Impacts of warm equal-channel angular pressing on microstructure and mechanical properties of granular pearlitic steel

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Abstract:

Equal-channel angular pressing (ECAP) of granular pearlite high carbon steel at 650 ℃ via Bc route has been thoroughly investigated. Together with microstructural evolution investigated through scanning- and transmission- electron microscopies, the micro-tensile and microhardness testing were carried out for their mechanical properties. After four passes of warm deformation, the formed ultra-microduplex structure is found to contain both ferrite grains of size ~0.45 μm and cementite particles with the diameter of ~0.3 μm. The corresponding microhardness and tensile strength are observed to increase first followed by a decrement with the number of deformations passes. Meanwhile, yield strength and yield ratio increase with the ECAP passes, along with a slight decrease in the elongation. The fracture morphology has also been changed from many deep dimples before ECAP to many small dimples after ECAP application, denoting a typical ductile fracture of the pearlitic steel.

Keywords: Equal-channel angular pressing; Granular pearlite; Microstructure; Mechanical property; Fracture morphology

1. Introduction

Though endowed with excellent strength and wear resistance, high carbon steels are limited in application fields due to its poor toughness and ductility. Grain refinement, which simultaneously enhances strength and toughness of the steels, is widely considered in material engineering for next generation steels. So far, many techniques have been developed to refine grains within high carbon steels. A few of them involve equal-channel angular pressing (ECAP) [1], high pressure torsion [2], ball milling [3], thermo-mechanical processes [4] and warm cross wedge rolling [5]. Amongst them, ECAP is considered as the most promising method, thanks to its scaling-up potentials to large species. It also provides homogeneous microstructures and negligible reductions of cross-sectional dimensions of samples [6].

Wetscher et al. [7] reported an increase in the tensile strength with the ECAP passes after a complete deformation of the pearlitic R260 steel rail via ECAP at room temperature. The fracture toughness and fatigue crack propagation rate showed a strong azimuthally anisotropy, which was found to be closely related with the deformed carburizing layer. Huang et al. [8] obtained ultrafine-grained microstructures in the same type of 65Mn steel by five passes of ECAP via route C at 650 ℃. They obtained an average grain size of around 0.3 μm in addition to the formation of huge number of defects due to the severe plastic deformation. Wang et al. [9] studied the cementite spheroidizing process of 65Mn steel by ECAP at 650 ℃ via route C. The cementite showed an observed acceleration of spheroidization characteristics during the warm deformation process. After five passes of ECAP, an ultrafine microduplex structure consisting of both the ferrite and cementite was noted wherein the grain size was suppressed down to sub-micrometer level. Xiong et al. [10-12] investigated fracture morphology and mechanical properties of high carbon steels by ECAP at

650 ℃ via route Bc. After two passes of ECAP, the microhardness and tensile strength reached the maximum value and then decreased with the pass number. An ultrafine (α+θ) microduplex structure was observed after four passes of ECAP, and the cementite lamellae was noted to be fully spheroidized. A gradual change from brittle fracture to typical ductile fracture was also noted in the tensile fracture. During the deformation, the microstructure of pearlite changed from planar lamellae to three-dimensional equiaxed grains. Nanoindentation hardness and Young's modulus of the equiaxed submicron ferrite phase were also found to be slightly higher than the original lamellar pearlite's. The nanoindentation hardness showed a clear base effect, while the Young's modulus was not sensitive to the base effect.

Despite these successes in materials engineering, the above researches were intended mainly on the lamellar pearlite structure with high strength and deformation resistance. It has been noticed that the lamellar pearlite is easy to fracture during the deformation process because of their poor toughness and ductility. The counterpart granular pearlite has more tolerance against deformation due to excellent plasticity and toughness. This enable the granular perlite a more conducive subject in severe plastic deformation than the lamellar. In addition to these aspects, a systematic study consisting of microstructure and mechanical properties of such high carbon steels with granular pearlite structures after ECAP are still lacking. In this work, the T8 steel with a completely granular pearlitic structure has been investigated by ECAP at 650 ℃ via Bc route . Together with the revelation of underlying microstructure evolution mechanism, a systematic study of the mechanical properties is also covered. This work will hopefully provide an experimental support to the future ECAP treatment of high carbon steels and further widen their application fields.

