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Development of Martensitic Stainless Steel Seamless Pipe for Linepipe Application*



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1 Introduction

In recent years, oil and gas wells have been developed in increasingly severe corrosion environments characterized by high temperature, high partial pressure of CO_2, and high concentration of chloride ions, and in some cases, also containing H_2S. For this reason, the prevention of CO_2 corrosion and sulfide stress cracking (SSC) in OCTG material have become an important task. In pipelines, the material of flowlines and gatheringlines, which are used to transport oil or gas before corrosive substances and water are removed, is required to provide the same level of corrosion resistance as OCTG material. Conventionally, pipelines of this type have generally been protected from corrosion by injecting a corrosion inhibitor into the pipeline[1] or by adopting a corrosion resistant pipe material such as duplex stainless steel.[2] However, inhibitor injection involves various problems. In particular, the operating cost is high in offshore pipelines, the effectiveness of inhibitor is unstable at high temperatures, and leaks of inhibitor are a cause of environmental pollution. Although duplex stainless steel has excellent corrosion resistance, the cost of the material is extremely high, control of welding heat input is difficult, and in many cases, the corrosion resistance of the material is higher than necessary.

Martensitic stainless steels have rarely been used in pipelines because their weldability is generally low, requiring preheating and post welding heat treatment (PWHT) in the welding process. However, these materials show appropriate CO_2 corrosion resistance and are comparatively low in cost. This suggests that if martensitic stainless steel pipes with excellent weldability could be developed, considerable demand could be expected as an economical product which enables users to reduce the material cost, laying cost, and operating cost of pipelines.

From these backgrounds, two types of martensitic stainless steel seamless pipes with excellent weldability and corrosion resistance were developed. One is an 11Cr steel pipe for CO_2 environments, which offers corrosion resistance superior to that of 13Cr OCTG, and the other is a 12Cr steel pipe with good SSC resistance for CO_2 + slight H_2S environments. This report describes the development of these martensitic stainless steel seamless pipes and their respective features.

2 Development of 11Cr Steel Pipe for Linepipe in CO_2 Environments

2.1 Target Properties

The target properties in the development of a marten-

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sitic stainless steel seamless pipe for linepipes in CO_2 environments are presented below.

(1) Weldability: Weldable without preheating or PWHT
(2) CO_2 corrosion resistance: Equal to or better than 0.2C-13Cr steel pipe for OCTG
(3) Low temperature toughness: Charpy absorbed energy of 100J or higher at −40°C in both the base material and the welds
(4) Strength: Yield strength (YS) of 360 MPa or higher; namely, grade X52 or higher in API5L of the American Petroleum Institute (API)
(5) Hot workability: Possible to manufacture seamless pipe by the Mannesmann process

2.2 Laboratory Study

2.2.1 Concept of alloy design

The alloy design of the new steel pipe was carried out based on knowledge of the effect of alloying elements on the weldability, hot workability, and corrosion resistance of martensitic stainless steel.

Generally, when martensitic stainless steels are welded, cracking occurs due to the hardening which accompanies the martensite transformation in the heat affected zone (HAZ) and due to hydrogen which is dissolved in the weld metal during welding.[3] Accordingly, one possible method of preventing weld cracking from the material side is suppressing the hardening which accompanies the martensite transformation by reducing the contents of C and N. In this connection, it has been reported that cracks in the welds can be prevented in martensitic stainless steels by holding the total content of C and N to 0.04% or under.[4] Moreover, reducing the C content can also be expected to improve resistance to CO_2 corrosion by reducing the amount of Cr carbides.[5]

Hot workability is an extremely important factor for deciding the surface quality of seamless pipes. Hot workability is drastically reduced and surface quality is deteriorated if delta-ferrite is formed in the manufacture of seamless pipes of martensitic stainless steel, and in some cases, pipemaking becomes impossible.[6] Thus, adding the content of austenite former elements such as Ni, Mn, Cu, etc. or reducing the content of ferrite formers such as Cr, Si, etc. is effective in improving hot workability.

Based on these points, laboratory heats with a basic composition of 0.01C-(11,12)Cr-Ni-Cu-0.01N were prepared. The chemical composition of the respective steels is shown in **Table 1**. The heats were hot-rolled to a thickness of 15 mm, air-quenched, and tempered.

