Yield stress of duplex stainless steel specimens estimated using a compound Hall–Petch equation

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Abstract

In this study, the 0.2% yield stress of duplex stainless steel was evaluated using a compound Hall–Petch equation. The compound Hall–Petch equation was derived from four types of duplex stainless steel, which contained 0.2–64.4 wt\% $\delta$-ferrite phase, had different chemical compositions and were annealed at different temperatures. Intragranular yield stress was measured with an ultra-microhardness tester and evaluated with the yield stress model proposed by Dao \textit{et al.} Grain size, volume fraction and texture were monitored by electron backscattering diffraction measurement. The $k_\gamma$ constant in the compound equation for duplex stainless steel agrees well with that for $\gamma$-phase SUS316L steel in the temperature range of 1323–1473 K. The derived compound Hall–Petch equation predicts that the yield stress will be in good agreement with the experimental results for the Cr, Mn, Si, Ni and N solid-solution states. We find that the intragranular yield stress of the $\delta$-phase of duplex stainless steel is rather sensitive to the chemical composition and annealing conditions, which is attributed to the size misfit parameter.

Keywords: 0.2\% yield stress, duplex stainless steel, compound Hall–Petch equation, intragranular yield stress, ultra-microhardness tester, grain size, volume fraction, electron backscattering diffraction, solid solution, size misfit parameter

1. Introduction

Duplex stainless steel has high strength and superior corrosion resistance. Owing to this, it has been widely applied in many industrial facilities, such as chemical plants and water-power plants [1–3]. However, it exhibits a much lower elongation than austenitic stainless steel. Recently, controlling the microstructure features of duplex stainless steel has been emphasized and investigated in depth in order to increase the ductility of duplex stainless steel. One important microstructure feature of duplex stainless steel is the volume fraction of the austenite phase. An increased austenite volume fraction results in an increased elongation for $\delta/\gamma$ duplex-phase stainless steel [4]. However, a decrease in $\delta$-ferrite volume fraction reduces yield stress. In addition to the volume fractions of the two phases, grain size and other microstructural features are also considered to have a significant effect on the strength of duplex stainless steel. Thus, an accurate estimation of the yield stress of duplex stainless steel is important for optimizing the properties of this steel.

Nam \textit{et al.} [5] proposed the compound Hall–Petch equation to estimate the yield stress of drawn pearlitic steel as follows:

$$\sigma_\gamma = (\sigma_{c0} + k_c t_c^{-1/2}) V_c + (\sigma_{f0} + k_f t_f^{-1/2}) V_f. \quad (1)$$
Here, \( \sigma_y \) is the 0.2% yield stress of drawn pearlitic steel; \( \sigma_{0y} \) and \( \sigma_{0a} \) are the friction stresses of the lamellar cementite and ferrite phase, respectively; \( k_y \) and \( k_a \) are the correspondent Hall–Petch constants of the two phases; \( t_c \) and \( t_f \) are thicknesses, and \( V_c \) and \( V_f \) are the volume fractions of the cementite and ferrite phases, respectively. The values calculated using equation (1) show good agreement with the experimental results for pearlitic steel. In deep-drawn pearlitic steel, a regular lamellar microstructure and distinctly different strengths of the component phases facilitate the estimation of yield stress using the compound Hall–Petch equation. In contrast, more microstructural features should be taken into account for \( \delta/\gamma \) duplex-phase stainless steel, such as the texture of each phase formed after thermomechanical processing [6, 7].

The yield stress of \( \gamma \) or \( \delta(\alpha) \) single-phase steel has been well investigated, and its grain size dependence has been expressed using Hall–Petch equations. In SUS316L stainless steel, Kashyap and Tangri proposed a Hall–Petch relationship between 0.2% yield stress and \( \gamma \)-phase grain size [8]. The relationship varied between two grain size ranges. For grains smaller than 6 \( \mu \)m, it was

\[
\sigma_y = \sigma_{0y} + 547D_y^{−1/2}.
\]

Here, \( \sigma_y \) is the 0.2% yield stress of SUS316L steel, \( \sigma_{0y} \) is the friction yield stress of the \( \gamma \)-phase and \( D_y \) is the grain size for the \( \gamma \)-phase. \( k_y \) in the Hall–Petch equation was obtained as 547 MPa \( \mu \)m\(^{-0.5}\).

For grains larger than 10 \( \mu \)m, the equation changed into

\[
\sigma_y = \sigma_{0y} + 121D_y^{−1/2}.
\]

In this case, \( k_y \) in the Hall–Petch equation was 121 MPa \( \mu \)m\(^{-0.5}\). Similarly, Tsuchida et al [9] obtained a Hall–Petch relationship with ferrite-phase grain size in low-carbon steel as

\[
\sigma_y = \sigma_{0a} + 475D_a^{−1/2}.
\]

Here, \( \sigma_y \) is the 0.2% yield stress of low-carbon steel, \( \sigma_{0a} \) is the friction yield stress of the ferrite phase and \( D_a \) is the grain size for the \( \alpha \)-phase. In most cases, friction yield stress is obtained by fitting the experimental dependence of yield stress on grain size using the Hall–Petch equation. However, this procedure seems impossible for duplex stainless steel, as the two stress constants cannot be obtained with one set of experimental results of yield stress variation with grain size.

