

Intergranular Corrosion Mechanism of Slightly-sensitized and UNSM-treated 316L Stainless Steel

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316L stainless steels have been widely used in many engineering fields, because of their high corrosion resistance and good mechanical properties. However, welding or aging treatment may induce intergranular corrosion and stress corrosion cracking etc. Since these types of corrosion are closely related to the formation of chromium carbide in grain boundaries, the alloys are controlled by methods such as the lowering of carbon content, solution heat treatment. This work focused on the intergranular corrosion mechanism of slightly-sensitized and Ultrasonic Nano-crystal Surface Modification (UNSM)-treated 316L stainless steel. Samples were sensitized for 1, 5, and 48 hours at 650 °C in N₂ gas atmosphere. Subsequently UNSM treatments were carried out on the surface of the samples. The results were discussed on the basis of the sensitization by chromium carbide and carbon segregation, the residual stress and grain refinement. Even though chromium carbide was not precipitated, the intergranular corrosion rate of 316L stainless steel was drastically increased with aging time, and it was confirmed that the increased intergranular corrosion rate of slightly-sensitized (not carbide formed) 316L stainless steel was due to the carbon segregation along the grain boundaries. However, UNSM treatment improved the intergranular corrosion resistance of aged stainless steels, and its improvement was due to the reduction of carbon segregation and the grain refinement of the outer surface, including the introduction of compressive residual stress.

Keywords : 316L stainless steel, intergranular corrosion, sensitization, UNSM, carbon segregation, residual stress

1. Introduction

Nuclear power plants have used austenitic stainless steels extensively for heat exchanger tubing, reactor internals, and valves, since these steels have good mechanical properties including strength, toughness, and high corrosion resistance^{1,2)}. However, because of the exposure to high temperature and high pressure, the internals of the nuclear power plants have experienced stress corrosion cracking under thermal and external stress. In particular, intergranular and transgranular stress corrosion crackings at the weldments have occurred mainly by chromium carbide precipitation and the depletion of chromium³⁾.

From the thermodynamical point of view of chromium carbide precipitation, chromium carbide is very stable under 850 °C, the chromium diffusion rate is very fast to form the carbide over 500 °C and then the stainless steel

can be sensitized in this temperature range. Therefore, grain boundaries result in chromium carbide precipitation. If the sensitized stainless steel exposed to corrosive environments, the chromium depletion zone will preferentially corrode⁴⁾. From the kinetics point of view of the time-temperature-precipitation curve, carbide can precipitate in the range of 500 ~ 800 °C depending upon the sensitization time and the alloying system⁵⁾.

In order to mitigate the stress corrosion cracking of weldments, there are many applicable methods, which include substitution with high corrosion resistant materials, modification of water chemistry, and reduction of residual stress. Among these remedies, the reduction of residual stress from tensile stress to compressive stress can be obtained by surface treatment. Applicable and studied techniques to induce compressive residual stress on the weldments can be summarized as shot peening⁶⁻⁸⁾, laser shock peening^{9,10)}, water jet peening¹¹⁾, ultrasonic peening¹²⁾, and UNSM^{13,14)}. In a typical UNSM technique, a tungsten car-

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Table 1. Chemical composition of commercial 316L stainless steel (wt%)

Alloy	Cr	Mo	Ni	C	Mn	Si	P	S	Fe
316L	16.69	1.99	10.19	0.01	1.19	0.59	0.04	0.01	Bal

bide (WC) tip is attached to an ultrasonic horn, which strikes the specimen surface up to 20,000 or more times per second, with 1,000 to 10,000 shots per square millimeter in a very short time. These strikes, which can be described as micro cold forging, bring severe plastic deformation to surface layers, and thus induce nano-crystalline structure¹³. This is very similar to the ultrasonic technique mentioned by Mukhanov¹⁵, who used a magneto-strictive transducer instead of a piezo-transducer to provide ultrasonic vibratory energy. Compared with other mechanical surface nano-crystal treatments, UNSM produces the best hardness, and good surface roughness^{13,14}. The UNSM effects and their anticipated benefits are that the uniformed micro dimple pattern on the surface and nano-crystalline structure induce compressive residual stress in the subsurface layer. This change has the potential to simultaneously improve the static and fatigue strength, as well as the surface hardness and surface roughness of the work piece^{13,14,16}.

The nano-crystalline surface could increase the density of diffusion paths available for alloying elements to migrate and rapidly form a protective passive film^{17,18}. In addition, residual stress in the surface layer affects the critical current density and the current density for passivation which are closely related to the formation and retention of the passivation layer¹⁹. The high mechanical energy imparted by UNSM to the steel surface may also induce the diffusion of the alloying elements^{13,14,18} and therefore can affect the intergranular corrosion resistance of the stainless steels. We already reported that the intergranular corrosion properties by aging treatment was closely related to the formation of chromium carbides and carbon segregation, and UNSM treatment reduced the residual stress and thus improved the intergranular corrosion

resistance²⁰, but we couldn't propose the mechanism in detail between its resistance and carbon segregation.

This work focused on the effects of sensitization and UNSM on the microstructure and intergranular corrosion resistance of 316L stainless steel and elucidated the importance of carbon segregation in grain boundaries on the intergranular corrosion.

2. Experimental Procedures

2.1 Materials and UNSM treatment

Table 1 gives the composition of the material investigated in this study, which is AISI 316L austenitic stainless steel. Aging treatments were performed at 650 °C for 1, 5, and 48 hours in N₂ protective atmosphere and then water quenched. Table 2 lists the conditions of the annealed and aged samples that were treated by UNSM.

