Hall-Petch relationship of interstitial-free steel with a wide grain size range processed by asymmetric rolling and subsequent annealing

Bin Fu, Chenghao Pei, Hongbo Pan, Yanhui Guo, Liming Fu and Aidang Shan

1 School of Materials Science and Engineering, Shanghai Institute of Technology, 100 Haiquan Road, Shanghai 201418, People’s Republic of China
2 Key Laboratory of Metallurgical Emission Reduction & Resources Recycling of Ministry of Education, Anhui University of Technology, Ma’anshan 243002, People’s Republic of China
3 The Department of Chemical and Materials Engineering, University of Alberta, Edmonton T6G2H5, Canada
4 School of Materials Science and Engineering, Shanghai Jiao Tong University, 800 Dongchuan Road, Shanghai 200240, People’s Republic of China

E-mail: 20130007@ahut.edu.cn and gyh415@126.com

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Abstract

Asymmetric rolling (ASR) is an efficient processing for fabricating ultrafine-grained (UFG) materials. In the present investigation, interstitial-free (IF) steels with the grain size ranged from 500 nm to 500 μm were obtained by asymmetric rolling and subsequent annealing. The evolution of microstructures and mechanical properties of IF steel were studied. Accordingly, the Hall–Petch relationship of IF steel with a wide grain size range was established. It was found that ultimate tensile strength (UTS) corresponds well to the Hall–Petch relationship over the whole grain size range. However, the yield strength (YS) and hardness deviated from the Hall–Petch relationship as the grain size is larger than 100 μm, which is mainly attributed to the slight effect of grain boundary as obstacle on the dislocation movement and/or pile-up under small deformation in coarse grain (CG).

1. Introduction

IF steel is widely used in the field of automobile due to its excellent deep drawability and high ductility [1–3]. However, IF steel usually exhibits relatively low yield strength (YS) compared with modern high strength steel such as QP, TRIP and DP steel, which limits its further application. Many efforts of grain refinement such as severe plastic deformation (SPD) and microalloying element adding have been conducted to improve the strength of IF steel [4–12]. Severe plastic deformation (SPD) is thought to be the most prominent method of grain refinement in metals and alloys. However, conventional SPD methods such as equal channel angular pressure (ECAP) and high pressure torsion (HPT) are difficult to be applied in the industry because the specimen is too small and the processing is complicated. Recently, asymmetric rolling (ASR) in which the speed of two work rolls are different has been performed to obtain ultrafine-grained (UFG) materials with a good combination of strength and ductility [13–17]. And, it is thought to be suitable for mass production.

The strengthening mechanism of grain refinement is resulted from the resistance of grain boundary to dislocations slip. For polycrystalline materials, the grain boundary strengthening could be described by the Hall–Petch relationship [18, 19]. The flow stress or hardness is related to the average grain size by the equation:

\[ \sigma_f = \sigma_0 + k d^{-1/2} \text{ or } H = H_0 + k d^{-1/2} \]  

(1)

Where \( \sigma_f \) is flow stress, \( d \) is average grain diameter, \( H \) is hardness, and \( \sigma_0, H_0 \) and \( k \) are independent coefficients. \( d \) can not only represent the grain size, but also can be regard as the size of sub-grain and polygonal line in the sub-grain structure.

Hall–Petch relationship has been investigated in many works, such as Cu [20], Al-Mg alloy [21] and IF steel [4, 22–24], which indicate that the SPD-processed metals and alloys still correspond to the Hall-Petch
relationship. However, there are also some studies exhibiting inverse Hall-Petch relationships [25, 26] when the grain size in the nanometer scale. Grain size in previous investigations is almost in the range of 0.6 ~ 20 μm. However, less effort has been focused on the grain size larger than 100 μm to the knowledge of authors [27]. The effect of grain boundary on the strength is not clear when the grain size is in the order of micrometer. Additionally, the slope of the Hall-Petch relationship with different SPD methods is different, which reflect the different effect of grain refinement strengthening [23, 28, 29]. It is shown that the slope of Hall-Petch relationship of IF steel processed by HPT is larger than that processed by ECAP [4, 5]. In this study, IF steels with a wide grain size range were fabricated by ASR and subsequent annealing. Accordingly, the Hall-Petch relationship over a wide range of grain size was established and discussed.

