

TEM ANALYSIS OF NANOMETER MULTI-LAYER STRUCTURE OF LOW CARBON STEEL

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In general considering, the special properties that nanostructured materials possess are mainly decided by the large quantity of existing grain boundaries [1]. Moreover, the substructure of nanocrystallines plays an important role, too. There are still different opinions on the substructure of nanocrystallines. By simulation, some held the idea that there is little or no dislocation in nanostructured materials [2]. However, electro-depositing nanometals analysis made by TEM showed that the substructure of nanocrystallines is still dislocation [3]. Obviously, to study the structure and the substructure of nanocrystallines is significant for probing into the essence of nanocrystallines deeply, revealing the special properties mechanism of nanostructured materials and developing nanostructured materials.

In the study of the structures of nanostructured materials, block full-compact nanocrystallines should be fabricated. In recent years, the research on full-compact block nanocrystalline-Ni, nanocrystalline-Cu and their super plasticity has been reviewed [4-5], but the substructures of them haven't been reported. Especially for traditional steels, grain size has been up to about 1-5 μm through super refinery [6-7]. However, some reports considered that 1 μm or so is a critical size for super refinery of steel materials to exceed [8]. It is of great

importance for full-compact steel materials to break through 1 μm limit to grain refinery, proceed to realize nanometrication, and further to research their structures and substructures.

In this study, low carbon steel plate that has nanometer multi-layer structure was fabricated by quenching-cold rolling low carbon steel. The structure and the substructure of the nanocrystallines are studied by TEM analysis method.

1. Experimental procedure

Commercial 15 steel is chosen. After electro slag remelting, and heat treatment at 950 $^{\circ}\text{C}$, quenched into ice salt water and then aged at 200 $^{\circ}\text{C}$, low carbon martensite is obtained. According to 85% total pressure, 15 steel is used, and rolled many times to form steel plate of 2mm thickness. After aging at 350 $^{\circ}\text{C}$ x2h, thin sheets of 0.5mm thickness are cut down by electrodischarge machining (EDM) along ST(short transverse) of the plate, which is thinned to about 50 μm mechanically. Then the steel sheets are polished at double-jet-electrolysis polishing device, thus TEM specimens were made. In this experiment, solution is 10% perchloric alcohol, voltage of 30V was applied, polishing temperature is 20 $^{\circ}\text{C}$, H-800 TEM was used to observe and analyze the specimens.

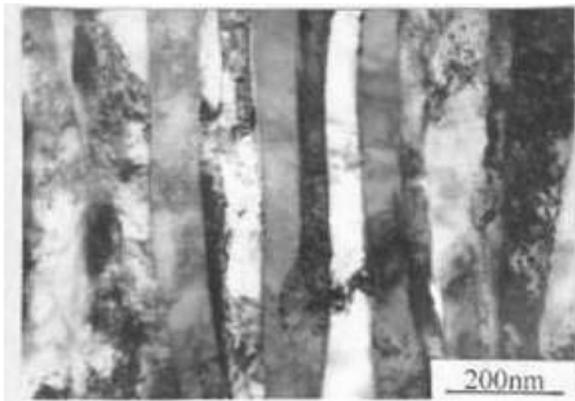


Fig 1-1. Stripe nanostructure on the STface

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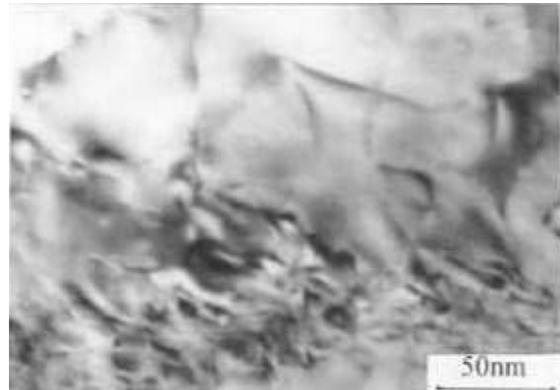


Fig 1-2. Dislocation structure on the longitudinal face

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2. Experimental results and analysis

