

Development of Heavy 9% Nickel Steel Plates with Superior Low-Temperature Toughness for LNG Storage Tanks

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Abstract:

A 9% nickel heavy plate for large LNG tanks was developed by applying intercritical quenching, an effective heat treatment method for improving the toughness of nickel-bearing steel. The intercritical quenching treatment can significantly suppress the temper embrittlement sensitivity and stabilize the large amount of transformed austenite of the steel. Commercial mill-manufactured 40 and 45 mm thick 9% Ni steel plates exhibit superior mechanical properties. Both the base metal and weld metal of these heavy plates demonstrate excellent toughness against brittle fracture initiation and propagation as specified for materials of LNG tanks.

1. Introduction

A third LNG boom is claimed to have arrived recently. Increasing demand for LNG to meet thermal power generation and city gas production has led to the installation of many LNG storage tanks in Japan. Aboveground LNG tanks built in Japan in the past had inner volumes of not exceeding 80,000 m³ in the main, but larger LNG tanks are now studied to raise the efficiency of land use, for instance, and those exceeding 140,000 m³ have already been built. Such large LNG tanks are most likely to be fabricated from 9% nickel steel plates ranging from 40 to 45 mm in thickness, which goes beyond the conventional application limit of 30 mm for nickel steel plates.

The 9% Ni steel was first applied in liquid oxygen containment vessels in 1952. Since then, it has been used mainly for the inner shell of LNG tanks as a ferritic cryogenic material. Its brittle fracture properties, which are closely related to the safety of structures, have been extensively investigated by many researchers¹⁾. The research results have demonstrated that the 9% Ni steel has satisfactory mechanical properties, at welded joints as well, for

application to aboveground LNG tanks.

The heavy 9% Ni steel plate, however, has only a very little record of actual manufacture for large LNG tanks. Further, since increasing plate thickness tends to deteriorate the fracture toughness of steel, technology must be established to ensure the stable manufacture of heavy 9% Ni steel plates comparable in low-temperature toughness to conventional 9% Ni steel plates.

Against the above background, Nippon Steel studied manufacturing a heavy 9% Ni steel plate with high toughness by an intercritical quenching method. The resultant heavy 9% Ni steel plate is discussed here, centering on its mechanical properties.

2. Study of Toughness Improvement

2.1 Factors governing toughness of 9% Ni steel

Nine percent nickel steel was melted in the laboratory, rolled into a 15 mm thick plate, quenched and tempered at different temperatures, and Charpy impact tested at -196°C. Fig. 1 shows the change of Charpy absorbed energy at -196°C with the tempering temperature. There clearly exists an optimum tempering temperature range of 550 to 600°C where the 9% Ni steel exhibits high toughness. In other words, the toughness of the 9% Ni steel sharply deteriorates when tempered at temperatures of under 550°C and above 600°C. The causes of embrittlement in

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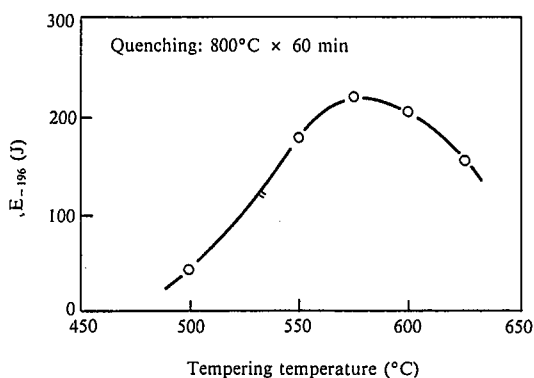


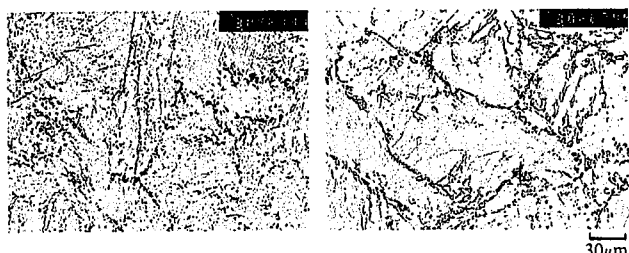
Fig. 1 Effect of tempering temperature (holding time 20 min) on toughness of quenched and tempered (QT) 9% Ni steel (0.05C-0.25Si-0.53Mn-9.09Ni)

these two regions are clear from past studies²⁾, namely:
 (1) 400 to 500°C: intergranular embrittlement (temper embrittlement)
 (2) 600°C and above: Embrittlement due to an instability of temper-formed austenite

Grain size and alloying elements are considered additional factors that affects the toughness of the base 9% Ni steel. The former depends on such process conditions as reheating, rolling and heat treating conditions, while the latter affects toughness through the factors (1) and (2) above. To improve the toughness of the 9% Ni steel, therefore, the two primary embrittlement factors must be controlled and appropriate process conditions must be selected.

The susceptibility of the 9% Ni steel to the temper embrittlement (1) can be effectively reduced by lowering the contents of phosphorus and other intergranular segregation elements as is well known. Manganese also enhances the embrittlement susceptibility, and must therefore be lowered as far as possible²⁾. Alloy additions must always take the strength-toughness balance into consideration.

