## Abridged version

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Weld Zone Toughness of ERW Ti-Stabilized 11% Cr Steel Pipe for Automobile Exhaust Systems

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The toughness of weld zone of ERW Ti-Stabilized 11% Cr steel pipe produced by a "chance-free" bulge roll (CRB) forming pilot mill was investigated. The weld zone, particularly HAZ, had its toughness markedly lower than in the base metal. The reason for this embrittlement was examined by investigating precipitant behavior of Ti and Cr through laboratory-scale various heat treatments. As a result, the embrittlement of HAZ is considered to have been due to the following metallurgical factors: (1) Cr-carbides formed during cooling by the partial dissolution of TiC at high welding temperatures, (2) solute C supersaturated by rapid cooling after welding, and (3) undissolved TiN remaining even at high welding temperatures. Consequently, a reduction in the carbon and nitrogen content would improve HAZ toughness of ERW pipe.

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# Weld Zone Toughness of ERW Ti-Stabilized 11% Cr Steels Pipe for Automobile Exhaust Systems





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#### 1 Introduction

Increasing quantities of stainless steel are now being used in automobile exhaust systems. It is known that titanium-stabilized 11% chromium steels (type 409 steel) have good formability, oxidation resistance and corrosion resistance, pipe in these steels being welded to manufacture automobile exhaust systems. Since exhaust systems are usually one of the last components to be designed, their shape is often complex to fit into limited available space. Consequently, exhaust systems are often subjected to severe bending and expansion stresses,

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while the material must possess good formability.

Defects in welded pipe during forming are often ascribed to insufficient toughness in the welded area, and many studies<sup>1-7)</sup> have so far been conducted on the toughness of TIG-welded pipe. However, these studies have mainly been related to steels with a chromium content of 16 wt.% or more, including SUS 430, and there have been few studies on low-chromium materials such as 11% Cr steels.

In addition, few studies have been made on the toughness of pipe produced by electric resistance welding (ERW). ERW pipe is produced at far higher welding speeds than TIG pipe, before being subjected to upsetting, and is consequently subjected to heating and rapid cooling over a very short period. A metallurgical examination of the material characteristics of the weld zone in titanium-stabilized steels that undergo this thermal history and in the area near such a weld zone is very important for understanding the formability of ERW pipe.

In this report, an examination was made of the effects of carbon, nitrogen and titanium on the toughness of the weld zone in ERW type 409 steel pipe regarding its thermal history and the precipitation behavior of these elements.

<sup>\*</sup> Originally published in Kawasaki Steel Giho, 22(1990)4, 251-256

## **2** Experimental Method

All the specimens of type 409 steel were vacuummelted in a 30-kg laboratory furnace, before being hot rolled and then cold rolled to a thickness of 1.5 mm according to the usual methods. Final annealing was conducted at 930°C for 3 min, and the chemical composition of each specimen is given in **Table 1**. Material specimens A, B, C, E, and F were formed into ERW pipe 42.7 mm in diameter by a CBR (chance-free bulge roll) forming pilot mill for a toughness investigation. The pipes were formed at a speed of 20 m/min and with an upset of 0.9 mm.

To simulate the thermal history of the ERW pipes sheets of the relevant specimen were heated in a direct current furnace to 900-1250°C at a heating rate of 50°C/s. After heating, each sheet was quenched with water or  $N_2$  gas to change the cooling rate before the toughness was investigated. Toughness was also investigated for samples heat-treated for 30 s in the temperature range of 500 to 1000°C after the initial heating to 1250°C and water quenching.

The toughness was evaluated by conducting a Charpy impact test (5 kg). Test pieces of 1.5 mm in thickness were obtained from the cold-rolled steel sheets and heat-treated steel sheets, and a 2-mm V-notch was made in the rolling direction after sampling at right angles to the rolling direction.

