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Limit of Dislocation Density and Ultra-Grain-Refining on Severe Deformation in Iron

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Abstract

It is well-known that severe deformation to metals causes a direct grain refinement of the matrix without special heat-treatments due to the mechanism of dynamic continuous recrystallization (DCR). However, the microstructural revolution during severe deformation is seemed to be different depending on the deformation mode, namely the direction of deformation. In general, multi-directional deformation is thought to be effective for the grain refinement caused through DCR. For instance, ultra-grain-refinement to 10nm has already achieved by mechanical milling treatment in a steel powder and hardness of mechanically milled steel powder is increased to around HV12GPa by such a marked grain refinement. On the other hand, hardness of iron never exceeds HV4GPa by the mode of uni-directional deformation such a conventional cold rolling. In this paper, a limit of dislocation density is discussed for iron which is severely strained by the mode of uni-directional deformation, and also the importance of multi-directional deformation on DCR will be mentioned in association with a significant work hardening behavior in mechanically milled iron powder.

Keywords: Iron, Grain refinement, Uni-directional deformation, Multi-directional deformation, Stored strain, Dislocation density, Grain size, Dynamic continuous recrystallization

Introduction

Grain refining is most important technology in practical metallic and grain refinement to around 1μm has already achieved in a steel sheet which was produced in a national project; SUPER METAL [1]. However, further grain refinement below 1μm is probably difficult in steel sheets which are produced by rolling process. To achieve ultra grain refinement to the nano-size level, the other processes have be developed. Severe deformation of metallic is one of useful technique, because very fine crystal grains can be directly formed through the mechanism of DCR. So far, accumulative roll bonding; ARB [2], equal channel angular pressing; ECAP[3], mechanical milling; MM [4], ect. have been tried to obtain none sized grains in metallic materials. In particular, ECAP is suitable for the basic research to investigate the difference in deformation mode, because we can set the direction of shear deformation as we want. The results on ECAP [3] indicate an importance of multi-directional deformation as well as the importance of stored strain.

In the other words, grain refinement by DCR is not expected by the repetition of uni-directional ECAP even if the stored strain is large enough. Mechanical milling of metallic powder is most effective in terms of multi- directional deformation, because powder particles are repeatedly forged from all direction with hard steel ball. For instance, ultra-grain-refinement to 10nm has already achieved by mechanical milling treatment in a steel powder and hardness of the mechanically milled steel powder is increased to around HV12GPa by such a marked grain refinement [5]. On the other hand, hardness of iron should never exceed HV4GPa by uni-directional deformation such a conventional cold rolling. In this paper, a limit of dislocation density is discussed for iron which has been severely strained by the mode of uni-directional deformation, and also the importance of multi-directional deformation on DCR will be mentioned in association with a significant work hardening behavior in mechanically milled iron powder.

Deformation mode in ECAP and MM

In conventional cold rolling, decrease in thickness is inevitable to increase the amount of strain. This means that there is a limit of deformation in such a deformation mode. In order to make a severe deformation possible, the other deformation mode should be applied, which does not accompany the decrease in thickness of materials. ECAP and

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MM are unique processes which enable unlimited endless deformation to metallic materials. The outline of ECAP and MM is displayed in Figure 1. In the case of ECAP (a), a rod-like specimen is put in a mold with angulated hole and then pressed to arrowed direction. Through the deformation at the corner of hole, a lot of shear strain is charged into the material without any dimensional change of the specimen. True strain (ε) stored in the material is dependent on the angle (θ) of hole ($\varepsilon = 1$ for $\theta = \pi / 2$). When this pressing has been repeated at the same manner, a large shear strain can be stored in the mode of uni-directional deformation. However, when a specimen was put in the mold at the different rotated position for each time, the specimen undergoes multi-directional deformation because shear direction is different each time depending on the setting manner of the specimen. It has been reported in aluminum alloys that grain refinement through DCR is hard to occur in the mode of uni-directional deformation $^{3)}$. On the other hand, MM process can be applied only for powder materials. The principle of deformation is substantially a high speed forging by hard steel balls. In an early stage of MM treatment, metallic powder is simply deformed by the forging action and the shapeof powder is changed from block to flaky. In a middle stage of MM treatment, agglomeration of flaky powder proceeds due to the kneading action shown in (b) of Figure 1. The shape of powder becomes again blocky. In the final stage of MM treatment, blocky particles are deformed from all direction through the forging action by hard steel balls. The process from middle to final stage is correspondent to multi-directional deformation. It should be noted that, in MM treatment, a lot of fresh interface is introduced into powder particles through the kneading action just like ARB process.