Another metallurgical characteristic of the 9% Ni steel is the presence of temper-formed austenite precipitated during tempering. The temper-formed austenite is stably present in the optimum tempering temperature range for the 9% Ni steel shown in Fig. 1. However, tempering at and above 600°C rapidly increases the amount of temper-formed austenite and lowers the alloy content therein. Therefore, when strained during cooling at -196°C or Charpy impact test, the temper-formed austenite transforms to martensite and brings about a loss of toughness³⁾. Photo 1 shows the state of carbides changing to austenite during tempering. At the optimum tempering temperature of 575°C, many carbides, dark in the photo, precipitate together with a small



(a) Q: 800°C (60min) + T: 575°C (20min) (b) Q: 800°C (60min) + T: 625°C (20min)

Photo 1 Change in microstructure of 9% Ni steel (QT) with tempering temperature

amount of temper-formed austenite at prior austenite grain boundaries and martensite lath boundaries. At the higher tempering temperature of 625°C, carbides completely change to temper-formed austenite.

Effective utilization of stable temper-formed austenite, therefore, is one of the essential requirements for improving the toughness of the 9% Ni steel.

2.2 Toughness improvement by intercritical quenching

Through its research on the toughness improvement of nickel-bearing steel, Nippon Steel developed in the early 1970s an intercritical quenching process (lamellarizing process) that can substantially improve the low-temperature toughness of the steel^{4,5)}. The nickel-bearing steel is conventionally manufactured by quenching and tempering (QT) and contains austenite formed during the process. The new process adds an additional stage of heating to the intercritical region and rapid cooling therefrom between the quenching and tempering stages. Termed the QLT process where L stands for lamellarizing, it can remarkably improve the low-temperature toughness of the nickel-bearing steel.

In this study, application of the QLT process to the manufacture of heavy 9% Ni steel plates was investigated. The QLT process was traditionally outside the scope of JIS and ASTM, and its application to the 9% Ni steel remained in the research phase⁶⁾. Recently, however, JIS and ASTM standards relating to the 9% Ni steel were revised to permit the application of the QLT process to the 9% Ni steel.

Fig. 2 shows the results obtained when the QLT process was applied to the same 9% Ni steel plate as the one used to obtain the results of Fig. 1. As evident when compared with Fig. 1, the QLT process results in a high toughness of over 200 J in a wide tempering temperature region from 500 to 625°C. This may be attributed to the aforementioned inhibition of the two primary embrittlement factors for the 9% Ni steel.

The 9% Ni steel was microalloyed with 0.009 wt% phosphorus, melted in the laboratory, heat treated as specified, reheated to 425 to 525°C for embrittlement, and examined for resultant change in toughness in order to investigate the temper embrittlement susceptibility of QT and QLT steels. The results are given in Fig. 3. As can be seen from the figure, the QT and QLT steels are most extensively embrittled in the temperature region of 450 to 475°C. The toughness of the QT steel is reduced to about 50 J, while the embrittlement is recognizable but is very slight in the QLT steel. This means that the QLT steel has very high resistance to temper embrittlement.

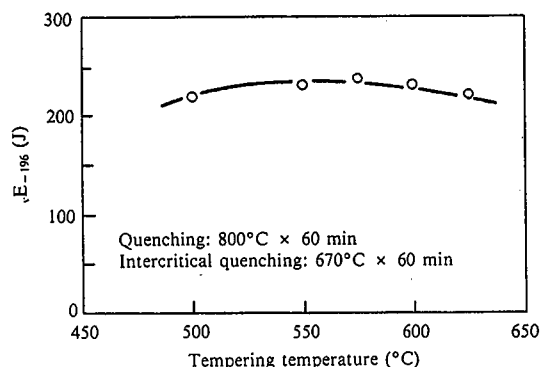


Fig. 2 Effect of tempering temperature on toughness of QLT steel

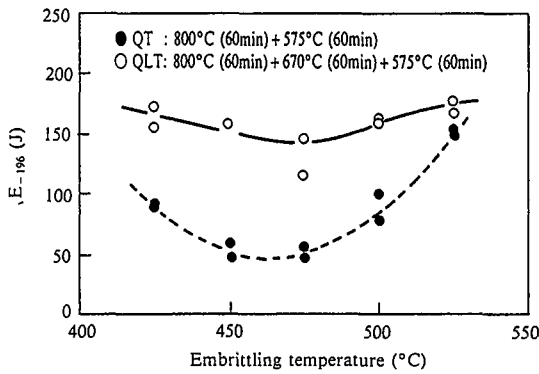


Fig. 3 Effect of embrittling temperature (holding time 1,200 min) on toughness of QT and QLT steels (0.05C-0.25Si-0.53Mn-0.009P-9.32Ni)

