Nanoindentation-Induced Deformation Behavior in the Vicinity of Single Grain Boundary of Interstitial-Free Steel

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Nanoindentation measurements are made on an interstitial-free steel to reveal the effect of single grain boundary on deformation behavior. Three different sites, “on grain boundary”, “near grain boundary” and “grain interior”, were probed to investigate the pop-in behavior on the initial loading curve and nanohardness. The typical pop-in load at the “grain interior” gives a maximum shear stress beneath the indenter as an order of ideal strength. The pop-in load at the “on grain boundary” is significantly smaller than that at the other sites, indicating that the grain boundary acts as an effective dislocation source with a lower applied shear stress. The nanohardness in the “grain interior” is about 20% lower than that at the other sites, suggesting that a single grain boundary has significant resistance to indentation-induced deformation.

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1. Introduction

Many studies regarding the grain boundary effect on the strength of a polycrystalline material have been performed because grain refinement is an effective strengthening factor for improving both strength and toughness. The grain boundary effect is usually explained in some conventional models such as the dislocation pile-up model\(^1\)\(^2\) and the dislocation density model.\(^3\)\(^4\) Experimental work is still necessary to understand the whole mechanism of the grain boundary effect especially on the deformation behavior in a small scale in the vicinity of a single grain boundary. Recent progress in nanoindentation technique makes it possible to probe the mechanical properties in a nano-scale. A previous study\(^5\) in niobium using nanoindentation technique showed a strain burst during indentation called the “pop-in” phenomenon when a plastic zone crossed a grain boundary, suggesting that a single grain boundary has a significant resistance to slip transfer. Another work\(^6\) in martensitic steel demonstrated visually that grain boundaries have some resistance to dislocation glide motion even for a low-angle grain boundary through nanoindentation with a transmission electron microscope. Additionally, the behavior of the dislocation-grain boundary interaction depends on the characteristics of the boundary. A quantitative evaluation on the grain boundary-related deformation behavior should be performed for further understanding. In the present study, we focus on a pop-in behavior on the loading curve at the initial stage of deformation at various sites such as “on grain boundary”, “near grain boundary” and “grain interior”, and consider the initiation of plastic deformation depending on various subsurface microstructures that are associated with dislocation nucleation/multiplication. Moreover, the nanohardness of the three sites is evaluated, and the relationship between the hardness and the efficiency of a single grain boundary is discussed.

2. Experimental

A Ti-added ultra-low carbon interstitial-free steel (hereafter called IF steel) was used. The chemical composition of the steel is shown in Table 1. An ingot was hot-rolled at 1213 K (940 °C) and then cooled in a furnace. The typical grain size of the sample is about a couple of hundred μm. All the specimen surfaces for nanoindentation testing were mechanically polished, and subsequently electropolished in a solution of 8% perchloric, 10% butylcellosolve, 60% ethanol, and 22% water at 273 K under a potential of 40 volts to remove the damaged layer. Nanoindentation experiments were carried out using a Hysitron, Inc. Triboindenter. A Berkovich indenter was employed, and the tip truncation was calibrated using a reference specimen of fused silica. Analyses for the tip calibration and the calculation of hardness were conducted using the Oliver and Pharr method.\(^7\) Probed sites and indent configurations on the specimen surfaces were confirmed before and after the indentation measurements with the scanning probe microscope (SPM) capabilities of a Triboindenter. Electron backscatter diffraction (EBSD) analysis was carried out to characterize the grain boundary using a Carl-Zeiss LEO-1550 Schottky Field Emission SEM fitted with a TexSEM Lab.

3. Results and Discussion

The indenter was probed on the “on grain boundary”, “near grain boundary” and “grain interior” on the specimen surface. Figure 1 shows the SPM images of the specimen surface after the indentation measurements were taken and shows the typical positions at the grain boundary. In Fig. 1(a), the indent mark is made on the grain boundary between grains 1 and 2. The EBSD analysis was used to obtain the characteristics of the grain boundary. The speci-

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**Table 1** Chemical composition of the Ti added ultra-low carbon interstitial free steel (mass%).

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<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>Ti</th>
<th>N</th>
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<td>0.0002</td>
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</table>
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Fig. 1 Scanning probe microscope images of the specimen surface of the interstitial-free steel after the indentation measurements. (a) the indent mark is made on the grain boundary, (b) the cross section profile along line A–A’, which is indicated on the top view image of (a), and (c) the indent mark is made near the grain boundary.

Fig. 2 Typical load-displacement curves for the “on grain boundary” and “grain interior”. Both curves have the obvious pop-in behavior.

