New Aspects of Sensitization Behavior in Recent 316 Type Austenitic Stainless Steels†

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Abstract

Intergranular precipitation behavior of 316 and 316L stainless steels after annealing at 600 - 900 °C for 5 min - 50 hrs were examined using transmission electron microscopy of carbon extraction replicas and thin foil techniques. Precipitated particles were identified by electron diffraction analysis. Chemical compositions of precipitated particles were measured from EDX - spectra by a semiquantitative method. When 316 steel was annealed at 750 - 850 °C for 15 min or longer, only $M_{23}C_6$ carbide was identified at grain boundaries. For 316L steel, however, three kinds of particles, i.e. Laves phase, $M_{23}C_6$ and a quasicrystal, were precipitated at the grain boundaries when annealed at 700 - 800 °C for 10 hrs or longer. Most of the precipitated particles at grain boundaries of annealed 316L steel were Laves phase. $M_{23}C_6$ precipitation caused Cr depletion at grain boundaries of the annealed 316 steel, but the formation of Laves phase did not induce the Cr depletion at grain boundaries of annealed 316L steel. Although no Cr depletion occurred, the grain boundaries of annealed 316L steel were attacked in Oxalic acid etch tests and Strauss tests, probably because of electrical potential difference between the Laves phases and matrix, and/or low Cr contents in Laves phases. After single-pass welding with cooling rates higher than 0.07 °C/s, the weld HAZs of both 316 and 316L seem to be free of sensitivity to intergranular attack.

KEY WORDS: Stainless Steel, Annealing, Precipitation, Sensitization, Cr-depletion

1. Introduction

Austenitic stainless steels are used extensively in chemical, petrochemical, thermal and nuclear power industries because of their excellent corrosion resistance and good mechanical properties at elevated temperatures. Sensitization in unstabilized austenitic stainless steels refers to the loss of intergranular corrosion resistance of these steels, and generally occurs upon slow cooling from solution annealing temperatures (1000 - 1200 °C) or upon heating the steel in the temperature range of 600 - 850 °C for some time. Bain et al. [1)] suggested that sensitization occurs because chromium is depleted from regions of the alloy matrix adjacent to grain boundaries where chromium rich carbides, $M_{23}C_6$, have precipitated in these regions. $M_{23}C_6$ carbide formation causes the local chromium content to fall below 13 wt% at grain boundaries and in the matrix adjacent to grain boundaries, and leads to a loss of resistance to corrosion. Bruemmer et al.[2)] found that sensitization was a main reason for intergranular stress-corrosion cracking (IGSCC) in austenitic stainless steels.

Because the sensitization is a potential cause for the functional failure of stainless steel structures or containers, many studies have been made to understand the factors influencing the sensitization of stainless steels. Mulford et al.[3)] indicated that additions of N, Mn, or Mo or any combination of these elements could improve resistance to sensitization. Nitrogen additions appear to retard the nucleation and growth of carbides at grain boundaries. Molybdenum acts by changing the electrochemical response of the alloy, so that more chromium depletion is required to cause sensitization. However, Ni, Si and P seem to enhance the sensitization[3,4)]. Raising nickel contents will increase carbon activity and decrease carbon solubility. Silicon also increases carbon activity, like nickel, but its influence is much stronger. Segregation of phosphorous to grain boundaries appears to repel nitrogen and/or independently lower the corrosion resistance of stainless

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Transactions of JWRI is published by Joining & Welding Research Institute of Osaka Uni., Ibaraki, Osaka 567, Japan.

steels. Of all elements in stainless steels, the carbon is one of the most important, and the rate of sensitization is always dependent on the carbon content in the steels[3].

Beside the alloying elements, deformation or strain in steels has a significant effect on sensitization. Advani et al.[5,6] pointed out that deformation accelerates the development of grain boundary chromium depletion, or sensitization. The acceleration in sensitization is a function of the amount of strain in the material and temperature of isothermal sensitization treatment. Intergranular depletion is accelerated for strain below 20 %, while strain above 20 % enhances both intergranular and transgranular depletion.

In weld heat affected zone (HAZ) of stainless steels, the sensitization is almost dependent on the peak temperature and cooling rate imposed by weld thermal cycles[7]. A critical peak temperature was found to be 950 °C. Peak temperatures either above or below this critical temperature resulted in lower degree of sensitization. Continuous cooling sensitization development increased with decreasing cooling rate, and occurred primarily in the temperature range between 900 to 750 °C. Because of high cooling rates in real welding processes, however, the Cr depletion in the weldment did not seem to occur, but weld thermal cycles could promote the nucleation of carbides at grain boundaries[8,9]. The carbides nucleated by short exposure in the temperature range 500-850 °C without a detrimental degree of Cr depletion can probably grow when kept at lower temperatures for a long time and thereby cause a severe degree of Cr depletion[9].

Although many studies have been made to investigate sensitization behavior and a large amount of information has been known, the sensitization behavior in some recent stainless steels have not been clarified completely. Some studies showed that Cr-depletion did not occur even though carbides precipitated at grain boundaries[8]. This implies that the types and chemical compositions of the precipitates should be carefully checked when dealing with sensitization behavior in stainless steels. Few studies, however, have investigated the types and chemical compositions of precipitates at grain boundaries and their effects on Cr depletion.

In this paper, a study of particles precipitation at grain boundaries of 316 type steels is presented. The effects of precipitation on Cr depletion and intergranular attack are discussed.

2. Experimental Details

2.1 Materials

Austenitic stainless steels 316 and 316L were used in this study. Their chemical compositions are showed in Table 1. The as-received 316 steel plate (10 mm in thickness), containing 0.05 wt% carbon content, was solution annealed at 1040 °C and subsequently water quenched. The as-received 316L steel plate (10 mm in thickness), containing a lower carbon content (0.021 wt%), was solution annealed at 1100 °C and water quenched. The steel plates were machined into specimens of 6 mm in diameter and 55 mm in length, and supplied for heat treatment and HAZ simulation.

