Solidification Crack Susceptibility in Weld Metals of Fully Austenitic Stainless Steels (Report VIII)†

– Effect of Nitrogen on Cracking in SUS 304 Weld Metal –

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Abstract

This investigation was undertaken to obtain a better understanding of the effect of nitrogen (N) on the solidification crack susceptibility of AISI (SUS) 304 austenitic-ferritic stainless steel weld metals, based on ductility curves at high temperatures, solidification process and microsegregation of phosphorus (P) and sulphur (S). It was confirmed that an increase in N had a detrimental effect on the cracking susceptibility of SUS 304 weld metals. This harmful effect of N was interpreted in terms of remarkable decreases in primary and residual δ-ferrite content and consequently an increased degree of microsegregation of P in particular.

KEY WORDS: (Austenitic Stainless Steels) (Weld Metals) (Hot Cracking) (Weldability Tests) (Nitrogen)

1. Introduction

Since nitrogen (N) enables yield strength of austenitic stainless steels to increase significantly and offers high modules at low temperatures[1),2)], nitrogen-modified grades containing 0.1 to 0.25%N have been standardized as SUS 304Nl, SUS 316N, etc. in Japanese Industrial Standard (JIS)[3)]. In the previous paper[4)] the effect of N on the cracking susceptibility of weld metals was investigated by using AISI 310S (JIS: SUS 310S) fully austenitic stainless steels, and according to ductility curves at high temperatures and simple hot cracking test result it was revealed that N had a neutral or slightly detrimental effect on the solidification cracking propensity of weld metals. On the other hand, N is a potent austenitizer[5), 6)], and thus it has been reported that an increase in N exerts a deleterious effect on the solidification cracking susceptibility of austenitic-ferritic stainless steel weld metals[5), 7)]. The influence of N was frequently discussed only in terms of the amount of residual δ-ferrite at room temperature and as a result it has been concluded that a good correlation exists between cracking susceptibility and δ-ferrite content[7)]. This may be right in that residual δ-ferrite content is more or less associated with solidification process and δ-ferrite content at high temperatures[8)]. There are, however, few fundamental results dealing with the effect of N on cracking susceptibility on the basis of ductility properties at high temperatures, solidification process and microsegregation.

Therefore, in this study, the effect of N on cracking susceptibility was investigated by the use of SUS 304 austenitic stainless steels, from the viewpoints of the interrelationship among ductility curve or BTR (brittleness temperature range), solidification process and microsegregation of phosphorus (P) and sulphur (S), in addition to the relations with residual δ-ferrite content.

2. Experimental Procedure

2.1 Materials used

The materials used are SUS 304 type austenitic stainless steels, the chemical compositions of which are shown in **Table 1**. SUS 304-P1 (0.056%P) and SUS 304-P2 (0.121%P) were employed to provide a better assessment of cracking susceptibility, since P was found to be one of the most harmful elements in weld metals of fully austenitic stainless steels in the previous reports[8), 10)]. N addition was made by utilizing Ar-5%N_2 and Ar-20%N_2 shielding gas in place of Ar gas during GTA welding. The

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Table 1 Chemical compositions of materials used.

Materials (SUS)	Composition (wt%)								
	C	Si	Mn	P	S	Cr	Ni	N	O
304-(A)	0.072	0.52	0.91	0.030	0.005	18.12	8.74	0.023	0.008
304-(B)	0.052	0.75	0.97	0.026	0.005	18.26	8.97	0.019	0.005
304-P1	0.05	0.58	0.94	0.056	0.006	18.50	9.10	0.026	—
304-P2	0.06	0.61	0.99	0.121	0.005	18.60	9.10	0.021	—
310S-(A)	0.060	0.55	0.97	0.001	0.003	24.62	20.78	0.020	0.005
310S-(B)	0.078	0.93	1.56	0.021	0.007	25.06	20.30	0.029	0.015
310S-(C)	0.063	0.69	1.15	0.023	0.003	24.60	20.20	0.052	0.043
310S-P1	0.06	0.77	1.04	0.055	0.005	23.75	19.55	0.033	—
310S-P2	0.06	0.79	1.05	0.109	0.003	23.90	19.80	0.033	—

N contents are in the range of about 0.015 to 0.025, 0.09 to 0.12 and 0.15 to 0.18% for shielding gases of Ar, Ar-5%N_2 and Ar-20%N_2, respectively. The N content in weld metal made in Ar shielding gas showed a tendency to be decreased compared with that in base metal.