2. Experimental

2.1. Materials preparation and processing

The materials used in this study was hot-rolled IF steel with the dimension of 100 mm (length) × 25 mm (width) × 20 mm (thickness), and its chemical composition is shown in Table 1. The hot-rolled IF steel was annealed at 900 °C for 1 h and 1000 °C for 12 h, followed by furnace cooling. The samples after annealing at 900 °C were then cold rolled to the final thickness of 1 mm with the total reduction of 95% by ASR through 35 passes. The rolling process was performed using two-high asymmetric rolling mill with the roller diameter of 180 mm in Shanghai Institute of Technology. The rotational speed of top and bottom roller was 33 rpm and 27.5 rpm, respectively, i.e., the speed ratio of ASR is 1.2. The ASRed plates were then annealed within the temperature range from 400 °C to 800 °C by the interval of 100 °C for 1 h followed by air cooling.

2.2. Microstructure

The samples for optical microscopy were etched in an electrolyte consisting of 4 ml HNO3 and 96 ml CH3COOH for about 40 s. The observed sections for optical microscopy were perpendicular to the transverse direction (TD) of the rolled samples.

Electron backscatter diffraction (EBSD) is performed to study the details of the microstructure evolution of IF steel samples on the cross section (RD-ND planes) using a high-resolution scanning electron microscope (SEM, FEI Quanta 200 FEG) equipped with backscattered electrons at 10 kV. The EBSD inspection area of samples annealed below 1000 °C was 40 × 40 μm, and the others were 150 × 150 μm. The step sizes are 100 nm and 250 nm, respectively.

The grain sizes of the samples under different annealing conditions were determined by Channel 5 and Image J software, respectively. For samples annealed below 700 °C, the average grain size was measured using Channel 5, while the others were measured using Image J software. Area method was used to calculate the grain sizes. Assuming that each grain is a regular hexagon. Then take double the side length of the regular hexagon as the average grain size. At least 500 grains were used for statistics. The recrystallization fraction of the samples was also determined by Channel 5.

2.3. Mechanical properties

Hardness was measured on the ND-RD, ND-RD, RD-TD planes of each sample by Buehler Micromet Hardness Tester with a Vickers diamond pyramid indenter under 200 g load for 15 s. At least 10 points were measured on each plane to calculate the mean value of hardness.

The tensile testing samples were cut from samples in the longitudinal RD by electrical discharge machining. The gauge length and gauge width of the tensile specimen was 25 mm and 3 mm, respectively. The tensile tests at ambient temperature were carried out by Shimadzu AG-10 kN tensile testing machine at the strain rate of 0.8 × 10⁻³ s⁻¹. 0.2% proof stress was used as yield strength.

3. Results and discussion

3.1. Microstructures

3.1.1. OM

The optical micrographs of IF steel processed by different conditions are shown in Figure 1. It is found that the dark lines almost aligned in one direction which are interpreted as deformation bands [30] in Figure 1(a), indicating that the IF steel has subjected to considerable severe plastic deformation during cold rolling. After annealing at 400 °C, the microstructure in Figure 1(b) did not change significantly and the deformed microstructure still dominated. As the annealing temperature increased to 500 °C, it is shown in Figure 1(c) that the dark areas of metallographic corrosion are significantly increased which indicates that the nucleation points of recrystallized grains gradually increased. Moreover, the dark area is further enlarged after annealing at 600 °C
shown in figure 1(d), i.e., the new grains nucleated around severely deformed structure increasingly. The structure is fully filled with recrystallized grains after annealing at 700 °C in figure 1(e). When annealing temperature increases to 800 °C, the grains begin to grow further (figure 1(f)).
In order to obtain large grains of IF steel, furnace cooling was used to reduce the position of ferrite nucleation. Figures 1(g) and 1(h) show the optical micrographs of hot-rolled IF steel obtained by annealing at 900 °C for 1 h and 1000 °C for 12 h followed by furnace cooling, respectively. Obviously, it can be found that larger grains can be obtained at higher temperatures. However, there are still some relatively small grains existing around the large grains because some ferrites nucleated too late to grow during the transformation of austenite to ferrite.