2. Experimental procedures

2.1 Experimental material

A commercially available T8 steel was selected for the present work. Its chemical composition is tabulated in Table 1. To ensure a complete evolution of granular pearlite, a set of T8 steel blocks was prepared and placed at 740 ℃ for 2 h in the furnace. The blocks were further cooled down and hold at 680 ℃ for 8 h, down to 500 ℃ in the furnace cooling, and then air cooling outside of the furnace. It is worth to mention here that during the heat treatment process, anti-oxidants treatments were applied to the surface of the material to prevent decarburization.

Table 1. The chemical composition of experimental T8 steels (wt. %) [13]

C	S	Si	Mn	P	Cr	Ni	Cu	Fe
0.82	0.012	0.244	0.334	0.011	0.09	0.043	0.124	Balance

2.2 Experimental process

The present ECAP experiments were performed at the Institute of Structural Materials, Beijing Iron and Steel Research Institute. Samples selected for ECAP were machined into cylindrical bars with 49 mm in length and 8.3 mm in diameter. Further, the intersecting angle between the two channels and the angle of the outer arc at the intersection were selected to be 120° and 30°, respectively. Systematically, four passes of ECAP were carried out on these samples at 650 ℃ using the Bc route. The sample was further rotated 90° along the longitudinal axis between the passes [14-16]. 18Ni (350) martensite aging steel was chosen as the die material. Before the experiment, the mould was preheated at a temperature of about 200 ℃, and the temperature of the sample and die was determined with an infrared thermometer gun. The selected sample and the chosen die were further coated with MoS_2 and graphite for lubrication before being kept in the entrance channel at the selected temperature [17]. After each pass of ECAP, the sample was machined again to remove the front and end warping parts. Meanwhile, the middle part of the sample was machined precisely to facilitate the subsequent extrusion deformation.

To probe metallographic investigation, the selected samples were cut from transverse cross-sections by using the conventional wire-electrode cutting method from the bars before and after the application of ECAP. The sample geometries and orientations are shown in Figure 1. They were then successively polished. Afterwards, these samples were etched in a 4 vol.% nitric acid solution. The microstructure analysis was carried out on a scanning electron microscope (SEM, JSM-5610LV). Moreover, a transmission electron microscope (TEM: JEM-2010) was employed to investigate the microstructure evolution of ECAPed samples. The TEM experiment was operated at 200 kV. For this experiment, a mechanically polished 40 μm thin foil of these samples was prepared by using a well-known double jet electrolytic thinning process (30 V, 50 mA) in a mixture of 93 vol.% acetic acid and 7 vol.% perchloric acid (temperature below -30 ℃). The sample with lengths of 6 mm and cross-sectional areas of 2 mm×1mm was sectioned along the longitudinal axes for the aimed mini-tensile test. This mini-tensile test was performed on a micro-tensile testing machine specified by Instron 5948R. The morphology characterization of the fracture surfaces was carried out by using JSM-5610LV SEM. Further, the microhardness characteristic was performed by using MH-3 Vickers microhardness tester with a 200g normal load and 10s holding time on the as-polished regions of selected sample. To ensure the data repeatability, an average microhardness value was selected based on five indentation measurements.