2.2 Experimental procedure

The Trans-Varestraint test was performed on the plates of 3 mm thickness during GTA welding under Ar, Ar-5%N_2 or Ar-20%N_2 shielding gas, where the welding conditions were I=100 A, E=12.5 V and v=150 mm/min. The effect of N on cracking susceptibility was assessed by the BTR and ductility curve obtained by the Trans-Varestraint test. Some specimens were water-quenched during welding and in some cases immediately after testing to preserve solidification structures at high temperatures. Microstructures and the amounts of δ-ferrite, phosphides and sulphides in weld metals were investigated by light microscope (LM), scanning electron microscope (SEM) and energy dispersive X-ray spectroscope (EDX).

3. Experimental Results and Discussion

3.1 Effect of nitrogen or δ-ferrite on cracking susceptibility of SUS 304 type stainless steels

3.1.1 Brittleness temperature range and ductility curve

Figure 1 shows the effect of N on the ductility curves for commercially available SUS 304 weld metals. Residual δ-ferrite content in room temperature microstructures was shown for reference. In the case of SUS 304-(A), the BTR at $\epsilon \fallingdotseq 2.5\%$ increased from 55 to 150°C and $_B\epsilon_{min}$ decreased drastically from 0.7 to 0.25% with an increase in N content from about 0.02 to 0.09% (a decrease in δ-ferrite content from 4.8 to 0.5%). As the N content increased to about 0.15% (δ-ferrite content: 0%), the BTR at $\epsilon \fallingdotseq 0.2$ to 2.5% expanded to about 100 to 155°C and $_B\epsilon_{min}$ lowered to about 0.1%. That is to say, it was found that the feature of the ductility curve for SUS 304-(A) fully austenitic weld metals with 0.15%N came to correspond to that for SUS 310S weld metal. On the other hand, in the case of SUS 304-(B) weld metals the increase in N content from 0.015 to 0.15% (the decrease in δ-ferrite content from 5.6 to 1.3%) made the BTR increase from about 50 to 75°C and simultaneously rendered $_B\epsilon_{min}$ lower from about 0.7 to 0.35%. By comparing SUS 304-(A) with SUS 304-(B) at 0.15% of N content, N itself has little effect on cracking susceptibility but the effect of N on a reduction in δ-ferrite amount may be more essential, as has been stated before[7].

Figure 2 indicates the effect of N on SUS 304-P1 (0.056%P) and SUS 304-P2 (0.121%P) weld metals, which were produced in Ar and Ar-20%N_2 shielding gas. As the N content was increased from 0.025 to 0.18% and from 0.02 to 0.16%, in the case of SUS 304-P1 the BTR at $\epsilon \fallingdotseq 2.5\%$ widened extremely from 70 (δ-ferrite content: 5.5%) to 265°C (δ-ferrite: 1.2%) and $_B\epsilon_{min}$ lowered from about 0.7 to 0.08%, while in the case of SUS 304-P2 the BTR at $\epsilon \fallingdotseq 2.5\%$ broadened from 75 (δ-ferrite content: 6%) to 145°C (δ-ferrite content: 3.2%) but $_B\epsilon_{min}$ decreased to a considerable extent from 0.7 to 0.3%. Consequently the effect of δ-ferrite is considered to be more predominant than the effects of N and P.

The BTR, one of the most reasonable indexes to assess cracking susceptibility[9], was summarized in the relation between residual δ-ferrite content and P content in **Fig. 3**, as is often the case with residual δ-ferrite[11]. The data[9,10] on the BTR of SUS 310S type weld metals are also given at 0% of δ-ferrite for reference, and a region showing the BTR of 120°C and less is obtained as a region resistant to cracking, because practical cracking can be diminished for the BTR of 120°C or below[10,12]. It is judged that commercial SUS 304 weld metal (0.026%P)

