Effect of nitrogen content on temperature dependence of grain refinement strengthening in austenitic stainless steel

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Since type 316L austenitic stainless steel shows great ductility and elongation even at low temperature, it is often used in the cryogenic environment as structural materials such as liquid hydrogen's storage or conducting magnets in nuclear fusion reactor. In order to enhance the strength of type 316L steel, the grain refinement has been considered. It is well known that the yield stress of polycrystalline metallic materials follows the Hall-Petch relationship ($\sigma_y=\sigma_0+k_y d^{-1/2}$), where σ_y is the yield stress, σ_0 is the friction stress, k_y is the Hall-Petch coefficient, and d is the mean grain size. In our previous research, we found that nitrogen addition increases k_y of austenitic steel at ambient temperature. In this study, we focused on the temperature dependence of Hall-Petch coefficient, k_y, to utilize the grain refinement strengthening at low temperature in austenitic stainless steels containing nitrogen, and then the effect of nitrogen content on the k_y was discussed. As a result, not only the σ_0 is increased by the stronger Cottrell-atmosphere which enhanced by the solid solution nitrogen atoms but also the k_y increased with the addition of nitrogen at a temperature range of 293–77 K, and the increase in grain refinement strengthening caused by nitrogen was greatly enhanced at low-temperature. This result suggests that the combined use of nitrogen addition and grain refining was extremely effective for strengthening austenitic steel at low temperature.

Keywords: *Hall-Petch relationship, Nitrogen addition, austenite stainless steel, Grain boundary segregation*

1. Introduction

Since SUS316L (Fe-18Cr-12Ni-2Mo steel) austenitic stainless steel shows great ductility and elongation at extreme low-temperature, it is often used in the cryogenic environment as structural materials such as liquid hydrogen's storage or conducting magnets in nuclear fusion reactor. In order to enhance the strength of SUS316L steel at ambient temperature and cryogenic temperature, the grain refinement is effective. It is well known that the yield stress of polycrystalline metallic materials follows the Hall-Petch relationship[1-2] ($\sigma_y = \sigma_0 + k_y{*}d^{-1/2}$), which σ_y is the yield stress, σ_0 is the friction stress, k_y is the Hall-Petch coefficient, and d is the mean grain size. The k_y represents the hardening effect of grain refinement strengthening and is an important parameter to evaluate the yield stress of polycrystalline metals. With a higher value of k_y, a higher yield stress can be predicted even without a grain refinement. In our previous research, we found that nitrogen addition increases k_y of austenitic steel at ambient temperature. In this study, we focused on the temperature and nitrogen concentration dependence of Hall-Petch coefficient, k_y, to utilize grain refinement strengthening in high-nitrogen austenitic stainless steel.

2. Experiment

Conventional SUS316L steel was subjected to solution nitriding treatment at 1473 K for 20 h in various partial pressure of N_2 gas atmosphere (0.45N, 0.35N, 0.16N steel). The chemical composition of the Base steel and 0.16N steel are listed in Table 1, the composition except nitrogen of the 0.45N and 0.35N steels are almost the same with those of 0.16N steel. In addition, SUS316L was solution-treated at 1473 K for 20 h in an Ar gas atmosphere (Base steel). To obtain the specimens with different grain size, these specimens were subjected to 66.7% cold rolling, and the recrystallization annealing was performed in austenite single-phase region at 1473 K for 60–3600 s, followed by water cooling (**Fig.1**). Tensile testing was performed at an initial strain rate of 10^{-3} s^{-1} for plate test pieces at room temperature and cryogenic temperature in liquid nitrogen (77 K). The grain boundary was calculated from the optical microscope image by using quadrature method.

Table.1 Chemical composition of alloys (mass%)

	N	**Cr**	**Ni**	**C**	**Si**
Base	**0.024**	**17.42**	**12.09**	0.007	0.69
0.16N	**0.162**	**18.00**	**12.00**	0.0015	0.71
	Mn	Mo	P	S	Fe
Base	0.93	2.06	0.036	0.002	Bal.
0.16N	1.00	2.01	<0.01	0.0003	Bal.

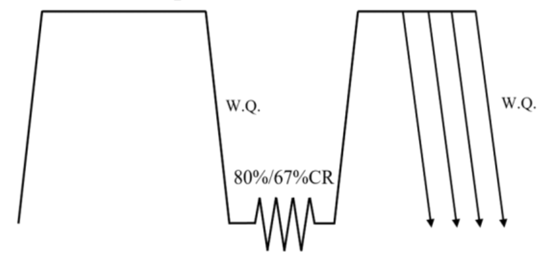

Fig.1 Heat treatment diagram

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3. Results

Solution nitriding treatment is one of the methods to add nitrogen atoms to the steel; the specimen is heated in N_2 atmosphere. It is also possible to control the N content of the specimens by mixing the Ar gas with N_2 gas to change the partial pressure of the N_2 gas. **Figure 2** shows the relationship between the square root of N_2 gas partial pressure and the nitrogen contents after the solution nitriding treatment, shows a linear relationship, which obeys the Sievert's law (Equation 1).

$$S = 1.36 * P_{N2}^{1/2} \ldots (1)$$

Fig.2 Relationship between N_2 gas partial pressure while nitriding and N contents