3.1.2. EBSD
Figure 2 shows grain boundary character distribution maps obtained from EBSD. The low-angle grain boundaries (LAGBs, $2^\circ < \theta < 15^\circ$) and high-angle grain boundaries (HAGBs, $\theta \geq 15^\circ$) were signified by the red and black lines, respectively.

It is found in figure 2(a) that the microstructure of the sample is mainly composed of slender grains and sub-grains after ASR. Obviously, some grains are broken and some grains are elongated during the rolling process. Also, the microstructure is dominated by LABs. There was no obvious change in microstructure, and the proportion of LABs still dominated after annealing at 400 °C (figure 2(b)). Further, the grains grow slightly and LABs decreases after annealing at 500 °C (figure 2(c)). It is shown that LABs in some grains has orientations close to the $\alpha$-fiber, while the $\alpha$-fiber continuously changes to the $\gamma$-fiber with increasing annealing temperature [15, 16]. As the annealing temperature increased to 600 °C, the microstructure is characterized by the elongated grains and recrystallized fine grains shown in figure 2(d). The formation mechanism may be due to different texture components after cold rolling, which have different storage energies, and therefore recover and recrystallize at different rates [4, 31]. Furthermore, it can be clearly seen from figure 2(e) that the recrystallization completed at 700 °C with large grain size. As the annealing temperature increases to 800 °C, the grains grow into coarser grains (figure 2(f)).

3.1.3. Grain size distribution
Grain size distribution determined by software (Channel 5 and Image J) are presented in figures 3(a)–(f).

The distribution of as-rolled IF steel exhibits rather narrow range of grain sizes (figure 3(a)). It demonstrates that the microstructure mainly consists of refined grains less than 1 $\mu$m. It is obvious that UFG IF steel was obtained by ASR. As the annealing temperature increases, the grain size distribution gradually becomes broader because the microstructure of the grains has begun to recover and recrystallize. The broadening of the grain size distribution occurs firstly at recovery stage of 400 °C (figure 3(b)), but the grain size below 1 $\mu$m still dominated. The grain size distribution gradually approaches to the right and the proportion of grain size larger than 1 $\mu$m gradually increases due to the sub-grain growth and partially recrystallization during the annealing at 500 °C and
It could be found in figure 3(c) that recrystallization completed after annealing at 700 °C and the highest fraction of the grain size distribution is around 8 ~ 15 μm. The recrystallized grains grew further at 800 °C and the proportion of grain sizes below 10 μm is significantly reduced (figure 3(f)). It can be found that the grain size above 100 μm accounts for nearly half after annealing at 900 °C (figure 3(g)). After annealing at 1000 °C, most of the grain sizes were more than 100 μm, i.e., micron-sized grains dominated (figure 3(h)). The average grain size ($\bar{d}$) as the function of annealing temperature is plotted in figure 4. The dependence of the recrystallization fraction on the annealing temperature can be well described as S shape.
3.2. Mechanical properties

3.2.1. Stress-strain curves

The stress–strain curves of IF steel after rolling and subsequent annealing are shown in Figure 5. Detailed tensile properties information of samples with different conditions are given in Table 2.

There was a great enhancement in strength after ASR. The YS and UTS of IF steel after ASR were 698 and 773 MPa, respectively. The YS was approximately 7 times of the CG IF steel at 900 °C for 1 h. Similar improvement of strength has been reported in IF steel after ARB, in which UTS reached 660 MPa [6]. It was proposed [32] that the strength of IF steel can be estimated by combination of contributions from dislocation strengthening and grain refinement strengthening. The additional shear strain induced by ASR could increase the degree of grain fragmentation, thereby improving the efficiency of grain refinement [7, 17]. In addition, a large amount of dislocations (work hardening) generated during ASR, which could also improve the strength of IF steel.

The YS drop slightly after annealing at 400 °C and 500 °C. The recovery resulted in the annihilation and rearrangement of deformation defect and there were mainly dislocation polygonization and subgrain coalescence. It can be seen that a gradual decrease in strength but a substantial increase in ductility after annealing at 600 °C. The change in mechanical properties was mainly attributed to partial recrystallization. The further reduction of YS was due to the increasing volume fraction of recrystallized grains after annealing at 700 °C ~ 800 °C. Figure 6 represents the evolution of YS and elongation with the variation of annealing temperature. There was an inverse relationship between the elongation and YS. The sample after annealed at
600 °C for 1 h have a strength of 422 MPa and reasonable elongation of 13.8%. Additionally, compared with full annealing state, the strength of the sample annealed at 700 °C for 1 h increased by more than 100 MPa while maintaining an almost constant elongation of 45%.

