Work-Hardening Mechanism during Super-Heavy Plastic Deformation in Mechanically Milled Iron Powder

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Mechanical milling using a high energy ball mill was applied to a commercial pure iron powder to introduce a large deformation strain into the matrix. Microstructural changes during deformation and annealing were investigated for the mechanically milled powder, by means of X-ray diffractometry, transmission electron microscopy and hardness testing, in order to clarify the work-hardening mechanism of iron during super-heavy plastic deformation. Through the mechanical milling of 360 ks, nanocrystalline grains of 20–50 nm were formed within the powder, and the resultant powder was work-hardened to Hv950. Hardness of the mechanically milled powder mainly depends on the crystallite size(d); the size of dislocation cells, subgrains and nanocrystalline grains. In the mechanically milled powder, a linear relationship was confirmed between hardness and the reciprocal of crystallite size (d^{-1}), supporting the strain hardening to Hv500. While it was also confirmed that the work-hardening mechanism of iron gradually changes from strain hardening to grain refining strengthening in the hardness range of around Hv600, where crystalline grains are refined less than 100 nm through heavy deformation. In the hardness range above Hv600, the Hall-Petch relationship was realized to the crystalline grain size.

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I. Introduction

Work-hardening of metals is usually explained by the decrease in mobility of dislocations owing to the formation of dislocation cells, namely, strain hardening. The strain hardening is known to be a linear function of cell size (d^{-1}).

On the other hand, a great attention is paid to the ultra grain refining to 1 μm or less which can be achieved by super-heavy plastic deformation of metals; wire drawing⁽¹⁾⁽²⁾, equal-channel angular pressing (ECAP)⁽³⁾⁽⁴⁾, mechanical milling (MM)^{(5)–(15)}, etc. Of these techniques, MM has a great advantage of giving an extremely large strain into metals through the cyclic process of milling action; forging, agglomeration and fracture of powder particles, although there is a troublesome problem on consolidation of powder. In many metals such as pure iron⁽⁶⁾⁽⁷⁾, Fe–Cu alloy⁽⁸⁾⁽⁹⁾ and Fe–C alloy⁽¹⁰⁾⁽¹¹⁾, it has been reported that grain size is decreased to a few tens nm or less after MM treatment. Hardness of the nanocrystalline materials increases with decreasing grain size on the Hall-Petch relationship for the grain refining strengthening⁽⁶⁾⁽⁷⁾. But there is an open question about the detail of strengthening mechanism of the nanocrystalline materials produced by super-heavy plastic deformation: Of strain hardening and grain refining strengthening, which is the main mechanism on

strengthening of the nanocrystalline materials?

In this paper, microstructural changes during deformation and annealing were investigated for a commercial pure iron powder heavily cold-worked through MM in order to clarify the work-hardening mechanism of iron during super-heavy plastic deformation.

II. Experimental Method

A commercial pure iron powder (99.2 mass%Fe, and a particle size of -100 mesh) was used as a starting material. The amount of oxygen in the iron powder was about 0.3 mass%[†]. The iron powder (100 g) was put into a stainless steel pot (SUS304, 0.4 liter volume) with steel balls (SUJ2, 10 mm in diameter), and then mechanically milled in an argon gas atmosphere with a planetary ball mill up to 360 ks. The weight ratio of the steel balls to the iron powder was 12. As can be seen in **Table 1**, the total amount of C and N atoms is less than 0.1 mass% even in the specimen mechanically milled for 360 ks, hence the influence of contamination from milling media and/or atmosphere is considered to be negligible on hardening behavior of the iron powder during MM. Annealing of the mechanically milled iron (MM-Fe) powder was carried out in a hydrogen gas at-

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[†] Most of oxygen in the iron powder exists as oxides like FeO/Fe₃O₄, Cr₂O₃, MnO, SiO₂, etc. and the volume fraction of these oxides can be estimated to be as small as about 0.02 in total. The contribution of the dispersion strengthening by the oxides is thus considered to be relatively small to work-hardening of the iron.

Table 1 Chemical compositions of mechanically milled iron powders (mass%).

	C	N	Cr	Si	Mn	P	S
0 s	0.007	—	0.03	0.04	0.24	0.006	0.005
36 ks	0.005	0.001	0.10	0.05	0.26	0.006	0.008
108 ks	0.018	0.003	0.22	0.05	0.26	0.006	0.008
360 ks	0.022	0.070	—	—	—	—	—

mosphere to avoid oxidizing.

