Investigation of the indentation size effect based on measurement of the geometrically necessary dislocation density by electron backscatter diffraction

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Abstract
In this study, we investigated the mechanism of the indentation size effect based on measurement of the geometrically necessary (GN) dislocation density. The GN dislocation density was measured around impressions by electron backscatter diffraction (EBSD). Indentation tests were performed for two types of single crystal Ni with different crystal orientations: (001) and (111). Difference between (111) and (001) orientation are small in the relationship of the hardness and the penetration depth. However, the deformation behavior and distribution of the GN dislocation density are different for the (001) and (111) orientations. For the (111) orientation, the GN dislocation density increases with decreasing hardness. However, the GN dislocation density for the (001) orientation increases with increasing hardness. The mechanism of the indentation size effect for the (001) orientation can be attributed to the increase of the GN dislocation density. In addition, we investigated the effect of the indenter shape on the indentation size effect. Indentation tests were performed with different apex angles. The hardness using an indenter with a large apex angle is smaller than that using an indenter with a small apex angle. The GN dislocation density increases with decreasing apex angle. The mechanism of the indentation size effect for the apex angle can be attributed to the increase of the GN dislocation density. We prepared another indenter with a dull tip. The hardness using the dull indenter is larger than that using the sharp indenter. The GN dislocation density distribution changes with the indenter sharpness but the GN dislocation density is similar. For the dull indenter, variation of the GN dislocation density has a smaller effect on the indentation size effect than the increase of the resistance because of the different indenter shape.

Keywords: Indentation tests, Indentation size effect, Geometrically necessary dislocation, Electron backscatter diffraction, Indenter shape

1. Introduction

Indentation tests are a convenient way to evaluate the mechanical properties of small materials. They are used to evaluate the strengths of thin films, the local mechanical properties of weld zones, and so forth. Many studies using the indentation method have been reported (Nakashima et al., 2016; Yonezu et al., 2006; Sakaue et al., 2012). The local stress–strain relationship can be evaluated using the multi-indentation method, which is performed by indentation with multiple apex angles (Ozeki et al., 2014; Komori et al., 2012). However, numerous indentation studies have reported an increase in the hardness with decreasing depth of penetration, which is known as the indentation size effect. The causes of the indentation size effect are considered to be the radius of the indenter tip, roughness of the specimen surface, and so forth (Kim et al., 2007; Swadener et al., 2002). Miyahara et al. (1998) proposed a method to determine the macrohardness from test results that include the indentation size effect. It is known that there is a large difference...
between macroscale and microscale strengths and phenomena. For example, a sudden displacement jump called “pop-in” occurs when the penetration depth is a few nanometers (Gerberich, 1996; Durst et al., 2008). Pop-in is interpreted in dislocation nucleation behavior. Then, stress which causes pop-in is corresponding to ideal strength. When the penetration depth is a few micrometers or sub-micrometer, it has been proposed that the indentation size effect mechanism is based on geometrically necessary (GN) dislocation (Ashby, 1970; Fleck et al., 1994). This concept is different from pop-in behavior. Then, Nix and Gao (1998) proposed a method to predict the indentation size effect by strain gradient theory. This method has been used in many studies of the indentation size effect (Qu et al., 2006; Huang et al., 2005).

As mentioned above, the indentation size effect at the microscale can be explained by strain gradient theory. However, almost all of the research on the indentation size effect based on strain gradient theory has been theoretical, and there has been no experimental research of measurement of the GN dislocation density. Electron backscatter diffraction (EBSD) is a microstructure observation technique that has rapidly progressed in recent years (Schwartz et al., 2009; Kamaya et al., 2005; Wilkinson and Britton, 2012; Yamagita et al., 2010; Hasunuma et al., 2015; Sakakibara et al., 2010; Nomura et al., 2012; Fujiyama et al., 2012; Wright et al., 2011). The GN dislocation density can be easily measured by EBSD (Calcagnotto et al., 2010; Moussa et al., 2015; Wright et al., 2015). Numerous studies have measured the GN dislocation density of indentation (Kysar et al., 2007; Demir et al., 2009; Rester et al., 2007). Demir et al. (2009) performed indentation tests of a Ni(111) single crystal and measured GN dislocation. Their results showed that the GN dislocation density increases with decreasing penetration depth. They suggested that the indentation size effect cannot be explained by strain gradient theory and the GN dislocation density. However, further investigation is required because their indentation tests were performed with only one type of indenter and only the (111) crystal orientation. In particular, indentation tests need to be performed for other crystal orientations that have a large influence on the dislocation movement. In addition, indentation tests should be performed for different apex angles and with indenters with different tip shapes because the distribution of the GN dislocation density around the impression varies. The relationship between the GN dislocation density and the hardness should then be investigated in detail.

