EBSD- and ECCI-based Assessments of Inhomogeneous Plastic Strain Evolution Coupled with Digital Image Correlation

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We measured the local grain orientation gradient and dislocation density using electron backscatter diffraction (EBSD) measurement and electron channeling contrast imaging (ECCI) to obtain strain maps near a stress concentration source in pure nickel (Ni) as a face-centered cubic (FCC) model sample. In particular, we obtained the relationship amongst the grain orientation spread (GOS), dislocation density, and equivalent plastic strain on the sample surface, which were obtained by EBSD, ECCI, and digital image correlation (DIC), respectively. After obtaining the GOS-strain and dislocation density-strain relationships, the strain distribution in the interior of the sample was also determined by measuring the GOS and dislocation density, which were linearly correlated. The dislocation density-strain relationship exhibited a relatively small deviation.

KEY WORDS: electron backscatter diffraction; electron channeling contrast imaging; digital image correlation; grain orientation spread; dislocation density; plastic strain.

1. Introduction

The local plastic strain evolution plays an important role in the ductile fracture of structural metallic components such as steel. For instance, the failure of dual-phase steel1,2) and austenitic transformation-induced-plasticity steel3,4) are predominantly controlled by the deformation-induced micro-damage evolution associated with the plastic straining at the damage tip. Specifically, the final failure is caused by damage such as void and crack coalescence and growth. In particular, the heterogeneous plastic deformation occurs at the crack tip, owing to the stress concentration. Hence, the microstructural plastic strain and its gradient have been recognized as key factors in understanding the underlying mechanism of the ductile fracture and ductile crack growth phenomena. The plastic strain evolution depending on the crack opening/propagation is different between the surface and the interior of the material, owing to the difference of the stress field. To discuss the crack propagation, it is necessary to measure the plastic strain distribution of not only the surface, but also of the material’s interior.

In the numerical approach, the stress-strain relationship is analyzed using the finite element method (FEM). Moreover, the crystal plasticity FEM, which considers the effects of the crystal structure and interfacial structure, has also been developed.5–7) In the experimental approach, the digital image correlation (DIC) method using artificial fine particles or a microstructural pattern as a random pattern is used to measure the local strain.8–10) However, the DIC method is an image-based strain analysis conducted on the sample’s surface. Thus, it cannot be used to measure the strain distribution in the sample’s interior. For this problem in the DIC method, there are two solutions for carrying out plastic strain measurements: (1) three-dimensional tomography11) and (2) measuring the grain orientation gradient or dislocation density of a deformed material.12,13) In this study, we discuss the latter method.

First, we explain the plastic strain measurement by the grain orientation gradient of the deformed material. The electron backscatter diffraction (EBSD) method is known as the grain orientation mapping method. In particular, the Kernel average misorientation, (KAM), grain reference orientation deviation (GROD), and grain orientation spread (GOS) are obtained from the grain orientation by the EBSD measurement, which enables the estimation of the local plastic strain.14–16)

The KAM is a parameter representing the average misorientation with the surrounding pixels. The KAM is represented by Eq. (1), as follows:

\[ \text{KAM} = \frac{\sum_{i=1}^{6} \gamma_i}{6} \]

where \( \gamma_i \) represents the misorientations. The pixels of the EBSD measurement used in this study are hexagonal. Hence, the KAM is the average misorientation between an arbitrary hexagonal pixel and the surrounding six pixels. The KAM corresponds to the geometrically necessary (GN) dislocation density. Hence, the KAM has a quantitative correspondence with the plastic strain arising from the dislocation motion.17) However, when the plastic strain gradient
is monotonic, as shown in Fig. 1, the KAM calculated as the average misorientation from orientations of surrounding pixels corresponds to the slope of the plastic strain. Therefore, the KAM has a constant value despite the presence of a non-monotonic plastic strain gradient. In this case, the asymptote point of the KAM is the elasto-plastic boundary.

