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NANOCRYSTALLIZATION OF CARBON STEELS BY SHOT PEENING AND DRILLING

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Abstract. The formation of nanocrystalline (NC) structure in steels by shot peening and drilling was investigated. NC layers, several microns thick, with grain size of around 20 nm were successfully produced in steels by both shot peening and drilling. By annealing, the NC structure showed substantially slow grain growth without recrystallization. Comparing shot peening and drilling, it was found that the NC structure formed irrespective of the deformation temperature. As a common deformation condition to produce the NC structure, a large strain gradient is proposed.

1. INTRODUCTION

Large efforts have been devoted to produce ultrafine-grained materials, especially nanocrystalline (NC, grain size smaller than 100 nm) materials, since the materials are expected to possess superior mechanical properties in the simple chemical compositions. To produce the NC materials, various severe plastic deformation processes have been proposed, such as ball milling (BM) [1], high pressure torsion [2], sliding wear [3,4] and shot peening (SP) [5-7]. From our previous BM [8,9] and SP [10,11] experiments in various steels, it was indicated that the NC structure regions have the following characteristics: (1) equiaxed grains of around 20 nm, (2) extremely high hardness, (3) separated from adjacent deformed structure regions with sharp boundaries and (4) no recrystallization and substantially slow grain growth by annealing. In our recent study [12], it was found that such NC structure with grain size of a few 10 nm formed by the intense plastic deformation of drilling. It was also reported that the NC layers formed by other cutting processes [13], e.g. turning [14] and reaming [15].

In the present study, the formation of NC structure in steels by SP and drilling are compared, and the common condition to produce the NC structure by deformation is discussed.

2. EXPERIMENTAL PROCEDURES

The materials used in this study were Fe-0.10%C, Fe-0.16%C, Fe-0.23%C, Fe-0.45%C and Fe-0.56%C (in mass %) with martensite structure. The martensite structure was obtained by austenitized at 1273K for 3.6 ks and quenched into ice water. The Fe-0.56%C steels tempered at 673 and 873K

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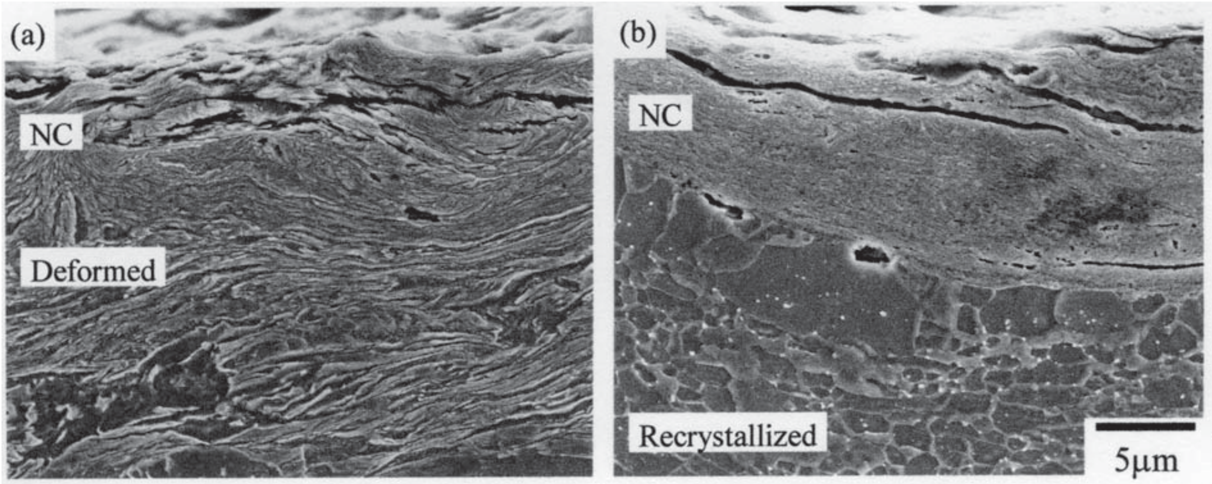

Fig. 1. The SEM micrographs of the NC layer formed in the as-quenched Fe-0.10%C steel after (a) SP (Coverage: 10000 % (Shot peening time: 100 s)) and (b) subsequent annealing at 873K for 3.6 ks.