In this study, we investigated the mechanism of the indentation size effect based on measurement of the GN dislocation density. First, indentation tests were performed for two Ni single crystals with different crystal orientations. The GN dislocation density was then measured around the impression by EBSD. Thus, the effect of the crystal orientation on the relationship between the GN dislocation density and the hardness was determined. Second, to change the distribution of the GN dislocation density, indentation tests were performed with various apex angles and indenter tip shapes. The relationship between the hardness and the GN dislocation density was then investigated in detail. Finally, the experimental results were compared with strain gradient theory to investigate the mechanism of the indentation size effect.

2. Effect of the crystal orientation on the hardness and GN dislocation distribution

2.1 Measurement of the hardness

First, the effect of the crystal orientation on the hardness and GN dislocation distribution was investigated. A Ni single crystal was used for the indentation tests. The crystal structure of Ni is face centered cubic (FCC). Indentation tests were performed for two Ni single crystals with different crystal orientations: (001) and (111). The specimen surfaces were polished with colloidal silica (particle size 0.04 μm) after diamond suspension polishing (abrasive grain sizes 9, 3, and 1 μm). The indentation tests were performed with a DUH-211 Dynamic Ultra Micro Hardness Tester (Shimadzu, Kyoto, Japan) using a triangular pyramid indenter with an apex angle of φ = 115°. The relationship between the indenter direction and the crystal orientation is shown in Fig. 1. The hardness was evaluated by the JIS Z 2255 ultralow loaded hardness (HTL) (Japanese Industrial Standards Committee, 2003):

![Fig. 1 Coordinate and crystal orientations for the hardness tests.](image-url)
\[ HTL = 0.102 \times \frac{3 - \tan^2(\phi/2)}{9 \tan(\phi/2)} \times 10^5 \times \frac{F}{h_{\text{max}}^2} \]  

(1)

where \( F \) and \( h_{\text{max}} \) are the load and maximum penetration depth, respectively. The indentation test results are shown in Fig. 2. \( HTL \) increases with decreasing penetration depth \( h \), thus the indentation size effect occurs. Difference between (111) and (001) orientation are small in the relationship of \( HTL \) and \( h \). Therefore, the test results reveal that the crystal orientation does not affect the indentation size effect, although the reason is not clear. One possible reason is because the FCC crystal has many slip systems.

### 2.2 Measurement of the GN dislocation density

The GN dislocation densities of the impressions described in Section 2.1 were measured by EBSD. The EBSD measurements were performed with an ERA-600FE or ERA-8900FE field emission scanning electron microscope (Elionix, Kanagawa, Japan) equipped with an EDAX-Digi View orientation image microscopy (OIM) detector (Elionix). The OIM data were obtained by TSL OIM Data Collection 5.1 software. The step size was 0.067–0.25 \( h_{\text{max}} \). The specimen surfaces were repeatedly polished to measure the crystal orientation at the point whose distance from the deepest point of the impression is \( h \). The surfaces were polished with colloidal silica (particle size 0.04 \( \mu \)m) after diamond suspension polishing (abrasive grain sizes 9, 3, and 1 \( \mu \)m). There are many researches which investigate 3D micro structure using cyclic polishing (Yamagishi et al., 2010). It is noted that colloidal silica polishing is mechanochemical polishing so we can remove surface with no machined surface layer.

