Influence of Annealing on Strength of Ultrafine Grained Low Carbon Steels by ECAP

T. Akita[1,a], M. Gotoh[1,b], S.V. Dobatkin[2,c], K. Kitagawa[1,d] and Y. Hirose[1,e]

[1]Graduate school of Natural Science and Technology, Kanazawa University,

Kakuma, Kanazawa, Ishikawa, 9201192, Japan

[2]Moscow State Institute of Steel and Alloys (Technological University),

Leninsky pr.4, Moscow, 119049, Russia

[a]akita@hakusankiko.co.jp, [b]masahide@cs.s.kanazawa-u.ac.jp, [c]dobatkin@ultra.imet.ac.ru

[d]kitagawa@t.kanazawa-u.ac.jp, [e]hirose@kenroku.kanazawa-u.ac.jp

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Abstract. In the present study, ultra fine-grained low carbon steel samples were processed by equal channel angular pressing (ECAP). Mechanical properties of the specimens annealed statically at several temperatures were evaluated by tensile and hardness test. In addition, grain sizes of the specimens were measured by SEM-electron back scattering pattern (SEM-EBSP) and X-ray diffraction analysis. Differential scanning calorimetry (DSC) measurement also evaluated thermal reactions in anneal process of the specimen. As a result, the grain size was changed at the temperature between 550^{o}C and 600^{o}C drastically and the tensile strength also became lower at the same temperature. The relation between yield stress and averaged grain diameter of specimens obeyed the Hall-Petch relation except the normalized specimen. Behavior of grain growth and recovery in structural observation by EBSP corresponded to reaction signal of the DSC curve.

Introduction

Ultra fine-grained metals achieve very high yield strength according to the Hall-Petch relation. Severe plastic deformation (SPD) is effective method to obtain such fine-grained materials. Equal channel angular pressing (ECAP) is a typical technique for obtaining ultra fine-grained bulk metals [1]. ECAP has been applied to produce various ultra fine-grained bulk metals, after Segal et al. introduced that technique. The light metals such as Cu and Al were applied to ECAP at first because of its high deformation capability. Moreover, Fe and steels had been applied as the technique was improved in recent years. Grain boundaries of ultra fine-graied materials by ECAP are in non-equilibrium state. Therefore the enhanced grain growth would be expected in ultra fine-graied materials compare with usual grained ones produced by the conventional cold working process. It would influence on mechanical properties of materials in addition.

In the present study, the ultra fine-grained low carbon steel samples were processed by ECAP. Mechanical properties were evaluated after static annealing at several temperatures. SEM-electron back scattering pattern (SEM-EBSP) analysis was carried out to obtain several structural information of the ultra fine-grained steel and X-ray diffraction analysis was adapted to the steel samples. Differential scanning calorimetry (DSC) curves were obtained in order to compare with structural information obtained by EBSP.

Experimental

The present ultra fine-grained steel was processed by ECAP, one of typical severe plastic deformation technique used most widely for fabrication of bulk ultra fine-grained metals. Mn rich low carbon steel was used in the present investigation. The chemical composition of the material is shown in table 1. Before ECAP, the base steel was normalized at 920^{o}C for 30 min in electrical furnace and cooled at air atmosphere. The structure of normalized steel was dual phase structure consisted of ferrite and pearlite, both phase of grain size were about 10 μm. The ECAP of the sample of 20 mm in diameter and 80 mm in length was conducted using a die with route Bc at 300C^{o}, the angle of intersection between two channels was Φ=110^{o} and the angle representing the outer arc of curvature where the two channels intersect was ψ=0^{o}. By a general relationship allowing one to calculate the strain value of the billet during ECAP for N pass [1], the accumulated strain for 4 passes in the present study was about 3.2.

The static annealing of ECAPed specimens were carried out at the temperatures of 500^{o}C, 550^{o}C and 600^{o}C by the electrical furnace for 5 hours in air. All de-carbonized layers were removed from the specimen surface by machining or electrical polishing in order to avoid the influence on experimental data.

