Chromium Nitride Precipitation Behavior in Weld Heat-affected Zone of High Nitrogen Stainless Steel

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High nitrogen stainless steels (HNS) containing about 1 mass% (%) nitrogen without adding Mn were manufactured by pressurized electro-slag remelting method. The chromium nitride precipitation behaviors at the weld heat-affected zone (HAZ) of HNS with different compositions were investigated. We also evaluated the localized corrosion resistance of the welded joints.

The nitride precipitates were identified as Cr$_2$N containing a small amount of Mo and Fe by TEM/EDS analyses. Time–temperature–precipitation curves of some HNS were obtained. It was found that for 23Cr–4Ni–2Mo–1N steel, aging for 2 s in the temperature range between 1173 K and 1373 K caused Cr$_2$N precipitation and decreased the critical pitting corrosion temperature (CPT) in the 6% FeCl$_3$ solution. Precipitation was delayed to 4 s by decreasing the nitrogen content from 1 to 0.8% and was further slightly delayed to over 5 s by adding the pre-deformation followed by the thermal treatment of 23Cr–4Ni–2Mo–0.8N steel.

Precipitation at the HAZ was accelerated by a continuous thermal cycle test compared to the prediction based on the additivity rule, and that the existence of a ferritic phase affected acceleration was estimated.

Joints welded by laser and minimum heat input conditioned plasma arc welding showed a 348 K CPT in a 6% FeCl$_3$ solution and no crevice corrosion occurred at 308 K in artificial seawater. The CPT dropped notably against holding time above 1073 K at the HAZ. The CPT drop was slightly relieved by decreasing nitrogen content in the base metal from 1 to 0.8%.

KEY WORDS: nitrogen; stainless steel; Cr$_2$N; time–temperature–precipitation curve; welding; heat-affected zone; localized corrosion resistance.

1. Introduction

High nitrogen-bearing stainless steels (HNS) containing from 0.5 to 1 mass% (%) nitrogen have been developed. Adding nitrogen to austenitic stainless steels suitably consolidates localized corrosion resistance and improves mechanical properties. HNS present problems in weldability however, due to nitrogen supersaturation in the solid state below the solvent temperature range and, in some cases, in the molten state. The HNS those basic compositions were defined as 24%Cr–6%Mn–17%Ni–4.5%Mo–0.5%N steel$^1$) or 24%Cr–3.5%Mn–22%Ni–7%Mo–0.5%N steel$^1$) were studied their weldability. It was reported that these HNS could be welded without large degradation in localized corrosion resistance by using a Ni base alloy containing high Cr and Mo as a welding consumable. No significant precipitation occurred at the heat-affected zone (HAZ). Time–temperature–precipitation (TTP) curves of 18%Cr–18% Mn–0.6–1.2%N steels,$^{31}$ 18%Cr–18%Mn–2%Mo–0.9%N steel,$^{31}$ 19%Cr–5%Mn–5%Ni–0.7%N steel$^{31}$ and 21%Cr–25%Mo–0.9%N$^{30}$ steel showed that it took over 10 s to precipitate chromium nitride, so it was estimated that precipitation would not occur within heat cycle of the conventional arc welding.

HNS containing about 1% nitrogen without addition of Mn are being developed. The basic composition of one of these HNS is 23%Cr–4%Ni–2%Mo–1%N. This HNS was manufactured by a pressurized electro-slag remelting (ESR) method. This HNS showed superior localized corrosion resistance and mechanical properties, e.g., total resistance to crevice corrosion in seawater, and tensile strength of 1 000 MPa.$^7$) The weldability of these HNS is an important engineering issue. Blowhole generation in the weld metal was suppressed and localized corrosion resistance was improved by using high Cr and Mo containing welding consumables.$^9$) An other important problem in welding Cr–Ni–Mo–N system steels is to suppress the very rapid precipitation of chromium nitride at the HAZ, due to the temperature range at which the austenitic single phase is stable, which is narrower than that of Cr–Mn–N system steels. Chromium nitride precipitation severely degrades localized corrosion resistance. In this report, we evaluated and compared chromium nitride precipitation behavior in several kinds of compositional Cr–Ni–Mo–N steels at the HAZ and evaluated localized corrosion resistance of these HNS welded joints.
2. Experimental Procedure