On slip system \( n \), the GN dislocation density \( \rho_{\text{GN}}^{(n)} \) can be expressed as (Fleck et al., 1994; Ohashi, 2002)

\[ \rho_{\text{GN}}^{(n)} = \sqrt{\left(\rho_{\text{GN,screw}}^{(n)}\right)^2 + \left(\rho_{\text{GN,edge}}^{(n)}\right)^2} \]  

(2)

\[ \rho_{\text{GN,screw}}^{(n)} = \frac{1}{b} \frac{\partial y^{(n)}}{\partial \zeta} \]  

(3)

\[ \rho_{\text{GN,edge}}^{(n)} = \frac{1}{b} \frac{\partial y^{(n)}}{\partial \zeta} \]  

(4)

where \( \rho_{\text{GN,screw}}^{(n)} \) and \( \rho_{\text{GN,edge}}^{(n)} \) are the screw and edge components of the GN dislocations on the \( n \)th slip system, respectively. \( \zeta \) and \( \xi \) are the directions perpendicular and parallel to the slip direction on the slip plane, respectively. The Burgers vector \( b = 0.256 \) nm (Koslowski et al., 2011). \( y^{(n)} \) is the plastic shear strain on the \( n \)th slip system. From the EBSD data, the GN dislocation density was calculated using the dislocation density tensor proposed by Nye (1953), the local misorientation angle, and so forth (Motz et al., 2005; Moussa et al., 2015). In this study, the local misorientation was used because it is easy to calculate. Using the local misorientation, the GN dislocation density \( \rho_{\text{GN}} \)
can be expressed as (Moussa et al., 2015)

\[ P_{\alpha} = \frac{\alpha \theta}{\partial b} \]  

(5)

where \( \alpha, d, \) and \( \omega \) are a constant, the step size, and the local misorientation, respectively. The kernel average misorientation (KAM) is generally used to measure the local misorientation (Moussa et al., 2015). The KAM is the average misorientation angle of a given point with all of its neighbors (Sakakibara et al., 2010; Wright et al., 2011):

\[ KAM = \frac{\sum_{i=1}^{n} \alpha_i}{n_b} \]  

(6)

where \( \alpha_i \) and \( n_b \) are the misorientation angle between its neighbors and the number of neighboring measurement points, respectively. However, the KAM is sensitive to the size step (Moussa et al., 2015). In addition, the effect of noise on the GN dislocation density increases with decreasing the step size. Therefore, \( \theta \) is defined as the misorientation between reference orientation and individual orientation, when reference orientation in this study is orientation before experiment, i.e. (001) or (111). Then, \( \theta \) was used to calculate the GN dislocation density:

\[ P_{\alpha} = \frac{1}{b} \sqrt{\left( \frac{\partial \theta}{\partial x} \right)^2 + \left( \frac{\partial \theta}{\partial y} \right)^2 + \left( \frac{\partial \theta}{\partial z} \right)^2} \]  

(7)

The definitions of the \( x, y, \) and \( z \) axes are shown in Fig. 1. The method to calculate \( \partial \theta/\partial x \) is as follows. First, the crystal orientation is measured by EBSD, and \( \theta \) is determined. \( \theta \) is then approximated during five steps by a quadratic equation in the \( x \) axis direction using the least squares method. \( \partial \theta/\partial x \) is calculated by differentiating that approximation with respect to \( x \). This method for approximating \( \theta \) from multistep data reduces the noise and step size dependence. \( \partial \theta/\partial y \) is calculated using the same method as that used for \( \partial \theta/\partial x \). \( \partial \theta/\partial z \) can be calculated by repeatedly polishing and measuring \( \theta \). However, it is very difficult to measure \( \partial \theta/\partial z \) with high accuracy and space resolution because the number of polishings becomes large. Therefore, \( \partial \theta/\partial z \) was ignored and \( \rho_{\alpha N} \) was calculated from \( \partial \theta/\partial x \) and \( \partial \theta/\partial y \):

\[ P_{\alpha} = \frac{1}{b} \sqrt{\left( \frac{\partial \theta}{\partial x} \right)^2 + \left( \frac{\partial \theta}{\partial y} \right)^2} \]  

(8)

The experimental results were then qualitatively compared. In Section 2.4, we will show that \( \partial \theta/\partial z \) has no effect on \( \rho_{\alpha N} \) qualitatively.

