

# Comparison of Nanocrystalline Surface Layer in Steels Formed by Air Blast and Ultrasonic Shot Peening\*1,\*2

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Surface nanocrystallization in various steels by shot peening (both air blast (ABSP) and ultrasonic (USSP)) was investigated. In all the shot-peened specimens, the equiaxed nanocrystals with grain size of several 10nm were observed at the surface regions. The depth of nanocrystalline (NC) layers was several  $\mu\text{m}$ . The NC layers have extremely high hardness and were separated from the deformed structure regions just under the NC layers with sharp boundaries. By annealing, the NC layers show the substantially slow grain growth without recrystallization. These characteristics are similar to those observed in the specimens treated by ball milling, ball drop and particle impact deformation. Comparing ABSP and USSP at the similar peening condition, the produced volume of NC region in ABSP is larger than that in USSP.

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## 1. Introduction

Nanocrystallization of bulk structural materials by severe plastic deformation (SPD) processes has been drawn strong attentions, since nanocrystalline (NC) materials (grain size smaller than 100 nm) have superior mechanical and physical properties to those of coarse-grained counterparts. Various SPD processes have been proposed to produce the NC materials, such as ball milling (BM),<sup>1-4)</sup> ball drop deformation (BD),<sup>4,5)</sup> particle impact deformation (PI),<sup>6)</sup> high pressure torsion,<sup>7)</sup> sliding wear<sup>8,9)</sup> and shot peening.<sup>6,10-15)</sup> Among these techniques, the shot peening has considerable importance since it is a popular process in industries and it can produce the nanostructured surface layer with a high productivity. From our previous BM, BD and PI experiments in steels,<sup>2-6)</sup> it was found that the NC region has the following characteristics: 1) extremely high hardness (8~13 GPa), 2) separated from deformed structure region with sharp boundary, 3) dissolution of cementite when it exists and 4) no recrystallization and slow grain growth by annealing.

In the present study, the surface nanocrystallization in various steels by shot peening (both air blast and ultrasonic) was investigated. The NC regions formed by these techniques were compared with those produced by BM, BD and PI.

## 2. Experimental Procedures

The materials used were Fe-0.03C (Fe-0.031C-0.052Si-0.054Mn in mass% hereafter, ferrite structure), Fe-0.10C (Fe-0.099C-0.20Si-1.37Mn, martensite), Fe-0.80C (Fe-0.800C-0.20Si-1.33Mn, either pearlite or spheroidite) and Fe-3.3Si (Fe-0.027C-3.29Si-0.01Mn, ferrite). Air blast shot peening (ABSP) was carried out using two different shot size, *i.e.*  $\phi$  0.05 mm (cast steel (Fe-1.0C, HV 7.8 GPa), shot velocity: 190 m/s) and  $\phi$  0.3 mm (SUS304 (Fe-18Cr-8Ni, HV

5.3 GPa), shot velocity: 100 m/s). Coverage (the area fraction of specimen surface deformed by shots) larger than 50% was estimated by multiplying shot peening time by % coverage for one second measured at the state of 50% coverage. Ultrasonic shot peening (USSP) was performed using SUJ2 (Fe-1C-1.5Cr, HV 7.8 GPa) shot ( $\phi$  0.4 mm). In the USSP experiment, the shots were placed in the vibrating chamber (frequency: 20 kHz, amplitude: 90  $\mu\text{m}$ ), with which the shots were resonated. The projection distance was 10 mm, and the shot velocity was less than 20 m/s. The details of conditions in ABSP and USSP were described in the previous paper.<sup>10)</sup> Annealing of nanocrystallized specimens was carried out at 873 K for 3.6 ks by sealing in a quartz tube under a pure Ar protective atmosphere. Specimens were characterized by SEM, TEM and Vickers microhardness tester. Specimens for SEM observations were etched by 5% Nital.

