Effect of \(\gamma\)-Phase Stability on Local Deformation Energy of \(\alpha\)-\(\gamma\) Duplex Stainless Steel

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The effect of austenite (\(\gamma\)) phase stability on the local deformation region on the deformation energy of ferrite (\(\alpha\))-\(\gamma\) duplex stainless steel is investigated using tensile testing. The uniform deformation energy values of both the \(\gamma\)-stable and unstable duplex stainless steels are similar, while the local deformation energy of \(\gamma\)-stable duplex stainless steel is larger than that of \(\gamma\)-unstable duplex stainless steel. In \(\gamma\)-unstable duplex stainless steel, the difference in nano-indentation hardness between the \(\alpha\) and transformed martensite (\(\alpha’\)) phases increases remarkably beneath the fractured surface. Low-voltage electron microscopy observations reveal that voids grow rapidly at the grain boundaries between the \(\alpha\) and \(\alpha’\) phases beneath the fractured surface, resulting in a decrease in the local deformation energy in \(\gamma\)-unstable duplex stainless steel.

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1. Introduction

Ferrite (\(\alpha\))–austenite (\(\gamma\)) duplex stainless steel is widely used in chemical plants, precision machinery, and medical instruments owing to its excellent strength, ductility, and corrosion resistance. This material is broadly classified into \(\gamma\)-unstable and \(\gamma\)-stable types. In the \(\gamma\)-unstable steel, transformation-induced plasticity (TRIP) occurs\(^1\), where the \(\gamma\) phase transforms into the martensite (\(\alpha’\)) phase; the \(\gamma\)-stable steel does not show TRIP behavior. Many studies have shown that the TRIP effect in \(\gamma\)-unstable steel causes greater total elongation and described the mechanisms involved. In 1975, Nakamura et al.\(^{21}\) revealed that the TRIP effect can be used to increase total elongation in tensile tests.

Both uniform and local elongations contribute to the total elongation and these are controlled by different microstructural behaviors. In the uniform elongation deformation region, work hardening behavior is dominant that causes crystal slips and grain rotations prevails, along with dislocation increases and movements. In the local elongation deformation region, work hardening continues, generating voids that grow in size as plastic deformation continues. When certain critical conditions are reached, the voids rapidly merge, resulting in fracture.

The mechanisms underlying the observed uniform deformation behavior have now been clarified theoretically and experimentally and discussed with respect to dislocation theory. Local deformation occurs under the complicated influences of mechanical and microstructural factors including multiaxial stress after necking and deformability of the matrix structure, where the formation, growth, and connection of inner micro-voids are the fundamental processes causing deformation. The micro-void formation begins at secondary-phase particles, such as nonmetallic inclusions and precipitates or crystal grain boundaries. Many studies have investigated local deformation by considering voids in steel materials\(^{3–5}\) void growth and connection models\(^{6–8}\) and numerical calculations based on fracture mechanics\(^{9,10}\). However, only a few studies discuss the relationship between mechanical and microstructural factors.

As mentioned above, the mechanical and microstructural factors that control uniform elongation are completely different to those controlling local elongation. However, many previous studies discuss the material properties using the total elongation as an indicator. A study by Nakamura et al.\(^2\) also discusses the TRIP effect with reference only to increasing total elongation. Therefore, it is not clear whether the TRIP phenomenon increases local elongation in duplex stainless steels. In order to identify the major factors controlling local elongation, it is proposed that it is necessary to consider both the mechanical properties and microstructure, observing the formation and growth of voids and elucidating the differences in work-hardening behavior between the two phases. Furthermore, it is essential to establish an experimental method for observing micro-voids and a microscopy method for evaluating work-hardening behavior in the respective phases of the two-phase structure.

Previously, the authors presented a method for detecting voids of the order of 0.1 micro meters using the secondary electron (SE) detector and angle-selective backscattered (AsB) electron detector of a low-voltage field emission scanning electron microscope (FE-SEM)\(^{15}\). In addition, we demonstrated that nano-indentation testing is an effective tool for analyzing plastic deformation behavior of a microstructure after tensile testing\(^{4,11}\).