### 2.3 Results of the GN dislocation measurements

Figures 3 and 4 show the distributions of \( \theta \) for the (001) and (111) orientations, respectively. Figures of inside in Figs. 3 and 4 were observed after repeatedly polished. As it is impossible to determine the impression on EBSD image, image quality, which is EBSD parameter that indicates quality of the Kikuchi pattern, was used, because image quality was decreased in impression (Calcagnotto et al., 2010). The impression is defined as the area whose image quality is lower than 1100. For the (001) orientation, misorientation occurs on the surface and inside the material. Plastic deformation occurs around impression so misorientation also occurs. The area where misorientation occurs extends from the center to the side of the impressions. In particular, it extends in the [T T 0] and [1 T 0] directions, which are the slip directions of FCC materials. Inside the material, \( \theta \) is large under the impression. In the case of the (001) orientation, all slip systems are active. Therefore, misorientation occurs on the surface and inside the material. In contrast, for the (111) orientation, \( \theta \) is large under the impression but there is no misorientation at the surface. This is thought to be because the indent direction corresponds to the normal direction for slip place and slip systems in (111) are not active. Figure 5 shows height distribution of Fig. 4(a) along A-A’ shown in Fig. 1. This height distribution was observed by atomic force microscopy. There is pile-up but this pile up is very small. Therefore, the effect of pile up on EBSD observation is thought to be small.

Figure 6 shows the GN dislocation density map of the (001) orientations. There is a high GN dislocation density area near the edge of the impression. There are GN dislocation at the surface and inside the materials because all slip
The maximum dislocation density calculated with the least squares method is shown by the solid line for the (001) orientation with $\phi = 115^\circ$. The area where misorientation occurs extends from the center to the side of the impression. Figure 7 shows the distributions of the GN dislocation density along the lines A′–A′ and B–B′ shown in Fig. 1. These data have large scatterings. The solid lines are cubic curves approximated by the least squares method. It should be noted that an approximate line calculated without the data is greatly different from the plots. The results of indentation tests for the (001) orientation with $h_{\text{max}} = 1\,\mu m$ are shown in Fig. 7. The GN dislocation density increases toward the center of the impression. In Fig. 7(a), the relationship between $\rho_{\text{GN}}$ and $y/h_{\text{max}}$ for the surface is similar to that inside the material. However, the impression inside the material is smaller than that at the surface, as shown in Fig. 6. Therefore, the experimental data inside the material are closer to $y/h_{\text{max}} = 0$ than those of the surface. Consequently, the maximum...
This trend corresponds to the experimental results. The solid lines are cubic curves approximated by the least squares method. The relationships between the GN dislocation density and $y/h_{\text{max}}$ at the surface and inside the material are similar.

Fig. 7 GN dislocation density distributions for the (001) orientation along (a) A–A’ and (b) B–B’ at the surface and inside the material. The points are the experimental results. The solid lines are cubic curves approximated by the least squares method. The relationships between the GN dislocation density and $y/h_{\text{max}}$ at the surface and inside the material are similar.

Fig. 8 GN dislocation density distributions along A–A’ for the (001) orientation with different $h_{\text{max}}$ values. The GN dislocation density for small $h_{\text{max}}$ is higher than for large $h_{\text{max}}$.

Fig. 9 GN dislocation density map for the (111) orientation with $\phi = 115^\circ$. There is an area of high GN dislocation density inside the material but not at the surface.

The value of the GN dislocation density for the surface is larger than that inside the material. In Fig. 7(a) and (b), the GN dislocation densities along A–A’ and B–B’ are quantitatively different but the trends are the same. The relationship between $y/h_{\text{max}}$ and $\rho_{\text{GN}}$ for the surface is similar to that inside the material, thus only the results at the surface will be discussed below. Figure 8 shows a comparison of the GN dislocation distributions for different $h_{\text{max}}$ values. The GN dislocation density for small $h_{\text{max}}$ is higher than that for large $h_{\text{max}}$. This trend corresponds to strain gradient theory.

