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Nanocrystallization in Steels by Air Blast Shot Peening

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Surface nanocrystallization in various steels by shot peening (both air blast and ultrasonic) was investigated. Nanocrystalline layers with several μm thick were successfully fabricated by these methods. In all the case, the nanocrystalline layers have extremely high hardness and separated from adjacent deformed structure regions with sharp boundaries. By annealing, the nanocrystalline layers showed substantially slow grain growth without recrystallization. Those characteristics are similar to those observed in the samples treated by ball milling, ball drop and particle impact deformation. When nanocrystalline structure was produced by shot peening, the strains were found to be not simple compression but complex multi-directional deformation.
Key words: nanocrystalline, severe plastic deformation, air blast shot peening, ultrasonic shot peening, steel.

1. INTRODUCTION

Nanocrystalline (NC) materials (grain size smaller than 100 nm) have attracted considerable scientific interests in the last decade, since NC materials have often superior mechanical and physical properties to those of coarse-grained counterparts. Various severe plastic deformation (SPD) processes have been proposed to produce the NC materials, such as ball milling (BM) [1-4], ball drop deformation (BD) [4,5], particle impact deformation (PI) [6], high pressure torsion [7], sliding wear [8,9] and shot peening [6,10-14]. Among these techniques, the shot peening has considerable importance since it is a popular process in industries and it can produce nanostructured surface layer with a high productivity.

From our previous BM, BD and PI experiments in steels [2-6], it was found that the NC regions have the following characteristics: 1) extremely high hardness (8 ~ 13 GPa), 2) separated from deformed structure (DS) region with sharp boundaries, 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 [2-6].

2. EXPERIMENTAL PROCEDURES

The materials used in the present study were Fe-3.3Si (Fe - 0.027C - 3.29Si - 0.01Mn in mass% hereafter, ferrite structure), Fe-0.03C (Fe - 0.031C - 0.052Si - 0.054Mn, ferrite), a 590 MPa class high tensile strength steel of Fe-0.05C (Fe - 0.05C - 0.01Si - 1.29Mn, ferrite), Fe-0.10C (Fe - 0.099C - 0.20Si - 1.37Mn, martensite), Fe-0.45C (Fe - 0.45C - 0.20Si - 0.95Mn - 0.20Cr, ferrite plus pearlite) and Fe-0.80C (Fe - 0.800C - 0.20Si - 1.33Mn, either pearlite or spheroidite). The martensite structure in Fe-0.10C was obtained by austenitized at 1273 K for 5.4 ks and quenched into water. The pearlite structure in Fe-0.80C was obtained by austenitizing the specimens at 1223 K for 1.8 ks and then kept at 873 K for 0.3 ks to transform to pearlite structure. To obtain the spheroidite structure in Fe-0.80C, the specimens were austenitized at 1173 K for 3.6 ks and quenched into water to obtain martensite structure, and then tempered at 983 K for 79.2 ks.

The shot peening equipments are illustrated in Fig. 1. Air blast shot peening (ABSP, Fig. 1 (a)) was carried out in various conditions (Table I). The coverage is the area fraction of specimen surface deformed by shots. Coverage 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, Fig. 1 (b)) was performed using JIS SUJ2 shot (ϕ 0.4 mm). In the USSP experiment, the shots were placed in the chamber vibrated (frequency: 20 kHz, amplitude: 90 μm) by a generator, with which the shots were resonated. The projection distance was 10 mm, and coverage rate was 20 %/s. Annealing of nanocrystallized specimens was carried out at 873

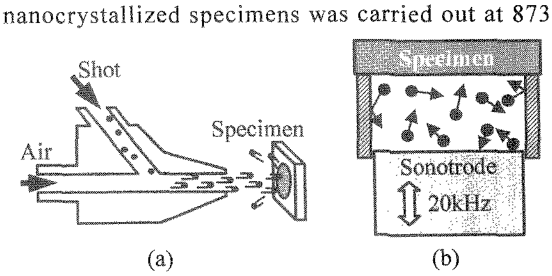

Fig. 1 Equipments used in the present study. (a) ABSP and (b) USSP.