The ERW pipe was cut into round slices and developed; after flattening, the excess weld metal was cut off, and a 2-mm V-notch was made in the rolling direction at the center of the weld zone (seam), 0.25 mm from the seam, 0.50 mm from the seam, or in the base metal  $180^{\circ}$  opposite to the weld zone ( $180^{\circ}$  zone), before the Charpy impact characteristics were investigated.

The microstructure of each specimen was observed under an optical microscope after etching in picric and hydrochloric acid. The grain size was determined from photographs of the microstructures according to the

 Table 1
 Chemical compositions of specimens of type

 409
 (wt. %)

	C	Cr	Ti	N	
A	0.006	11.8	0.14	0.011	
В	0.011	11.7	0.14	0.010	
С	0.015	11.8	0.14	0.010	
D	0.020	11.5	0.15	0.010	
Е	0.011	11.8	0.14	0.004	
F	0.010	11.8	0.15	0.013	
G	0.020	10.9	0.28	0.010	
н	0.011	11.4	0.23	0.011	

Note(1) Other compositions average varied in ranges as follows: 0.4~0.6 wt.% Si, 0.3~0.5 wt.% Mn, 0.03 wt.% P, 0.005 wt.% S, 0.002~0.005 wt.% O cross-cut method (JIS G-0052). The precipitates were determined by observing thin films and carbon extraction replicas under an electron microscope. To investigate the relationship between the toughness and precipitates after heating to high temperatures, the precipitates were electrolytically extracted at a constant potential according to the 10% AA-methanol process, before the insoluble titanium and insoluble chromium were quantitatively analyzed by ICP and the insoluble nitrogen was quantitatively measured by chemical analysis. Precipitates not less than 0.01  $\mu$ m in diameter are considered to have been extracted and analyzed.<sup>8)</sup> Because no insoluble aluminum was detected, the amount of titanium that precipitated as TiN from insoluble nitrogen was calculated by assuming that all the nitrogen was fixed by titanium.

#### **3** Experimental Results and Discussion

## 3.1 Characteristics of the ERW Pipe Welds Zone

Figure 1 shows an example of the Charpy impact values (absorbed energy) in the seam of the ERW pipe, in the HAZ 0.25 mm from the seam, in the HAZ 0.50 mm from the seam, and in the base metal  $180^{\circ}$  opposite to the seam. Figure 2 shows the change in the ductile-brittle transition temperature from the seam outward. It is apparent from these figures that the toughness of the weld zone was much lower than that of the base metal and  $180^{\circ}$  zone. Although the toughness of the seam may be lower than that of the HAZ under some welding conditions, the phenomenon of the toughness of the seam is one of the features of the weld zone of ERW pipe. In the case of TIG-welded pipe, grain growth is noticeable in the weld bead, the toughness of



Fig. 1 Result of Charpy impact tests at the seam, HAZ, and the opposite side of the seam in the ERW pipe (0.009C-0.007N-0.23Ti-11Cr)



Distance from the seam (mm)

Fig. 2 Distribution of ductile-brittle transition temperature along the circumference of ERW pipe (0.009C-0.007N-0.23Ti-11Cr)



(b) Post annealed (930°C × 10 min)

Photo 1 Macrostructures of transverse cross section of an ERW pipe (Etching reagent: picric and hydrochloric acid)

the bead with coarse grains being the lowest.

The macrostructure of the weld zone of ERW pipe made by the pilot mill is shown in **Photo 1**(a). The bead remaining uncut in this weld.