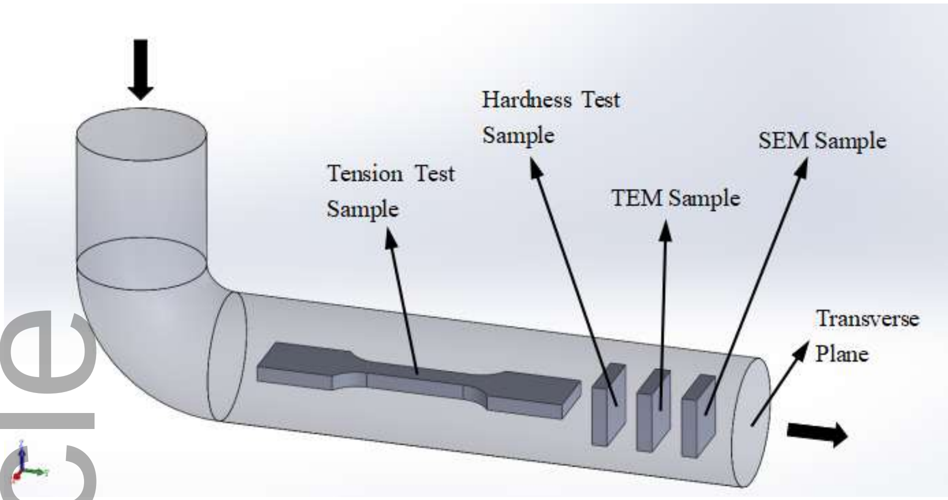


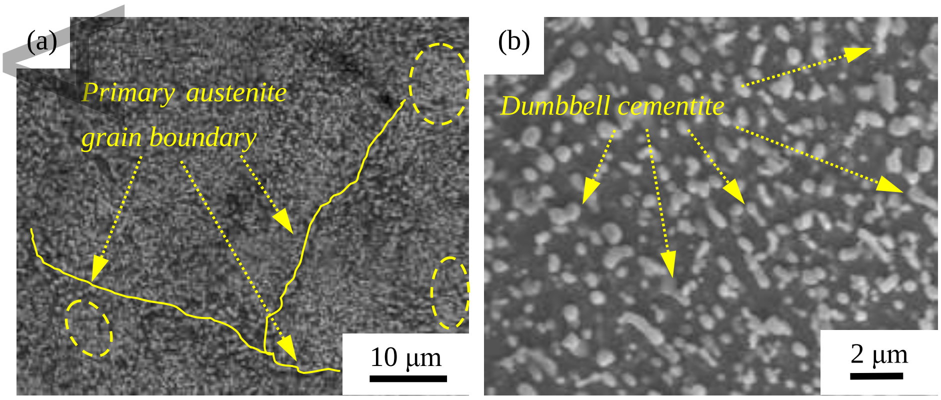
Figure 1. Sample geometries and orientations in the ECAP-processed steel

3. Results and discussion

3.1 Microstructure before and after ECAP

Figure 2 displays the measured SEM micrographs of the studied samples under different stages of ECAP application i.e. before and after passes of ECAP. As shown in Figure 2a, the cementite lamellae in the original granular pearlitic steel are mostly spheroidized with only a small fraction of remaining lamellae. The shape of the cementite is shown by a short bar with an average size of about 750 nm. After one pass of ECAP, because of shear strain, the cementite particle size becomes smaller and the corresponding structure changes from short bars to dumbbells. At the same time, however, the presence of large cementite particle is also noted with the size of ~450 nm. Figure 2b shows the elongation of the ferrite phase and refinements in the grains. After two passes, the cementite particle further fractures with reduced average size of ~300 nm. More cementite particles are distributed on ferrite matrix and the grain boundary of ferrite becomes relatively regular and straight, as also depicted in Figure 2c. After four passes, the shear strain increases, and the cementite particles were completely broken. In order to coordinate the severe deformation between ferrite and cementite, a large number of dislocations occur in ferrite. This provides a channel for the rapid diffusion of C atoms, prompting C atoms in the cementite to continuously enter the dislocation, and gather around the dislocation to form a Cottrell air mass, leading to the dissolution of the cementite [18,19]. Similar changes have also been reported in the experiments of lamellar pearlite steel after equal-channel angular pressing [10]. Further, along with a bimodal distribution in the cementite particle size, the size of cementite particles inside the ferrite grain is noted to be about 90 nm. Moreover, the bulky cementite particles are seen to be formed mainly at ferrite grain boundaries with a uniform size of about 1 μm, as can also be visualized in Figure 2d.

During the warm deformation of ECAPed samples, a huge number of dislocations were created to share deformations between the cementite and ferrite. The cementite' carbon atoms are expected to enter into those created dislocations, which further contribute to the formation of a Cottrell atmosphere. Such process has been reported to reduce the energy and therefore leads to a partial dissolution of the cementite [20,21]. This subsequently results in the appearance of the dynamic recovery and dynamic recrystallization. This further results in a decrease in the dislocation density and the re-precipitation of the cementite inside of the ferrite matrix. At the same time, due to the occurrence of the Ostwald ripening [22,23], this leads to a decrease in cementite particle numbers and increase of the particle sizes [24].