Figure 9 shows the GN dislocation density distribution for the (111) orientation. Comparisons of the GN dislocation densities shown in Fig. 9 along A–A’ of Fig. 1 for the surface and inside the material are shown in Figs. 10 ($h_{\text{max}} = 0.5 \mu m$) and 11 ($h_{\text{max}} = 3 \mu m$). The solid lines are linear approximations of the data. There is no area of high GN dislocation density at the surface, but there is an area of high GN dislocation density inside the material under the impression. This is because slip systems in (111) are not active. In particular, the maximum GN dislocation density occurs at the side of the impression. In addition, the GN dislocation density for small $h_{\text{max}}$ is lower than that for large $h_{\text{max}}$. This trend contradicts from strain gradient theory.
Effect of $h_2$.

In addition, the orientation excludes the number of polishing with increasing $h_2$. Therefore, the GN dislocation density was calculated using Eq. (8), which excludes $\partial \theta \zeta$. The effect of $\partial \theta \zeta$ on the GN dislocation density was investigated. Figure 12 shows the GN dislocation density calculated by Eq. (7), which includes $\partial \theta \zeta$. The method for determining $\partial \theta \zeta$ is as follows. First, the crystal orientation $\theta$ is measured at the point whose distance from the deepest point of impression is $h_t$ by repeated polishing. Next, the difference between $\theta$ at $h_t$ and at the surface is calculated at each measurement point. Finally, these values are divided by $(h_{\text{max}} - h_t)$ to obtain $\partial \theta \zeta$. Comparing Figs. 6, 9 and 12, the GN dislocation density with $\partial \theta \zeta$ is higher than that without $\partial \theta \zeta$. However, the shapes of the high GN dislocation density areas are the same with and without $\partial \theta \zeta$. In addition, with $\partial \theta \zeta$, the GN dislocation density decreases with increasing $h_{\text{max}}$ for the (001) orientation, and it increases with increasing $h_{\text{max}}$ for the (111) orientation. Therefore, $\partial \theta \zeta$ quantitatively affects the GN dislocation density but it has no effect on the qualitative trend.

2.4 Effect of $\partial \theta \zeta$ on the GN dislocation density

As mentioned in Section 2.2, it is very difficult to measure $\partial \theta \zeta$ with high accuracy and space resolution because the number of polishings becomes large. Therefore, the GN dislocation density was calculated using Eq. (8), which excludes $\partial \theta \zeta$. The method for determining $\partial \theta \zeta$ is as follows. First, the crystal orientation $\theta$ is measured at the point whose distance from the deepest point of impression is $h_t$ by repeated polishing. Next, the difference between $\theta$ at $h_t$ and at the surface is calculated at each measurement point. Finally, these values are divided by $(h_{\text{max}} - h_t)$ to obtain $\partial \theta \zeta$. Comparing Figs. 6, 9 and 12, the GN dislocation density with $\partial \theta \zeta$ is higher than that without $\partial \theta \zeta$. However, the shapes of the high GN dislocation density areas are the same with and without $\partial \theta \zeta$. In addition, with $\partial \theta \zeta$, the GN dislocation density decreases with increasing $h_{\text{max}}$ for the (001) orientation, and it increases with increasing $h_{\text{max}}$ for the (111) orientation. Therefore, $\partial \theta \zeta$ quantitatively affects the GN dislocation density but it has no effect on the qualitative trend.
3. Effect of the indenter shape on the distribution of the GN dislocation density

3.1 Effect of the apex angle on the GN dislocation density

Indentation tests were performed to investigate the effect of the indenter shape on the GN dislocation density for the (001) orientation, whose relationship between the GN dislocation density and $h_{\text{max}}$ corresponds to strain gradient theory. First, the effect of the apex angle on the GN dislocation density was investigated. Indentation tests were performed for the (001) orientation using indenters with $\phi = 100^\circ$, $115^\circ$, and $118^\circ$. The other conditions were the same as those specified in Section 2.1. It should be noted that the JIS Z 2255 ultralow loaded hardness is not applicable for indentation with $\phi = 100^\circ$ and $118^\circ$, but it was calculated by Eq. (1). Figure 13 shows the indentation results for various apex angles. The small apex angle indenter ($\phi = 100^\circ$) has a greater indentation size effect than the large apex angle indenter ($\phi = 118^\circ$).

