Technical Report

Effect of Retained Austenite Phase on Ductility of Martensitic Stainless Steel

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Abstract

In order to improve the processability of martensitic stainless steel, utilization of the transformation induced plasticity (TRIP) effect of the retained austenite phase was examined. A complex structure where about 30% is the austenite phase and the rest is the martensitic phase could be acquired by applying a quenching and partitioning method to martensitic stainless steel. The mechanical properties of the quenching material and partitioning material, and stability of the retained austenite phase, were examined. The balance between the tensile strength and ductility improved greatly because the partitioning process along with the accompanying TRIP effect increases stability of the retained austenite phase.

1. Introduction

Martensitic stainless steel has excellent properties such as high strength and wear resistance and is applied to machine parts and other uses in relatively mild corrosive environments. It cannot be said, however, that it has sufficient toughness and formability for structural members. The properties of martensitic stainless steel can be adjusted by heat treatment, but it is difficult for conventional heat treatment methods to simultaneously achieve both strength and ductility, two contradicting properties.

Research has been actively carried out by utilizing the transformation induced plasticity (TRIP) effect of the retained austenite (γ) phase as a means of improving formability without impairing strength.[1–3] The quenching and partitioning (Q&P) method[4, 5] is available as one means for retaining the γ phase. The Q&P method refers to a heat treatment method consisting of: interrupting quenching at an intermediate temperature between the Ms and the Mf points to partially transform austenite to martensite, reheating to 400 to 500°C, and holding for a short time to diffuse the supersaturated solute carbon (C) in the martensite (M) matrix into the untransformed γ phase. As a result, the Ms point of the untransformed γ phase drops below room temperature, and the γ phase stabilizes and remains at room temperature. However, continuous annealing is required to interrupt the martensitic transformation and concentrate the C in the untransformed γ phase. The volume fraction of the retained γ phase depends on the quenching temperature and the holding temperature and time after reheating. Stable production by the continuous annealing process is not easy. There are few reports in which the Q&P method with these properties and problems was applied to stainless steel to improve the formability of the steel or was investigated for its effects on mechanical properties of the steel.

In this report, we describe the results of the studies we conducted to improve the formability of stainless steel by the Q&P method and to examine the effects of the forming stability of the retained γ phase on the mechanical properties of stainless steel.

2. Concept of Microstructure Control and Target Properties

Figure 1 schematically shows the Q&P method in this report. The γ phase is finely dispersed in the M phase by quenching. The C supersaturated in the M phase is diffused in the γ phase by partitioning. The final outcome is a duplex microstructure of the tempered M phase and the retained γ phase. In this report, we attempted to divide continuous annealing into the quenching treatment and the C partitioning treatment to solve the production stability issue of continuous annealing. We also tried to apply the Q&P method to stainless steel. When assuming a quenched microstructure, we referred to the Schaeffler diagram[6] shown in **Fig. 2**. The Schaeffler diagram shows the relationship between a weld metal microstructure and the effects of alloying elements by converting the effects of alloying elements into the effects of the Ni and Cr. We used the Schaeffler diagram as

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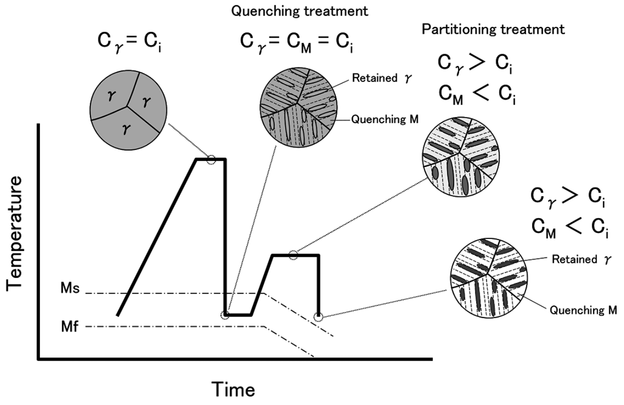