Table I Conditions of ABSP.

Condition	Shot velocity [m/s]	Blasting pressure [MPa]	Shot			Peening time per 100 % in coverage [s]
			Diameter [mm]	Composition [mass%]	Vickers hardness [GPa]	
(1)	190	0.5	0.05	Fe-1.0C-1.3Si-1.0Mn	7.8	1
(2)	100	0.45	0.3	Fe-18Cr-8Ni (JIS SUS304)	5.3	0.6
(3)	100	0.5	0.8	Fe-0.8C-0.25Si-0.45Mn	6.9	4

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 (load of 0.25 N for 15 s). Specimens for SEM observations were etched by 5 % Nital.

3. RESULTS AND DISCUSSIONS

3.1 Air blast shot peening

Figure 2 shows a typical nanocrystalline (NC) layer of Fe-3.3Si specimen by ABSP (Condition (1) in Table I, 1000 % in coverage). The sharp boundary between NC and DS regions is seen, similar to that observed in ball milled Fe-3.3Si powder. To examine the grain size and orientation of grains in NC layer, TEM samples were prepared parallel to the specimen surface. TEM dark filed (DF) image and selected area diffraction (SAD) rings taken from the NC region near the surface of the specimen are shown in Fig. 3. The DF image shows that size of grains is less than 20 nm, and the SAD rings

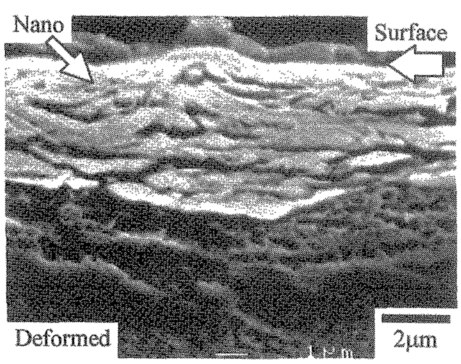

Fig. 2 SEM micrograph of Fe-3.3Si specimen after ABSP (Condition (1), 1000 % in coverage).

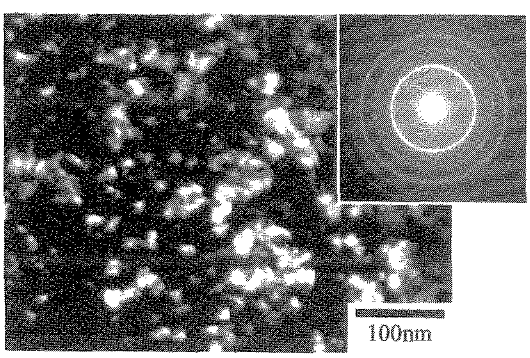

Fig. 3 TEM DF image and (c) SAD rings (ϕ 1.2 μm in aperture size) of NC layer in Fe-3.3Si specimen after ABSP (Condition (1), 6000 % in coverage).

indicate that the grains are randomly oriented. Since bcc structure in Fe-3.3Si alloy is stable up to its melting point, NC structure formed by the ABSP is due to severe plastic deformation and not concerned martensite thermally induced. Figure 4 shows a cross section of SEM micrograph of Fe-0.05C high tensile strength steel after ABSP (Condition (1), 1000 % in coverage). The hardness of the NC region near surface is as high as 6.8 GPa, which is much higher than that of the interior region (2.6 GPa). The hardness of 6.8 GPa indicates the grain size is less than 100 nm according to the Hall-Petch relationship for iron (HV [GPa] = 0.36 + $59d^{-1/2}$ [d: nm]). Figure 5 shows cross sectional SEM micrographs of various carbon steels after ABSP (Condition (1)). It is noted that nanocrystallization occurs by ABSP irrespective of the carbon content or initial structure of steels. These NC regions are separated with sharp boundary between DS regions. In the NC region of Fe-0.80C pearlite (or spheroidite) steels shown in Fig. 5 (c) (or (d)), cementite lamellae (or particles) are invisible in the NC region near surface, indicating the dissolution of cementite. These structures are similar to those observed in the specimens after BM, BD or PI [2-6]. Figure 6 shows cross sectional SEM micrographs of these ABSPed specimens after annealing at 873 K for 3.6 ks. Prior NC regions keep much finer structure than that of DS regions, which is similar to those observed in BM or BD experiments [2-5].