2.2.2 Weldability

Weldability was evaluated from the cracking sensitivity of the HAZ in a y-groove weld cracking test. A commercially-available lime-type coated electrode for martensitic stainless steel was used as the welding material. Preheating was performed at temperatures of 30, 70, and 100°C. **Table 2** shows the test conditions together with the results of the y-groove cracking test. In steels with C and N contents reduced to 0.01%, no cracking was detected in any cross-section in observations 120 h after welding, even at a preheating temperature of 30°C, showing that these steels had satisfactory weldability. However, welding cracking occurred in steels with C or N contents of 0.03%, and was thought to have been caused by hardening of the HAZ or hydro-

Table 1 Chemical compositions of steels used in laboratory study (mass%)

	Material		C	Cr	Ni	Cu	Mo	N
1	Laboratory heats		0.01-0.03	11-12	0-2.0	0-1.5	-	0.01-0.03
2	Production heats	0.2C-13Cr	0.2	13	-	-	-	0.02
3		Low C-13Cr	0.02	13	4.0	-	1.0	0.05

Table 2 Results of y-groove cracking test for 11 and 12Cr steels

Material		Preheating temperature 30°C	70°C	100°C
0.03C-0.01N	11Cr-1.0Ni-0.5Cu	Crack	Crack	Crack
0.01C-0.03N		Crack	Crack	Crack
0.01C-0.01N	12Cr-1.0Ni-0.5Cu	No crack	No crack	No crack
	12Cr-1.0Ni-1.0Cu	No crack	No crack	No crack
	12Cr-2.0Ni-0.5Cu	No crack	No crack	No crack

Plate thickness: 15 mm
Welding material: Type 410H SMAW, 4 ϕ (Diffusible hydrogen; 4.28 cc/100 g)
Welding condition: Current; 160A, Voltage; 24-26V, Speed; 150 mm/min
Test condition: Room temperature; 30°C, Humidity; 60%RH

gen. These results confirmed that weldability can be improved by reducing the content of C and N, and showed that welding without cracking is possible, even without preheating, if the C and N contents of the steel are reduced to 0.01%.

2.2.3 Mechanical properties

Figure 1 shows the relationship between the Charpy absorbed energy at 0°C and YS when 12Cr steels having various Cu contents from 0–1.5% were tempered for 30 min at 600–700°C, below the Ac_1 transformation point. Both high toughness and strength of the YS550 MPa grade namely, X80 grade, can be obtained by tempering these steels under appropriate conditions. Steels with Cu contents of 1.0% or higher tend to show high strength and low toughness due to their high resistance to temper softening, suggesting that this level of Cu is disadvantageous for stable toughness.

2.2.4 CO_2 corrosion resistance

A corrosion test was performed using an autoclave in order to evaluate resistance to CO_2 corrosion. The test was performed by immersing 3 mmt × 25 mmw × 50 mml specimens in a 20%NaCl solution saturated with CO_2 at 3.0 MPa, and then holding at 80°C for 7 d. Resistance to CO_2 corrosion was evaluated by the corrosion rate (mm/y) obtained by conversion from the weight loss rate. As comparison steels, a 0.2C-13Cr steel pipe for OCTG and a 0.02C-13Cr-4Ni-Mo steel pipe for OCTG for high-temperature high-CO_2 environments were tested at the same time.

Figure 2 shows the results of the CO_2 corrosion test. The Cu-added steels showed a general corrosion morphology. The corrosion rate decreased as the content of Cr and Ni increased or the content of C decreased. The results obtained by a regression equation were arranged as a CO_2 corrosion index (CCI), which was defined by Cr-10C + 2Ni. On the other hand, pitting occurred in the Cu free steel, and consequently, this material showed a high corrosion rate. These results showed that CO_2 corrosion resistance superior to that of 0.2C-13Cr steel for OCTG can be obtained, even when the Cr content is reduced to 11%, if Cu is added and the CCI is increased to 12% or higher.

2.3 Trial Production of Pipe and Properties of the Pipe

Considering the results of the laboratory study described above and hot workability, steel with a composition of 0.01C-11Cr-1.5Ni-0.5Cu-0.01N was melted in a converter and produced into seamless pipes with an outer diameter of 273 mm and a wall thickness of 12.7 mm. After pipemaking, YS was adjusted to X80 grade by quenching and tempering the pipes.

