



# Annealing-induced Hardening in a Nanostructured Low-carbon Steel Prepared by Using Dynamic Plastic Deformation

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Lamellar nanostructures were induced in a plain martensitic low-carbon steel by using dynamic plastic deformation at room temperature. The nanostructured steel was hardened after annealing at 673 K for 20 min, with a tensile strength increased from 1.2 GPa to 1.6 GPa. Both the remained nanostructures and annealing-induced precipitates in nano-scale play key roles in the hardening.

**KEY WORDS:** Nanostructure; Annealing; Precipitation hardening; Low-carbon steel; Dynamic plastic deformation

## 1. Introduction

Plastic deformation has been proven to be an effective approach to strengthen metallic materials due to the strain-induced grain refinement and high density of defects. The grain size of ferrite steel can be reduced to 100–200 nm by using plastic deformation methods, such as high pressure torsion (HPT)<sup>[1]</sup>, equal-channel angular pressing (ECAP)<sup>[2]</sup>, accumulative roll-bonding (ARB)<sup>[3]</sup>. These ultrafine-grained (UFG) ferritic steels exhibit an enhanced tensile strength as high as 1 GPa. After annealing, strengths of these UFG ferritic steels usually decrease monotonously with an increasing annealing temperature<sup>[4,5]</sup>. In the plain low-carbon steels with lath martensite, nano-scale lamellar structures were obtained through cold rolling, resulting in the tensile strength as high as 1.5 GPa<sup>[6]</sup>. Consistent with the UFG ferritic steels mentioned above, softening was also observed in the nanostructured low-carbon steel with martensite after annealing. The annealing-induced softening was usually attributed to a decrease of defect density and microstructure coarsening at elevated temperatures.

Nanostructures were obtained in various metals and alloys by using dynamic plastic deformation (DPD)<sup>[7,8]</sup>. In a DPD-

processed nanostructured plain low-carbon steel, we found annealing-induced hardening, rather than softening as usually observed in deformed metals. The objective of the present study is to identify the origin of such an annealing-induced hardening.

## 2. Experimental

A commercial plain low-carbon steel with a carbon concentration of 0.2 wt% was used in this work. Its chemical composition is listed in [Table 1](#). The as-received material was annealed at 1173 K for 2 h and then quenched in water to obtain the microstructure of lath martensite. The initial samples before DPD treatment are cylinders of 10 mm in diameter and 17 mm in height. The details of DPD set-ups and process can be found in our previous work<sup>[7,9]</sup>. During DPD processing, the cylindrical samples are compressed with impact loading repeatedly. The DPD strain is calculated by the expression  $\epsilon = \ln(h_0/h)$ , where  $h_0$  and  $h$  are the initial and final heights of DPD samples, respectively. The strain rate of each impact is estimated to be  $10^2$ – $10^3$  s<sup>-1</sup>. In this work, the low-carbon martensitic steel sample was deformed to a strain of 1.7 by using DPD treatment at room temperature. Three groups of DPD samples were annealed at 673 K, 773 K and 873 K for 20 min, respectively. The water quenched samples without deformations (WQ for short) were also annealed with corresponding parameters for comparison.

Transmission electron microscopy (TEM) was used to characterize the microstructures of the steel samples, with a JEOL JEM-2010 microscope operated at 200 kV. The TEM samples were cut parallel to the loading directions and prepared with

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**Table 1** Chemical composition of the low-carbon steel studied in this work (wt%)

C	Mn	Si	S	P	Ni	Cr	Cu
0.205	0.510	0.73	0.0050	0.0190	0.05	0.7	0.14

twin-jet. Back-scattered electron (BSE) signals in scanning electron microscopy (SEM) are used to obtain electronic channeling contrast (ECC) from the electrochemically polished surfaces which depend on the orientation of grains in the matrix and could show annealed microstructures clearly for large areas. Dog-bone tensile samples with the gauge dimensions of 5 mm × 1 mm × 0.5 mm were cut perpendicular to the loading directions, which means parallel to the surface of samples. The tensile tests under a strain rate of  $5 \times 10^{-3} \text{ s}^{-1}$  were conducted on an Instron 8848 micro-tester with a laser extensometer at room temperature.