### 3.2.2. Hardness

The hardness of IF steel measured on various planes of different specimens is shown in Table 3. The hardness on different plane of the specimen presented a little difference which can be expressed as $HV_{(RD-TD)} < HV_{(ND-RD)} < HV_{(TD-ND)}$. It means that the mechanical properties of IF steel in this study exhibited some anisotropy which might be attributed to the different crystallographic textures in rolled IF steel [30]. In addition, compared with the fully annealed state, the hardness of IF steel increased by more than 3 times after ASR.

#### Table 2. Tensile properties of the IF steel samples under different conditions.

| Condition        | YS (MPa) | UTS (MPa) | Elongation (%) |
|------------------|----------|-----------|----------------|
| As-rolled        | 698.5 ± 6.8 | 773.2 ± 5.4 | 6.5 ± 0.2 |
| 400 °C/1 h       | 598.5 ± 10.5 | 684.8 ± 3.6 | 7.9 ± 0.5 |
| 500 °C/1 h       | 523.8 ± 4.6 | 605.7 ± 6.3 | 8.2 ± 0.4 |
| 600 °C/1 h       | 422.8 ± 5.6 | 493.7 ± 5.8 | 13.8 ± 0.3 |
| 700 °C/1 h       | 114.3 ± 4.1 | 348.1 ± 4.6 | 44.6 ± 0.5 |
| 800 °C/1 h       | 107.9 ± 3.5 | 306.8 ± 3.7 | 46.4 ± 1.2 |
| 900 °C/1 h       | 94.6 ± 5.9  | 230.5 ± 5.4 | 45.5 ± 0.8 |
| 1000 °C/12 h     | 101.2 ± 3.4 | 225.8 ± 4.6 | 48.3 ± 1.5 |

#### Table 3. Hardness of IF steel samples on various planes under different conditions.

| Condition       | Hardness                     |
|-----------------|------------------------------|
|                 | RD-TD plane | ND-RD plane | TD-ND plane | Average value |
| As-rolled       | 197.6 ± 2.1 | 210.9 ± 5.0 | 217.6 ± 4.9 | 208.7 ± 4.0  |
| 400 °C/1 h      | 187.7 ± 3.7 | 192.3 ± 6.7 | 201.7 ± 2.2 | 193.8 ± 4.2  |
| 500 °C/1 h      | 165.4 ± 7.0 | 172.1 ± 4.7 | 184.2 ± 5.8 | 173.8 ± 5.8  |
| 600 °C/1 h      | 135.8 ± 4.1 | 141.6 ± 3.4 | 161.7 ± 4.4 | 146.4 ± 3.9  |
| 700 °C/1 h      | 65.6 ± 4.3  | 69.7 ± 1.9  | 84.6 ± 2.2  | 73.3 ± 2.8   |
| 800 °C/1 h      | 61.5 ± 1.3  | 64.3 ± 2.3  | 77.2 ± 4.0  | 67.6 ± 2.5   |
| 900 °C/1 h      | 60.5 ± 2.1  | 62.9 ± 1.4  | 64.2 ± 1.6  | 62.5 ± 1.7   |
| 1000 °C/12 h    | 60.2 ± 3.2  | 63.4 ± 0.9  | 62.8 ± 1.9  | 62.1 ± 2.0   |
Figure 7 shows the variation of hardness of IF steel with different annealing temperatures. The result shows that the hardness decreases slightly as the annealing temperature increased from 400 °C to 600 °C. However, it decreases significantly after annealing at 700 °C, which is mainly attributed to the fully recrystallization. Eventually, the hardness begins to stabilize which is similar to the change of YS. Correspondingly, the hardness on various planes of IF steel annealed at different temperatures basically conforms to the same rule.