Recently, Dao et al [10] proposed a comprehensive computational model for estimating the local yield stress of materials during nanoindentation-induced deformation. By measuring the load curve change with indenter depth up to the highest peak load, as shown in figure 1, the yield stress of the locally indented phase can be deduced by applying data of elastic deformation at the unloading area displacement \( h-h_f \) [11]. The expression developed for yield stress involves the relationship between the load \( P \) and the depth \( h \) during loading as well as the Young’s modulus \( E \) deduced during unloading. As the derived intragranular yield stress is independent of grain size for the \( \gamma \)- and \( \delta \)-phases, the obtained intragranular yield stress reflects the friction yield stress \( \sigma_{f\gamma} \) and \( \sigma_{f\alpha} \), which are required in the Hall–Petch equation.

The purpose of this study is to evaluate the applicability of the Hall–Petch relationship to the analysis of different types of duplex stainless steel with various volume fractions of the \( \delta \)-phase, as well as to determine the appropriate grain size for each phase, by controlling the composition of the steel and annealing temperature. To establish a reliable simulation equation for yield stress, Dao et al’s equation for yield stress is used to estimate intragranular yield stress for each phase.

2. Experimental procedure

Four duplex stainless steel specimens, with nominal compositions, namely, Fe-17Cr-12Ni, Fe-19Cr-12Ni, Fe-22Cr-12Ni and Fe-25Cr-7Ni-0.15N, were investigated and compared with SUS316L single-phase austenite stainless steel. The chemical compositions of these alloys are listed in table 1. The carbon contents were 0.028 wt\% in SUS316L, 0.016 wt\% in Fe-17Cr-12Ni, 0.019 wt\% Fe-19Cr-12Ni, 0.045 wt\% in Fe-22Cr-12Ni and 0.011 wt\% in Fe-25Cr-7Ni-0.15N. The three alloys Fe-17Cr-12Ni, Fe-19Cr-12Ni and Fe-22Cr-12Ni were prepared in argon atmosphere using an induction-melting furnace with a capacity of 20 kg. The alloy Fe-25Cr-7Ni-0.15N was industrial steel purchased from Sumitomo Metals, Ltd. Ingots of these alloys were homogenized at 1473 K for 1 h and hot-forged into 20-mm-thick slabs. After solid-solution annealing at 1323 K for 1 h, samples with a 20 \( \times \) 20 mm\(^2\) cross section were cut from the annealed slabs and rolled at room temperature. Cold-rolled samples with a 2 \( \times \) 21 mm\(^2\) cross section were finally obtained with an accumulated rolling reduction of 90\%. To vary the microstructure of the steel specimens, the cold-rolled samples were then annealed for 1 h at 1323, 1473 and 1573 K.

Tensile test on the annealed samples was conducted at room temperature along the rolling direction (RD) of the rolled steel plates. The tensile specimens were 2-mm-thick plates with a 35 \( \times \) 6 mm\(^2\) gauge section. Electron backscattering diffraction (EBSD) measurement was performed on the transverse-direction (TD) sections.
of the rolled and annealed samples. Samples for EBSD measurement were electrochemically polished in perchloric acid/butoxyethanol/ethanol solution. EBSD measurement was performed with a Carl–Zeiss LEO–1550 scanning electron microscopy (SEM) system equipped with an orientation–imaging microscopy (OIM) system from TexSEM Laboratories Inc.

The intragranular yield stress of the γ- and δ-phases was measured in the normal direction (ND) using a Shimadzu DUH-211 ultra-micro hardness tester with a Berkovich indenter, at a maximum load of 98 mN. Intragranular yield stress was calculated according to Dao et al.’s algorithm, using the slope load, the maximum load under test load and concavity depth after unloading. In the first step, a unique solution for the reduced Young’s modulus $E^*$ was applied, as shown in the following equation, which is the same as Oliver’s equation.

$$E^* = \frac{1}{C^*} \frac{dp_u}{dh} \bigg|_{h_o}.$$  

(5)

Here, the constant $C^* = 1.167$ because the Berkovich indenter takes into account the elasto-plastic finite deformation prior to unloading. The true projected contact area $A_m$ is taken as $24.56h^2$ where $h$ is the displacement in the unloading curves. The unloading slope $dp_u/dh$ is determined as shown in figure 1. The next step is to determine the yield stress at a plastic strain of 0.033, $\sigma_{0.033}$, with the following equation; $\sigma_{0.033}$ is chosen because true stress can be determined regardless of the strain hardening exponent $n$:

$$C = \sigma_{0.033} \times \Pi_1.$$  

(6)

The polynomial function $\Pi_1$ can be obtained by fitting the relationship between $\sigma_{0.033}$ and $E^*$; $C$ is the slope of the line through the load $P$ and the displacement $h$ during loading. For example, in the case shown in figure 1, $C = 53.2$ is deduced using $P = 53.2h^2$. Therefore, $\sigma_{0.033}$ can be derived by fitting to the change in $\Pi_1$ including $\sigma_{0.033}$. In other words, it is not allowed to change $\Pi_1$ for intragranular yield stress. $n$ is determined using

$$\Pi_2 \left( \frac{E^*}{\sigma_t}, n \right) = \frac{1}{E^*} \frac{dp_u}{dh} \bigg|_{h_o}.$$  

