Transactions of the Korean Nuclear Society Spring Meeting
Jeju, Korea, May 12-13, 2016

Ordering Reaction and Its Effect on Microstructure Variation in 316L Stainless Steel

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1. Introduction

The short range ordering (SRO) reaction in 316 stainless steel has been investigated by a resistivity change at 400-500^{o}C [1]. It is reported that the SRO reaction occurs in a Ni-Cr-Fe (Ni-base alloys) and Fe-Cr-Ni (stainless steel) alloys. This is important since the SRO reaction is an avoidable process during reaction environment.

It is reported that the SRO reaction causes a lattice contraction [2] and a dimensional change of structure [3], and produces an additional stress [4], and provides a driving force for a primary water stress corrosion cracking (PWSCC) in Alloy 600 [4, 5].

In this study, the ordering reaction in 316L is systematically studied by a differential scanning calorimeter (DSC), and the activation energy for the ordering reaction is determined, and the effect of ordering treatment at 475℃ on microstructure is investigated.

2. Experimental

The chemical composition of 316L stainless steel is shown in Table 1.

The specific heat (Cp) of 316L is determined using a water quenched (WQ) from 1100℃, a furnace cooled (FC), and an ordering treated 475℃ for 5,500hrs after WQ. The cold work is applied by cold rolling by 10, 20, and 40% in thickness. The as-received, 10, 20, and 40% cold rolled specimens are ordering treated at 475℃ for 24 hrs in order to investigate the microstructure variation by the ordering treatment.

The WQ specimens are differently scanned by DSC, as shown in table 2. The activation energy for the ordering reaction is calculated as follows;

$$\ln ((\alpha_2 T_1^2/\alpha_1 T_2^2) = Q/R(1/T_1-1/T_2) \qquad (1)$$

where T_1 and T_2 are peak temperatures at scan rate α_1 and α_2 and R is gas constant.

The microstructure variation before and after ordering treatment at 475℃ for 24hrs is examined by electron back scattered diffraction (EBSD). The specimens are prepared by grinding and electro-polishing in 10% perchloric acid + methanol solution.

Table 1. Chemical composition of SS316L (wt. %).

elements	Cr	Ni	Mo	Mn	Si	C
Composition [%]	17.29	12.19	2.06	1.19	0.69	0.008
Spec.[%]	16.0~ 18.0	12.0~ 15.0	2.0~ 3.0	2.0	1.0	0.03

Table 2. Peak temperature variation with heating rate in water quenched 316L.

Material condition	Heating Rate (α, ℃/min)	Peak Temperature (Tp, ℃)	1/T [K^{-1}]	α/Tp2
water quenched 316L	5	519.7	0.00126	7.954E-06
	10	540	0.00123	1.512E-05
	20	548.8	0.00122	2.960E-05
	40	567.3	0.00119	5.663E-05

3. Results and Discussions

The microstructure and grain orientation of as-received 316L is shown in Fig. 1.The red line denotes a sigma 3 boundary, this is generally twin boundaries.

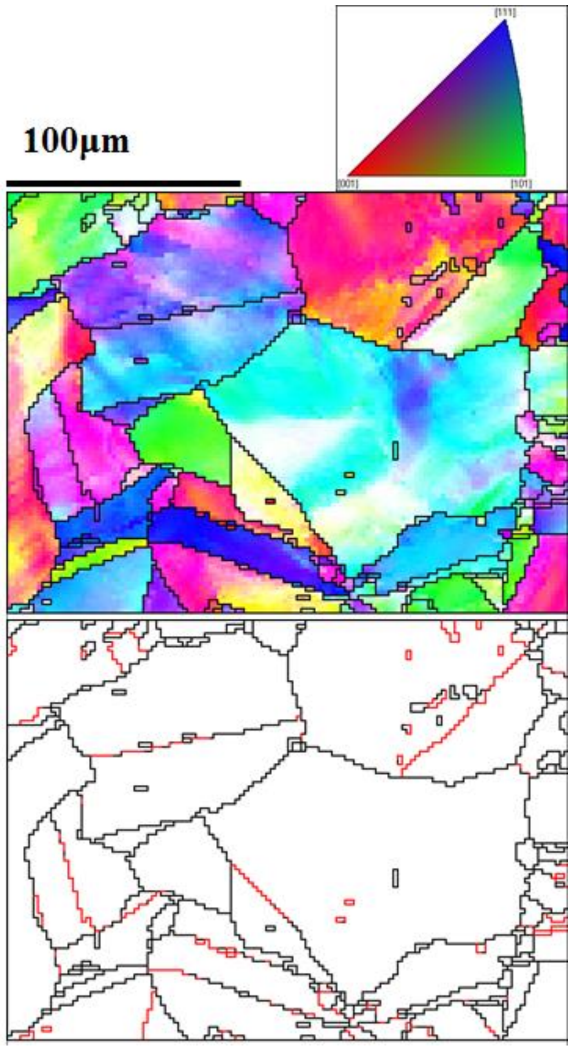

Fig. 1. The microstructure and grain orientation map in as-received 316L.