## 3. Results and Discussions

Figure 1 shows cross sectional SEM micrographs of various carbon steels processed by ABSP using  $\phi$  0.05 mm shot. From TEM observations, the equiaxed nanocrystals with grain size of several 10nm were confirmed in the nanocrystalline (NC) surface layers. The NC layers have extremely high hardness and were separated from the adjacent deformed structure (DS) regions with sharp boundaries.<sup>6,10)</sup> It is noted that the nanocrystallization occurs by ABSP irrespective of the carbon content or the initial structure of steels. In the NC region of Fe-0.80C pearlite (or spheroidite) steels shown in Figs. 1(c) (or (d)), cementite lamellae (or particles) are invisible in the NC region, indicating the dissolution of cementite took place. After annealing at 873 K for 3.6 ks, the NC regions still kept much finer structure than that of the DS regions.<sup>6,10)</sup> These structures and the annealing behavior are similar to those observed in the specimens after BM, BD or PI.<sup>2-6)</sup> The effect of coverage on the thickness of NC layer formed by ABSP was investigated. Figure 2 shows the cross sectional SEM micrographs of Fe-3.3Si steel processed by ABSP using  $\phi$  0.3 mm shot and annealed. The thickness of NC layer at the

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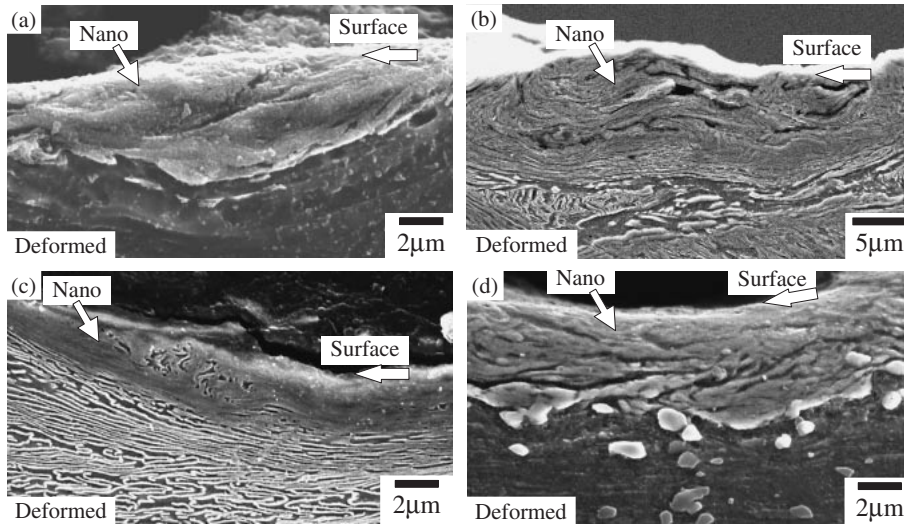


Fig. 1 SEM micrographs of NC layers formed in various steels after ABSP using  $\phi$  0.05 mm shot. (a) Fe-0.03C (ferrite, 80% cold rolled, 1000% in coverage), (b) Fe-0.10C (martensite, 6000% in coverage), (c) Fe-0.80C (pearlite, 82% cold rolled, 1000% in coverage) and (d) Fe-0.80C (spheroidite, 84% cold rolled, 1000% in coverage).

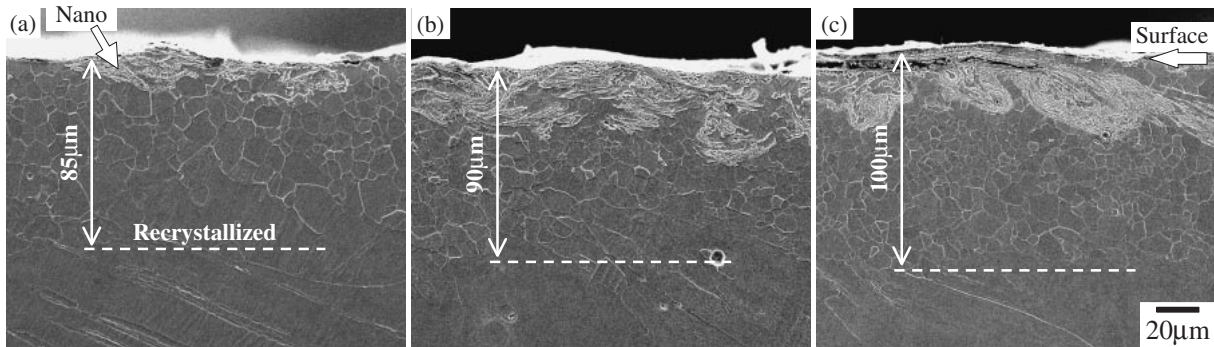


Fig. 2 The effect of coverage on the thickness of NC layer formed by ABSP. Fe-3.3Si steel was ABSPed using  $\phi$  0.3 mm shot and subsequent annealed. (a) 3000%, (b) 10000% and (c) 30000% in coverage.