Work hardening behavior of iron by uni-directional deformation

Cold rolling is simplest way to charge uni-deformation to metallic materials but it is sure that there is a limit of the amount of deformation. The deformation by cold rolling is likely limited to around 95% to keep sufficient thickness. Stored strain is about 3 in true strain for 95% reduction in thickness. However, ECAP enable further deformation. Azushima et al have reported mechanical properties of iron that has provided to ECAP treatment up to 10 times; (stored strain: about 10 in true strain $^{6)}$. Figure 2 shows the relation between 0.2% proof stress and stored strain in iron deformed by cold rolling or ECAP. Work hardening is significant in the early stage of deformation but becomes small in the latter stage (ε>3). A possibility of further work hardening is still remained but strengthening over 1.1GPa will not be so easy. The work hardening behavior can be followed by the equation 1.

$$\sigma_{0.2}[GPa] = 0.1 + 0.45 * \varepsilon^{0.3} \qquad (1)$$

Estimation of the limit of dislocation density in iron

It is well-known that work hardening is caused by an increase of dislocation and that the strength is represented by the following Bailey-Hirsch relationship as a function of dislocation density (ρ).

$$\sigma_{0.2}[GPa] = \sigma_0 + \alpha G b \sqrt{\rho} \qquad (2)$$

σ_0 denotes an original strength of materials. The character α, G and b is an unknown constant, shear modulus and Burgers vector of dislocation, respectively. Evaluation of dislocation density is not so easy but we can roughly evaluate it by means of X-ray diffraction analysis $^{7)}$. Figure 3 shows a relation between $\sqrt{\rho}$ and $\sigma_{0.2}$ in iron cold rolled up to 90% reduction in thickness and an ultra low-carbon martensitic steel. Clear Bailey-Hirsch relationship is found and strength is represented by the Eq. 3.

$$\sigma_{0.2}[GPa] = 0.1 + 1.04 * 10^{-8} \sqrt{\rho} \qquad (3)$$

As for iron and steels, G and b are known as 80GPa and 0.25nm, respectively, hence the comparison between the Eq. 2 and 3 gives the value of α as α=0.5.

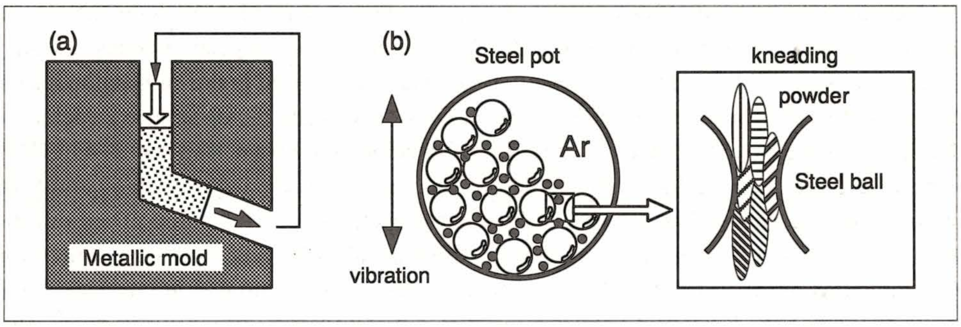

Fig. 1. Schematic illustration of Equal Channel Angular Pressing(ECAP) (a) and Mechanical Milling (b).

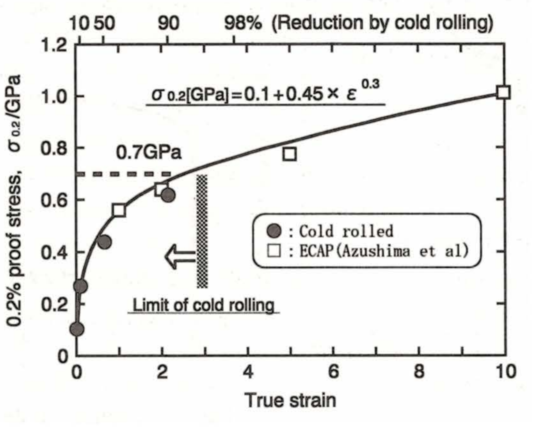

Fig. 2. Work hardening behavior of iron by cold rolling or equal channel angular pressing.

