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Journal of Alloys and Compounds 434–435 (2007) 290–293

Journal of ALLOYS AND COMPOUNDS

www.elsevier.com/locate/jallcom

Role of strain gradient on the formation of nanocrystalline structure produced by severe plastic deformation

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Available online 22 September 2006

Abstract

In the present study, the formation of nanocrystalline structure by high speed drilling was investigated. Several micrometers thick nanocrystalline layer was observed at the top surface of drilled hole. Nanocrystalline layers showed high hardness and good thermal stability. The estimation of necessary dislocation density to produce nanocrystalline structure and the comparison between the estimated and measured strain gradient near the drilled hole surface was conducted. It is proposed that deformation with a large strain gradient is an important condition to produce nanocrystalline structure.

Keywords: Nanostructured material; Microstructure; Strain gradient; Severe plastic deformation

1. Introduction

Nanocrystalline materials, as a new class of materials with potentially new and superior properties, have attracted considerable scientific interest in the last few decades. To produce the nanocrystalline structure, various severe plastic deformation methods have been proposed. However, among these processes, nanocrystalline structure is obtained only by those with large strain gradient such as ball milling [1,2], high-pressure torsion (HPT) [3,4], particle impact deformation [5] and shot peening [6,7]. It has been gradually recognized that nanocrystalline structure cannot be produced by ECAP or ARB in which homogeneous deformation occurs. This suggests that deformation with large strain gradient is a critical condition for the formation of nanocrystalline structure. In the present study, high speed drilling was employed to produce the nanocrystalline structure near the drilled hole surface. The dislocation density for nanocrystalline structure formation and strain gradient near the drilled hole surface is estimated.

2. Experimental procedures

The material used in the present study was an Fe–0.56% C steel. Before drilling, the specimen was austenized at 1273 K for 3.6 ks and quenching into ice water to obtain martensite structure (7.8 GPa in Vickers hardness). The drilling experiments were performed using a sintered carbide drill with diameter in 5.0 mm. The cutting speed was 80 m/min and feed rate was 0.05 mm/rev. Oil mist was used as coolant. The thermal stability of surface structure was studied by annealing the drilled specimen at 673 K for 3.6 ks. Specimens were characterized by SEM, TEM, XRD and Vickers microhardness tester.

3. Results

The cross-sectional microstructure near the drilled hole surface is shown in Fig. 1. Featureless layer is seen near the hole surface. The hardness of the featureless layer (10.6 GPa) is much higher than that of matrix (7.8 GPa) as shown in Fig. 1a. Fig. 1b–d show the high magnification SEM morphology at different depth from drilled hole surface (marked in Fig. 1a as b–d). Fig. 1b shows the top surface layer with featureless structure. Beneath the featureless layer, the structure containing deeply etched islands elongated along the cutting direction appears (Fig. 1c). The hardness of this layer (9.2 GPa) is lower than that of the top surface layer. From the XRD analysis, the amount of retained austenite at the drill hole surface was found to be increased substantially by drilling. This indicates that re-austenization at the hole surface occurred during drilling. The island structure is considered as retained ferrite and surrounding

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doi:10.1016/j.jallcom.2006.08.167