Fig. 1 Schematic of the quenching and partitioning method

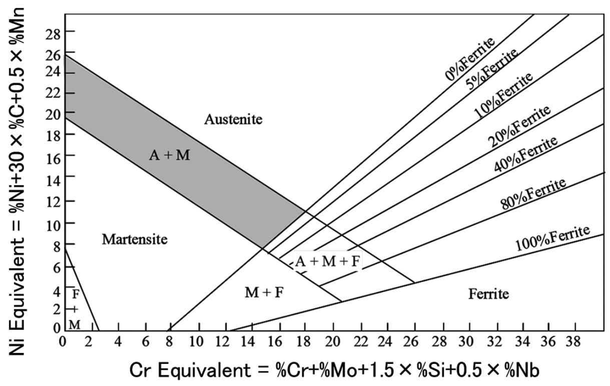

Fig. 2 Schaeffler diagram

a guideline for designing the composition of the subject steel. As the composition of the subject steel, the Ni equivalent and the Cr equivalent were adjusted to a duplex region of the M phase and the γ phase. The Ms point[7] that affects the M transformation amount during quenching was adjusted and set to 100°C or below so that the entire microstructure should not consist of the M phase alone. However, the Schaeffler equation does not consider the N that has a large impact on the Ni equivalent and the Ms point. Therefore, in our alloy design, the Ni equivalent equation[8] obtained by Delong[9] was used to determine the N equivalent that reflects the effect of the N.

We targeted a tensile strength of 1 200 MPa or more and a ductility of 20% or more as the target properties of the subject steel to improve the balance between tensile strength and ductility. These numerical values were set to ensure strength equivalent to that of the martensitic stainless steel SUS410 and ductility equivalent to that of tempered-rolled metastable austenitic stainless steel such as the SUS304. To achieve the target properties, a duplex microstructure containing a combined C+N content of 0.15% or more and consisting of the γ phase in a volume fraction of about 30% and the M phase in the remaining volume fraction was determined as the target microstructure by referring to the past findings.[10]

3. Experimental Methods

Table 1 shows the chemical composition of the subject steel. We designed the composition of the subject steel as follows: The Cr equivalent and the Ni equivalent were adjusted by using Equations (1) and (2), respectively, to achieve an M phase-γ phase duplex microstructure on the Schaeffler diagram. Also, the Ms point was set to about 90°C by using Equation (3) to leave about 30% of the γ phase at room temperature. An ingot was melted in a vacuum melting furnace, held at 1 230°C for 2 h, and hot rolled to a 4 mm thick plate on a reversible hot rolling mill. After that, the plate was again hot rolled, annealed, and cold rolled to a thickness of 1.0 mm. The cold-rolled sheet was annealed at 1 050°C, quenched with a water-cooled copper plate, and partitioned at 500°C.

Table 1 Chemical composition of subject steel and comparative material SUS301

(mass%)

	C	Si	Mn	Ni	Cr	N	Cr eq.	Ni eq.	Ms (°C)
Subject steel	0.09	0.5	2.0	4.2	14.6	0.07	15.7	10.0	87.3
SUS301	0.10	0.7	0.8	6.4	16.5	0.03	–	–	–

$$\mathrm{Cr\,eq} = \mathrm{Cr} + \mathrm{Mo} + 1.5\mathrm{Si} + 0.5\mathrm{Nb} \quad (1)$$

$$\mathrm{Ni\,eq} = \mathrm{Ni} + 30\,(\mathrm{C}+\mathrm{N}) + 0.5\mathrm{Mn} \quad (2)$$

$$\mathrm{Ms}\ (^\circ\mathrm{C}) = \{3\,000\,[0.068-(\mathrm{C}+\mathrm{N})]+50\,(0.47-\mathrm{Si}) +60\,(1.33-\mathrm{Mn})+110\,(8.9-\mathrm{Ni}-\mathrm{Cu}) +75\,(14.6-\mathrm{Cr}-\mathrm{Mo})-32\}\times(5/9) \quad (3)$$

JIS13B test specimens (L direction) as specified in the JIS Z 2201 were prepared and uniaxial tensile tested at a strain rate of $2.1\times10^{-2}\mathrm{s}^{-1}$. The mechanical properties of the subject steel were determined from the stress-strain curves obtained. The strain rate was kept constant because the change in the strain rate would affect the forming heat and vary the TRIP effect.