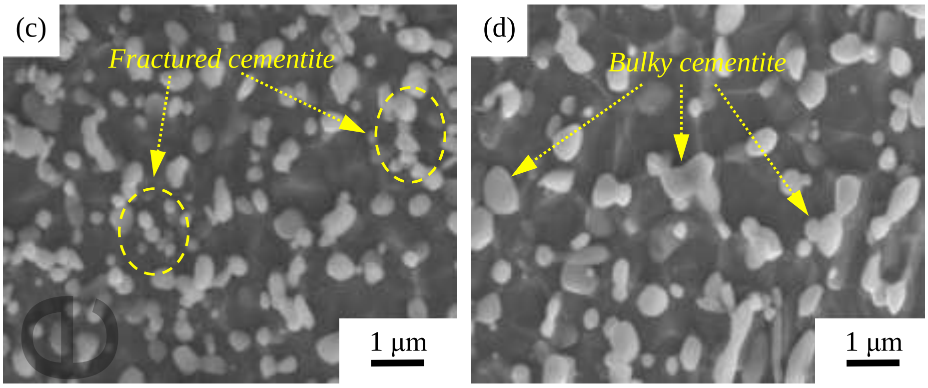


Figure 2. SEM micrographs of granular pearlitic steel before and after different passes of ECAP

(a) before ECAP; (b) after one pass; (c) after two passes; (d) after four passes

Figure 3 depicts TEM micrographs of granular pearlite steel under different stages of ECAP application. Before ECAP, majority of the cementite are observed to be fully spheroidized. However, a small number of cementite lamellae still exists in the short rods form along with an uneven particle size distribution, as also shown in Figure 3a. After one pass of ECAP, the cementite is noted to be evenly distributed in the ferrite matrix. Meanwhile, high-density dislocations are piled up as induced by severe deformation (Figure 3b). After two passes, the stacked high-density dislocations form the dislocation cells, which is also shown in Figure 3c. Such dislocation cells generally lead to the formation of sub-grains, which further refine ferrite grains. Moreover, the cementite has further spheroidized with the particle size of about 300 nm. The dislocation density after four passes is significantly lower than that of same after two passes of deformation. This is because when subgrains migrated from the small-angle to the large-angle grain boundary, a huge number of dislocations are expected to be expended along with strain accumulation [25,26]. The rate of dislocation annihilation is also higher than that of the dislocation proliferation. Meanwhile, because of the continuous dynamic recrystallization, a few fractions of fine grains are also noted to be increased with the increase in the ECAP pass numbers, and the corresponding microstructure gradually becomes homogeneous. The equiaxed ferrite grains have been found to show clear sharp boundaries with an average grain size of ~450 nm. The particle size of corresponding cementite is observed to be about 300 nm.

During the process of warm deformation, huge number of dislocations have been created with an increase in the ECAP passes because of the shear strain. This further supports the high-density tangled dislocation formation and accelerates the dissolution of the cementite. Furthermore, because of the continuous occurrence of dynamic recrystallization in the ferrite matrix, significant decrease in the dislocation density and re-precipitation of the cementite inside the ferrite matrix have been observed. It also suggests the refinement of the cementite particles. This phenomenon has widely been observed in warm deformations of carbon steel [27,28].