The effect of coverage on the thickness of NC layer formed by ABSP was investigated. Figure 7 shows cross sectional SEM micrographs of Fe-3.3Si steel after ABSP (Condition (2)) and subsequent annealing at 873 K for 3.6 ks. The thickness of NC layer at the spacimen surface increased with coverage (here one second of shot

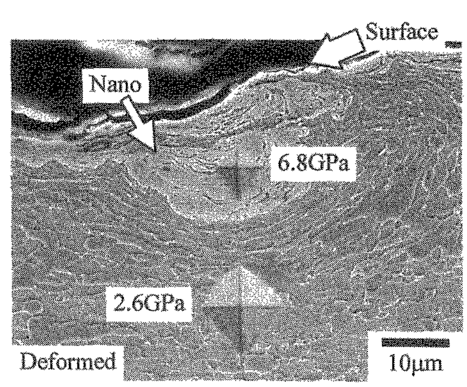

Fig. 4 SEM micrograph of NC region formed in high tensile strength steel (Fe-0.05C) after shot peening (Condition (1), 1000 % in coverage).

peening corresponds to 100 % in coverage). While, in the recrystallized structure (RS) layer the grain size reduced and thickness of RS layer increase slightly. RS layer received certain degree of strain by ABSP to onset recrystallization. The mean recrystallized grain size decreased from 6.9 μm (3000 % in coverage) to 5.4 μm (30000 %), indicates that the strain in this layer increased with coverage.

The effect of shot size on the thickness of NC layer formed by ABSP was investigated using Fe-3.3Si steel. Two different sizes of shots were used, *i.e.* ϕ 0.8 mm (Fe-0.8C) and ϕ 0.3 mm (SUS304). Figure 8 is cross sectional SEM micrographs of Fe-3.3Si specimens after ABSP (Condition (2) or (3)) and subsequent annealing. The same coverage (3000 %) and the shot speed (100 m/s) were employed in both cases. The NC layer formed by ϕ 0.8 mm shot is thicker than that of ϕ 0.3 mm shot, and the mean recrystallized grain size at prior DS region of ϕ 0.8 mm shot (4.0 μm) is smaller than that of ϕ 0.3 mm shot