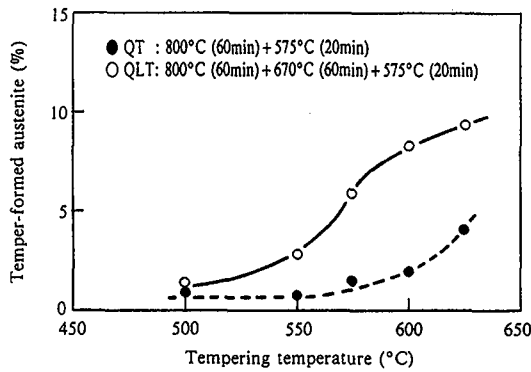


Fig. 4 Effect of tempering temperature (for 20 min) on amount of temper-formed austenite after subzero treatment in QT and QLT steels (0.05C-0.25Si-0.53Mn-9.09Ni)

The QT and QLT steel specimens tempered as shown in Figs. 1 and 2, respectively, were then subzero treated, and examined by X-ray diffraction for the amount of temper-formed austenite. Fig. 4 shows the results. In the case of the QT steel, the amount of temper-formed austenite indicated by the closed circle (●) is 1 to 2% in the optimum tempering temperature region of 575 to 600°C, and it increases to about 4% at the tempering temperature of 625°C where the loss of toughness is recognized. This result agrees well with the change of carbide into austenite as shown in Photo 1, and indicates that the presence of temper-formed austenite in a small amount is instrumental in improving the toughness of the 9% Ni steel. This is confirmed also by Ohoka et al³⁾.

In the case of the QLT steel, the amount of temper-formed austenite which is indicated by the open circle (○) is higher than that in the QT steel at all the tempering temperatures concerned. At 625°C, the temper-formed austenite is present in an amount of about 10%, but the loss of toughness is not at all recognizable at such a high tempering temperature. Generally, the enrichment of alloying elements is necessary to stabilize the temper-formed austenite. The wide difference in the stability of temper-formed austenite between the QT and QLT steels at the same tempering temperature suggests that the QT and QLT steels are greatly different in the form of austenite precipitation after the intercritical quenching treatment.

Photo 2 shows microstructures of the QLT steel observed by transmission electron microscope. As compared with that of the QT steel, the microstructure of the QLT steel reveal the follow-

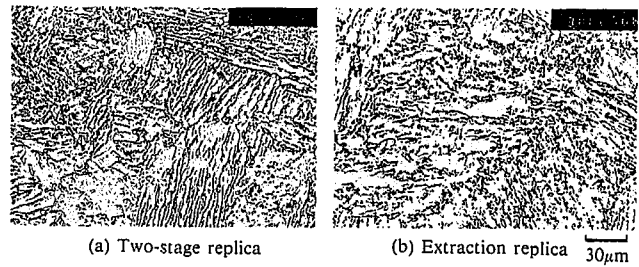


Photo 2 Microstructures of QLT steel (Q: 800°C, L: 670°C, T: 600°C)

ing characteristics:

- (1) Lamellar ferrite, which transformed to austenite along prior austenite grain boundaries and martensite lath boundaries during intercritical quenching (lamellarizing) and was again tempered, is finely formed (prior austenite grain boundaries are difficult to discriminate).
- (2) Fine carbide particles (some containing austenite) are present in the fine lamellar ferrite structure.

Namely, the fine lamellar ferrite transformed to austenite on intercritical heating, so that it is enriched in such elements as carbon and nitrogen.

Therefore, when the lamellar ferrite is quenched in that condition, there is obtained martensite where the alloying elements are enriched, and austenite formed by tempering the martensite is believed to be present in a stabler form than austenite formed in the QT steel. Further, since many temper-formed austenite precipitation sites are present in the fine lamellar ferrite, fine austenite is present in a greater amount than in the QT steel as shown in Fig. 4. The austenite thus formed is proved to be extremely stable⁵⁾. In addition, since ferrite that remained unchanged during the lamellarizing treatment is tempered again, the toughness of the ferrite matrix may improve with sufficient dislocation recovery.

On top of that, low-temperature temper embrittlement is considered to be significantly reduced by a large increase in grain-boundary area with microstructural refinement and by reduction in prior austenite grain boundary embrittlement with formation of lamellar ferrite structure on the prior austenite grain boundary.

Factors in the toughness improvement of the QLT steel may be summarized as follows:

- (1) Grain size refinement by the formation of fine ferrite structure
- (2) Massive precipitation of stable, fine temper-formed austenite
- (3) Presence of fully tempered ferrite matrix
- (4) Reduction in grain-boundary embrittlement by precipitation of temper-formed austenite on prior austenite grain boundaries

The above discussion indicates the possibility of manufacturing 9% Ni steel plates of excellent toughness through the application of lamellarizing treatment.

