

Influence of Si Content on Hot Cracking Sensitivity of Weld Heat Affected Zone in Incoloy 800

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Synopsis

The influence of Si content on the cracking sensitivity of the weld heat affected zone (HAZ) in the Si bearing Incoloy 800 was examined and assessed through hot ductility characteristics using the weld thermal cycle simulator. The result showed that the HAZ cracking sensitivity of Si bearing Incoloy 800 was low enough when the Si content is less than 2%, but when 2% is exceeded, the brittle temperature range (BTR) was expanded and the material became sensitive to HAZ cracking. The type of cracking found within the BTR is the intergranular liquation cracking, and some enrichment of the solute elements was observed in the liquated grain boundary. In materials with less than 2% of Si with low cracking sensitivity, this liquation boundary can be observed as the ghost boundary which is caused by the migration of the boundary during weld thermal cycle. In the materials with Si content of more than 2% with high cracking sensitivity, no ghost boundary was found, and the precipitation of Cr carbide was found in the liquated grain boundary, suggesting that the liquation of the grain boundary was also affected by the constitutional liquation due to Cr carbide. The phenomena described above were discussed from the viewpoint of the diffusion theory.

1. Introduction

In the welding of the austenitic Cr-Ni stainless steel (referred to as Cr-Ni steel), Si is known to be a harmful element that increases the hot cracking tendency of the weld metal¹⁾. Already in 1952, Bishop and others²⁾ proposed that the maximum content of Si in the 19%Cr-13%Ni steel (with 0.12-0.16%C) should be 0.3%. Gooch and others³⁾ also pointed out that Si content should be kept under 0.3% in the 310s (25%Cr-20%Ni) material.

Such limitation of Si content, however, was made chiefly in the hot cracking tendency of the weld metal, and it is not clear if the same limitation should be applied to the hot cracking of the weld heat affected zone (HAZ). Although the influence of Si content in the HAZ cracking of the Cr-Ni steel has been judged as harmful just like in the weld metal, the judgement is based on the analogical inference from the experience of weld metal. There seem to have been no report that made a positive study of the influence of Si on the HAZ cracking of Cr-Ni steel.

Si has been traditionally used as the element to add to heat-resistant materials as it improves, for instance the resistance to scaling when several percent is added to the Cr-Ni steel. Recently the high-Si Cr-Ni steel that contains 2 to 4% of Si is gathering attention again as materials for automobile exhaust gas treatment devices, heating system and nuclear waste material treatment facilities. When using these materials, the measures prevent the hot cracking of the weld metal is taken, such as by adjusting the base metal composition so the γ -phase contains several percent of α -phase to form a dual phase even in the fusion welding without using the filler metal, or by adjusting the filler metal composition, so the weld metal contains several percent of α -phase when used with fully austenitic base metal. Thus, the hazard of the hot cracking of weld metal is somehow

avoided, but whether this is sufficient for preventing the HAZ cracking is still unknown. Therefore, it seems quite significant to clarify the HAZ cracking tendency of the high-Si Cr-Ni steel because demands for high-Si materials is increasing lately.

For this purpose, the author chose the Incoloy 800 as the material that retains fully austenitic structure even when used as filler metal, and conducted hot ductility tests using the weld thermal cycle simulator in order to examine the influence of Si on the HAZ cracking sensitivity.

2. Materials

Table 1 shows the chemical composition of the Incoloy 800 used in this test. The content of Si was kept under 4%, which is the content found in commercial high-Si Cr-Ni steel. The materials were melted in a high frequency furnace and cast as 10Kg square ingots, and then hot rolled into round bars with a diameter of 10mm (forging ratio:9) and then they were solution treated at 1100°C.