The Cp variations in 316L are shown in Fig. 2. The exothermic reaction appears at 500-550^{o}C in WQ. The amount of released energy (FC-WQ) is calculated as 3.9J/g in WQ. The transition occurs in FC specimen at 470-520^{o}C. The endothermic reaction occurs in the ordering treated specimen. The amount of the absorbed energy (order-WQ) is 3.8 J/g. The exothermic and endothermic reactions are due to the formation of SRO and the disordering of SRO respectively. This fact is consistent with the previous investigation in Ni-Cr-Fe alloys [6, 7].

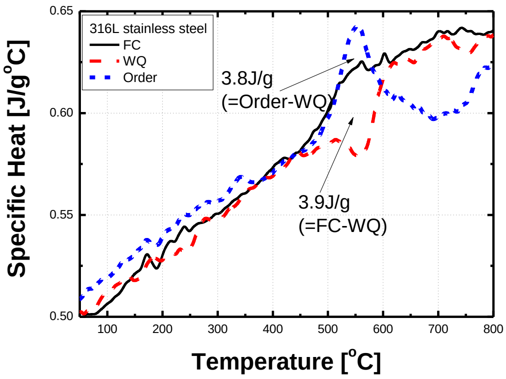

Fig. 2. The specific heat (Cp) variation with treatment conditions, in furnace cooled (FC), water quenched (red dash line), and ordering treated (blue dot line) SS316L.

The DSC results are shown in table. 2. Fig. 3 shows the calculated result of the activation energy for the ordering reaction in WQ 316L, Q ordering in 316L = 234 kJ/mol (2.42 eV/atom).. This seems to be a reasonable value since the ordering reaction is controlled by a substitutional diffusion. However, this is smaller than that of Q = 309 kJ/mol reported by Stanley et al. [1]. It seems that this difference may come from material and experimental procedure. The WQ 316L is used once in DSC scanning, whereas the 316 is used and the resistivity measurement is applied repeatedly at 400-520^{o}C by Stanley et al.

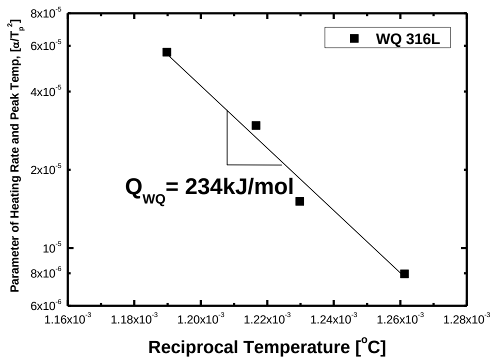

Fig. 3. The activation energy for ordering in WQ 316L.

The effect of cold rolling on Cp variation is plotted in Fig. 4. The released energy increases with amount of cold rolling. The released energy is calculated as 3.5, 5.5, and 8.8 J/g in 10, 20, 40% cold rolled specimen, respectively. This can be explained by the fact that the number of disordering bond increases with the amount of cold rolling due to the disordering by cold rolling. The other important fact is that the temperature range of the exothermic reaction is shifted by cold rolling from 500-550^{o}C to 200-520^{o}C. This means that the cold rolling lowers the ordering temperature and changes the kinetics of ordering significantly. Again, this means the ordering takes place simultaneously during deformation at 200-600^{o}C.

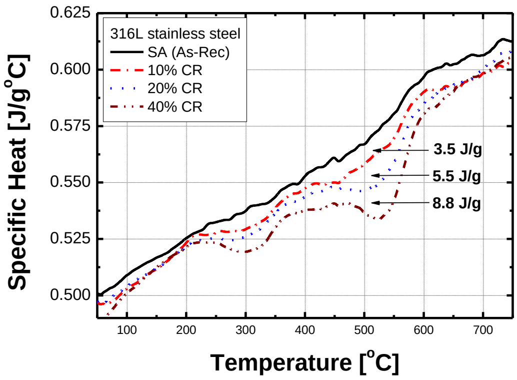

Fig. 4. Comparisons of Cp variation with amount of cold rolling in 316L.

The microstructure and grain orientation map in 20% cold rolled 316L and ordering treated 316L are shown in Fig. 5 and 6, respectively. The 20% cold rolled 316L shows a refined grain structure (Fig. 5), compared with as-received 316L shown in Fig. 1. The ordering treatment at 475℃ for 24hrs causes a grain growth unexpectedly, as shown in Fig. 6.

Although all results are not shown, the effect of the ordering treatment appeared similarly in as-received and 10% cold rolled 316L. The grain structure does not change before and after cold rolling. In addition, the ordering treatment causes twinning mainly.

However, the grain structure changes abruptly before and after 20% and 40% cold rolling. A recrystallization is occurred by cold rolling. This is an unexpected result, since the 20% and 40% cold work is applied only. Furthermore, the ordering treatment causes grain growth after ordering treatment at 475℃-24hrs. It is thought that the ordering temperature is too low to induce grain growth in 316L.