In this paper, the authors report the results of testing two types of duplex stainless steel with different Ni contents that do not show the TRIP phenomenon in the uniform elongation region. The above-mentioned experimental methods were used to investigate the effect of the TRIP phenomenon in the local elongation region under high strain on the formation and growth of voids and the elongation behavior. Be-
cause elongation is inversely correlated with strength, it is not an appropriate measure for evaluating the deformation properties of materials with different strengths. In a tensile test, dynamic characteristics appear according to the applied external force. Assuming that a test specimen is a single system, the first law of thermodynamics applies; therefore, the work (dW) done during tensile testing can be regarded as a physical quantity that influences the change in the internal energy (dU) of the system[12]. Generally, the input and output of heat (dQ) can be ignored in standard tensile tests[5]; therefore, it is possible to obtain dU by determining dW. Hence, dU can be used to evaluate the deformation behavior of specimens with different strengths. For these reasons, this study does not use local elongation itself, but rather evaluates the local deformation energy or the dU value of the system in the local elongation region obtained from the stress-strain curves from tensile testing[4,12,13].

2. Experimental Methods

2.1 Test materials

Two types of commercially available α-γ duplex stainless steel with different Ni contents were used here; one of these steels was γ-stable and the other γ-unstable in the local elongation region. The chemical compositions of these steels are shown in Table 1. Specimens for tensile testing were taken from the steel sheets in the direction parallel to the rolling direction. A plane perpendicular to the transverse direction of the base material was wet-ground with #2500 emery paper and then polished to a mirror finish using diamond paste and colloidal silica and then etched with aqua regia. Figure 1 shows the microstructures of treated specimens observed using optical microscopy.

Next, electron backscatter diffraction (EBSD) images under an accelerating voltage of 15 kV were obtained for the γ-stable duplex stainless steel using a Carl Zeiss Ultra55 FE-SEM. We confirmed using electron diffraction orientation imaging microscopy (OIM) that the white and gray phases shown in Fig. 1 corresponded to the α and γ phases, respectively, as shown in Fig. 2. The image shown in Fig. 2 (a) was taken with an optical microscope and shows part of the Vickers indentation used as a marker on the left side. Figures 2 (b) and (c) show the inverse pole figures (IPFs) of the area indicated in (a), corresponding to the α and γ phases, respectively. It was found that the white region corresponded to the α phase and the gray region to the γ phase; the ratio of the α phase to γ phase was 6:4 in the γ-stable steel and 7:3 in the γ-unstable steel.

2.2 Tensile testing

Cylindrical specimens 6 mm in diameter, as shown in Fig. 3, were prepared for tensile testing. The tensile tests were conducted with an initial strain velocity of 1.0 × 10^{-3} s^{-1} over three cycles. Gauge marks were made 25 mm apart on the parallel portions of the specimens. The marked areas were imaged with a camera to record change in the displacements. From the tensile test, the 0.2% yield strength, tensile strength, uniform elongation, local elongation, and uniform and local deformation energies (as defined in Fig. 4) were determined.

2.3 Nano-indentation hardness tests

After the tensile tests, specimens for hardness testing were prepared, as shown in Fig. 5. The plane of the specimen perpendicular to the fractured surface was embedded in resin, buffed, and polished with colloidal silica. Changes in the hardness (H_{RT}) of the specimen as a function of distance from the fractured surface were measured using an Elionix ENT-2100 nano-indentation tester under a test load of 9.81 mN. Figure 6 shows a schematic diagram of the posi-

### Table 1

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>N</th>
<th>Cr</th>
<th>Mo</th>
<th>Al</th>
<th>N</th>
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<td>A</td>
<td>0.02</td>
<td>0.50</td>
<td>0.75</td>
<td>7.1</td>
<td>24.9</td>
<td>3.0</td>
<td>0.02</td>
<td>0.11</td>
</tr>
<tr>
<td>B</td>
<td>0.02</td>
<td>0.35</td>
<td>0.98</td>
<td>5.4</td>
<td>22.5</td>
<td>3.2</td>
<td>0.03</td>
<td>0.15</td>
</tr>
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</table>

Fig. 1 Optical micrographs of (a) γ-stable and (b) γ-unstable steels tested.

Fig. 2 EBSD analysis results of γ-stable duplex stainless steel: (a) Optical micrograph, (b) IPF image of bcc, (c) IPF image of fcc.

Fig. 3 Dimension of tensile test specimen.
tions on the sample at which hardness measurements were taken. Starting on a point about 100 μm from the fractured surface, measurements were taken at points on lines at intervals of 20 μm up to a point about 1.5 mm from the fractured surface, and then on lines at intervals of 500 μm. Five measurements were taken in the transverse direction of the specimen at intervals of 20 μm between test points on each line, and the average of the five measurements on each line was compared to the specimen deformation. The deformation was defined as the true strain $\varepsilon_x$ at a distance $x$ from the fractured surface, as shown by eq. (1) below:

$$\varepsilon_x = 2 \ln \left( \frac{D_0}{D} \right),$$

(1)

where $D_0$ is the initial diameter of the specimen (here, 6 mm) and $D$ is the diameter at distance $x$ where the hardness measurement was taken after the tensile test.