2.2 Intergranular corrosion tests

The intergranular corrosion rate was determined by Huey test (ASTM A262 Practice C)²¹. The weight loss per unit area was measured after each of five 48-hour boils in 65 % nitric acid. The intergranular corrosion rate was calculated as the average corrosion rate of five boils in millimeters per year (mmpy). In order to differentiate the effect of UNSM treatment, we used the immersion time of 9 or 3-hours, besides of 48 hours.

The degree of sensitization (DOS) was determined by double loop electrochemical potentiokinetic reactivation test (DL-EPR). The test solution was 1M H₂SO₄ + 0.005M KSCN⁴, and the solution temperature of 30 ± 1 °C, and scan rate of 0.833 mV s⁻¹ were used.

2.3 Microstructure analysis

Microstructure was studied by optical microscope (OM) using Zeiss Axiotech 100HD, scanning electron microscopy (SEM) at 20kV using Tescan Vega II LMU, EPMA using Oxford EPMA-1600, and EBSD with step size of 1µm at 30kV using Oxford Lyra 3 XMH. The etching solution was aqua regia (10mL of HNO₃ + 30mL of HCl).

2.4 Residual stress measurement

The surface residual stress was evaluated using an X-ray diffraction method employing a Cu target operated at 40 kV and 30 mA using Rigaku D/MAX RAPID-S.

Table 2. Condition of UNSM treatment¹³

Alloy	316L
Amplitude	30 µm
Static load	10 N
Pitch	0.07 mm
Speed	1000 mm/min
Tip diameter	2.38 mm (WC)

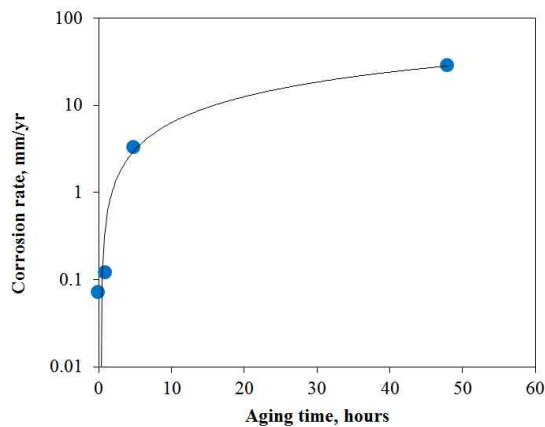
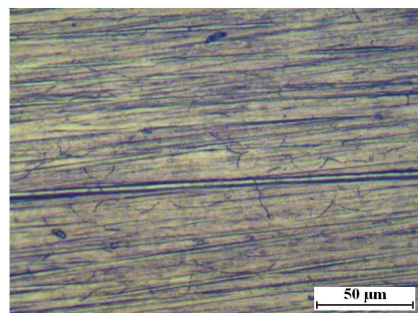
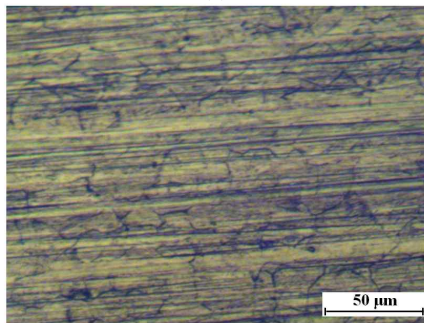


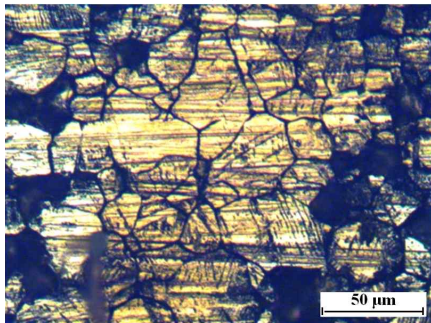
Fig. 1. Effect of aging time at 650 °C on the intergranular corrosion rate of 316L stainless steel determined by ASTM A 262 practice C.



(a)



(b)



(c)

Fig. 2. Corroded surface appearance of 316L stainless steel aged at 650 °C after one 48 period tested by ASTM A262 practice C: (a) 0 h-aged, (b) 1 h-aged, (c) 5 h-aged.

The residual stress was calculated by $\sin^2\psi$ method with the Residual stress analysis II program.

3. Results

Intergranular corrosion of stainless steel is known to occur due to electrochemical potential difference between the matrix and Cr depleted zone in the vicinity of the grain boundary area. According to the conventional intergranular corrosion mechanism, Cr depletion and consequent intergranular corrosion are induced by the formation of Cr-carbide along grain boundaries²²⁻²⁴. Intergranular corrosion in the stabilized stainless steels is also induced by Cr depletion, due to the segregation of solute Cr atoms near grain boundary carbides such as TiC, (Ti, Nb)C, or NbC, but not due to the formation of Cr-carbide²⁵⁻²⁸. Therefore, the general commercial recommendation to prevent intergranular corrosion of stainless steel is the reduction in the content of carbon to less than 0.03 wt%²².

Fig. 1 shows that increasing aging time (t) at 650 °C greatly facilitated the intergranular corrosion of 316L stainless steel, even at the short aging time of 1 and 5 hours. In this figure, the intergranular corrosion rate and coefficient of determination (R^2) can be derived as follows :

$$\begin{aligned} \text{Intergranular corrosion rate, mm/yr} = \\ - 0.0016t^2 + 0.6669t - 0.2003 \end{aligned} \quad (1)$$

(t ; hour, $R^2 = 0.9996$)