for 3.6 ks were also used, and their Vickers hardnesses after tempering were 4.5 GPa and 2.7 GPa. The air blast SP was carried out by using the shots of < 50 μm in diameter (Shot material: cast steel (Fe-1.0%C, HV 6.9 GPa), Shot velocity: 190 m/s, Coverage: 6000 – 10000%). Coverage is the area fraction of specimen surface deformed by shot bombarding. The coverage higher than 50% was estimated by multiplying SP time by percent coverage for one second measured at the state of 50% coverage. The drilling was performed by using the sintered carbide drills with 2.5 mm and 5.0 mm in diameter. The cutting speed was 20 m/min and 80 m/min, and the feed rate was 0.05 mm/rev. Oil mist was used as coolant. Specimens were characterized by SEM, TEM and Vickers microhardness tester. The specimens etched by 5% Nital were observed by SEM.

3. RESULTS

3.1. Shot peening

Fig. 1 is the SEM micrographs of the as-quenched Fe-0.10%C steel after SP and subsequent annealing. It can be seen that the near-surface region is significantly deformed (Fig. 1a). In this region, precipitation of cementite particles is hard to observe, indicates that the temperature rise by deformation is not significant. Fig. 2 shows the TEM dark field

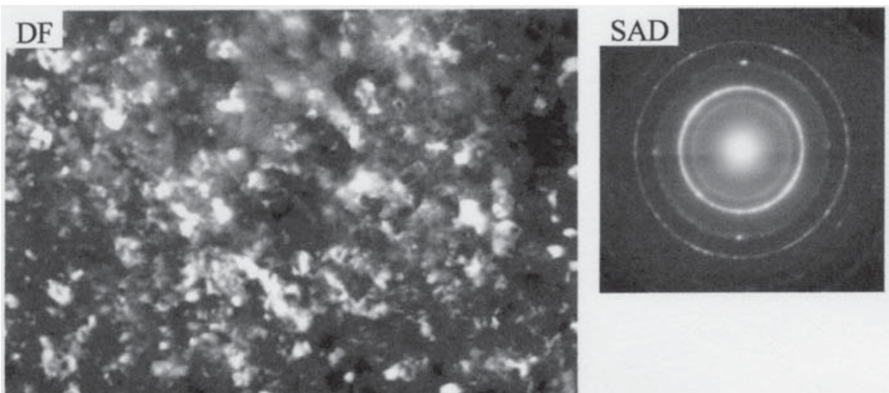

Fig. 2. The TEM DF image and SAD rings (∅ 1.2 μm in aperture size) of the NC layer formed in the as-quenched Fe-0.10%C steel after SP (Coverage: 6000% (Shot peening time: 60 s)).

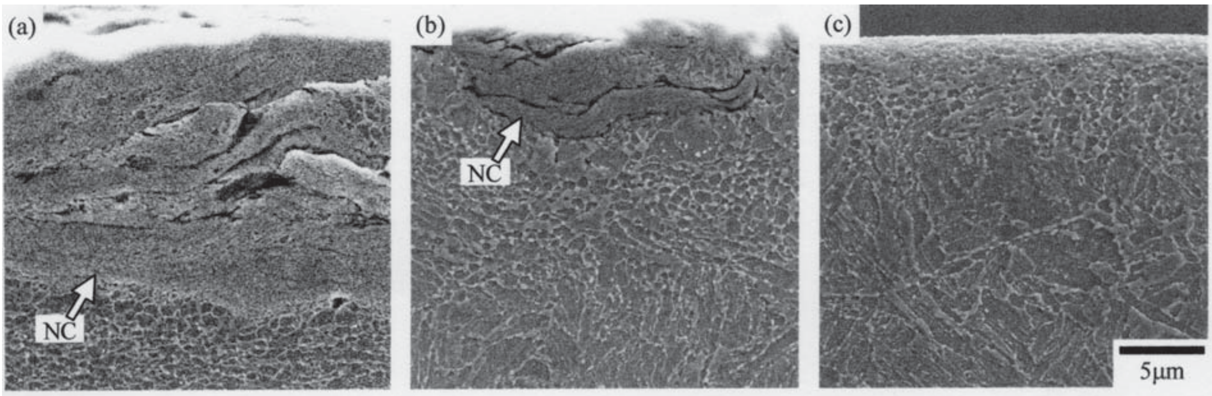

Fig. 3. The SEM micrographs of the NC layers formed in the as-quenched martensite steels of (a) Fe-0.16%C (HV 3.5 GPa), (b) Fe-0.23%C (4.6 GPa) and (c) Fe-0.45%C (7.1 GPa) after SP (Coverage: 10000 % (Shot peening time: 100 s)) and subsequent annealing at 873K for 3.6 ks.