When producing ERW pipe, the temperature-rise time is very short and the fusion zone is squeezed out. Therefore, grain growth is slight even in the weld parts. For this reason, the cause of the decrease in toughness of the weld parts cannot be ascribed to grain size, but



Fig. 3 Effect of C content on the ductile-brittle transition temperature and grain size number

mainly to either the thermal history of welding or upsetting strain (or to a combination of both of them). If an upsetting strain remains, grain growth may sometimes occur in this area, although this depends on the amount of strain and reheating conditions. Photo 1(b) shows the microstructure of a section after heat treating the ERW pipe at 930°C for 10 min. Grain growth is hardly noticable in the upset part near the seam, and it can be judged that the upsetting strains were almost completely relieved during pipemaking. On the other hand, grain growth is noticeable in the zones more than 0.5 mm to the right and left of the seam, and this indicates that the working strain during pipemaking remained to a large extent in these zones. From the fact that the reduction in toughness was greater in the seam and its vicinity than in the areas with large residual strain, it is judged that this reduction in toughness was caused by changes due to the thermal history of welding, and not by upsetting.

Therefore, ERW pipe was made from steels with different carbon and nitrogen contents, and the effects of the carbon and nitrogen contents on the toughness of the HAZ were investigated. The effect of carbon content is shown in Fig. 3; the HAZ toughness increased with decreasing carbon content. In this case, the grain size was almost constant, regardless of the carbon content, and it can be considered that the grain size had no effect on toughness. On the other hand, the toughness of the base metal decreased due to grain coarsening in the samples with lower carbon content. Furthermore, although it is apparent from Fig. 4 that a reduction in the nitrogen content improved the toughness of both the base metal and the HAZ, this effect was greater in the HAZ. Because the nitrogen content has scarcely any effect on the grain size, it can be assumed that the pres-

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Fig. 4 Effect of N content on the ductile-brittle transition temperature and grain size number

ence of nitrogen itself had a direct effect on the toughness of the base metal. (As will be described later, almost all nitrogen existed as TiN and, therefore, TiN may have influenced the toughness.) As already mentioned, the HAZ and base metal were affected by the carbon and nitrogen contents in different manners, and the effects were especially great in the samples that had undergone a thermal history of rapid heating and quenching. This suggested the necessity to correlate the dissolution and precipitation behavior of these elements to toughness.

## 3.2 Relationship between Precipitates and Toughness after Heating and Quenching

Although the cooling rate of the ERW pipe weld zone was not measured, the average cooling rate from 1200 to 700°C was estimated at approximately 500°C/s from heat transfer calculations, in which the heat transfer from the weld in the circumferential direction was taken into consideration. (Calculation was made assuming that heating was stopped after a temperature rise to 1400°C, and that the cooling was regarded as by radiation and circumferential secondary heat transfer. The heat transfer in the longitudinal direction of the pipe was ignored.) This cooling rate is lower than the water quenching rate that was used for the 1.5 mm $t \times 60$  mm  $\times 100$  mm sheet in this experiment, and is higher than the N<sub>2</sub> gas quenching rate.

The temperature-rising rate of ERW pipe weld zone is in the order of thousands of degrees centigrade per second and cannot be reproduced experimentally. However, the cooling rate can be represented by water quenching and gas quenching.

Samples were heated at a temperature-rise rate of 50°C/s, and water-quenched immediately after the sample temperature had reached 900-1250°C. The insol-

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uble titanium and the titanium in precipitates of TiN in these samples were analyzed, with the results shown in Fig. 5. Those analyzed as insoluble titanium contained not only carbonitrides of titanium, Ti(C, N), but also titanium sulfide, titanium oxide and other compounds. Insoluble titanium decreased with increasing quenching temperature, and this tendency was especially noticeable above 1000°C. According to thermodynamic data,<sup>9)</sup> at 1250°C, some TiC is dissolved and in the solute state. In the results shown in Fig. 5, a deviation from the equilibrium state is conceivable because the holding time was not sufficient at each temperature. However, the dissolution of TiC, etc. into the solute state proceeded with the increase in temperature attained, and carbon was in a supersaturated solute condition at room temperature after cooling. On the other hand, titanium as TiN shows scarcely any difference from the analytical value obtrained in the base metal, even by quenching from 1250°C. In other words, the amount of TiN scarcely changed in the thermal history of welding, this also being apparent from the thermodynamic data. Figure 6 shows the Charpy impact values of sheets of material samples A, B, C and D with different carbon content obtained after water quenching from 1250°C. The impact values were evaluated by the absorbed energy at the two levels of temperature of 0°C and  $-40^{\circ}$ C. Analytical values of titanium as TiN and the carbon content for estimating the solute carbon not fixed as TiC are also shown in the same figure. Because solute carbon (sol C) cannot be found by analysis, it was found approximately by the following equation:

