Effect of dislocation and grain boundary on deformation mechanism in ultrafine-grained interstitial-free steel

K Nakazawa1,3, S Itoh1, T Matsunaga2, Y Matsukawa2, Y Satoh1, Y Murase3, H Abe2
1School of Engineering, Tohoku University, Miyagi, Japan
2Institute for Materials Research, Tohoku University, Miyagi, Japan
3National Institute for Materials Science, Ibaraki, Japan
E-mail: NAKAZAWA.Kasane@nims.go.jp

Abstract. Ultrafine-grained interstitial-free steel fabricated by the accumulative roll-bonding method was subjected to tensile tests and analyses of AFM, TEM and XRD to identify the effects of interaction between dislocations and grain boundaries (GB) on the deformation mechanism. The AFM analyses indicated that the main deformation mechanism of this material changed from dislocation motion to grain boundary sliding (GBS) with decreasing strain rate. TEM observations and XRD analysis revealed that dislocations piled up at GB and the dislocation density decreased with increasing strain. Those suggest the dislocations are absorbed into GB during deformation, activating slip-induced GBS.

1. Introduction

Ultrafine-grained (UFG) metals with grain size of <1 µm have been developed and have higher mechanical strength than coarse-grained (CG) ones. In addition, it has been reported that the properties of UFG metals are affected by grain boundaries (GB) because the volume fraction of it increases with decreasing grain size and GB introduced by severe plastic deformation (SPD) processes are non-equilibrium [1]. In fact, ductility and fracture strain are often reduced in UFG metals, severely limiting their application. To extend the use of these metals, the deformation mechanism must be understood.

Two kinds of deformation model have been proposed to predict the deformation mechanism in UFG metals. Kato et al. suggested the model that dislocations bow out from GB at a strain rate of >10^{-4} s^{-1} [2]. Blum et al. proposed the model that GB store dislocations at a strain rate of <10^{-3} s^{-1} [3]. Recently, the authors reported that grain boundary sliding (GBS) was activated at a strain rate of <10^{-3} s^{-1} in UFG interstitial-free (IF) steel [4], but the detailed mechanism remains unknown.

In this paper, UFG IF steel fabricated by the accumulative roll-bonding (ARB) method was subjected to tensile tests and several analyses to clarify the deformation mechanism in UFG IF steel compared with CG IF steel in terms of the interaction between dislocations and GB at room temperature.

2. Materials and methods

Ti-added ultra-low carbon IF steel (0.002C-0.003O-0.01S-0.002N-0.02P-0.05Cr-0.12 Mn-0.03Ti) with grain size of 16 µm was used for the 5-cycle ARB process at 823 K to fabricate a UFG sample with typical grain thickness of 0.4 µm. Tensile specimens with the gauge length of 6 mm were produced with the loading direction corresponding to the rolling direction. Then, these specimens were annealed at 723 K for 0.5 h. On the other hand, as-received steel was used as the CG sample which was annealed at 903 K for 1 h. The gauge length for the specimens was 10 mm. Tensile tests were conducted at strain rates of 10^{-5}–10^{-2} s^{-1} at room temperature, during which strain was measured using strain gauges mounted on the surface.

After the tensile tests, atomic force microscope (AFM) analyses were conducted to calculate the contribution of GBS to the deformation using the following equation [5]:

\[ \text{GB contribution} = \frac{1}{6 \lambda_{GB}} \int F_{GB} \, dA \]

where \( \lambda_{GB} \) is the GB thickness and \( F_{GB} \) is the force on the GB.
\[ \varepsilon_{gb} = f v / d_l \]  
where \( \varepsilon_{gb} \) is the strain generated by GBS, \( v \) is the height of surface steps measured by AFM, \( f \) is the geometrical factor of 1.1, and \( d_l \) is the grain length.

Next, X-ray diffraction (XRD) analyses were performed to examine the change of dislocation density (\( \rho \)) using the Williamson-Hall plot (equation (2)) [6] and equation (3) [7]:

\[ \beta \cos \theta / \lambda = (2 \varepsilon \sin \theta / \lambda) + (0.9 / D) \]  
\[ \rho = (14.4 \varepsilon^2) / b^2 \]

where \( D \) is the average crystallite size, \( \lambda \) is the wavelength of 0.154 nm (CuK\( \alpha \)), \( \beta \) is the full width at half maximum of diffraction peak, \( \varepsilon \) is the strain in the sample, \( \theta \) is Bragg’s angle, and \( b \) is the Burgers vector of 0.248 nm.

In addition, transmission electron microscope (TEM) observations were performed at a voltage of 200 kV using a LaB\(_6\) gun to observe the micro structure in CG and UFG specimens after the tensile tests up to \( \sigma_{0.2} \) at a strain rate of \( 10^{-5} \) s\(^{-1}\).

### 3. Results

Figure 1 in AFM measurements shows surface height profiles for CG and UFG [4] specimens deformed up to \( \varepsilon = 0.002 \) at the strain rate of \( 10^{-5} \) s\(^{-1}\). The solid line shows a surface profile on the dashed line. In the CG specimen, intra-granular deformation is significant because slip bands, showing arrows in the figure, appeared slightly. However, GBS is predominant in the UFG specimen because the grain’s shape can be clearly observed. Then, AFM analysis also shows the contribution of GBS to plastic strain (\( \varepsilon_{gb}/\varepsilon_{pl} \)) as a function of strain rate for each specimen in Fig. 2. Although the ratio of \( \varepsilon_{gb}/\varepsilon_{pl} \) in the CG specimen was a few percent and less dependent on strain rate, the ratio in UFG specimen decreased drastically from 0.76 to 0.12 as the strain rate increased from \( 10^{-5} \) to \( 10^{-2} \) s\(^{-1}\). Fig. 2 also shows the strain rate dependence of the work-hardening exponent (\( n \)). The \( n \) value in CG specimen did not change almost with increasing strain rate, whereas, the value in UFG specimen decreased. These results indicate that the deformation mechanism in UFG specimen changes by strain rate and the contribution of GBS is enhanced at lower strain rate.