(DF) image and selected area diffraction (SAD) rings taken from the top surface region in the Fe-0.10%C steel after SP. The DF image shows that grain size is around 20 nm. The random orientation of grains is confirmed from the uniform contrast of SAD rings. This TEM investigation indicates the shot-peened surface region is clearly nanocrystallized. After annealing at 873 K for 3.6 ks (Fig. 1b), the NC layer kept ultrafine grained structure, while recrystallization took place at the deformed structure region. The NC layer was separated from the recrystallized structure region by a sharp boundary.

In the SP experiments [10,11], it was found that the NC layer was produced by SP irrespective of the type of steels (composition (Fe-C, Fe-Si) and microstructure (ferrite, martensite, pearlite, spheroidite)). The increasing in the kinetic energy per one shot, such as the increasing in the shot velocity and/or the shot size, was effective to increase the thickness of NC layer. It is also found that the NC layer can be obtained when the initial specimen hardness is lower than that of shot. Fig. 3 shows the microstructures of the as-quenched martensite steels, Fe-0.16%C (HV 3.5 GPa), Fe-0.23%C (4.6 GPa) and Fe-0.45%C (7.1 GPa), after SP and subsequent annealing at 873K for 3.6 ks. In these specimens, the thickness of NC layer decreased with the increasing in the initial hardness. In the hardest Fe-0.45%C specimen (Fig. 3c), the recrystallized structure layer was only seen at the top surface with several microns thick. The hardness of shot used in this experiment was HV 6.9 GPa, wherefore the specimens with higher hardness than shot hardness were difficult to deform.

3.2. Drilling

Fig. 4 is the SEM micrographs of the drill holes in the as-quenched Fe-0.56%C steel with different cutting speed. At low cutting speed (Fig. 4a), only a deformed structure layer is seen at the top surface. While, a featureless structure layer about 10 μm thick is seen at high cutting speed (Fig. 4b). It is also found that the featureless structure layer forms when the initial hardness of specimen is high. Fig. 5 shows the microstructures of the hole surface in the Fe-0.56%C steels with different hardness after drilling. The featureless structure layer increased with initial hardness of specimen.

Fig. 6 is the SEM micrograph showing the hardness indentations near the drill hole surface of as-quenched Fe-0.56%C specimen. The hardness of the featureless layer near the top surface was 11.3 GPa. This hardness at the top surface was much higher than that of the as-quenched specimen (7.8 GPa). The hardness decreased with depth and became minimum of around 5.0 GPa at the inner region adjoining to the featureless layer. At the further inner region, the hardness increased with depth and saturated to the starting value (7.8 GPa). Fig. 7a is the low magnification SEM micrograph showing the entire area at the hole surface affected by drilling, and Figs. 7b – 7d are the enlarged SEM micrographs taken at different depth marked in Fig. 7a. Fig. 8a shows the TEM DF image taken from the surface region. The DF image shows equiaxed NC structure with grain size of around 20 nm, is similar to the NC structure formed by SP. Beneath the NC region, submicron-crystalline (SMC) island structure is observed as shown in Fig. 7c. The hardness