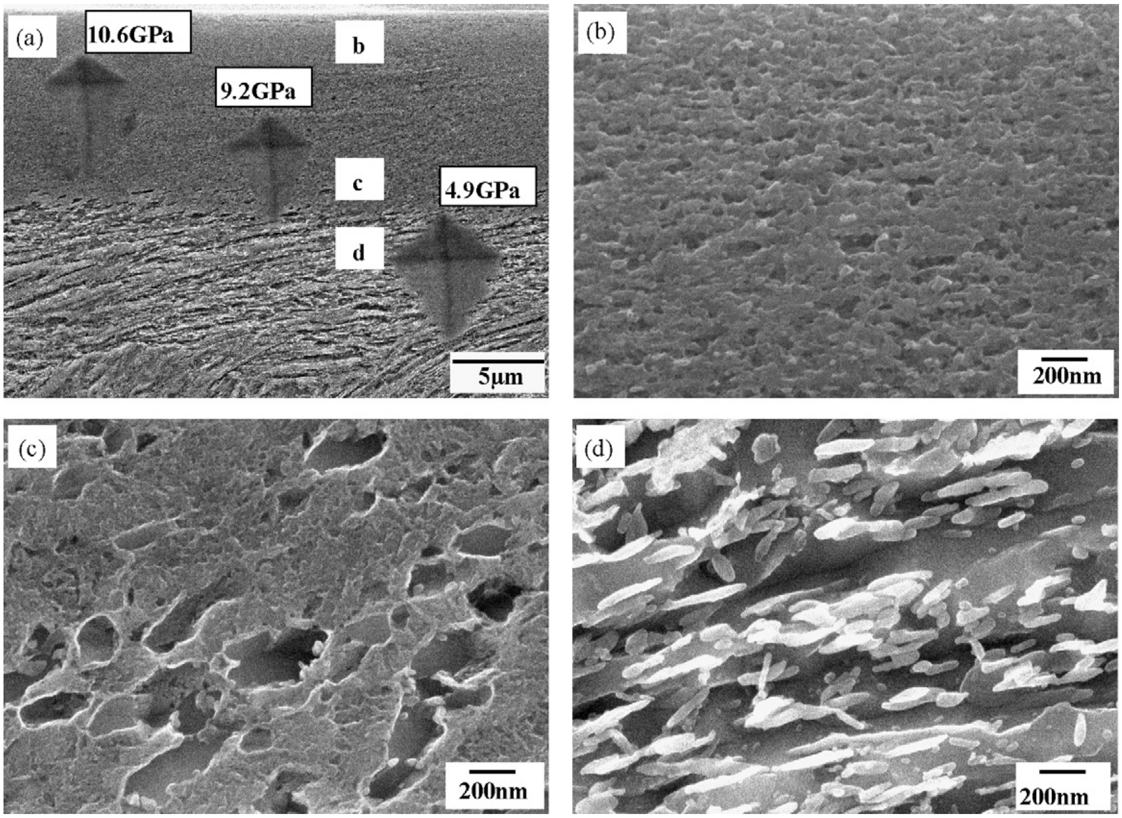

Fig. 1. SEM micrographs of drilled Fe–0.56% steel hole surface. (a) Low magnification with the hardness indentation, (b–d) show high magnification of areas marked in (a with b) featureless structure layer at the top surface, (c) islands layer underneath the feartureless layer, (d) rod like cementite particles at the elongated ferrite grain boundaries in recrystallized structure layer.

is re-austenized region. Fig. 1d shows the structure underneath the island layer which has lower hardness (4.9 GPa). Elongated submicron size ferrite grains with rod shaped cementite particles are at the grain boundaries. These structure features suggest that this area was heavily deformed and the temperature of this area was raised close to A_1 resulting in dynamically recrystallized ferrite structure. Fig. 2 shows TEM micrographs and a selected area diffraction (SAD) pattern (aperture of Ø 1.2 μm) of the featureless layer. Equiaxed grains with the size of about 20 nm are seen. The nearly continuous rings of SAD indicate that the grains are in random orientation. Fig. 3 shows SEM micrographs after annealed at 673 K. The re-austenitzed region in the as-drilled state is clearly divided into nanocrystalline and submicron layers. The nanocrystalline layer remaines featureless. In submicron grain layer, cementite particles with size of several 10 nm precipitated at the grain boundaries. In the recrystallized structure layer, there is almost no detectable change after annealing.