Mechanical tensile test of the samples of 5 mm in width, 1 mm in thickness and 10 mm in gage length was performed by tensile test machine at a crosshead velocity of 5 mm/min. Micro Vickers hardness was measured at a load of 300 gf. The structural examination was performed by SEM-EBSP analysis and SEM secondary electron image. The EBSP images of the as-ECAPed, the 500^{o}C annealing and the 550^{o}C annealing specimen were obtained from 15 x 15 μm^{2} area at the interval of 0.02 μm with hexagonal grid, whereas that of the 600^{o}C annealing specimen was obtained from 30 x 30 μm^{2} area at the interval of 0.10 μm. Williamson-Hall (WH) plots of the specimens were made through the X-ray diffraction experiment using Cu-Kα radiation. Differential scanning calorimetry (DSC) curves were obtained at the condition of ΔT=2 K/min in order to compare with structural information obtained by EBSP.

Table 1. Chemical composition of present material [mass%].

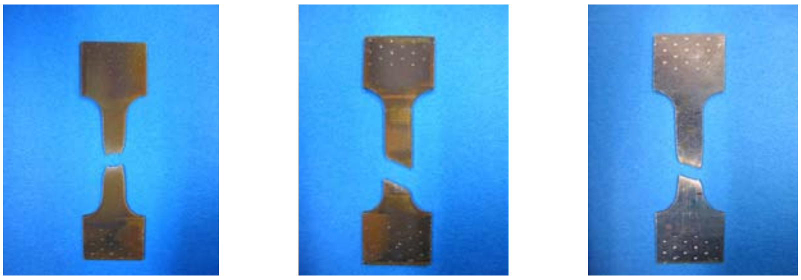
C	Si	Mn	Ni	S	P	Cr	N	Cu	Al	V	Nb	Ti
0.09	0.62	1.3	0.1	0.008	0.012	0.1	0.003	0.1	0.026	0.08	0.01	0.021

Results and Discussion

Mechanical Properties. Some mechanical properties of present specimens are shown in table 2. The yield stress and the ultra tensile strength after ECAP were about 2 times higher than before ECAP though the total elongation after ECAP was shorter than before ECAP. The mechanical properties were slightly changed for the annealing up to 550^{o}C, however their properties were extremely affected by the annealing at 600^{o}C. Moreover, this annealing influenced the micro Vickers hardness of each specimen as well as the yield stress. Figure 1 shows some photographs of the specimen after tensile test. The normalized specimen was broken with necking phenomena as well as ordinary ductile steels, while the as-ECAPed specimen had brittleness. Because the fracture occurred at an angle of 45^{o} to the axial load axis, it was thought that the specimen was fractured at once after the material was satisfied with the von Mises yield criterion. The specimens after annealing were fractured the same as the as-ECAPed specimen except the 600^{o}C annealing, and the necking was slightly existed in the 600^{o}C annealing specimen.

Table 2. Mechanical properties by tensile and hardness test.

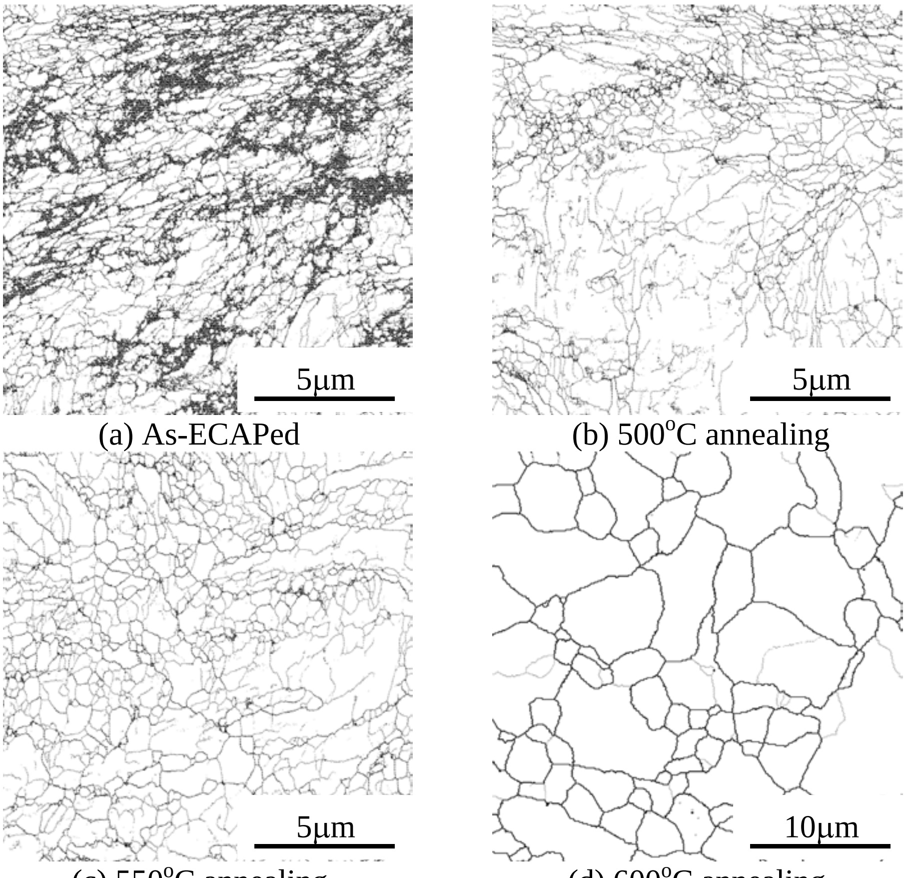
	Yield stress, [MPa]	Ultra tensile strength, [MPa]	Elongation, [%]	Vickers hardness, [GPa]
Normalized specimen	369	505	36	1.9
As-ECAPed	965	972	15	3.3
500^{o}C annealing	917	921	16	3.1
550^{o}C annealing	790	863	16	2.9
600^{o}C annealing	559	632	21	1.6