EBSD of the impressions was then performed, and GN dislocation density was measured. The measurement conditions were the same as those specified in Section 2.2. The GN dislocation density was calculated using Eq. (8). It was determined at the surface because the relationship between $\rho_{\text{GN}}$ and $y/h_{\text{max}}$ at the surface is similar to that inside the material, as explained in Section 2.3. Figure 14 shows the distribution of $\theta$. The result with $\phi = 115^\circ$ is shown in Fig. 3. The area where misorientation occurs increases with increasing $\phi$. The GN dislocation distribution is shown in Fig. 15. The result with $\phi = 115^\circ$ is shown in Fig. 6. Figure 16 shows the GN dislocation density along A–A′. The solid lines are cubic curve approximations by the least squares method. The GN dislocation density increases with decreasing $\phi$.

![Graph showing the relationship between HTL and $h$](image)

**Fig.13** Effect of the apex angle on the indentation size effect. A small apex angle indenter has a greater indentation size effect than a large apex angle indenter.

![Graph showing the relationship between $F$ and $h$](image)

**Fig.14** Distribution of the crystal orientation at the surface for different apex angles ($h_{\text{max}} = 1 \mu$m). The area where misorientation occurs increases with increasing $\phi$.

![Graph showing the distribution of GN dislocation density](image)

**Fig.15** Distributions of the GN dislocation density at the surface for different apex angles ($h_{\text{max}} = 1 \mu$m). The GN dislocation density increases with decreasing $\phi$. 

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3.2 Effect of the indenter tip shape on the GN dislocation density

To investigate the effect of the indenter tip shape on the GN dislocation density, indentation tests were performed with a \( \phi = 100^\circ \) indenter that was worn and the tip was dull because the indenter had been used for indentation tests of a diamond-like carbon coating (Nakayama et al., 2009). The shape of the indenter tip was observed by atomic force microscopy by transferring the shape of the indenter tip to pure aluminum. Figure 17(a) shows the three-dimensional shape of a sharp indenter with \( \phi = 100^\circ \) and Fig. 17(b) shows the shape of the dull indenter. The C–C’ section is shown in Fig. 17(c). Comparing the sharp and dull indenters, the dull indenter is worn by about 0.2 \( \mu \)m. Figure 18 shows the indentation results using the dull indenter. HTL of dull indenter is higher than that of the sharp indenter.

![Figure 16 GN dislocation density distributions along A–A’ for different apex angles.](image)

![Figure 17 Three-dimensional shapes of the (a) sharp and (b) dull indenters, and (c) the height distribution along the C–C’ line.](image)

![Figure 18 Comparison of indentation test results between sharp and dull indenters. HTL of the dull indenter is higher than that of the sharp indenter.](image)
The GN dislocation density was then measured around the impression made by the dull indenter. Figure 19 shows the GN dislocation distribution around the impression made by the dull indenter, and Fig. 20 shows the GN dislocation distribution along A–A’. The solid lines are cubic curve approximations by the least squares method. The shape of the impression is not an equilateral triangle because the indenter tip is dull. Comparing Figs. 15, 19 and 20, the GN dislocation distributions of the sharp and dull indenters are different.

4. Mechanism of the indentation size effect

The relationship between the representative GN dislocation density, HTL and $h_{\text{max}}$ was investigated using the HTL data (Figs. 2, 13, and 18) and GN dislocation density data (Figs. 6, 9, 15, and 19). This relationship was compared with strain gradient theory. The calculation method for the representative GN dislocation density is as follows. For the (001) orientation, the average ($\rho_0$) and standard deviation ($\sigma_0$) of the GN dislocation density at the surface are determined before the indentation tests. The GN dislocation density is then measured after the indentation tests. The high GN dislocation density area is defined as the area whose GN dislocation density is larger than $\rho_0 + 2 \sigma_0$. Finally, the average GN dislocation density is calculated for the high GN dislocation density area, which is defined as the representative GN dislocation density. We used the values $\rho_0 = 0.154 \times 10^{16}$ m$^{-2}$ and $\sigma_0 = 0.0972 \times 10^{16}$ m$^{-2}$. For the (111) orientation, the representative GN dislocation density is defined as the average of the GN dislocation density under the impression. These calculation methods cannot be used for quantitative comparison, but they can be used for qualitative comparison.