sol C = total C - 
$$\frac{12.01}{47.88}$$
 (insol Ti  
-  $\frac{47.88}{14.00}$  × insol N) · · · · · (1)

The amount of carbon in a supersaturated solute state decreases with decreasing total carbon content. Even when the carbon content was decreased, the grains after



Fig. 6 Effect of C content on the results of chemical analyses, Charpy impact value, and grain size number

heating to 1250°C coarsened to almost the same degree and there was no difference in this respect. Furthermore, although TiN that worsened toughness (as will be described later) was a little high, the toughness improved with decreasing carbon content. In this process, solute titanium increases by the amount of dissolution of TiC at the same time. However, solute titanium existed from the start in an amount of about 0.1%, and the increase in solute titanium from TiC was small. Practically, when solute titanium content was 0.10 to 0.11% in all the samples. Therefore, the amount of solute titanium is considered constant in them.

A similar investigation was made into samples with different nitrogen content after water quencing from 1250°C, the results being shown in Fig. 7. The amount of titanium as TiN decreased with decreasing nitrogen, and the toughness improved accordingly. At this time, there was no difference in the grain size. It might be thought that the insoluble nitrogen had almost the same value as the total nitrogen content, and that the nitrogen was all in the form of TiN. Therefore, a reduction of TiN.

From the foregoing, it was found that in the pipe weld zone quenched from a high temperature, the amounts of supersaturated solute carbon and of TiN had a great effect on its toughness.

In the HAZ of the weld, the maximum attained temperature and the cooling rate from this temperature are determined by the distance from the seam. A difference



Fig. 7 Effect of N content on the results of chemical analyses, Charpy impact value, and grain size number

in the attained temperature produces a difference in the amount of TiC that is dissolved into the solute state. However, if the cooling rate was low, the steel could be stabilized by titanium again, and the embrittlement could be suppressed. Therefore, the reprecipitation behavior in the cooling process is important for investigating the reasons for a change in toughness.

## 3.3 Relationship between the Cooling Rate and the Precipitates and Toughness after Heating

The precipitation during cooling was investigated by cooling samples after heating to a high temperatures by  $N_2$  gas injection. The cooling rate by  $N_2$  gas was about 100°C/s, so that it was slower than the actual cooling rate at the weld part of ERW pipe.

Figure 8 shows analytical values of the insoluble chromium that depended on the difference in quenching temperature and on the cooling method. The analyzed chromium would contain not only chromium carbides but also other chromium precipitates. There was a clear difference depending on the cooling rate; the higher the cooling rate, the larger the amount of precipitates. This difference was especially great at quenching temperatures 1100°C or over, at which the dissolution of TiC into the solution state was most marked. There was no difference due to the cooling rate, although a difference between the insoluble titanium, which approximates to the amount of precipitated TiC, and titanium as TiN (titanium compounds other than TiN) was apparent. It appears that when the quenching time was low, TiC did not dissolve into the solute state and remained unchanged.

As will be described later, chromium carbide can greatly worsen the toughness.<sup>7,10)</sup> Although in Fig. 8 there is a difference in the amount of insoluble chro-

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Quenching temperature ("C)

Fig. 8 Effect of quenching temperature and quenching rate on the results of chemical analyses (steel G: 0.02C-0.28Ti-0.01N)



Fig. 9 Results of Charpy impact tests for waterquenched and reheated specimens (steel H: 0.011C-0.23Ti-0.011N)

any difference in the transition temperature because of the existence of considerable amounts of insoluble chromium, even in the water-quenched steel.