The moment m was measured with a vibrating sample magnetometer and was substituted together with the sample weight W into Equation (4) to obtain the saturation magnetization σ. The theoretical saturation magnetization σs was calculated by the regression equation of Equation (5) from the alloy composition. The two saturation magnetization values were substituted into Equation (6) to calculate the M phase content V_M of the sample. The M phase content was subtracted from the total composition to calculate the γ phase content.[11, 12]

$$\sigma = (\mathrm{m/W})\ (\mathrm{emu/g}) \quad (4)$$

$$\sigma\mathrm{s} = 214.5-3.12\,(\mathrm{Cr}+\mathrm{Mo}+0.5\mathrm{Ni})-12\mathrm{C}-1.9\mathrm{Mn} -6\mathrm{N}-3\mathrm{P}-7\mathrm{S}-2.6\mathrm{Si}-2.3\mathrm{Cu}\ (\mathrm{emu/g}) \quad (5)$$

$$V_M\,(\%) = (\sigma/\sigma\mathrm{s}) \times 100\ (\%) \quad (6)$$

where the content of each element is given in mass%.

The cross-sectional center of each specimen was mirror polished and observed with a JEOL JSM-7000F field emission scanning microscope at an accelerating voltage of 20 kV and a step size of 0.2 to 0.8 μm. The obtained electron beam backscatter diffraction (EBSD) images were analyzed for crystal orientation with a TSL OIM analysis system.

4. Experimental Results

Figure 3 shows the microstructure controlled by the Q&P method. Plate-like and massive γ grains about 10 μm in size were uniformly dispersed in the M phase. A duplex microstructure containing the retained γ phase in a volume fraction of 27% and close to the target volume fraction of 30% was obtained. **Figure 4** shows the 0.2% proof stress, tensile strength, and ductility behavior of the quenched specimens and the specimens partitioned at 500°C for 0 to 3 600 s. Partitioning increased the 0.2% proof stress by about 500 N/mm² and decreased the tensile strength by about 200 N/mm². The two properties changed little with the elapse of the partitioning time and showed almost constant values. The ductility significantly in-

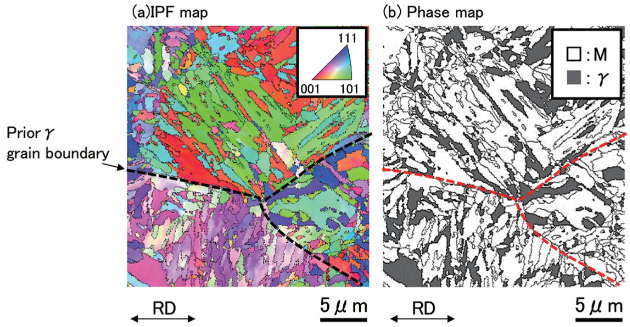

Fig. 3 Metallographic structure controlled by quenching and partitioning method

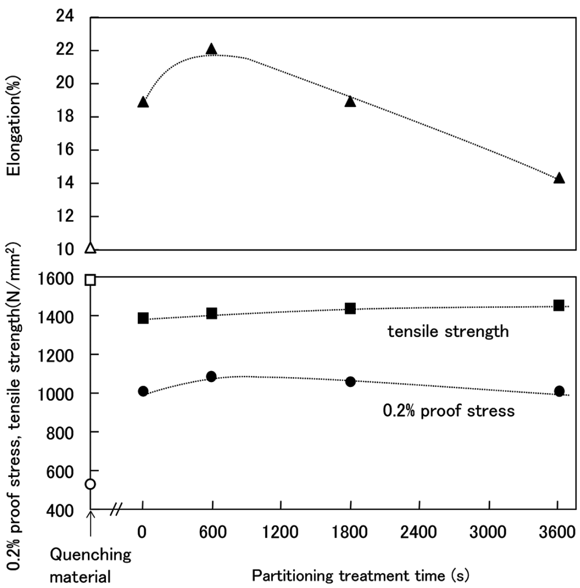

Fig. 4 Effect of partitioning processing time at 500°C on 0.2% proof stress and tensile strength, ductility of the sample