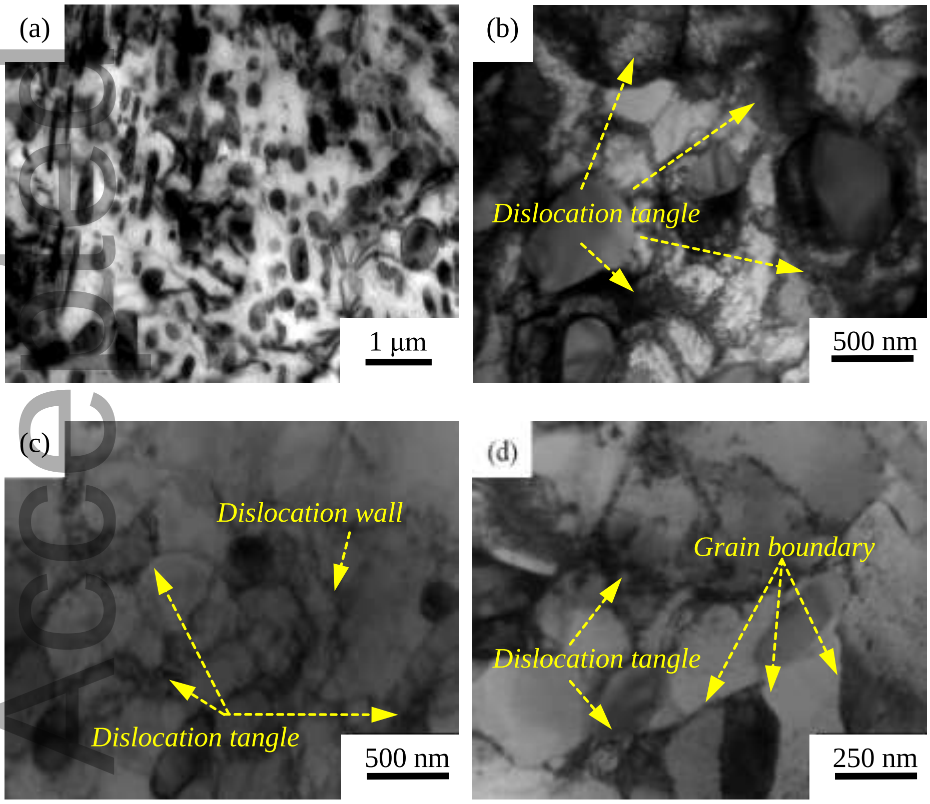


Figure 3. TEM micrographs of granular pearlite steel before and after different passes of ECAP (a) before ECAP; (b) after one pass; (c) after two passes; (d) after four passes

3.2 Mechanical Properties

Figure 4 represents the microhardness of granular pearlite steel under different stages of ECAP application i.e. before and after serial passes of ECAP. After one pass, the microhardness of granular pearlite steel shows an increase from 195 HV (before ECAP) to 241 HV at a rate of 24%. After two passes, the microhardness jumps to 41% i.e. 275 HV. Further, after four passes, the microhardness decreases to 258 HV, a 32% increment compared with the sample before ECAP.

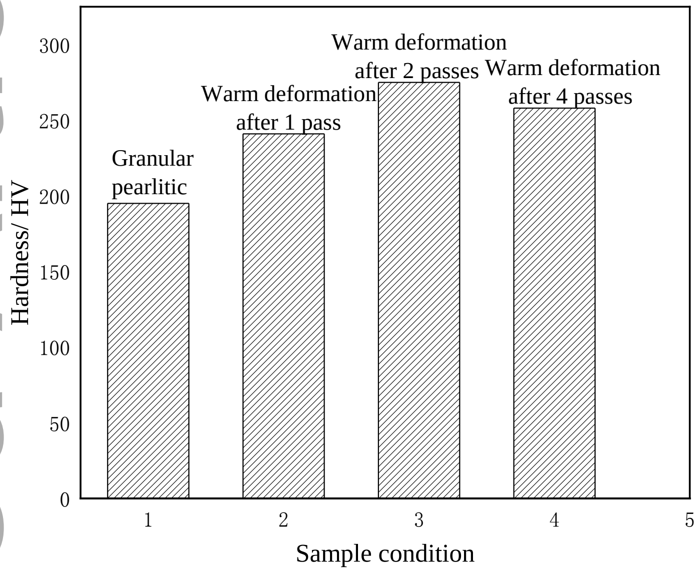


Figure 4. Microhardness of granular pearlitic steel before and after different passes of ECAP

As also envision in Figure 4, the microhardness of the granular pearlite steel show increase with the strain up to a peak value (two passes) followed a decrement after that. At the initial stage of deformation, the high-density dislocations were formed, and dislocations piled up, leading to refinements of the cementite and ferrite, which improved the microhardness. After two passes, the pile-up of dislocations is aggravated, and more cementite particles are spheroidized. Further, the ferrite is seen to be refined. Under the joint action of dislocation strengthening and grain refinement, the microhardness reached the maximum. After four passes, the rate of dislocation annihilation is higher than that of the dislocation multiplication [29]. Due to the dynamic recrystallization of the ferrite, the dislocation density has been reduced. Meanwhile, the grain refinement slightly decreases the microhardness of the sample, though the value is still higher than that of the original granular pearlite steel before ECAP application.