The method for calculation of the GN dislocation density and HTL based on strain gradient theory is as follows. For a conical indenter, the equations for the relationships between the GN dislocation density and $h_{\text{max}}$, and HTL and the GN dislocation density are (Swadener et al., 2002)

$$\rho_{\text{GN}} = \frac{3\lambda^2}{2b} h_{\text{max}}^{-1}$$ (9)

$$\text{HTL} = \text{HTL}_{0}\sqrt{1+\frac{\bar{\rho}_{\text{GN}}}{\rho_{\text{SS}}}}$$ (10)

with

![Image](image_url)

Fig.19 GN dislocation density distribution around the impression made by the dull indenter ($\phi = 100^\circ$). The GN dislocation distributions for the sharp and dull indenters are different.
\[ \rho_{ss} = \left\{ \frac{HTL_0}{(3\sqrt{3}\alpha h)} \right\}^2 \]  

(11)

where \(HTL_0\), \(\vec{\tau}\), and \(\rho_s\) are the macroscopic \(HTL\), Nye factor, and statistically stored dislocation density, respectively. For a conical indenter (Fig. 21), the constant \(A = l/r\). For a FCC metal, the geometric constant \(\alpha\) is usually in the range 0.3–0.6. In this study, we used shear modulus \(\mu = 75\) GPa for Ni (Ardell et al., 1976). The GN dislocation density and \(HTL\) were calculated for indenters with \(\phi = 100^\circ, 115^\circ, \) and \(118^\circ\). Equations (9) and (10) do not apply to this study because a triangular pyramid indenter was used. Therefore, we used a conical indenter whose ratio of \(h_t\) to the contact area was the same as that of a triangular pyramid indenter. In other words, triangular pyramid indenters with \(\phi = 100^\circ, 115^\circ, \) and \(118^\circ\) correspond to conical indenters with cone angles of 52.67°, 70.30°, and 77.37°, and we used \(A = 1.31, 2.79,\) and 4.46, respectively. We used \(HTL_0 = 90\) based on the horizontal zone of \(HTL\) in Fig. 2. We used \(\vec{\tau} = 2\) and \(\alpha = 0.5\) (Swadener et al., 2002). It should be noted that strain gradient theory cannot be used for the dull indenter because the indenter tip shape is very complex. The calculated and experimental results were only qualitatively compared because the \(\rho_{GN}\) values of the calculations and experiments are different.

Figure 22 shows the relationships between the GN dislocation density and \(HTL\), and the GN dislocation density and \(h_{max}\). The points are the experimental data and dashed lines are the trends of the plots. The solid lines are the results calculated based on strain gradient theory. First, we will discuss the experimental results. For the indentation tests with the (001) orientation and the sharp indenter, the GN dislocation density increases with decreasing \(h_{max}\), and \(HTL\) increases with increasing GN dislocation density. For the same \(h_{max}\), \(\rho_{GN}\) of the indenter with a small apex angle is
higher than that of the indenter with a large apex angle. The relationship between the GN dislocation density and HTL does not depend on $h_{\text{max}}$ and the apex angle. These trends of the experimental results are also observed in results calculated by strain gradient theory. It should be noted that the experimental and calculated results are quantitatively different because definition of the GN dislocation densities of the calculations and experiments are different. In contrast, for the experimental results with the (111) orientation, the GN dislocation density decreases with decreasing $h_{\text{max}}$, and the GN dislocation density decreases with decreasing HTL. These trends of the experimental results are different from the results calculated by strain gradient theory. In this study, indentation tests were performed for the (111) orientation using indenter with only $\phi = 115^\circ$. However, for the (001) orientation, the relationship between the GN dislocation density and HTL does not depend on the apex angle. In addition, $\phi = 115^\circ$ is the median of the $\phi = 100, 115$ and $118^\circ$. Therefore, the experimental results with the (111) orientation using indenter with $\phi = 115^\circ$ is considered to show general trend for the (111) orientation. For the same GN dislocation density, HTL for the dull indenter is higher than that for the sharp indenter. For the same $h_{\text{max}}$, the GN dislocation density for the dull indenter is similar to that for the sharp indenter.