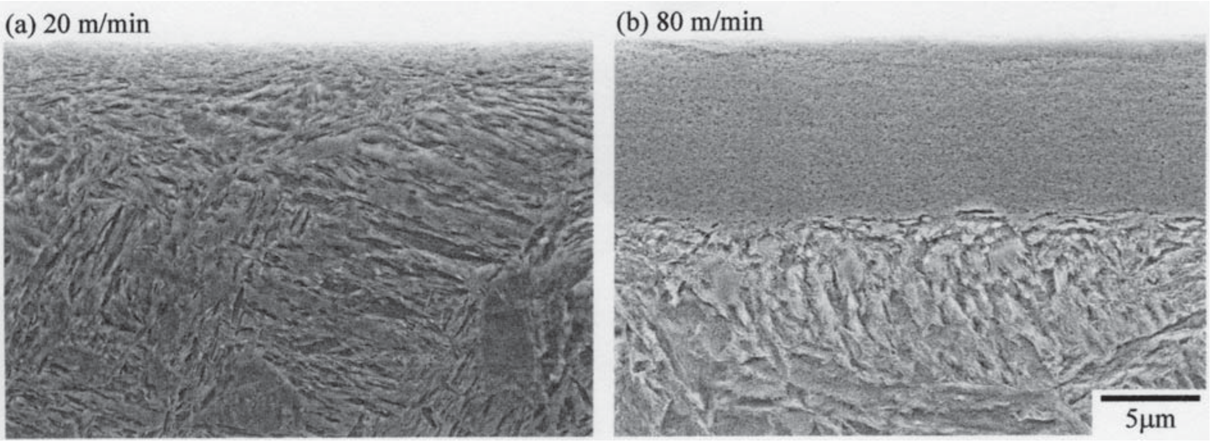

Fig. 4. The SEM micrographs of the drill hole surface in the as-quenched Fe-0.56%C steel after drilling (Cutting speed: (a) 20 m/min and (b) 80 m/min, Drill diameter: 5.0 mm).

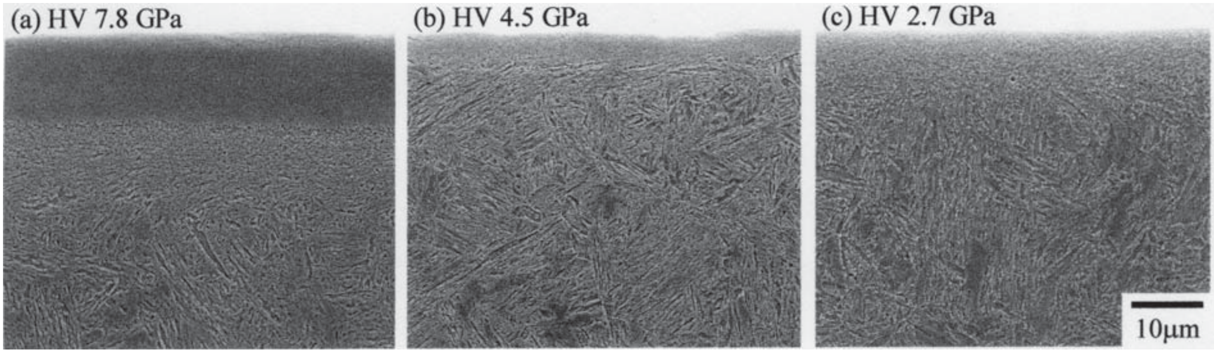

Fig. 5. The SEM micrographs of the drill hole surface in the Fe-0.56%C steel after drilling (Cutting speed: 80 m/min, Drill diameter: 5.0 mm). (a) As-quenched (HV 7.8 GPa), (b) tempered at 673K for 3.6 ks (4.5 GPa) and (c) tempered 873K for 3.6 ks (2.7 GPa).

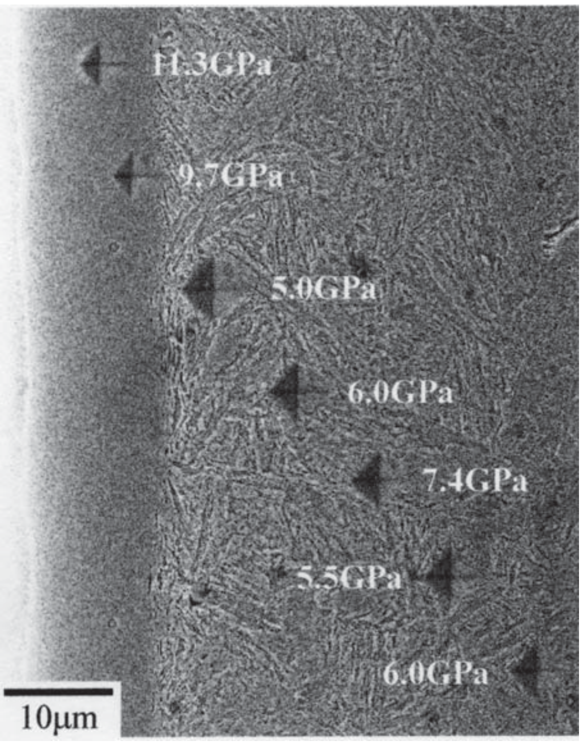

Fig. 6. The microstructure and hardness of the drill hole surface in the as-quenched Fe-0.56%C steel after drilling (Cutting speed: 80 m/min, Drill diameter: 5.0 mm).