(7)

The dimensionless function $\Pi_2(E^*/\sigma_t, n)$ relates three fitting parameters: $n, \sigma_{0.033}$, and $E^*$. Finally, $\sigma_t$ is derived from $\sigma_{0.033}$ and $n$ using

$$\sigma_{0.033} = \frac{\sigma_t}{\left( 1 + \frac{E^*}{\sigma_t} \right)^{0.033}}.$$  

(8)

where $E = 197$ GPa for the γ-phase and $E = 200$ GPa for the δ-phase [12]. The 0.2% yield stress after the tensile test is compared with the Hall–Petch relationship for duplex stainless steel where the volume fraction for the δ-phase, the grain size of each phase, and intragranular yield stress were taken from the ultra-micro hardness measurements.

3. Experimental results and discussion

3.1. Mechanical properties

Figure 2 shows the stress-strain curves for SUS316L (Fe-16Cr-16Ni), Fe-17Cr-12Ni, Fe-19Cr-12Ni, Fe-22Cr-12Ni and Fe-25Cr-7Ni-0.15N steel specimens that were cold-rolled and annealed at 1323, 1473 and 1573 K. As shown in table 2, the increase in the Cr content of the steel specimens clearly improves tensile strength and yield stress owing to the higher fraction of the δ-phase. Also, the increase in annealing temperature from 1323 to 1473 K clearly increases the yield strength of three steel specimens, while further increase in annealing temperature causes different effects in the different steel specimens. On the other hand, total elongation to rupture tends to decrease with increasing annealing temperature and Cr content of the steel specimens, while elongation for Fe-22Cr-12Ni remains almost unchanged by the annealing.

3.2. Microstructural analysis

Figure 3 shows the OIM images of the TD plane in the γ- and δ-phases of the Fe-17Cr-12Ni, Fe-19Cr-12Ni, Fe-22Cr-12Ni and Fe-25Cr-7Ni-0.15N steel specimens annealed at 1323 K. Grain orientations along the ND in both phases in these steel specimens are shown in standard inverse pole figure color. To distinguish between the γ- and δ-phases, the former is defined as the fcc phase and the latter as the bcc phase. The grain boundary is indicated for grains with a misorientation $\theta \geq 15^\circ$. Several data analyses use a cleanup parameter mode to obtain higher-accuracy data. In this mode, grain tolerance angle set at 5° and minimum grain size at 2 μm. Figure 3 reveals annealing twinning boundaries in the γ-phase grains, and grain size was obtained after removing such twinning boundaries. With an increase in Cr content from 17 to 22 wt%, γ-phase grain size decreases, while δ-grain size increases. Figure 4 shows the OIM images of the TD plane in the γ- and δ-phases of Fe-22Cr-12Ni steel annealed at 1323, 1473 and 1573 K. The grain sizes of the γ- and δ-phases, as well as the volume fraction of the δ-phase, increase with annealing temperature. In addition, a strong ND/⟨110⟩ texture of the γ-phase and the ND/⟨111⟩ texture of the δ-phase appeared in the annealed microstructure, which are typical annealing.

Table 1. Chemical compositions of the steel specimens studied (in wt%).

| Alloy          | C  | Si | Mn | P   | S  | Ni | Cr | Mo | N   |
|---------------|----|----|----|-----|----|----|----|----|----|
| SUS316L       | 0.028 | 0.45 | 0.72 | <0.005 | 0.002 | 16.2 | 16.4 | 2.80 | 0.002 |
| Fe-17Cr-12Ni  | 0.016 | 0.44 | 0.72 | <0.005 | 0.002 | 12.0 | 16.5 | 2.84 | 0.002 |
| Fe-19Cr-12Ni  | 0.019 | 0.45 | 0.72 | <0.005 | 0.002 | 12.1 | 19.3 | 2.83 | 0.002 |
| Fe-22Cr-12Ni  | 0.045 | 0.41 | 0.67 | <0.005 | 0.005 | 12.0 | 21.8 | 2.84 | 0.005 |
| Fe-25Cr-7Ni-0.15N | 0.011 | 0.49 | 0.86 | <0.005 | 0.001 | 7.22 | 25.3 | 3.14 | 0.15 |
and were well homogenized. Fe-17Cr-12Ni, Fe-19Cr-12Ni, Fe-22Cr-12Ni and Fe-25Cr-7Ni-0.15N steel specimens, cold-rolled and annealed at 1323, 1473 and 1573 K. The grain sizes for the Fe-17Cr-12Ni, Fe-19Cr-12Ni, Fe-22Cr-12Ni and Fe-25Cr-7Ni-0.15N steel specimens with annealing temperature. The grain sizes for the γ- and δ-phases in the SUS316L, Fe-17Cr-12Ni, Fe-19Cr-12Ni, Fe-22Cr-12Ni steel specimens. With those quantitative microstructural features, we can consider the applicability of the compound Hall–Petch relationship (equation (1)), to the estimation of 0.2% yield stress in the steel specimens. First, kγ in the Hall–Petch equation for the single γ-phase in SUS316L steel is obtained as 164 MPa μm−0.5. This kγ is nearly the same as that reported by Kashyap et al. in large γ-phase grains. σγ0,γ is therefore calculated by applying the Hall–Petch equation, σγ = σγ0,γ + 164Dγ−1/2, to the determination of the experimental yield strength of SUS316L steel shown in table 3. The average σγ0,γ was deduced as 211 MPa, which is quite similar to that reported by Kashyap and Tangri. Thus, the yield stress of SUS316L can be defined as