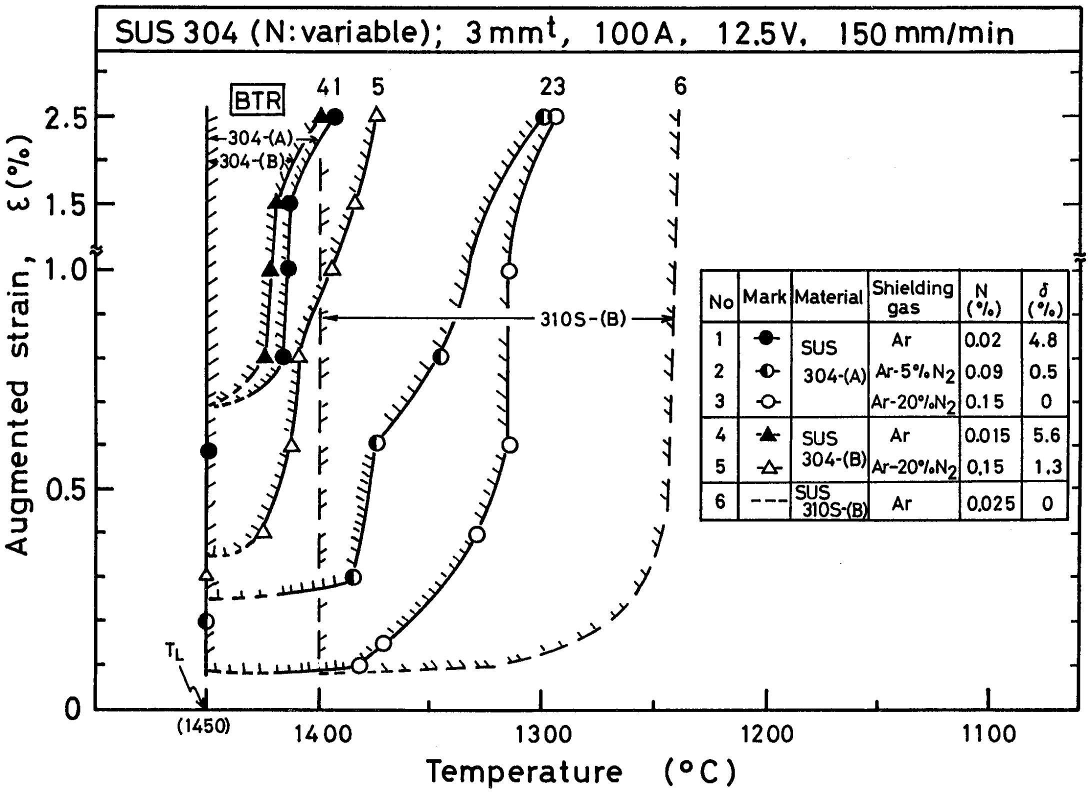


Fig. 1 Effect of N content or δ-ferrite content on ductility curves of BTR for weld metals of commercial SUS 304 steels.

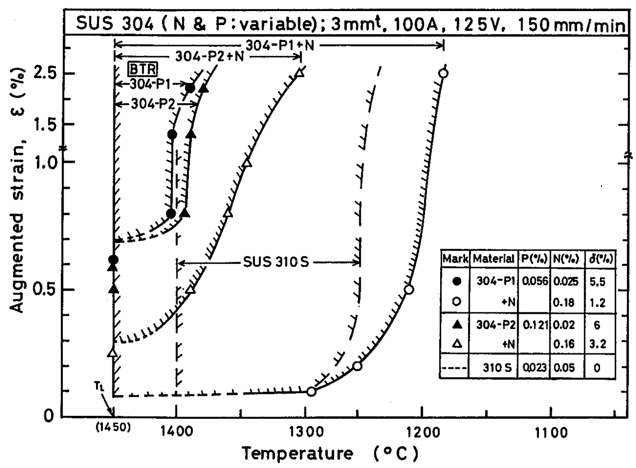


Fig. 2 Effect of N content or δ-ferrite content on ductility curves of BTR for weld metals of SUS 304 type steels containing 0.056 and 0.121%P.