of this region (9.7 GPa) was slightly lower than that of the top surface NC region. In the TEM bright field (BF) image taken from the island structure region (Fig. 8b), the equiaxed grain with size of around 100 - 200 nm and twined martensite structure (marked by circles) can be seen. In Fig. 7c, the SMC island structure consists of the deeply etched and the smooth surface area. It is considered that the smooth surface grains correspond to re-austenitized grains. (The re-austenitized grains are difficult to etch in comparison with the ferrite grains due to much carbon content probably.) As shown in Fig. 7a, the island structure starts to appear at the region of around 8 μm in depth from the drill hole surface and extends to around 15 μm, and the volume fraction of re-austenitized grains with smooth surface decreases with the depth. It is considered, therefore, that the maximum temperature decreased with the depth. The highest temperature reached was Ac_3 at the area of 8 μm deep and Ac_1 at the 15 μm deep from the surface. It is assumed that the maximum temperature of the featureless structure region near the top surface was much higher than

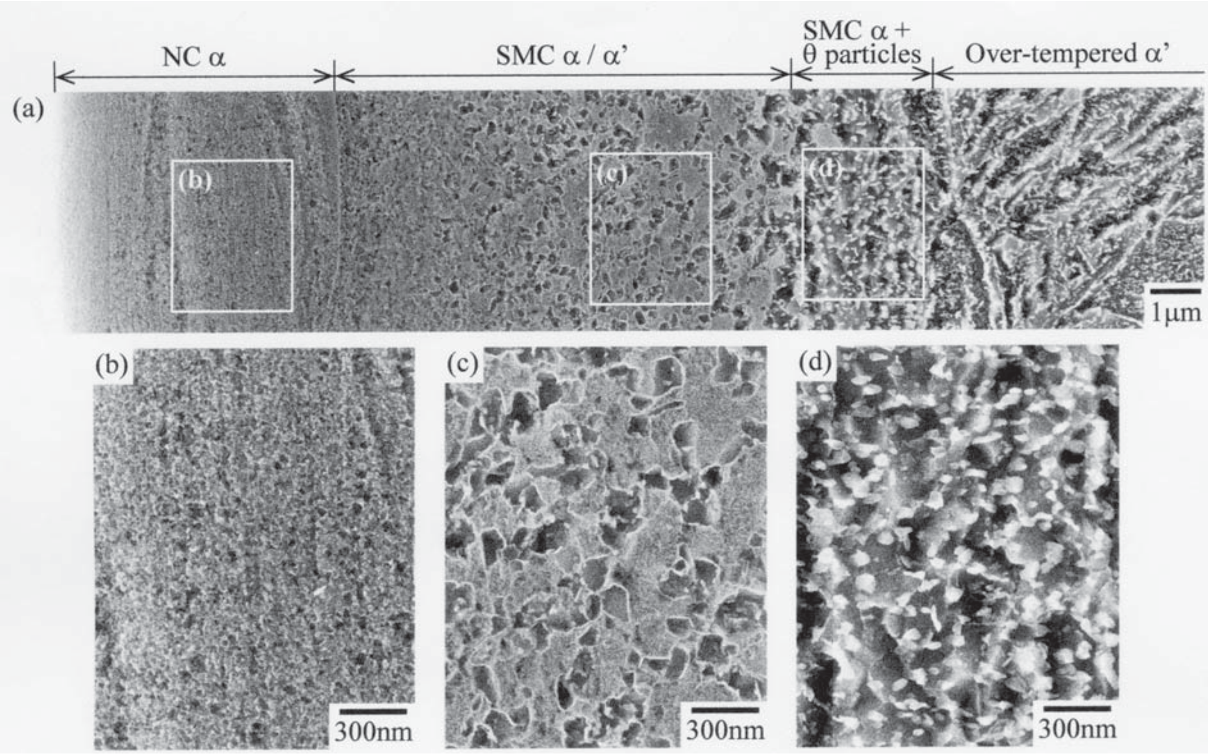

Fig. 7. The SEM micrograph ((a) low magnification) of the drill hole surface in the as-quenched Fe-0.56%C steel after drilling (Cutting speed: 80 m/min, Drill diameter: 5.0 mm). The enlarged micrographs at (b) NC α, (c) SMC α/α' and (d) SMC α + θ particles structure layers.