(a) Normalized (b) As-ECAPed (c) 600°C annealing

Figure 1. Fractured specimens after tensile test.

Microstructure. The grain boundary maps of specimens obtained by EBSP analysis are shown in fig.2. High angle boundaries (HAGB's), which are over 15 degrees, and low angle boundaries, which are between 2 degrees and 15 degrees, are indicated by black lines and gray lines, respectively. The picture of the as-ECAPed specimen was remarkable about black lines, that is, it is indicated that a lot of refinement grains had HAGB's. Hansen explained that grain subdivision involved with GNB (Geometry Necessary Boundary) and IBD (Incidental Dislocation Boundary) during plastic deformation was the cause of continuous structural refinement in fcc metals [2,3]. After annealing, recognizable black lines of the as-ECAPed specimen were eliminated a lot. Moreover, recrystallization took place gradually as the annealing temperature increased up to 550°C. Over 600°C, the heat given hastened the grain growth more, and then, almost all low angle boundaries disappeared. As for the morphology of grain size, it was not homogeneous, small and big grains were in the specimen together.

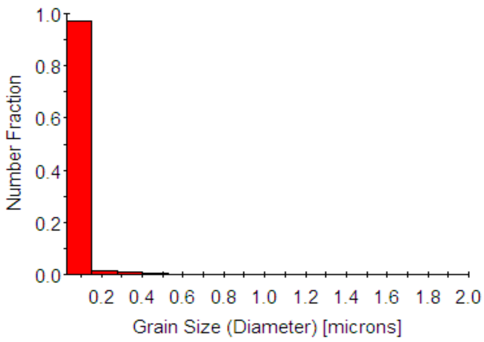


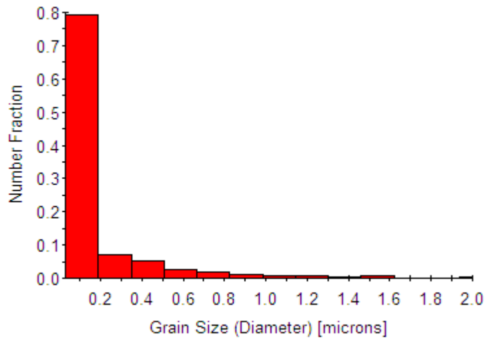
(a) As-ECAPed (b) 500°C annealing

(c) 550°C annealing (d) 600°C annealing

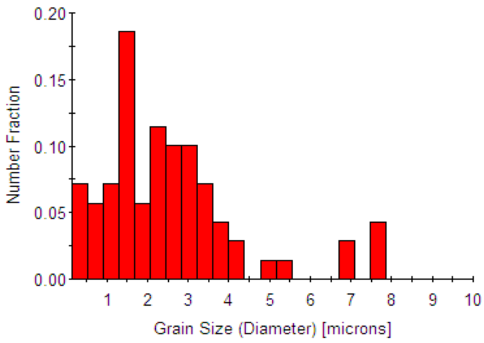
Figure 2. Grain boundary maps of specimens.