3. Results of Manufacturing 40 and 45 mm Thick 9% Ni Steel Plates with Superior Low-temperature Toughness

3.1 Steel plate manufacturing process and base metal service performance test results

For a procedure test, the nine percent nickel steel was melted in a 250 ton basic oxygen furnace, ingot cast, slabbed, rolled to 40 and 45 mm thick plates at a plate mill, and was mill tested to confirm its service performance properties. Table 1 gives the

Table 1 Chemical composition of test steels (mass%)

Plate thickness	C	Si	Mn	P	S	Ni
40mm	0.05	0.25	0.57	0.002	0.001	9.46
45mm	0.05	0.24	0.56	0.002	0.001	9.39

Table 2 Results of base-metal mechanical tests

Plate thickness (mm)	Thick-ness lo-cation	Orienta-tion*1	Tensile test*2				Charpy impact test (-196°C)*3	
			0.2% off-set yield strength (MPa)	Tensile strength (MPa)	Elonga-tion (%)	Reduc-tion in area (%)	Energy (J)	Shear area of fracture (%)
40	t/4	L	619	737	33	82	281	100
			635	734	34	79	279	100
		T	621	728	33	79	266	100
			624	734	32	79	259	100
	t/2	L	632	734	34	81	299	100
			618	734	35	81	268	100
		T	622	737	34	80	278	100
			624	738	34	78	247	100
45	t/4	L	608	712	34	83	279	100
			610	713	34	83	286	100
		T	615	713	33	82	276	100
			615	713	33	82	291	100
	t/2	L	616	712	33	83	289	100
			608	713	33	82	299	100
		T	613	715	33	82	295	100
			611	712	33	83	269	100
JIS specified value			≥590	695-830	≥21		Min ≥ 34	Ave ≥ 41

*1: L: Rolling direction, T: Direction normal to rolling direction
 *2: JIS No. 4 specimens
 *3: JIS No. 4 specimens

chemical compositions of the test plates.

The steel plates were QLT treated and tested for various service performance properties.

The test results on base-metal mechanical properties are listed in Table 2. Each of the 40 and 50 mm thick 9% Ni steel plates fully meets the JIS specified values. The impact absorbed energy is above 250 J, and all specimens exhibit 100% shear area of fracture.

Table 3 shows the through-thickness tensile test results on the 40 and 45 mm thick 9% Ni steel plates at room temperature and -196°C, respectively. Even at -196°C, the reduction of area is more than 60%, with fairly small anisotropy.

Table 4 gives the results of impact test after strain aging. The rate of decrease in average absorbed energy with respect to the base metal is as shown in Fig. 5. The specimens were aged by prestraining to 0, 3, or 5% in the rolling direction, holding at 250°C for 1 h, and cooling in air. The specimens were taken at quarter- and mid-thickness locations in both the longitudinal and transverse orientations and were impact tested at -196°C. All the specimens tested demonstrated that even after 5% strain aging, toughness is as high as more than 250 J, and is no less than 90% of that of the base metal. This test result shows that the loss of toughness due to stain aging is small.

3.2 Test results on welded joints

This section describes the results of performance tests conducted on welded joints of 40 mm thick 9% Ni steel plates.

Table 5 shows the test welding conditions. Automatic tungsten inert gas arc welding (TIG) and shielded metal arc welding (SMAW) were performed in the vertical position, and submerged

Table 3 Results of tensile test in thickness direction

Plate thickness (mm)	Specimen	Orienta-tion*1	Tempera-ture (°C)	Tensile strength (MPa)	Reduction in area (%)
40	Diameter of parallel portion: 10 mm	Z	RT	723	77
			-196	723	75
	Length of parallel portion: 35 mm		RT	1126	66
			-196	1129	60

*1 Z: Thickness direction

Table 4 Results of Charpy impact test after strain aging at -196°C

Plate thick-ness (mm)	Thick-ness location	Orientation	0% strain aging		3% strain aging		5% strain aging	
			Energy (J)	Shear area of fracture (%)	Energy (J)	Shear area of fracture (%)	Energy (J)	Shear area of fracture (%)
40	t/4	L	281	100	279	100	279	100
			295	100	260	100	248	100
		303	100	276	100	284	100	
		T	250	100	274	100	264	100
	284		100	279	100	264	100	
	t/2	L	273	100	253	100	251	100
			275	100	265	100	262	100
		T	299	100	251	100	276	100
234			100	259	100	259	100	
45	t/4	L	258	100	290	100	269	100
			230	100	265	100	266	100
		T	261	100			261	100
			265	100			257	100
	t/2	L	250	100			250	100
			276	100			276	100
		T	265	100			265	100

Table 5 Test plate welding conditions

Welding process	Plate thickness (mm)	Welding material	Current (A)	Voltage (V)	Speed (cm/min)	Heat input (kJ/min)	Interpass temperature (°C)
Automatic TIG	40	NITTETSU FILLER 196	220-290	10	4.4-8.0	2.5-2.9	<100
SMAW		YAWATA WELD B(M)	130-140	25	5.0-7.3	3.0-3.8	<100
SAW		NITTETSU FILLER 190 × NITTETSU FLUX 10H	360-380	25-26	35-70	1.1-1.3	<100

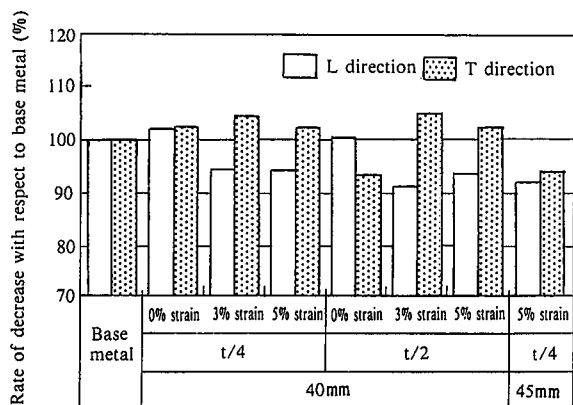


Fig. 5 Rate of decrease in absorbed energy by strain aging

arc welding (SAW) was conducted in the horizontal position. All the welded joints were made using 70% Ni austenitic filler metal.