X-ray diffraction (XRD) was performed with a diffractometer using $\text{CoK}\alpha$ radiation ($\lambda = 0.1790$ nm) and graphite monochromator, and the XRD lines were separated to $K_{\alpha 1}$ and $K_{\alpha 2}$ components by Rachinger's method⁽¹⁶⁾. Crystallite size ($d_{hkl}^{\dagger\dagger}$; the size of crystalline grains bounded by high angle boundaries, dislocation cells or subgrains within powder particles) and local strain (ε_{hkl} ; internal elastic strain produced by dislocations, lattice distortion, etc.) were estimated on the basis of the Hall-Williamson plot⁽¹⁷⁾ given by the eq. (1).

$$\beta_s \cos \theta / \lambda = (2\varepsilon_{hkl}) \sin \theta / \lambda + K / d_{hkl} \quad (1)$$

where θ is Bragg angle, K is the Scherrer constant (≈ 0.9 for crystals of cubical shape), and β_s is the full width at the half maximum height (FWHM) of $K_{\alpha 1}$ line with the correction of instrumental line broadening. In this study, the FWHM values were measured for α -Fe (110) and (220) reflections. The instrumental line broadening was obtained from the FWHM value (β_r) of $K_{\alpha 1}$ line of an annealed iron powder and removed from measured FWHM value (β_m) of $K_{\alpha 1}$ line of MM-Fe powder on the following eq. (2)⁽¹⁸⁾.

$$\beta_s^2 = \beta_m^2 - \beta_r^2 \quad (2)$$

Microstructure of MM-Fe powder was observed with transmission electron microscope (TEM) at the acceleration voltage of 200 kV for bulk samples prepared by the cold-pressing method proposed by Ameyama *et al.*⁽¹²⁾: MM-Fe powder was loaded into a cylindric stainless steel vessel and then pressed at room temperature. Disks of $\phi 3$ mm were cut out from the pressed samples by electro-spark machining and then sliced to films of about 0.1 mm in thickness. Some of them were subjected to annealing. These films were finally thinned by jet-polishing in the electrolyte solution of 90% acetic and 10% perchloric acid under the electrolytic current of about 90 mA. Mean crystalline grain size was also measured from the dark field images of TEM. Hardness of MM-Fe powder was represented by the average of eight measurements in micro-Vickers hardness testing (Load; 0.245–0.49N) for the sample prepared by the following process: Powder particles were buried in an epoxy resin and their cross sections were prepared by polishing with buff and ceramic paste.

III. Experimental Results

1. Microstructures of mechanically milled iron powders

Figure 1 shows the change in hardness of MM-Fe powder⁽⁷⁾. In this figure, the increment of hardness by 98% cold-rolling is also shown for a commercial pure iron sheet. The reduction area of 98% (true strain = 4.5) is usually in a category of heavy plastic deformation for bulk material, nevertheless hardness of the iron sheet is as small as Hv280. On the other hand, hardness of the MM-Fe powder greatly increases with elongated milling time, and finally reaches the level of Hv950. This result indicates that conventional heavy deformation is just at the initial stage of work-hardening of iron. As the MM-Fe powder is hardened, XRD lines were confirmed to broaden appreciably. Figure 2 gives the changes in local

Fig. 1 Change in Vickers hardness of an iron powder with mechanical milling. Increment of hardness by 98% cold-rolling is shown in this figure for a bulk iron.

Fig. 2 Changes in local strain and crystallite size measured by X-ray diffractometry as a function of hardness in mechanically milled iron powders.

^{††} The crystallite size (d_{hkl}) is defined as the cube root of the volume.

Fig. 3 Transmission electron micrographs of an iron powder work-hardened to Hv470 by mechanical milling: Bright (a) and dark (b) field images, and the diffraction pattern obtained from the selected area 1.5 μm in diameter.