After the nano-indentation tests, an EBSD image of the specimen was obtained using a Hitachi High-Technologies SU6600 scanning electron microscope (SEM). The image data were analyzed with an orientation-imaging microscope (OIM) to identify the phases present at the indentation and determine the hardness values of the $\alpha$ and $\gamma$ phases and their relationship with $\varepsilon_x$. Hardness measurements taken on the grain boundaries and on or around voids were excluded from the calculations.

2.4 Observations of voids

SEM specimens were prepared by cutting a cross-sectional test piece from the fractured sample using a micro-cutter. The plane perpendicular to the fractured surface was wet-ground sequentially using No. 150 to No. 2000 emery paper, and then buffed with alumina paste to a mirror finish. Next, the specimen was subjected to argon sputtering to remove any surface contamination and damaged layers (treatment time of 3 min; accelerating voltage of 4 kV; angle of inclination of 80°; rotation speed of 25 rpm) using a Hitachi High-Technologies IM-3000 ion milling system. An AsB image of the specimen was obtained using a Carl Zeiss Ultra55 FE-SEM at an accelerating voltage of 5 kV to observe voids down to 0.1 μm in size. Image analysis was performed in order to quantify the number and area ratio of the voids using ImageJ software. In addition, an EBSD image was obtained at an accelerating voltage of 15 kV and the points of void initiation were analyzed using OIM.

3. Results

3.1 Strength, elongation, and deformation energy

Figure 7 shows examples of stress–strain curves obtained...
from the tensile testing undertaken for three times per each steel. These results show that the γ-stable duplex stainless steel had a greater 0.2% proof stress and tensile strength than the γ-unstable steel. This was attributed to the fact that the γ-stable specimen used here had a harder γ phase (as discussed later with reference to Fig. 15) and a higher total fraction of γ phase. The total elongation was highest for the γ-stable steel. Considering the uniform and local elongations separately, both of which were components of total elongation, the γ-unstable steel had a greater uniform elongation and smaller local elongation than the γ-stable steel. The main purpose of this study was to clarify the effects of γ phase transformation in the local deformation area on the local deformation energy. Following is a discussion of the experimental results from this perspective. Figure 8 shows ductile fracture energies determined from the measured stress–strain curves. The uniform deformation energy of both types of steel was almost equal, whereas the γ-stable steel showed about a 10% larger local deformation energy.

### 3.2 Observations of voids

Figure 9 shows the relationship between εₓ and the percentage area of voids, Sv, or the number of voids, Nv, per unit area (μm²). The void measurement area was 700 μm in length and 30 μm in width for each εₓ value. Both the γ-stable and γ-unstable steels showed an increase in the number of voids with increasing strain. The γ-unstable steel showed a sharp increase in Sv at εₓ immediately beneath the fractured surface, whereas the γ-stable steel showed only a slight increase. Next, the relationship between εₓ and average void size (Sv/Nv) was calculated to observe the void growth behavior, as shown in Fig. 10. The γ-stable steel showed little void growth, even just before the fracture occurred, whereas the γ-unstable steel showed rapid void growth immediately before fracture. The observed positions nearest to the fracture surface were drawn in the micrographs of longitudinally sectioned in Fig. 10 (see arrows). This analysis revealed that the void growth behavior differed significantly between γ-stable and γ-unstable steels.

### 3.3 Observations of voids using EBSD imaging

Figure 11 (a) to (d) show the results of the EBSD analysis of the cross-section of the γ-unstable steel at a low true strain of εₓ = 0.28. Figure 11 (a) shows an SEM image of the plane, (c) is the result of an EBSD analysis of the entire test region, and (b) and (d) are the results of extracting only bcc and fcc structures, respectively. These results reveal that the voids were generated from the bcc interface. After analysis of the other fields of the microscope, it was found that 80% of the voids were created from the biphase interface. Figure 12 shows the results of the same EBSD analysis of the cross-section of the γ-stable steel at a true strain of εₓ = 1.20. The results suggest that the voids were generated within the γ phase or from the γ grain boundary. The analysis of the other fields of the microscope confirmed that 90% of the voids were created within the γ phase or from the γ grain boundary.