Ac_3 and the NC structure was probably formed in the austenite state. At the further inner region (Fig. 7d), equiaxed SMC ferrite grains with cementite particles at the grain boundaries are seen. It is considered that this area was deformed and then heated to just below Ac_1. The equiaxed ferrite grains suggest that recrystallization (static and/or dynamic) took place. At the region at about 18 μm away from the surface, martensite structure is maintained without deformation as shown in Fig. 7a. The precipitated cementite particles suggest that this region was heated to a certain extent to form tempered structure.

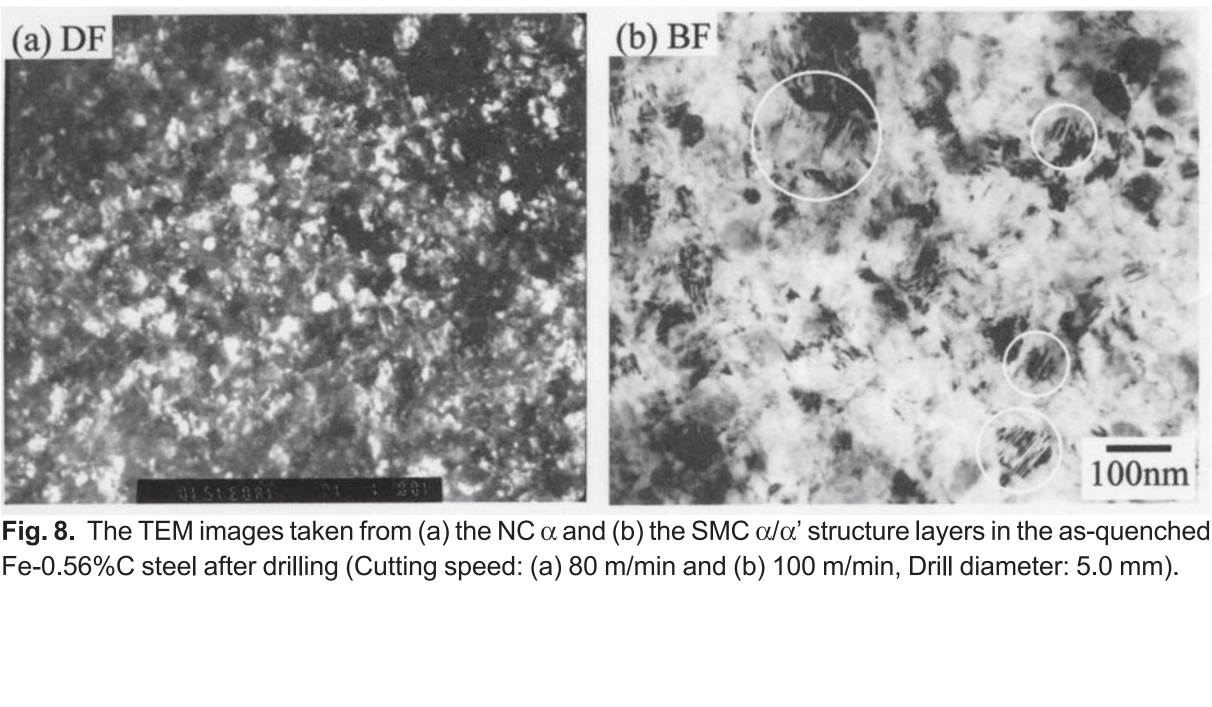

Fig. 8. The TEM images taken from (a) the NC α and (b) the SMC α/α' structure layers in the as-quenched Fe-0.56%C steel after drilling (Cutting speed: (a) 80 m/min and (b) 100 m/min, Drill diameter: 5.0 mm).