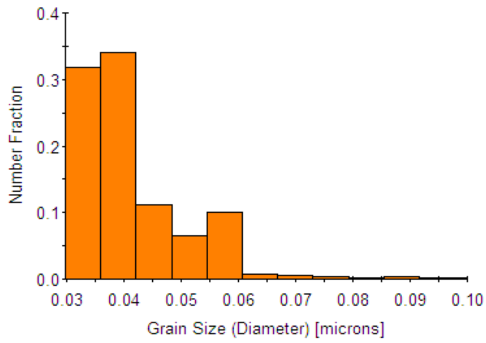
Figure 3 shows the histograms of grain diameter of the as-ECAPed, the 550°C annealing and the 600°C annealing specimen. The graph of 500°C specimen and 550°C specimen closely resembled one another in distribution. The grain size of the as-ECAPed specimen was almost under 200 nm. Further, although many small grains under 200 nm remained, annealing up to 550°C increased the number fraction of grain size over 200 nm. The distribution of grain size of the 600°C annealing specimen extended compared with another annealing specimens. In addition, the shape of distribution was lognormal distribution [4], the distribution of another ECAPed specimen was also lognormal distribution, for instance, as shown in fig.3 (d). With respect to the average grain size, the average size of the as-ECAPed, the 500°C annealing, the 550°C annealing and the 600°C annealing specimen were 51 nm, 96 nm, 157 nm and 2.6 μm respectively. Figure 4 shows the SEM secondary electron images of the as-ECAPed and the 600°C annealing specimen. The picture of the as-ECAPed specimen is able to divide into two parts; one of the parts consists of black grains and white expanding particles, the other one is occupied by only black grains. In addition, it is seem that the part with expanding white particles in fig.4 (a) corresponds to the part occupied by black lines in fig.2 (a), that is, HAGB's. Figure 4 (b) is the high magnification micrograph of the as-ECAPed specimen. White expanding particles is not understood at present, but they may be cementite particles divided from perlite by SPD. On the other hand, the fine white dots could be found in the ferrite grains, and they may be also cementite particles. After 600°C annealing, spherical white particles were recognized.





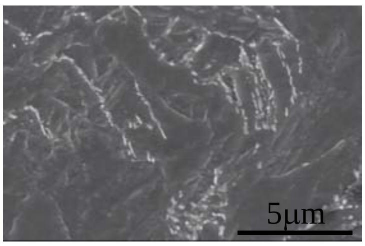
(a) As-ECAPed (b) 550°C annealing

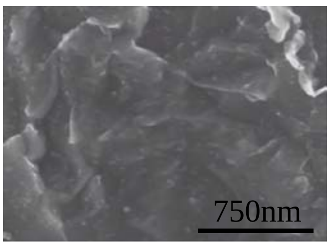


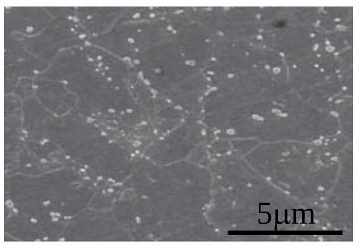


(c) 600°C annealing (d) As-EBAPed (under 100nm)

Figure 3. Histograms of grain diameter.







(a) As-ECAPed (b) As-ECAPed (High magnification) (c) 600°C annealing

Figure 4. SEM micrographs.

Hall-Petch Stress-Grain Size Relation. The relation between grain size and yield stress of specimens are shown in fig.5. The dots of the as-ECAPed and another annealing specimens were on the straight line according to the Hall-Patch relation. However, only the normalized specimen was not satisfied with this straight correlation. On the other words, two correlations exist in this figure, and they are distinguished under 1 μm from over 1μm. Although understanding of this phenomenon has not been clear yet, this have been found out and discussed in many literatures until now [5].