Table 1 Chemical compositions of experimental materials

Material	Chemical composition (mass %)							
	C	Si	Mn	P	S	Ni	Cr	Mo
316	0.05	0.47	0.86	0.026	0.001	11.56	17.55	2.10
316L	0.021	0.62	1.10	0.027	0.004	12.20	17.47	2.10

2.2 Heat treatment and HAZ simulation

The specimens of 316 and 316L steels were isothermally treated (or annealed) in a temperature range of 600 to 900 °C. Annealing time was 5 min - 20 h for 316 steel and 30 min - 50 h for 316L steel, respectively. The heat treatment was performed in a thermal/mechanical simulator, Gleeble 1500, when the annealing time was 5 - 30 min, or in an electrical furnace when the annealing time was 1 - 50 h, and followed by fast cooling or water quenching.

Single-pass weld HAZs of 316 steel were simulated with the Gleeble 1500. The peak temperature of thermal cycles for simulation was 950 °C, which was a critical temperature for sensitization[7]. Cooling rates were in the range of 1 to 0.05 °C/s for simulating weld HAZs with various heat inputs.

2.3 Metallography and analysis

Transmission electron micrography (TEM) of carbon extraction replicas and thin foils was used for determining precipitation behavior, identifying precipitated particles and analyzing chemical compositions of the particles. The transmission electron microscope employed was a JEM-1200 FX (JEOL), equipped with an EDX - analyzer and operating at 200 KV. The chemical composition of particles extracted in carbon replicas was calculated from the EDX - spectra according to the semiquantitative standardless method.

In addition, chromium concentration profiles across grain boundaries were measured on thin foils by using the EDX analyzer, in order to investigate whether or not the chromium depletion existed at grain boundaries where precipitation occurred.

2.4 Intergranular attack test

An oxalic acid etch test (ASTM A 262A or JIS G 0571) was used for determining susceptibility to intergranular attack. According to ASTM A 262, this test is a rapid method for identifying the specimens which are certain to be free of susceptibility to intergranular attack. That is to say, the specimens which can pass this test are free of intergranular attack and those fail in this test should be further judged by other tests such as Strauss test. In this study, the oxalic acid etch test was taken to determine the safe ranges of heat treatment or cooling rates during welding.

For the specimens which were attacked by the oxalic acid etch test and exhibited no Cr depletion at grain boundaries, the Strauss test (ASTM A262E or JIS G 0575) was used to confirm the susceptibility to intergranular attack.

3. Results and Discussion

3.1 Precipitation behavior

The intergranular precipitation behavior of annealed 316 and 316L steels can be illustrated by C - curves, as shown in **Fig. 1**. At the right side of the C curves, heat treatment induced intergranular precipitation. The C curve nose of 316 steel is at a position of higher temperature and shorter time, compared with that of 316L steel. This tendency is in accordance with the results obtained by other researchers[9], suggesting that decreasing the carbon content of stainless steels can retard the intergranular precipitation and improve the resistance of the stainless steels to corrosion.

TEM micrographs of carbon replicas from annealed 316 steel are illustrated in **Fig. 2**. When the annealing time was 10 minutes or shorter, no intergranular precipitation was observed irrespective of annealing temperatures. When annealing temperatures are 750 - 850 °C, heat treatment for 15 min or longer led to intergranular precipitation. At temperatures higher than 900 °C or lower than 650 °C, no precipitation occurred.

TEM micrographs of intergranular precipitation in annealed 316L steel are showed in **Figs. 3** and **4**. Fig. 3 reveals the precipitation behavior with various annealing times, keeping annealing temperature at 750 °C. When the annealing time was 5 hours or longer, precipitation occurred. Keeping the annealing time for 10 hours, the precipitation phenomena at various temperatures can be understood from Fig. 4. In this case, when heat treatment temperatures are 700 - 800 °C, precipitation occurred.

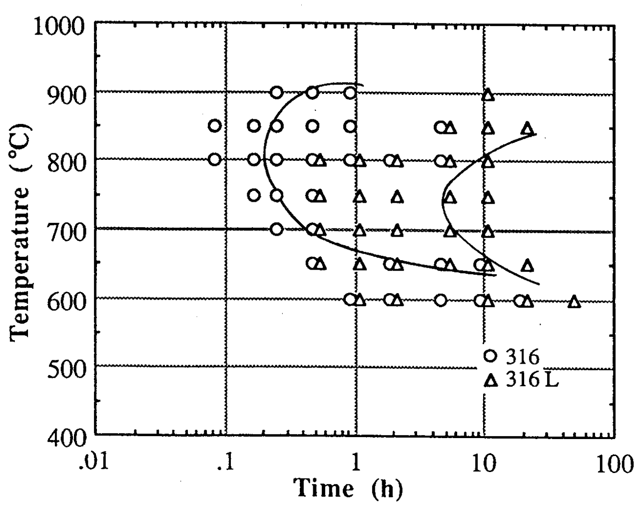


Fig. 1 Diagram of temperature-time-precipitation (TTP)