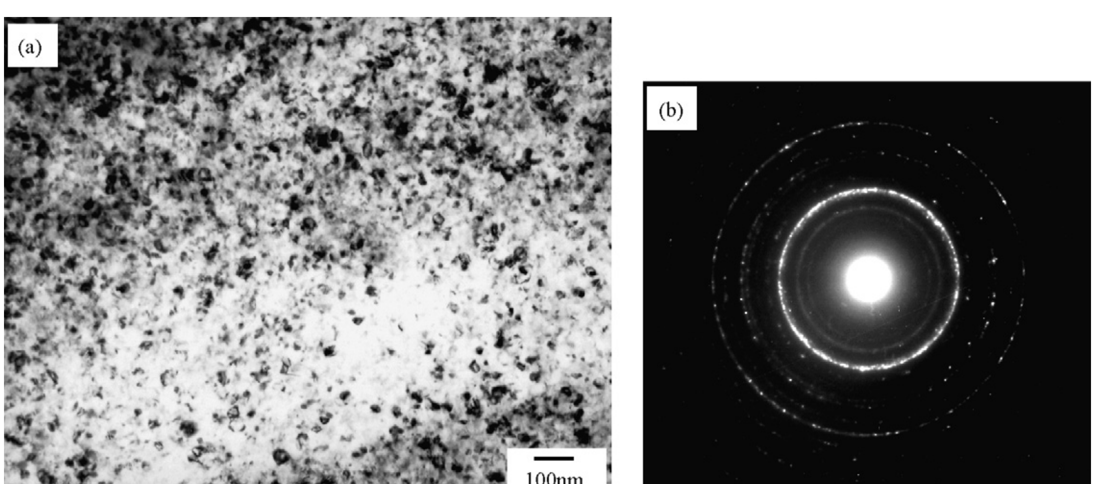

Fig. 2. (a) Bright field TEM image of the top surface of drilled hole in Fe–0.56% C steel, (b) the corresponding selected area diffraction pattern.

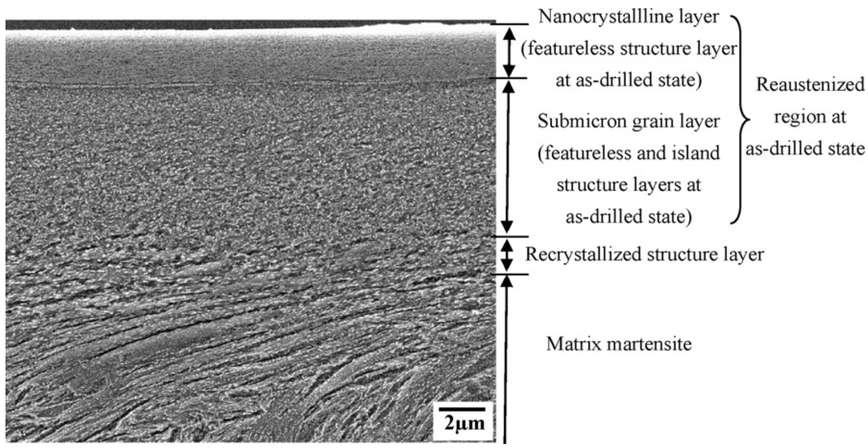

Fig. 3. SEM micrograph of drilled Fe–0.56% C steel after annealing at 673 K for 3.6 ks.

4. Discussions

The nanocrystalline structure formation mechanism by severe plastic deformation has received strong attention [1,4,5]. Tao et al. [6] studied the surface nanostructure in Fe produced by surface mechanical attrition treatment and proposed that the nanostructure formation involves formation of dense dislocation cell wall (DDW) and dislocation tangles (DT) in original grains and in the refined cells as well, and then evolution to highly misoriented grain boundaries. Yin et al. [2] suggested that the nanocrystlline ferrite formed through a transition from dislocation cell wall created by work hardening during ball milling of pure Fe to grain boundary. From those and other previous works, it has been commonly realized that storing high density of dislocation is a key to forming nanocrystalline structures by severe plastic deformation. Plastic deformation of metals occurs by the formation, movement and storage of dislocations. Theoretically, a correlation between deformation pattern and stored dislocations has been proposed by introducing the concept of statistically stored dislocation (SSD) and geometrically necessary dislocations (GND) [8,9]. The SSDs generally develop in homogenous deformation and accumulate by random mutual trapping. The recovery is easy to occur for SSDs since the same number of dislocations of different sign distribute randomly. However, the GNDs ensure compatibility of deformation and accommodate strain gradients in the case of nonhomogeneous deformation. The density of GNDs is directly proportional to strain gradient. Thus, high strain gradient is beneficial for obtaining high dislocation density and grain refinement during deformation. Therefore, the role of strain gradient seems quite important for the formation of nanocrystalline structure.