Table 6 shows the tensile test results on welded joints. All welded joints exhibited high tensile strength values of more than 730 MPa, and were broken at the location of the soft weld metal.

Charpy impact test results on automatic TIG, SMAW, and SAW welded joints at -196°C are shown in Figs. 6, 7, and 8, respectively. Since the weld metal (WM) deposited by using the 70% Ni welding material was lower in strength than the base metal, cracks shifted toward the weld metal when the welded joint was tested near the fusion line (FL). Although the absorbed energy

Table 6 Tensile test results on welded joints

Welding process	Specimen	Tensile strength (MPa)	Breaking location*1
Automatic TIG	JIS Z 3121 No. 1 specimen	756	WM
		752	WM
SMAW		752	WM
		743	WM
SAW		737	WM
		748	WM

*1 WM: Weld metal

value of the welded joint is thus largely influenced by that of the weld metal, the absorbed energy value of the fusion line is more than 90 J even for SMAW joints with weld metal of relatively less toughness and is more than 150 J for automatic TIG joints with weld metal of good toughness. The shear area of fracture was 100% for all specimens.

3.3 Test results on fracture toughness

Described hereunder are the results of investigation made on the performances of the base metal and welded joint against the brittle fracture initiation and propagation. The purpose was to evaluate the safety of heavy 9% Ni steel plates as materials of

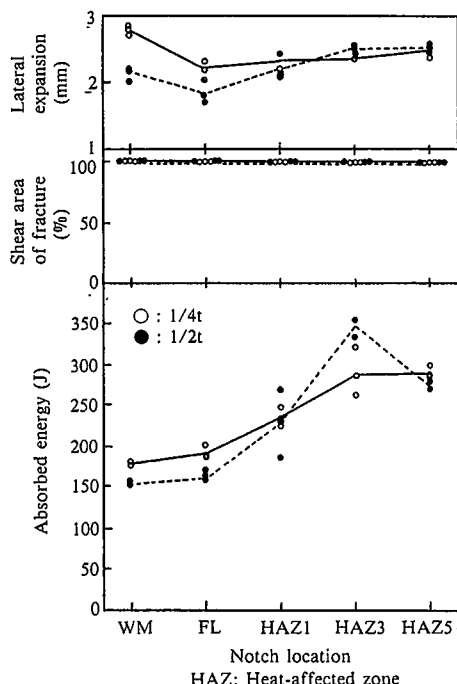


Fig. 6 Charpy impact test results on automatic tungsten inert gas arc welded joints