Measured engineering stress-strain curves for the granular pearlite steel under the different stages of ECAP application are plotted in the Figure 5. The tensile strength of granular pearlite steel shows an increase with the pass numbers. After two passes of deformation, the tensile strength reaches to its maximum value and then decline after that. Table 2 summarizes the mechanical properties of granular pearlite steel under the different stages of ECAP application (i.e. before and after serial passes of ECAP). As can be noted, after one pass, the tensile strength of granular pearlite steel increases from 589 MPa to 660 MPa. The tensile strength reaches to a maximum of 870 MPa after 2 passes. After four passes, the tensile strength slightly reduces to 782 MPa, which is still 31% more than that of the original granular pearlite steel before ECAP application. The yield strength increases with the application of ECAP passes. After four passes, the yield strength reaches to 649 MPa from an original value of 289 MPa, with an increase of about 125%. The yield ratio increases by 73% from 0.48 (before ECAP) to 0.83, which is mainly due to the grain refinement. Despite these merits, the elongation has been noted to decreased from 20.10% (before ECAP) to 16.80%.

The change in the mechanical properties is attributed to combined effects of work hardening, grain refinement and spheroidization of the remaining cementite lamellae. Work hardening improves the microhardness and tensile strength of pearlite steel. However, spheroidization of the remaining cementite lamellae and recrystallization of the ferrite reduce hardness during the warm deformation. During the initial stage of warm deformation, the work hardening effect causes an increase in the tensile strength and microhardness, a prior effect to the combined effect of recrystallization and spheroidizations. After two passes, the high-density dislocations have been noted to formed dislocation entanglements and dislocation cells, resulting in the peak tensile strength and microhardness. With further straining, the granular pearlite steel shows continuous dynamic recrystallization. In the process of warm deformation, the refined grain will grow to a certain extent, and the number of substructure defects such as dislocation in the deformed grain will also be reduced. Meanwhile, the remaining cementite lamellae tends to be fully spheroidized, and thus, the tensile strength and microhardness have been seen to be decreased quite a bit. The trend of plasticity is contrary to that of tensile strength. In line with present observation, the plasticity of granular pearlite is also reported to be better than that of the lamellar pearlite. The continuous increase in the yield strength and yield ratio have been attributed to the different microstructure observed under different stages of ECAP application i.e. different number of ECAP passes. Likewise, such phenomena have also been reported in eutectoid steel during hot deformation [30,31].

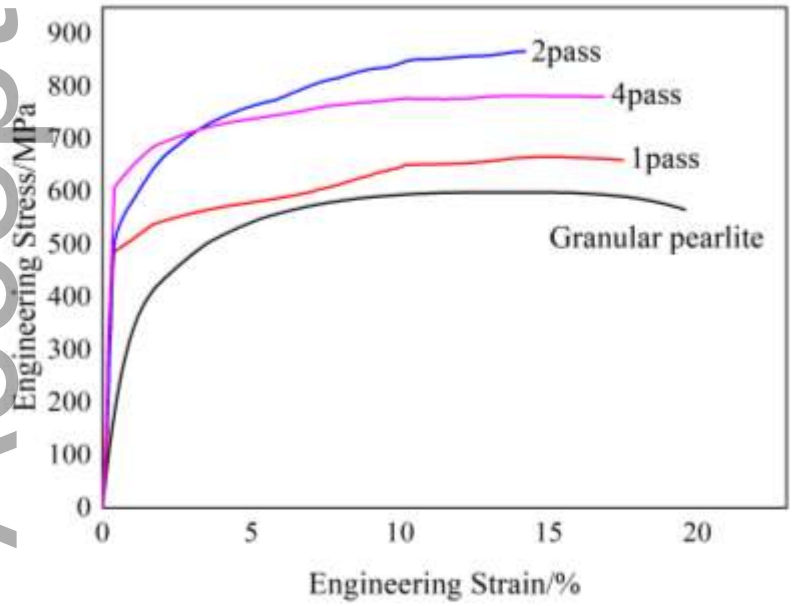


Figure 5. Engineering stress-strain curves of the mini-tensile specimens before and after ECAP