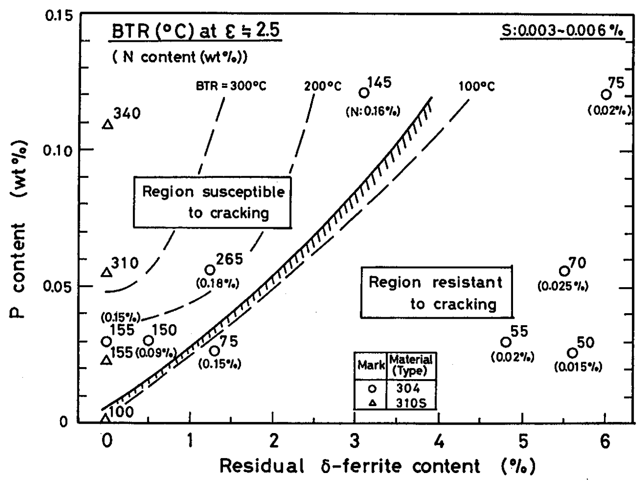
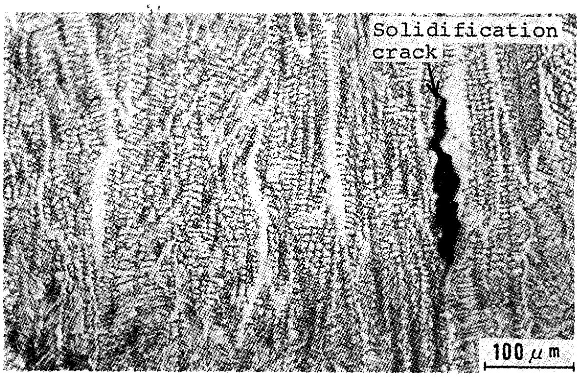


Fig. 3 Relationship between residual δ-ferrite content (%), P content and BTR at $\epsilon = 2.5\%$ in SUS 304 and for reference SUS 310S, showing region resistant to cracking.

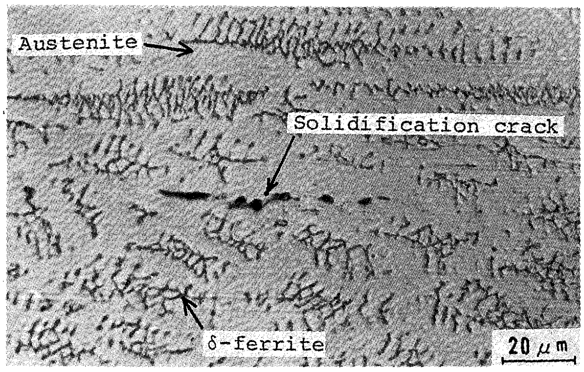
containing about 1.3% δ-ferrite is more resistant to cracking than modified SUS 310S fully austenitic weld metal with about 0.001%P and 0.003%S. In other words, the cracking susceptibility of fully austenitic weld metal achieved by extremely low content of P and S can be easily obtained by producing SUS 304 with more than 1.5% δ-ferrite if δ-ferrite is permitted. It is also understood from this figure that the presence of δ-ferrite in microstructures of SUS 304 weld metals signifies great importance in reducing cracking problems, and that P is highly detrimental to cracking in weld metals containing little or no δ-ferrite. These are in good agreement with the previous papers[9), 10)].

3.1.2 Effect of N on location of cracking

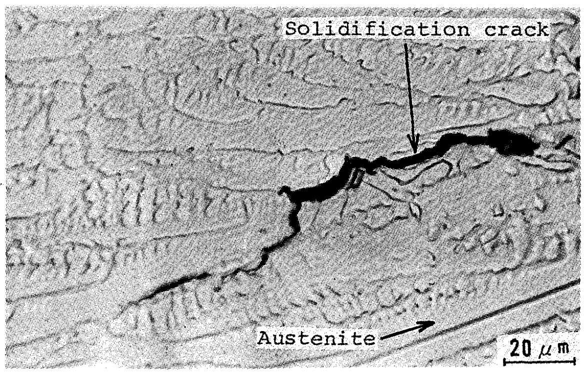
Weld beads were quenched by watering immediately after the Trans-Varestraint test during welding in Ar or Ar-20%N_2 shielding gas, to observe the relationship between microstructures and location of cracks. **Figure 4**(a), (b) and (c) show the microstructures of SUS 304-(B) weld metal with 0.015%N at $\epsilon=1.5\%$, SUS 304-(B) weld metal with 0.15%N at $\epsilon=0.6\%$ and SUS 304-(A) weld metal with 0.15%N at $\epsilon=0.15\%$. In Fig. 4 cracks are seen black, and are relatively small because of smaller augmented-strains. In Fig. 4(a) δ-ferrite is also black in tangled shape and the other white part is austenite matrix, and in the right side a crack is seen to have occurred mainly at the solidification grain boundary of primary δ-ferrite, to have propagated along it, to have turned aside to the boundary between δ-ferrite and austenite and to have been instantly arrested there, as discussed and explained in detail in the previous reports[12,13]. It should be also noted that a little wider white regions are recognized in the upper part of the crack and in other three parts corresponding to solidification grain boundaries of primary δ-ferrite. That is to say, in SUS 304 weld metal the mechanism of backfilling[14,15] or healing[16] should be taken into consideration as the effect of δ-ferrite in preventing cracking in addition to the mechanism to arrest cracking propagation due to the transformation of δ-ferrite to austenite. The mechanism of backfilling or healing should be investigated in more detail.