To further clarify the precipitation behavior during the cooling process, a precipitation treatment was carried out at 500-1000°C for 30 s in specimens that had been water-quenched from 1250°C. The results of the Charpy impact test are shown in Fig. 9, and the absorbed energy at 0°C and 20°C, and analytical values of the precipitates are shown in Fig. 10. It is apparent that the decrease in toughness was most marked at about 700°C, and that the amount of insoluble chromium increased with decreasing toughness. The higher

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the temperature, the larger the amount of (insoluble Ti) – (Ti as TiN), which approximates to the amount of TiC. This could have been due to the supersaturated solute carbon, etc. being fixed by titanium again. The toughness of those specimens subjected to a precipitation treatment at 900 and 1000°C, in which no insoluble chromium was detected, was higher than that of the quenched specimens.

It is apparent from the foregoing results that insoluble chromium had a great effect on the decrease in toughness,<sup>7,10)</sup> and it was apparent that, even in titanium-stabilized steels, chromium-base precipitates were formed in the cooling process after heating to high temperatures. It has been stated that the precipitation of chromium carbide was most accelerated at about  $800^{\circ}$ C,<sup>11,14)</sup> and it appears that the insoluble chromium we found was chromium carbide.

The results of an observation of the precipitates under a transmission electron microscope are shown in **Photo 2.** The base metal shows coarse precipitates within grains, with no precipitates being apparent at the grain boundaries. These precipitates may have been Ti(C, N), etc. This Ti(C, N) observed in the base metal did not exist in those specimens water-qienched from 1250°C, the precipitates instead being formed at the grain boundaries. These precipitates were chromium carbide and were coarsened at the grain boundaries when the precipitation treatment was conducted at 700°C for 30 s after quenching, fine precipitates with matching strain being formed within the grains.

**Photo 3** shows the carbon extraction replica of a sample subjected to precipitation treatment at  $700^{\circ}$ C, with featherlike carbide precipitates accumulated densely at the grain boundaries. From an EDX analysis, it was



(a) As annealed (b) After water (c) After aged quenched from at 700°C for 30s 1 250°C

Photo 2 Transmission electron micrographs of type 409 steel (steel G; 0.02C-0.28Ti-0.01N)



Photo 3 X-ray microanalysis of aged specimen of type 409 steel

found that these precipitates were composed of chromium, titanium, and iron.

#### 3.4 Precipitation Behavior of Chromium Carbide

As previously mentioned, steel G was composed of a considerable amount of chromium carbide precipitates, even in the samples water-quenched beginning at 1250°C. Therefore, an examination was made to determine whether the precipitation of carbide would be possible in such a short time.

The theoretical calculation by Arai et al.<sup>14</sup> is well known for analyses related to the precipitation of chromium carbide in ferritic stainless steels,<sup>11-15</sup> and Tomimura et al.<sup>15</sup> have conducted analyses by converting this theoretical expression into an approximated expression. In this report also, the precipitation of chromium carbide was examined in 11% Cr-0.01% C ferritic stainless steels by using a similar approximated expression.

Since precipitation was concentrated at the grain boundaries, as shown in Photo 3, the theoretical expression (molar concentration of carbon fixed as carbide at the grain boundaries,  $\Delta X_{\rm C}$ ) introduced by Arai et al. is used:

$$\Delta X_{\rm C} = \frac{6}{23} \times \frac{6}{r_0} \times \frac{X_{\rm Cr}^0 - X_{\rm Cr}^i}{Y_{\rm Cr} - X_{\rm Cr}^i} \times \left(\frac{D_{\rm Cr}t}{\pi}\right)^{1/2} \dots \dots \dots (2)$$

where  $r_0$ : Grain size (cm)