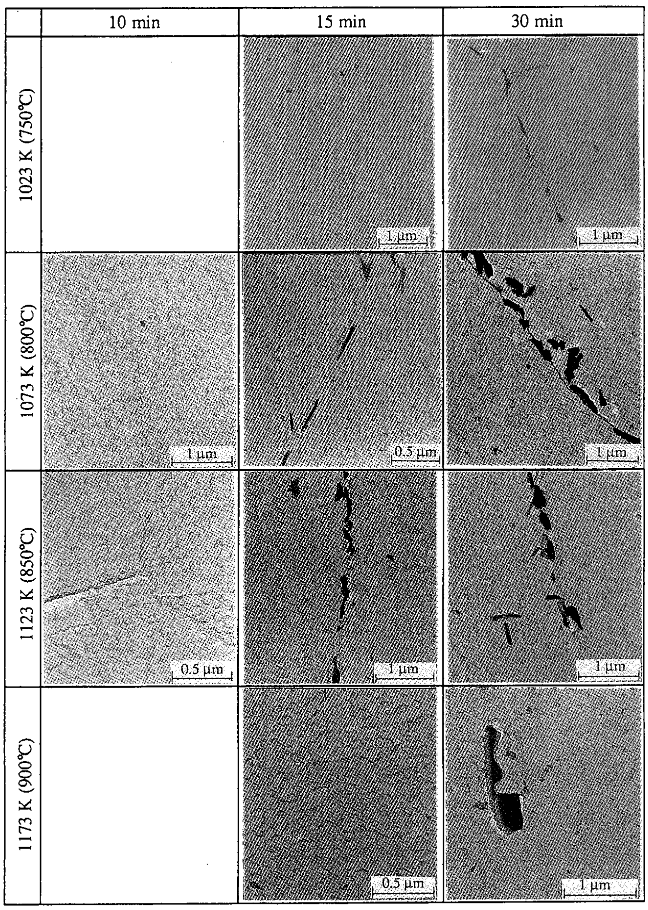


Fig. 2 Effect of annealing temperature and time on intergranular precipitation of 316 steel