(a) SUS304-(B) (0.015%N, ϵ≒1.5%)



(b) SUS304-(B) (0.15%N, ϵ≒0.6%)



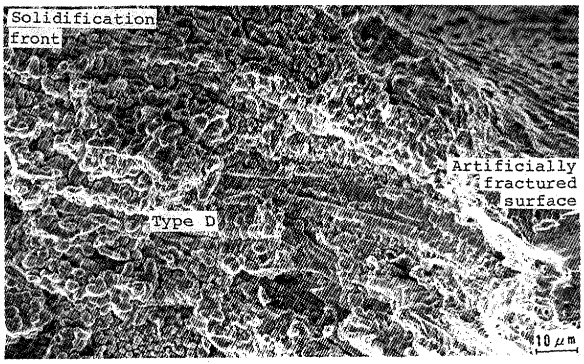
(c) SUS304-(A) (0.15%N, ϵ≒0.15%)

Fig. 4 Microstructures of SUS 304 weld metals subjected to Trans-Varestraint test, showing cracking in SUS 304-(B) (in Ar, at $\epsilon=2.5\%$) (a), in SUS 304-(B) (in Ar-20%N, at $\epsilon=0.6\%$) (b) and in SUS 304-(A) (in Ar-20%N, at $\epsilon=0.15\%$) (c).

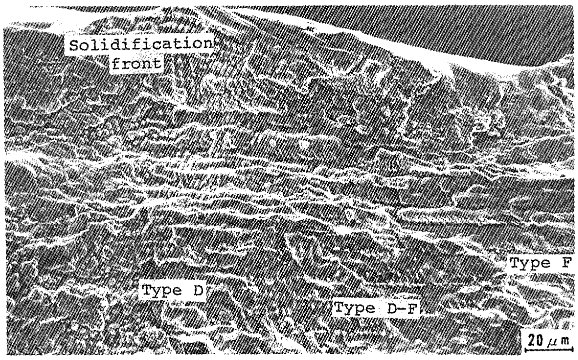
It is observed in SUS 304-(B) weld metals made in Ar-20%N_2 shielding gas that primary solidification of δ-ferrite took place and resulted in lacy δ-ferrite in microstructures, and cracking occurred along the solidification grain boundary of primary δ-ferrite as shown in Fig. 4(b). On the other hand, as shown in Fig. 4(c), in SUS 304-(A) weld metal produced in Ar-20%N_2 gas, primary solidification of austenite occurred and cracking was found along solidification grain boundaries of primary austenite, which is just the same behavior as SUS 310S mentioned previously[17].

Subsequently the surfaces of cracks which occurred at $\epsilon=3.75\%$ in the Trans-Varestraint test were observed by the SEM to reveal the effect of N on cracking surface morphology. **Figure 5**(a), (b) and (d) show the crack surfaces of SUS 304-(A) weld metals produced in Ar (0.02%N; 4.8%δ), Ar-5%N_2 (0.09%N; 0.5%δ) and Ar-20%N_2 (0.15%N; 0%δ) shielding gas, respectively. In the case of 0.02%N in Fig. 5(a), Type D dendritic pattern[13,17] can be observed all over the surface.

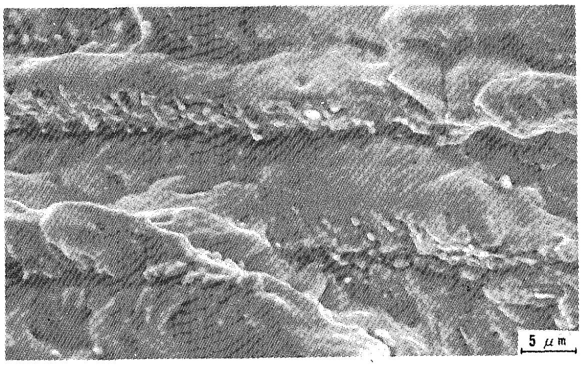
In the case of 0.09%N in Fig. 5(b), the crack surface apparently shows the three typical features of dendritic Type D, transient Type D-F and smooth, flat Type F surface according to a drop in temperature[17]; however, Type F region is narrow, and small projections are observed on Type D-F and Type F surface, as shown in higher magnification in Fig. 5(c). This feature is a little different from the crack surfaces of SUS 310S weld metal, and it is interpreted from the behavior that cracking propagates along the migrated grain boundary pinned by δ-ferrite transforming into austenite, although in the case of a smaller augmented-strain cracking is arrested at the temperature range of Type D-F surface. On the other hand, for 0.15%N in Fig. 5(d), the crack surface is charac-