2.1. Materials

Table 1 shows compositions of base metals used in our study. 23%Cr–4%Ni–2%Mo–1%N, 23%Cr–4%Ni–2%Mo–0.8%N and 27%Cr–3%Ni–1%N steels were melted by pressurized ESR followed by forging, hot rolling and solution treatment. Table 2 shows compositions of welding consumables used. Steels containing high Cr and Mo were used as welding consumables in order to increase nitrogen solubility in the molten state and also to improve the localized corrosion resistance of the weld metal.

2.2. Experimental Methods

Welding of HNS was performed by gas tungsten arc (GTA) welding, CO2 laser welding and plasma arc welding under the conditions of one-side, one-pass, butt welding. The HNS base metals were machined to plates of 4 mm thickness and supplied for welding tests. Table 3 summarizes welding conditions. Using a high-frequency induction furnace with helium gas jet attachment, we rapidly heated and cooled a HNS rod specimen 3 mm in diameter and 10 mm long to define the TTP curve. The localized corrosion resistance of welded joints was evaluated using pitting corrosion tests and crevice corrosion tests. In the pitting corrosion test, the specimen was immersed in 6% FeCl3 solution for 86.4 ks and the critical pitting corrosion temperature (CPT) was measured by step-wise increasing of the test temperature at 5 K intervals. In the crevice corrosion test, the welded joint specimen was drilled at the center of the weld bead and a couple of multiple crevice device was attached to the top and bottom surface of the specimen, which was then immersed in artificial seawater at 308 K and maintained at a potential for 172.8 ks. The critical crevice corrosion potential (CCP) was measured by increasing the potential at 50 mV intervals.

3. Results and Discussion

3.1. Structure and Composition of the Precipitates

At the grain boundary in the HAZ of 23%Cr–4%Ni–2%Mo–1%N steel welded by GTA, precipitates were observed and resulted in the degradation of localized corrosion resistance. The microstructure and compositions of the precipitates were analyzed by TEM/EDS using the specimen aged at 1 173 K for 2 s. The results of images by optical microscope and TEM, electron diffraction pattern and EDS analysis were shown in Fig. 1. This specimen had already degraded pitting corrosion resistance. Granular precipitates about 20 nm in diameter were observed at the grain boundary. The ratio of the length occupied by these precipitates on the grain boundary was about 20%. From the electron diffraction pattern, precipitates were determined to be \( \beta \)-Cr2N. From EDS analysis, these precipitates included small amounts of Mo and Fe.

3.2. Cr2N Precipitation Behavior at HAZ

Figure 2 shows TTP curves for different compositional HNS and their phase diagrams calculated by thermo-calc. Here, \( T_{\text{Cr}_2\text{N}} \) and \( T_{\text{Cr}} \) represent the maximum temperature at which \( \text{Cr}_2\text{N} \) and ferritic phase precipitate equilibriumly, as shown in the phase diagrams. \( \Delta \text{Mcr} \) represents the difference in the Cr content between in the austenitic phase equilibrated with \( \text{Cr}_2\text{N} \) and in the bulk of the base metal at 1 173 K. The Cr content in the austenitic phase equilibrated with \( \text{Cr}_2\text{N} \) was calculated by thermo-calc. In the present study, occurrence of the precipitation was judged by a drop of over 5 K of CPT in 6% FeCl3 solution compared to that of the solution-treated base metal. In 23%Cr–4%Ni–

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Fig. 1. Results of TEM/EDS analyses for precipitates at grain boundary. (23Cr–4Ni–2Mo–1N steel, aged at 1173 K for 2 s)