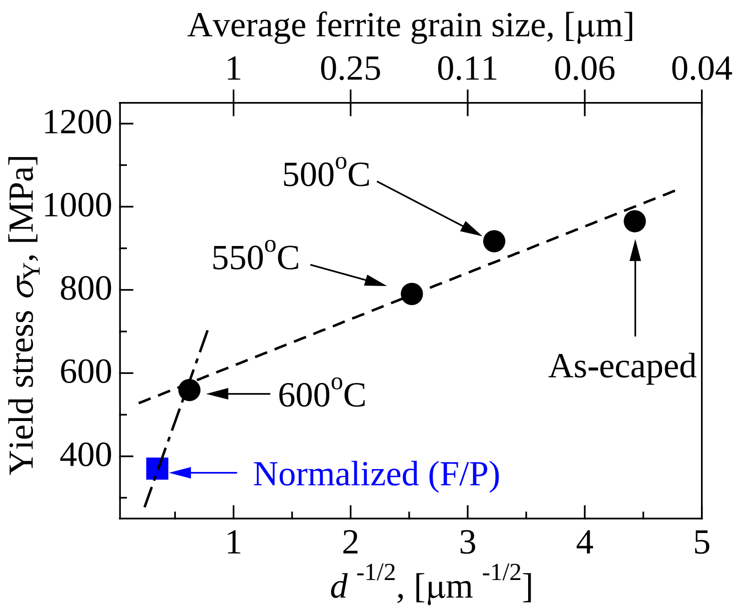


Figure 5. Relation between grain size and yield stress on present specimens.

Recovery and Recrystallization by Annealing. The parameter of micro strain ε_v and volume weighted average diameter d_v, which is related with grain size, were obtained by WH integral breadth method with X-ray diffraction experiment. Table 3 shows the results of measurement. The accumulated micro strain by SPD fully decreased with annealing up to 500°C. Moreover, the average diameter after annealing increased as well as the result of EBSP. Absolute value of the volume weighted average diameter also closely resembled the average diameter by EBSP. Figure 6 shows DSC curves of the as-ECAPed specimen by using the normalized specimen as a reference material. Here, the second run means that the specimen used in the first run is applied to the second experiment. In the first run, a little peak was found out at the temperature between 570°C and 640°C. This reaction is endothermic, then, it is suggested that the grain growth took place in the present experiment [6]. That is, the rapid grain growth found in EBSP analysis was in accord with the result of DSC. In comparison between the first run and the second run, the behavior of curve was different over 350°C obviously. From the result, it is supposed that the recovery phenomena took place over 350°C. Accordingly, the gradual grain growth by annealing under 550°C found in fig.2 (a)~(c) is expected with DSC curve.

Table 3. Volume weighted average diameter and micro strain of as-ECAPed and 500°C annealing specimen.

	d_v, [nm]	ε_v, [x10^{-6}]
As-ECAPed	67	1855
500°C annealing	99	27

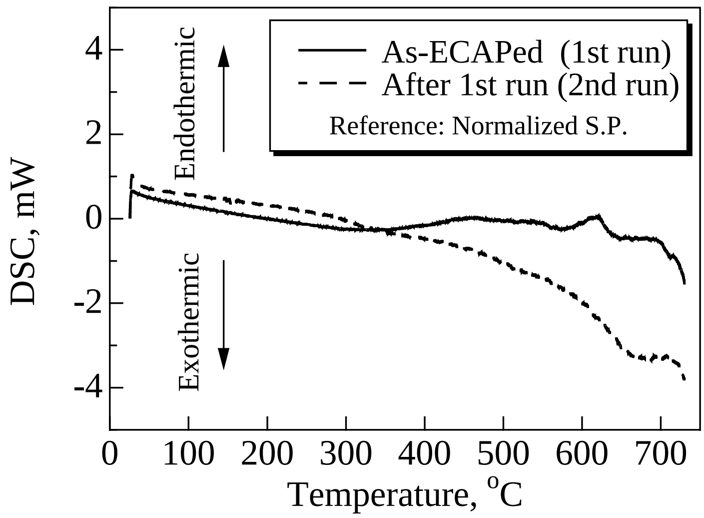


Figure 6. Thermal reaction of as-ECAPed specimen by DSC curve.

Summary

The ultra fine-grained low carbon steel samples were processed by ECAP. Mechanical properties of the specimens annealed statically at several temperatures were evaluated by tensile and hardness test. In addition, grain sizes of the specimens were measured by EBSP and X-ray diffraction analysis. DSC measurement also evaluated thermal reactions in anneal process of the specimen. The yield stress and the ultra tensile strength after ECAP were about 2 times higher than before ECAP due to existence of ultra fine grains. They were slightly changed for the annealing up to 550^{o}C, extremely affected by the annealing at 600^{o}C. Annealing changed grain sizes of the specimens, the grain size of the 600^{o}C annealing specimen also became the largest of all annealed specimen. The relation between yield stress and averaged grain diameter of the as-ECAPed specimen and its annealing specimens obeyed the Hall-Petch relation. Behavior of grain growth and recovery in structural observation by EBSP corresponded to reaction signal of the DSC curve.

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