Fig. 4 Transmission electron micrographs of an iron powder work-hardened to Hv950 by mechanical milling: Bright (a) and dark (b) field images, and the diffraction pattern obtained from the selected area 1.5 μm in diameter.

strain and crystallite size measured by the XRD as a function of hardness of MM-Fe powder⁽⁷⁾. With an increase of hardness in the MM-Fe powder, local strain rises to about 0.8% and tends to drop again in the hardness range of more than Hv800, while the crystallite size monotonically decreases to a few tens nm. The saturation of local strain in nanocrystalline materials has been observed in the other mechanically milled powders of Fe-Cu alloys⁽⁹⁾, AlRu intermetallic compound⁽¹⁴⁾, etc. **Figure 3** is TEM images of a MM-Fe powder work-hardened to Hv470. In such a middle stage of work-hardening, the substructure of deformed iron is characterized by fine crystallites bounded by low angle boundaries; cell walls and/or subgrain boundaries, as shown in the bright field image (a). The dark field image (b) reveals that there are two kinds of crystalline grains within the powder particle. One is “agglomerated grain” which consists of some dislocation cells and/or subgrains of 100 nm or less (arrow a). The other is “individual nanocrystalline grain” with large misorientations to the matrix and without the substructures (arrow b). The size of individual grains is about 50 nm or less. These crystalline grains are thought to be formed through local random motion of dislocations during cy-

clic plastic deformation of powder particles. Jang *et al.*⁽⁶⁾ have studied structural evolution of an iron powder during MM using shaker mill. They pointed out that dislocation cells are refined to about 10 nm at the initial stage of MM, and the dislocation cell walls are gradually changed to high angle boundaries during subsequent MM treatment, and finally individual nanocrystalline grains of about 6 nm are formed. **Figure 4** is TEM images of a MM-Fe powder work-hardened to Hv950. The diffraction pattern, which was obtained from the selected area of approximately 1.5 μm in diameter, shows that there are a lot of crystalline grains with different crystallographic orientations within the small area. Unclear contrast in the bright field image (a) is due to a complicated internal elastic strain (local strain of 0.7%). In the dark field image (b), we can see that individual nanocrystalline grains of about 20 nm are formed at random and agglomerated grains are also refined to about 50 nm in the size. The size of subgrains, which construct the agglomerated grains, is also about 20 nm. The crystallite size measured by the XRD was 24 nm for this specimen, and this means that the crystallite size represents the size of individual grains and of subgrains within the agglomerated grains. It must be also empha-

Fig. 5 Schematic illustration showing change in crystalline grain size distribution in an iron powder during mechanical milling.

sized that few dislocations are observed within individual grains refined to about 20 nm, despite containing a large elastic strain. In case of iron, elastic theory predicts that individual small particles become free from dislocations when the radius of small particles is less than 23 nm⁽¹⁹⁾, and this reasonably agrees with the above experimental value. Such a feature is the typical of nanocrystalline materials produced through super-heavy plastic deformation of metals⁽³⁾⁽¹¹⁾⁽¹³⁾.

Figure 5 illustrates the change in crystalline grain size distribution with MM treatment. When milling time was not long enough, the type of crystalline grains observed is of agglomerated grains and the size of agglomerated grains is large. With elongated milling time, the grain size distribution shifts toward the smaller side and the volume fraction of individual grains increases. At final stage, all of crystalline grains should be the type of individual grains with an ultimate grain size (about 20 nm or less for iron). In this study, however, it was difficult to obtain such an ultimate microstructure owing to the limitation of machine power of ball-mill machine.

2. Changes in microstructures of mechanically milled iron powders with annealing

Since annealing is helpful for releasing local strain, we can obtain further information about the relation between microstructure and local strain in MM-Fe powders by observing the structural evolution during annealing. **Figure 6** shows changes in hardness of the MM-Fe powders work-hardened to Hv340-950, as a function of annealing temperature⁽⁷⁾. Data of 98% cold-rolled iron sheet is also shown for reference. In the cold-rolled iron sheet, abrupt softening takes place at around 800 K in response to recrystallization. The MM-Fe powder work-hardened to Hv340 undergoes the similar softening. While in the MM-Fe powder work-hardened to Hv470, two softening stages appear: First one is abrupt softening between 700 and 900 K, and second one is gradual softening above 900 K. With increasing the initial hardness of MM-Fe powder, the abrupt softening at around 900 K becomes not significant in the overall softening

Fig. 6 Changes in hardness of iron powders work-hardened to several hardness levels by mechanical milling as a function of annealing temperature. Hardness change of a bulk iron subjected to 98% cold-rolling is also shown in this figure for reference.

Fig. 7 Changes in local strain and crystallite size measured by X-ray diffractometry as a function of annealing temperature in an iron powder work-hardened to Hv470 by mechanical milling.

curve. In the MM-Fe powder work-hardened to Hv950, the curve is characterized by a continuous softening from 600 to 1200 K.