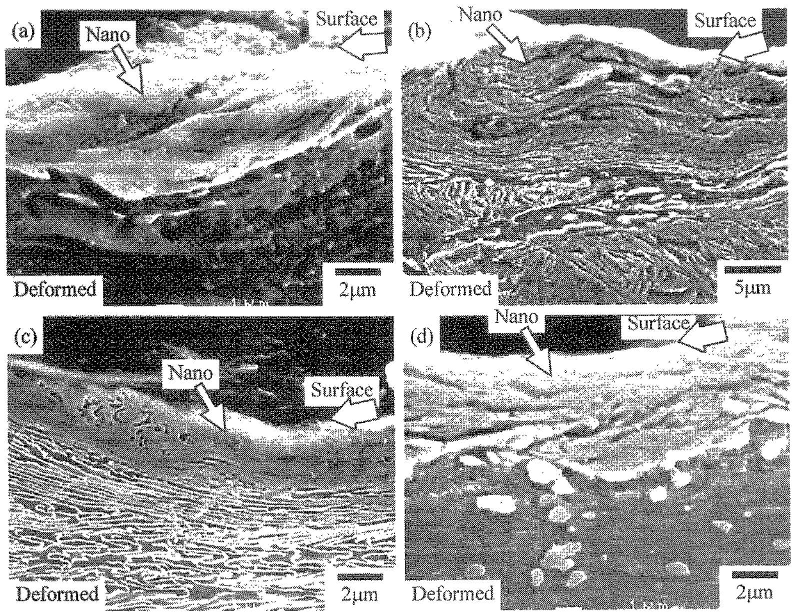

Fig. 5 SEM micrographs of NC layers formed in various steels after ABSP (Condition (1)). (a) Fe-0.03C (ferrite, 80 % cold rolled, 1000 % in coverage), (b) Fe-0.10C (martensite, 6000 % in coverage), (c) Fe-0.80C (pearlite, 82 % cold rolled, 1000 % in coverage) and (d) Fe-0.80C (spheroidite, 84 % cold rolled, 1000 % in coverage).

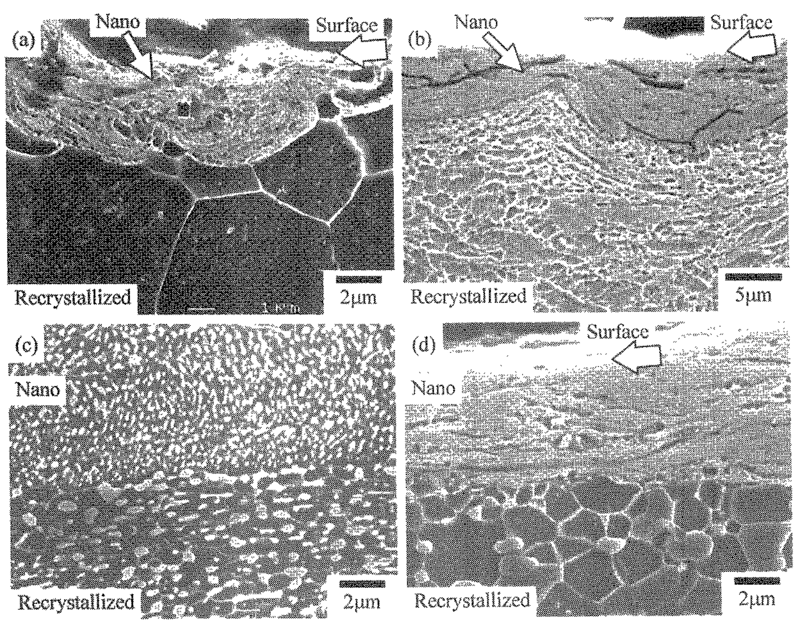

Fig. 6 SEM micrographs of NC layers formed in various steels after ABSP (Condition (1)) and subsequent annealing. (a) Fe-0.03C (ferrite, 80 % cold rolled, 1000 % in coverage), (b) Fe-0.10C (martensite, 6000 % in coverage), (c) Fe-0.80C (pearlite, 82 % cold rolled, 1000 % in coverage) and (d) Fe-0.80C (spheroidite, 84 % cold rolled, 1000 % in coverage).

