Formation of Homogeneous Lamellar Structure with Nano-scale Grains under Material Surfaces by Fine Particle Bombarding

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ABSTRACT
In this study, the effects of the fine particle bombarding treatment: FPB on crystal structure in the material, were studied. After the FPB treatment, a lamellar work-hardening domain (a lamellar structure) was observed on the specimen surface with the scanning electron microscope: SEM. The crystal orientations in the domain of the lamellar structures were analyzed by the electron backscattered diffraction method: EBSD method with SEM. From the results of Image quality map: IQ map and inverse pole figure map: IPF map obtained by the EBSD method, the several refined crystal grains of sub-micro meter or less in diameter were observed in the lamellar structure. However, the conventional FPB processing locally forms the lamellar structures. In this study, the specimens were moved at the constant speed during the FPB processing and we succeeded in the formation of continuous lamellar structure on specimen surfaces. The observations and hardness test of the microstructure was conducted. The crystal grains in a lamellar structure were nanocrystallized and the hardness in the lamellar structure raised more than 5 times prior to FPB treatment.

KEY WORDS
Fine Particle Bombarding, Lamellar structure, Nanocrystallization, EBSD measurement

INTRODUCTION
The previous shot-peening treatments have spread industrially as the effective method to give the compressive residual stress to a material surface, in order to increase the fatigue strength of the machine components[Suzuki, H., 2001]. Recently, FPB treatment is paid attention as the surface modification techniqu. In the Fine Particle Bombarding treatment : FPB treatment, beads of 50 - 200 μm in diameter at 100m/s or more are impacted on the matrerial surface. Therefore, various effects of FPB treatment has been reported, such as the control of surface roughness, refined structure, phase transformation and diffusion of beads material etc[Kagaya, C., 2006]. Especially, the nano crystallization of materials has received more attention and several results have been already reported [Altenberger, I., 1999]. However, the refinement of crystal grain, the transformation of microstructure or the formation mechanism of nano-scale grains by FPB treatment are not almost clarified. On the other hand, it is thought that the refined crystal grain exists in the lamellar structure after the FPB processing and the continuous lamellar structures on matrial surfaces effectively improves the static and fatigue strengths of materials.

In this study, microstructure of a material surface after the FPB treatment was observed with the EBSD method. In addition, by moving materials during the FPB processing, the formation of the continuous lamellar structure on the material surfaces was tried.

EXPERIMENTAL PROCEDURE

Materials and specimens

In this study, pure iron (SUYP-1: JIS C2504) and rolled steel for general structure (SS400: JIS G3101) were used. Table 1 shows the chemical composition of these materials. These materials were cut off into specimens with 30×30×7mm^3 and they were heat-treated in the electric furnace. Heat treatment condtion was sumalized in Table 2. The sufaces were polised and lapped, and then FPB processing was conduced. FPB treatment conditions were shown in Table 3. For FPB treatment, a direct pressure type FPB machine (FUJI KIHAN CO.,LTD) was used.

FPB proccessing for constitution of continuous lamellar structure

Specimens were clamped by the vice fixed on the work table as shown in Fig. 1 and the work table was moved at the three types of conditions during FPB processing.The moving conditions are surmalized in Table 4.

Table 1. Chemical composition of materials

	Chemical composition (mass.%)				
Material	C	Si	Mn	P	S
SUYP-1	0.01	0.09	0.12	0.015	0.005
SS400	0.17	0.14	0.60	0.17	0.07

Table 2. Heat treatment of materials

Material	Heat treatment condition	HV
SUYP-1	Annealed (950°C, 3.6ks)	90
SS400	Annealed (650°C, 3.6ks)	140

Table 3. Fine particle bombarding conditions

Material of shot	FHS	Air pressure (MPa)	0.2~0.8
Diameter of shot (μm)	50~100	Bombarding time (sec)	10~100
Hardness of shot (HV)	900	Nozzle (mm)	φ7×φ7
Coverage (%)	>100	Incidence distance (mm)	40, 100