Fig.1 is 15 steel's TEM image on the longitudinal face and the ST face after 85% cold rolling-aging. Fig.1-1 is the morphology at the ST face. As indicated in Fig.1-1, grains become stripe morphology, and the grain width whose average size is about 75nm has reached nanometer level. On the longitudinal face, the oriented dislocation distributed in the great size grain as shown in Fig.1-2. The formation of this structure is because that after the quenching of low carbon steel, lath martensite tissue can be obtained, and the average width of the lath is about 0.2 μ m. Thin pancake grain shape is formed after being cold rolled-aged; the average

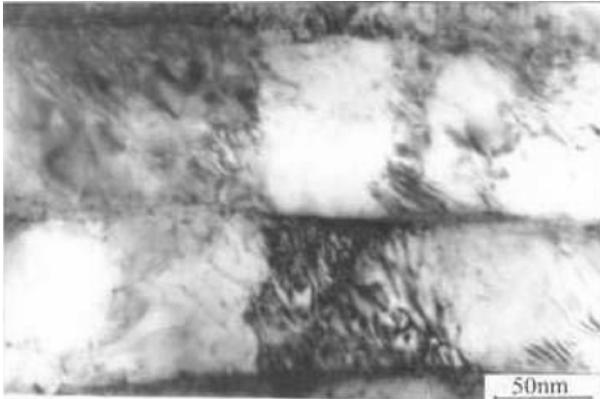


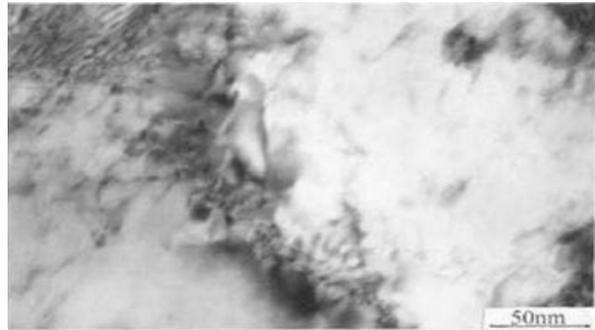
Fig 2. Dislocations and sub cells in nano-structures

length of great-sized grains in the direction of longitudinal ace was 1000nm, at the ST face, the lath width reduces to about 80nm, and the multi-layer structure that possesses nanosize in one-dimensional direction was formed.

Nanocrystalline substructure morphology of the rolling sheet's lateral face can be seen in fig.2. The substructure inside grains is still dislocation. In a same grain, sub cells were formed by the division of the dislocation. The size of the sub cells is between hundreds of and tens of nanometers. Fig.3 is the dislocations morphology on the longitudinal face. As shown in fig.3-1, dislocation density is lower, cell walls are formed in the place of dislocation concentration, and the grains on two sides of the cell wall have greater misorientation. In fig.3-2, dislocation density is higher and dislocations are distributed evenly. No cell wall is formed. Dislocation's Burgers vector was measured being the type of $1/2\langle 111 \rangle$ by the method bright field conjunct dark field.

As indicated in fig.4, large quantities of Mohr stripes appear on the longitudinal face.

The cause is that after great deformation



materials are rolled into thin pancake grain shapes. In the direction of rolling thickness,

Fig 3-1. Cell walls and the morphology of dislocations

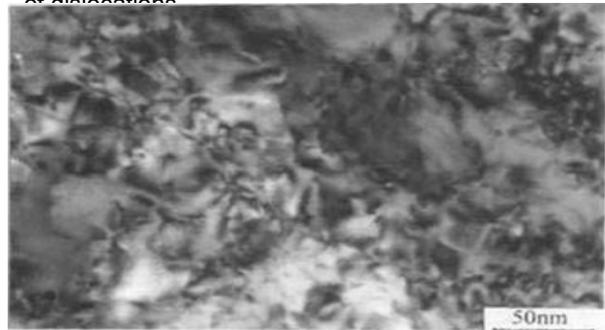


Fig 3-2. High-density dislocations

grains are nanometricated. So there are two layers grains of different orientation in the TEM

samples of 200-300 nanometer thickness. Their different crystal faces overlap so as to form new great cycle structure. As a result, such Mohr stripes occur in the TEM image.