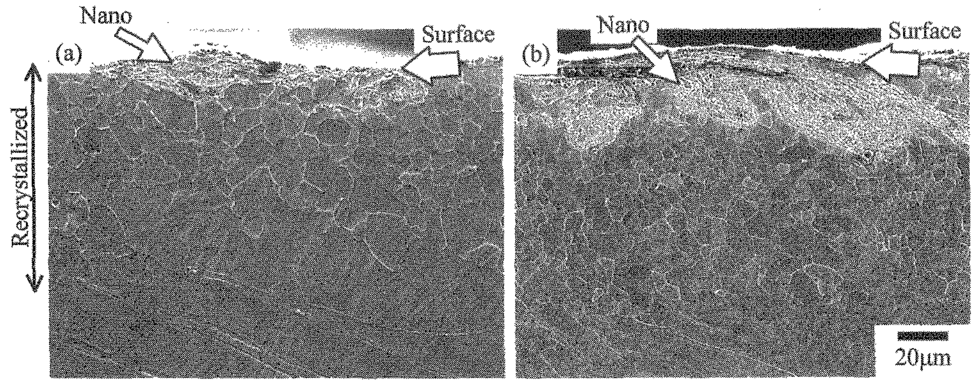

Fig. 7 The effect of coverage on the thickness of NC layer formed by ABSP (Condition (2)). Fe-3.3Si specimens were ABSPed with (a) 3000 % or (b) 30000 % in coverage and subsequent annealed.

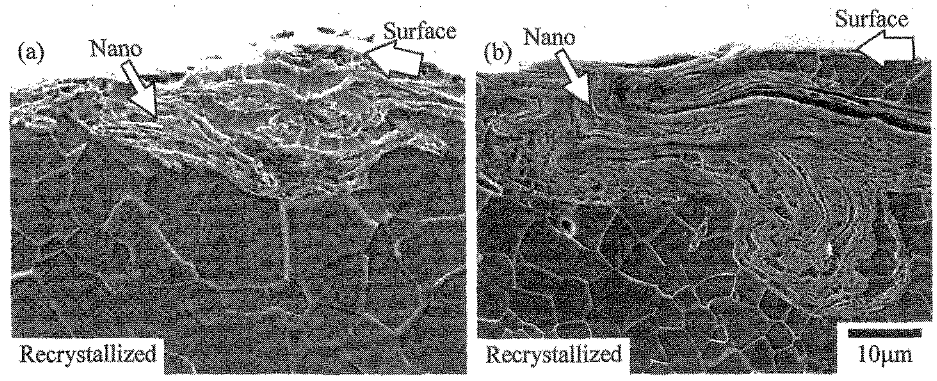

Fig. 8 The effect of shot size on the thickness of NC layer formed by ABSP (Condition (2) or (3)). Fe-3.3Si specimens were ABSPed (shot: (a) ϕ 0.8 mm (Fe-0.8C), (b) ϕ 0.3 mm (SUS304), shot speed: 100 m/s, coverage: 3000 %) and subsequent annealing. Fig. 8 (a) is the enlarged micrograph of Fig. 7 (a).

(6.9 μm). These results are considered to be due to the kinetic energy per one shot. (ϕ 0.8 mm shot is about 19 times lager than that of the ϕ 0.3 mm shot.) The hardness of shot also might be responsible to the produce microstructure. The hardness of ϕ 0.8 mm (Fe-0.8C) shot is 6.9 GPa, and that of ϕ 0.3 mm (SUS304) is 5.3 GPa. Since the hardness of these shots is harder than the initial hardness of specimens, the effect of the shot hardness on the thickness of NC layer is considered to be small. In summary, it is possible to produce surface NC layers with several 10 μm thick by ABSP if the coverage, shot size and shot speed are properly controlled.

3.2 Ultrasonic shot peening

The formation of NC regions by USSP was examined. Figure 9 and 10 shows cross sectional SEM micrographs of Fe-0.03C and Fe-0.80C spheroidite steels after USSP, respectively. As is seen, the formation of NC regions can be seen in both specimens. Comparing with ABSP, the NC regions formed by USSP is aggregated morphology rather than layered. They form locally as clusters and tend to extend inwards from surface instead of forming layers at the surface. Various possible reasons of this difference in the morphology of NC regions in the two shot peening processes can be proposed, such as shot speed, shot direction, coverage per second, specimen temperature and so on. In USSP, shot speed is less than 20m/s and has a wide distribution. On the other hand, in ABSP shot speed is around 100m/s and has a narrow distribution. The impact angle of shot to specimen is random in USSP and almost perpendicular in ABSP. The coverage rate is 20 %/s in USSP and 100 %/s in ABSP. Since the impact energy of one shot in ABSP is larger than in USSP, the heat generation in specimen is much larger in ABSP than in USSP. The temperature of the specimen is higher in ABSP than in USSP. The temperature gradient at surface is also higher in ABSP than in USSP. It is considered that in the USSP the specimen temperature is rather constant