The GROD exhibits a transgranular deformation gradient on the basis of the average orientation for each grain, and is represented by Eq. (2), as follows:

\[ \text{GROD} = \theta_i - \theta_{\text{AVE}} \]  

where \( \theta_i \) is the orientation of \( i \)th pixel in a grain, and \( \theta_{\text{AVE}} \) is a reference value defined as the average orientation of each grain. The GROD qualitatively corresponds to the plastic strain distribution.\(^{18} \) The two-way arrows shown in Fig. 1 represent the GROD. By comparing (a) and (b) in Fig. 1, it can be seen that the GROD at (a) is larger than that at (b), which is consistent with the plastic strain gradient. However, compared with (c), point (b) has a larger plastic strain with a smaller GROD. This mismatch between the GROD and the plastic strain is attributed to the grain-character-dependent average orientation, which is used as the reference value. Because the reference value is not constant in each grain, the GROD cannot be quantitatively compared between the different grains. Therefore, the GROD is not suitable for quantitatively analyzing the plastic strain distribution of multiple grains. The GOS is defined by Eq. (3),\(^{16} \) as follows:

\[ \text{GOS} = \sum_{i=1}^{n} (\theta_i - \theta_{\text{AVE}}) / n \]  

where \( n \) is the number of pixels in a grain. Hence, GOS is the average GROD of each pixel in a grain. The GOS is constant in each grain; therefore, the spatial resolution is extremely lower than that of the KAM and GROD. However, the GOS enables the quantitative evaluation of local plastic strain for each grain, as shown in Fig. 1, and thus has good correlation with a degree of grain-scale deformation.

Secondly, the dislocation density measured by a transmission electron microscope (TEM) is another type of microstructure information and enables the estimation of the plastic strain.\(^{19,20} \) There exists an intrinsic relationship between the dislocation density and the plastic strain, which is represented by Eq. (4), as follows:

\[ \gamma = \rho bx \]  

where \( \gamma \) is the plastic shear strain, \( \rho \) is the dislocation density, \( b \) is the Burgers vector, and \( x \) is the average moving distance of dislocation. Therefore, the plastic strain distribution can be estimated from the dislocation density by measuring the relationship between the plastic strain and the dislocation density. However, if Eq. (4) is used for polycrystalline and microstructurally complex samples, we must specify the extraction position of the thin films in the bulk sample and conduct wide-range/multiple observations. Therefore, the plastic strain measurement using TEM requires a substantial amount of effort. Hence, instead of the TEM, we conducted electron channeling contrast imaging (ECCI) using scanning electron microscopy (SEM).\(^{21,22} \) The ECCI can visualize the dislocations of a bulk sample, which enables the measurement of the dislocation density in a bulk sample across a wide observation range. In this study, we present the results and a discussion with regard to the following two points.

1) The crystallographic orientation gradient and dislocation density around the stress concentration area were measured on the sample’s surface using EBSD/ECCI. The plastic strain measurement using EBSD/ECCI was demonstrated by combining the abovementioned measurements with DIC analysis.

2) In the sample’s interior, wherein the DIC method cannot be used, the EBSD/ECCI measurements were used to estimate the inhomogeneous plastic strain distribution around the stress concentration area.

2. Experiment

2.1. Material and Sample

The objective of this study was to develop a microstructure-based strain mapping technique to clarify the ductile fracture mechanism in steel. For the sake of simplicity, the present material was selected based on the following guidelines to exclude any factors that complicate the relationship between the dislocation density and the strain.

1) To avoid the occurrence of strong dislocation pinning and depinning, which alter the average moving distance of
dislocation, interstitials such as carbon and nitrogen must be removed.

2) In addition to the dislocation motion/multiplication, the twinning deformation and deformation-induced martensitic transformation also provide strain. Therefore, the extremely low stacking fault energy metals or metastable austenitic steels must be avoided.

3) It is currently uncertain how the strain-dislocation density relationship changes by the cross slip effects and the formation of dislocation aggregates such as cell. Therefore, body-centered cubic metals, which easily exhibit cross slip, should be avoided.

Thus, pure nickel (Ni; purity of 99.4%), which is a pure metal with a stable structure and the same crystal structure as austenite, was the material used in this study. The influence of microstructural and compositional complexities, which entail the abovementioned issues, will be investigated in future work. In the final heat treatment, a thin plate with a thickness of 1 mm was annealed at 800°C for 30 min and subsequently furnace-cooled. Then, two types of samples were prepared by an electric discharge machine (EDM).