Fig.5 is X-ray diffraction and selected-area electron diffraction pattern of multi-layer structure. It can be seen that this kind of

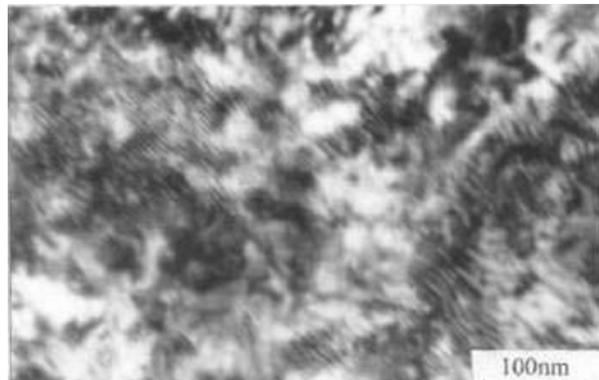


Fig 4. Mohr stripes on the longitudinal face

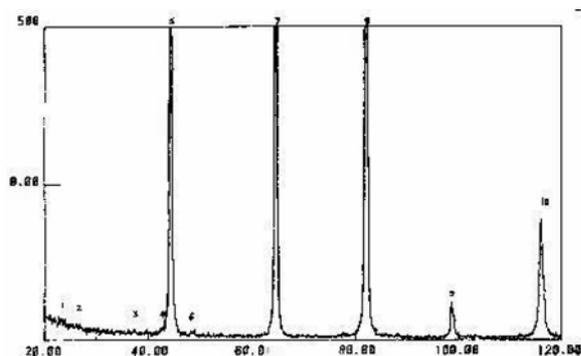


Fig 5-1. X-ray diffraction chart

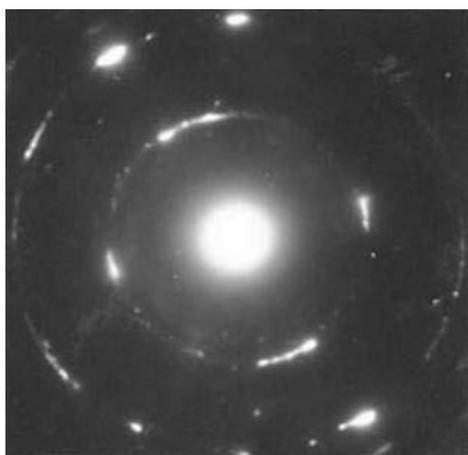


Fig 5. Diffraction analysis of nanometer multi-layer structure, material possesses bcc structure and {100}<110>texture

nanocrystalline still remains body-centered cubic structure, and what's more, due to cold-rolling, material forms deformed texture whose type is {100}<110>.

4. Conclusions

After cold rolling-aging, 15 steel nanometer multi-layer structure was formed. The atoms still arrange in body-centered cubic and the substructure is mainly dislocation whose Burgers vector is $1/2\langle 111 \rangle$ type. The sub cells were formed by concentrated dislocation at both the longitudinal face and the ST face.

REFERENCES

1. Glitter H, Prig Mater Sic, 1989, 33:223.
2. Schitz J., Di Tolla F.D, Jacobsen K.W: Nature, 1998, 391:561.
3. Chen R.S, Cuo J.T, Yin W.M., Zhou J.Y.: Acta Mater Sin 1998,34:1121.
4. McFadden S.X., Mishra R.S., et.al: Nature, 1998, 3 91:684.
5. Lu L, Sui M.L, Lu K.: Science, 2000, 287: 1463.
6. Yang Wangyue, Hu Anmin, Sun Zuqing: Acta Metal Sin, 2000, 43:21.
7. Belykov, H. Miura, T. Sakai: Scr Mater, 2000, 43:1060.
8. Maki T., Tessu Hagane, 1995, 81:547.

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