To evaluate the properties of the welds, a girth welded joint was made in the trial pipes. The welding was performed by the methods which are most generally applied in actual work with this steel grade. Namely, 25Cr type duplex stainless steel welding wire was used, and welded joints were made by GTAW (gas tungsten arc welding) in all passes, and by GTAW in the first pass followed by GMAW (gas metal arc welding) in the second pass. The chemical composition of the welding wire and base material are shown in **Table 3**. The welding conditions are shown in **Table 4**. Although preheating and PWHT were omitted in all welding, no weld cracks occurred in any of the joints. Thus, it was possible to weld the pipes by either the GTAW or GMAW without cracking, even when preheating and PWHT were not applied and it can therefore be said that the material has excellent weldability.

The results of a tensile test and side bend test of the welded joints are shown in **Table 5**. In the tensile test, the test piece was a full-thickness specimen with a width of 25 mm in the gauge section, from which the reinforcement had been removed. Both the joint made by

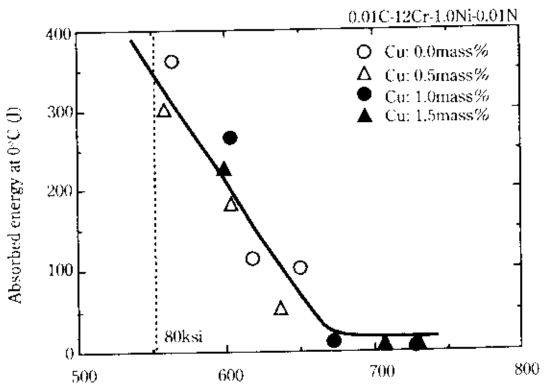

Fig. 1 Relation between Charpy absorbed energy at 0°C and yield strength of 12Cr steels with various Cu contents

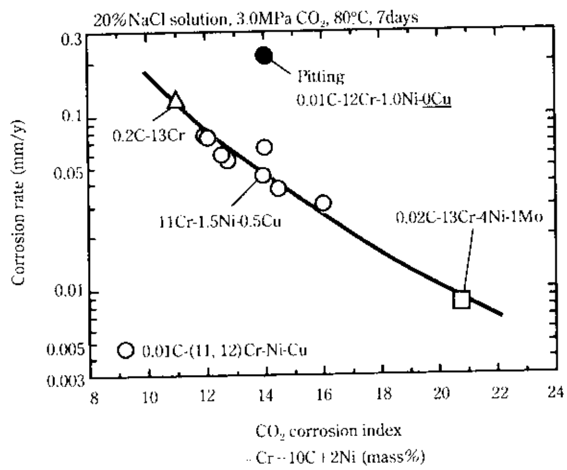

Fig. 2 Relation between corrosion rate and CO_2 corrosion index

Table 3 Chemical compositions of base metal and welding wires for girth welding of 11Cr-1.5Ni-0.5Cu steel pipe

(mass%)

Material	C	Si	Mn	Cr	Ni	Cu	Mo	N
11Cr base metal	0.01	0.16	1.13	11.1	1.5	0.48	-	0.01
GTAW filler wire	0.01	0.30	0.38	25.3	9.5	-	4.0	0.27
GMAW wire	0.02	0.33	0.41	25.1	9.6	-	4.0	0.27

Table 4 Girth welding conditions for 11Cr-1.5Ni-0.5Cu steel pipe

Welding method	Pass	Diameter of welding wire (mm)	Welding current (A)	Welding voltage (V)	Welding speed (cm/min)	Heat input (kJ/cm)	Shielding gas
GTAW	1-7	2.0, 2.4	150-190	13.5-16.5	5.3-7.2	20-35	Ar
GTAW + GMAW	1 (GTAW)	2.0	146	13.5	4.0	30	Ar
	2 (GMAW)	1.2	150	14.0	6.5	19	Ar

Table 5 Mechanical properties of girth welded joints of 11Cr-1.5Ni-0.5Cu steel pipe

Welded joint/Base metal	Tensile test				Side bend test
	YS (MPa)	TS (MPa)	El (%)	Fracture position	
GTAW	-	717	24	BM	No crack
GTAW + GMAW	-	748	25	BM	No crack
Base metal	593	726	22	-	-

GTAW in all passes and the joint made by GTAW + GMAW fractured in the base metal, and no large differences were observed between them. In the side bend test, full-thickness specimens 4 mm thick, with the reinforcement removed, were bent with a radius of 8 mm. No cracking was observed in either joint, even when bent to 180°.