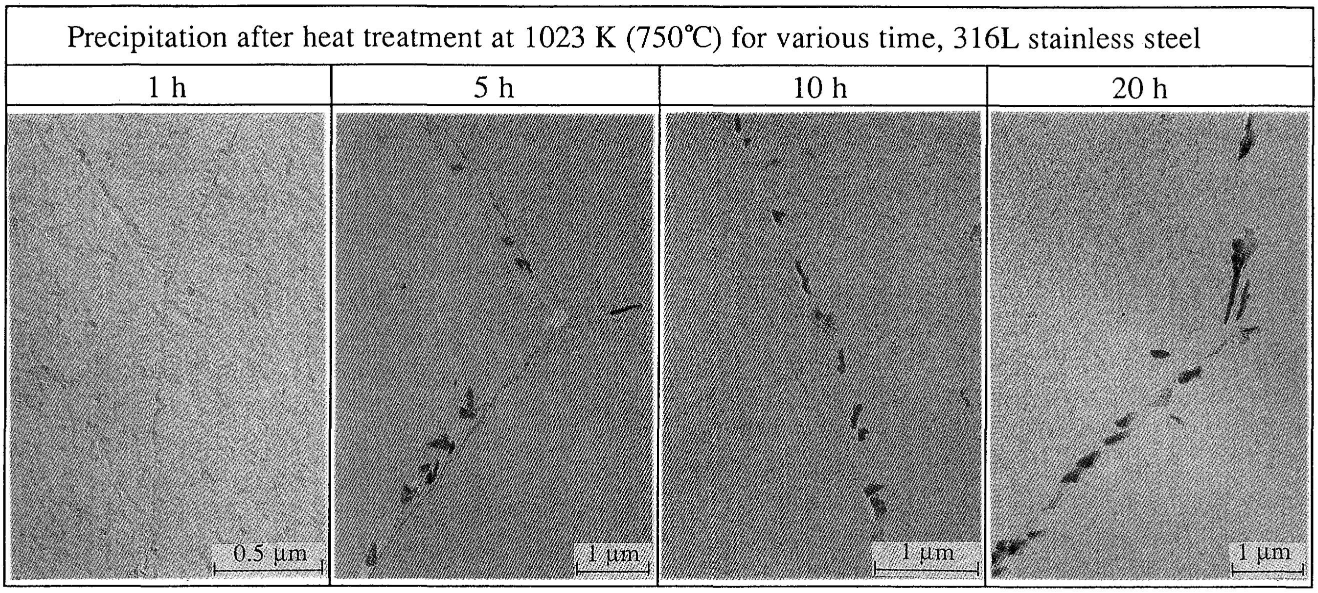


Fig. 3 Effect of annealing time on intergranular precipitation of 316L steel

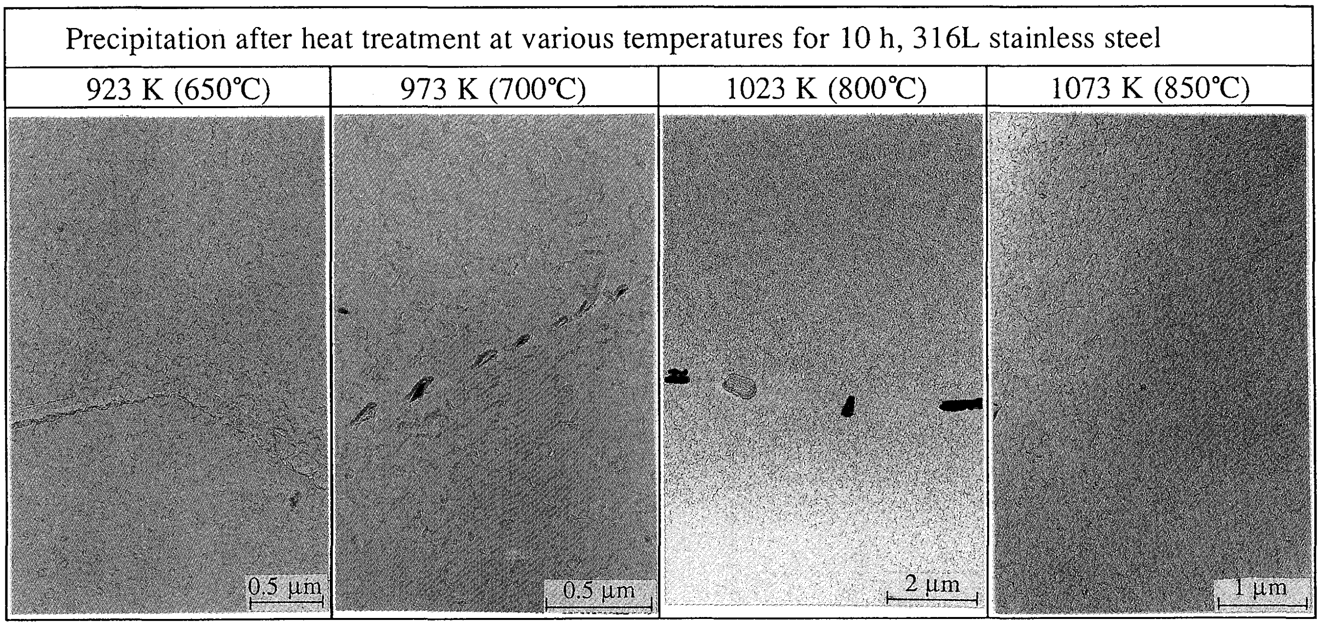


Fig. 4 Effect of annealing temperature on intergranular precipitation of 316L steel

3.2 Precipitated particles

One kind of particle was determined at the grain boundaries of annealed 316 steel. Although the particles on carbon replica films took various shapes such as bar, triangle or quadrilateral, the chemical composition measurements and electron diffraction analyses demonstrated that all these particles were chromium enriched carbides $M_{23}C_6$. **Fig. 5** shows an electron diffraction pattern of these carbides (Fig. 5 (c)) and its resolution (Fig. 5 (d)). The TEM extraction replica micrographs of intergranularly precipitated carbides corresponding to the diffraction pattern in Fig. 5 (c) are given in Fig. 5 (a) (low magnification) and Fig. 5 (b) (high magnification).

The chemical compositions of $M_{23}C_6$ carbides at various heat treatment conditions are documented in **Tables 2 and 3**. For each heat treatment condition, 10 -

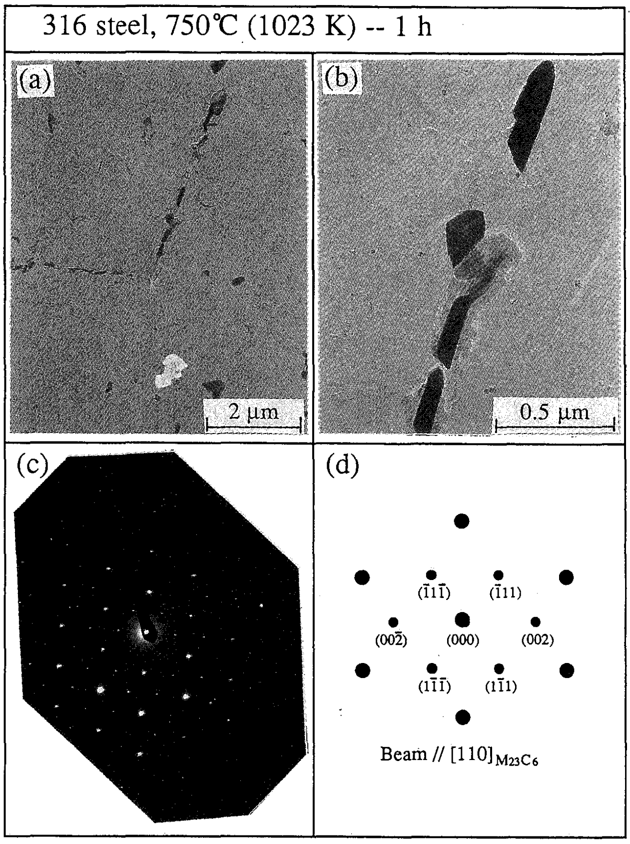


Fig. 5 TEM micrographs and electron diffraction analysis of intergranular particles $M_{23}C_6$ in annealed 316 steel

Table 2 Chemical compositions of intergranular particles in annealed 316 steel (annealing temperature: 750 °C)

No. of specimen	Time (h)	Chemical composition (mass %)				Note
		Cr	Fe	Ni	Mo	
B11	0.25	66.83	20.30	2.17	10.70	
B3	0.5	63.43	22.51	2.38	11.69	
B10	1	69.80	18.29	1.90	10.21	
B18	5	70.02	15.76	2.60	11.63	
B25	10	67.15	19.40	2.83	10.92	

20 particles were analyzed, and the average chemical compositions were used in Tables 2 and 3. The Cr content in $M_{23}C_6$ is in the range of 63 - 71 wt%, depending on heat treatment. Mo content in $M_{23}C_6$ is in the range of 9 - 12 wt%. Both Cr and Mo contents in $M_{23}C_6$ are much higher than those in the as-received material.