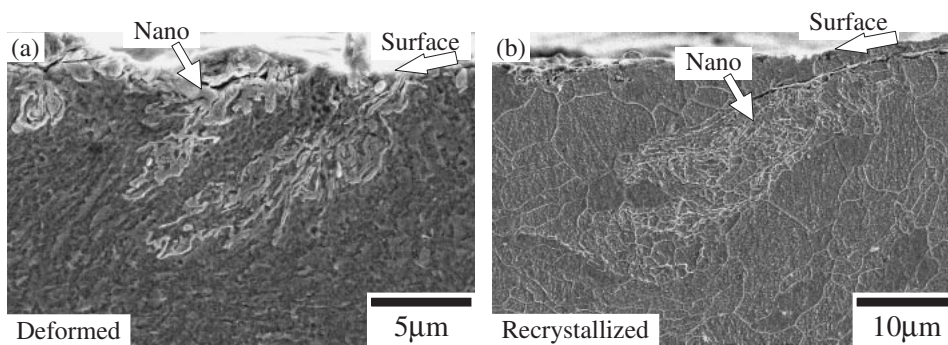


Fig. 3 SEM micrographs of Fe-0.03C specimen after (a) USSP (coverage: 36000%, peening time: 1800 s) and (b) subsequent annealing.

specimen surface increased with coverage. While, the thickness of the recrystallized structure (RS) layer increases slightly with coverage. The mean recrystallized grain size decreased from  $6.9\mu\text{m}$  (3000% in coverage) to  $6.0\mu\text{m}$  (10000%) and  $5.4\mu\text{m}$  (30000%), indicates that the strain in these layers increased with coverage.

Figure 3 shows the typical microstructures of Fe-0.03C specimen after USSP and annealing. The formation of NC regions can be seen. These structures and the annealing

behavior are similar to those observed in the specimens processed by ABSP. Figure 4 shows the comparison of the morphology of NC regions in Fe-0.80C spheroidite steel after APSP and USSP. Comparing these NC regions, the specimen processed by USSP showed the aggregated morphology and the NC regions tend to extend inwards from the surface instead of the forming layers like that observed in ABSP. The shot peening conditions of ABSP and USSP are summarized in Table 1. In ABSP, the shot velocity is around 100 m/s and

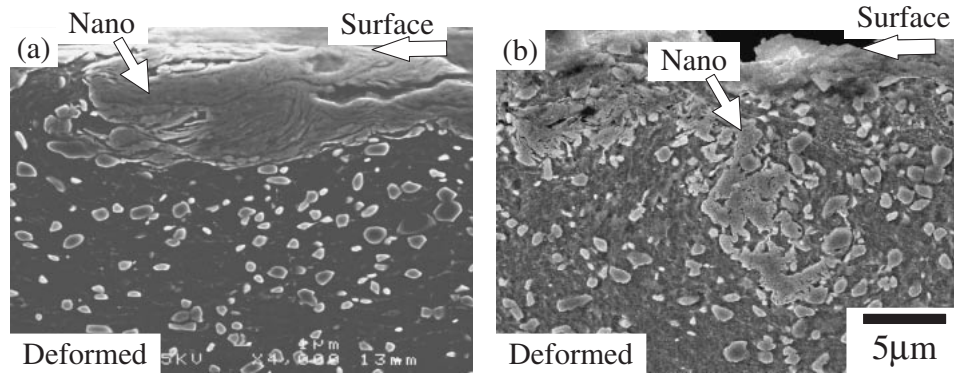


Fig. 4 Comparison of morphology of NC regions in Fe-0.80C spheroidite steel in (a) ABSP (shot:  $\phi$  0.05 mm, Fe-1.0%C, shot velocity: 190 m/s, coverage: 6000%, peening time: 60 s) and (b) USSP (shot:  $\phi$  0.4 mm, SUJ2, shot velocity: <20 m/s, coverage: 108000%, peening time: 5400 s).

Table 1 Comparison of shot peening conditions of ABSP and USSP.