The hardness distribution of the all-GTAW and GTAW + GMAW welded joints is shown in **Fig. 3**. Hardness was measured from the bead center to the base material at intervals of 1 mm, at the center of the plate thickness and at positions 1 mm from the inner and outer surfaces. The maximum hardness was approximately HV310 in the HAZ. No large differences were found between the all-GTAW joint and the GTAW + GMAW joint.

The results of a Charpy test of the all-GTAW and GTAW + GMAW welded joints are shown in **Fig. 4**. The test was performed at the bond, HAZ, and base material. The notch in the bond and HAZ was at the position shown in the figure. The toughness of the HAZ was higher than that of the base material. Although the bond showed somewhat lower toughness than the base material, an absorbed energy of 100 J at −40°C was obtained.

The results of a CO_2 corrosion test of the welded joints are shown in **Table 6**. The test was performed

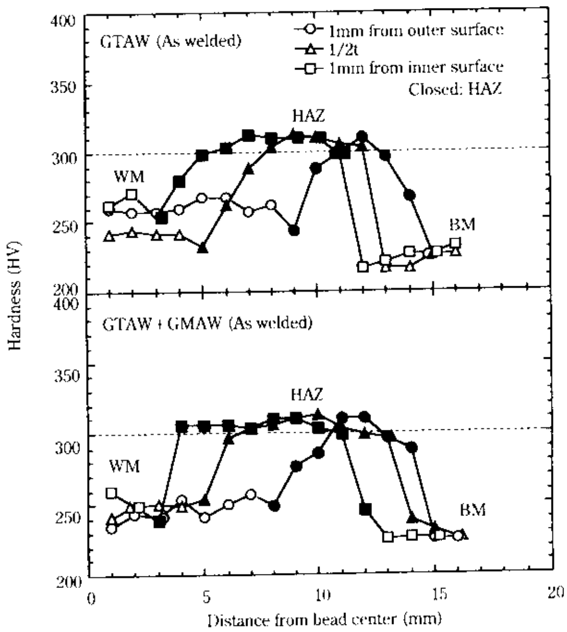

Fig. 3 Hardness distribution in welded joint of 11CR-1.5Ni-0.5Cu steel pipe

using specimens 3 mmt × 25 mmw × 50 mml taken from the inner surface of the pipe at the joint. The specimens were immersed in a 10%NaCl solution saturated

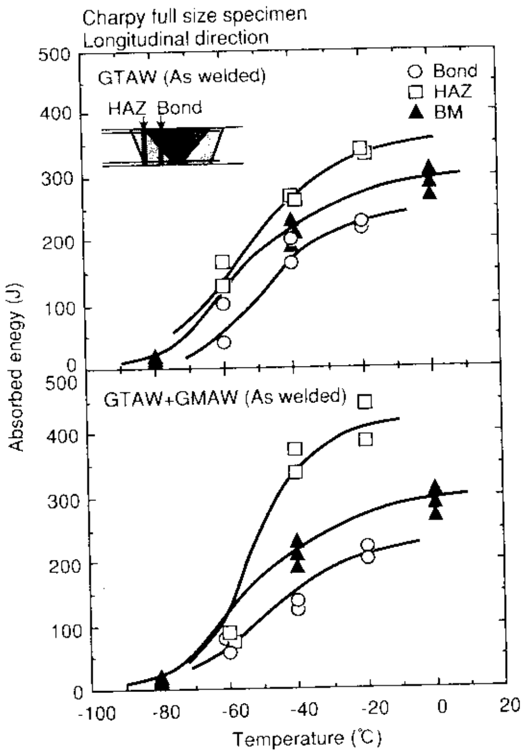

Fig. 4 Relation between Charpy absorbed energy and temperature of 11Cr-1.5Ni-0.5Cu steel pipe welded joint

Table 6 Results of CO_2 corrosion test for welded joints of 11Cr-1.5Ni-0.5Cu steel pipe

Material			Corrosion rate (mm/y)
11Cr-1.5Ni-0.5Cu	Welded joint	GTAW	0.030
		GTAW + GMAW	0.029
	Base metal		0.033
0.2C-13Cr			0.059

10%NaCl solution, 3.0 MPa CO_2, 80°C, 168 h

with CO_2 at 3.0 MPa and held at 80°C for 7 d. As comparison materials, the base material of the 11Cr steel pipe and a 0.2C-13Cr steel pipe for OCTG were tested at the same time. The corrosion rate of both joints was approximately 1/2 that of the 0.2C-13Cr pipe for OCTG, and no preferential corrosion of the welds was observed.