The strain gradient necessary to obtain nanocrystalline structure was roughly estimated as follows. According to literature [11], the grain boundary energy (E_{gb}) per unit volume of grains is expressed by

$$E_{\mathrm{gb}} = \frac{4\sigma}{\pi^{1/2} d} \tag{1}$$

where σ is the grain boundary energy per unit area and d is the grain size. The dislocation energy (E_{dis}) per unit volume of grains is given by

$$E_{\mathrm{dis}} = \rho \frac{\mu b^2}{2} \tag{2}$$

where ρ is the dislocation density , μ the shear modulus and b is the magnitude of a Burgers vector. It is assumed that all the dislocations introduced by the strain gradient are GNDs only and all the dislocation energy transfers into grain boundary energy, i.e., the ρ is equal to GND density (ρ_{G}) and $E_{\mathrm{gb}} = E_{\mathrm{dis}}$. The ρ_{G} with equivalent energy density to E_{gb} is therefore expressed as

$$\rho_{\mathrm{G}} = \frac{8\sigma}{\pi^{1/2} \mu b^2 d} \tag{3}$$

The strain gradient (χ) corresponds to this GND density [12] is

$$\chi = \rho_{\mathrm{G}} b = \frac{8\sigma b}{\pi^{1/2} \mu b^2 d} \tag{4}$$

For pure Fe, taking $\sigma = 0.6\,\mathrm{J/m^2}$, $\mu = 80\,\mathrm{GPa}$, $b = 0.25\,\mathrm{nm}$, the estimated strain gradient necessary to obtain nanocrystalline structure with grain size 100 nm is$1.4\,\mu\mathrm{m}^{-1}$.

Since most of the dislocation structures transform to grain boundary structures during drilling, GND density is not measurable experimentally. In the present study, the comparison between the estimated and measured strain gradient of the drilled hole surface was conducted as follows. Fig. 4a is a cross-section SEM micrograph near the drilled hole surface. The structure perpendicular to the drilled surface was selected. The magnitude of displacement (marked by dash curve in Fig. 4a) generated by drilling was measured. Following Heilmann et al. [10], an exponential function was adopted to describe the displacement of deformed structure. The selected coordinate axis is shown in Fig. 4a. Measured displacements (y) were fitted to the following equation:

$$y = y_0 \exp(-kx) \tag{5}$$

where y_0 is displacement at the top surface and k is a constant. The fitted equation and a corresponding curve are shown in Fig. 4b. The shear strain γ was obtained by differentiating y with depth x and shown in Fig. 4c. The strain gradient χ was