Air blast shot peening		Ultrasonic shot peening
	Schematic illustration of equipment	
$\phi$ 0.05, 0.3 mm	Shot size	$\phi$ 0.4 mm
High (> 100 m/s) Velocity distribution - Small	Shot velocity	Low (< 20 m/s) Velocity distribution - Large
Single-direction ( $\sim 90^\circ$ )	Impact direction	Multi-direction
100, 170 % / s	Coverage rate	20 % / s

has a narrow distribution. On the other hand, in USSP the shot velocity is less than 20 m/s and has a wide distribution. The impact direction of shot to specimen is almost perpendicular in ABSP and random in USSP. The impact energy of one shot in ABSP is much larger than that in USSP. The heat generation in specimen, therefore, is larger in ABSP than in USSP, and also the temperature gradient at the surface is larger in ABSP than in USSP. It is considered that the specimen temperature in USSP is rather constant through the depth since the coverage rate (per peening time) in USSP is smaller than that in ABSP. The deformation in USSP preferentially occurs around the grain boundaries and the NC regions tend to extend toward inside of the specimens. While, in the ABSP the temperature gradient at the surface is large and the deformation preferentially occurs parallel to the specimen surface and results in the layered morphology. Figure 5 shows the cross sectional SEM micrographs of Fe-3.3Si steel after APSP and USSP. The produced volume of NC regions in ABSP is larger than that in USSP, even though the coverage in ABSP is about 80% of that in USSP. As are shown in Fig. 5, the recrystallized grain is  $5.4\mu\text{m}$  in ABSP and  $4.9\mu\text{m}$  size in USSP. The thickness of recrystallized layer is  $100\mu\text{m}$  in ABSP and  $35\mu\text{m}$  in USSP. These results indicate that a larger strain is given at a thin surface layer in USSP than that in ABSP. This difference is due to the lower

surface temperature in USSP than in ABSP since the impact energy of one shot in USSP is much smaller than that in ABSP.

Lu *et al.*<sup>13–15)</sup> have reported the surface nanocrystallization in various steels by USSP. In their experiments, the surface NC (grain size of about 10 nm) layer was formed by USSP (50 Hz,  $\phi$  8 mm shot, 1.8 ks of peening time). They reported that the grain size continuously decreased toward the surface and there is no discontinuous change in structure. The sharp boundaries along the NC region observed in the present study were not realized. They might overlook the boundaries between NC and DS regions since the area they observed by TEM is much smaller than ours. The differences are caused by the experimental conditions.

#### 4. Conclusions

The NC layers with several  $\mu\text{m}$  thick were successfully produced in various steels by both ABSP and USSP. The NC layers have extremely high hardness and were separated with sharp boundaries from the adjacent DS regions. By annealing, the NC layers show the substantially slow grain growth without recrystallization. These characteristics are similar to those observed the specimens after BM, BD and PI. Comparing ABSP and USSP at the same coverage, the



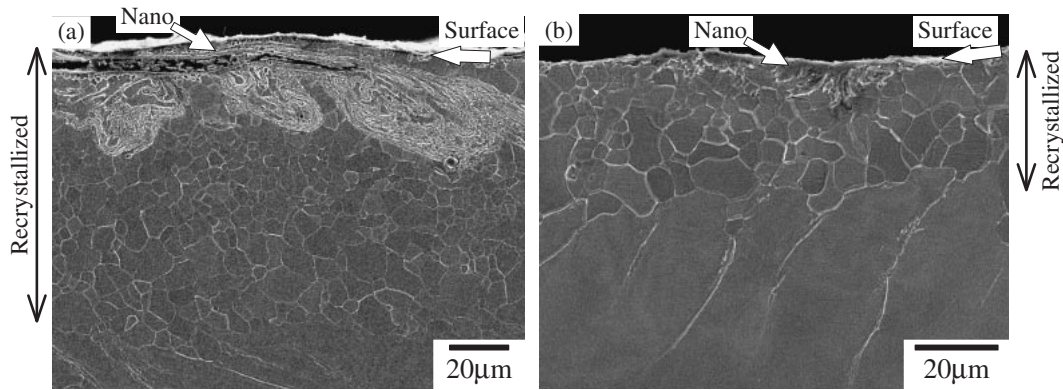


Fig. 5 SEM micrographs of Fe-3.3Si steel shot-peened and annealed. (a) ABSP (shot:  $\phi$  0.3 mm, SUS304, shot velocity: 100 m/s, coverage: 30000%, peening time: 180 s) and (b) USSP (shot:  $\phi$  0.4 mm, SUJ2, shot velocity: <20 m/s, coverage: 36000%, peening time: 1800 s).

produced volume of NC region is larger in ABSP than in USSP. The DS region is thicker and the strain is smaller in ABSP than in USSP.

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