2.4 Summary

The above test results confirmed that the newly-developed 11Cr steel pipe possesses adequate weldability, mechanical properties, and corrosion resistance for practical application as linepipe in CO_2 environments.

3 Development of 12Cr Steel Pipe for Linepipe in CO_2 + Slight H_2S Environments

3.1 Target Properties

The target in the development of a martensitic stainless steel seamless pipe for linepipe in CO_2 + slight H_2S environments was a pipe which combined SSC resistance with the target properties of the martensitic stainless steel seamless pipe for linepipe in CO_2 environments described above. Specifically, the target properties for development were as follows.

(1)-(5) Same as the target properties of the pipe for CO_2 environments

(6) SSC resistance: SSC resistance in an environment with an H_2S partial pressure of 0.001 MPa, 5%NaCl solution, pH of 4.0

3.2 Concept of Alloy Design

With martensitic stainless steel, SSC originates from pitting in environments where a passivation film is formed. Accordingly, SSC resistance can be improved by increasing the pitting resistance of the steel. Mo is known to be an element which improves the pitting resistance of martensitic stainless steel. The effect of Mo and Ni on the SSC resistance of martensitic stainless steels for OCTG is shown in **Fig. 5**.[7] Ni did not affect the SSC test results. In contrast, when the Mo content is increased from 1% to 2%, the limit line for the occurrence of SSC shifts toward the low pH, high H_2S partial pressure side, in other words, the severe environment side. This shows that a 1% addition of Mo is adequate for the environment which was adopted as the development target here, i.e. H_2S partial pressure of 0.001 MPa, 5%NaCl solution, pH of 4.0. However, because the pitting resistance of the HAZ is considered to be lower than that of the base material, 2% Mo was added to the

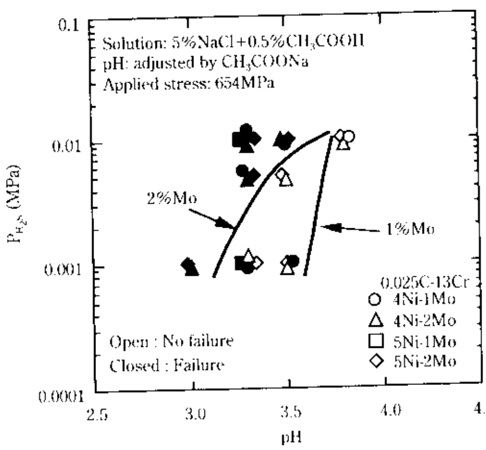

Fig. 5 Effects of Ni and Mo content on SSC resistance of 0.025C-13Cr steels

newly developed material to ensure stable pitting resistance.

Excellent weldability was secured by reducing the C and N contents to 0.01%, as with the 11Cr steel pipe discussed above. To compensate for the reduction in hot workability which was predicted due to the addition of Mo and reduction in the contents of C and N, 5% Ni was added.

Based on the above thinking, 0.01C-12Cr-5Ni-2Mo-0.01N was adopted as the chemical composition of the martensitic stainless steel seamless pipe for linepipe in CO_2 + slight H_2S environments.

3.3 Trial Production of Pipe and Properties of the Pipe

Seamless pipes with an outer diameter of 273 mm and wall thickness of 12.7 mm were manufactured using steel with a composition of 0.01C-12Cr-5Ni-2Mo-0.01N melted in a converter. The manufactured pipes were air-quenched and tempered to adjust YS to X80 grade. To evaluate the properties of the welds, girth welded joints were made in the trial pipes. As with the welded joints in the above-mentioned 11Cr pipes, 25Cr duplex stainless steel was used as the welding wire, and joints were welded by GTAW in all passes and GTAW in the first pass, followed by GMAW in the second pass. The chemical composition of the welding wire and base material is shown in **Table 7**; the welding conditions are shown in **Table 8**. Preheating and PWHT were not performed. As with the 11Cr steel pipes, no cracking occurred in any of the welds with these pipes, and excellent weldability was confirmed.

