Effect of V, Nb and Ti Addition and Annealing Temperature on Microstructure and Tensile Properties of AISI 301L Stainless Steel

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The effects of V, Nb and Ti additions and the annealing temperatures on the microstructure and tensile properties of AISI 301L stainless steel have been investigated. The grain size of 0.9 μm was obtained by adding 0.5 mass% V with the annealing at 850°C for 30 s, while those of original 301L annealed at 850 and 1 000°C were 2.7 and 9.8 μm, respectively. The grain size decreased as V and Nb increased up to 0.5 and 0.1 mass%, respectively, with the increase in the number density of the precipitate particles. The additions of V over 0.5 mass%, Nb over 0.1 mass% and Ti did not affect the grain size because these additions hardly contributed to the increase in the particle number density. The grain size also decreased with the decrease in the annealing temperature from 1 000 to 850°C. These grain refinements were achieved mainly by the pinning effect of the precipitates and the decrease in the mobility of the grain boundary itself with the decrease in the annealing temperature. The difference of the pinning effect by V, Nb and Ti was explained by the gap of the solubility limit of these elements between the temperatures of the solution treatment and annealing. 0.2% proof stress increased from 400 to 750 N/mm² as the amount of V and Nb increased and as the annealing temperature decreased. These strengthenings were explained mainly by the grain refinement in accordance with the Hall-Petch relationship and the influences of other strengthening mechanisms were estimated as quite small.

KEY WORDS: AISI 301L; austenitic stainless steel; Vanadium; Niobium; Titanium; grain refinement; precipitation.

1. Introduction

The grain refinement of steels is recognized as an effective method for strengthening with small deterioration in ductility and toughness. Therefore, the grain refinement has been studied for a long time, and is still a field being studied widely now. In metastable austenitic stainless steels, it has been known that the fine grains were obtained by strain-induced martensite (α') transformation and the following reversion transforming process from α' to austenite (γ). Takaki et al. have succeeded in obtaining the γ grains of 1 μm or less by the annealing for 600 s at less than 1 000 K (727°C) after strong cold rolling of about 90% reduction in Fe–15.5Cr–10Ni steel. In an industrial perspective, strong cold rolling of 90% reduction would be difficult and the annealing time would be preferable to be short, like under several tens of seconds. In that case, higher annealing temperature would be needed for an enough reverse transformation from α' to γ. However, the annealing at high temperature generally accompanies rapid grain growth. Therefore, understanding of the grain growth and the way of controlling them must be also essential if the industrial production was taken into consideration.

Making use of the pinning effect by fine carbides and nitrides has been known as a technique for suppressing the grain growth. Thus, Vanadium (V), Niobium (Nb) and Titanium (Ti), the free energies of whose carbide and nitride formation are relatively low, have been used in commercial carbon steels. For taking advantage of the pinning effect with nitrides of V, Nb and Ti, steels that contain high Nitrogen (N) are supposed to be suitable as base steels. However, there are still few reports about the effect of these micro alloying elements on the metastable austenitic stainless steels that contain relatively high N like AISI 301L stainless steel.

The aim of this study is to clarify the lower limit of the grain size obtained by using the pinning effect of V, Nb and Ti in high N metastable austenitic stainless steel. For this purpose, the effects of these micro alloying elements and the annealing temperatures on the microstructure and the tensile properties of AISI 301L were investigated.

2. Experimental

The chemical compositions of the specimens are presented in Table 1. Steels based on AISI 301L stainless steel were alloyed with the different contents of V, Nb and Ti. The contents of V, Nb and Ti were ranging from 0.002 to 1.0 mass%, 0.002 to 0.3 mass% and 0.002 to 0.3 mass%, respectively. The cast ingots were mechanically ground to 40 mm in thickness, hot rolled at 1 200°C, annealed and cold-rolled. Afterwards, the solution treatment at 1 150°C...
for 600 s and water quenching for preventing the carbide and nitride precipitation during the cooling, cold-rolling by 67% reduction in thickness at the room temperature of 25°C and annealing at 850, 900 and 1 000°C for 30 s were given in the process illustrated in Fig. 1. Testing specimens were prepared from the annealed sheet of 0.4 mm thickness.