\[ \sigma_{y,\gamma} = 211 + 164D_{\gamma}^{-1/2}. \]  

(9)

However, the Fe-22Cr-12Ni steel clearly shows smaller γ-phase grains than Fe-19Cr-12Ni steel. The grain size for the γ-phase decreases gradually with increasing Cr content, and the order of decrease is Fe-17Cr-12Ni > Fe-19Cr-12Ni > Fe-22Cr-12Ni. As shown in figure 5(b), the volume fraction of the δ-phase increases almost linearly with annealing temperature in the Fe-19Cr-12Ni and Fe-22Cr-12Ni steel specimens. The volume fraction of the δ-phase in Fe-22Cr-12Ni steel is a little larger than that in Fe-19Cr-12Ni steel. On the other hand, the volume fraction of the δ-phase increases with increasing Cr content, and the order of increase is Fe-17Cr-12Ni < Fe-19Cr-12Ni < Fe-22Cr-12Ni < Fe-25Cr-7Ni-0.15N.

### 3.3. Derivation of compound Hall–Petch equation

Table 3 shows a summary of the obtained results for the volume fraction of the δ-phase and grain sizes of the γ- and δ-phases in the SUS316L, Fe-17Cr-12Ni, Fe-19Cr-12Ni, Fe-22Cr-12Ni and Fe-25Cr-7Ni-0.15N steel specimens. With those quantitative microstructural features, we can consider the applicability of the compound Hall–Petch relationship (equation (1)), to the estimation of 0.2% yield stress in the steel specimens. First, kγ in the Hall–Petch equation for the single γ-phase in SUS316L steel is obtained as 164 MPa μm−0.5. This kγ is nearly the same as that reported by Kashyap et al. in large γ-phase grains. \( \sigma_{\gamma,\gamma} \) is therefore calculated by applying the Hall–Petch equation, \( \sigma_{\gamma} = \sigma_{\gamma,\gamma} + 164D_{\gamma}^{-1/2} \), to the determination of the experimental yield strength of SUS316L steel shown in table 3. The average \( \sigma_{\gamma,\gamma} \) was deduced as 211 MPa, which is quite similar to that reported by Kashyap and Tangri. Thus, the yield stress of SUS316L can be defined as

\[ \sigma_{y,\gamma} = 211 + 164D_{\gamma}^{-1/2}. \]  

(9)

Supposing that the composite principle of yield strength is suitable for duplex stainless steel, the measured yield stress of steel can be expressed as

\[ \sigma_y = f_y \times \sigma_{y,\gamma} + f_\delta \times \sigma_{y,\delta}. \]  

(10)
Figure 3. OIM images of TD sections of samples, cold-rolled and annealed at 1323 K: (a) γ- and (b) δ-phases of Fe-17Cr-12Ni, (c) γ- and (d) δ-phases of Fe-19Cr-12Ni, (e) γ- and (f) δ-phases of Fe-22Cr-12Ni, and (g) γ- and (h) δ-phases of Fe-25Cr-7Ni-0.15N duplex steel. In particular, an inverse pole figure map was rotated on the RD axis by 90° from the TD plane to the ND plane.

Here, \( f_\gamma \) is the γ-phase fraction, \( f_\delta \) is the δ-phase fraction, \( \sigma_{\gamma,\gamma} \) is the 0.2% yield stress of the γ-phase and \( \sigma_{\gamma,\delta} \) is the 0.2% yield stress of the δ-phase in duplex stainless steel. Therefore \( \sigma_{\gamma,\delta} \) can be obtained by applying equation (11) to the determination of experimental yield stress \( \sigma_\gamma \) as follows:

\[
\sigma_{\gamma,\delta} = \frac{\sigma_\gamma - f_\gamma \times (211 + 164D_\gamma^{-1/2})}{f_\delta}. \tag{11}
\]