Table 1 Chemical composition of materials tested

No	Aim of Si cont.	C	Si	Mn	P	S	Cr	Ni
1	0.4	0.058	0.43	0.76	0.011	0.005	20.89	32.04
2	0.6	0.052	0.76	0.75	0.014	0.008	20.24	32.34
3	0.9	0.049	0.96	0.76	0.012	0.007	20.42	32.34
4	1.5	0.048	1.59	0.82	0.010	0.009	20.93	32.34
5	2.0	0.048	2.23	0.79	0.012	0.009	20.84	32.34
6	3.0	0.041	3.12	0.85	0.014	0.007	20.95	32.60
7	4.0	0.044	4.32	0.84	0.013	0.006	20.97	32.08

3. Experimental procedure

The materials described in Section 2 were machined into necessary amount of bar-shaped test pieces of 8mm in diameter and 70mm in length. Then the test pieces were put into a weld thermal cycle simulator and were exposed to the thermal cycle that has a heating rate of 180°C/sec., a cooling rate of 80°C/sec., duration at the maximum temperature (T_{max}) of 3 sec., and rapid tensile tests were conducted on heating and on cooling¹⁾. In the hot ductility tests in general, the ductility (reduction of area was adopted in this test) gradually increases with the rise of temperature, and at a certain temperature the ductility suddenly drops. This temperature is the liquation initiation temperature (LIT), which is estimated to take place at about half value of maximum ductility¹⁴⁾. As the temperature continues to rise, there comes a point "NDT" at which the ductility becomes nil, but the material still retains some strength. At a higher temperature "NST", even the strength becomes nil. In the hot ductility test, the test pieces were heated at once to NST, and tensile tests were conducted on cooling to determine the temperature range between the solidus and the temperature at which the ductility starts to recover rapidly from nil (called L.BTR). This temperature range is called the brittle temperature range (BTR), and is used to assess the sensitivity to HAZ cracking which is usually caused by the liquation in the grain boundary, often called the "liquation cracking". In many cases the HAZ cracking found in Cr-Ni steel is this liquation cracking. Another type of hot cracking in the HAZ is the so-called ductility dip cracking, which is seen on cooling after the ductility recovers once and then drops again. But the ductility in ordinary Cr-Ni steel reaches several tens of percent even when in the ductility dip temperature range, and therefore very few cases of ductility dip cracking in the HAZ are found. Taking all this into account, first of all LIT, NDT, NST and L.BTR were obtained from tests on heating and cooling in the thermal cycle. Then BTR was calculated from the L.BTR and solidus which is measured by thermal analysis, similar to the Jackson's method¹²⁾, and the influence of Si content on the HAZ cracking sensitivity in Incoloy 800 was assessed from the relation between the BTR and the Si content.

4. Results

Figure 1 summarize the results of hot ductility test, and Figure 2 shows LIT, NST, L.BTR and solidus together with the relationship between the Si content.

As Figure 2 shows, the BTR of Incoloy 800 that contains 2% or less of Si is constant at about 700, narrower than the critical BTR of 1000 adopted by the authors as the criteria in another study of HAZ cracking sensitivity of Cr-Ni steel¹³⁾. It seems safe to say this material has little hazard the HAZ cracking when the Si content is less than 2%, however, with 3% or more of Si, BTR starts to expand and exceeds the critical value of 1000, increasing the hazard of HAZ cracking. Yet, the increase of the Si content in excess of 3% shows only a mild widening of BTR, and therefore further increase of Si seems not likely to cause an extreme increase of the liquation cracking in the HAZ.

5. Discussion

5-1 Microstructure of fractured area

In order to discuss the cracking tendency from the aspect of the materials' property, firstly the microstructure of the fractured area that failed at NST was observed. Consequently, it was observed that the grain boundary was etched somewhat thickly, clearly indicating that the grain boundary liquation had taken place. It was also apparent that the failure occurred along this liquated boundary. This means that the brittleness within BTR was caused by the grain boundary liquation. As to the type of

fracture, the materials with less than 2% of Si judged to be without hazard of cracking generated fine crackings that run roughly parallel to the fractured surface, whereas no such phenomenon was observed in the materials containing more than 3% of Si judged to have a hazard of cracking. This suggests that the stress of the fracture affected the area several grains distant from the fractured surface in case of materials containing 2% or less of Si. It seems that the strength at the moment of fracture differs depending on the Si content even when the temperature is at NST, at which the boundary is in the same liquid film state.