### 3. Results

#### 3.1. Microstructures of the DPD steel with nanostructures

The microstructure of the DPD samples with a strain of 1.7 is characterized by lamellae with an aspect ratio of 15, as shown in Fig. 1(a). The lamellae are parallel to each other and perpendicular to the loading direction. Dislocation slipping dominates the plastic deformation and microstructure refinement in the martensitic low-carbon steel during DPD and deformation twinning is not found. The average thickness of lamellae is 84 nm calculated by Gauss fitting with the thickness distribution in Fig. 1(b). Nanometer scaled microstructures are obtained which resemble those obtained in cold rolled martensitic low-carbon steels<sup>[6]</sup>, and are finer than those in the ferritic low-carbon steels processed by plastic deformations such as ECAP<sup>[2,5]</sup> and ARB<sup>[3]</sup> etc. The nanostructures obtained during DPD are mainly with the benefits of the initial lath microstructures scaled in several hundreds nanometers and the supersaturated carbon in the matrix. High density of dislocations, another typical feature of the cold deformed metals, is also observed accumulating on the lamellar boundaries and tangling in the spaces between the boundaries. It has been confirmed in other metals that higher strain rate in DPD than that in conventional deformation methods with lower strain rate is beneficial to the

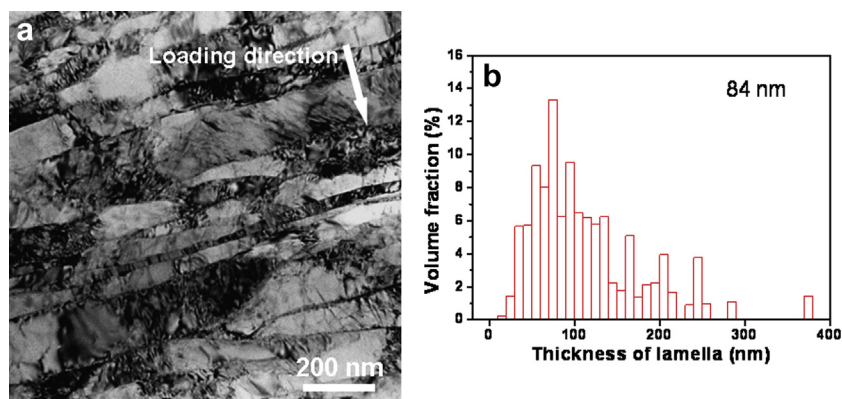
formation of higher defect density and finer microstructures in the matrix<sup>[10,11]</sup>.

#### 3.2. Microstructures of annealed samples

Microstructural observations of the annealed samples were conducted with SEM on the WQ and DPD samples (Fig. 2(a) and (b)) for comparisons. Characteristic size distributions of microstructures of DPD samples are shown in Fig. 2(c). The microstructures of WQ samples do not change much after annealing. Sub-microstructures in the regions of original austenite grains can be observed in the samples annealed at 673 K and 773 K. Fully recrystallized microstructures are obtained after annealing at 873 K for 20 min, but the grain size keeps nearly unchanged. In contrast, remarkable changes of microstructures take place in the DPD samples after annealing. The lamellar structures still exist after annealing at 673 K for 20 min. The average thickness of lamellae measured with SEM-ECC images is about 111 nm. The detailed observations under TEM (Fig. 3(a)) show that there is no obvious change in microstructure. The average thickness of lamellae is 86 nm, identical to that of the as-deformed state. High density of dislocations induced by the DPD is still remaining. Carbide particles with the size of several nanometers were observed at the lamellar boundaries and inside the lamellae, as arrowed in Fig. 3(b). When annealed at 773 K, recrystallization takes place uniformly and equiaxed grains are obtained. The average grain size is about 242 nm. The grain size distributions of equiaxed grains (in DPD samples annealed at 773 K and 873 K) were weighted with the square of grain sizes. Some of ultrafine grains grew into coarse grains embedded in the ultrafine-grained matrix of the sample annealed at 873 K. Carbide particles could be observed with SEM, which are arranged in line perpendicular to loading directions in coarse grains or pinned at the grain boundaries of ultrafine grains.

#### 3.3. Tensile properties and annealing-induced hardening

The as-quenched samples exhibit a lower strength and a higher tensile ductility (Fig. 4(a)) compared with those in martensitic low-carbon steels reported in literature<sup>[6,12]</sup>. This is attributed to a long holding duration (2 h) at 1173 K before water quenching, for ensuring a sufficient high ductility for cold deformation and uniform distribution of carbon in the matrix.



**Fig. 1** A typical cross-sectional TEM bright-field image of the DPD martensitic plain low-carbon steel (a) and the thickness distribution of lamellar structures measured from cross-sectional TEM bright-field images (b).

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