However, according to the TTT diagram of 316L stainless steel,⁴) it is well known that if chromium carbide was not precipitated in grain boundaries and chromium depleted zone was not formed, the stainless steels doesn't be sensitized,. In order to identify the occurrence of intergranular corrosion, surface appearance after Huey test was observed. Fig. 2 shows the corroded surface appearance of 316L stainless steel aged at 650 °C after one 48 h period tested by ASTM A262 practice C (note that the corrosion rate in Fig. 1 was determined after five 48 h period immersion tests. but the surface appearance after one 48 h period test was shown to differentiate the effect of aging time because of the high corrosion rate by five 48 h period tests). After one 48 h immersion test, the annealed specimen (0 h-aged) showed a scratched surface and 1 h-aged specimen revealed grain boundaries, but many grains were detached in the 5 h-aged specimen, and also, in the case of 48 h-aged specimen²⁰), it was confirmed that grains and grain boundaries were severely corroded and detached. On the basis of the TTT diagram⁵) and proposed mechanisms²²⁻²⁴), the high corrosion rate and severely corroded appearance in the 48 h-aged specimen were under-

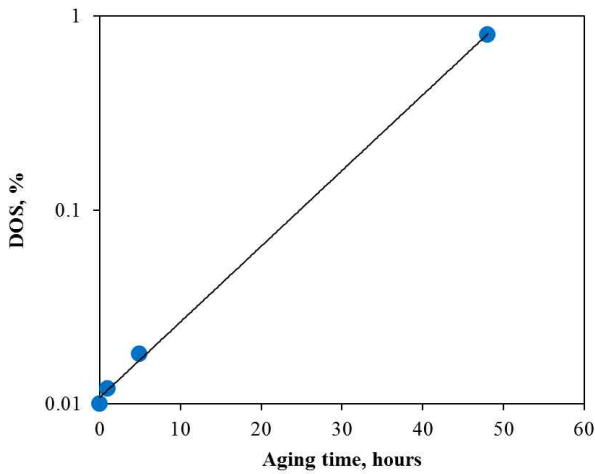


Fig. 3. Effect of aging time at 650 °C on the degree of sensitization of 316L stainless steel determined by DL-EPR test in 1 M H₂SO₄ + 0.005 M KSCN at 30 °C.

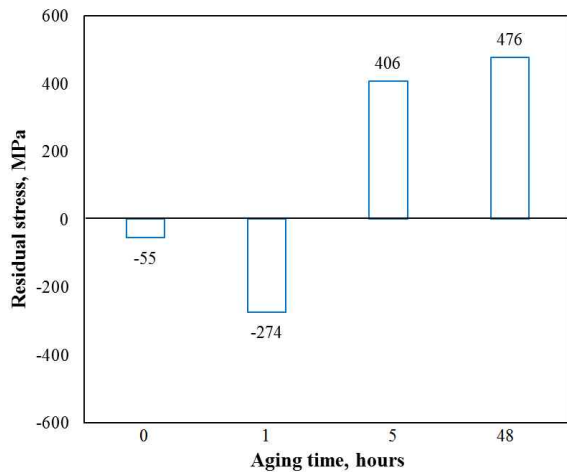


Fig. 4. Effect of aging time at 650 °C on the residual stress of 316L stainless steel determined by X-ray diffraction method.

stood, but why did the 5 h-aged specimen reveal a high corrosion rate and severely detached grains? Even though from the thermodynamical aspect, there was no possibility that chromium carbide could form.

Moreover, intergranular corrosion is closely related to the degree of sensitization of stainless steels. The degree of sensitization can be quantitatively determined using a DL-EPR test. Fig. 3 shows the degree of sensitization determined by the DL-EPR test. Similar to the corrosion rate in Fig. 1, 48 h-aged specimen showed high DOS. The degree of sensitization and coefficient of determination (R^2) can be derived as follows:

$$\text{DOS, \%} = 0.0108e^{0.0899t} \quad (2)$$

(t: hour, $R^2 = 0.9992$)

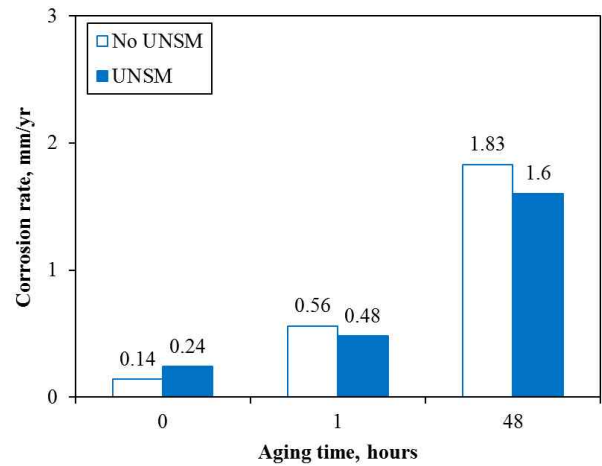


Fig. 5. Effect of UNSM treatment on the intergranular corrosion rate of 316L stainless steel aged at 650 °C determined by ASTM A262 practice C: 0 h-aged specimen, after five 48 h test periods; 1 h-aged specimen, after five 9 h test periods; 48 h-aged specimen, after five 3 h test periods.

In general, it is well known that the precipitation of chromium carbide and the depletion of chromium near grain boundaries induces intergranular corrosion of stainless steels²⁹. This sensitization process needs a critical time^{4,5}. Therefore, if this mechanism is correct, the specimen aged shorter than the transformation starting time in the TTT diagram should not be sensitized or corroded. However, Figs. 1, 2, and 3 show that intergranular corrosion and sensitization occurred in even shortly aged specimens. Comparison of Figs 1 and 3 indicates that intergranular corrosion is closely related to the degree of sensitization but their relationship is not linear. This means that other factors may affect the intergranular corrosion. Residual stress in the alloys can affect the corrosion properties including stress corrosion cracking and corrosion fatigue. Recently, it was reported that compressive residual stress may improve the intergranular corrosion resistance^{19,30,31}. Fig. 4 shows the effect of aging time on the residual stress of the surface of 316L stainless steel. Comparison of Figs 1 and 4 indicates that increasing the residual stress from negative to positive direction generally increased the intergranular corrosion rate.