It is understood that the ordering reaction occur simultaneously at 200-600℃ region when deformation is applied (Fig. 4). This means that the lattice contraction due to the ordering reaction occurs during cold rolling, if the 316L is heated at above 200℃ during rolling. This phenomenon is reported in the section of self-induced strain [8]. The self-induced strain in 316L is thought to be the lattice contraction due to the ordering reaction during the ordering treatment at 475℃ -24hrs.

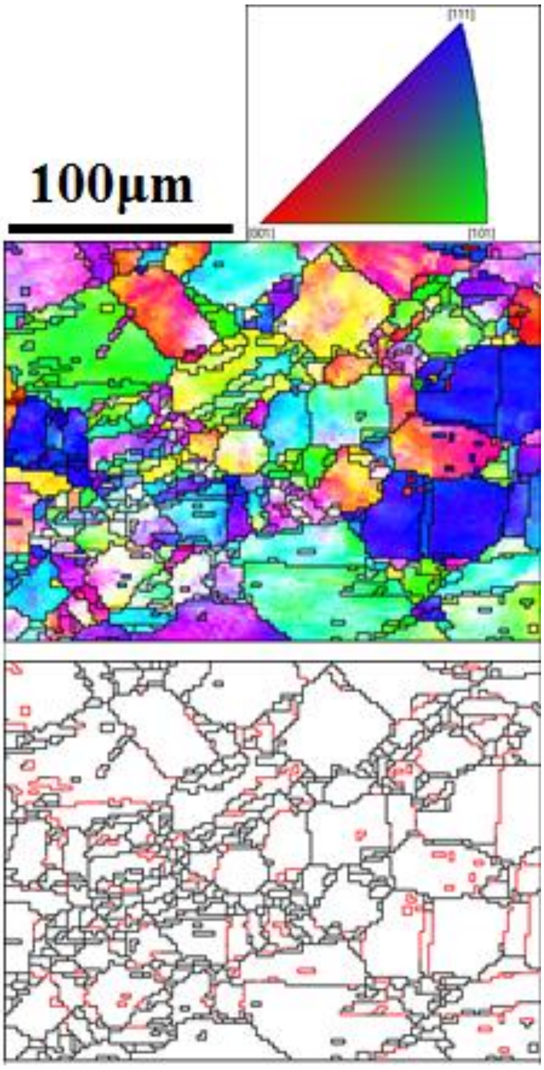

Fig. 5. The microstructure and grain orientation map in 20% cold rolled 316L before ordering treatment.

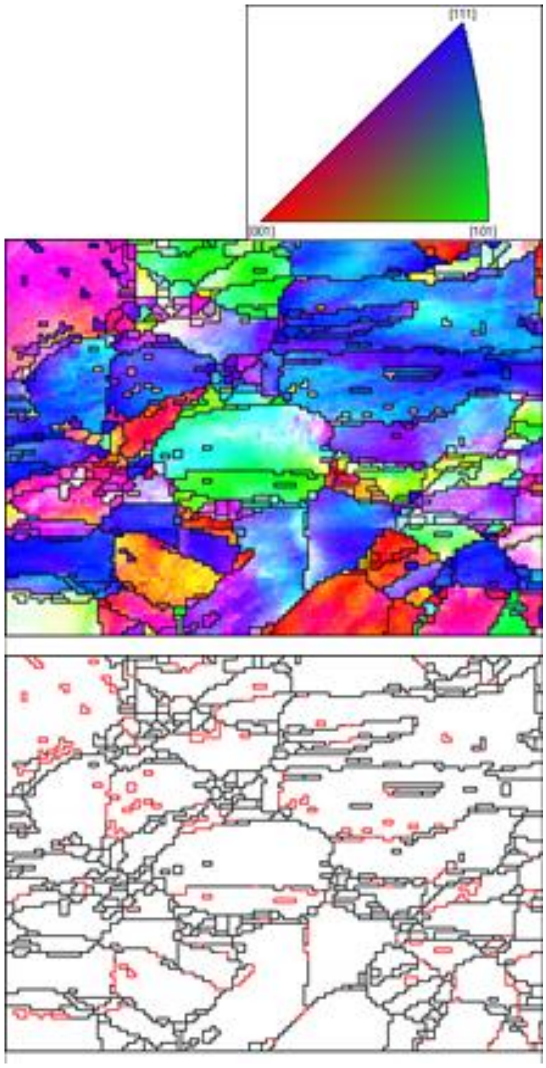

Fig. 6. The microstructure and grain orientation map in 20% cold rolled 316L after ordering treatment (475℃ - 24Hrs).

4. Conclusions

1. The ordering reaction in WQ 316L occurs at 450-550℃, this seems to be formation of sigma phase (FeCr).
2. The activation energy for the ordering reaction in WQ 316L is Q = 234 kJ/mol (2.42 eV/atom).
3. The cold work lowers the ordering temperature from 450-550 ℃ to 200-600 ℃ region, this is due to the driving force for the ordering reaction accumulated during cold rolling.
4. The strain induced ordering occurs at 200-600 ℃ region simultaneously when deformation is applied.
5. The ordering treatment at 475℃ causes twinning in as-received and 10% cold rolled 316L and grain growth in 20% and 40% cold rolled materials.

Acknowledgements

This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIP).

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