Microstructural Development and Mechanical Properties of Multi-pass Cold Drawn Hypereutectoid

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Abstract. In the present work, processes of multi-pass cold drawn were conducted on hypereutectoid pearlite steel wires with carbon content of 1.02 w.t.%. Relationship between microstructural development and mechanical properties of hypereutectoid steel wires after drawing was investigated. It was found that when the strain was relatively small, lamellar morphology of the pearlite in the steel wire after cold drawn remained, and the spacing of lamella was refined. With the increase of the compression ratio, coordinated deformation of ferrite and cementite occurred and pearlite lamella were bended and folded. As the strain increased, fibrous morphology of pearlite lamella was observed in longitudinal section of the wire. High magnification TEM observation revealed the fragmentation of cementite and a large number of nano-crystalline. Tensile strength, microhardness and torsion strength of the steel wires after multi-pass drawn process increased significantly with the increase of accumulated strain. The results of XRD analysis show that the <110> peak of the ferrite shifted to the left and widened following the multi-pass cold drawn deformation, indicating that the lattice constant of ferrite was increased. The increase of lattice constant of ferrite was the result of the distortion of ferrite lattice due to the dissolution of carbon atoms.

1. Introduction

Owing to its abundant resources, low cost, excellent mechanical performance, high strength and good ductility, hypereutectoid steel wires have been widely used in piano string[1], automobile tires[2] and suspension bridge cable[3].

To further improve the mechanical properties of hypereutectoid steel wires, much effort has been made to investigate the microstructural evolution and its effect on the mechanical property of the wires. The strength of cold drawn steel wires can be effectively increased by the refinement of interlamellar spacing, the increment of carbon content, the addition of alloying elements, and the increase of deformation strain[4-5].

It was shown that with the drawing process going, the orientation of ferrite and cementite platelets turned to drawn axial gradually, followed by the decrease of pearlite spacing[6]. Finally, the pearlite lamellar will be refined as the fibrous drawn morphology[7]. In addition to pearlite lamellar refinement, dislocation multiplication in ferrite was also proposed by Lenasson et al.[8].

Pearlite is a two-phased, lamellar structure composed of alternating layers of ferrite and cementite. It has been recently emerged revealing that cementite lamellae also exhibited remarkable ductile
behavior during drawing[9]. In the process of drawing, in addition to the changes in the spacing between the pearlite laminates, there will also be some coordination deformation between ferrite and cementite[10].

It was found that cementite platelets were divided into nanometer-sized grains as a result of the dissolution of cementite after the serious deformation of wires[11-12]. Under further drawing, the amorphous cementite will be found and distributed in the carbon-rich region between ferrite grains[13-14].

In this article, the changes of microstructure and mechanical properties of multi-channel pull-out hypereutectoid steel wire were analyzed. And the relationship between cementite recrystallization and mechanical properties of the hypereutectoid steel wires were discussed.

2. Experimental methods
Materials used in this study were hot-rolled hypereutectoid steel rods with a composition in the mass fraction (%) of Fe-1.02C-0.297Cr-0.215Si-0.31Mn-0.0041S-0.0089P. After pickling and phosphating, the rods (5.5 mm in diameter) were cold drawn to 1.35 mm in diameter by multi-pass drawing process (the total reduction of 75.5 %, $\varepsilon=2.38$). The temperature of hypereutectoid steel wires during drawing was below 100 °C.

The torsion tests of steel wires were performed on CTT500 torsion tester at room temperature with a rotating speed of 720 °/min. The length of the test wires is 50 times of the wire diameter. Both the torque and torsional angle were recorded in real time. Five identical samples were tested under each condition.

For microstructural observation, after standard grinding, polishing and electro-polishing were applied to prepare samples, the specimens were etched by 4% nitric acid alcohol solution and examined using FEI Siron-400 scanning electron microscope (SEM).

TEM samples were prepared by GATAN 691 Precision Ion Polishing System (PIPS) after the thickness of the samples was mechanically thinned to below 40 µm. Sample was cooled by liquid nitrogen in PIPS to avoid the temperature rise. Ion angle and energy were set to 3° and 2 Kev at the final stage of TEM sample preparation to minimize microstructure damage.

3. Results
3.1 Microstructure of hypereutectoid steel wires
Fig. 1 shows the microstructures of the cross-section (a and b) and longitudinal-section (c and d) of the hypereutectoid steel wire. The strain is 0, 1.34, 0, 1.34 in sequence. In fig. 1 a and c, it can be seen that the lamellar structure of hypereutectoid steel wire consists of the white part cementite and the black part ferrite, who stay parallel to each other. And the grain orientations of the pearlite are homogeneous. The selected areas in fig. 1 marked by the red circular line show the microstructure evolution of the lamellar structure. It can be seen from fig. 1 b and d that with the increase of the strain, the pearlite interlaminar spacing decreases gradually, and the ferrite and cementite coordinate deform violently. Additionally, the lamellar structure is bent or broken into pieces for those who perpendicular to the drawing direction as can be seen in fig. 1 b and d, while it shows the fibrous morphology for those who parallel to the drawing direction as can be seen in fig. 1 d.