3.3. Hall-Petch relationship
Hall-Petch relationship was proposed firstly as the relationship between yield stress and grain size. Then, it was applied to the ultimate tensile strength which also corresponds to the Hall-Petch relation very well in many researches [32, 33]. In figure 8, the Hall-Petch relationship is based on the data fitting from YS, UTS, hardness and average grain size.

Generally, UTS conforms to the Hall-Petch relationship over the whole range of grain size. Meanwhile, both YS and hardness are linearly related when the grain size is in the range of 0.5–100 μm, which is also consistent with the Hall-Petch relationship.

The strength of IF steel should consider the effect of dislocation strengthening beside the grain boundary strengthening, especially to the severely deformed materials, which could be described as,

$$\sigma_f = \sigma_0 + \sigma_{diss} + \sigma_L$$  

(2)
where $\sigma_{\text{dis}}$ is the strengthening contribution from dislocation, $\sigma_{g}$ is the strengthening contribution from grain boundary, which could be described as Hall-Petch relationship (equation (1)).

The strengthening due to dislocations is also proportional to the inverse square root of grain size after the polycrystals were severely deformed and grain refined by the SPD, which could be drawn as [28],

$$
\sigma_{\text{dis}} = \alpha G b^\frac{1}{2} A \delta^\frac{1}{2}
$$

(3)

Where $\alpha$ is a constant, $G$ is the shear modulus, $b$ is the Burgers vector, $A$ is expected to be a stable value.

Therefore, it is obviously shown that both the $\sigma_{\text{dis}}$ and $\sigma_{g}$ are proportional to the inverse square root of grain size, and thus UTS and YS also correspond to the Hall-Petch relationship.

UTS is dependent on the work hardening behavior and the value of UTS-YS is indicative of work-hardening behavior which is also related to the grain size [34, 35]. It is shown that the slope of UTS (392 MPa \cdot \mu m^{0.5}) is lower than that of YS (488 MPa \cdot \mu m^{0.5}) in figure 8(b). Furthermore, the value of UTS-YS at fine grain size is lower than that at coarse grain size, which indicates better work hardening behavior was obtained at coarse grain size. The UFG metallic materials processed by SPD usually show bad ductility and low work hardening ability [10, 34].

However, it is noted that both YS and hardness almost remain constant regardless of grain size when the grain size is larger than 100 $\mu$m, which deviated from the linear Hall-Petch relationship.

3.3.1. Difference of Hall-Petch relationship between YS and UTS in coarse grains

As mentioned above, both the YS and hardness deviated from the Hall-Petch relationship over the grain size larger than 100 $\mu$m (figure 8). It is suggested that the effect of grain boundary on the dislocation movement might be different under different degree of plastic deformation.

Plastic deformation is mainly carried out by the movement of dislocations within the individual grains. Meanwhile, dislocations can move through the crystal grains and can interact with each other. Grain boundaries often act as obstacles to hinder their transmission, leading to the dislocation pile-up near the grain boundary, and thus strengthening the materials. It could also described as Hall-Petch relationship based on the theory dislocation pile-up [36].

Figure 9 shows the schematic diagram of dislocation distribution in small grain (SG, $d < 100 \mu$m) and CG ($d > 100 \mu$m) under different level of plastic deformation, respectively. The CG in the present study was obtained by high temperature annealing, and thus the dislocation density in the grains is very low [32, 33].
YS is the stress under small deformation, as well as hardness. Although dislocations accumulate rapidly in the CG during the small plastic deformation, CG has enough space to accommodate the dislocation multiplication (figure 9(a)). Therefore, grain boundary has slight effect on dislocation multiplication and movement, which leads to the almost no change of YS with grain size larger than 100 μm. Conversely, due to the SG with insufficient space, the dislocation movement in a short distance would be hindered by grain boundaries to form dislocation pile-up (figure 9(b)). Thus, the change of YS with grain size of less than 100 μm conforms to the Hall-Petch relationship. Additionally, during the process of large plastic deformation, an amount of dislocations were accumulated in CG to maintain plastic deformation, which could not be accommodated anymore in the individual grains, resulting in the dislocation pile-up at the grain boundaries (figure 9(c)). That is, the grain boundary strengthening takes effect. Hence, the dislocations pile-up model described by the equation (1) became valid (figure 8(b)) and the UTS conforms to the Hall-Petch relationship [33]. Similarly, dislocations pile-up model was also applicable to the SG grain boundaries during large plastic deformation (figure 9(d)).