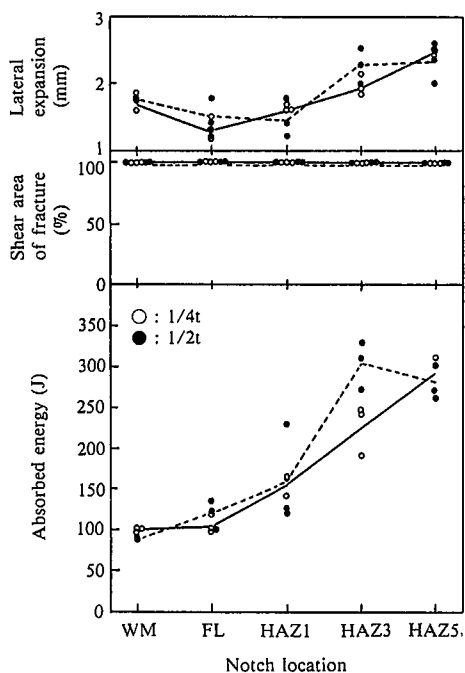


Fig. 7 Charpy impact test results of shielded metal arc welded joints

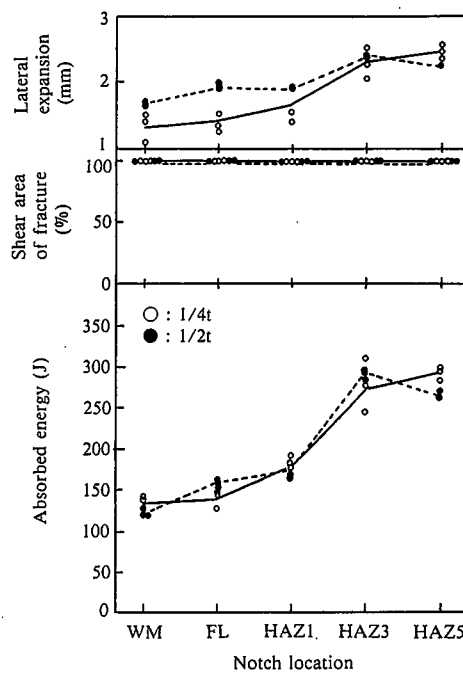


Fig. 8 Charpy impact test results of submerged arc welded joints

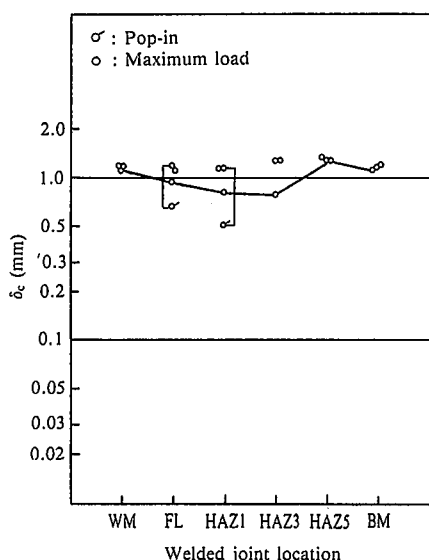


Fig. 9 CTOD test results on automatic TIG welded joints at -165°C

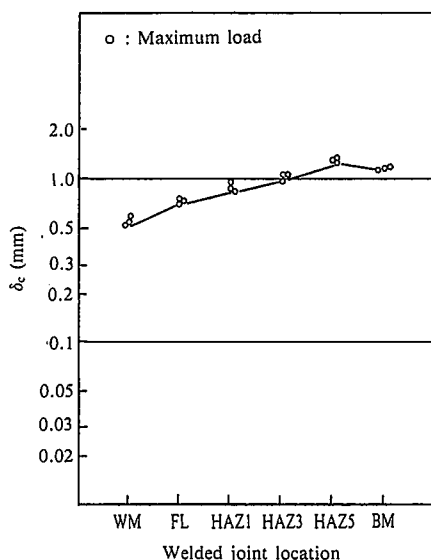


Fig. 10 CTOD test results on SMAW welded joints at -165°C

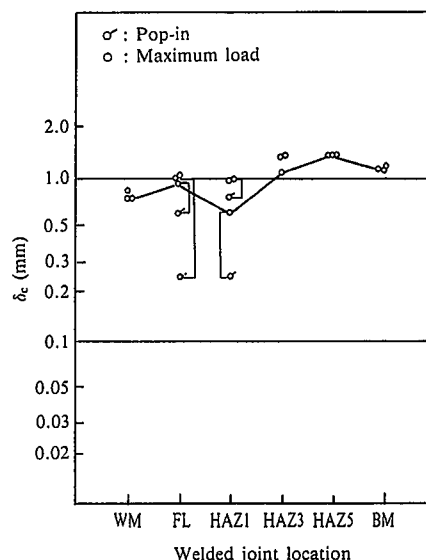


Fig. 11 CTOD test results of SAW welded joints at -165°C

construction for large LNG tanks.

3.3.1 Fracture toughness against brittle crack initiation

Table 7 shows the results of CTOD test on base metal conducted according to BS 5762-1979. The CTOD value is higher than 1 mm at the LNG temperature of -165°C and more than 0.4 mm even at -196°C .

The CTOD test results on automatic TIG, SMAW, and SAW welded joints at the LNG temperature are shown in Figs. 9, 10, and 11, respectively. Pop-in is recognized in automatic TIG and SAW joints. The minimum CTOD value at pop-in is more than 0.2 mm, and the critical CTOD (δ_c) obtained from the maximum load is more than 0.5 mm.

The conventional method of evaluating the fracture toughness against brittle crack initiation involved determining the local strain acting at the crack tip and studying the necessary toughness and δ_c value, considering the actual operating conditions of LNG tanks and other factors, according to WES-2805: Method of Assessment for Defects in Welded Joints with Respect to Brittle Fracture. Recently, Machida et al⁷⁾ investigated by a similar method the required fracture toughness of 40 mm thick steel plates used for side shells of 140,000-m³ class tanks. According to their results, the required value of δ_c was 0.085 mm at the cross of a vertical joint and a horizontal joint where residual welding stress is most severely superimposed on the membrane stress during a short-cycle earthquake, the most punishing design condition. The CTOD of the base metal and weld metal of the heavy 9% Ni steel plate is far higher than 0.085 mm as described earlier. In other words, heavy 9% Ni steel plates have excellent fracture toughness against brittle crack initiation as materials of construction for LNG tanks.

As a test method capable of more closely simulating the fracture toughness against brittle crack initiation of actual welded joints than the CTOD test, the through notched cross-joint wide plate tensile test was conducted using automatic TIG and SMAW joints. Fig. 12 shows the configuration of the test sample. The test results are given in Table 8, and a typical fracture surface is shown in Photo 3. The test introduced a notch twice as large as the plate thickness as a through flaw and measured the frac-

Table 7 CTOD test results of base metal

Plate thickness (mm)	Specimen orientation	Temperature ($^{\circ}\text{C}$)	δ_c (mm)		
			>1.172	>1.184	>1.132
40	L	-165	>1.172	>1.184	>1.132
		-196	0.571	0.528	0.514
	T	-165	1.051	1.123	1.071
		-196	0.409	0.429	0.475

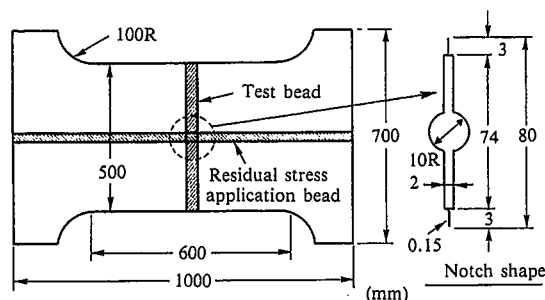


Fig. 12 Shape of specimen for through notched cross-joint wide plate tensile test

ture stress at the LNG temperature. For reference, the crack opening displacement at 7 mm apart from the crack tip was measured with a clip gage, and δ_c at the onset of fracture was calculated by the BSC model. With both the automatic TIG and SMAW joints, pop-in produced a fine fracture, and the crack immediately propagated through the weld metal and led to the failure of the welded joint.