Fig. 2. Phase diagrams and TTP curves of HNS.
2%Mo–1%N steel, aging at the temperature range from 1 173 K to 1 373 K for 2 s caused CPT drop compared to that of the base metal. The aging time that caused CPT drop could be slightly delayed to as long as 4 s by reducing the N content from 1 to 0.8%. In the case of 27%Cr–3%Ni–1%N steel, the minimum aging time that caused the CPT drop was not changed from that of 23%Cr–4%Ni–2%Mo–1%N steel but the temperature range was narrower. In 25%Cr–20%Ni–0.3%N steel, precipitation required aging exceeding 10 s. The shape of the TTP curve is expected to be changed by not only TsCr2N but also by ΔMcr. The aim of 27%Cr–3%Ni–1%N steel was to decrease TsCr2N. According to the phase diagram calculated by thermo-calc, TsCr2N of 27%Cr–3%Ni–1%N steel was estimated as 1 300 K that was 120 K lower than that of 23%Cr–4%Ni–2%Mo–1%N steel. In contrast, ΔMcr at 1 173 K was estimated at 6.9%, about 2% grater than that of 23%Cr–4%Ni–2%Mo–1%N steel. The large value of ΔMcr introduces the large gradient of the Cr content near the Cr2N precipitates. This causes the fast Cr diffusion from the bulk of the base metal to Cr2N precipitates. It was estimated that from this reason, the minimum aging time of 27%Cr–3%Ni–1%N steel causing the CPT drop was not changed much compared to that of 23%Cr–4%Ni–2%Mo–1%N steel. Due to these results, lower TsCr2N and smaller ΔMcr had to be taken into account in selecting the HNS composition in order to suppress the sensitivity of degradation in corrosion resistance by Cr2N precipitation.

### 3.3. The Effect of Mechanical and Thermal Treatment on TTP Curve

It was reported that the intergranular corrosion of austenitic stainless steels caused by chromium carbide precipitation could be improved by increasing the frequency of coherent and low interfacial energetic boundaries introduced by annealing twins.10 The method to induce many twin boundaries was cold rolling followed by heat treatment. In this investigation, the effect of the mechanical and thermal treatment on chromium nitride precipitation behavior was examined. After 23%Cr–4%Ni–2%Mo–0.8%N steel specimens were mechanically reduced their thickness by cold rolling, then they were annealed at 1 473 K. TTP curves of mechanically and thermally treated specimens were compared to those of only solution treated base metal specimens. The results are shown in Fig. 3. The aging time to cause CPT drop by chromium nitride precipitation was delayed over 5 s by two treatments, one was 5% cold rolling followed by annealing at 1 473 K for 36 ks and the other was 4 times of 5% cold rolling followed by the annealing at 1 473 K for 1.8 ks. The grain boundary of mechanically and thermally treated specimens was characterized by Electron Back Scattering Pattern/Orientation Imaging Microscopy analyses as shown in Fig. 4. From these analyses, the line fraction of the coincidence site lattice grain boundaries with low sigma (Σ=29), meaning high coherency and low grain boundary energy, for mechanically and thermally treated specimens was characterized by Electron Back Scattering Pattern/Orientation Imaging Microscopy analyses as shown in Fig. 4. From these analyses, the line fraction of the coincidence site lattice grain boundaries with low sigma (Σ=29), meaning high coherency and low grain boundary energy, for mechanically and thermally treated specimens was estimated to become slightly larger compared to that of the only solution treated base metal specimens. But a random grain boundaries still existed at some fractions. The mechanism of the precipitation delay cannot be explained by the fraction of coincidence site lattice grain boundaries alone. It is estimated that grain size and alloy element segregation at a grain boundary might also influence the delay in the precipitation. Further detailed study is needed on the structure and compositions of...
the grain boundaries to decisively confirm the mechanism.