Table 3 Chemical compositions of intergranular particles in annealed 316 steel (annealing temperature: 800, 850, 900 °C)

No. of specimen	Time (h)	Chemical composition (mass %)				Note
		Cr	Fe	Ni	Mo	
800℃						
B52	0.17	--	--	--	--	no particle
B24	0.25	67.89	18.79	2.30	11.03	
B5	0.5	65.17	22.13	2.81	9.89	
B12	1	66.59	21.33	1.33	10.75	
850℃						
B34	0.25	67.45	18.64	1.94	11.96	
B33	0.5	68.61	20.01	2.05	9.30	
B32	1	68.38	16.68	2.60	11.35	
900℃						
B50	0.25	--	--	--	--	no particle
B53	0.5	69.54	18.68	1.93	9.98	
B54	1	70.68	17.08	1.86	10.30	

Table 4 Chemical compositions of intergranular particles in annealed 316L steel (annealing temperature: 750 °C)

No. of specimen	Time (h)	Chemical composition (mass %)					Note
		Cr	Fe	Ni	Mo	Si	
A4	0.5	--	--	--	--	--	no particle
A10	1	14.81	32.61	--	45.85	6.74	a few, small
A19	5	28.89	40.60	3.54	23.68	3.22	a few
		16.54	32.59	4.29	41.71	4.87	
A18	10	70.87	16.93	2.29	9.78	--	a few
		13.60	34.93	3.95	41.94	5.88	
A31	20	27.96	42.25	3.61	23.29	2.89	a few
		13.92	35.23	3.65	42.08	5.13	

At grain boundaries of annealed 316L steel, three kinds of particle were identified from both chemical composition measurement and electron diffraction analysis. These three kinds of particle contain Cr at about 13 - 18, 27 - 29 and 63 - 71 wt%, respectively, as can be seen from **Tables 4** and **5**. The particles containing about 63 - 71 wt% chromium are $M_{23}C_6$ carbides, the same as those precipitated in 316 steel.

An electron diffraction pattern of the particles containing 27 - 29 wt% Cr and about 23 wt% Mo is given in **Fig. 6 (a)**. The TEM micrographs of the

Table 5 Chemical compositions of intergranular particles in annealed 316L steel (annealing time: 10 hrs)

No. of specimen	Tem. (°C)	Chemical composition (mass %)					Note
		Cr	Fe	Ni	Mo	Si	
A23	600	--	--	--	--	--	no particle
A25	650	--	--	--	--	--	no particle
A20	700	65.59	17.37	3.22	12.89	0.34	a few
		18.83	30.96	3.72	40.55	5.97	
A18	750	70.87	16.93	2.29	9.78	--	a few
		13.60	34.93	3.95	41.94	5.88	
A28	800	13.11	36.35	4.10	42.38	4.06	
A35	850	--	--	--	--	--	no particle

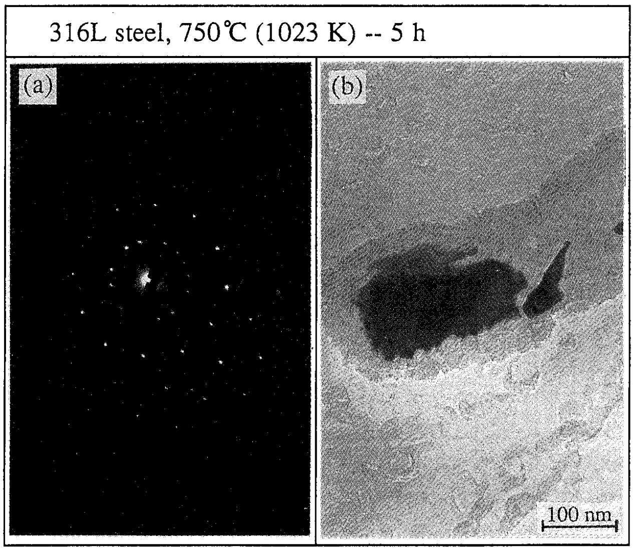


Fig. 6 TEM micrographs and electron diffraction analysis of intergranular particles containing 27-29 wt% Cr in annealed 316 steel

particles corresponding to the diffraction pattern in Fig. 6 (a) is showed in **Fig. 6 (b)**. The diffraction pattern is difficult to resolve. According to some studies[10], the particles having such diffraction patterns are probably quasicrystals.

The particles containing 13 - 18 wt% Cr and 41 - 45 wt% Mo were identified as Laves phase (η). An electron diffraction pattern of these particles and its resolution are given in **Fig. 7 (c)** and **(d)**. The TEM micrographs of corresponding intergranular particles are showed in **Fig. 7 (a)** and **(b)**.

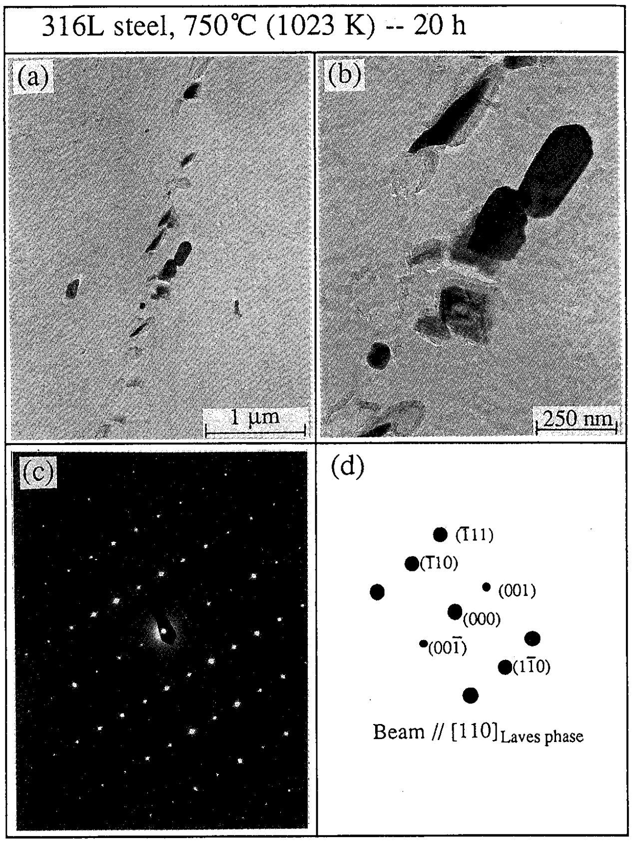
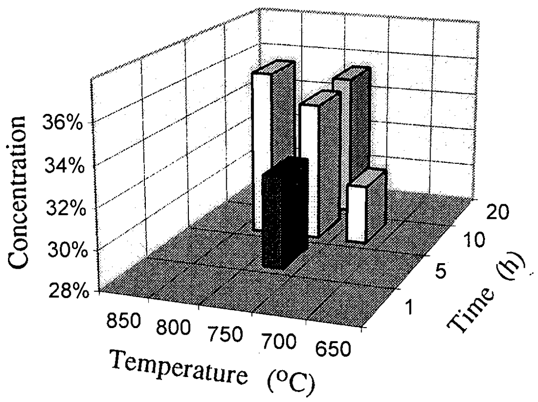
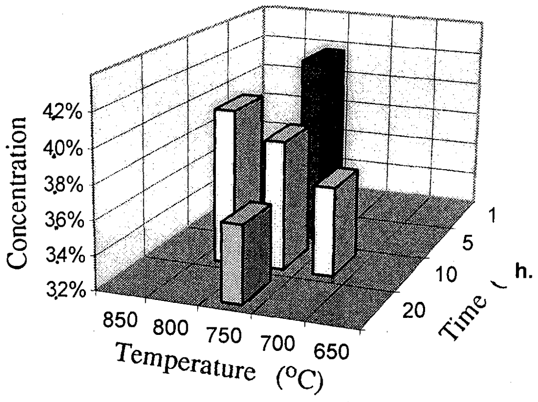