Figure 2(a) shows the shape of the sample used in the tensile testing conducted to measure the mechanical properties. The gauge dimensions were 4 mm x 1 mm x 30 mm. Before the tensile test, the sample was ground by emery paper (#1000) to remove the stress concentration resulting from the surface scratches. The tensile test was conducted at 20°C with an initial strain rate of 1.0 x 10^{-4} s^{-1}. Figure 3 shows the engineering stress-strain curve. The 0.2% proof stress and ultimate tensile strength were 79 MPa and 401 MPa, respectively, which indicates the occurrence of significant strain hardening. Figure 2(b) shows the shape of the specimen used in the in situ SEM under tensile loading. The gauge dimensions were 2 mm x 1 mm x 20 mm. This sample (Fig. 2(b)) was ground and polished by emery paper (#1000) and diamond suspension (9 μm, 3 μm). Moreover, in this study, a notch with a groove width of 0.1 mm and groove length of 0.5 mm was introduced at the side of the sample using EDM to induce heterogeneous plastic strain. The stress concentration part near this notch was subjected to plastic strain distribution analysis. Finally, the sample surface was polished by colloidal silica to carry out EBSD/ECCI measurement.

### 2.2. Measurement of Strain Evolution Behavior

To measure the local strain near the notch using the DIC method, a random pattern was provided on the sample’s surface. In this study, colloidal silica with a particle size of 50 nm was used as the random pattern. The tensile test for characterizing the strain evolution was conducted at 20°C with an initial strain rate of 2.5 x 10^{-4} s^{-1}. This initial strain rate was estimated without considering the notch effect. The maximum load was 90 N. The sample surface was observed by in situ SEM using an in-lens detector for the secondary electron. The observation was conducted at 5 kV with a probe current of 10 pA and working distance of 15 mm. The images were used to carry out local strain measurement with VIC-2D (Correlated Solution, Inc.). The step size and subset size for the DIC analysis were set to 10 pixels and 31 x 31 pixels, respectively.

### 2.3. EBSD/ECCI Measurements of Sample Surface and Interior

To carry out an observation after the tensile test, the sample was polished by colloidal silica to remove the random pattern of the colloidal silica used for the DIC. The EBSD measurement system (OIM ver. 7.x) was used to analyze the crystal orientation near the notch on the surface of the sample. The observation was conducted at 20 kV with a probe current of 10 nA and working distance of 15 mm.
In the EBSD measurement, the step size was set to 0.2 \( \mu \text{m} \).

ECC imaging was conducted at 30 kV with a probe current of 10 nA and a working distance of 2.7 mm. Each grain near the notch on the sample surface was observed using ECCI, and the images were used to carry out the dislocation density measurement, as will be described later. The contrast in the ECC images depends greatly on the lattice distortion of a grain. Hence, near the grain boundaries, where the lattice distortion is larger than that in the grain interior, it is difficult to obtain clear dislocation images with the same sample tilt angle and contrast as in the center of the grain. In this study, the dislocation densities of multiple grains had to be measured by imaging the individual dislocation using a relatively simple procedure. Therefore, the image-based dislocation density measurement was conducted by observing around the center of each grain. The dislocation density was calculated by counting the number of interceptions between the dislocation lines and the meshes drawn in the ECC images, as follows:24)

\[
\rho = \frac{n_1/l_1 + n_2/l_2}{t} \quad \text{................. (5)}
\]

where \( l_1 \) and \( l_2 \) are the mesh lengths, \( n_1 \) and \( n_2 \) are the number of interceptions between the dislocation lines and the meshes, and \( t \) is the penetration depth of the electron beam. In this study, \( t \) was set to 90 nm.22) After the ECCI analysis, the DIC and EBSD/ECCI correlations were evaluated for the sample’s surface.

Similarly, to perform EBSD/ECCI measurements in the interior of the sample, the thickness of the sample was reduced to half. Specifically, the sample was ground and polished in the thickness direction using emery paper (#1000), diamond suspension (9 \( \mu \text{m}, 3 \mu \text{m} \)), and colloidal silica. The EBSD/ECCI measurements in the sample’s interior were performed under the same condition as that on sample’s surface.