In a typical UNSM technique, a tungsten carbide tip strikes the specimen surface up to 20,000 or more times per second, with 1,000 to 10,000 shots per square millimeter in a very short time. These strikes bring severe plastic deformation to surface layers and thus induce nano-crystalline structure.¹³ Fig. 5 shows the effect of UNSM treatment on the intergranular corrosion rate of 316L stainless steel aged at 650 °C determined by ASTM A262 practice C (note that corrosion rates in this figure

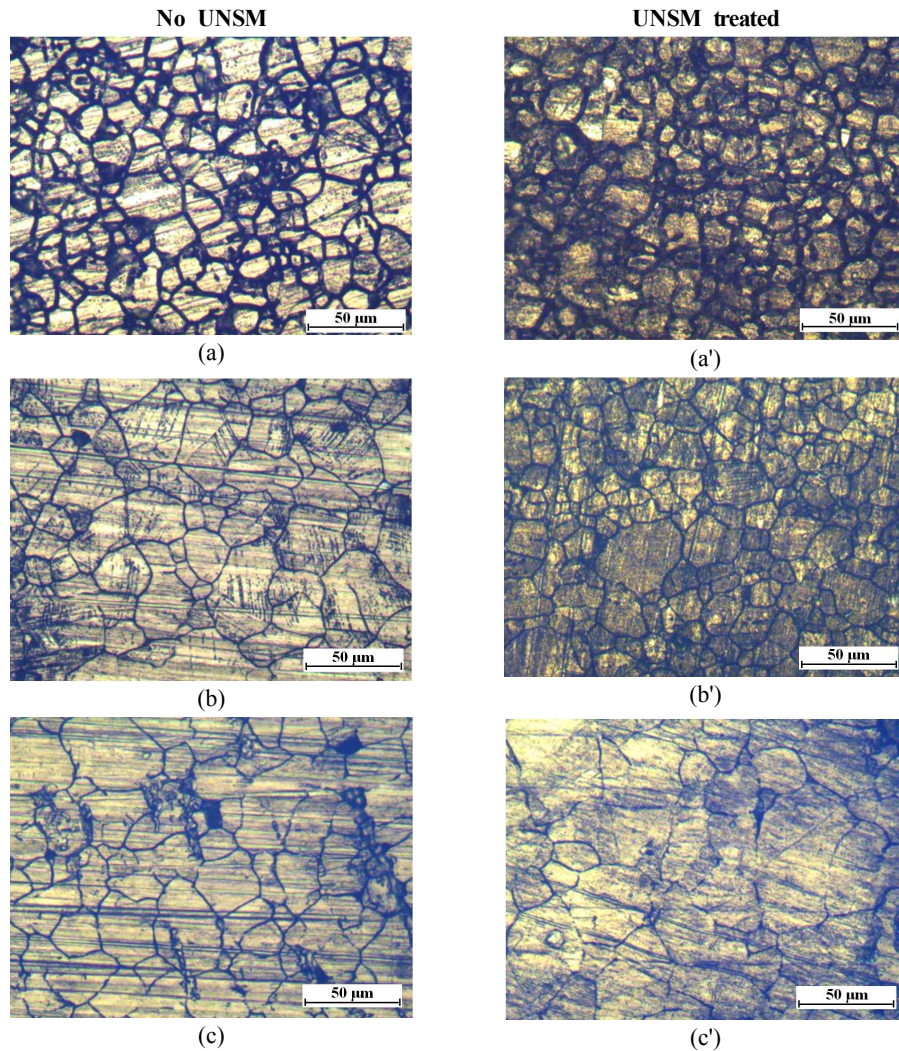


Fig. 6. Corroded surface appearance of 316L stainless steel tested by ASTM A262 practice C; (a, a') after five 48 h test periods of 0 h-aged specimen, (b, b') after five 9 h test periods of 1 h-aged specimen, and (c, c') after five 3 h test periods of 48 h-aged specimen.

mean the rates after five 48 h test periods for 0 h-aged specimen, and after five 9 h test periods for 1 h-aged specimen, and after five 3 h test periods for 48 h-aged specimen in order to differentiate the effect of UNSM on intergranular corrosion because of the formation of very thin surface layer by UNSM treatment)¹³⁾. UNSM treatment slightly increased the intergranular corrosion rate of the 0 h-aged specimen, but the treatment reduced the corrosion rate of the 1 h-aged and 48 h-aged specimens. Fig. 6 reveals the corroded surface appearance of 316L stainless steel tested by ASTM A262 practice C. In Fig. 6, (a and a') are the optical micrographs after five 48 h test periods of 0 h-aged specimen, (b and b') are the optical micrographs after five 9 h test periods of 1 h-aged specimen, and (c and c') are the optical micrographs after five 3 h test periods of 48 h-aged specimen. The degrees of

corrosion in Fig. 6 coincide with the corrosion rates in Fig. 5.

Fig. 7 reveals the effect of UNSM treatment on the degree of sensitization by a DL-EPR test. UNSM treatment did reduce the degree of sensitization even including the non-aged specimen. Reduced DOS should improve the intergranular corrosion resistance but Fig. 5 shows that the rate of the non-aged specimen increased. This implies that intergranular corrosion is closely related to the degree of sensitization but other factors may affect the intergranular corrosion as described above.