The grain boundary at NST is thought to be completely covered by liquid film of roughly an uniform thickness amounting to several percent of liquid

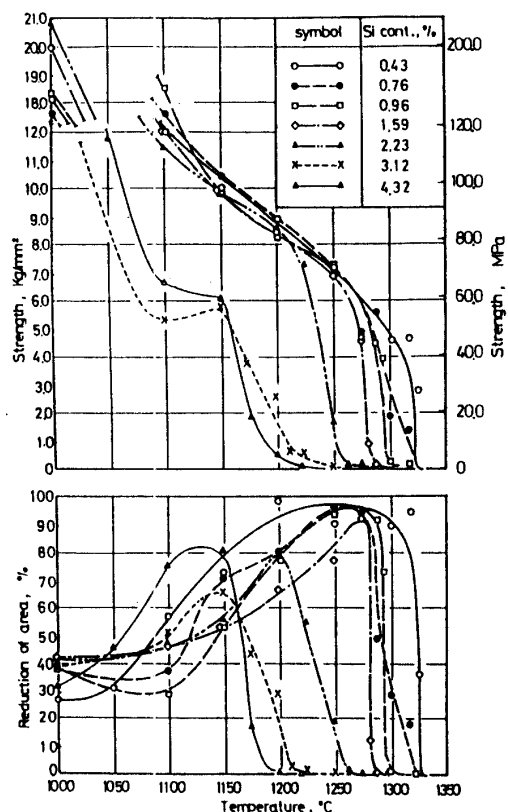


Figure 1. The effect of Si content on hot ductility characteristics of Incoloy 800, tested on cooling of thermal cycle.

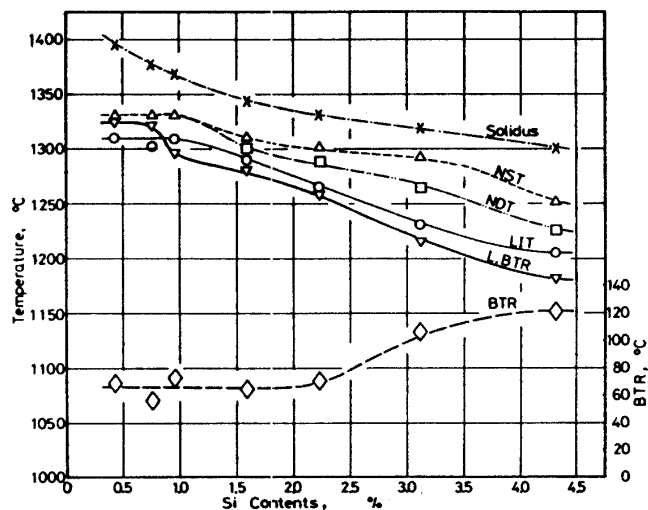


Figure 2. Effect of Si contents on solidus, NST, NDT, LIT, L.BTR, and BTR of Incoloy 800.

in volume⁶⁾. In this liquid film state, according to Chadwicks and others⁷⁾, the tensile strength σ of a metal can be represented by the next equation;

$$\sigma = \frac{2\gamma_L}{h} \quad (1)$$

in which h stands for thickness of the liquid film, and γ_L stands for surface tension of the liquid film. In the equation (1), σ is a function of γ_L when " h " is constant, and γ_L in turn changes depending on the composition of the liquid film. Therefore the tensile strength of a metal in liquid film state is affected by the composition of the liquid film provided that the thickness of the film is constant. In light of this, the different type of fracture at NST by the content of Si seems to suggest that the composition of the liquid film differs depending on the Si content, and a close connection between the composition of the liquid film and the cracking sensitivity can be expected.