3.4. Precipitation Behavior under Continuous Cooling

TTP curves mentioned above show precipitation behavior under isothermal heat treatment. To study the weldability of HNS, it is important to understand chromium nitride precipitation behavior under continuous cooling. Precipitation behavior is reportedly subject to the additivity rule if precipitation rate is controlled by a thermally active process such as Cr diffusion. If so, the occurrence of precipitation can be predicted from the TTP curve and thermal cycle of welding. The thermal cycle was divided into short time segments, $\Delta t$, and the precipitation time corresponding to the temperature of each segment $C_T$ was determined from the TTP curve. If the summation of $\Delta t/C_T$ exceeded unity, it was estimated that precipitation causing degradation of the corrosion resistance occurred.

As mentioned above, the mechanically and thermally treated 23%Cr–4%Ni–2%Mo–0.8%N steel required over 5 s to precipitate chromium nitride and to degrade pitting corrosion resistance. On the other hand, laser welding or plasma arc welding at a restricted condition enabled welding at a high cooling rate. By these welding methods, the holding time above 1 073 K could be decreased to less than 5 s, for example. We expected that the HAZ formed in laser welding or plasma arc welding using the mechanically and thermally treated 23%Cr–4%Ni–2%Mo–0.8%N steel showed no precipitation and as good pitting corrosion resistance as that of the base metal. But the result was not consistent with our expectation as shown in Fig. 5. In Fig. 5, a example of thermal cycle of plasma arc welding was shown. This was one of small heat input conditioned and the holding time above 1 073 K was restricted to 3 s. The TTP curve and CPT of the welded joints compared to that of the base metal were also shown in Fig. 5. In this case, the base metal was 23%Cr–4%Ni–2%Mo–0.8% steel 5% rolled and annealed at 1 473 K for 36 ks. From the TTP curve and the thermal cycle of welding, $\Sigma \Delta t/C_T$ that was derived from the additivity rule was 0.4 and less than unity. But this welded joint showed a 343 K CPT and pitting at the HAZ, though the base metal did not pit below 348 K, the maximum temperature examined thus far. So this welded joint caused the CPT drop compared to that of the base metal. This means that the additivity rule cannot apply to Cr$_2$N precipitation behavior of this steel.

It was reported that in the stainless steel weld metal having a duplex austenitic and ferritic structure, chromium carbide precipitated faster at lower temperature range compared to that of an austenitic single structure. This was probably due to lower chromium carbide/ferrite interfacial free energy and faster Cr diffusion in the ferrite phase than in the austenitic phase. Cr$_2$N precipitation may have been accelerated for the same reason. Actually in this investigation, the ferritic phase was observed. Figure 6 shows the thermal cycle of a plasma arc welding and the results of SEM and X-ray diffraction analyses in the HAZ. This specimen was held above 1 073 K for 8.5 s, a holding time slightly longer than that shown in Fig. 5. It was estimated that the deviation from the additivity rule and the acceleration of Cr$_2$N precipitation were due to the existence of the
ferritic phase. The TTP curve of the HNS was estimated by summing the nucleation time \( t \) and the growth time \( T_D \). The former was derived from kinetics theory of solid-solid nucleation at grain boundary,\(^\text{13)\} \) and the latter was estimated under the assumption that the growth of Cr\(_2\)N precipitates was controlled by one-dimensional Cr diffusion from inside a grain to the grain boundary.\(^\text{9)\} \) The nucleation time \( t \) and the growth time \( T_D \) are expressed as follows:

\[
\frac{t}{H_1} = \frac{8}{k} \cdot T \cdot \frac{s}{M / \text{Cr}_2\text{N}} \cdot a^4 \left( \frac{K_j / L_j}{V_{\text{Cr}_2\text{N}}^2 \cdot D \cdot X_0^{\text{Cr}}} \right)
\]

\(
\Delta G_v = k \cdot T \cdot \ln \left( \frac{X_0^{\text{Cr}} / X_i^{\text{Cr}}}{V_{\text{Cr}_2\text{N}}} \right)
\)

\( k \): Boltzmann number (1.38 \times 10^{-23} \text{ J/K})

\( T \): Temperature

\( \sigma_{\text{MOL}} \): Interphase-interfacial free energy (\( \gamma / \text{Cr}_2\text{N} \) 0.67 J/m\(^2\),\(^\text{14)\} \) \( \alpha / \text{Cr}_2\text{N} \) 0.13 J/m\(^2\).\(^\text{12)\} \) Under the assumption that those value are equal to those of chromium carbide.)