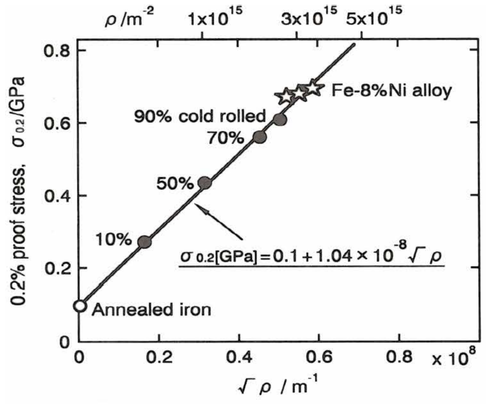

Fig. 3. Relation between dislocation density (ρ) and 0.2% yield stress in iron with cold rolling and ultra low carbon matensitic steel; Fe-8%Ni alloy.

On the other hand, everyone knows there is certain limit on dislocation density, however dose not known an exact limit. Combination of the Eq. 1 and 3 constructs the following Eq. 4 which shows a relation between ρ and ε.

$$\rho = 1.87 * 10^{15} \varepsilon^{0.6} \qquad (4)$$

Figure4 shows the change in ρ as a function of stored strain ε based on the above Eq. 4. It is very interesting that a lot of dislocations are introduced just in an early deformations stage and ρ exceeds 10^{15}/m^2 at the stored strain; ε=1. After that, an increase of dislocation tends to level off and the value of ρ seems not to go over 10^{16}/m^2. Thus, we may conclude that the limit of dislocation density is about 10^{16}/m^2 at the maximum and the expected strength should be 1.1 GPa in 0.2% proof stress, as far as the deformation is performed by a uni-directional deformation mode.

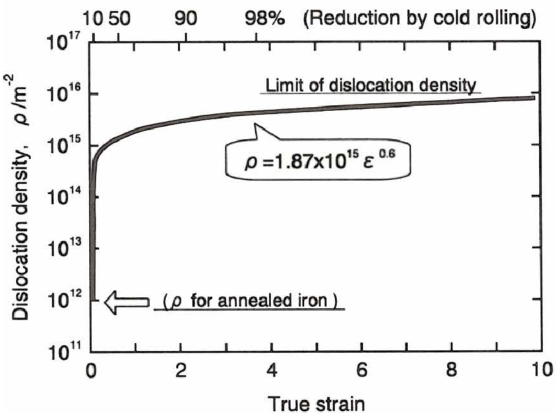

Fig. 4. Change in dislocation density as a function of true strain charged by one-directional deformation.

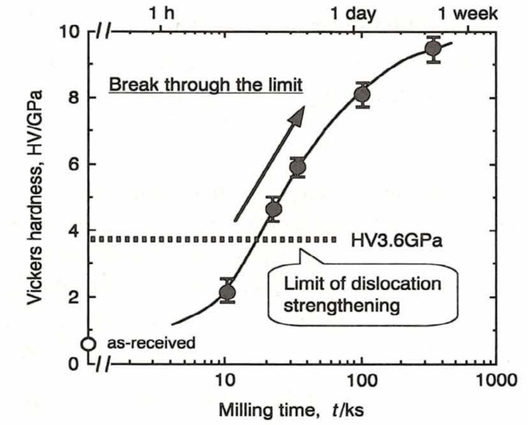

Fig. 5. Change in hardness of iron powder as a function of mechanical milling time.