Residual stress in the alloys can affect the corrosion properties. Shot peening^{6,7,8)}, laser shock peening^{9,10)}, water jet peening¹¹⁾, ultrasonic peening¹²⁾, and UNSM^{13,14)} can reduce the residual stress from tensile stress to compressive stress. Fig. 8 shows the effect of UNSM treatment

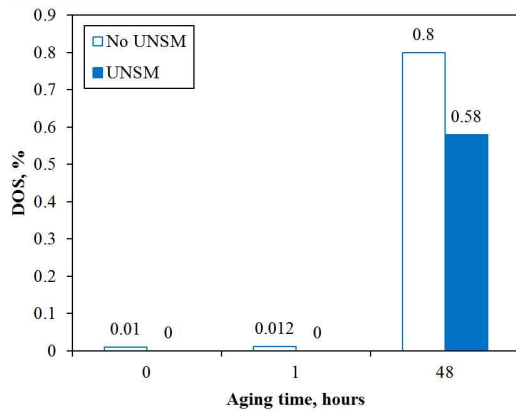


Fig. 7. Effect of UNSM treatment on the degree of sensitization of 316L stainless steel aged at 650 °C determined by DL-EPR test in 1 M H₂SO₄ + 0.005 M KSCN at 30 °C.

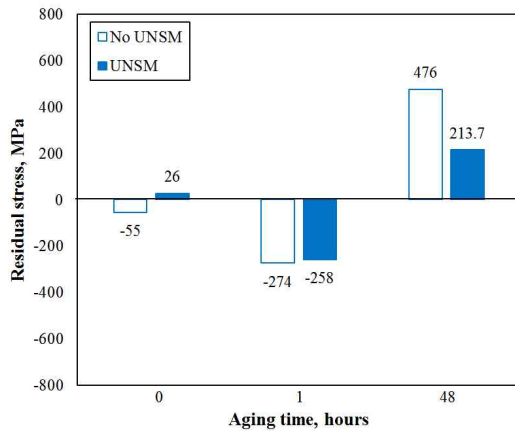


Fig. 8. Effect of UNSM treatment on the residual stress of 316L stainless steel aged at 650 °C determined by X-ray diffraction method.

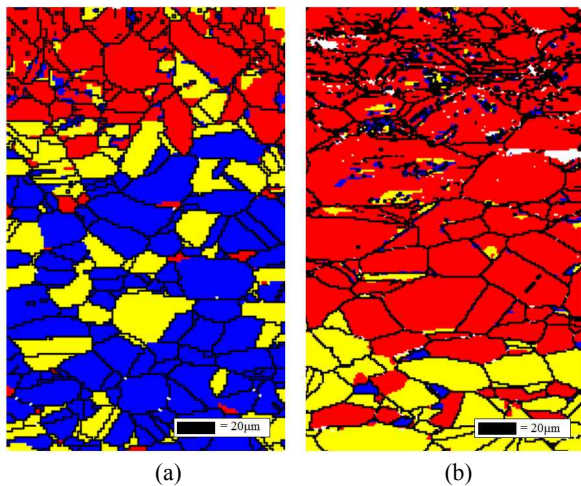


Fig. 9. The effect of UNSM treatment on deformed fraction of the cross section of 316L stainless steel aged at 650 °C for 48 hours determined by EBSD with step size of 1 µm at 30 kV: (a) No UNSM, and (b) UNSM.

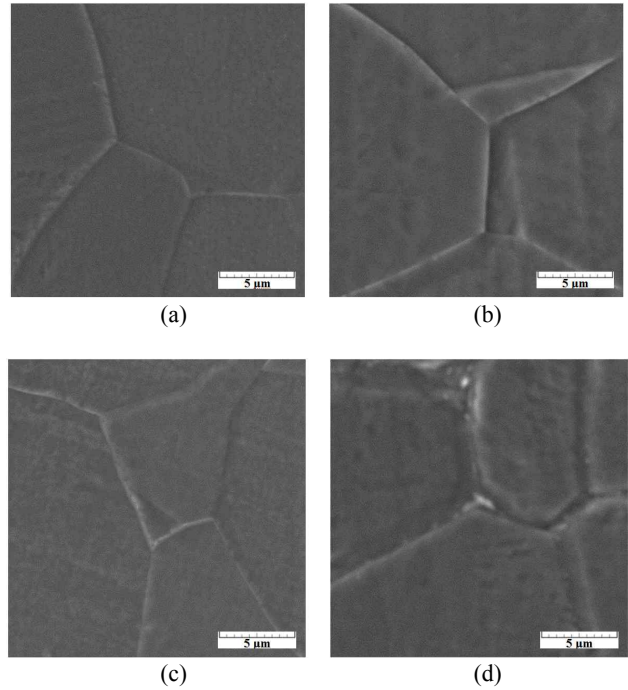


Fig. 10. SEM image showing grain boundary of 316L stainless steel with aging time at 650 °C: (a) 0 h-aged, (b) 1 h-aged, (c) 5 h-aged, and (d) 48 h-aged.

on the residual stress of the surface. In the case of 48 h-aged specimen, the residual stress was greatly reduced from tensile to compressive stress. Figs 5, 6, and 7 show that UNSM treatment improved the intergranular corrosion resistance of the aged specimen which was followed by the reduction of DOS and the induced compressive stress. However, in the case of the non-aged specimen, UNSM treatment increased the corrosion rate which was followed by increase of the residual stress from compressive to tensile, even though the UNSM treatment reduced the DOS.

Grain boundary engineering proposed by T. Watanabe³²⁾, means the intentional modification of the grain boundary structure to improve the materials properties; and also, UNSM treatment can refine the grain size of the surface¹³⁾. Fig. 9 shows the deformed fraction determined by EBSD on the cross-section treated by UNSM for the 48 h-aged specimen. UNSM treatment increased the ratios of deformed area in the 48 h-aged specimen, but it seems that in Fig. 7, the increased ratios of deformed area did not affect the intergranular corrosion.