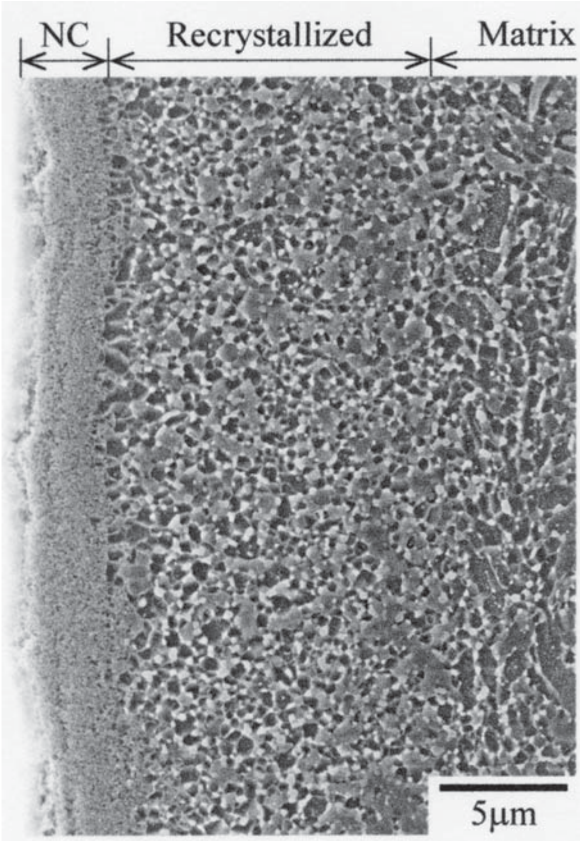

Fig. 9. The SEM micrograph of the drill hole surface in the as-quenched Fe-0.56%C steel after drilling (Cutting speed: 80 m/min, Drill diameter: 2.5 mm) and subsequent annealing at 873K for 3.6 ks.

Fig. 9 is the SEM micrographs near the hole surface of as-quenched Fe-0.56%C steel after drilling and annealing at 873K for 3.6 ks. The near-surface NC layer kept ultrafine grained structure, while recrystallization took place in the re-austenitized SMC layer underneath the NC layer. It is noteworthy that the annealed structures of the NC and the re-austenitized SMC layers were completely different although it was difficult to distinguish these layers before annealing.

4. DISCUSSION

In our experiments, BM [8,9], SP [10,11] and drilling [12] were demonstrated to be possible processes to produce NC structure. A common condition in these processes is considered to be non-uniform deformation. Ashby [16] introduced the distinction between statistically stored dislocations (SSD) and geometrically necessary dislocations (GND): the former is generally stored by uniform deformation, while the latter is developed with strain gradients in non-uniform deformation. The GND are hard to annihilate since all the dislocations have the same sign associated with the direction of strain gradient. Therefore, it seems that the GND are hard to recover even at high temperature generated by drilling. The density of GND ρ_{GND} is proportional to the shear strain gradient $\chi = d\gamma/dz$: $\rho_{GND} = \chi / b$, where b is the magnitude of Burgers vector. The minimum χ necessary to form the NC structure with grain size of 100 nm in iron was estimated to be 1.3 μm^{-1} from the ρ_{GND} which has the same energy density as the grain boundary energy per unit volume in the iron with grain size of 100 nm. Here, the χ at the hole surface of drilled specimen is evaluated to compare the estimated χ. Fig. 10a is the SEM micrographs of the drill hole surface perpendicular to the drilling direction. The remarkable plastic flow of martensite structure to the cutting direction can be seen. When it is presumable that the relevant structure was initially straight and perpendicular to the surface, the curve of plastic flow is described roughly by an exponential function of depth. The measured displacement x of martensite structure from the initial position by drilling are shown in Fig. 10b as a function of depth z from the top surface. The x is expressed by the following equation: $x\,[\mu m] = x_s\, exp(-k\, z)$, where x_s (= 71.3 μm) is the displacement at the top surface and k (= - 0.341) is a constant. The γ is given by differentiating the x by the z (Fig. 10c). The shear strain at the top surface is estimated to be around 24 (14 in equivalent strain). The χ is also obtained by differentiating the γ by the z as shown in Fig. 10d. In the NC layer, the amount of χ is lager than the estimated value (1.3 μm^{-1}). This indicates that to produce NC structure deformation with larger strain gradient than around 1 μm^{-1} is an important condition.

