

## Surface nanostructure formation mechanism of 45 steel induced by supersonic fine particles pombarding

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**Abstract:** By means of supersonic fine particles bombarding (SFPB), a nanostructured surface layer up to 15  $\mu\text{m}$  was fabricated on a 45 steel plate with ferrite and pearlite phases. To reveal the grain refinement mechanism of SFPB-treated 45 steel, microstructure features of various sections in the treated surface were systematically characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Grain size increases with an increase of depth from the treated surface. Plastic deformation and grain refinement processes are accompanied by an increase in strain. Plastic deformation in the proeutectoid ferrite phases has precedence over the pearlite phases. Grain refinement in the ferrite phases involves: the onset of dislocation lines (DLs), dislocation tangles (DTs) and dense dislocation walls (DDWs) in the original grains; the formation of fine lamellar and roughly equiaxed cells separated by DDWs; by dislocation annihilation and rearrangement, the transformation of DDWs into subboundaries and boundaries and the formation of submicron grains or subgrains; the successive subdivision of grains to finer and finer scale, resulting in the formation of highly misoriented nano-grains. By contrast, eutectoid cementite phase accommodated strain in a sequence as follows: onset of elongated, bended and shear deformation under deformation stress of ferrites, short and thin cementites with a width of about 20-50 nm and discontinuous length were formed. Shorter and thinner cementites were developed into ultra-fine pieces under the action of high density dislocation and strains. At the top surface, some cementites were decomposed under severe plastic deformation. Experimental evidences and analysis indicate that surface nanocrystallization of 45 steel results from dislocation activities, high strains and high strain rate are necessary for the formation of nanocrystallites.

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**Key words:** surface nanocrystallization; grain refinement mechanism; microstructure; 45 steel

### 1. Introduction

In most cases, the failures of engineering materials initiate from the materials surface, and are very sensitive to the structure and properties of the material surface. Therefore, optimization of the surface structure and properties may effectively enhance the global behavior of materials. With the increasing evidences of unique properties for nanocrystalline materials, it is reasonable to achieve surface modification to improve significantly the overall properties and behavior of the materials by the generation of a nanostructured surface layer. Surface self-nanocrystalline [1], which was referred in 1999 by Lu, is to transform the original coarse-grained surface layer of a bulk material into nano-sized grains while keeping the overall composition and shape of materials unchanged, which will greatly enhance the surface properties. Such a surface

nanocrystallization will provide a new approach to the application of nano-technology in engineering materials. In addition, many conventional surface treatment techniques are applicable for synthesizing nanostructures in the surface by modifying the processing parameters, such as shot peening, hammer peening, surface rolling.

The previous investigations demonstrate that surface mechanical treatment is an effective approach to create surface self-nanocrystalline [2]. By means of surface mechanical treatment, the nanostructured surface layers have been obtained on annealed materials and low hardness materials including pure metals (Fe, Al, Cu, and Ti) and stainless steel [3-5]. Supersonic fine particles bombarding (SFPB) [6] is a kind of new mechanically induced surface self-nanocrystallization device. Because of high efficiency and flexibility, the

SFPB set-up is suited for surface nanocrystalline of components with large surface area and complex shapes, so that it possesses potential industrial application value.

This study aims at studying the grain refinement mechanism of hypoeutectoid 45 steel possessing two-phase structures of proeutectoid ferrite and pearlite during the SFPB process. The investigation is of interest from an academic point, because it has two-phase structures, whereas all the ferrous metals studied up to now have a single-phase structure (Fe, stainless steel). In single-phase Fe, the dislocation glide is the main deformation mode and it has been found to dominate the grain subdivision process down to the nanometer scale by surface mechanical treatment [7]. However, detailed investigation on surface nanocrystalline of 45 steel with pearlite and ferrite phases remains open. So developing an understanding of the origin and mechanism of grain refinement has intrinsic merit. A 45 steel plate was treated by SFPB, by observing the microstructure feature at different depth from the deformed surface, the microstructural evolution was examined to establish the mechanism of grain refinement and strain accommodation during SFPB.

## 2. Experimental procedures

The material used in this investigation was a hypoeutectoid 45 steel plate of 6 mm thickness, with the following chemical composition (wt%): C 0.42, Mn 0.65, Si 0.27. The initial structure is a bcc ferrite and a pearlite with grain sizes in the range of 10-30  $\mu\text{m}$ , and the distance between segments in pearlite is about 0.4-0.5  $\mu\text{m}$ . The samples of 20 mm $\times$ 10 mm $\times$ 6 mm were prepared for the SFPB treatment.

The SFPB set-up and procedure refers to the published report [6]. The main parameters of the SFPB process were chosen as follows: the pressure of air-flow was about 1.5 MPa; the temperature of airflow was about 40-50°C; the diameter of steel shot was 0.4-0.6 mm; and the processing duration was 150 s.

The microstructural evolution of samples was characterized by different techniques. Cross-sectional observations of the treated 45 steel samples were performed on a QUANTA200 scanning electron microscope (SEM). The cross-sections were mechanically polished, and finally etched at room temperature in a 4% nital. X-ray diffraction (XRD) of the surface layer in the SFPB treated sample was carried out on D/MAX2400 X-ray diffractometer with Cu K $\alpha$  radiation, in the step scan mode. The average grain size and mean microstrain were calculated in terms of the dif-

fraction line broadening of bcc ferrite (110), (200), (211), (220), (310), and (222) Bragg reflection peaks, using the Scherrer-Wilson equation [8]. Using repeated electrochemical etching, the treated surface layer was removed layer-by-layer, so that the microstrain evolution along the depth from the treated surface was determined by means of XRD analyses. Transmission electron microscopy (TEM) investigation was carried out on a JEOL-2000FX microscope operating at 160 kV. Plane-view and cross-section thin foils for TEM observations were prepared by means of cutting, grinding, dimpling, and a final ion thinning at low temperatures.

## 3. Result and discussion

### 3.1. Cross-sectional observations

The cross-sectional SEM microstructures of the SFPB-treated specimens are shown in Fig. 1. It can be seen that the plastic deformations on the surface are produced, and plastic flows in the deformation region are found, which indicates that the plastic deformation mode of 45 steel is mainly dislocation slip. The plastic deformation is inhomogeneous along the depth. On the top surface, deformation is very severe, and a layer of ultra-fine structure is formed close to the top surface. With increasing depth, a severe deformation layer is gradually replaced by single slips lines unto free deformed region. Fig. 1(b) shows a contrast of the super-fine structure layer on the top surface of an SFPBed specimen to the matrix structure, which shows that the structure of top surface is evidently fined, and it is difficult to distinguish phase interface of ferrite and pearlite, which is outside the resolution of SEM. The thickness of the deformed layer is about 60-70  $\mu\text{m}$ .

### 3.2. Grain size and depth

XRD, TEM and SEM analyses are used to determine the grain (or cell) size. Fig. 2 shows the measured average grain (or cell) size and microstrain as functions of depth in the SFPB treated sample. The grains were remarkably refined into the nanometer regime (<100 nm) within the outer surface of about 15  $\mu\text{m}$  thick. The submicron grains and subgrains are present in the sub-surface of the layer of about 15-40  $\mu\text{m}$  deep regime. Grain size increases with an increase of depth from the treated surface. The microstrain determined from XRD analysis decreases significantly along the depth in the surface layer and gradually drops to zero at about 70  $\mu\text{m}$  deep. It can be noted that the gradient structure results from a gradual decrease in the applied strain and strain rate from the top surface layer to the matrix. Microstructure characteristic

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