Figure 3 is a partial enlargement of the microstructure of the fractured area. As Figure 3 shows, in the low-Si material containing less than 2% of Si, the trace of migration of the boundary (ghost boundary) occurred on cooling in the thermal cycle was observed apart from

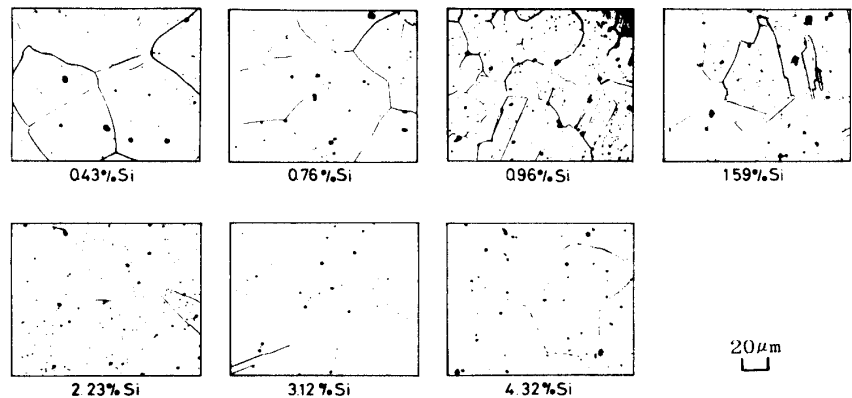


Figure 3. Photomicrographs of Incoloy 800 with various Si content, solution treated at 1100°C.

the liquated boundary. In the ghost boundary, carbides were found which must have precipitated on cooling. In the material which has more than 3% of Si, such ghost boundary was hardly found, and there was a clear difference in microstructure between the low-Si range (less than 2%) and the high-Si range (more than 3%). Such difference in the migration of the grain boundary depending on the content of Si is attributable to the difference of difficulty and velocity of migration after the grain boundary solidified.

For the grain boundary to separate from the solidified liquation boundary and to migrate, the driving force " P " of the boundary migration has to be larger than the force " F " of the solute elements in the liquated boundary that serves to pull back the boundary. The latter force, F , is described by Lücke and others⁸⁾ as follows;

$$F = n_B' \cdot f_0 \quad (2)$$

in which n_B' stands for the number of solute atoms per unit area, f_0 stands for the maximum attraction between the solute atom and the grain boundary (dyne). If the lattice constant is represented by " a " and the concentration of the solute atom in the grain boundary is represented by C_B , n_B' is described as follows;

$$n_B' = \frac{4\sqrt{2}}{a} C_B \quad (3)$$

As described later, C_B is proportionate to the average concentration of the solute C_0 . That means the separation of the grain boundary from liquated boundary is the more difficult when C_0 is larger, and it is imaginable that the boundary of the material with higher Si content (more than about 3%) was more difficult to migrate than the one in the low Si material. On the other hand, even if the boundary did separate itself from the liquated boundary, that does not cause actual migration when the migration velocity is

extremely slow. According to Lücke and Detert⁸⁾, the migration velocity G of the grain boundary in an alloy is described in the following equation;

$$G = \frac{P}{kT} \cdot \frac{a^2}{4\sqrt{2}} \cdot \frac{D_0^i}{C_0} \exp\left(-\frac{Q_D^i + V}{kT}\right) \quad (4)$$

in which P stands for the driving force in the boundary (N/cm^2), k for Boltzmann constant (J/k), T for absolute temperature ($^\circ\text{K}$), D_0^i for frequency factor of the lattice diffusion coefficient of the solute atom ($\text{cm}^2/\text{sec.}$), Q_D^i for activation energy for lattice diffusion of the solute atom (J) and V for interactive energy between grain boundary and solute atoms (J). As is apparent from the equation (4), the migration velocity of the grain boundary " G " is in inverse proportion to the average solute concentration C_0 in the material, and it is imaginable that the high solute concentration of the high-Si materials slowed the migration velocity of the grain boundary and made the migration difficult. Since the grain boundary migration is the diffusion process, the distance of diffusion (x_i) of the solute atoms on cooling of the weld thermal cycle can be approximated by the step function as follows;