The specimens were mechanically polished with emery papers, buffed with diamond paste, and then electrolytically etched with 35% aqueous Nitric acid solution for microstructure observation. Observation was carried out in the middle of the thickness of the specimens by means of an optical microscope (OM) and a scanning electron microscope (SEM). The average nominal diameter of the grains (only named “the grain size” at the following) of the specimens was determined by using the planimetric method (ASTM E112) with the SEM images. Several specimens were electro polished to thin films and observed with a transmitting electron microscope (TEM). The extraction replicas for observing the precipitate particles were prepared by chemical etching of the specimens covered with a carbon deposition film on their surface. These replicas were observed with the TEM and the kind of the particles was identified from electron diffraction patterns and energy dispersive X-ray spectroscopy analysis. The TEM images of the extraction replica were analyzed with the image analysis software for determining the particle number density per unit area and the average particle diameter (circle equivalent diameter).

The X-ray diffractions using Co-Kα line were used to obtain the volume fraction of α' phase ($V_{α'}$). $V_{α'}$ was estimated with using Direct comparison method\(^7\) by Eqs. (1) and (2). $I_{α'}$ or $I_{γ}$ was the integral intensity of a diffraction line from α' or γ respectively, $v$ was the volume fraction of unit cell, $F$ was the structure factor, $p$ was the multiplicity factor, $θ$ was Bragg angle, and $e^{-2M}$ was the temperature factor. The diffraction peaks of (200) and (211) of α' phase and (200) and (220) of γ phase were used. $V_{α'}$ was estimated separately with using the ratio of $R_{α'}/R_{γ}$ of the sets of α'(200) and γ(200), and α'(211) and γ(220), and the average value of two $V_{α'}$ was taken as the representative value.

\[
V_{α'} = \frac{I_{α'}}{I_{α'} + I_{γ} \left( \frac{R_{γ}}{R_{α'}} \right)} \tag{1}
\]

\[
R = \left( \frac{1}{v} \right) \left[ p \left( \frac{\cos^2 \theta}{\sin^2 \theta \cos \theta} \right) \right] e^{-2M} \tag{2}
\]

JIS 13 B tensile specimens (12.5 mm in width and 50 mm in original gauge length) were prepared along the rolling direction to examine the tensile properties, such as 0.2% proof stress and total elongation. An Instron tensile testing

| Table 1. Chemical compositions of specimens used in this study (in mass%, bal. Fe). |
|----------------|----------------|----------------|----------------|----------------|----------------|
|                | C             | Cr            | Ni            | Si            | Mn            | N             | V             | Nb            | Ti            |
| 301L           | 0.03          | 17.1          | 6.2           | 0.6           | 1.2           | 0.12          | <0.002        | <0.002        | <0.002        |
| 01V            | 0.03          | 17.3          | 6.1           | 0.5           | 1.2           | 0.11          | 0.21          | <0.002        | <0.002        |
| 02V            | 0.03          | 17.4          | 6.1           | 0.5           | 1.2           | 0.11          | 0.31          | <0.002        | <0.002        |
| 03V            | 0.03          | 17.2          | 6.1           | 0.6           | 1.2           | 0.12          | 0.48          | <0.002        | <0.002        |
| 05V            | 0.03          | 17.4          | 6.1           | 0.6           | 1.2           | 0.13          | 0.61          | <0.002        | <0.002        |
| 06V            | 0.03          | 17.4          | 6.2           | 0.6           | 1.2           | 0.12          | 0.71          | <0.002        | <0.002        |
| 07V            | 0.03          | 17.2          | 6.1           | 0.6           | 1.2           | 0.12          | 1.01          | <0.002        | <0.002        |
| 005Nb          | 0.03          | 17.2          | 6.2           | 0.5           | 1.3           | 0.12          | 0.05          | <0.002        | <0.002        |
| 01Nb           | 0.03          | 17.1          | 6.0           | 0.6           | 1.2           | 0.11          | 0.10          | <0.002        | <0.002        |
| 02Nb           | 0.03          | 16.7          | 6.6           | 0.6           | 1.2           | 0.11          | 0.19          | <0.002        | <0.002        |
| 03Nb           | 0.03          | 16.6          | 6.6           | 0.5           | 1.2           | 0.10          | 0.29          | <0.002        | <0.002        |
| 01Ti           | 0.03          | 17.4          | 6.2           | 0.5           | 1.2           | 0.11          | 0.09          | <0.002        | <0.002        |
| 02Ti           | 0.03          | 17.1          | 6.1           | 0.5           | 1.2           | 0.10          | 0.15          | <0.002        | <0.002        |
| 03Ti           | 0.03          | 17.3          | 6.2           | 0.5           | 1.2           | 0.08          | 0.29          | <0.002        | <0.002        |