4. Discussion

As described above, aging heat treatment sensitized the austenitic 316L stainless steel and increased its inter-

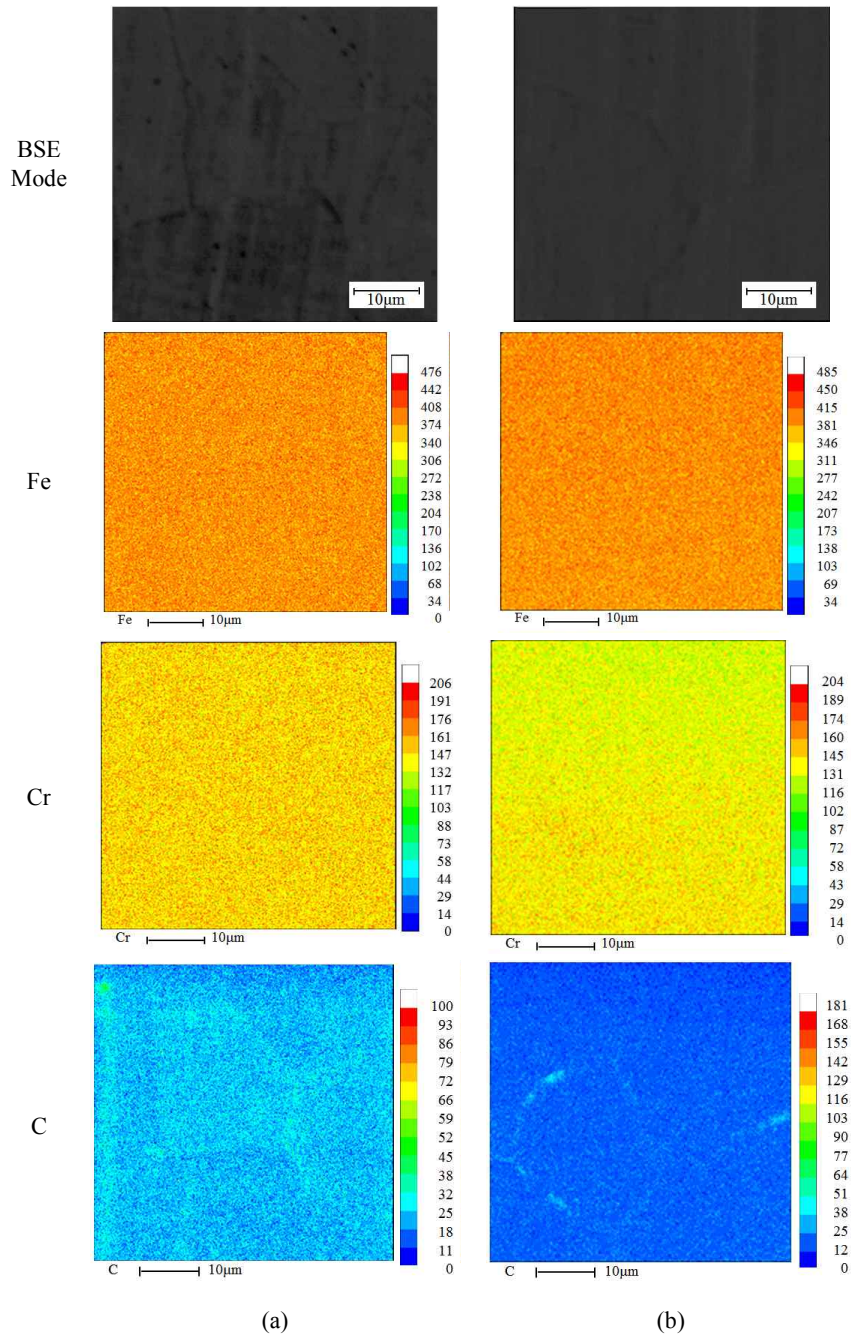


Fig. 11. Effect of aging time at 650 °C on grain boundary segregation of 316L stainless steel analyzed by EPMA: (a) 1 h-aged, (b) 5 h-aged.

granular corrosion rate and also UNSM treatment refined the grain and reduced the degree of sensitization, and improved its intergranular corrosion rate. In this paper, we first focused on the relationship between chromium carbide formation and intergranular corrosion. Even though from the thermodynamical aspect there was no possibility that chromium carbide could form^{4,5)}, we asked why the

5 h-aged specimen reveal a high corrosion rate in Fig. 1 and severely detached grains in Fig. 2.

Fig. 10 shows the SEM images showing the grain boundary of 316L stainless steel with aging time at 650 °C. In the non-aged, 1 h-aged, and 5 h-aged specimens, no precipitation was observed at the grain boundaries; but in the 48 h-aged specimen, precipitation was observed at

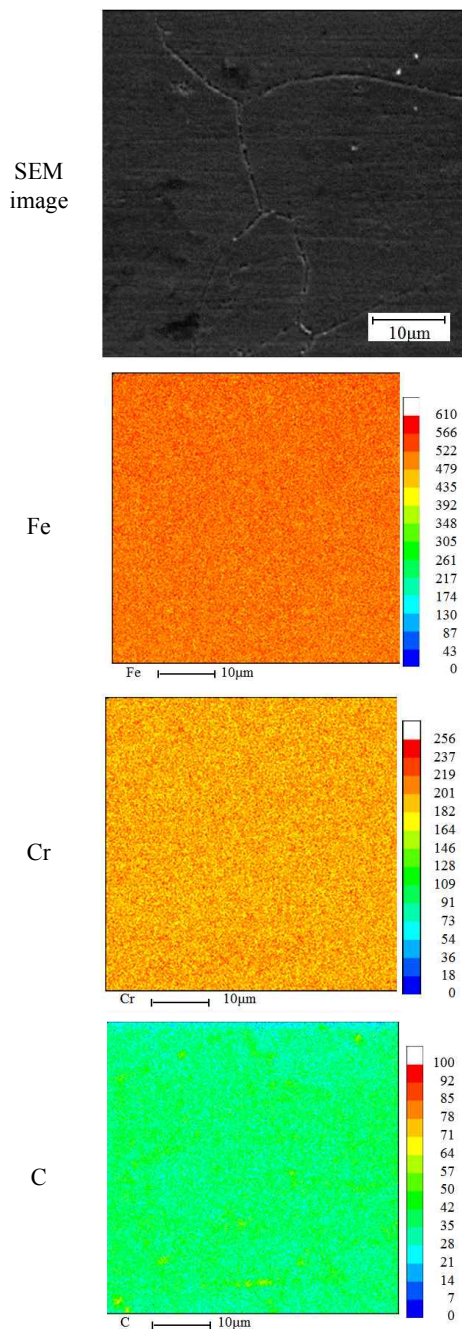


Fig. 12. Grain boundary segregation of UNSM-treated surface in 48 h-aged specimen analyzed by EPMA.