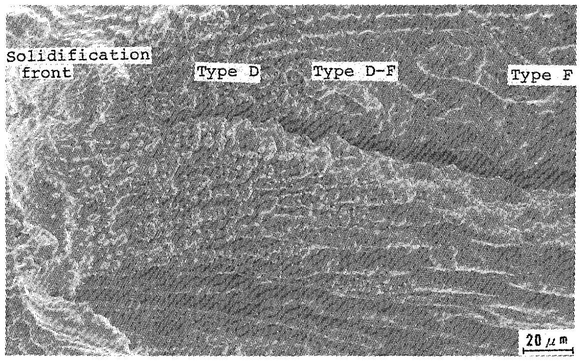
(a) Ar



(b) Ar-5%N_2



(c) Ar-5%N_2



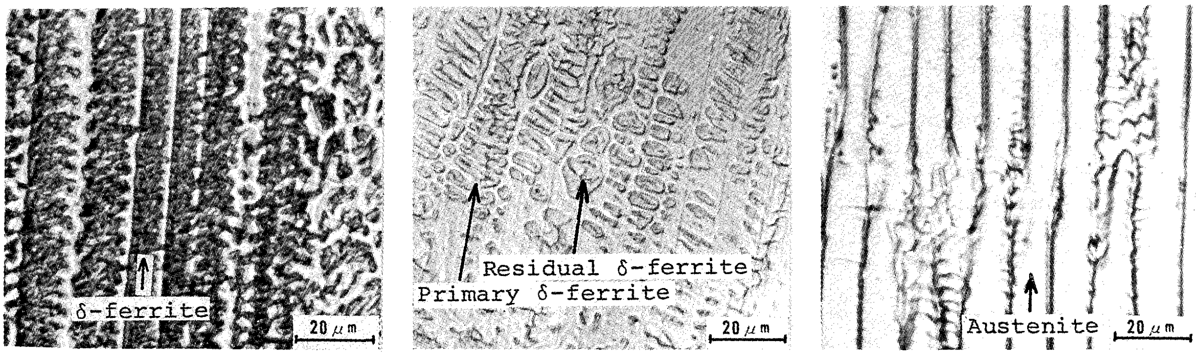
(d) Ar-20%N_2

Fig. 5 Surfaces of cracks occurring at ϵ=3.75% in Trans-Varestraint test in SUS 304-(A) weld metals made in Ar(a), Ar-5%N_2(b), (c), and Ar-20%N_2(d).

terized by the variation in Type D, Type D-F and Type F surface with a temperature drop, which is similar to the feature of SUS 310S crack surface[17]. It is regarded from the observation of crack surfaces that austenite solidifies to the last stages of solidification in SUS 304-(A) weld metal containing 0.15%N.

3.2 Effect of nitrogen on solidification microstructure and microsegregation

Sheets of 3 mm thickness during welding were quenched into water to observe the effect of N on microstructures at high temperatures during welding. **Figure 6**(a), (b) and (c) show the quenched microstructures of SUS 304-(A) weld metals welded in Ar, Ar-5%N_2 and Ar-20%N_2 shielding gas, respectively. Besides, **Figure 7** shows their EDX results indicating relative intensity of Fe, Cr and Ni on quenched weld metals. From Fig. 6(a) and Fig. 7(a) in the case of Ar shielding gas, it is judged that primary solidification of δ-ferrite occurs and subsequently peritectic/divorced-eutectic reaction would take place. From Fig. 6(b) and Fig. 7(b) in the case of Ar-5%N_2 gas,



(a) Ar (b) Ar-5%N_2 (c) Ar-20%N_2

Fig. 6 Microstructures of SUS 304-(A) weld metals quenched during welding in shielding gases of Ar(a), Ar-5%N_2(b) and Ar-20%N_2(c).