* FHS: Fine high speed steel shot

Table 4. Moving conditions for FPB processing

Condition	Hold time, t_h (sec)	Moving distance d_s (mm)
A	1	1
B	1	2
C	2	1

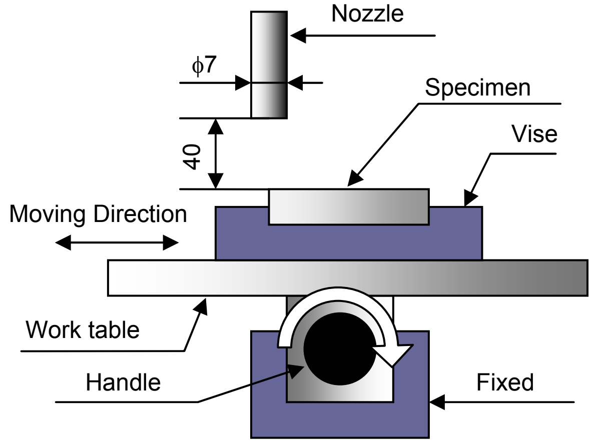


Fig. 1. Schematic of FPB processing.

Microstructure observation

After FPB treatments, cross sections of specimens were obseved by the optical microscope and the scanning electron microscope (SEM). Cross sections were mirror finished, and etched by 10% Naytal liquid.

Hardness measurement

Hardness distribution on cross section of specimens was measured by micro Vickers hardness test (Matsuzawa Seiki, MZT-522) with a load of 5mN. The measurement region was from the specimen surface to 60μm, and the distance between measurement points was 5μm.

Nano-scale grain evaluated by EBSD method

The evaluations of grain size and crystal orientation after FPB treatments were analized by the electron backscattered diffraction method: EBSD method. When an electron beam is focused on the specimens in SEM, the electrons are inelastically scattered in specimens and diffracted from the set of parallel lattice planes. Then, Kikuchi pattern is formed on the EBSD screen and the crystal orientations are determined by the analysis of Kikuchi patterns. In this study, automatic crystal orientation measurement was carried out by field emission scanning electron microscope: FE-SEM (JEOL JSM-7000F) with EBSD system (TSL OIM 4.6). In this equipment, a maximum spatial resolution for EBSD measurement is considered to be about 2 nm. In this study, the measurement step was 50nm.Threfore, nano-scale size grains can be evaluated by EBSD method.

EXPERIMENTAL RESULTS AND DISCUSSION

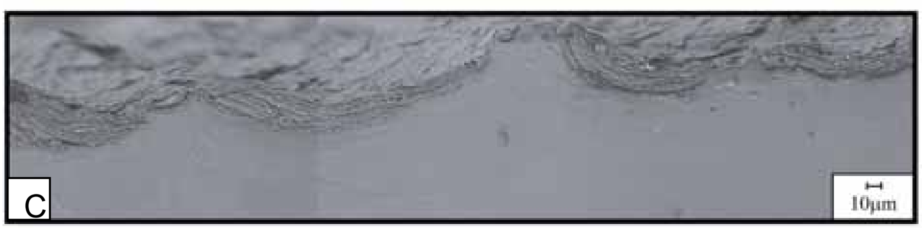
Microstructure observation

Figure 2 shows SEM micrographs of the cross section of pure iron after FPB treatment for various conditions. (a) , (b) and (c) are the micrographs for the conditions A, B, and C, respectively. Close to the materials surface, the continuous lamellar structure was formed by moving the specimen during FPB processing. On each moving conditions, the regions of the formed lamellar structure for conditions A, B, and C, were 40μm, 30μm and 20μm from the specimen surface, respectively. It is expected that the specimen surface after the FPB treatment locally become concavo-convex shape. By the additional FPB treatment, the micro-buckling or the fold of the micro convex parts is occurred and lamellar structure is formed on specimen surfaces. Figure 3 shows the enlargement of the lamellar structure shown in Fig. 2(a). Refined crystal grains with 100~500nm in diameter were observed inside the lamellar structure in the region of about 10μm from the surface.

Figure 4 shows the SEM micrograph of SS400 specimens performed FPB treatment at condition as same as pure iron. In this case, the incidence distance in FPB condition was 100mm and the defferent distance from the condition of pure iron. Therefore, the lamellar structures different from pure iron were locally formed.