creased with partitioning, reached a maximum at 600 s, then decreased, and became about 14% at 3600 s. The stress-strain curves of the quenched specimens and the 500°C × 600 s partitioned specimens are shown in **Fig. 5**. The mechanical properties of the respective specimens are shown in **Table 2**. The γ volume fractions before the tensile test, at nominal strains of 5% and 15%, and at fracture are also shown in Fig. 5. The quenched specimens greatly work hardened after yielding and exhibited a fracture elongation of about 12%. The partitioned specimens exhibited a higher proof stress than the quenched specimens, slowly work hardened, and exhibited a fracture elongation of about 22%. In the quenched specimens, the γ phase was initially present in a volume fraction of 27%. Near the fracture region, the γ phase all transformed to the strain-induced martensite (α') and became 0% in volume fraction. In the partitioned specimens, the γ phase finally remained in a volume fraction of 11%. This confirmed that the quenched specimens were likely to cause the stress-induced α' transformation, that is, partitioning increased the stability of the metastable retained γ phase. The increase in the 0.2% proof stress by partitioning may be ascribed to the decrease in the mobile dislocation density in the quenched M phase and the improvement in the proof stress of the retained γ phase with the increase in the solute C content. Partitioning decreased the tensile strength, probably because the stress-induced transformed α' content decreased with the increase in the stability of the retained γ phase in addition to the tempering of the quenched M phase.

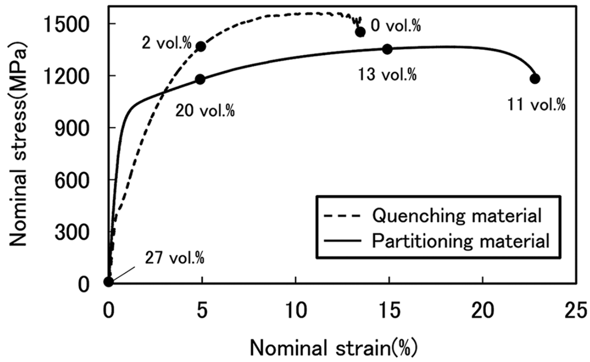

Fig. 5 Stress-strain curve of quenching material and partitioning material at 500°C × 600 seconds

Table 2 Mechanical properties of quenching material and partitioning material

	0.2%PS (N/mm²)	TS (N/mm²)	El (%)	Hardness (Hv30)
Quenching material	423	1532	12	407
Partitioning material	895	1336	22	386

5. Discussion

We confirmed that partitioning improves the balance between tensile strength and ductility. As conventionally known, the diffusion of the C from the M phase to the γ phase is presumed to have increased the stability of the γ phase[13] and changed the action of the TRIP effect. Here, the change in the forming stability of the retained γ by partitioning is quantitatively discussed from the viewpoint of the Md_{30}[14] shown in Equation (7) by using 500°C × 600 s partitioned specimens. The Md_{30} indicates the temperature at which 50% of the γ phase transforms to the stress-induced α' phase when subjected to a true strain of 0.3. The lower the Md_{30}, the more stable the γ phase is and the less likely the γ phase transforms to the stress-induced martensite (α'). Here, because the total elongation of the subject steel is about 20%, we cannot measure the Md_{30} that requires the application of a true strain of 0.3 (equivalent to a nominal strain of 35%). Therefore, we defined the Md_{15} as the temperature at which 50% of the γ phase transforms to the stress-induced α' phase at a true strain of 0.15. Assuming that the difference between the Md_{15} and Md_{30} temperatures was equivalent for both the subject steel and the SUS301, we calculated the Md_{30} and C of the subject steel by introducing the measured Md_{15} (subject steel), Md_{15} (SUS301), and Md_{30} (SUS301) into the following Equation (8). We assume here that the nitrogen (N), an interstitial and austenite stabilizing element like the C, is not diffused by partitioning. This is because when we checked the C and N concentration distributions before and after partitioning with an electron probe microanalyzer (EPMA), we could not recognize the concentration of the N in the γ phase with respect to the C. Because it is unlikely that the N does not diffuse at all, another investigation is in order.