Fig. 2 presents TEM micrographs of pearlite morphology. The strain is 0, 1.34, 0, 1.34 in sequence. Fig. 2 a and b are the bright-field image of the hypereutectoid steel wires, of which white part is cementite and the black part is ferrite, show the dislocation arrangement after large strain. A number of high density dislocation cells were observed in ferrite lamellae. It is worthy to be mentioned that, not only the ferrite lamellae, but also the cementite lamellae have a reduction in thickness with the increase of the strain. In addition, it has a different reduction level of thickness in different areas. Fig. 2 c and d are the dark-field image of the hypereutectoid steel wires, of which present the evolution of fractured cementite. It can be seen that a number of nano-crystal cementite is distributed in cementite lamellae unevenly. Meanwhile, cementite lamellae were bent due to stress concentration caused by
these dislocations. It can be observed in fig. 2 d that the different deformation degree of the cementite lamellae is associated to the amount of fractured cementite. As is marked in the red box line, the area which has the high density fractured cementite has the lower deformation degree.

**Figure 1.** SEM images of hypereutectoid steel wires. (a) and (b) The cross-section morphology. (c) and (d) The longitudinal-section morphology. (strain: a, c=0; b, d=1.34)

### 3.2 Mechanical properties

Table 1 presents the torsion test results of multi-pass drawing steel wires. Fig. 3 shows the engineering strain-torsional strength/turns curves of the multi-pass cold drawn treated steel wires. It can be seen from table 1 that the strain rates go along with the increase of drawing turns, which results in the reduction of diameter of steel wire. It can be seen from fig. 3 that with the strain increase from 0 to 2.81, torsional strength increases from 1126.83 MPa to 2527.22 MPa. As is shown in fig. 3 the black broken line, the variation trend of torsional strength is increasing, which means the work hardening rate of hypereutectoid steel wires is improved with the increase of strain. It is worthy to be mentioned that the torsional turns firstly increased from 15 to 20 turns and then decreased to 13 turns with the increase of strain, which is different from the direct proportional relationship between engineering strain and torsional strength during the whole multi-pass drawing process.
Figure 2. TEM micrographs of cold drawn hypereutectoid steel wires. (a) and (b) Bright-Field image. (c) and (d) Dark-Field image (strain: a, c=0; b, d=1.34)

Table 1. The torsion test results of multi-pass drawing steel wires

| Strain | Diameter (mm) | Torsional turns | Torsional strength (MPa) |
|--------|---------------|-----------------|--------------------------|
| 0      | 5.5           | 15              | 1126.83                  |
| 0.66   | 3.96          | 18              | 1298.54                  |
| 1.34   | 2.81          | 20              | 1586.85                  |
| 2.2    | 1.83          | 14              | 2000.16                  |
| 2.81   | 1.35          | 13              | 2527.22                  |

Figure 3. Engineering strain-torsional strength/turns curves of the multi-pass cold drawn treated steel wires
4. Discussion

4.1 Microstructure of hypereutectoid steel wires
It can be seen from fig. 1 and fig. 2 that with the increase of the strain, the pearlite interlaminar spacing decreases gradually, and the ferrite and cementite coordinate deform violently. And the lamellar structure is bent or broken into pieces for those who perpendicular to the drawing direction, while it shows the fibrous morphology for those who parallel to the drawing direction. Meanwhile, a number of high density dislocation cells were observed in ferrite lamellae, while a number of nano-crystal cementite is distributed in cementite lamellae unevenly.

It can be draw from the above experimental results that with the increase of the strain, a number of high density dislocation cells and nano-crystal cementite were formed in ferrite lamellae and cementite lamellae, respectively, which directly resulted in the deformation of pearlite. Those grains who parallel to the drawing direction show the fibrous morphology, while those who perpendicular to the drawing direction is bent or broken into pieces.

4.2 Mechanical properties
It can be seen from fig. 3 that with the strain increase from 0 to 2.81, torsional strength increases from 1126.83 MPa to 2527.22 MPa, while the torsional turns firstly increased from 15 to 20 turns and then decreased to 13 turns with the increase of strain. It can be observed in fig. 2 that with the increase of strain, a number of high density dislocation cells were observed in ferrite lamellae, which directly results in the increase of torsional strength. However, with the dislocation density increases above the yield point, as a result of crack formation that resulted in torsion delamination, the torsional turns decrease from 20 turns to 13 turns.

5. Conclusion
With the intention of investigating the microstructure evolution and mechanical properties of hypereutectoid steel wires in a large strain range, the high carbon steel wires with a carbon content of 1.02 wt.% is investigated. The conclusions are summarized as follows:
1. With the increase of the strain, the pearlite interlaminar spacing decreases gradually, and the ferrite and cementite coordinate deform violently. Additionally, the lamellar structure is bent or broken into pieces for those who perpendicular to the drawing direction, while it shows the fibrous morphology for those who parallel to the drawing direction.
2. A number of high density dislocation cells were observed in ferrite lamellae. It is worthy to be mentioned that, not only the ferrite lamellae, but also the cementite lamellae have a reduction in thickness with the increase of the strain. In addition, it has a different reduction level of thickness in different areas.
3. It can be seen that a number of nano-crystal cementite is distributed in cementite lamellae unevenly. Meanwhile, cementite lamellae were bent due to stress concentration caused by these dislocations. Different deformation degree of the cementite lamellae is associated to the amount of fractured cementite.
4. The strain rates go along with the increase of drawing turns, which results in the reduction of diameter of steel wire. With the strain increase from 0 to 2.81, torsional strength increases from 1126.83 MPa to 2527.22 MPa. The variation trend of torsional strength is increasing, which means the work hardening rate of hypereutectoid steel wires is improved with the increase of strain. The torsional turns firstly increased from 15 to 20 turns and then decreased to 13 turns with the increase of strain, which is related to the high density dislocation cells deformed after drawing.

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