The changes in crystallite size and local strain of the MM-Fe powder work-hardened to Hv470 are shown in **Fig. 7** as a function of annealing temperature⁽⁷⁾. There is no change in crystallite size in the temperature range below 700 K, but most of local strain is released by the annealing to 600 K. The decrease in local strain is thought to be ascribed to dislocation annihilation and polygonization[†]. Above 700 K, the crystallite abruptly becomes larger in the size, corresponding to the abrupt softening. This result indicates that hardness of the MM-Fe powder is affected hardly by the local strain, but greatly by the crystallite size. **Figure 8** shows the

[†] Polygonization results in decreasing total strain energy because dislocations configure each other to cancel their own elastic strain.

Fig. 8 Transmission electron micrographs showing microstructural change with 1.8 ks isochronal annealing for an iron powder work-hardened to Hv470 by mechanical milling. Diffraction patterns were obtained from the selected area $1.5\ \mu\text{m}$ in diameter.

microstructural change before and after the abrupt softening in the MM-Fe powder. Compared the TEM images (a) with Fig. 3(a), no significant change is observed on the size of subgrains and/or dislocation cells. It is again confirmed that, in this MM-Fe powder, the crystallite size corresponds to the size of subgrains and/or dislocation cells which construct agglomerated grains. On the contrary, the number of these crystallites is reduced after the annealing at 873 K (b). The annealing of 923 K–1.8 ks results in the formation of fresh grains of about $0.7\ \mu\text{m}$, as shown in Fig. 9. These findings lead to a conclusion that the softening between 700 and 900 K in Fig. 6 is caused by the growth of the dislocation cells and/or subgrains and the recrystallization involving the movement of high angle boundaries. The gradual softening above 900 K is attributed to the normal grain growth. For the MM-Fe powder work-hardened to Hv950, the changes in crystallite size and local strain are also shown in Fig. 10 as a function of annealing temperature⁽⁷⁾. As described in Fig. 4, the crystallite size agrees with that of individual nanocrystalline grains or of subgrains within agglomerated grains. The crystallites start to grow at the low temperature of about 600 K,

Fig. 9 Transmission electron micrograph showing the formation of fresh grains in an iron powder work-hardened to Hv470. The powder was annealed at 923 K for 1.8 ks.

where a half of local strain is released. The behavior of continuous softening is just corresponding to the change in crystallite size. Figure 11 shows microstructural

change by annealing for the MM-Fe powder. On the annealing of 673 K–1.8 ks, marked change is not observed in the size of nanocrystalline grains (mean crystalline

Fig. 10 Changes in local strain and crystallite size measured by X-ray diffractometry as a function of annealing temperature in an iron powder work-hardened to Hv950 by mechanical milling.

grain size; 37 nm), but the diffraction contrast in Fig. 11(a) becomes clearer in comparison with Fig. 4(a). After the annealing of 923 K–1.8 ks, which results in almost full release of local strain, the individual grains grow and agglomerated grains change to the fresh grains without subgrain boundaries. Mean crystalline grain size is about 70 nm for this specimen, thus corresponding to the crystallite size measured by XRD. The diffraction contrast in the Fig. 11(b) becomes clearer corresponding to the disappearance of local strain.

As a result, it should be noted that, hardness of the MM-Fe powders scarcely depends on the amount of local strain but mainly on the crystallite size; the size of dislocation cells, subgrains and nanocrystalline grains.

IV. Discussion

1. Origin of local strain in mechanically milled iron powders

We have already shown that local strain scarcely affects hardness of the MM-Fe powders. In this section, the origin of local strain will be discussed in association

Fig. 11 Transmission electron micrographs showing microstructural change with 1.8 ks isochronal annealing for an iron powder work-hardened to Hv950 by mechanical milling. Diffraction patterns were obtained from the selected area 1.5 μm in diameter.

Fig. 12 Changes in local strain and crystallite size measured by X-ray diffractometry as a function of annealing time at 673 K in an iron powder work-hardened to Hv750 by mechanical milling. Hardness change of the iron powder with the annealing is also shown in this figure.