$$Md_{30} = 551 - 462\,(C+N) - 9.2 - 8.1Mn - 13.7Cr - 29\,(Ni+Cu) - 18.2Mo \quad (7)$$

Md_{30} (subject steel) − Md_{15} (subject steel)

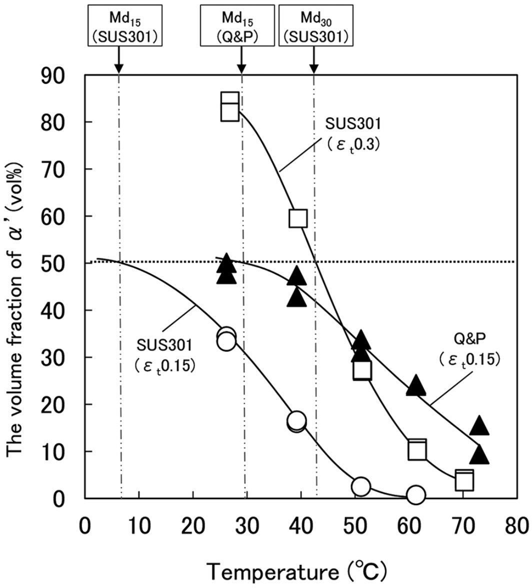

Fig. 6 Effect of tensile test temperature on strain-induced martensite quantity when a true strain of 0.15 and 0.30 was added

$$= Md_{30}(SUS301) - Md_{15}(SUS301) \qquad (8)$$

Figure 6 shows the Md_{15} and Md_{30} of the subject steel and the SUS301 obtained from the tensile test and the stress-induced α' content at various temperatures. The Md_{15} and Md_{30} of the SUS301 were 7°C and 42°C, respectively. The Md_{15} of the subject steel was 30°C. The Md_{30} and C content of the subject steel calculated by introducing these values into Equations (7) and (8) were 63°C and 0.22%, respectively. Assuming that the added C (0.09%) was uniformly dissolved in solid solution in the quenched specimens, partitioning is presumed to have diffused 0.13%C from the M phase into the γ phase, increased the C concentration in the γ phase to 0.22%, and decreased the Md_{30} of the γ phase from about 125°C to 63°C. Because the Md_{30} of the subject steel after partitioning is relatively close to that of the SUS301, it can be said that partitioning changed the microstructure of the subject steel to a duplex microstructure in which the 27% metastable γ phase with stability close to that of the γ phase in the SUS301 was uniformly dispersed in the tempered M phase. These results suggest that as partitioning changed the stability of the retained γ phase to close to that of the γ phase in the SUS301, the TRIP effect worked to improve the ductility.

6. Conclusions

We studied the effect of the retained γ phase on the strength-ductility balance of martensitic stainless steel. We utilized the Q&P method as a means for obtaining the retained γ phase. We also attempted to divide the quenching and partitioning treatments conventionally performed in the continuous annealing process. As a result, we obtained the following findings:

(1) When the Ms point was set to 90°C, the quenched microstructure changed to a duplex microstructure composed of the γ phase in a volume fraction of about 30% and the M phase in the remaining volume fraction.

(2) The quenched specimens showed a tensile strength of about 1 500 MPa and a ductility of 12%. The 500°C × 600 s partitioned specimens showed a ductility of 22%, a tensile strength of about 1 400 MPa, and an improved strength-ductility balance.

(3) We estimated that the 500°C × 600 s partitioning decreased the Md_{30} to about 65°C and diffused 0.13% of the C from the M phase to the γ phase. Since the Md_{30} of the retained γ phase after partitioning was close to that of the retained γ phase in the SUS301, a metastable austenitic stainless steel with excellent ductility, we inferred that the improvement in the ductility by partitioning was due to the TRIP effect of the retained γ phase.

(4) The quenching treatment and the partitioning treatment can be separated. When the steel is processed through the respective steps under appropriate conditions, its production stability is expected to improve.

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