Fig. 1. Schematic diagram of the process in this study.

Fig. 2. OM images of (a) 301L, (b) 05V, (c) 10V, (d) 01Nb and (e) 03Ti annealed at 1 000°C for 30 s.
machine was used for the tensile tests under the following operating conditions (strain rate = 9.0×10⁻⁴/s and temperature = 25°C).

3. Results

3.1. Effect of V, Nb and Ti Addition on Grain Size and Precipitation

Figure 2 represents the OM images of specimens annealed at 1000°C for 30 s. The grain size decreased drastically with the addition of V and Nb. As shown in Fig. 3, the grain size decreased from about 10 to 3 μm as the amount of V increased from 0.002 to 0.5 mass%, although the grain sizes of the specimens that contained V exceeding 0.5 mass% (06V, 07V and 10V) were nearly equal to that of 05V. The grain size also decreased to 4.5 μm as the amount of Nb increased up to 0.1 mass%, although the grain sizes were not affected by the Nb addition exceeding 0.1 mass%. The grain sizes were not affected by the Ti addition up to 0.3 mass%.

Figure 4 represents the TEM images of the extraction replica from 301L, 05V and 10V. Figure 5 shows the effect of the V amount on the grain size and the number density of the precipitate particles. The precipitate particles were hardly observed in 301L and 01V. According to the result that the grain sizes of 301L and 01V in Fig. 5 were almost the same, the effect of the solute V on the grain size seemed to be very small in our study. The fine particles of about 20 nm were observed in the specimens that contained V from 0.2 to 0.5 mass%, and the number density of these particles increased as the amount of V increased in this range. In the specimens containing V over 0.5 mass% (06V, 07V and 10V), the coarse particles of about 1 μm were observed in...
addition to the fine particles. The size of the fine particles observed in 06V, 07V and 10V was the same as that in 05V. Both of the fine particles and the coarse particles were identified as V(C, N). The particle number densities of 06V, 07V and 10V were nearly equal to that of 05V because those coarse particles were few as a number.

As shown in Fig. 5, the tendency of the particle number density against V content shows the inverse correlation to that of the grain size against V content. Figure 6 shows the relationship between the grain size and the particle number density of the specimens annealed at 1000°C. The grain size decreased as the number density of the particles increased, regardless of the kind of additional elements.

3.2. Effect of Annealing Temperature on Grain Size and Precipitation

As shown in Fig. 7, the grain sizes of 301L and the V added steels decreased with the decrease in the annealing temperature. In addition, regardless of the annealing temperature, the grain sizes of 06V, 07V and 10V were nearly equal to that of 05V. Those tendencies of the grain size against V, Nb and Ti content do not depend on the annealing temperature, although the absolute values of the grain sizes were different.