Work hardening behavior in mechanically milled iron powder

Figure 5 displays a change in hardness of iron powder as a function of milling time. The hardness corresponding to $\sigma_{0.2}$=1.1GPa is HV3.6GPa (conversion relationship: HV=($\sigma_{0.2}$+0.1)*3[GPa]). It should be noted that iron powder can be greatly hardened by MM treatment and the hardness of MM iron powder easily goes over HV3.6GPa. After 360ks MM treatment, hardness of iron powder reaches about HV10GPa finally, that is almost same as the hardness of cementite (Fe_3C) 8). On the reason for such as a marked work hardening, we have to discuss from both points of the difference in deformation mode and stored strain. It is not easy to evaluate the strain stored within MM iron powder but we tried it by comparing the hardness of MM iron powder with cold rolled iron sheets. As a result, a mean strain rate of 0.2/ks was obtained for the MM machine used for this investigation. Figure 6 shows a change in expected stored strain ε as a function of milling time. This is a very rough evaluation, but it is sure that an enormous strain contributes to the abnormal hardening shown in Figure 5. In order to evaluate the effect of deformation mode, work hardening behavior has to be compared for both deformation mode of uni-directional and multi-directional deformation in the same Figure as a function of stored strain. Figure 7 displays the difference in work hardening behavior for MM treatment and uni-directional deformation. MM treatment is found to be more effective for increasing hardness. This result suggests an occurrence of another strengthening mechanism in MM treatment. Figure 8 shows the microstructure of MM iron powder with hardness HV4.7GPa. Bright field image (a) indicates the existence of high density of dislocation and also subgrains or dislocation cells. While, dark field image (b) clearly shows the existence of fine crystalline grain of the size 0.1~0.2 μm. This means that new small crystalline grain of formed within iron powder during MM treatment through the mechanism of DCR. Figure 9 shows the microstructure of MM iron powder with hardness HV9.5GPa. It is difficult to distinguish crystalline grains in the bright field image (a) but very fine grains of the size 10~30nm are obviously observed in the dark filed image (b). X-ray diffraction analysis has given the mean grain size as 25nm. Although the existence of declination structure is confirmed within some grains [9], it is sure that ultra grain refinement to such as a nano-size level results in marked hardening to around HV10GPa.

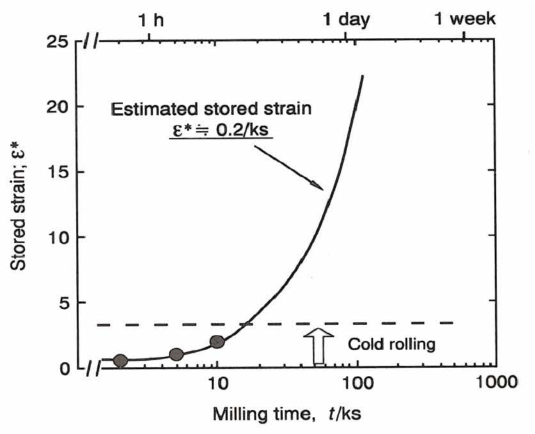

Fig. 6. Relation between mechanical milling time and strain which has been stored within mechanically milled iron powder. Stored strain was estimated by comparing the hardness of iron powder with cold rolled iron (ε<3).*

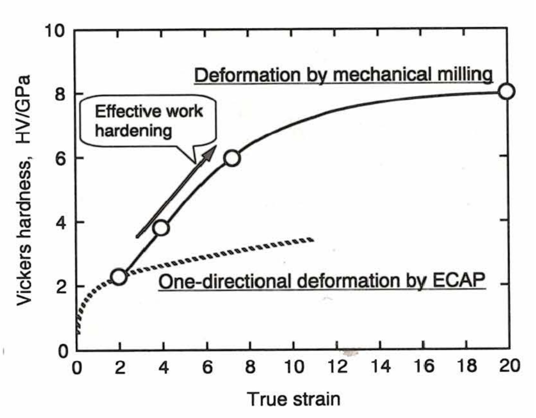

Fig. 7. Difference in work hardening behavior depending on deformation mode.

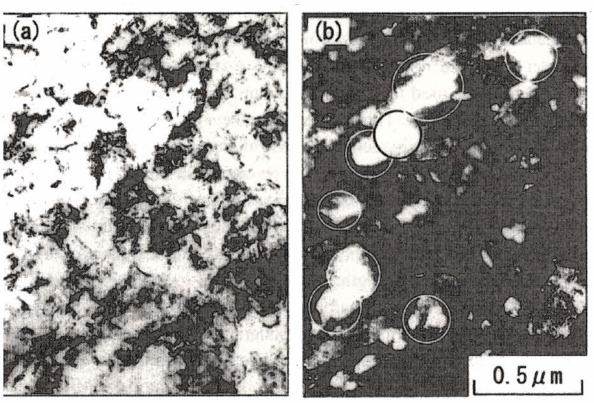

Fig. 8. TEM images showing the existence of fine crystalline grains which were formed within iron powder (HV4.7GPa) through mechanical milling treatment. Bright field (a), Dark field (b).