Table 2. Mechanical properties of granular pearlite before and after ECAP

	pearlite	1 pass	2 passes	4 passes
Hardness/ HV	195	241	275	258
Yield strength ($\sigma_{s(0.2)}$)/ MPa	289	491	590	649
Tensile strength (σ_b)/ MPa	598	660	870	782
$\sigma_{s(0.2)}/\sigma_b$	0.48	0.68	0.74	0.83
Elongation (δ)/ %	20.10	17.50	15.26	16.80

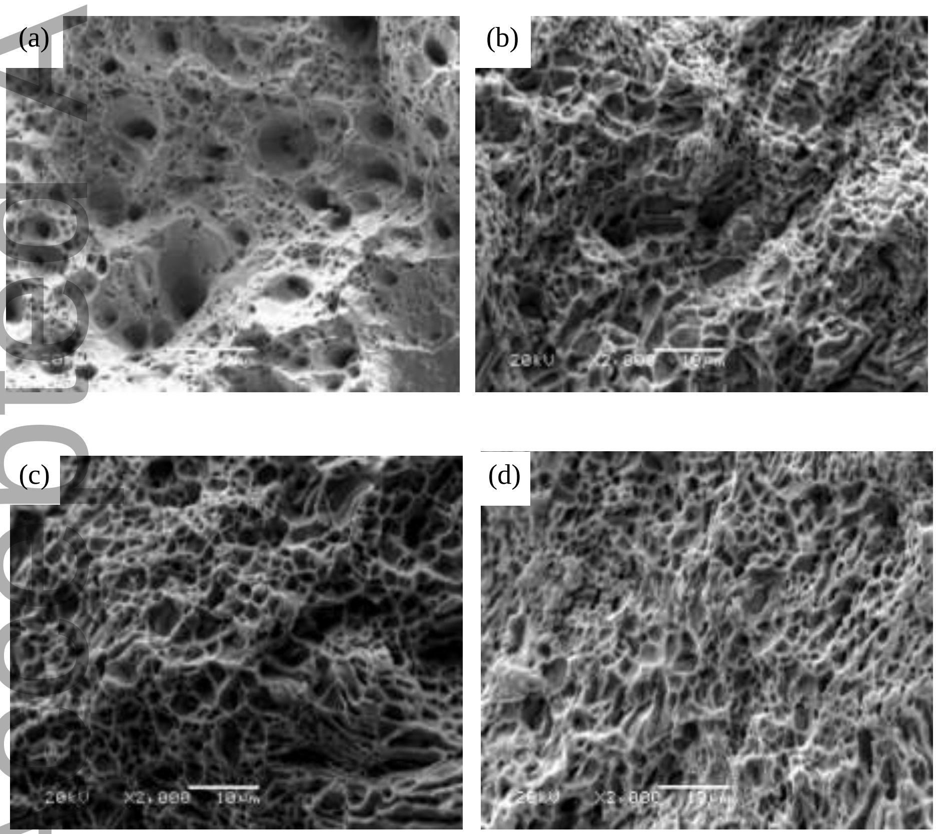


Figure 6. Fracture surface morphology of granular pearlite steel before and after different passes of ECAP (a) before ECAP; (b) after one pass; (c) after two passes; (d) after four passes

Figure 6 shows the fracture surface morphology characteristics of granular pearlite steel under different stages of ECAP application. From Figure 6a, the fracture is seen to be composed of huge number of large and deep dimples with an average size of about 10 μm, which also refer to a good toughness of the sample. After one pass, some small dimples turn out, as noted in Figure 6b. With the increase in ECAP passes, the number of dimples gradually increased, and the depth of the dimples have been decreased. The spheroidization of cementite increases, as also seen in Figure 6c. After four passes, the size and depth of these dimples are seen to be relatively uniform. The average size of about 6 μm is noted for a few large and deep dimples, while an average size of about 1 μm was seen for a huge number of small and shallow dimples distributed uniformly, as visualized in Figure 6d. During the warm deformation, with the increase in ECAP passes, the plastic deformation is aggravated and sizes of the ferrite and cementite are further refined down to sub-micron [32], resulting in the decrease in the dimple size and increase in the dimple number. Similar type of observations was also reported in pure Cu after ECAP at room temperature [33]. Compared with the original granular pearlite high carbon steel, the plasticity of ultra-fine grain high carbon steel slightly reduces.