Figure 8 represents the TEM images of 301L, 05V and 01Nb annealed at 1000 and 850°C. Figure 9 shows the effect of the annealing temperature on the grain size of 301L, 05V and 01Nb. The grain size of each steel decreased with the decrease in the annealing temperature. At the all annealing temperature, the grain size was small in the order of 05V, 01Nb and 301L.
The fine precipitate particles were observed in 301L, 05V and 01Nb and the number of these particles was higher in the steels annealed at 850°C than those annealed at 1000°C, as shown in Fig. 10. The average size of these fine particles was roughly 20 nm and the differences among the steels and the annealing temperatures were small. Figure 11 shows the relationship between the grain size and the particle number density. The grain size decreased as the particle number density increased in the both annealing temperatures. This relationship revealed that the grain size was strongly affected by the pinning effect of the precipitate particles. However, the grain sizes seemed to be different depending on the annealing temperature even if the particle number density was the same, which indicated that the reason of this difference in the grain size could not be explained by only the number density of the particles.

3.3. Tensile Properties

Figure 12 shows that 0.2% proof stress increased with the decrease in the annealing temperature and was high in the order of 05V, 01Nb and 301L under the same annealing temperature. Figure 13 shows that 0.2% proof stress increased from about 400 to 740 N/mm² as the grain size decreased in accordance with the Hall-Petch relationship. 0.2% proof stress of 05V annealed at 850°C, whose grain size was the smallest, was the highest in this study.

Figure 14 shows the relationship between elongation and 0.2% proof stress. The decrease in elongation corresponded to the increase in 0.2% proof stress, regardless of the alloy composition.

4. Discussions

4.1. Mechanism of Grain Refinement

In this study, the grain size decreased as the particle number density increased. The relationship between the grain size and the particle dispersion has been studied by many groups after Zener’s first investigation. Nishizawa et al. have investigated this relationship precisely and obtained Eq. (3), where $D$ is the average size of grains pinned by the particles, $\alpha$ is the coefficient, $d$ is the average size of the particles and $f$ is the volume fraction of the particles.

$$D \approx \alpha \cdot \frac{d}{f^{2/3}}$$

Figure 15 shows the relationship between the grain size $D$ and $df^{2/3}$, which is the variable in Eq. (3). $D$ was measured with the SEM images, $d$ was measured with the TEM images of the extraction replicas and $f$ was estimated with Thermo-calc. The grain size decreased as $df^{2/3}$ decreased, which meant that the grain size decreased as the particle size decreased and the volume fraction of the particles increased. The grain size should have been constant and independent on the annealing temperature, according to Eq. (3), which assumed the equilibrium of the grain growth and the pinning effect by the particles. However, in this study, the grain sizes of the specimens annealed at 1000 and 850°C were not plotted on the same line in Fig. 15. This would indicate that the
grain growth and the pinning effect did not reach the equilibrium because the annealing time of 30 s was considerably short. In other words, the mobility of the grain boundary, which depended on the annealing temperature, would have strongly affected the difference in the grain sizes.

To summarize the discussion above, the grain refinement by the V and Nb addition and the annealing at the lower temperature was achieved by the overlay of the pinning effect by the precipitate particles and the decrease in the mobility of the grain boundary itself.

4.2. Correspondence of Precipitation Behavior and Phase Diagram

As mentioned before, the grain sizes of the specimens that contained V over 0.5 mass% (06V, 07V and 10V) and Nb over 0.1 mass% (02Nb and 03Nb) were nearly equal to those of 05V and 01Nb, respectively. This would be because the addition of both V over 0.5 mass% and Nb over 0.1 mass% hardly contributed to the increase in the particle number density. The grain size was not affected by the addition of Ti up to 0.3 mass% because the particles in the Ti added steels were very few as a number.