(a) Condition A

(b) Condition B



(c) Condition C

Fig. 2. SEM micrographs of SUYP-1 specimen for various FPB consitions.

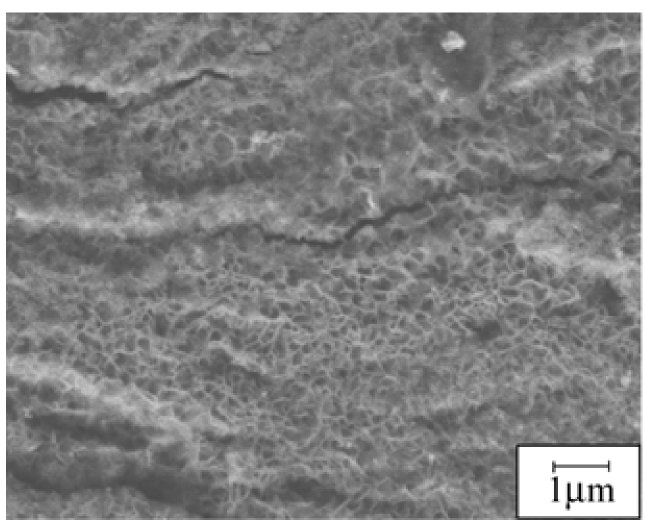


Fig. 3. SEM micrograph of SUYP-1 specimen with lamellar structure after FPB treatment (Condition A).

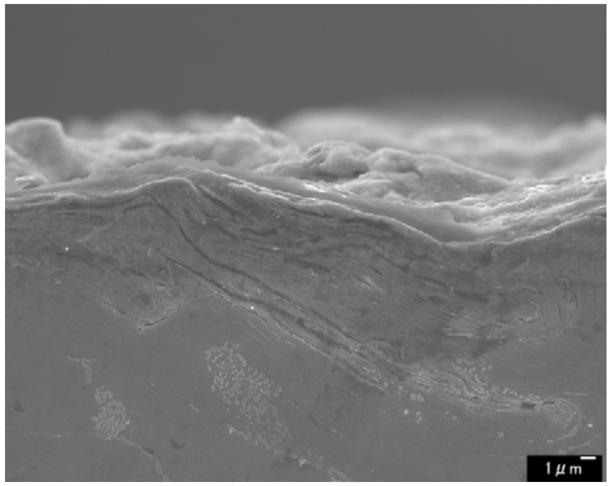


Fig. 4. SEM micrograph of SS400 specimen with lamellar structure after FPB treatment (Condition A).

Hardness measurement

Figure 5 shows the hardness distribution of pure iron close to the surface in each treatment condition. The abscissa is the distance from the specimen surface, the ordinate is Vickers hardness. Vickers hardness was mesured at FPB treatment surface to inside 60μm. As shown in Fig. 5, the hardness in the lamellar structure was more than 5 times the value before FPB treatment for each condition. The region corresponded to the width of lamellar structure (Fig. 3) and the position of maximum hardness was not on the FPB treatment surface but on a little inside from surface. The hardness has decreased gradually from in the outside of lamellar structure. The reason is thought that the nano-crystal grains or layers are formed in a lamellar structure.

Evaluation of the refined crystal grain by EBSD method

Figure 6 shows the EBSD measurement result of the lamellar structure after the FPB treatment. (a), (b) and (c) indicates the SEM micrograph of measurement area, the image quality map: IQ map, and Inverse pole figure map: IPF map, respectively. In this measurement, the average of IQ value was 128 and that of confidential index value was