Table 3 shows the \( \sigma_{\gamma,\delta} \) results calculated using equation (1). \( k_\delta \) for the δ-phase in the Fe-22Cr-12Ni and Fe-19Cr-12Ni steel specimens can be obtained from the linear plot of \( \sigma_{\gamma,\delta} \) versus \( D_\gamma^{-1/2} \). There is a large difference in \( k \) between these two steel specimens, which is too large even considering the small difference in Cr content. Two possible reasons can be considered for this difference. One is the difference in δ-phase texture formed between the steel specimens at each annealing temperature. A parameter needs to be added into equation (11) to account for the grain size variation and thus the texture changes with annealing temperature. Another reason is the change in intragranular yield stress for the δ-phase in duplex steel with annealing conditions and Cr content.
As shown in Table 3, the ND/(110) texture fraction of the γ-phase and the ND/(111) texture fraction of the δ-phase with 15° tolerance change with annealing temperature. Particularly, both fractions in Fe-22Cr-12Ni steel clearly increase after annealing at 1573 K. To determine the effect of texture intensification on the yield stress of the phase, the average Taylor factor $M$, which is related to the critical resolved shear stress of each phase, is calculated under the uniaxial tensile deformation condition. Table 3 also shows a list of the average Taylor factors $M$ for the γ- and δ-phases in the steel specimens used. Despite the variations in the texture intensities of the γ- and δ-phases with steel type and annealing temperature, the average Taylor factor $M$ remains almost constant at 3.0. Thus the observed intensified texture in duplex stainless steel has negligible effect on yield stress, and the intragranular yield stress $\sigma_y^\gamma$ deduced from the Hall–Petch relationship can be used to evaluate the variation in $k$.

### 3.4. Derivation of intragranular yield stress

Figure 6 shows the microstructure of Fe-22Cr-12Ni steel annealed at 1573 K, after ultra-micro hardness test. Pyramid indenterations are obtained in the γ- and δ-phase grains by distinguishing between the phase contrast and the twins in the γ-phase. By using equations (5)–(8), the average intragranular yield stresses for each phase and under different annealing condition are therefore obtained and are shown in figures 7(a) and (b). Intragranular yield stresses are quite similar between the γ- and δ-phases of the Fe-17Cr-12Ni, Fe-19Cr-12Ni and Fe-22Cr-12Ni steels specimens annealed at 1323 K; however, they are significantly larger than that of Fe-25Cr-7Ni-0.15N steel. The intragranular yield stress of the γ-phase in Fe-19Cr-12Ni steel decreases between 1323 and 1473 K and remains nearly constant even with further increase in annealing temperature to 1573 K. In contrast, stress in Fe-22Cr-12Ni steel monotonically increases with annealing temperature. The behavior between 1323 and 1473 K, but not at 1573 K, is the same in the single phase of SUS316L steel. Figures 3 and 4 show uniform microstructures of the γ-phase after annealing between 1323 and 1473 K. The intragranular yield stress of the δ-phase in Fe-19Cr-12Ni steel was nearly constant, but that in Fe-22Cr-12Ni steel clearly increases with annealing temperature. Using the friction yield...
shows the EPMA concentrations of several steel specimens annealed at different temperatures. The Cr, Ni and Mo contents in the γ- and δ-phases vary for all specimens. In particular, the amounts of Cr and Ni are different between the γ- and δ-phases of Fe-22Cr-12Ni steel. Also, the γ-phase of Fe-25Cr-7Ni-0.15N shows a high amount of nitrogen. Thus, $k_γ$ for the γ-phase of duplex stainless steel is calculated by multiplying the ratio of the elemental components by the initial $k_γ$ of 164 MPa $\mu$m$^{-0.5}$. For example, the hardening coefficient is 193.8 for the γ-phase of Fe-22Cr-12Ni steel annealed at 1323 K; it is 194.5 for the γ-phase of SUS316L steel. Thus, the $k_γ$ value for Fe-22Cr-12Ni steel annealed at 1323 K is 164 MPa $\mu$m$^{-0.5}$ × 193.5/194.5 = 163 MPa $\mu$m$^{-0.5}$.

Table 5 shows the $k_γ$ values calculated using the ratio of the elemental components, as well as the $k_δ$ values obtained using equation (12). There is a variation in $k_γ$; in particular, the $k_γ$ of 239 MPa $\mu$m$^{-0.5}$ for Fe-25Cr-7Ni-0.15N steel is higher than those for other steels. This difference might be due to the solid solution of nitrogen. On the other hand, the $k_δ$ values calculated with equation (12) show the same tendency except for the $k_δ$ of Fe-22Cr-12Ni steel annealed at 1573 K, which is within 909 ~ 985 MPa $\mu$m$^{-0.5}$. The low $k_δ$ of Fe-22Cr-12Ni steel annealed at 1573 K might be related to the higher-than 60% volume fraction of the δ-phase, i.e. 64.4%. In such a case, a different procedure will be needed to calculate hardening coefficient.

The good accordance observed in the results of the steel specimens used indicates that a compound Hall–Petch relationship is valid for different types of duplex stainless steel with large variations in both the volume fraction and grain size of the δ-phase. The average $k_δ$ values are 941 MPa $\mu$m$^{-0.5}$ for Fe-17Cr-12Ni, 975 MPa $\mu$m$^{-0.5}$ for Fe-19Cr-12Ni, 909 MPa $\mu$m$^{-0.5}$ for Fe-22Cr-12Ni and 954 MPa $\mu$m$^{-0.5}$ for Fe-25Cr-7Ni-0.15N. Figure 8 shows a comparison of the calculated and measured stresses, revealing a small difference between them.