### 3.4 Nano-indentation hardness

Figures 13 (a) and (b) show the nano-indentation hardness measurements of the γ-stable and γ-unstable duplex stainless steels, respectively, after tensile testing. Both steels showed higher hardness values closer to the fractured surface. As commonly observed, the γ phase showed a higher work-hardening rate than the α phase. The data points showed as solid squares in this figure represent the hardness values of the α’ (martensite) phase. The γ-unstable steel showed higher hardness values than the γ-stable steel, which is attributed to the TRIP behavior of the γ phase when it transforms into the α’ phase at εₓ = 1.57. This phenomenon is discussed in more detail in the following section.

### 3.5 Strain-induced transformation

Figure 14 shows EBSD images of the specimens at a position 0.2 mm from the fractured surface where εₓ = 1.57. Figure 14 (a), (b), and (c) show IPF images of both the ferrite and austenite phases, only the ferrite phase, and only the austenite phase at εₓ = 1.57, respectively.
austenite phase, respectively. In Fig. 14 (c), the areas surrounded by solid and dashed yellow lines represent a phase subject to twinning deformation and a phase with substantial noise, respectively. Investigating the orientation of the grain boundary in the area surrounded by the solid yellow line, it was found to have a $\Sigma 3$ structure, which is proof of twinning deformation. The area indicated by the dashed yellow line was speculated to be strain-induced martensite. Figure 15 shows the pole figures of $\gamma$[111] and $\alpha$[011] in the same area$^3$. As the positions of the circled points were very similar for both figures, it was confirmed that the $\gamma$[111] plane was parallel to the $\alpha$[011] plane. Hence, it was confirmed that the area indicated by the dashed yellow line in Fig. 14 (c) underwent martensitic transformation. Therefore, it is
clear that the γ-unstable steel tested in this study showed TRIP phenomenon at high strain.

4. Discussion

4.1 Hardness ratio and void growth behavior

From the hardness relationship shown in Fig. 13, the hardness ratio of the α-phase or strain-induced α` phase to the γ phase was determined for γ-stable and γ-unstable steels, as shown in Fig. 16. The γ-stable steel showed a slight increase in the hardness ratio, whereas the γ-unstable steel showed a sharp increase in the hardness ratio near the fracture surface. Together with the results confirming the martensitic transformation (Figs. 14 and 15), these data suggest that TRIP behavior occurred in the high-strain region (immediately before fracture) where $\epsilon_x \geq 1.5$. This sharp increase in hardness ratio is thought to have caused the rapid growth of voids in the γ-unstable steel, as shown in Fig. 10.

4.2 Factors controlling local deformation energy

No significant difference in uniform deformation energy was observed between the γ-stable and γ-unstable steels; however, a larger local deformation energy was measured for the γ-stable steel, as shown in Fig. 8. Meanwhile, the work-hardening behavior in the γ and the α phases of the γ-stable and the γ-unstable steels was identical, irrespective of the uniform or local deformation region, as shown in Fig. 13. Hence, the two steels showed an equal change in inner energy attributable to the formation of dislocations during uniform and local deformation of the matrix phase during tensile tests. Accordingly, the difference in local deformation energy between the two steels may be attributable to the different void growth processes. As shown in Fig. 10, the γ-stable steel showed extremely little void growth until close to fracture, whereas the γ-unstable steel showed gradual void growth starting at very small $\epsilon_x$ values and reaching final fracture at a relatively low $\epsilon_x$. Hence, the γ-unstable steel experienced rapid void growth, beginning in the low-strain region, resulting in low local deformation energy.

5. Conclusions

We studied the effects of γ-stability on deformation energies determined from elongation and stress–strain curves obtained during tensile testing, in the local deformation region where a high strain was applied, using γ-stable α-γ and γ-unstable α-γ duplex stainless steels. The major conclusions are given below.

(1) In the local elongation region, the local deformation energy of the γ-stable stainless steel was larger than that of the γ-unstable stainless steel, in which the γ phase transforms into the α` phase.

(2) In the γ-unstable stainless steel, voids were generated mainly at the interface between the two phases. For the γ-stable stainless steel, voids were generated within the...
γ phase or at the γ grain boundary.

(3) The γ-stable stainless steel showed an extremely small amount of void growth up until a point close to fracture and rapid void growth immediately before fracture. Meanwhile, the γ-unstable stainless steel showed gradual void growth starting at low strain and rapid void growth immediately before fracture.

(4) In the γ-unstable steel, the creation of a strain-induced α’ phase before the moment of fracture increased the hardness ratio of the two phases. From these results, we reached the conclusion that, in order to increase the local deformation energy of α-γ duplex stainless steel, it is important to ensure strain stability in the local deformation region of the austenite phase.

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