The following discussion aims to clarify the reason why the grain size was not affected by the excessive addition of V and Nb and the addition of Ti, and the degree of the pinning effects by V, Nb and Ti were different. Figure 16 shows the phase diagrams calculated by using Thermo-calc. According to Fig. 16(a), the solubility limit of V at the solution treatment temperature of 1150°C is about 0.5 mass% and a part of the added V precipitates as fine V(C, N)s during the annealing carried out at the temperatures lower than that of the solution treatment. Therefore, as long as the amount of added V is not exceeding 0.5 mass%, the difference between the amount of added V and the solubility limit of V at the annealing temperature contributes to the grain refinement as fine V(C, N)s. Hence, the grain size decreased as the amount of added V increased in this range with the increase in the number density of the precipitate particles. However, the added V over 0.5 mass% could not exist as the solid solution but as V(C, N)s and these V(C, N)s would rapidly grow up during the solution treatment. These grown up particles hardly influence the grain size because these particles are quite coarse and few as a number. The behavior of Nb can be explained similarly with Fig. 16(b) as that of V. As shown in Fig. 16(c), Ti could not exist as the solid solution during the solution treatment at all but form TiN, which would also grow up rapidly. Furthermore, it was confirmed that a part of added Ti had already precipitated before hot rolling process. Thus, it is supposed to be difficult to use the pinning effect with Ti for the stainless steels that contain relatively high N like AISI 301L.

In this study, the smallest grain size was obtained by V addition because large quantities of the fine precipitate particles were dispersed. The quantity of the precipitates corresponded to the gap of the solubility limit of the elements between the temperature of the solution treatment and the annealing. Hence, the reason why V was the most effective elements in V, Nb and Ti for the pinning effect can be explained by that the gap of the solubility limit of V between those temperatures was the largest.

4.3. Estimation of the Amount of Strengthenings

The relationship between 0.2% proof stress \( \sigma_{0.2} \) and the grain size \( D \) of 301L annealed at 1000, 900 and 850°C was given by Eq. (4). On the other hand, that relationship of 301L, 05V and 01Nb altogether was given by Eq. (5). The Hall-Petch coefficients in Eqs. (4) and (5) were 485 and 489, respectively and these two coefficients were almost equal. The agreement of these two coefficients indicates that the increment of 0.2% proof stress by the V and Nb addition is explained only by the decrease in the grain size, as well as that of 301L.

\[
\sigma_{0.2} (301L) = 485 \times D^{-1/2} + 247 \quad (4)
\]

\[
\sigma_{0.2} (301L, 05V \text{ and } 01Nb) = 489 \times D^{-1/2} + 253 \quad (5)
\]

According to the XRD measurements, the volume fractions of \( \alpha' \) in 301L, 05V and 01Nb annealed at 850°C and above were very few of less than 10% while those in pre-annealed specimens were above 90%. In addition, the full widths of a half maximum of these annealed specimens were very narrow of less than 0.2° while those of pre-
annealed specimens were about 0.7°. These XRD results indicated that these annealed specimens consisted of mainly diffusionally reversed γ grains. Therefore, the effects of the phase and the residual strains on 0.2% proof stress were supposed to be quite small.

As mentioned above, the additives, such as V and Nb, must have contributed to the increase in 0.2% proof stress by decreasing the grain size. In addition to the effect of the grain refinement, a part of added V and Nb might have contributed to the particle dispersion strengthening and the solid solute strengthening. However, neither of these stiffenings by the addition of V and Nb appeared in the Hall–Petch coefficients in Eqs. (4) and (5). In other words, 0.2% proof stresses of V and Nb added steels would have been higher, if the solute strengthening and the particle dispersion strengthening had contributed to 0.2% proof stresses. For clarifying which strengthening mechanism was effective, the amounts of the stiffenings by the particle dispersion, the grain refinement, and the solid solution were estimated and compared.