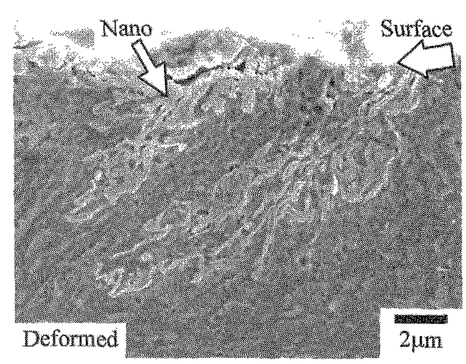

Fig. 9 SEM micrograph of Fe-0.03C specimen after USSP for 1.8 ks (36000% in coverage).

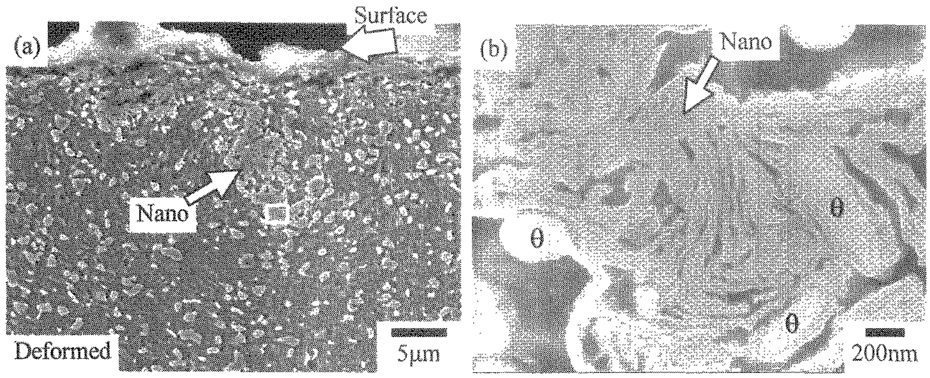

Fig. 10 SEM micrographs of Fe-0.80C spheroidite steel after USSP for 5.4 ks (108000% in coverage). (a) low magnification and (b) high magnification of NC region.

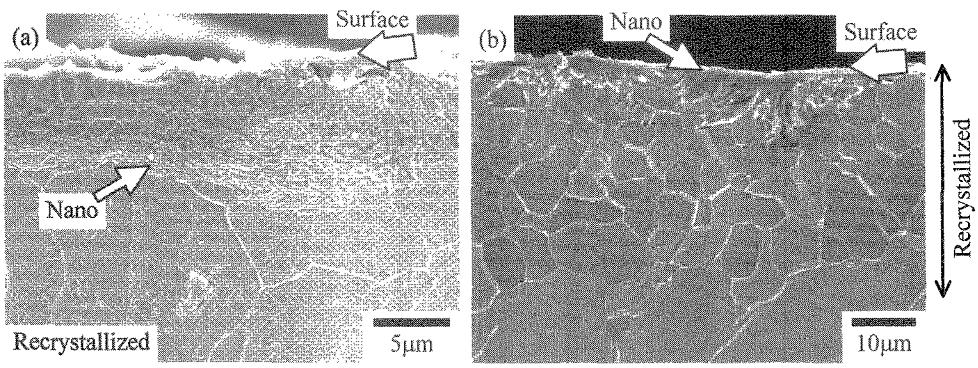

Fig. 11 SEM micrographs of (a) Fe-0.03C and (b) Fe-3.3Si specimens after USSP for 1.8 ks (36000% in coverage) and subsequent annealing.

through the depth and deformation occurs preferentially around the grain boundaries. Thus the NC regions tend to extend toward inside of the specimens. While, in the ABSP the temperature gradient at surface is large and deformation occurs preferentially parallel to specimen surface and form NC layers.