4 Conclusion

To conclude, this work presents a systematic study of the impacts of warm ECAP passes on the microstructure and mechanical properties of granular pearlitic steel. The primary results are summarized as follows.

1. An ultra-microduplex structure of granular pearlite steel has been observed after four ECAP passes via Bc route with the characteristics of ~450 nm sized equiaxed ferrite grains and ~300 nm diameter sized cementite particles.

2. Due to the joint action of work hardening and the grain refinement, the microhardness and tensile strength of the granular pearlite were observed to be increased to a peak value (two passes) followed by a slight decrease. After two passes of ECAP, the microhardness and the ultimate tensile

strength increased from 195 HV and 598 MPa (before ECAP) to 275 HV and 870 MPa, respectively. The yield strength and the yield ratio increased from 0.48 and 298 MPa (before ECAP) to 0.83 and 649 MPa after four passes, respectively. However, the elongation was observed to be slightly decreased.

3. With increase in the ECAP pass application, significant grain refinement leads to a gradual change in tensile fracture morphology from a large number of deep dimples (before ECAP application) to many small dimples (after ECAP application).

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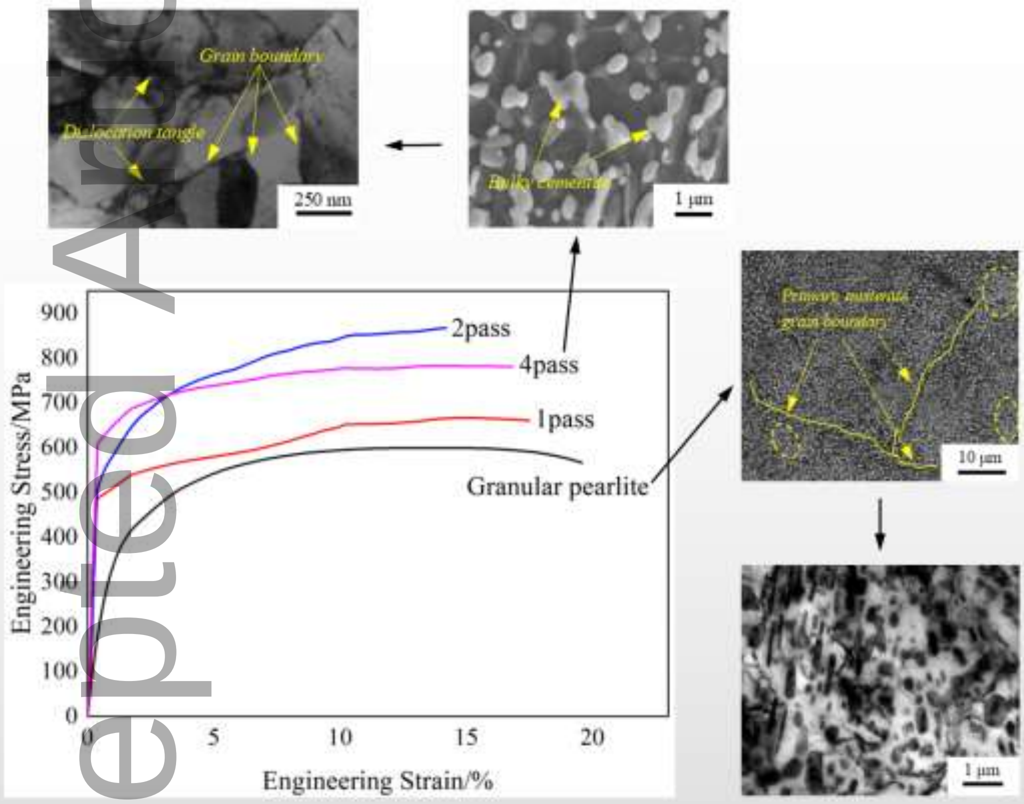
Preparation of ultra-microduplex structure by warm equal-channel angular pressing (ECAP) is one of the most effective methods for producing high quality granular pearlitic steel. This investigation provides the first-of-its-kind systematic study of microstructural evolution and its correlation to the mechanical property of the granular pearlitic steel under various ECAP passes.



Impacts of warm equal-channel angular pressing on microstructure and mechanical properties of granular pearlitic steel