the grain boundaries. From Figs 1 and 10, we confirmed that no precipitation was formed in the shortly aged specimen, but its intergranular corrosion rate drastically increased.

Fig. 11 shows the effect of aging time at 650 °C on the grain boundary segregation of 316L stainless steel analyzed by EPMA. Increasing the aging time depleted the iron and enriched the chromium and segregated and en-

riched the carbon. The high intergranular corrosion rate of the 48 h-aged specimen was already interpreted on the basis of elemental segregation and enrichment²⁰). However, in the 5 h-aged specimen, chromium was not enriched, but carbon was segregated in grain boundaries. Therefore, we also confirmed that the increased intergranular corrosion rate of the 5 h-aged specimen was due to the segregation of carbon in the grain boundaries. Fig. 12 shows the effect of UNSM treatment on the grain boundary segregation of surface in the 48 h-aged specimen analyzed by EPMA (EPMA result about non-UNSM treated specimen was described in the previous report²⁰). As described before, the UNSM treatment in the 48 h-aged specimen greatly reduced carbon (also chromium and iron) segregation and enrichment in the grain boundaries. Namely, it is considered that the high mechanical energy of UNSM treatment can diffuse the segregated carbon (also chromium and iron), and reduce the segregation of carbon (also chromium and iron) in grain boundaries, and thus decrease its intergranular corrosion rate. This behavior was confirmed by the result for the cross-section. Fig. 13 shows the effect of UNSM treatment on the grain boundary segregation of the cross-section in 48 h-aged specimen analyzed by EPMA. As for the result on the surface in Fig. 12, UNSM treatment greatly reduced carbon (also chromium and iron) segregation and enrichment in the grain boundaries in the 48 h-aged specimen.

However, residual stress is one of the important factors that affect intergranular corrosion.²⁰) The intergranular corrosion rate by aging in Fig. 1 was attributed to the summation of the increased degree of sensitization in Fig. 3, and the increased residual stress in Fig. 4. The intergranular corrosion rate by UNSM treatment in Fig. 5 was due to the change of residual stress. That is, UNSM treatment reduced the degree of sensitization of non-aged specimen in Fig. 7, but conversely increased the intergranular corrosion rate in Fig. 5 a little, and this behavior can be related to the increased residual stress by UNSM treatment in Fig. 8. Therefore, note that tensile residual stress facilitates intergranular corrosion, while compressive residual stress suppressed intergranular corrosion.

We can therefore propose a new intergranular corrosion model by aging and UNSM treatment; Fig. 14 shows the intergranular corrosion model of 316L stainless steel with aging time – increasing aging time at sensitization temperature range first segregates free carbon in grain boundaries, and then segregates it more and more with time. Finally, after the critical time to form any carbide, carbon segregates in grain boundaries and enriches to form metallic carbides. In summary, carbon segregates in grain boundaries with aging time, and thereafter the carbide formed