The formation of NC regions by USSP can be confirmed by the cementite dissolution and annealing behaviors. The deformation or dissolution behavior of cementite particles during nanocrystallization was realized to be the same with other deformation processes [3-6]. During nanocrystallization, cementite particles keep their shape until the surrounding ferrite become NC grained structure. The enlarged micrograph (Fig. 10 (b)) shows the cementite particles surrounded by NC ferrite. Figure 11 shows cross sectional SEM micrographs of Fe-0.03C and Fe-3.3Si specimens after USSP for 1.8 ks (36000% in coverage) and subsequent annealing at 873 K for 3.6 ks, respectively. The grain size of the prior NC regions near surface is much smaller than that of the recrystallized prior DS regions. This annealing behavior is similar to those observed in the ABSP specimens.

In the DS regions, certain differences were recognized between the USSP and ABSP. The recrystallized grain size in USSP is smaller than that in ABSP, while the depth of recrystallized region in USSP is smaller than in ABSP. As are shown in Fig. 11 (b) and Fig. 7(b), the recrystallized grain size in Fe-3.3Si is 4.9 μm in USSP and 5.4 μm in ABSP. The thickness of recrystallized layer is 35 μm in USSP and 100 μm in ABSP. These results indicating that in USSP the larger strain remains in a shorter distance from surface than in ABSP. This is probably due to the lower temperature of specimen during USSP than that of ABSP.

K. Lu *et al.* [12-14] has reported the surface nanocrystallization in various steels by USSP. In their experiments, the surface NC layer (grain size of about 10 nm in the top surface layer) formed by USSP (50 Hz, ϕ 8 mm of shot size) for 1.8 ks. They reported that grain size continuously decreases toward surface, and there is no discontinuous change in structure. The clear boundaries along the NC region observed in the present study was not recognized. The reason of this difference about NC region between theirs and ours are not clear. Since their observations are made by TEM and not SEM, the area they observed is much smaller than ours. The NC region in their samples is considered to be quite small since their highest hardness is 3.8 GPa which is far lower than that expected for NC pure Fe. There is a high possibility that they might overlook the boundaries of NC regions. The differences may arise from the differences in experimental conditions. They used larger shot and lower frequency than the present study. The atmosphere is in vacuum in theirs and in air in the present study. At this moment, it is difficult to determine true origin of the difference observed.

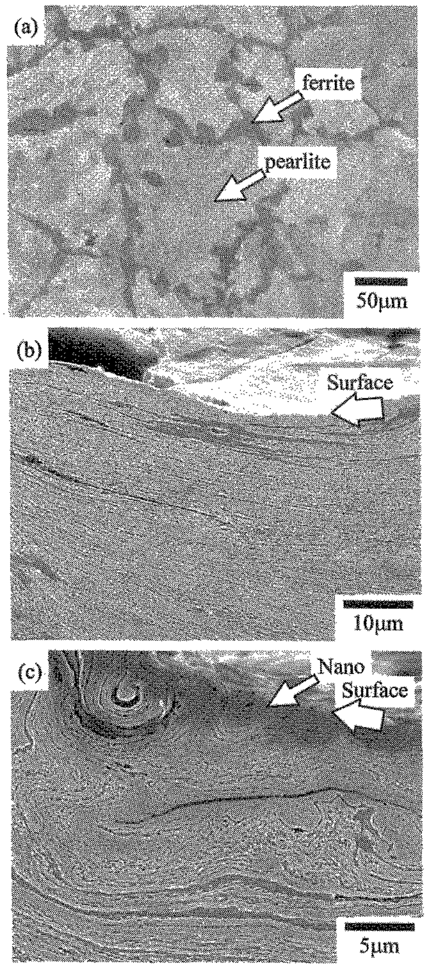

Fig. 12 SEM micrographs of Fe-0.45C steel with ferrite and pearlite structure (a) before and (b), (c) after ABSP (Condition (3), 6000 % in coverage).