The tensile test, side bend test, hardness measurement, and Charpy test of the joints were performed following the same procedure as that used with the 11Cr steel pipes.

The results of the tensile test and side bend test of the welded joints are shown in **Table 9**. In the tensile test, both joints fractured in the base material, and no large differences were observed between the two. In the side bend test, no cracking occurred.

The hardness distribution of the all-GTAW and GTAW + GMAW joints is shown in **Fig. 6**. The maximum hardness was approximately HV330 in the HAZ, which was a somewhat higher value than the maximum hardness of the welded joints in the 11Cr steel pipes. This was caused by an increase in hardness after quenching due to the addition of Ni and Mo. However, no large differences were found between the two joints in the 12Cr steel pipes.

The results of the Charpy test of the all-GTAW and GTAW + GMAW joints are shown in **Fig. 7**. Although the bond showed a somewhat lower value than other positions, an absorbed energy of 200 J or more at −80°C was obtained, and excellent low temperature toughness was confirmed.

Table 7 Chemical compositions of base metal and welding wires for girth welding of 12Cr-5Ni-2Mo steel (mass%)

Material	C	Cr	Ni	Cu	Mo	N
12Cr base metal	0.01	12.0	5.1	-	2.0	0.01
GTAW filler wire	0.01	25.3	9.5	-	4.0	0.27
GMAW wire	0.02	25.1	9.6	-	4.0	0.27

Table 8 Girth welding conditions for 12Cr-5Ni-2Mo steel pipe

Welding method	Pass	Diameter welding wire (mm)	Welding current (A)	Welding voltage (V)	Welding speed (cm/min)	Heat input (kJ/cm)	Shielding gas
GTAW	1-6	2.0, 2.4	145-191	14.0-16.5	4.0-7.4	18-33	Ar
GTAW + GMAW	1 (GTAW)	2.0	148	13.5	4.4	27	Ar
	2 (GMAW)	1.2	145	15.0	7.5	17	Ar

Table 9 Mechanical properties of girth welded joints of 12Cr-5Ni-2.0Mo steel pipe

Welded joint/Base metal	Tensile test				Side bend test
	YS (MPa)	TS (MPa)	El (%)	Fracture position	
GTAW	-	845	30	BM	No crack
GTAW + GMAW	-	856	30	BM	No crack
Base metal	634	827	34	-	-