![Fig. 1. AFM image in (a) CG and (b) UFG specimens deformed up to \( \varepsilon = 0.002 \) at the strain rate of \( 10^{-5} \) s\(^{-1}\). The solid line shows a profile of surface roughness on the dashed line.](image)
Fig. 2. Strain rate dependence of the ratio of $\frac{\varepsilon_{gb}}{\varepsilon_{pl}}$ at $\varepsilon = 0.002$ and the work-hardening exponent in CG and UFG specimens.

Fig. 3. Change in dislocation density during plastic deformation of (a) CG and (b) UFG specimens [8].

Fig. 4. TEM micrographs in (a) CG and (b) UFG specimens deformed up to $\varepsilon = 0.002$ at the strain rate of $10^{-5}$ s$^{-1}$.

Figure 3 shows the relation of dislocation density as a function of $\varepsilon_{pl}$ obtained by XRD analyses for (a) CG specimen tested at the strain rate of $10^{-5}$ s$^{-1}$ and (b) UFG specimens tested at $10^{-5}$ s$^{-1}$ and $10^{-2}$ s$^{-1}$. The dislocation density increased after the tensile test in the CG specimen, whereas that in the UFG specimens decreased in test at the strain rate of $10^{-5}$ s$^{-1}$ and was almost stable at the strain rate of $10^{-2}$ s$^{-1}$.

TEM observations confirmed the XRD analyses because the CG specimen shows tangled dislocations and a cell structure within grains, whereas the UFG specimen contains...
dislocations piled up mainly at GB at $\varepsilon = 0.002$ (Fig. 4).

4. Discussion

It has been reported that the GB introduced by SPD processes have higher GB energy than those in CG metals and can absorb piled-up dislocations [9], and that this dislocation absorption activates “slip-induced GBS” with only 15 kJ/mol even at room temperature [10].

In the present study, the deformation mechanism in UFG IF steel was researched by comparing to CG specimens. TEM observation showed that dislocations piled up at GB without the cell structure in UFG specimens. Decrease in dislocation density and increase in the contribution of GBS to plastic strain after deformation at lower strain rate indicates that slip-induced GBS can be activated by dislocation absorption into GB in UFG IF steel.

Additionally, it is considered that the amount of GBS is sensitive to strain rate as shown in Fig. 2. At high strain rates, the dislocation adsorption rate is slower than its multiplication rate and dislocations pile up at GB, resulting in less GBS. On the other hand, at low strain rates, the amount of dislocation absorption increases because there is sufficient time to absorb the piled-up dislocations. The balance between multiplication and absorption of dislocations is the key to the deformation mechanism.

5. Conclusion

Tensile tests followed by AFM, TEM and XRD analyses were performed at room temperature to get insights into the deformation mechanism of UFG IF steel, and the following conclusions were obtained:

(1) Dislocations pile up and are absorbed at GB efficiently in UFG specimen because of relatively high GB energy. This mechanism is attributable to slip-induced GBS.

(2) The generation of GBS to the plastic deformation is influenced by strain rate.

Acknowledgments

The authors sincerely appreciate technical support of AFM observations from Prof. Yutaka Watanabe (Tohoku University) and funding support from 21th ISIJ Research Promotion Grant (incl. Ishihara/Asada Grant) of the Iron and Steel Institute of Japan.

References

[1] Zhu Y and Langdon T 2005 Influence of grain size on deformation mechanisms: an extension to nanocrystalline materials Mater. Sci. Eng. A, 409 p 234
[2] Kato M, Fujii T, Onaka S 2008 Dislocation bow-out model for yield stress of ultra-fine grained materials Mater. Trans. 49 p 1278
[3] Blum W and Zeng X 2009 A simple dislocation model of deformation resistance of ultrafine-grained materials explaining Hall-Petch strengthening and enhanced strain rate sensitivity Acta Mater. 57 p 1966
[4] Matsunaga T, Itoh S, Satoh Y, Abe H 2013 Effect of strain rate on deformation mechanism for ultrafine-grained interstitial-free steel Mater. Sci. Eng. A 576 p 267
[5] Kumar P, Xu C, Langdon T 2005 The significance of grain boundary sliding in the superplastic Zn-22% Al after processing by ECAP Mater. Sci. Eng. A 410-411 p 447
[6] Williamson G and Hall W 1953 X-ray line broadening field aluminum and wolfram Acta Metal. 1 p 22
[7] Williamson G and Smallman R 1956 Dislocation density in some annealed and cold-worked metals from measurement on the X-ray debye-scherrer spectrum Phil. Mag. 1 p 34
[8] Itoh S et al, ISIJ Int. Submitted
[9] Mompiou F et al 2013 Inter- and intragranular plasticity mechanisms in ultrafine-grained Al thin films: an in situ TEM study Acta Mater. 61 p 266
[10] Koike J, Ohyama R, Kobayashi T, Suzuki M, Maruyama K 2003 Grain-boundary sliding in AZ31 magnesium alloys at room temperature to 523 K Mater. Trans. 44 p 445