Fig. 7 TEM micrographs and electron diffraction analysis of intergranular particles (Laves phase) in annealed 316 steel

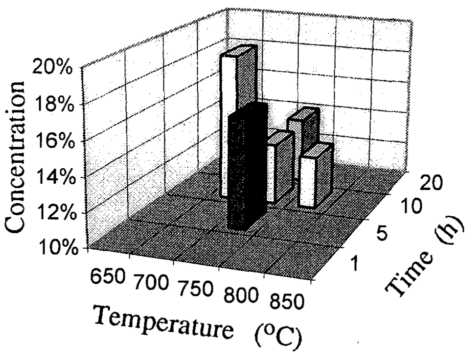
From the measurement results, it can be concluded that most intergranularly precipitated particles in annealed 316L steel are Laves phase (above 90 %). $M_{23}C_6$ and the other kinds of particles (quasicrystals) are few.



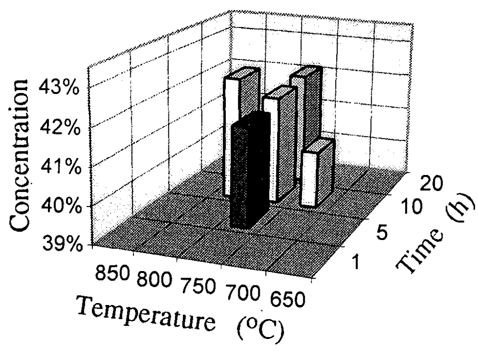
(a) Fe



(b) Ni



(c) Cr



(d) Mo

Fig. 8 Effect of annealing temperature and time on Fe, Ni, Mo and Cr concentrations of intergranular particles in annealed 316L steel

It can also be seen from Tables 4 and 5 that chemical compositions of Laves phase changed with heat treatment conditions. **Fig. 8** shows the changing levels of Fe, Ni, Mo and Cr contents with heat treatment temperature and time. The Mo content in Laves phases seems to increase with heat treatment temperature. Cr content decreases with increase in heat treatment time and temperature.

It should also be noted that as well as Fe, Ni, Mo and Cr identified in the Laves phases, but as reported in other papers[4,11] Si was also found in Laves phase in the present study. The Si content in Laves phase was about 4 - 7 wt%.

3.3 Chromium depletion at grain boundaries

Chromium concentrations across grain boundaries were measured on thin foils. As shown in **Fig. 9**, Cr concentration measurements were made in a line normal to the grain boundary and between two intergranular particles, with a distance step of about 50 nm. In order to investigate the effect of precipitation on Cr depletion at grain boundaries, the 316 steel samples annealed at 750 °C for 0.5 and 1 hour and the 316L steel samples annealed at 750 °C for 10 and 20 hours were used.

Figure 10 shows a Cr concentration profile across a grain boundary of 316 steel annealed at 750 °C for 1 hour. Cr depletion was confirmed at the grain boundary. The measurements on other grain boundaries show similar results. The Cr content at the depleted region is very low (about 13 wt%).

However, Cr depletion did not occur in 316L steel when annealed at 750 °C for 10 and 20 hours, as can be seen from **Fig. 11**. Although particles precipitated