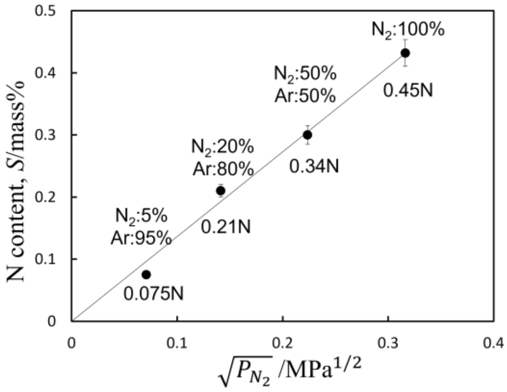


Figure 3 shows the Hall-Petch relationship of 0.45N, 0.35N, 0.16N steels and the base steel at ambient temperature. The yield strength increases with the grain refinement and the amount of nitrogen. The σ_0 of those specimens, which is the intercept of Hall-Petch lines, is enhanced with the increasing of nitrogen addition. The value of k_y, which is the slope of these lines and calculated with the least-square method, is 640, 537, 466, and 288 MPa·μm$^{-1/2}$, respectively. The increase of k_y would be explained by the enhancement of critical grain boundary shear strength. $^{3)}$

Fig.3 Hall-Petch relationship at RT in SUS316L with various N contents.

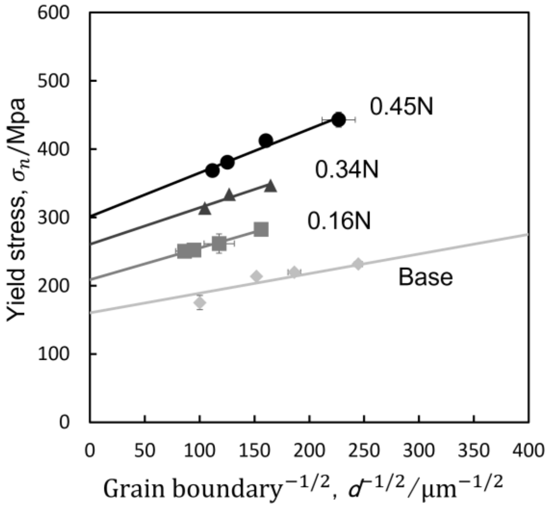


Figure 4 shows the Hall-Petch relationship of 0.45N, 0.35N, 0.16N steels and the base steel at 77K. The yield strength increases with the grain refinement and the amount of nitrogen, which shows the same tendency with the situation of room temperature. The value of k_y at 77K is 1630, 1550, 1310, and 690 MPa·μm$^{-1/2}$, which all shows a higher amount than that at room temperature.

Fig.4 Hall-Petch relationship at RT in SUS316L with various N contents.

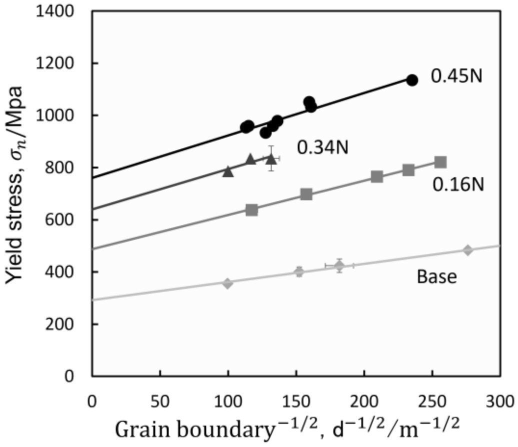
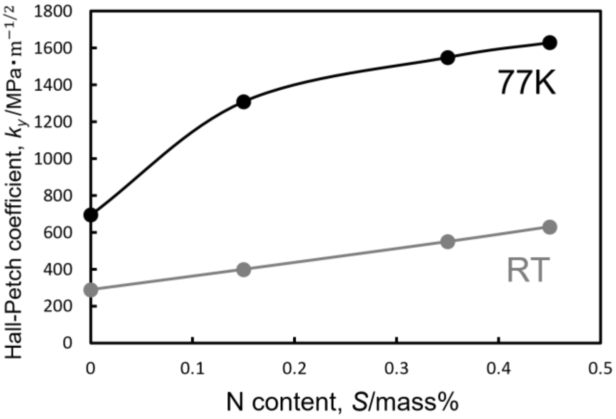


Figure 5 shows the changes in the k_y as a function of nitrogen content at room temperature and 77 K. At room temperature, the k_y monotonically increases with increasing nitrogen content. On the other hand, at 77 K, the increment is more significant than that at room temperature. The k_y is defined as the followed equation, and as a result of considering the parameters which may affect the k_y, the increment of k_y at 77 K would be explained by the improvement of critical grain boundary shear strength (τ_{cr}) , which due to the grain boundary segregation of nitrogen atoms, because the parameters except τ_{cr} hardly change depending on the nitrogen content and temperature.

$$k_y = M * (2Gb\tau_{cr} / \pi k)^{1/2} \ldots (2)$$

Fig.5 Contrast of Hall-Petch coefficient between 77K & RT with the influence of varied N contents.



4 Conclusion

This study aims to estimate the effect of nitrogen addition on the Hall-Petch coefficient at room temperature and 77K. Based on experimental observations and analyses, the main conclusions have been drawn as follows.

(1) At room temperature, not only the σ_0 but also the k_y

has been improved by nitrogen addition, which can be consider as the influence of grain boundary segregation of nitrogen atoms.

(2) At 77K, the improvement of σ_0 and k_y, shows at room temperature, becomes greater. It indicates that the effect of grain refinement with N addition shows the temperature dependence.

(3) The present results suggest that the combine of nitrogen addition and grain refinement to strengthen the austenitic steel at cryogenic temperature is effective.

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