with the substructures of MM-Fe powders. **Figure 12** shows changes in crystallite size, local strain and hardness during isothermal annealing at 673 K for the MM-Fe powder with the initial hardness of Hv750. There are no significant changes in the hardness and the crystallite size to the annealing time of 10 ks at least. The crystallite size is kept at about 50 nm. On the other hand, local strain drops abruptly at the early annealing stage of 0.6 ks, but local strain (0.3%) remains stably during the subsequent annealing. Similar result has already been obtained in Fe-24 mass%Cr-15 mass%Y₂O₃ alloy with nanocrystalline ferrite grains of about 20 nm⁽¹⁵⁾. In this case, local strain (about 0.5%) remains stably in correspondent to the ferrite grain size (about 20 nm), but it disappears in response to the abrupt grain growth from 20 to 100 nm which occurs at around 1200 K. These findings suggest that a certain amount of local strain is necessary to keep a nanocrystalline grain structure stable in terms of geometrical structure. **Figure 13** summarizes the changes in local strain and crystallite size before and after the annealing of 673 K-1.8 ks as a function of hardness of the MM-Fe powders. In the MM-Fe powders with the hardness of less than Hv500, in which the crystallites are thought to be corresponding to dislocation cells and/or subgrains, local strain almost fully disappears after the annealing. While in the MM-Fe powders with the hardness of more than Hv500, local strain is retained even after the annealing in relation to the decrease of the crystallite size. This indicates that the retained local strain probably comes from the area around the boundaries of crystallites in the MM-Fe powders. **Figure 14** schematically illustrates the microstructural change by

Fig. 13 Changes in local strain and crystallite size measured by X-ray diffractometry with annealing of 673 K-1.8 ks as a function of Vickers hardness of mechanically milled iron powders.

Fig. 14 Illustration showing microstructural change with annealing in an iron subjected to super-heavy plastic deformation by mechanical milling.

annealing in a MM-Fe powder with nanocrystalline grains. From above findings, we can interpret the origin of the local strain as follows. In as-milled powder, there are two strained fields; One is around dislocations, especially “geometrically-necessary dislocations”⁽²⁰⁾ in the cell walls and/or the subgrain boundaries within agglomerated grains. The other is around the grain boundaries which contain a large amount of lattice distortion and geometrically-necessary dislocations to accommodate the misfit between or among adjacent grains. With regard to grain boundary structure of nanocrystalline materials fabricated through super-heavy plastic deformation, Valiev *et al.*⁽³⁾ have recently pointed out that there is an elastically distorted layer around the grain boundaries. Besides, Ameyama *et al.*⁽¹³⁾ showed in mechanically milled powder that the grain boundary is characterized by complicated wavy interfaces. Such grain boundaries are thought to have a great number of defects

in comparison with common flat grain boundaries. Authors refer the area around the grain boundaries as “grain boundary layer”. When all of grains are refined to the ultimate grain size (about 20 nm or less for iron), which never allow the existence of dislocations within grains, local strain will arise only from the grain boundary layer. On annealing of the MM-Fe powders, local strain is released in response to recovery of the dislocations and of the defects around the grain boundary area, but a certain amount of local strain is kept for accommodating the misfit of the boundaries of nanocrystalline grains. Such strain might be released through the grain growth.

2. Strengthening mechanism of mechanically milled iron powders

The strain hardening of iron at heavy deformation has been investigated by wire drawing up to the true strain of 7, and it was observed that there is a linear dependence of the strength on the transverse cell size (d^{-1}) in the size range from 0.5 to 0.1 μm , where the flow stress reaches about 1.4 GPa ($\approx \text{Hv}470^\dagger$)⁽²¹⁾. Hardness of the MM-Fe powders is summarized in Fig. 15 as a function of the reciprocal crystallite size (d^{-1}) measured by the XRD. In the MM-Fe powders, which have dislocation cells and/or subgrains within large agglomerated grains, almost linear relationship is observed to the crystallite size of 60 nm, thus supporting the strain hardening to Hv500. While in the MM-Fe powders with the initial hardness of more than Hv600, the hardness exhibits significantly positive deviations from the linear line.

On the other hand, hardness of nanocrystalline materials is known to follow the Hall-Petch relationship on grain refining strengthening, when the crystalline grains are random in the crystallography⁽⁶⁾. Relations between Vickers hardness and the reciprocal square root of the sizes of crystalline grain (solid symbols) and crystallite (open symbols) in MM-Fe powders are shown in Fig. 16. The crystalline grain size and the crystallite size here correspond to the values measured by TEM observation and XRD respectively. Data of bulk iron produced from a MM-Fe powder⁽²²⁾ are also plotted in this figure. All of data are almost on the same line to 30 nm, excepting data of dislocation cells and/or subgrains.

These findings lead to a conclusion that the work-hardening mechanism of iron gradually changes from strain hardening to the grain refining strengthening in the hardness range of around Hv600, where crystalline grains are refined to less than 100 nm through heavy plastic deformation.