3.3.2. Comparison of the slope in Hall-Petch relationship with different processing

Figure 10 shows the comparison of Hall-Petch relationship obtained by different processing. In general, Hall-Petch relationship with different processing methods shows significant difference, especially the slope of Hall-Petch relationship. It is thought that the magnitude of the slope of Hall-Petch relationship reflects the strengthening effect of grain size and dislocation, which is directly related to the strengthening mechanism. The slope of Hall-Petch relationship of ASR-processed IF steel obtained in present work is 460. Similar slope (k = 488) as well as yield strength were obtained by Yong et al [17] because the same ASR processing was used. Furthermore, the Hall-Petch relationship of ECAP-processed IF steel is basically coincided with the present study [22]. It has been reported that many factors such as texture, misorientation and processing conditions [29, 37] may also affect the slope of Hall-Petch relation, resulting in the slight difference of slope between ECAP and ASR.

However, the slope (k = 530) processed by HPT [4] is a litter larger than the present study, which is also the largest value among these processing methods shown in figure 10. It was found that IF steels with different grain size were processed by HPT without any annealing treatment, which results in a large amount of dislocations accumulated in the samples. Both the grain refinement strengthening and dislocation strengthening contribute to the large magnitude of slope, while the dislocation density of the annealed samples after ASR in the present study were relatively low.

Interestingly, ARB-processed samples with different grain size show relatively smaller value of slope compared with other processing methods, although the yield stress maintains in a relatively high level. The sheet were cut into two pieces, and then stacked together during ARB. In order to obtain the well-bonded bulk materials, it usually needed to be heated before roll-bonding, which would inevitably introduce metal oxide layer in the sample [38]. It is suggested that the metal oxide layers play a dominant role in strengthening [5, 36], thus resulting in the relatively high value of yield strength compared with other processing when the grain size smaller than 1 μm. However, the slope of ARB is only 138, which is significantly lower than other processing.
The smaller grain size was obtained by the increasing of cycles, and correspondingly the metal oxide layer also increases, which substantially reduce the effect of grain size on the yield strength due to the limit of ductility [39].

4. Conclusion

In the present study, IF steels with the grain size ranged from 500 nm to 500 μm were obtained by ASR and subsequent annealing. The evolution of microstructure and mechanical properties of IF steel was studied. And the Hall-Petch relationship over the wide range of grain size was established. The following conclusions can be drawn:

(1) Ultrafine-grained IF steel was obtained due to the additional shear strain induced by ASR, resulting in the significant increase of hardness and strength. High ultimate tensile strength of 773.2 MPa was achieved after ASR, which is mainly attributed to the ultrafine grains and dislocation multiplication. Comprehensive mechanical properties with different grain size could be controlled through subsequent annealing process.

(2) The UTS corresponds to Hall-Petch relationship over the whole range of grain size. However, Hall-Petch relationship of YS and hardness strongly depend on grain size. It still conforms to Hall-Petch relationship very well as the grain size is smaller than 100 μm, while deviates when the grain size is larger than 100 μm. This phenomenon is mainly due to the different effect of grain boundary on the dislocation movement and/or pile-up in grain with different size level when subjected to small and large plastic deformation.

(3) The characteristics of Hall-Petch relationship mainly include two aspects of strength level and slope, which are directly related to the strengthening mechanism, and therefore are influenced significantly by the processing method. The Hall–Petch relationship of IF steel with different grain size processed by severe plastic deformation (ECAP or ASR) and subsequent annealing is similar because the grain boundary strengthening is the main strengthening mechanism. However, the slope as well as strength of the HPT-processed IF steel without annealing treatment were increased due to the combined contribution of grain boundary strengthening and dislocation strengthening. In addition, relatively high strength and low slope of ARB-processed IF steel is mainly attributed to metal oxide layer generated during the heating and/or hot rolling process.

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Conflicts of interest

The authors declare no conflict of interest.

ORCID iDs

Bin Fu https://orcid.org/0000-0001-9675-6328
Chenghao Pei https://orcid.org/0000-0002-8230-8433
Liming Fu https://orcid.org/0000-0002-2340-2710

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