3. Results

3.1. DIC Analysis

As shown in Fig. 4, the position of each grain was determined by superimposing the DIC result on the ECC image, by which the average plastic strain in each grain was calculated. Figure 4 shows the equivalent plastic strain (\( \varepsilon_{eq} \)) distribution after applying the maximum load of 90 N and subsequently unloading. Additionally, \( \varepsilon_{eq} \) was calculated by assuming the plane strain condition, although the mechanical condition on the surface was the plane stress condition. This is because the two-dimensional DIC does not provide information for \( \varepsilon_s \). The solution to the problem stemming from the difference between the plane stress and the strain conditions will be investigated in future work. In Fig. 4, the strain gradient was observed at the notch tip. The strain distribution was asymmetrical perhaps owing to the introduction of twisting during loading. The strain decreased from 1.80% to 0.20% as the distance from the notch increased. In this inhomogeneous strain distribution, the left half area of the image, where the strain gradient was particularly large, was considered for plastic strain measurements using EBSD/ECCI.

3.2. EBSD Measurement

The objective of this study was to evaluate the crystallographic misorientation distribution at the grain-size scale, namely, a spatial resolution below the grain size is not necessary. Therefore, GOS, which has a good correlation with plastic strain, was used. Figure 5 shows the GOS map near the notch obtained by the EBSD measurement (Fig. 5(a) on the surface and Fig. 5(b) in the sample’s interior). The details of Figs. 5(a’) and 5(b’) will be described later.

Figure 6 shows the relationship between the GOS and the average \( \varepsilon_{eq} \) for each grain obtained by the DIC on the sample’s surface. The measured GOS of grain H was high, owing to the presence of the subgrain boundary (Fig. 5(a’)) (the details will be described later). Therefore, the red circle, which shows a value without the effect of the subgrain boundary, is also plotted in Fig. 6. Hereafter, the GOS without the subgrain effect was used. Additionally, Eq. (6) was obtained by linear approximation as the relationship between the GOS and the \( \varepsilon_{eq} \) of each grain.

\[
GOS = 0.329\varepsilon_{eq} + 0.118 \quad \text{................. (6)}
\]

The units for GOS and \( \varepsilon_{eq} \) are [°] and [%], respectively. This equation was used to calculate the \( \varepsilon_{eq} \) of the sample’s interior from the GOS, where DIC analysis cannot be carried out. The result shown in Fig. 7 indicates that the plastic strain tended to decrease as the distance from the notch tip increased. However, by comparing grains 1 and 2 shown in Fig. 5(b’), it was found that the plastic strain of grain 2 was larger than that of grain 1, even though the distance from the notch tip of grain 2 was larger than that of grain 1. The reason for this unusual trend will be discussed later. Additionally, we must note here that the \( \varepsilon_{eq} \) on the sample’s surface was measured by assuming the plane strain condition as described above. This assumption becomes problematic when the interior strain of the sample is determined by Eq. (6). Because this problem will be investigated in future work, it is inherent in the discussion that follows.
Fig. 5. GOS maps coupled with grain boundary maps near the notch after unloading, as obtained by the EBSD measurement (a) on the surface and (b) in the interior of the sample. The GOS errors were caused by (a’) the presence of the subgrain boundary in grain H, and (b’) the grain size difference between grain 1 and grain 2. (Online version in color.)

Fig. 6. Relationship between the grain orientation spread, GOS, and local plastic strain $\varepsilon_{eq}$ on the sample’s surface. The data shown in Figs. 4 and 5(a) were used. The red circle indicates the GOS value after considering the effect of the subgrain boundary shown in Fig. 5(a’). (Online version in color.)

Fig. 7. EBSD-based assessment of the relationship between the local plastic strain $\varepsilon_{eq}$ in the sample’s interior, as calculated by Eq. (6), and distance from notch tip L. The GOS data shown in Fig. 5(b) were used. (Online version in color.)
3.3. ECCI Observation

Figure 8 shows the initial microstructure of the sample surface before deformation, as obtained by the ECCI method. Figure 8(c) was used to measure the initial dislocation density. The yellow lines in the figure indicate the mesh. As a result of the dislocation density measurements using Eq. (5), the initial dislocation density was determined to be $4.69 \times 10^{12} \text{ m}^{-2}$. Figure 9 shows the dislocation structure (a) on the sample surface and (b) in the sample’s interior, as obtained using the same ECCI method for each grain of the sample after the deformation.