$$\sum x_i = \sum \sqrt{D_0 \exp\left(-\frac{Q}{RT_i}\right) t_i} \quad (5)$$

in which D_0 stands for diffusion constant (m^2/sec), Q for activation energy of diffusion (J/mol), R for gas constant ($\text{J/mol}\cdot\text{K}$), T_i for temperature in the i -th step when weld thermal cycle is divided into n for step function approximation (K), t_i for time at i -th step when the weld thermal cycle is divided into n for step function approximation. As is shown in the results described in Section 4, the so-called low-Si material with less than 2% of Si has the high solidifying point, and therefore the solute atom has enough temperature and time to move in diffusion. Thus, it is considered that the boundary of the low Si material is, on cooling of the weld thermal cycle, easily removed and can migrate the weld thermal cycle, easily removed and can migrate from the region of liquation boundary, and consequently, the liquated boundary remained as a ghost boundary in the microstructure. On the other hand, in case of the high-Si materials with more than 3% of Si, the solute atoms do not have enough time and temperature to diffuse due to low solidifying point, and consequently the liquated boundary cools down without migrating, causing the carbides to precipitate in the liquated boundary. As is described above, the properties of the liquated boundary (observed by the optical microscope) directly relevant to the cracking sensitivity are all related to the composition of the liquated boundary, and there seems to be a very close relationship between the cracking sensitivity and the composition of the liquated boundary.

5-2 Scanning analysis of the liquated boundary using EPMA

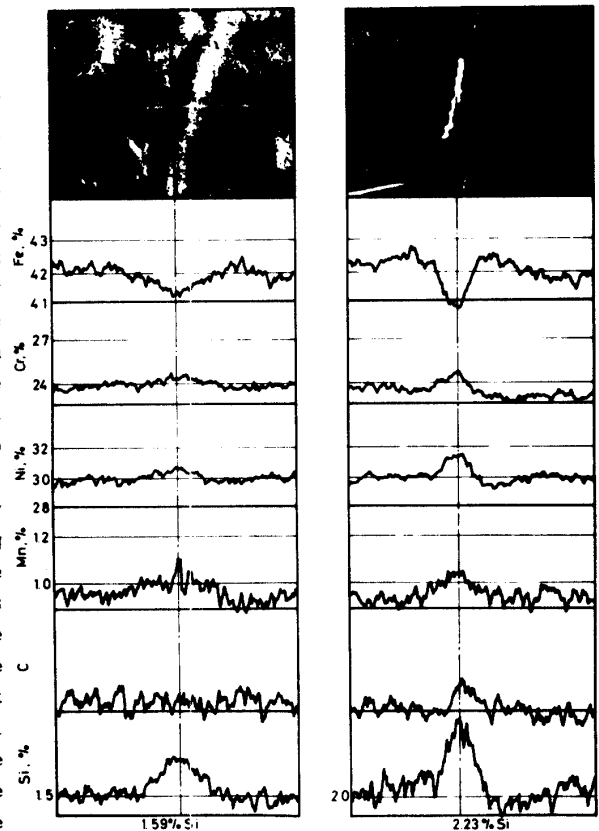


Figure 4. Example of scanning electron probe microanalysis of grain boundary of Incoloy 800 with different Si contents.

For each material heated to NST, cross-scanning of the liquated boundary was conducted at 10 points to examine the changes in concentration of Fe, Cr, Ni, Mn, C, and Si using EPMA. Figure 4 shows an example of the result of scanning and Figure 5 shows the result of the statistical treatment⁹⁾ of the 10 scans to represent the change of composition from the matrix with a probability of 95.4%. As is clear from Figure 5, Si, Ni, C, Cr, and Mn increased in the liquated boundary as the Si content increased. Enrichment of these element was remarkable in the materials containing more than 3% of Si, and especially C, Ni, and Si increased considerably.