All the nanoindentation data described in the present study were carried out on grains 1 and 2 as well as the grain boundary between them. Figure 2 shows typical load-displacement curves for the “on grain boundary” and “grain interior”. Both curves have the obvious pop-in behavior, and the pop-in load $P^c$ and the depth of excursion $\Delta h$ can be defined as indicated in the figure. $P^c$ and the corresponding $\Delta h$ are larger for the “grain interior” than that for the “on grain boundary”. To understand the pop-in behavior, it is important to reveal the deformation mode of the initial loading curve before the pop-in event. If the pop-in behavior is associated with a drastic dislocation nucleation or multiplication as suggested in previous studies,8–10) the initial loading curve should indicate pure elastic deformation. According to the Hertz contact theory,11) the relation between load $P$ and penetration depth $h$ is expressed as

$$h = \sqrt{\frac{9}{16E^*}} \frac{P^2}{E^*R_i^2 + E^*R_c^2},$$

where $R_i$ and $R_c$ are the curvatures of the indenter tip and specimen surface, respectively, and $E^*$ is the reduced modulus in the equation

$$\frac{1}{E^*} = \frac{1 - \nu_s^2}{E_s} + \frac{(1 - \nu_i^2)}{E_i},$$

where $E_s$ and $\nu_s$ are the Young’s modulus and the Poisson’s ratio for the specimen and $E_i$ and $\nu_i$ are the same parameters for the indenter. When $R_s$ is assumed to be infinity, eq. (1) can be reformulated as

$$P = \frac{4}{3}E^*R_i^2h^3.$$
210 GPa into eq. (3), which is the average reduced modulus obtained from the unloading curve analysis by the Oliver and Pharr method.\(^7\) \(R_i\) is roughly estimated to be 230 nm. The value of \(R_i\) is consistent with a following estimation from the calibration on the tip area function. The area function of the indenter was determined as

\[
A = 24.5h_c^2 + 5.33 \times 10^2 h_c, \tag{4}
\]

where \(A\) is a contact area and \(h_c\) is a contact depth. Since the area function for the ideal indenter geometry is \(A = 24.5h_c^2\), the difference in depth between the ideal indenter and the actual indenter, which is named as truncated depth \(h_t\), can be calculated as approximately 10 nm. Meanwhile, the truncated depth is also calculated to be 6.3 nm from the \(R_i\) using an equation\(^{12}\)

\[
h_t = \frac{R_i}{8} \cot^2 \alpha, \tag{5}
\]

where \(\alpha\) is the angle between the centered axis and the side face of the indenter, and is 65° for the Berkovich type. Additionally, the estimated \(R_i\) is almost the same with a typical value shown in the literatures.\(^{12,13}\) Therefore, the estimated \(R_i\) can be regarded as a reasonable value. On the other hand, the maximum shear stress \(\tau_{\text{max}}\) beneath the indenter is given as\(^{10}\)

\[
\tau_{\text{max}} = 0.18 \left(\frac{E^*}{R_i}\right)^{\frac{3}{2}} P^{\frac{1}{2}}. \tag{6}
\]

Using the values of \(E^* = 210\) GPa, \(R_i = 230\) nm, and \(P = 300\) \(\mu\)N as a typical \(P^c_{\text{interior}}\) for the “grain interior” shown in Fig. 2, \(\tau_{\text{max}}\) is calculated to be 11.3 GPa. Since the shear modulus \(\mu\) of ferrite is approximately 83 GPa, a value of 7.3 is obtained as the ratio of \(\mu/\tau_{\text{max}}\), meaning that \(\tau_{\text{max}}\) is an order of an ideal strength of the ferrite phase. In addition, the maximum shear stress \(\tau_{\text{max}}\) is located exactly on the indentation axis beneath the indenter and the distance \(z_0\) from the original specimen surface can be obtained with the formula\(^{10}\)

\[
z_0 = 0.47 \sqrt{\frac{3PR_i}{4E^*}}. \tag{7}
\]

Substituting the values \(E^*, R_i\) and \(P^c_{\text{interior}}, z_0\) becomes 29.5 nm, which definitely corresponds to the subsurface region. Therefore, the pop-in behavior of the IF steel at the grain interior described in this study is attributed to the nucleation of dislocations within the defect free zone just beneath the indenter. For the “on grain boundary”, we obtain \(z_0 = 23.4\) nm by using the \(P^c_{\text{on GB}} = 150\) \(\mu\)N that was estimated in Fig. 2. The \(z_0\) value is also deep enough not to be influenced by any irregular surface.