Figure 10 shows the relationship between the dislocation density and the average $\varepsilon_{\text{eq}}$ for each grain, as obtained by the DIC on the sample’s surface. Additionally, as a result of linear approximation from each point, Eq. (7) was obtained as the relationship between the dislocation density and the $\varepsilon_{\text{eq}}$ of each grain, as follows:

$$\rho = 2.00 \times 10^{12} \varepsilon_{\text{eq}} + 4.69 \times 10^{12} \text{........ (7)}$$

The unit for $\rho$ and $\varepsilon_{\text{eq}}$ are $[\text{m}^{-2}]$ and $[\%]$, respectively. Similar to Section 3.2, this equation was used to calculate the $\varepsilon_{\text{eq}}$ of the sample’s interior from the dislocation density, and

![Figure 8](image1)

![Figure 9](image2)
4. Discussion

From the coefficients of determination $R^2$ in Figs. 6 and 10, it was found that the scatter of correlation between the GOS-DIC strain were larger than those between the dislocation density-DIC strain. First, we discuss the cause of error for each method. Next, we propose the plastic strain measurement method using EBSD/ECCI.

4.1. Error in GOS Calculation

4.1.1. Influence of Grain Boundary Determination

First, we note the grain H shown in Fig. 5(a’). In terms of the EBSD measurement characteristics, the subgrain boundaries with a misorientation below 5° were not identified as the grain boundaries. Instead, the subgrain boundaries were characterized as the misorientation of the grain interior. Hence, when a subgrain boundary with a misorientation below 5° was adjacent to the target grain, the GOS was over-estimated. Therefore, the GOS of grain H, which was adjacent to the subgrain boundary, was higher than that of the surrounding grains. Grain H and the adjacent grain striding across the subgrain boundary were identified as the same grain. Thus, the same GOS was assigned to these two grains. This problem was solved by extracting only grain H to measure the GOS again, and the result is presented in Fig. 6.

4.1.2. Grain Size Dependence

Secondly, we discuss grains 1 and 2 shown in Fig. 5(b’). This figure shows that the GOS of grain 2 was larger than that of grain 1. From a macroscopic viewpoint with regard to the plastic strain gradient, the plastic strain distribution obtained by the DIC analysis, as shown in Fig. 4, revealed that the plastic strain decreased monotonically as the distance from the notch tip increased. Therefore, the grain misorientation of each grain decreased as the distance from the notch tip increased. Moreover, from the result of the plastic strain in the sample’s interior, which was measured by the dislocation density shown in Fig. 11, the dislocation densities of grains 1 and 2 were approximately the same. Thus, for these grains, it is unlikely that the orientation change in grain 2 was greater than that of grain 1. As shown in Eq. (3), the GOS does not consider the effect of the target area (grain size), but is rather grain-area-dependent. More specifically, the GOS corresponds to the integrated value of the plastic strain within each grain divided by the grain area, which is problematic with regard to obtaining a quantitative correlation between the GOS and the strain. For example, we here consider two grains with a similar average plastic strain. When a plastic strain gradient exists as shown in Fig. 12, even if the average plastic strain is the same, the difference in the transgranular plastic strain increases as the grain size increases, and the GOS increases correspondingly. Hence, comparing and calculating the plastic strain of grains with largely different grain sizes may cause large errors. Because the grain size observed in this study did not consider a three-dimensional shape, it could not be concluded whether the plastic strain error was actually caused by the effect of the grain size. However, this is considered to be one of the most possible causes.

4.2. Error of Dislocation Density Measurement

Here, the effect of the grain boundaries is considered as a factor of error for the plastic strain calculated from the dislocation density. The dislocation density was only measured in the central area of the grains considered in this study. However, considering the distribution of dislocation that accumulates during the deformation, the GN dislocation density near the grain boundary is higher than that in the center of the grain, so as to satisfy the strain compatibility condition. If the grain size is small, the center of the grain is strongly affected by the high GN dislocation density near the grain boundary. Thus, the relationship between the dislocation density and the plastic strain has a grain size dependency, which introduces error (Fig. 13). Moreover,
the crystallographic orientation dependence of the formation behavior of dislocation substructures, such as cells, can also introduce error into the plastic strain-dislocation density relationship. However, as shown in Figs. 10 and 11, the dislocation density changed monotonically as a function of the DIC-plastic strain and distance from the notch tip. Note that the coefficient of determination was high in the dislocation density-strain relationship. These facts suggest that, in this case, the error caused by the abovementioned factor was small. Additionally, the recovery phenomenon is another significant factor that can introduce error. During the large plastic deformation, the dislocation density does not monotonically increase against the plastic strain because the effect of the dislocation density reduction, which is caused by the recovery, became significant owing to the multiplication of dislocation. Thus, for applicability to large deformation, it is necessary to measure another relationship between the dislocation density and the plastic strain.