Past research⁸⁾ identified this crack propagation behavior as a phenomenon commonly observed in the 9% Ni steel welded using austenitic weld metal and clarified that all the cracks shift into the weaker weld metal in welded joint specimens. Given the brittle fracture of LNG tanks, the 9% Ni steel welded joints are not considered susceptible to a large-scale brittle fracture despite the occurrence of pop-in. The fracture stress at the maximum load will thus assume greater importance.

As can be seen in the test results shown in Table 8, both types of welded joints exhibit a fracture stress of more than 660 MPa,

Table 8 Results of through notched cross-joint wide plate tensile test

Welding process	Notch location	Plate thickness (mm)	Plate width (mm)	Test temperature (°C)	Gross stress (MPa)	Net stress (MPa)	δ_c (mm)	Remarks
Automatic TIG	FL	40.2	499.9	-165	676	802	2.32	Pop-in Maximum load
					686	815	3.08	
SMAW	FL	40.1	499.8	-165	584	695	0.48	Pop-in Maximum load
					665	790	1.58	

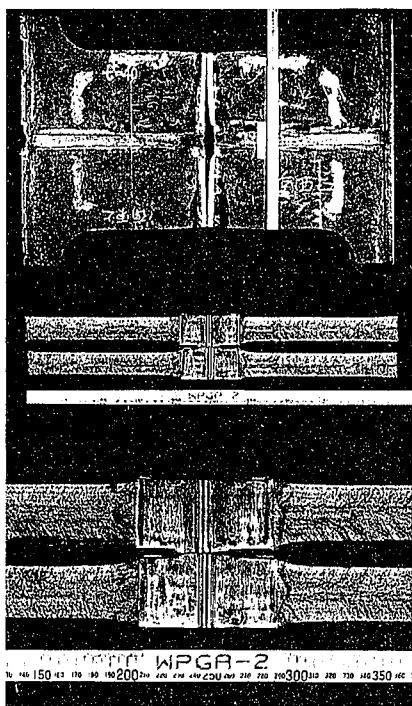


Photo 3 Fractographs of specimen for through notched cross-joint wide plate tensile test (automatic TIG joint, test temperature: -165°C)

and the stress is greater than 580 MPa even when having the pop-in. These values are far higher than the allowable stress of 375 MPa⁷⁾ during earthquakes as specified in LNG aboveground storage guidelines. Coupled with the CTOD test results discussed earlier, the through notched cross-welded wide plate tensile test results confirm that heavy 9% Ni steel plates have superior fracture toughness against brittle crack initiation in both the base metal and weld metal.

3.3.2 Brittle crack propagation arresting capability

In the storage of easy-to-vaporize liquids such as LNG, it is essential to arrest the brittle crack propagation in as short distance as possible when the crack could not be prevented from occurring. From this standpoint, the short crack arrest properties of the 9% Ni steel have been studied^{8,9)}. Among these studies, the validity of the duplex ESSO test method in evaluating actual brittle crack arrest properties has been verified. The duplex ESSO test was then conducted on the base metal and weld metal of the 9% Ni steel.

Shapes of duplex ESSO test specimen are shown in Fig. 13. The test plate and crack starter plate are butt welded using a 3.5% Ni welding material. Particularly with the welded-joint duplex ESSO test specimen, a 6 mm deep groove is provided on each side to introduce a crack into the fusion line of the test plate.