\( V_{\text{Cr}_2\text{N}} \): Moller volume of Cr\(_2\)N

\( \alpha \): Lattice parameter (\( \gamma / \text{Cr}_2\text{N} \) 3.6 \times 10^{-10} \text{ m}, \( \alpha / \text{Cr}_2\text{N} \) 2.4 \times 10^{-10} \text{ m})

\( D \): Cr diffusion coefficient (in \( \gamma 8.0 \times 10^{-6} \exp(-58500/1.987/T) \text{ m}^2/\text{s}^{15) \}) \) in \( \alpha 4.6 \times 10^{-5} \exp(-52500/1.987/T) \text{ m}^2/\text{s}^{15) \})

\( X_0^{\text{Cr}} \): Moller fraction of Cr in the matrix

\( X_i^{\text{Cr}} \): Moller fraction of Cr equilibrated with Cr\(_2\)N

\( K_j, L_j \): Geometrical parameter (Here, assumed as \( K_j = 0.31, L_j = 0.5 \))

\( T_D = L^2 R^2 \left( \pi / D \right) \times \left( X_{\text{Cr}_2\text{N}}^{\text{Cr}} \cdot M_{\text{Cr}} / (\rho \cdot M_{\text{Cr}}) \right)^2 / (4(X_0^{\text{Cr}} - X_i^{\text{Cr}})^2)
\)

\( L \): Width of precipitates (20 nm)

\( R \): Ratio of length occupied by precipitates on grain boundary (Here, assumed as 0.2.)

\( \rho_{\text{Cr}_2\text{N}} \): Density of Cr\(_2\)N (6.51 \times 10^3 \text{ kg/m}^3)

\( \rho_{\text{matrix}} \): Density of matrix (7.7 \times 10^3 \text{ kg/m}^3)

\( M_{\text{Cr}_2\text{N}}^{\text{Cr}} \): Moller fraction of Cr in Cr\(_2\)N

\( M_{\text{Cr}} \): Atomic mass of Cr (52 g/mol)

\( M_{\text{matrix}} \): Mean atomic mass of the matrix (56 g/mol)

The estimated precipitation time of Cr\(_2\)N precipitates is plotted as the TTP curves in Fig. 7. Here, the condition shown in Fig. 1 is assumed to be the initial stage degrading the pitting corrosion resistance. Equilibrated compositions were estimated by Thermo-calc. The solid line shows the TTP curve assuming that only austenitic phase exists, and dashed line assuming that both austenitic and ferritic phases exist at the start of aging. In the latter case, the short time as about 5 s holding at the temperature range from 973 to 1 173 K, that was relatively lower temperature range than the former case, cause precipitation due to acceleration.
caused by the existence of the ferritic phase. In Fig. 7, the same thermal cycle of the plasma arc welding shown in Fig. 5 was shown again. $\Sigma \Delta t / C_t$ of this thermal cycle derived from the additivity rule became unity when this double nose shape TTP curve was taken into account. This predicted the possibility of CPT degradation of the welded joint by Cr$_2$N precipitation and agreed well with the experimental data. So it was estimated that the HAZ of HNS passing through the heat cycle of welding caused ferritic phase precipitation at a relatively higher temperature range near 1273 K followed by the acceleration of Cr$_2$N precipitation at a relatively lower temperature range near 1073 K.

Precipitation of the ferritic phase was estimated to be harmful to suppress Cr$_2$N precipitation. So the maximum equilibrium ferritic phase precipitation temperature ($T_{Sf}$) must be decreased. As mentioned in Sec. 3.1, it is necessary to take into account the maximum equilibrium Cr$_2$N precipitation temperature ($T_{S_{Cr2N}}$) and the difference of Cr content between in the austenitic phase equilibrated with Cr$_2$N and in the bulk of the base metal ($\Delta M_{cr}$) and also $T_{S_a}$ in selecting HNS composition.