4. DISCUSSION

In the present study, it was found that surface NC layer can be produced by both ABSP and USSP. The necessary condition to form the NC structure by deformation was proposed to apply a strain lager than about 7 [6]. It is not clear how such a large strain is given by shot peening. Here, the deformation behavior during shot peening to induce surface NC layer is discussed. Figure 12 shows cross sectional SEM micrographs of Fe-0.45C with ferrite and pearlite structure before and after ABSP (Condition (3), 6000 % in coverage). In the early stage of shot peening, the area below the indentation uniformly deform as is shown in Fig. 12 (b). The microstructure appears as layers parallel to the indentation edge. The amount of strain seems a smooth function of depth. However, such uniform deformation cannot produce NC regions. In the early stage, shots produce inhomogeneity in height, hardness and temperature at specimen surface. In the later stage of peening, shots hit such non-uniform surface and produced highly complex structure as is shown in Fig. 12 (c). This indicating that multi-directional large strains were applied by shot peening which is a favorable condition to produce NC regions.

5. CONCLUSIONS

NC layers with several μm thick were successfully produced in various steels by both ABSP and USSP. The NC layers have extremely high hardness and separated with sharp boundaries from adjacent DS regions. By annealing, NC layers showed substantially slow grain growth without recrystallization. Those characteristics are similar to those observed the specimens after BM, BD and PI. Comparing ABSP and USSP, the produced volume of NC region is larger in ABSP than USSP. The DS region is thicker and with smaller strain in ABSP than in USSP. The origin of applying large strains enough to produce NC regions by shot peening was demonstrated to be a multi-directional deformation due to inhomogeneity at specimen surface produced by each shot.

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REFERENCES

[1] C.H.Moelle and H.J.Fecht, Nanostruct. Mater., 6, 421-24 (1995).
[2] J.Yin, M.Umemoto, Z.G.Liu and K.Tsuchiya, ISIJ Int., 41, 1389–96 (2001).
[3] Y.Xu, M.Umemoto and K.Tsuchiya, Mater. Trans., 43, 2205–12 (2002).
[4] Y.Todaka, M.Umemoto and K.Tsuchiya, ISIJ Int., 42, 1429–36 (2002).
[5] M.Umemoto, B.Huang, K.Tsuchiya and N.Suzuki, Scripta Mater., 46, 383-88 (2002).
[6] M.Umemoto, Y.Todaka and K.Tsuchiya, Mater.Trans., 44, 1488-93 (2003).
[7] R.Z.Valiev, Y.V.Ivanisenko, E.F.Rauch and B.Baudelet, Acta Mater., 44, 4705-12 (1996).
[8] P.Heilmann, W.A.T.Clark and D.A.Rigney, Acta Metall., 31, 1293-05 (1983).
[9] D.A.Hughes, D.B.Dawson, J.S.Korellis and L.I.Weingarten, Wear, 181-183, 458-68 (1995).
[10] I.Altenberger, B.Scholtes, U.Martin and H.Oettel, Mater. Sci. Eng. A, 264, 1-16 (1999).
[11] X.Y.Wang and D.Y.Li, Wear, 255, 836-45 (2003).
[12] N.R.Tao, Z.B.Wang, W.P.Tong, M.L.Sui, J.Lu and K.Lu, Acta Mater., 50, 4603-16 (2002).
[13] G.Liu, S.C.Wang, X.F.Lou, J.Lu and K.Lu, Scripta Mater., 44, 1791-95 (2001).
[14] H.W.Zhang, Z.K.Hei, G.Liu, J.Lu and K.Lu, Acta Mater., 51, 1871-81 (2003).

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