We will now discuss the mechanism of the indentation size effect based on the results of this study. Difference between (111) and (001) orientation are small in the relationship of HTL and $h$. However, there are large differences in the distributions of misorientation and GN dislocation densities for the (001) and (111) orientations. For the (001) orientation, the trend of the experimental results corresponds to strain gradient theory, as shown in Fig. 22. Therefore, for the (001) orientation, the reason for the indentation size effect is the increase of the GN dislocation density.

For the same GN dislocation density, HTL for the dull indenter is higher than that for the sharp indenter. For the same $h_{\text{max}}$, the GN dislocation density for the dull indenter is similar to that for the sharp indenter. These results indicate that the variation of the GN dislocation density has a smaller effect on the indentation size effect than the increase in the resistance owing to the different indenter shape.

The experimental results for the (111) orientation do not correspond to strain gradient theory. Therefore, the mechanism of the indentation size effect is not the increase of the GN dislocation density. For the (111) orientation, the mechanism of the indentation size effect is not clear. A possible mechanism of the indentation size effect is the distance between the indenter tip and the high GN dislocation density area. A schematic diagram of the mechanism of the indentation size effect for the (111) orientation is shown in Fig. 23. A high GN dislocation density area occurs under the impression edge. Therefore, the smaller the $h_{\text{max}}$ value, the closer the indenter tip and the high GN dislocation density area. The high GN dislocation density area prevents dislocation movement more when $h_{\text{max}}$ is small, and the indentation size effect occurs. However, this mechanism is hypothetical and further investigation is required.

Summarizing the above, indentation size effect is caused by GN dislocation. The effect of apex angle on indentation size effect is also explained by GN dislocation. However, indentation size effect is not only caused by GN dislocation density but also distribution of GN dislocation. Contribution of GN dislocation distribution remains unknown and requires more research. On the other hand, the increase of the hardness using the dull indenter is caused by the increase of the resistance owing to the different indenter shape.

5. Conclusion

We have investigated the mechanism of the indentation size effect based on measurement of the GN dislocation density. Indentation tests were performed for single crystal Ni under various conditions, and the indentation size effect was measured. The GN dislocation density was then measured by EBSD, and the relationship between the GN dislocation density and hardness was determined. The following aspects are clarified by the results.
(1) Difference between (111) and (001) orientation are small in the relationship of \textit{HTL} and \textit{h}. For the (001) orientation, there is high GN dislocation density near the impression. In contrast, for the (111) orientation, a high GN dislocation area occurs under the edge of the impression. The GN dislocation density increases with decreasing \textit{h}_{\text{max}} for the (001) orientation, whereas it decreases with decreasing \textit{h}_{\text{max}} for the (111) orientation.

(2) A small apex angle indenter has a larger indentation size effect than a large apex angle indenter. The GN dislocation density increases with decreasing apex angle. The relationship between the GN dislocation density and \textit{HTL} is not dependent on the apex angle. The indentation size effect of a worn dull indenter is larger than that of a sharp indenter. The GN dislocation density for the dull indenter is similar to that for the sharp indenter. Therefore, the increase of the hardness is caused by the increase of the resistance owing to the different indenter shape.

(3) For the (001) orientation, the trends of the relationships between the GN dislocation density, \textit{HTL}, and \textit{h}_{\text{max}} correspond to strain gradient theory. Therefore, for the (001) orientation, the reason for the indentation size effect is the increase of the GN dislocation density. In contrast, the trends for the (111) orientation do not correspond to strain gradient theory. Therefore, a mechanism other than the increase of the GN dislocation density is responsible for the indentation size effect for the (111) orientation.

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