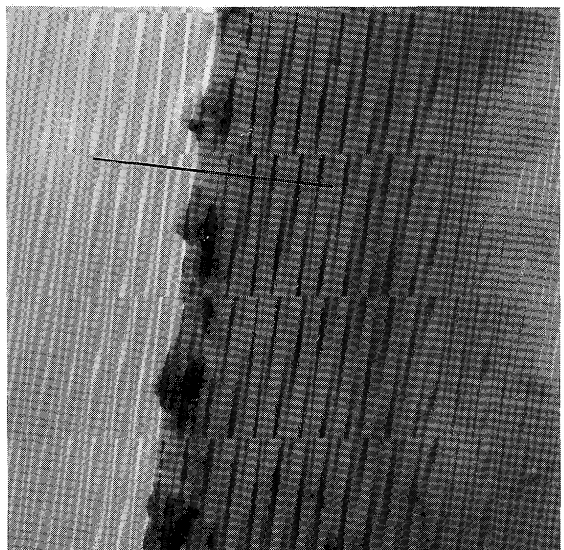


Fig. 9 TEM micrograph of intergranular particles in annealed 316 steel

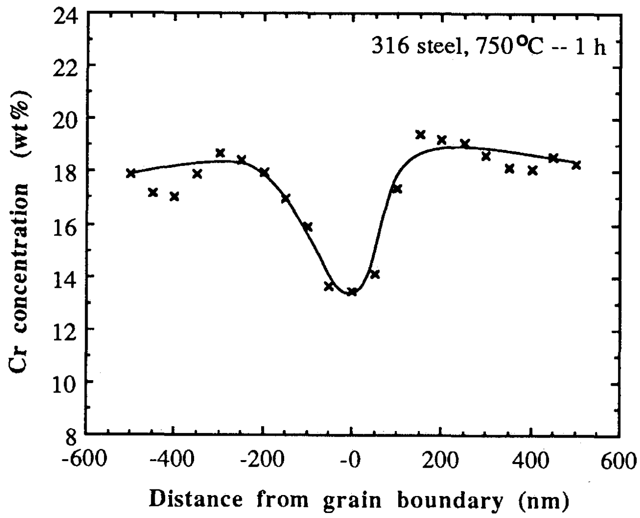


Fig. 10 Chromium concentration across a grain boundary

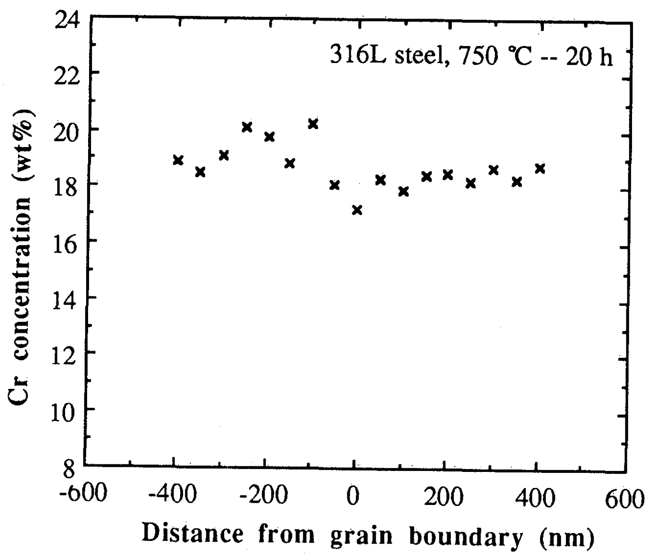


Fig. 11 Chromium concentration across a grain boundary

extensively at grain boundaries, most of the precipitated particles were Laves phase. Because the Cr content in Laves phase is quite low, in the range of 13 - 18 wt% in the present study, the formation of Laves did not induce the Cr depletion.

From above results, it can be concluded that not all intergranular precipitation leads to Cr depletion. The type and chemical compositions of precipitated particles have a strong influence on Cr depletion. $M_{23}C_6$ precipitation at grain boundaries could induce Cr depletion, while Laves phase precipitation did not contribute to Cr depletion.

3.4 Intergranular attacking susceptibility

The oxalic acid etch test was employed to detect the susceptibilities to intergranular attack of annealed 316 and 316L steels. The oxalic acid etch test results of annealed

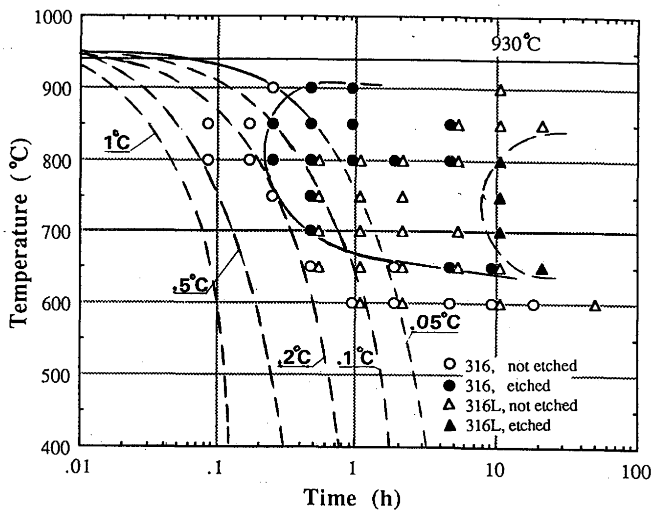


Fig. 12 Results of Oxalic acid etch test

316 and 316L steels are given in **Fig. 12**. In Fig. 12, the dark circles or triangles represent that the intergranular attack happened after oxalic acid etch test.

Comparing Fig. 12 with Fig. 1, it can be seen that the grain boundaries were attacked by oxalic acid etch test, usually when particles precipitated at grain boundaries. When 316L steel was annealed at 700 - 800 °C for 10 hours or longer, although Cr depletion did not form (see Fig. 11), the grain boundaries were attacked in the oxalic acid etch test because of extensive intergranular precipitation. This intergranular attack is attributed to electrical potential difference between the precipitated particles and the matrix.

Because the oxalic acid etch test is a test for identifying those specimens which are certain to be free of sensitivity to intergranular attack, the test results reveal the safe range of heat treatment. For 316L steel, the applicable heat treatment range is very wide. When the annealing time is less than 5 hrs, no intergranular attack can occur, irrespective of annealing temperatures.