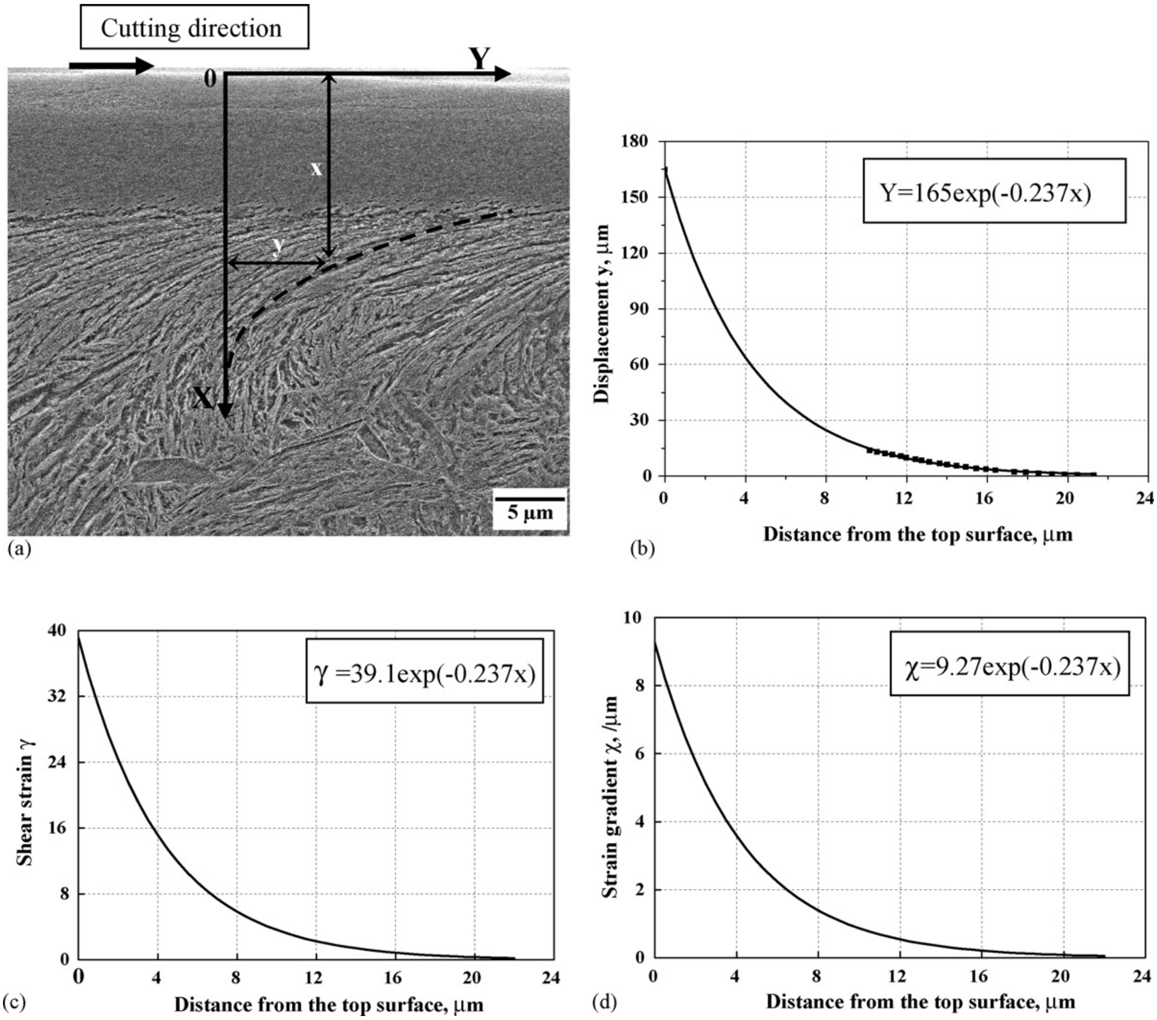

Fig. 4. Plastic flow structure near the drill hole surface in Fe–0.56% C steel. (a) SEM micrograph showing with flowed structure and coordinate system for analysis, (b), (c) and (d) the amount of displacement, shear strain and shear strain gradient as a function of depth, respectively.

obtained by differentiating γ with x and shown in Fig. 4d. The shear strain gradient at the top surface where 20 nm sized grains is observed is $9.27\,\mu m^{-1}$. This value corresponds well to the estimated strain gradient $7\,\mu m^{-1}$ for the grain size of 20 nm according to the above energy criterion. From the above calculation it is concluded that deformation with large strain gradients is an important condition to produce nanocrystalline structure. It is believed that the present analysis of the role of strain gradient on the formation of the nanocrystalline structures is also applicable to the other inhomogeneous deformation processes, such as shot peening and HPT.

5. Summary

Nanocrystalline structure layer several microns thick was obtained on the drilled hole surface by high speed drilling of Fe–0.56 C% steel in martensite structure. Nanocrystalline layers showed high hardness and good thermal stability. Estimated and measured strain gradients near the drilled hole surface show good correspondence. It is proposed that deformation with large strain gradient is an important condition to produce nanocrystalline structure by severe plastic deformation.

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