Cahn¹⁰⁾ assumed that the chemical potential μ_c of the solute atom in a grain boundary μ_b can be described in the following equation;

$$\mu_b = kT \ln C_B(x) + V(x) + C \quad (6)$$

and calculated the concentration of the solute atom C_B when the grain boundary is migrating at a constant velocity ($G : G > 0$) and stationary ($G = 0$), and showed that they are described as the function of the distance from the center of the grain boundary x .

If $G > 0$;

$$C_B = C_0 G \exp \left\{ - \frac{V(x)}{kT} - G \int_{x_0}^x \frac{d\eta}{D_i(\eta)} \right\} \times \int_{-\infty}^x \exp \left\{ \frac{V(\xi)}{kT} + G \int_{x_0}^{\xi} \frac{d\eta}{D_i(\eta)} \right\} \frac{d\xi}{D_i(\xi)} \quad (7)$$

If $G = 0$;

$$C_B = C_0 \exp \left\{ - \frac{V(x)}{kT} \right\} \quad (8)$$

From equation (7) and (8), if $G > 0$, concentration of solute atom C_B is asymmetric to the center of the grain boundary, and if $G = 0$, C_B is symmetric. At a constant temperature, the concentration of the solute atom C_B is higher when the boundary is stationary because the stationary boundary becomes closer to the equilibrium concentration than in the migrating boundary. Since migration of boundary in the Si bearing Incoloy 800 is found in the materials with less than 2% of Si and not in those with more than 3%, the equation (7) is applicable to the materials with less than 2% of Si and the equation (8) to those with more than 3%.

The quantitative rendering will be disregarded here. But qualitatively, it is well imaginable that the liquated boundary of the materials containing more than 3% of Si having higher concentration of the solute atom is stationary, and has more solute atoms than in the materials with less than 2% of Si. Results shown in Figure 5 match this estimation.

Thus, the increase of the solute atom concentration lowers the melting point in the grain boundary, and when the material contains more than 3% of Si, the melting (or solidifying) point is markedly decreased. The increase of the cracking sensitivity in such high-Si materials can be explained as due to the enrichment of the solute atom in the grain boundary

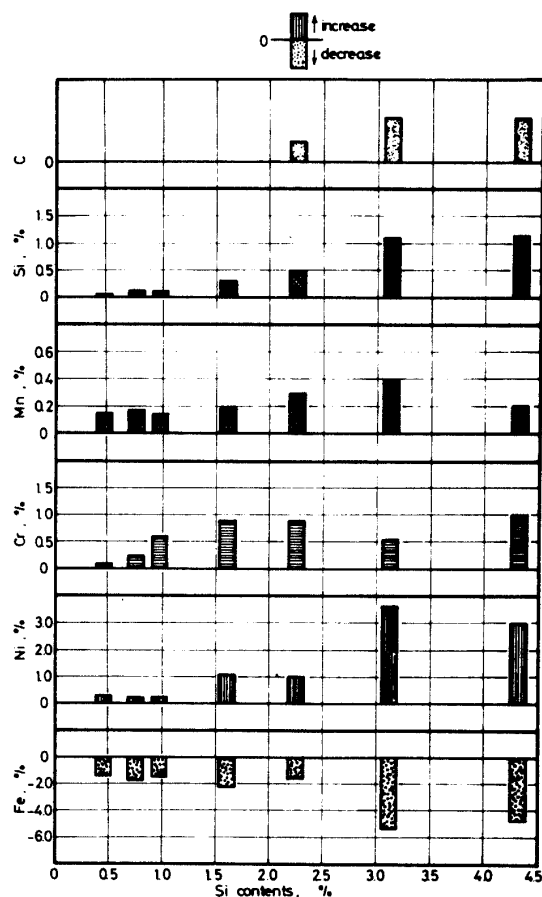


Figure 5. Composition changes from bulk structure in liquated grain boundary in Si bearing Incoloy 800, heated to NST.

