

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