5. CONCLUSIONS

(1) Nanocrystalline (NC) layer with grain size of around 20 nm was successfully produced in steels by shot peening or drilling. By annealing, the NC structure showed substantially slow grain growth without recrystallization.
(2) In shot peening, the NC layer was formed when the shot velocity and/or coverage were higher than those in the conventional operating condition. It was also found that the NC layer formed when the initial specimen hardness was lower than shot hardness.
(3) In drilling, the NC layer was formed under the condition of higher cutting speed than that in the

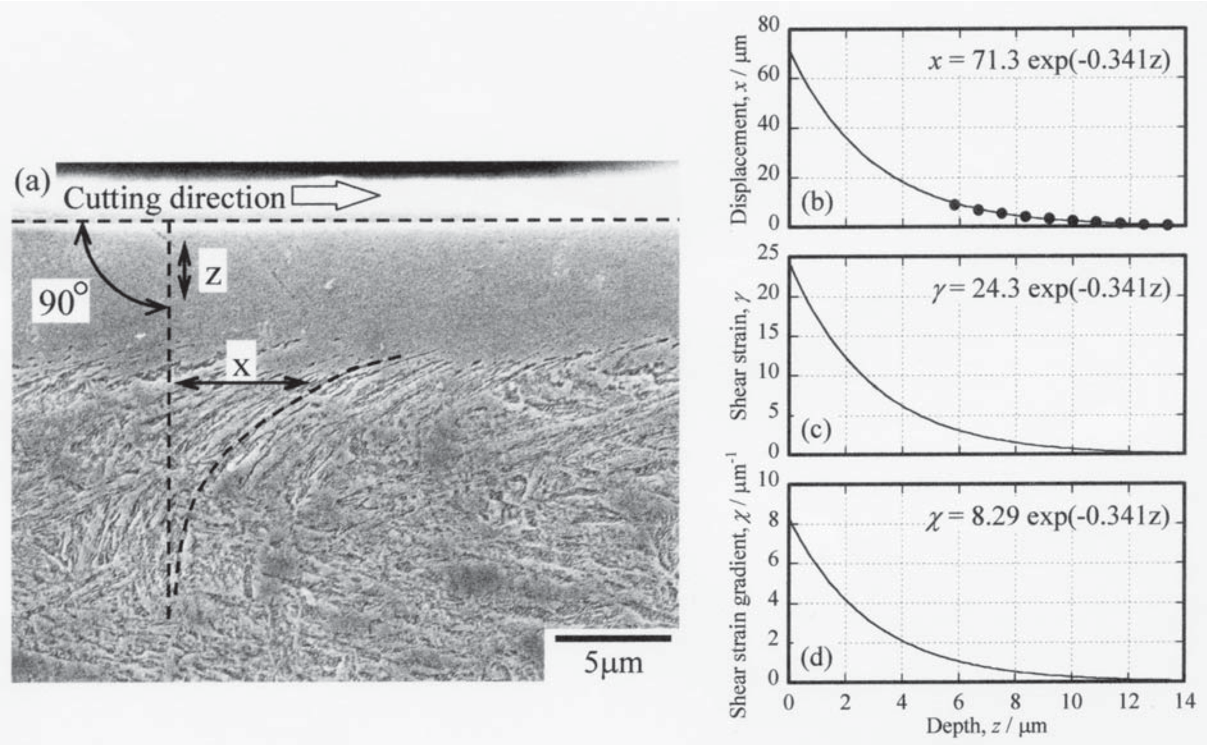

Fig. 10. (a) The SEM micrograph of the drill hole surface perpendicular to the drilling direction in the as-quenched Fe-0.56%C steel after drilling (Cutting speed: 80 m/min, Drill diameter: 2.5 mm). (b) The displacement *x* of martensite structure from the initial position, (c) the shear strain γ and (d) the shear strain gradient χ as a function of depth *z* from the top surface of drill hole.

conventional condition. The formation of NC layer was considered to take place in austenite condition (above Ac_3).

(4) As a common deformation condition to produce the NC structure, a large strain gradient was proposed. The minimum strain gradient necessary to produce 100 nm grained structure was estimated to be around 1 μm^{-1}.

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