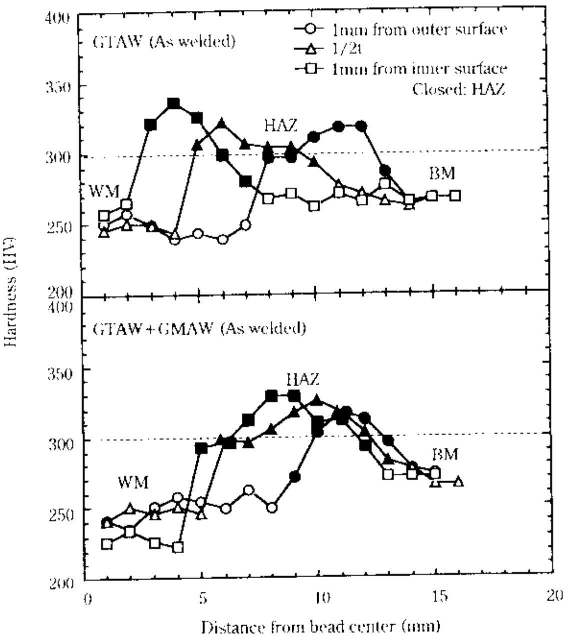

Fig. 6 Hardness distribution in welded joint of 12Cr-5Ni-2Mo steel pipe

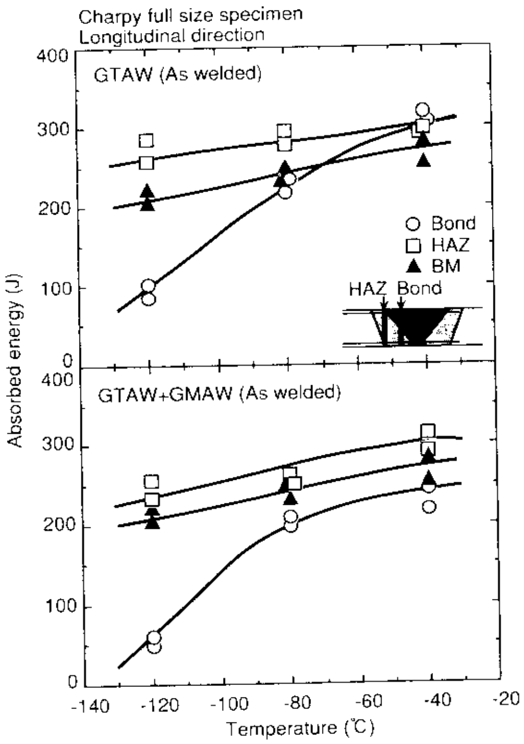

Fig. 7 Relation between Charpy absorbed energy and temperature of 12Cr-5Ni-2Mo steel pipe welded joint

An SSC test was performed by the constant load tensile SSC test method using the GMAW joint. The specimen was taken so that the welds were at the gauge center.

Table 10 SSC test results of GTAW + GMAW joint of 12Cr-5Ni-2Mo steel pipe

H_2S	Solution	pH 4.0	pH 4.5
0.001 MPa	5%NaCl+ 0.5%CH_3COOH	○	○
		○	○
0.002 MPa	5%NaCl+ 0.5%CH_3COOH	○	○
		○	○
0.002 MPa	10%NaCl+ 0.5%CH_3COOH	○	○
		○	○

pH was adjusted by CH_3COONa
Applied stress: 567 MPa
○ : No SSC
× : SSC

A mixed solution of either 5% or 10%NaCl and 0.5% CH_3COOH was prepared, and the pH was adjusted to 4.0 or 4.5 with CH_3COONa. The gas was H_2S at 0.001 or 0.002 MPa. The balance gas was CO_2, and the total pressure was set at 0.1 MPa. The applied stress was 567 MPa, which corresponds to 90% of the YS of the base material. The results of the SSC test are shown in **Table 10**. SSC did not occur even in a 10%NaCl solution with a pH of 4.0 and 0.002 MPa H_2S, confirming that welded joints of this steel possess satisfactory SSC resistance.

3.4 Summary

These test results confirmed that the newly-developed 12Cr steel pipe possesses adequate weldability, mechanical properties, and corrosion resistance for practical application as linepipe in CO_2 + slight H_2S environments.

4 Conclusion

Two types of martensitic stainless steel seamless pipes with excellent weldability and excellent corrosion resistance were developed for linepipe applications. For CO_2 environments, a composition of 0.01C-11Cr-1.5Ni-0.5Cu-0.01N was adopted. For CO_2 + slight H_2S environments, the composition adopted was 0.01C-12Cr-5Ni-2Mo-0.01N. The properties of these steel pipes and welded joints of the pipes using duplex stainless steel welding wires were as follows.

(1) Both types of pipe possess excellent weldability without requiring preheating and PWHT.
(2) It was possible to adjust the strength of both steels to X80.
(3) Welded joints in both types of steel pipe possesses adequate low temperature toughness for practical applications. Charpy absorbed energy is 100 J or more at −40°C in a welded joint of the 11Cr steel pipe for CO_2 environments and 200 J or more at −80°C in a

welded joint of the 12Cr steel pipe for CO_2 + slight H_2S environments.

(4) The welded joint of the 11Cr steel pipe for CO_2 environments possesses CO_2 corrosion resistance superior to that of 0.2C-13Cr steel for OCTG.

(5) The welded joint of the 12Cr steel pipe for CO_2 + slight H_2S environments shows no SSC in a 10%NaCl solution with a pH of 4.0 and 0.002 MPa H_2S, and thus possesses good SSC resistance.

Both types of steel pipe possess excellent weldability, mechanical properties, and corrosion resistance, as described above, and are therefore suitable as substitute materials for conventional flowlines in which corrosion protection by inhibitor injection or costly corrosion resistant materials such as duplex stainless steel pipe are now used.

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