### 3.5. Localized Corrosion Resistance of the HNS Welded Joints

CPT and CCP of HNS joints welded by CO$_2$ laser welding, plasma arc welding and GTA welding were measured. The results are shown in Figs. 8 and 9. The base metals of 23%Cr–4%Ni–2%Mo–1%N steel, 23%Cr–4%Ni–2%Mo–0.8%N steel, 27%Cr–3%Ni–1%N steel and 25%Cr–20%Ni–0.3% N steel were used. In all cases of these experiments, it was found that pitting corrosion and crevice corrosion caused at HAZ. Because steels containing high amounts Cr and Mo were used as welding consumables, the pitting corrosion resistance equivalent (PRE) of the weld metal was controlled at a higher value than that of the base metal, even taking into account the segregation of alloy elements. The horizontal axis in Figs. 8 and 9 shows the holding time above 1073 K at the HAZ. Only the laser welded joint and the plasma welded joint under the smallest heat input condition showed 348 K CPT, the maximum temperature in this experiment in the case of using 23%Cr–4%Ni–2%Mo–1%N steel as the base metal. The larger heat input plasma welded joints and the GTAW joints showed remarkable decrease in CPT. In 23%Cr–4%Ni–2%Mo–0.8%N steel, it gave a little improvement of the CPT drop against the holding time over 1073 K at the HAZ compared to the 23%Cr–4%Ni–2%Mo–1%N steel. The trend in the CPT drop with the mechanically and thermally treated 23%Cr–
4%Ni–2%Mo–0.8%N steel as the base metal showed further improvement compared to that of untreated steel having the same composition. In 25%Cr–20%Ni–0.3%N steel base metal, CPT was relatively low at small heat input conditions but the drop of the CPT at large heat input conditions was very small.

The CCP of HNS joints showed the same trend as the CPT. The laser welded joint and the plasma welded joints under smaller heat input condition showed no crevice corrosion at 308 K in artificial seawater in the case of 23%Cr–4%Ni–2%Mo–1%N steel, 23%Cr–4%Ni–2%Mo–0.8%N steel and 27%Cr–3%Ni–1%N steel.

4. Conclusion

The weldability of HNS containing about 1 mass% N with no Mn added is an important engineering issue. In this report, Cr$_2$N precipitation behavior at the HAZ of the HNS was investigated. Obtained results are summarized as follows.

(1) The precipitate at the grain boundary in HAZ was identified as Cr$_2$N containing slight amounts Mo and Fe by TEM/EDS analyses.

(2) TTP curves of the HNS were defined experimentally. From these, Cr$_2$N precipitation occurred in very short aging. In 23%Cr–4%Ni–2%Mo–1%N steel, aging for 2 s at the temperature range from 1 173 to 1 373 K caused precipitation and degraded localized corrosion resistance.

(3) Precipitation was slightly delayed to 4 s by decreasing nitrogen content in the base metal from 1 to 0.8 mass%.

(4) Precipitation was further slightly delayed to over 5 s by using mechanically and thermally treated 23%Cr–4%Ni–2%Mo–0.8%N steel as the base metal.

(5) Precipitation at the HAZ treated in a continuous thermal cycle was accelerated compared to prediction by the additivity rule accounting for the TTP curve and thermal cycle. The existence of the ferritic phase was estimated effectively to accelerate the precipitation.

(6) The welded joints by laser or the smallest heat input conditioned plasma arc welding showed a 348 K CPT in 6% FeCl$_3$ solution and no occurrence of crevice corrosion at 308 K in artificial seawater. The CPT dropped notably against holding time above 1 073 K at the HAZ. The CPT drop was slightly relieved by decreasing nitrogen content in the base metal from 1 to 0.8%.

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