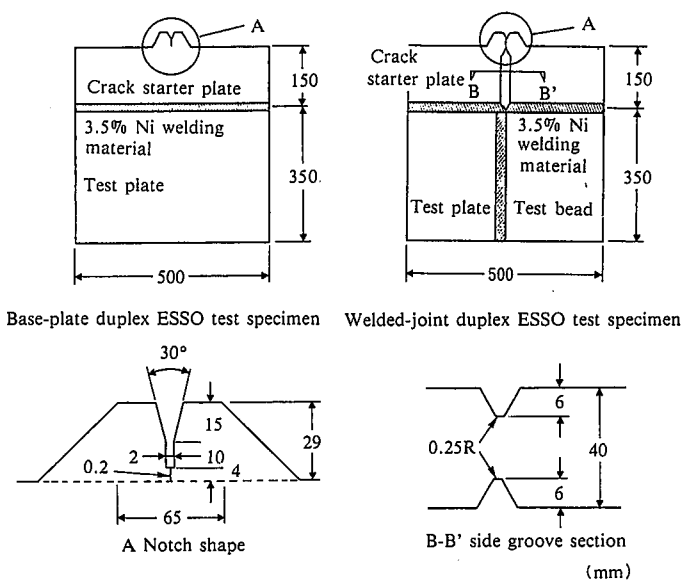


Fig. 13 Shapes of specimens for duplex ESSO test

Table 9 Results of duplex ESSO test

Test plate	Plate thickness (mm)	Test temperature (°C)	Applied stress (MPa)	Crack length (mm)	Applied K_{ca} (MPa·√m)	Judgment
Base metal	39.9	-165	392	170	282	No go
	40.0	-165	392	177	282	No go
	39.9	-196	392	173	282	No go
	39.9	-196	392	174	282	No go
	45.8	-165	392	172	282	No go
	45.8	-196	392	175	282	No go
Automatic TIG joint	40.4	-165	392	163	282	No go

The duplex ESSO test results are shown in Table 9, and the fracture surface of a duplex ESSO test specimen in Photo 4. Given the applied stress intensity factor K calculated from the starter distance of 150 mm, the welded joint section is found to have crack arrest toughness of more than 282 MPa·√m at the LNG temperature, and the base metal to have higher crack arrest toughness even at -196°C.

Machida et al.⁷⁾ discussed the required crack arrest toughness of 40 mm thick 9% Ni steel plates from the viewpoint of a short crack arrest distance. As a result, the toughness required to arrest brittle cracks initiated in the circumferential joint section and to intrude them to the base plate in normal direction to the joint is given by

$$K_{ca} = 0.38 \cdot \sigma + 75 \quad (1)$$

where K_{ca} (MPa·√m) is the required fracture toughness, and σ (MPa) is the membrane stress acting on the side shell of an LNG

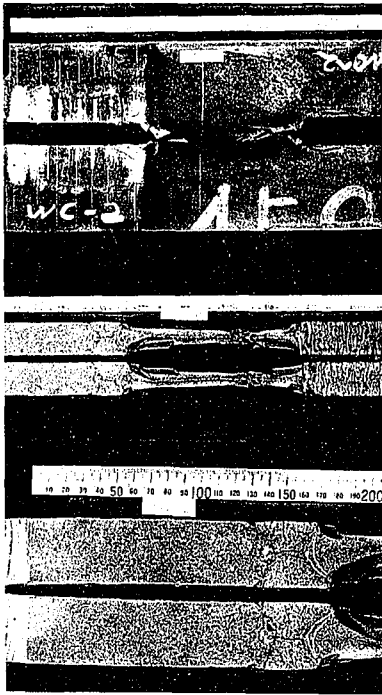


Photo 4 Fractographs of duplex ESSO test specimen (test temperature: -165°C , applied stress: 392 MPa, 40 mm thick base plate)

tank. This equation calculates the value of K by considering the residual stress in the circumferential joint and expresses, as a function of the membrane stress, the value of K at the point where it exhibits a maximum value in the vicinity of the weld interface. When calculated by Eq. (1), the required value of K_{ca} during an earthquake ($\sigma = 220$ MPa) is known to be $159 \text{ MPa}\cdot\sqrt{\text{m}}$. This value is clearly much smaller than the applied K value in the duplex ESSO test results described above. These results confirm that the heavy 9% Ni steel plates and their welded joints have high fracture toughness against brittle crack initiation and propagation for large LNG tanks.

4. Conclusions

An intercritical quenching process was studied for application to the manufacture of heavy 9% Ni steel plates with sufficiently high low-temperature toughness for large LNG tanks. The following results were obtained:

- (1) The QLT process applied to the 9% Ni steel suppresses temper embrittlement, stabilizes temper-formed austenite, and more stably imparts high low-temperature toughness than the conventional QT process.
- (2) The base-metal mechanical properties of trially mill-manufactured 40 and 45 mm thick 9% Ni steel plate fully conform to the applicable JIS specifications. Even after 5% strain aging, its Charpy impact test value is over 90% of its base metal value. The heavy 9% Ni steel plates thus exhibit stable impact properties. In the Charpy impact test on welded joints, the absorbed energy was good at more than 80 J at -196°C , and brittle fracture was observable on none of the specimens.
- (3) According to the CTOD test results, the critical CTOD value is more than 1 mm in the base metal and more than 0.5 mm in the weld metal. According to the results of through notched cross-welded wide plate tensile test with a crack length of 80

mm, the fracture stress is more than 580 MPa even at pop-in. Their fracture toughness against brittle crack initiation and propagation was studied as materials of construction for large LNG storage tanks in the light of WES-2805, and the result was that they fully satisfy the specifications of the standards in both the base metal and welded metal.

- (4) According to the duplex ESSO test results on the base metal and welded metal, the crack arrest toughness is more than $282 \text{ MPa}\cdot\sqrt{\text{m}}$ at -196°C for the base metal and at the LNG temperature for the welded metal. This crack arrest toughness is high enough to withstand short-cycle earthquakes.

As discussed above, 40 and 45 mm thick 9% Ni steel plates heat treated by the QLT process have excellent crack arrest properties at the LNG temperature and liquid nitrogen temperature. The plates are expected to find wide use as materials of construction for large LNG storage tanks and other cryogenic containment vessels.

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