For further understanding of the pop-in behavior, \(P^c\) is plotted as a function of \(\Delta h\) in Fig. 4. The square marks represent the \(P^c\) corresponding to the “grain interior”, the circles to the “near grain boundary” and the cross marks to the “on grain boundary”. The solid and open marks among the square and circle marks correspond to grains 1 and 2 defined in Fig. 1, respectively. A previous paper\(^{14}\) showed that the shear stress \(\tau\) beneath an indenter and \(\Delta h\) can be expressed as

\[
\tau = \frac{2\mu}{a} \Delta h + \gamma, \tag{8}
\]

where \(a\) is the lateral length of an indent mark and \(\gamma\) is the elastic strain remaining in the body after the pop-in event. Combining eqs. (6) and (8) with the condition \(\tau = \tau_{\text{max}}\) yields

\[
P^c = \left(\frac{1}{0.18}\right)^3 \left(\frac{R}{E^*}\right)^2 \left(\frac{2\mu}{a} \Delta h + \mu \gamma\right)^3. \tag{9}
\]

Figure 4 includes the fitted curves of eq. (9) for the data of each grain. The dotted and broken lines correspond to the data of grains 1 and 2, respectively. The data fit the line well, and there is no significant difference in the range of data of \(P^c\) or \(\Delta h\) for the “near grain boundary” and “grain interior”.

![Fig. 3 The initial loading curves below the pop-in load \(P^c\) in Fig. 2 replotted in a relation of \(P\) vs. \(h^{3/2}\).](image1)

![Fig. 4 A plot of the pop-in load \(P^c\) versus excursion depth \(\Delta h\).](image2)
The difference in the fitted curve between grains 1 and 2 may be attributed to an effect of crystallographic orientation. The plots for the “on grain boundary” have a much smaller range than those of the other grains. This suggests that the grain boundary is a site of dislocation nucleation with a lower applied shear stress compared to the grain interior. However, the shear stress is still in an order of the ideal strength of the ferrite phase.

Figure 5 shows the plastic nanohardness obtained from the $P-h$ curves for the three sites. The nanohardness is slightly lower in the grain interior than that at the other sites, suggesting that the single grain boundary has significant resistance to plastic deformation when the plastic zone includes the grain boundary. However, there is no remarkable difference in the nanohardness between the “on grain boundary” and “near grain boundary”, while the pop-in load is higher for the “near grain boundary” than for the “on grain boundary”. This result indicates that the nanohardness under the condition of a significant volume of plastic deformation is only attributed to the factors such as dislocation-dislocation interaction and dislocation-grain boundary interaction, and is not associated with the initial stage of plastic deformation that is characterized by the pop-in behavior. In other words, the “on grain boundary” condition may still includes some distance between an initiation site of plastic deformation and a grain boundary; hence the interaction between dislocations and grain boundary is the same situation with the “near grain boundary” case. Additionally, the nanohardness of grain 1 is pretty much the same as that of grain 2 at the “near grain boundary” and “grain interior”. In contrast, there is a remarkable difference in the relation of $P^c$ vs $\Delta h$ for the pop-in behavior described in Fig. 4. In the initial stage of plastic deformation leading to the pop-in event, a few of the activated slip systems may be limited; hence the effect of crystallographic orientation is considerably large. On the other hand, when the plastic deformation progresses, many slip systems are activated and the effect of crystallographic orientation becomes much smaller.

4. Conclusions

The effect of a single grain boundary on indentation-induced deformation was investigated for interstitial-free steel through nanoindentation technique. Indentation measurements were performed precisely on and near a grain boundary as well as the grain interior. Then the load-displacement curves were analyzed to reveal the pop-in behavior corresponding to the initiation of plastic deformation. Additionally, the nanohardness of the three sites was evaluated, and the relationship between the hardness and efficiency of the single grain boundary was discussed. The following conclusions were drawn.

1) The initial loading curves below the pop-in load can be fitted to the Hertz contact theory in this study. The typical pop-in load at the “grain interior” produces the maximum shear stress beneath the indenter as an order of ideal strength $\mu/6$. Therefore, the initial loading curves correspond to pure elastic deformation, and the pop-in behavior is associated with dislocation nucleation.

2) The relation between the pop-in load $P_c$ and the excursion depth $\Delta h$ is approximated to have the form of a three-degree polynomial equation. The pop-in load at the “on grain boundary” is significantly smaller than that at the “near grain boundary” and “grain interior”, suggesting that the grain boundary is an effective dislocation source.

3) The nanohardness at the “grain interior” is about 20% lower than that at the other sites because the single grain boundary of the IF steel has significant resistance to dislocation glide motion and a high potential to dislocation nucleation and the multiplication accelerates the dislocation interaction.

REFERENCES