3.6. Effect of solid solution strengthening on intragranular yield stress

A variation in $k_δ$ could be anticipated from the unequal coefficients for each element. In fact, the change in $k_δ$ with annealing temperature shows the same tendency as that of the $k_δ$ derived from the hardening coefficient, as shown in table 5.

The intragranular yield stresses of the δ-phase in the Fe-25Cr-7Ni-0.15N and Fe-22Cr-12Ni steel specimens show strong dependences on the volume fraction of the δ-phase. In particular, the increased intragranular yield stresses of these steel specimens are also related to the solid solution strengthening of steel. The amounts of Cr and Ni vary significantly in the δ-phase of the specimens, as shown in table 4. If we consider that Cr and Ni solid solution hardening affects the friction stresses of the δ-phase in the steel specimens, then it is natural to assert that the intragranular yield stresses are high for the δ-phases of the Fe-22Cr-12Ni and Fe-25Cr-7Ni-0.15N steel specimens.
Sieurin et al calculated the intragranular coefficients of Cr, Ni, Mn, Si and N using the Labusch-Nabarro relationship. The Labusch-Nabarro relationship results from the size misfit of Cr, Ni, Mn and Si, which affects intragranular yield stress. The size misfit and linear regression coefficients can be accounted for by an equation based on the Cr, Ni, Mn, Si and N content as follows:

$$
\Delta R_{\text{solid}}^{\delta/\gamma} = K_{\text{LN}} \sum_i \varepsilon_{b_i}^{4/3} \times \sigma_i^{2/3} + 77N_i^{1/2}
$$

$$
= K_{\text{LN}} \times \left( \varepsilon_{C_{\text{Cr}}}^{4/3} \times C_{\text{Cr}}^{2/3} + \varepsilon_{N_{\text{Ni}}}^{4/3} \times C_{\text{Ni}}^{2/3} + \varepsilon_{M_{\text{Mn}}}^{4/3} \times C_{\text{Mn}}^{2/3} + \varepsilon_{S_{\text{Si}}}^{4/3} \times C_{\text{Si}}^{2/3} \right) + A_N N^{1/2}.
$$

Here, $\Delta R_{\text{solid}}^{\delta/\gamma}$ is the yield stress difference associated with the solid solution of Cr, Ni, Mn, Si and N in the $\delta$-phase of all the steel specimens. $K_{\text{LN}}$ is the parameter of the model, $\varepsilon_{b_i}$ is the size misfit parameter, and $C_i, N_i$ pairs describe the chemical composition in atomic fractions. In this case, $K_{\text{LN}} = 18$ GPa, $\varepsilon_{C_{\text{Cr}}} = 0.031$, $\varepsilon_{N_{\text{Ni}}} = 0.016$, $\varepsilon_{M_{\text{Mn}}} = 0.073$, $\varepsilon_{S_{\text{Si}}} = 0.204$. Then $K_{\text{LN}} \sum_i \varepsilon_{b_i}^{4/3} \times \sigma_i^{2/3} = 4.3$ MPa is the difference in the intragranular yield stress in the $\delta$-phase annealed at 1323 K between the Fe-19Cr-12Ni and Fe-17Cr-12Ni steel specimens; this value is similar to the intragranular yield stress of the $\delta$-phase (5 MPa). In the case of the intragranular yield stress of the $\delta$-phase between the Fe-22Cr-12Ni and Fe-19Cr-12Ni steel specimens, this value is comparable to the measured value of 75 MPa, comparable to the measured value of 70 MPa. Thus, equation (13) can determine intragranular yield stress even if the volume fraction of the $\delta$-phase and annealing temperature change significantly in these duplex steel specimens. Figure 9 shows a comparison of the difference between the Fe-19Cr-12Ni, Fe-22Cr-12Ni, Fe-25Cr-7Ni-0.15N and Fe-17Cr-12Ni steel specimens in intragranular yield stress for solid solution as a result of the difference in intragranular yield stress with the size misfit.

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**Table 3. Summary of steel properties; $\sigma_{\gamma/\delta}$ and $\sigma_{\delta/\gamma}$ were deduced using the Hall–Petch relationship.**