For 316L steel, grain boundaries were attacked in the oxalic acid etch test when annealed at 750 °C for 10 hrs or longer, although no Cr depletion was detected, as shown by Fig. 11. In order to get a comprehensive understanding of the relationship between intergranular attack and Cr depletion, Strauss tests were made on 316L specimens which were annealed at 750 °C for 10, 20 and 50 hrs. The results demonstrated that 316L steel was also attacked intergranularly in the Strauss test although no Cr depletion occurred when annealed at 750 °C for 10, 20 and 50 hrs, as can be seen from **Fig. 13**. It can be seen from Fig. 13 that the intergranularly precipitated Laves phase was etched. This intergranular attack is probably

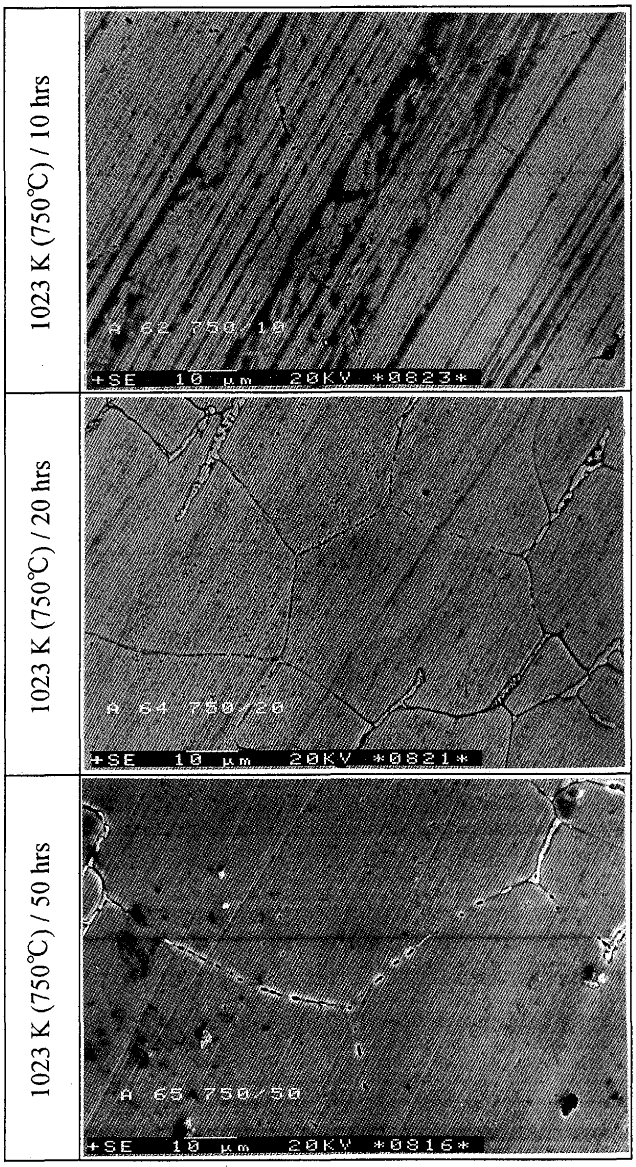


Fig. 13 SEM micrographs of 316L specimens etched by Strauss test

attributable to the low Cr content in Laves phase. Because Laves phase itself contains a low Cr content and precipitates extensively at grain boundaries when the annealing time is long enough (for example, longer than 10 hrs), the grain boundaries carrying Laves phases could be attacked.

Table 6 gives results of oxalic acid etch tests on simulated weld HAZs of 316 steel. It is known that only extremely slow cooling rates (0.07 °C/s or less) lead to intergranular attack of weld HAZs of 316 steel. This means that normal welding practice will not induce intergranular attack, because the cooling rates in the HAZs of practical welds are much higher. For 316L steel, it can be guessed that weld HAZs are also free of

Table 6 Oxalic etching results of continuously cooled samples with various cooling rate

No. of specimen	Cooling rate (K/s)	Result
B61	1.0	No etched
B62	0.5	No etched
B63	0.2	No etched
B64	0.1	No etched
B65	0.07	Etched
B66	0.05	Etched

sensitivity to intergranular attack after normal welding because of retarded precipitation behavior as can be seen from Fig. 1.

Of course, it should be pointed out that weld thermal cycles can probably promote the nucleation of precipitation at grain boundaries, and these nucleated particles can grow when the weldment is held at 300 - 400 °C (service temperature) for a long times[8,9], and this will probably lead to the intergranular attack.

4. Conclusions

The intergranular precipitation behavior in annealed 316 and 316L stainless steels were observed by means of TEM micrography of carbon replica and thin foil techniques. The precipitated particles were identified by electron diffraction analysis. Chemical compositions of intergranular particles were measured from EDX-spectra with a semiquantitative standardless method. The effects of intergranular precipitation on Cr depletion and intergranular attack were examined and the main conclusions are as follows:

(1) Time - temperature - precipitation (TTP) diagrams of 316 and 316L stainless steels were determined. For 316 steel, intergranular precipitation occurred when annealed at 750 - 850 °C for 15 min or longer. For 316L steel, annealing at 700 - 800 °C for 10 h or longer led to precipitation at grain boundaries.

(2) In the present heat treatment conditions, only Cr enriched carbides $M_{23}C_6$ were observed at grain boundaries of annealed 316 steel. At the grain boundaries of annealed 316L steel, three types of particles were identified, but most of the precipitated particles were Laves phase.

(3) The Laves phase contains not only Fe, Mo, Cr and Ni but also Si. The content of Mo in Laves phase

seems to increase with annealing temperature. The content of Cr seems decrease with an increase in annealing time and temperature.

(4) The micro-chemical composition profiles across grain boundaries of the selected specimens of 316 and 316L steels were measured. The Cr depletion at grain boundaries of 316 steel (annealed at 750 °C/0.5 and 1 h) was confirmed, while no Cr depletion at grain boundary of 316L steel (annealed at 750 °C/10 and 20h) was found.

(5) Although no depletion of Cr at grain boundaries of 316L steel after annealed at 750 °C/ 10 and 20 h was observed, the grain boundaries were attacked in both oxalic acid etch tests and Strauss tests, probably because of potential differences between Laves phases and the matrix and low Cr content in Laves phases.

(6) Weld HAZs of 316 and 316L steels, after experiencing welding processes with cooling rates higher than 0.07 °C/s, seem to be free of sensitivity to intergranular attack.

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