that occurs during the weld thermal cycle. In the liquated boundary of such materials, precipitation of carbides (identified as Cr_{23}C_6) was found. Such precipitation has been observed with the test specimen with more than 3% of Si (solution treated at 1100°C). This Cr_{23}C_6 in the test specimen is known to generate the so-called constitutional liquation in the process of the thermal cycle¹⁰⁾. The absence of grain boundary migration in the high-Si materials suggests that the constitutional liquation took place along with the enrichment of the solute atoms.

Although the Incoloy 800 used in this test have an increased amount of the silicate with the increase of the Si content, these silicates are not involved in the constitutional liquation in the boundary as is seen in Figure 6, and they seem to have no direct influence on the cracking sensitivity.

6. Summary and conclusion

In order to examine the influence of Si on the HAZ cracking sensitivity in the high-Si austenitic Cr-Ni steel, the Incoloy 800 was chosen as the material for hot ductility test. The results obtained were as follows:

- (1) The HAZ cracking sensitivity of the Incoloy 800 does not change when the content of Si is under about 2%, but the brittle temperature range (BTR) is increased when this level is exceeded, and at more than 3%, the critical BTR (100°C) is exceeded and the material becomes sensitive to HAZ cracking.
- (2) The dominant type of cracking is liquation cracking of the grain boundary.
- (3) In the HAZ of the materials with Si content of less than about 2%, the migration boundary is observable, but not in the high-Si range of more than about 3%. This seems to be caused by the increasing difficulty of migration when the solute atom concentration gets higher, and by consequent slowing down of the migration velocity, and by such factors as the low solidifying point (or melting point) in the liquated grain boundary which caused lack of time and temperature sufficient for the solute atoms to diffuse.
- (4) Enrichment of the solute atoms was observed in the liquated boundary, and especially so in the materials with more than about 3% of Si. This seems to be caused by the lack of grain boundary migration together with the high solute atom concentration.
- (5) The enrichment of the solute atom in the grain boundary lowers the melting point of grain boundary and widens BTR. The fact that BTR is especially wide in the materials with more than about 3% of Si is due to the marked degree of enrichment.
- (6) Precipitation of the carbide (Cr_{23}C_6) was observed in the liquated boundary of the materials with more than about 3% of Si that showed a increase in the cracking sensitivity. Cr_{23}C_6 was also detected in the grain boundary of as-received test piece (solution treated at 1100°C , water cooled). As the Cr_{23}C_6 in the base metal is known to lead to the constitutional liquation, the increase of the cracking sensitivity in the materials with more than about 3% of Si is also caused by constitutional liquation originated by a Cr carbide, in addition to the enrichment of solute atom described above.
- (7) Silicates were observed in the test specimen, and these showed a

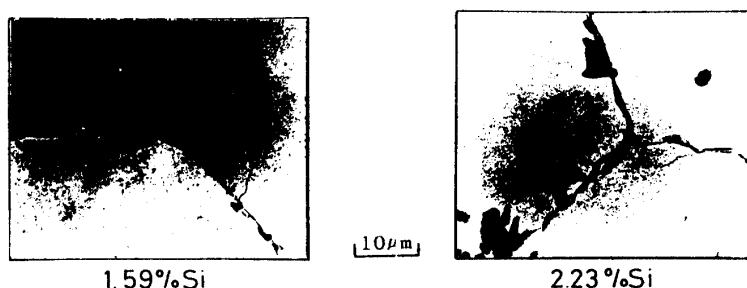


Figure 6. Examples of electron micrographs of liquated boundary by carbon extraction replica.

tendency to increase with the increase of Si in the material. However, these showed little tendency to affect the constitutional liquation and can be regarded as having no influence on the HAZ cracking.

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