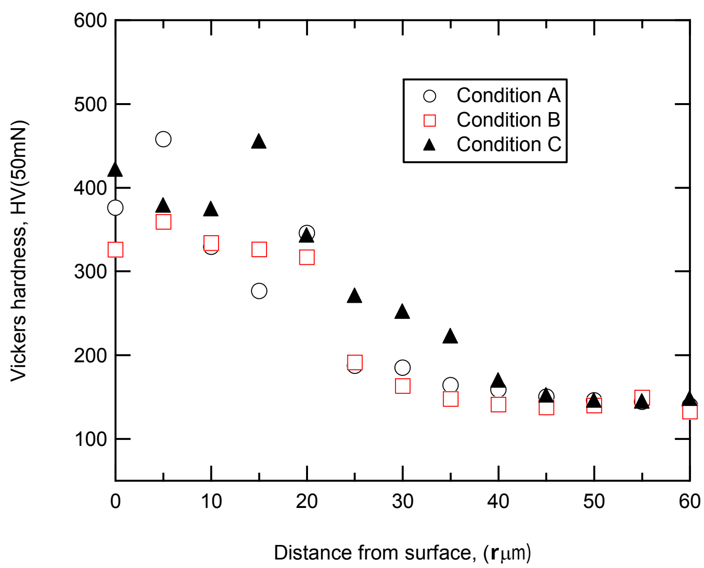
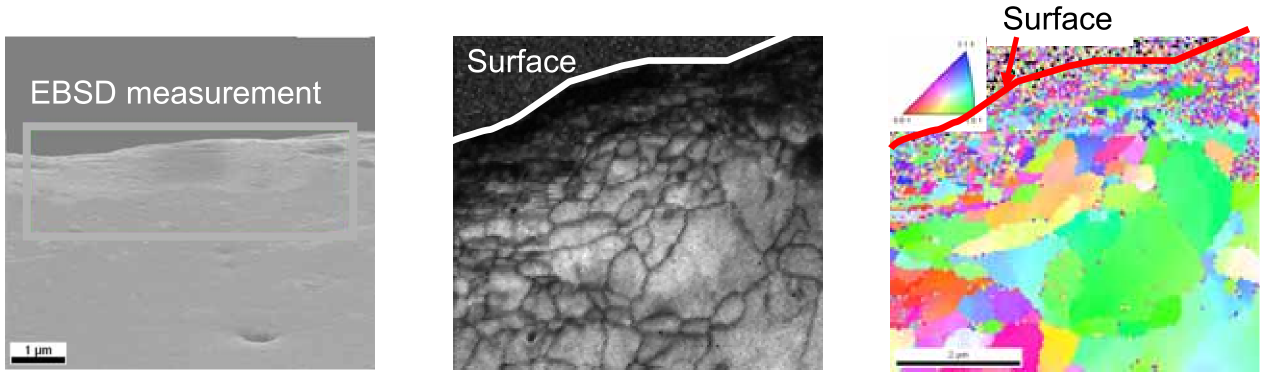


Fig. 5. Hardness distribution of SUYP-1 for various conditions.



(a) SEM micrograph (b) IQ map (c) IPF map

Fig. 6. EBSD measurements for SUYP-1 after FPB treatment.

0.56. The distribution of the grains was obviously observed with EBSD method and the grain size was gradually decreased toward to the specimen surface in Fig. 6(b). Near the specimen surface, the refined crystal grains of several 10 nm were able to be observed by EBSD method, as shown in Figs. 6(b) and (c). From IPF map, the surface lamellar structure had flattened nano-scale grains and the crystal orientation was random, as shown in Fig. 6(c). The average diameter of a crystal grain in measurement area was about 300nm. As most general method of the estimation of refined crystal grain size after the FPB treatment, the diffraction patterns obtained by the transmission electron microscope: TEM has been widely used. In this study, the refined crystal grains were observed more directly and simply with EBSD method than TEM. Therefore, EBSD method was the powerful and effective method to observe and analyze the refined crystal grains in lamellar structures.

And, it was reported that the temperature of material during the FPB processing rised to about 1000℃ [Miyahara, T., 2005]. Therefore, it is thought that the refined crystal grains were formed in a lamellar structure because of the synergistic effects of heat treatment and repeated heavy-worked process by FPB treatment.

CONCLUSIONS

FPB treatments were performed to pure iron (SUYP-1) and rolled steel for general structure (SS400) during moving the specimens, the crystal structure was observed with SEM and EBSD method. Results obtained from this study were summarized below.

(1) The continuous lamellar structure was formed on specimen surfaces by moving the specimen at constant speed during FPB processing.

(2) The hardness in the lamellar structure raised more than about 5 times of base material hardness, and the structure became refined.

(3) The refined crystal grains with 100nm or less and the crystal orientation were observed with EBSD method. EBSD method was the powerful tool to observe the refined crystal grains.

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