- $X_{Cr}^{0}$ : Molar concentration of Cr in the mother phase  $(X_{Cr}^{0} = 0.11)$
- $Y_{Cr}$ : Molar fraction of Cr related to metallic components of carbide
- $X_{Cr}^{i}$ : Molar concentration of mother-phase Cr in local equilibrium with carbide at the grain boundary
- $D_{Cr}$ : Diffusion coefficient of  $Cr^{16}$  (cm/s<sup>2</sup>)

t: Elapsed time (s)

Tomimura et al.<sup>15)</sup> obtained an approximate solution for this equation by supposing that, in a dual-phase stainless steel, carbide was  $Cr_{23}C_6$  not containing iron, i.e.,  $Y_{Cr} = 1$  and assuming that  $X_{Cr}^i = 0$ . Although iron and titanium were detected in carbide as mentioned earlier, we have also assumed in this report that  $Y_{Cr} = 1$ . However, we assumed for approximation that  $X_{Cr(11)}^i = (11/19)X_{Cr(19)}^i$ , and not that  $X_{Cr}^i = 0$ . For  $X_{Cr(19)}^i$ , we used the value for 0.01%C-19%Cr ferritic stainless steels found by Arai et al.<sup>14</sup> When  $\Delta X_C$  (molar concentration) of Eq. (2) is converted into the weight percentage of carbon and chromium that precipitated as  $Cr_{23}C_6$ , Eq. (3) is obtained:

$$\Delta X'_{\rm C} = 21.6 \times \Delta X_{\rm C}$$
  
$$\Delta X'_{\rm Cr} = 21.6 \times \Delta X_{\rm Cr} \times \frac{52 \times 23}{12 \times 6}$$
 (3)

- where  $\Delta X'_{C}$ : Weight of carbon fixed as carbide at the grain boundary (%)
  - $\Delta X'_{Cr}$ : Weight of chromium fixed as carbide at the grain boundary (%)

**Figure 11** shows the results of the calculation of chromium content as a function of reheating temperature and time, using a grain size of 0.008 cm after heating to  $1250^{\circ}$ C as  $r_0$ . The higher the reheating tempera-

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Reheating temperature (°C)

Fig. 11 Relation between calculated Cr content as  $Cr_{23}C_6$  precipitate,  $\Delta X'_{Cr}$ , and reheating temperatures (0.01C-11Cr)

ture and the longer the reheating time, the more  $X_{Cr}^i$ would approach  $X_{Cr}^0$ , with the result that the value of  $\Delta X'_C$  decreases. Therefore, the amount of precipitation would have a peak value at 800-850°C. This shows in 11% Cr steels without the addition of Ti that about 0.005% of chromium carbide precipitates when they are heated to 800°C and held for 0.36 s at this temperature. In its effect, the phenomenon may be complex in type 409 steel due to the competition of titanium and chromium in the fixing by carbon, the effect of iron in carbide (molar ratio  $Y_{Cr}$ ), etc. However, thorough precipitation occurs at the cooling rate from water quenching while the steel passes through the precipitation temperature range of 900-600°C.<sup>11,14</sup>

## 4 Conclusions

ERW pipes were made on a CBR forming pilot mill using a Ti-stabilized 11% Cr type 409 steel. Charpy impact tests were conducted on the weld zone of these ERW pipe samples and on sheets subjected to various heat treatments and the results were analyzed in relation to the precipitates and toughness of the welds. The results obtained are as follows:

- (1) The toughness of ERW pipe weld zone was lower than that of the base metal, the HAZ showing low values in particular.
- (2) The reason for the HAZ having low toughness values is thought to have been that TiC was dissolved by heating to high temperatures, solute carbon being precipitated as chromium carbide in the colling process, and that supersaturated carbon was present due to quenching.
- (3) TiN lowered the toughness in both the HAZ and the base metal.
- (4) Toughness in the HAZ was improved by reducing the carbon and nitrogen contents.

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