On the basis of the pile-up dislocation model in the Hall-Petch relationship, the minimum grain size ($D_{\min}$) being attained at room temperature is approximately expressed by the following eq. (3), by assuming that the grain size reaches a critical value when the number of pile-up dislocations (n) is equal to 1⁽²³⁾.

[†] The Vickers hardness number was converted by authors *et al.*, using the relation; σ_b (GPa) $\approx$ 0.003Hv.

Fig. 15 Relation between Vickers hardness and the reciprocal of crystallite size measured by X-ray diffractometry in mechanically milled iron powders.

Fig. 16 Relations between Vickers hardness and reciprocal square root of the sizes of crystalline grain (solid symbols) and crystallite (open symbols) in mechanically milled powders which were examined in this study. Data of bulk iron materials fabricated from mechanically milled powder are shown for Ref. (22).

$$D_{\min} = Gb / (K\tau^*) \quad (3)$$

where b is the Burgers vector (≈ 0.25 nm for iron), G is the shear modulus (≈ 80 GPa for iron), and the τ^* denotes the strength of a grain boundary for the onset of dislocation slip from the grain boundary. Then τ^* depends on the character of grain boundary of materials. K is the constant depending on the nature of dislocations (≈ 0.72 –1).

The yield stress varies with grain size yielding to the following eq. (4).

$$\tau_y = \tau_0 + (KGb\tau^*)^{0.5} \cdot D^{-0.5} \quad (4)$$

On the assumption that $\sigma_{0.2} = 2\tau_y$, the eq. (4) can be converted to

$$\sigma_{0.2} = \sigma_0 + 2(KGb\tau^*)^{0.5} \cdot D^{-0.5} \quad (5)$$

Since an experimental result; $\sigma_{0.2}$ (GPa) = $0.1 + 0.6 \times 10^{-3} \cdot D^{-0.5}$ ($\text{m}^{-0.5}$) has already been obtained in the grain size range up to $0.18 \mu\text{m}$ in the bulk iron produced from a MM-Fe powder⁽²²⁾, the comparison of this equation with the eq. (5) gives the value for τ^* as 3.2–4.5 GPa for $K=0.72$ –1. Finally, substituting the above value into the eq. (3), the value of $D_{\min}$ can be estimated to be 4–9 nm. When crystalline grains are refined to the size of $D_{\min}$, dislocations become hard to move within the grains. This means that the Hall-Petch relationship will be break down around this grain size range. That is, another deformation mode; grain boundary sliding might be activated in the grain size range.

Following the Hall-Petch relation described in Fig. 16, hardness of the iron should reach the level of Hv2000 at the $D_{\min}$ of 4–9 nm. However, in the grain size range below $20 \text{ nm}^{(11)(15)}$, hardness of nanocrystalline iron materials are much lower than the expected hardness. Hardness of nanocrystalline Fe–C alloys, for instance, levels off at around Hv1200 in the grain size range from 20 to 10 nm⁽¹¹⁾. This suggests the possibility of grain boundary sliding at room temperature. Languillaume *et al.*⁽²⁴⁾ have pointed out that, in nanocrystalline Ni_3Al produced by super-heavy deformation, the grain boundary structure largely affects the hardness of the material. This means that the strength of nanocrystalline irons depends not only on the grain size but also on the character of the grain boundaries.

V. Conclusions

Microstructural changes during plastic deformation and annealing were investigated in work-hardened mechanically milled iron powders, and then the work-hardening mechanism of iron during super-heavy plastic deformation was discussed. The findings of this study are as follows:

(1) Through the mechanical milling of 360 ks, nanocrystalline grains of 20–50 nm were formed within the powder, and the resultant powder was work-hardened to Hv950.

(2) Hardness of the mechanically milled iron powders scarcely depends on the amount of local strain but mainly on the crystallite size; the size of dislocation cells, subgrains and nanocrystalline grains.

(3) Local strain is thought to arise from two strained fields; One is around dislocations, especially geometrically-necessary dislocations in the cell walls

and/or the subgrain boundaries within crystalline grains. The other is around the grain boundaries which contain a large amount defects; lattice distortion and geometrically-necessary dislocations to accommodate the misfit between or among the adjacent grains. When all of grains are refined to an ultimate grain size (about 20 nm or less for iron), which never allow the existence of dislocations within grains, local strain might arise only from the latter.

(4) Work-hardening of iron follows the strain hardening mechanism in the hardness range up to Hv500, but above Hv600 it mainly follows the grain refining strengthening mechanism; the Hall-Petch relationship.

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