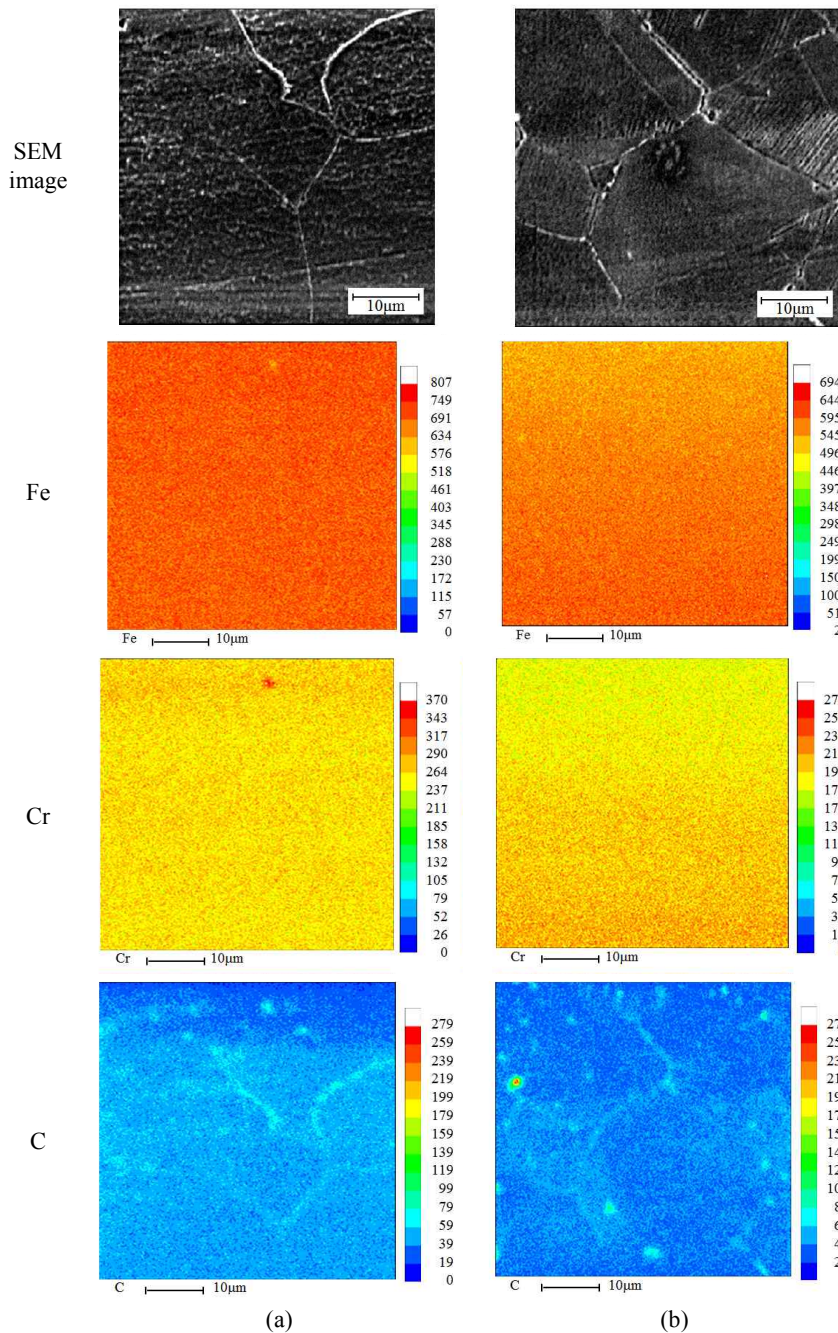


Fig. 13. Effect of UNSM on grain boundary segregation of the cross-section in 48 h-aged specimen analyzed by EPMA: (a) No UNSM, and (b) UNSM.

after the critical time results in tensile residual stress; Therefore, carbide formation (finally chromium depletion) and carbon segregation in grain boundaries increase intergranular corrosion, and residual stress may affect intergranular corrosion. Fig. 15 shows the mechanism of the improvement of intergranular corrosion by UNSM treatment for aged stainless steel – application of UNSM treat-

ment on the surface refines the grain size, eliminates the metallic carbides, and reduces the carbon segregation. Moreover, in depth, UNSM treatment refines the grain size and reduces the concentration of carbon segregation and metallic carbide in grain boundaries, and introduces compressive residual stress. These factors improve the intergranular corrosion resistance.

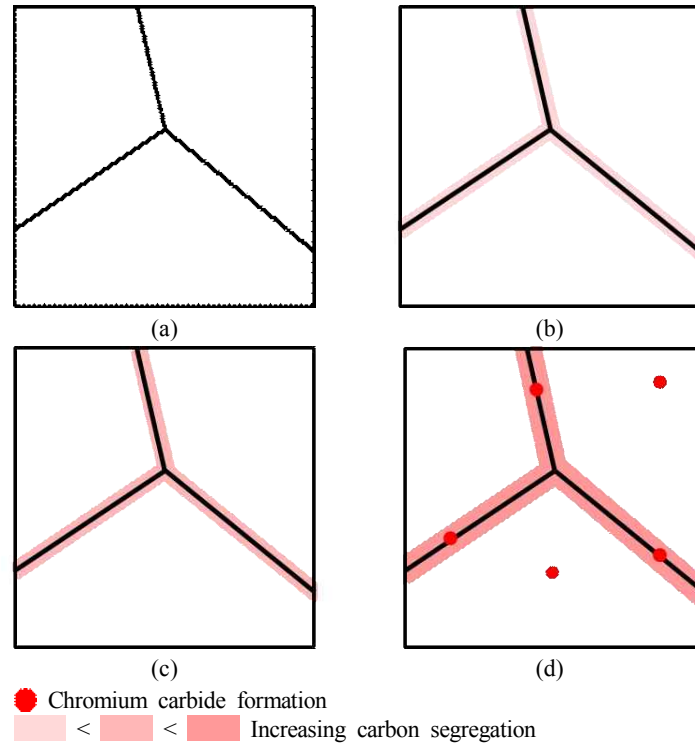


Fig. 14. Intergranular corrosion model by aging time of 316L stainless steel at sensitization temperature range: (a) 0 h-aged, (b) 1 h-aged, (c) 5 h-aged, and (d) 48 h-aged.

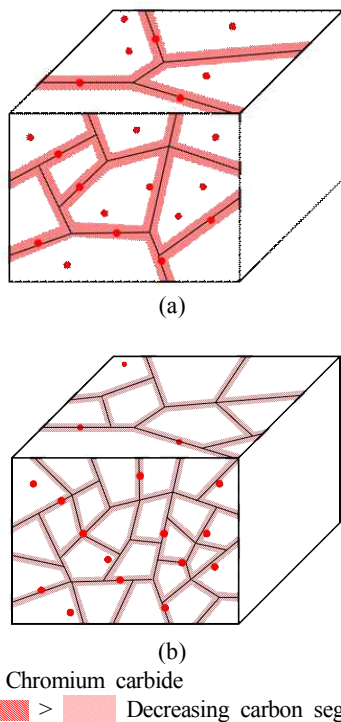


Fig. 15. Improvement of intergranular corrosion by UNSM treatment of aged 316L stainless steel: (a) No UNSM, and (b) UNSM.

5. Conclusions

- 1) Even though chromium carbide was not precipitated, the intergranular corrosion rate of 316L stainless steel was drastically increased with aging time, and it was confirmed that the increased intergranular corrosion rate of slightly-sensitized (not carbide formed) 316L stainless steel was due to the carbon segregation along the grain boundaries.
- 2) UNSM treatment improved the intergranular corrosion resistance of aged stainless steels, and its improvement was due to the reduction of carbon segregation and the grain refinement of the outer surface, including the introduction of compressive residual stress.

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