| Annealing temp. (K) | Phase | Volume fraction (%) | $\gamma$-grain size (µm) | $\delta$-grain size (µm) | NDf/110 in $\gamma$-phase $\theta = 15^\circ$ | NDf/111 in $\delta$-phase $\theta = 15^\circ$ | Taylor factor (M) | Measured $\sigma_y$ (MPa) | $k_b$ in $\sigma_{\gamma/\delta}$ (MPa μm$^{-0.5}$) | $\sigma_{\gamma/\delta}$ based on equation (11) (MPa μm$^{-0.5}$) | $k_b$ in $\sigma_{\delta/\gamma}$ (MPa μm$^{-0.5}$) |
|---------------------|-------|---------------------|--------------------------|--------------------------|---------------------------------------------|---------------------------------------------|-------------------|-----------------|-------------------------------|-------------------------------------------------|-------------------------------------------------|
| SUS316L             | 1323  | $\gamma$            | 49.8                     | 0.535                    | 226                                         | 164                                         | 209               |
|                     | 1473  | $\gamma$            | 114.9                    | 0.446                    | 246                                         |                                              |                   |
|                     | 1573  | $\gamma$            | 280.5                    | 0.215                    | 209                                         |                                              |                   |
| Fe-17Cr-12Ni        | 1323  | $\gamma$            | 99.8                     | 46.7                     | 0.643                                        | 3.03                                        | 236               | 164             | 886                           | 1501                                            | 157                                             |
|                     | 1473  | $\gamma$            | 92.3                     | 6.34                     | 0.523                                        | 3.04                                        | 321               | 164             | 1501                          | 1501                                            | 157                                             |
|                     | 1573  | $\gamma$            | 71.4                     | 8.34                     | 0.480                                        | 3.04                                        | 350               | 164             | 555                           | 555                                             | 157                                             |
| Fe-19Cr-12Ni        | 1323  | $\gamma$            | 62.2                     | 12.5                     | 0.545                                        | 3.00                                        | 311               | 164             | 555                           | 555                                             | 157                                             |
|                     | 1473  | $\gamma$            | 67.9                     | 3.45                     | 0.453                                        | 3.02                                        | 374               | 164             | 406                           | 406                                             | 157                                             |
|                     | 1573  | $\gamma$            | 54.4                     | 8.25                     | 0.372                                        | 3.03                                        | 414               | 164             | 532                           | 532                                             | 157                                             |
| Fe-22Cr-12Ni        | 1323  | $\delta$            | 32.1                     | 8.25                     | 0.338                                        | 3.03                                        | 414               | 164             | 588                           | 588                                             | 157                                             |
|                     | 1473  | $\delta$            | 45.6                     | 8.25                     | 0.369                                        | 3.03                                        | 393               | 164             | 532                           | 532                                             | 157                                             |
|                     | 1573  | $\delta$            | 64.4                     | 8.25                     | 0.717                                        | 3.06                                        | 532               | 164             | 588                           | 588                                             | 157                                             |
| Fe-25Cr-7Ni-0.15N   | 1323  | $\gamma$            | 42.4                     | 4.79                     | 0.427                                        | 3.02                                        | 572               | 164             | 468                           | 468                                             | 157                                             |

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**Figure 6.** Microstructure of Fe-22Cr-12Ni steel annealed at 1573 K after Vickers ultra-micro hardness test.
parameter. The other steel specimens also show the same tendency as intragranular yield stress. On the other hand, the \( \Delta \sigma_{\text{Fe-22Cr-12Ni}} - \sigma_{\text{Fe-17Cr-12Ni}} \), \( \sigma_{\text{Fe-22Cr-12Ni}} - \sigma_{\text{Fe-19Cr-12Ni}} \) values are negative for all the steel specimens annealed at 1323 K. This means that the intragranular yield stress is lower for Fe-22Cr-12Ni steel than for the Fe-19Cr-12Ni and Fe-17Cr-12Ni steel specimens. Then, the Cr or Mo content of the \( \delta \)-phase of Fe-19Cr-12Ni steel is higher than that of Fe-22Cr-12Ni steel. The finding may be attributed to the presence of trace amounts of the \( \sigma \)-phase. In general, the \( \sigma \)-phase appears at annealing temperatures between 923 and 1223 K. Thus, it could be produced upon heating to 1323 K during the annealing, but it is converted to the \( \delta \)-phase at 1323 K. However, if the \( \sigma \)-phase does not dissolve quickly at 1323 K in small amounts of the \( \delta \)-phase, then the Cr or Mo contents of the \( \delta \)-phase in the Fe-17Cr-12Ni and Fe-19Cr-12Ni steel specimens must be higher than that in Fe-22Cr-12Ni steel. For reference purposes, figure 10 shows a phase diagram of duplex stainless steel calculated with Thermo-calc software. According to the diagram, Fe-19Cr-12Ni should not have a \( \delta \)-phase at 1323 K; however, a \( \delta \)-phase is observed in figure 3(d). Therefore, the \( \gamma / \gamma + \delta \)

\[
\sigma_{\gamma, \delta} = \sigma_{\text{Fe-17Cr-12Ni}, \gamma, \delta} + \Delta \sigma_{\text{solid}}^{\text{Fe-22Cr-12Ni, \delta}}
\]

\[
= \sigma_{\text{Fe-17Cr-12Ni}, \gamma, \delta} + K_{\text{LN}} \sum_j \epsilon_i^{1/3} \times C_{i+j}^{2/3} + 77N_{i}^{1/2}
\]

\[
- K_{\text{LN}} \sum_j \epsilon_i^{1/3} \times C_{i+j}^{2/3} + 77N_{i}^{1/2},
\]

where \( C_i, N_i, N_j \) or \( C_j, N_j \) is the chemical composition of different alloys.