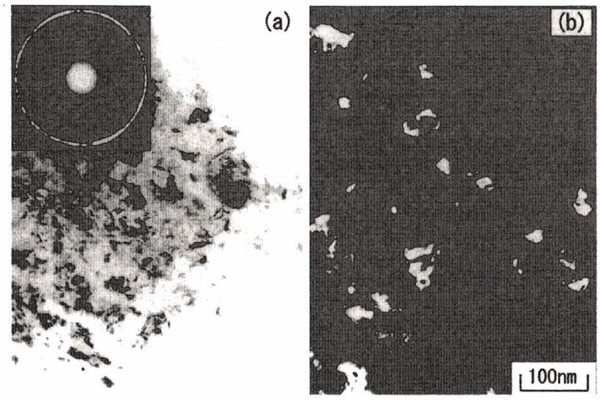

Fig. 9. Transmission electron micrographs of MM iron powder with hardness of HV9.5GPa. Bright field (a), Dark field (b).

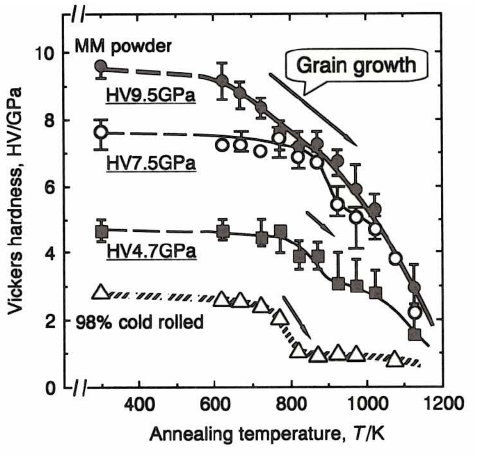

Fig. 10. Change in hardness of mechanically milled iron powder as a function of annealing temperature (annealing time: 30min).

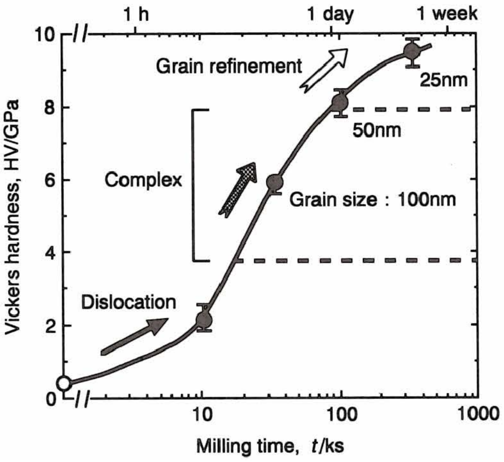

Fig. 11. Shift of strengthening mechanism in work hardening of mechanically milled iron.

Lattice defects such as dislocation and disclination might contribute to hardening somewhat. Annealing is useful for releasing lattice defects, so that only the contribution by grain refinement strengthening should be evaluated in annealed sampled without lattice defects. Figure 10 shows changes in hardness of MM iron powder as a function of annealing temperature. The result for 98% cold rolled iron sheet is also displayed in the Figure. Softening by incontinuous recrystallization, which appears at around 800K in the cold rolled sheet, is found at around 900K in MM iron powders. The retardation is due to finely dispersed iron-oxide (Fe_3O_4) particles which has precipitated on heating 10). Grain size of annealed MM iron powders can be kept fine even after incontinuous recrystallization due to the grain boundary pinning effect by oxide particles. In the case of MM iron powder of HV9.5GPa, clear incontinuous recrystallization is not observed any more. This means a great contribution of grain refinements strengthening in MM iron powders. In particular, the MM iron powder of HV9.5GPa is thought to be hardened mostly by the mechanism of grain refinement strengthening 11).

Finally, Figure 11 shows the shift of strengthening mechanism in the work hardening of MM iron powders. In the hardness level below HV3.6GPa, work hardening is caused by dislocation strengthening mechanism based on the Bailey-Hirsch relationship, while it is cause by grain refinement strengthening in the high hardness level above HV8GPa. In the middle hardness level, both of strengthening mechanisms contribute to work hardening.

Conclusion

1- In the case of uni-directional deformation of iron, the limit of dislocation density was estimated at around $10^{16}/m^2$ at the maximum and also achieved strength is evaluated at about 1.1 GPa in 0.2% proof stress and HV3.6GPa.

2- In mechanically milled iron powders, ultra grain refinement caused by dynamic continuous recrystallization was observed and it was found that the contribution of grain refinement strengthening is indispensable to realize a marked work hardening that exceeds the limit of dislocation strengthening.

3- On the appearance of dynamic continuous recrystallization, not only a large strain is required but also multi- directional deformation mode has to be applied.

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