4.3. Comparison of Interior Plastic Strain Analyses Calculated from EBSD-GOS and ECCI-dislocation Density

Figure 14 shows the relationship between the interior plastic strains calculated from the GOS and dislocation density. As can be seen, grains 1, 2, and 3 widely varied. Generally, from Eq. (4), when the mean dislocation motion distance is constant, the plastic strain is proportional to the dislocation density. Additionally, as can be seen in Fig. 10, the plastic strain and dislocation density had a linear relationship with a high coefficient of determination. Hence, because the plastic strain obtained by the dislocation density measurement was highly reliable, the deviation from the linear relationship shown in Fig. 14 was attributed to the plastic strain error associated with the problem of the GOS. As has been already pointed out, the high GOS of grain 2 was attributed to the large size of the grain. As can be seen in Fig. 5(b), the GOS values for grains 3 and 4 were low because the grain sizes were smaller compared with the surrounding grains.

4.4. Proposal: Plastic Strain Measurement Using EBSD and ECCI

First, we discuss the characteristics of EBSD measurement. Because EBSD is a method enabling the automatic measurements of grain orientations in a large number of wide areas, a statistical correlation with the plastic strain can be easily obtained. Therefore, the GOS is effective in evaluating the plastic strain statistically with a spatial resolution approximating the grain size. However, the presence of the subgrain boundary and the grain size dependency cause result scattering, and thus, statistical analysis with multiple data is required.

Secondly, we discuss the ECCI characteristics. The dislocation density measurement using the ECCI is very reliable for quantitatively estimating the local plastic strain. However, compared with the EBSD measurement, the dislocation density measurement requires a substantial amount of time for observation and analysis. Thus, for analyzing a large number of wide areas, it is effective to combine it with the EBSD measurement.

Hence, we propose the following method combining the EBSD and ECCI for measuring the plastic strain distribution. First, a sample with a deformation gradient is prepared and the relationship between the GOS and plastic strain is obtained by the EBSD and DIC. In this case, the unit area for the analysis is set to be sufficiently larger than the grain size, and the average GOS value of multiple grains in each unit area was used to reduce the error of the relationship between the GOS and the plastic strain. When the elastic strain effect induced by the grain boundaries and second phase particles, such as precipitates, is negligible, the GOS and dislocation density have a linear relationship with the same deformation as that considered in this study. Therefore, by measuring only the initial dislocation density using
the ECCI, the relationship between the dislocation density and the plastic strain can be obtained by the relationship between the GOS and the plastic strain. With this method, the internal plastic strain can be accurately measured with a reduced number of dislocation density measurements. We expect that this measurement can lead toward clarifying the ductile failure and ductile crack propagation mechanisms.

5. Conclusions

In this study, the GOS and dislocation density at the stress concentration area on the sample’s surface were measured using the EBSD and ECCI. By combining these results with DIC, we evaluated the correlation between the plastic strain and the grain orientation gradient/dislocation density, and demonstrated the plastic strain distribution measurement using the EBSD/ECCI. Moreover, the EBSD and ECCI measurements were used to measure the heterogeneous plastic strain distribution in the sample’s interior, wherein the DIC was not applicable. The following results were obtained.

(1) From the measurements using EBSD and ECCI at the stress concentration area on the sample’s surface, it was shown that the $\varepsilon_{eq}$ measured by the DIC had a linear relationship with the GOS and dislocation density, respectively.

(2) By measuring the stress concentration area in the sample’s interior, the internal plastic strain distribution was obtained from the relationship between $\varepsilon_{eq}$ and the GOS/dislocation density obtained on the sample’s surface. Based on this result, we found that the internal plastic strain tended to monotonically decrease as the distance from the notch tip increased.

(3) The presence of subgrain boundaries and the grain size dependence are considered as factors introducing the GOS error measured by the EBSD.

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