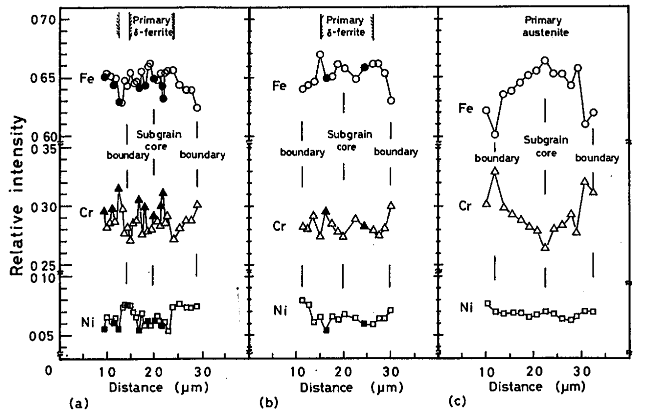
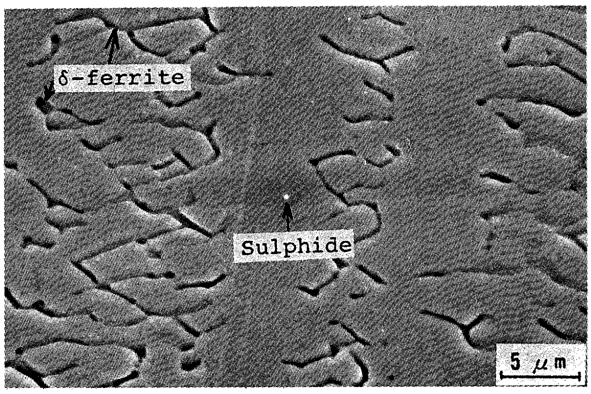


Fig. 7 Relative intensity of Fe, Cr and Ni distribution across cells in SUS 304-(A) weld metals quenched during welding in (a) Ar, (b) Ar-5%N_2 and (c) Ar-20%N_2.

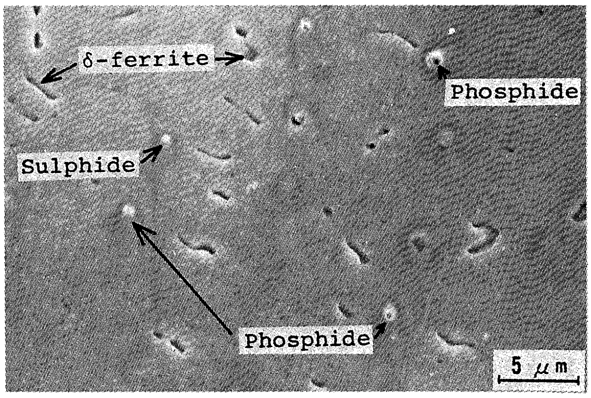
primary solidification of δ-ferrite, in which residual δ-ferrite is obviously observed, occurs as well, but the amount is smaller. On the other hand, in the case of Ar-20%N_2 gas, as shown in Fig. 6(c) and Fig. 7(c), fully austenitic microstructure is observed, and Cr and Ni segregate at solidification boundaries.

From the above results it is clearly understood that N is a powerful austenitizer and exerts a strong effect on a change from primary solidification of δ-ferrite to that of austenite in solidification process.

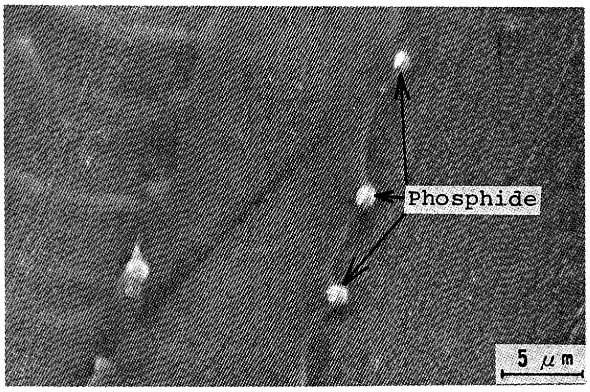
Figure 8(a), (b) and (c) show SEM microstructures of SUS 304-(A) weld metals welded in Ar (0.02%N; 4.8%δ), Ar-5%N_2 (0.09%N; 0.5%δ) and Ar-20%N_2 (0.15%N; 0%δ) shielding gas. Inclusions in the photos were observed in the granular, globular or elliptical shape and were identified as phosphides or sulphides by the EDX. The sizes of phosphides and sulphides are about 0.3 to 1 μm and 0.2 to 0.4 μm, respectively (The size of sulphides appears to be smaller because the S contents are smaller.) The counts of them were measured as an index of microsegregation of P and S. The result is tabulated in the relation of N content in **Table 2**. This table also shows a summary of the amount of primary δ-ferrite and residual δ-ferrite, and the BTR and $_B\epsilon_{min}$ obtained by the Trans-