On the other hand, \( \gamma \) intragranular yield stress for duplex stainless steel had almost the same value for all steel specimens except Fe-25Cr-7Ni-0.15N. Thus, it is difficult to develop an expression for \( \gamma \) intragranular yield stress taking into account the chemical composition of steel.
Table 5. Summary of steel properties; $k_γ$ for $γ$-phase and $k_δ$ for $δ$-phase were deduced using coefficients for each element.

| Annealing temp. (K) | $σ_{γ0}$ of intragranular steel (MPa) | $σ_{δ0}$ of intragranular steel (MPa) | $δ$ ferrite (%) | $γ$-grain size ($μm$) | $δ$-grain size ($μm$) | $k_γ$ based on equation (12) (MPa) | $σ_{γ0}$ based on equation (12) (MPa) | $k_δ$ based on equation (12) (MPa) | $k_δ$ based on hardening coefficient (MPa $μm^{-0.5}$) |
|---------------------|--------------------------------------|--------------------------------------|----------------|----------------------|----------------------|-----------------------------------|-----------------------------------|-----------------------------------|------------------------------------------------|
| SUS316L             | 1323                                 | 180                                 | 49.8           | 164                  | 1473                 | 186                              | 114.9                             | 164                              | 1573                             |
| Fe-17Cr-12Ni        | 1323                                 | 213                                 | 215 0.2        | 46.7                 | 164                  | 2.84                             | 152                               | 764                              | 941                              |
| Fe-17Cr-12Ni        | 1473                                 | 218                                 | 220 7.70       | 6.34                 | 164                  | 3.15                             | 166                               | 770                              | 985                              |
| Fe-17Cr-12Ni        | 1573                                 | 170                                 | 226 37.8       | 15.2                 | 164                  | 14.8                             | 167                               | 473                              | 948                              |
| Fe-17Cr-12Ni        | 1323                                 | 210                                 | 350 45.6       | 8.25                 | 166                  | 9.72                             | 162                               | 644                              | 916                              |
| Fe-17Cr-12Ni        | 1473                                 | 210                                 | 350 45.6       | 8.25                 | 166                  | 9.72                             | 162                               | 644                              | 916                              |
| Fe-17Cr-12Ni        | 1573                                 | 255                                 | 380 64.4       | 12.5                 | 170                  | 29.1                             | 170                               | 338                              | 1067                             |
| Fe-22Cr-12Ni        | 1323                                 | 174                                 | 140 32.1       | 3.45                 | 163                  | 3.72                             | 163                               | 611                              | 909                              |
| Fe-22Cr-12Ni        | 1473                                 | 210                                 | 350 45.6       | 8.25                 | 166                  | 9.72                             | 162                               | 644                              | 916                              |
| Fe-22Cr-12Ni        | 1573                                 | 255                                 | 380 64.4       | 12.5                 | 170                  | 29.1                             | 170                               | 338                              | 1067                             |
| Fe-25Cr-7Ni-0.15N    | 1323                                 | 345                                 | 335 57.6       | 4.79                 | 239                  | 8.68                             | 454                               | 954                              |

Figure 8. Comparison of calculated and measured yield stresses.

4. Conclusions

In this study, 0.2% yield stress of duplex stainless steel was evaluated using a compound Hall–Petch equation. The material included 0.2–64.4 wt% $δ$-ferrite that was cold-deformed and annealed. Intragranular yield stress was measured using an ultra-micro hardness tester. Grain size, volume fraction and texture were monitored by EBSD measurement.

1. Melted ingots were homogenized at 1473 K for 1 h, and hot-forged into 20-mm-thick slabs. After solid-solution annealing at 1323 K for 1 h, samples with a $20 \times 20 \ mm^2$ cross section were cut from the annealed slabs and rolled at room temperature. Samples with a $2 \times 21 \ mm^2$ cross section were finally obtained with an accumulated rolling reduction of 90%; they were further annealed for 1 h at 1323, 1473 and 1573 K. The grain sizes and the volume fractions of the $γ$- and $δ$-phases were observed by ESBD measurement. The $δ$-phase was finer than the $γ$-phase, and the $δ$-area fraction changed from 0.2 to 64.4 wt% depending on annealing temperature or chemical composition. The $γ$-grain shape was almost globular. However, the aspect ratio of $δ$-grains was small (0.38) because of the intensive cold rolling.
Figure 10. Phase diagram of duplex stainless steel used to calculate the increase in Cr content using Thermo-calc software.

2. Intragranular yield stress was evaluated from the relationship between the load $P$ and the depth $h$ in the loading area, and also in the unloading area. The $\delta$ intragranular yield stress of Fe-25Cr-7Ni-0.15N steel annealed at 1323 K and Fe-22Cr-12Ni steel annealed at 1573 K was higher than that for other annealed materials.

3. The 0.2% yield stress of duplex stainless steel was successfully evaluated using a compound Hall–Petch equation. In particular, the $k_{\gamma}$ of the $\gamma$-phase of duplex stainless steel matched well the $k_{\gamma}$ of the single phase of SUS316L steel. Changes in $k_{\delta}$ with annealing temperature were almost equivalent to those in the hardening coefficient. The compound Hall–Petch equation also showed the same value as the experimental yield stress, except for the yield stress for Fe-22Cr-12Ni steel annealed at 1573 K.

4. The difference in each intragranular yield stress shows good agreement with the equation based on the linear regression coefficient and size misfit parameter derived from the Labusch-Nabarro relationship.

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