First, the amount of the strengthening by the particle dispersion was estimated. It was assumed that the strengthening was not based on the cutting model, but Orowan’s model because the average particle sizes of 20 nm were too large to be cut. The amount of the strengthening by the particle dispersion Δσpd was estimated by Eq. (6), where β was the constant (0.8), G was the module of rigidity (70 GPa), b was the Burgers vector (0.25 nm) and λ was the mean free path of the particles. λ was estimated by Eq. (7), which assumed that the particles dispersed in an orderly manner (square distribution model).

\[
\Delta \sigma_{pd} = \frac{6 \beta G b}{\lambda} \quad \text{................................(6)}
\]

\[
\lambda = 1.25 \left( \frac{\pi}{6f} \right)^{1/2} d \quad \text{................................(7)}
\]

Secondly, the amount of the strengthening by the grain refinement Δσgr was estimated by Eq. (8) with the Hall–Petch coefficient in Eq. (4).

\[
\Delta \sigma_{gr} = 485 \times D^{1/2} \quad \text{................................(8)}
\]

Figure 17 shows the comparison between the estimated amounts of the stiffening by each mechanism. In the all specimens, the amounts of the strengthening by the grain refinement were higher than those by the particle dispersion. However, the amounts of the particle dispersion strengthening of 0.5V and 0.1Nb were estimated too high to be ignored as an error. For example, the amount of the particle dispersion strengthening of 0.5V annealed at 850°C was estimated as nearly 400 N/mm². Therefore, 0.2% proof stress of 0.5V and 0.1Nb would be much higher, if the strengthening by the grain refinement and the particle dispersion were simply added. Although there have been a lot of discussions about the addition rule of the strengthening,13–15) the present results that the strengthening by the particle dispersion did not appear support the idea that the stiffenings by those two mechanisms could not be simply added and were competitive.

Finally, the effect of the solid solution strengthening by the addition of V and Nb was discussed. Irvine et al.16) have reported that the solid solute V contributed to the increase in 0.2% proof stress of Cr–Ni austenitic stainless steels by about 10 MPa per atom%. In our study, the maximum additive amount of V was only 1.1 atom% (1.0 mass%) and a part of the added V existed as V(C, N)s after the annealing. According to Irvine’s estimation, the increase in 0.2% proof stress by the solid solute of V was estimated only as 11 MPa at most. The solid solution strengthening by the addition of Nb, whose maximum additive amount was only 0.18 atom% (0.3 mass%), was also supposed to be quite small.

To summarize the discussion above, the strengthening by the V and Nb additions and the annealing at low temperature was achieved mainly by the grain refinement and the influences of other factors such as the phase, the residual strains, the precipitates dispersion, and the solid solution seemed to be quite small in this study.

5. Conclusions

The effects of V, Nb and Ti additions and the annealing temperatures on the microstructure of AISI 301L metastable austenitic stainless steel was investigated for clarifying the lower limit of the grain size obtained by using the pinning effect. The tensile properties of these steels were also investigated. The conclusions are summarized follows.

(1) The grain size of 0.5 mass% V added steel annealed at 850°C for 30 s was quite small of about 0.9 μm, while those of original 301L annealed at 850 and 1000°C for 30 s were about 2.7 and 9.8 μm, respectively. The grain size decreased as the amount of V and Nb increased up to 0.5 and 0.1 mass%, respectively with the increase in the number density of precipitate particles. The additions of V over 0.5 mass%, Nb over 0.1 mass% and Ti did not affect the grain size because these additions did not contribute to the increase in the particle number density. The grain size also decreased with the decrease in the annealing temperature from 1000 to 850°C.

(2) The grain refinement was explained by the overlay of the pinning effect of the fine precipitate particles and the decrease in the mobility of the grain boundary itself with the decrease in the annealing temperature. The difference of the pinning effect among V, Nb and Ti added steels was explained by the gap of the solubility limit of these elements between the temperatures of the solution treatment and the annealing. V was the most effective element for the pinning
effect because the gap of its solubility limit was the largest.

(3) 0.2% proof stress increased from 400 to 750 N/mm² as the amount of V and Nb increased and as the annealing temperature decreased. These strengthenings were achieved mainly by the grain refinement in accordance with the Hall-Petch relationship. The influences of other strengthening mechanisms in this study were estimated as quite small, compared with that of the grain refinement.

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