(a) Ar



(b) Ar-5%N_2



(c) Ar-20%N_2

Fig. 8 SEM microstructures of SUS 304-(A) weld metals made in shielding gases of Ar(a), Ar-5%N_2(b) and Ar-20%N_2(c), showing formation of δ-ferrite, phosphides and sulphides.

Table 2 Summary of the effect of N content on primary and residial δ-ferrite content, amount of phosphides and sulphides, BTR at $\epsilon \fallingdotseq 2.5\%$, and $_B\epsilon_{min}$.

Materials (SUS)	Shielding gas	N content (%)	Primary δ-ferrite (%)	Residual δ-ferrite (%)	Inclusions (counts) enriched in			Sum ($\frac{counts}{0.1 mm^2}$)	BTR (°C) at ε= 2.5%	$_B\epsilon_{min}$ (%)
					P	P & S	S			
304-(A) (0.030%P, 0.005%S)	Ar	0.020	70	4.8	0	0	3	3	55	0.7
	Ar-5%N	0.09	30	0.5	6	2	7	15	150	0.25
	Ar-20%N	0.15	0	0	25	9	4	38	155	0.08
304-(B) (0.026%P, 0.005%S)	Ar	0.015	75	5.6	0	0	4	4	50	0.7
	Ar-20%N	0.15	50	1.3	1	6	5	12	75	0.35
310S-(B) (0.021%P, 0.007%S)	Ar	0.03	0	0	32	5	7	44	160	0.08

Varestraint test. According to the result of this table, the increase in N content reduces the amount of primary δ-ferrite and consequently both decreases the amount of residual δ-ferrite and increases the formation counts of phosphides in particular and sulphides at solidification grain boundaries and cellular dendritic boundaries, which results in increased cracking susceptibility. By comparing SUS 304-(A) weld metal made in Ar-5%N_2 gas with SUS 304-(B) weld metal made in Ar-20%N_2, an obvious difference in the BTR and $_B\epsilon_{min}$ in ductility curves can be recognized between them although microsegregation is considered to be almost equal. It is therefore interpreted that a little difference in the process of solidification and subsequent cooling, namely the presence of above 50% of primary δ-ferrite at the completion of solidification, exerts a great influence on the cracking resistance of SUS 304 stainless steels.

4. Conclusions

The following conclusions were drawn by the evaluation of the effect of N on the ductility curve or the BTR of SUS 304 weld metals and by the observation of microstructures:

(1) It was confirmed that N had such a strong effect on the condition of austenite formation that about 5% of residual δ-ferrite in the weld metal was decreased to about 1.5 to 0% with the addition of 0.09 to 0.18%N.

(2) It was revealed that the decrease in residual δ-ferrite content resulted from a decrease in primary δ-ferrite content from 70 – 75% to 0 – 50%, as the case may be.

(3) The degree of microsegregation of phosphides and sulphides, especially phosphides, was found to increase significantly with a reduction in primary δ-ferrite content during solidification.

(4) As the N content was gradually increased to about 0.2% in SUS 304 weld metal, the cracking susceptibility approached and finally was similar to that of SUS 310S weld metal. In fully austenitic weld metal of SUS 304 the location of cracking, the features of crack surfaces and the degree of microsegregation were almost the same as those in SUS 310S.

(5) The effect of N on cracking susceptibility was interpreted in terms of the variation in the solidification process. That is to say, the enhanced crack susceptibility was attributed to decreased primary δ-ferrite content, and consequently decreased residual δ-ferrite content and increased extent of microsegregation of P.

(6) Suggestions are made that more than 1.5% of residual δ-ferrite is required to reduce practical cracking problems in commercially available SUS 304 containing 0.04%P or below, and that the presence of more than 50% primary δ-ferrite at the last stages of solidification may be more important.

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