A STUDY OF FRACTURE TOUGHNESS AND MICROSTRUCTURES IN THE WELD HEAT-AFFECTED ZONE OF QLT-PROCESSED 9% Ni STEEL

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ABSTRACT

The objective of this study, based on the concept of fitness-for-purpose, is to evaluate the fracture toughness in X-grooved weld HAZ(heat-affected zone) of QLT(quenching, lamellarizing and tempering)-processed 9% Ni steel, qualitatively and quantitatively, and analyze the relation between the fracture toughness and microstructure. In general, CTOD test is widely used to determine the fracture toughness of steel weldments. But several problems of accuracy have been brought up. To avoid those, in this paper, modified CTOD testing method is proposed and used for weld HAZ for 9% Ni steel. In addition, microstructure of HAZ is observed and analyzed by OM, SEM and XRD.

From the results, HAZ toughness of QLT-9% Ni steel decreased as the evaluated region approaches the fusion line from base metal. The decrease in toughness was apparently partly caused by the reduction of the retained austenite content resulting from the decrease in nucleation sites for $\alpha \rightarrow \gamma$ reverse transformation due to the increasing fraction of coarse-grained region. On the other hand, due to the poor stability of retained austenite in the mixed zone of weld metal/base metal, toughness drop in F. L.~F. L.+3mm was larger than that in F. L.+5mm and F.L.+7mm with decreasing test temperature.

INTRODUCTION

The demand for LNG (Liquified Natural Gas) in the world is continuously increasing due to its convenience and advantage as a clean energy source with high energy density. Especially, the need for LNG in Korea has increased more rapidly than in other countries because of the fast economic growth and the change of life style in Korea. Therefore, steel company in Korea has also begun to produce large tonnage of 9% Ni steel, which is used for inner wall of LNG storage tank due to its excellent fracture toughness at 111K, LNG temperature.

Korean steel company, POSCO adapted the QLT heat treatment which is not standardized like QT, NNT and DQT heat treatment for 9% Ni steel. The QLT process, widely used for 3.5% and 5.5% Ni steel, includes lamellarizing heat treatment (quenching from the temperature between A_{C1} and A_{C3}) between quenching (from the temperature above A_{C3}) and tempering and considerably enhances cryogenic toughness. This is the result of the larger amount of retained austenite and the refinement of effective grain size.

This steel is welded by SAW, SMAW and GTAW process with 70% Ni based superalloy (Inconel type or Hastelloy type) as weld metal. Due to the fact that, generally, weld heat-affected zone (HAZ) is the weakest region of the most welded structures, HAZ toughness of QLT-9% Ni steel is very important for the safety performance of LNG storage tank. To evaluate the HAZ toughness of QT- 9% Ni steel, Charpy V-notch impact test and CTOD test have been carried out using K-grooved weldment or synthetic specimens.[1-3]

In this study, with the concept of fitness-for-purpose, fracture toughness of X-grooved HAZ for the real situation of LNG storage tank, was estimated by modified CTOD test newly proposed. The amount of retained austenite, the effective grain size and the fractographs of weld HAZ for QLT-9% Ni steel were also analyzed by XRD, OM, SEM.

MODIFICATION OF CTOD TEST FOR THICK WELDMENT WITH X-GROOVE

Before the development of fracture mechanics, Charpy V-notch impact test was commonly used to assess the fracture toughness of weldments. Charpy test has many advantages such as, specimen preparation and testing method are very simple and it is easy to select the notch location for the various regions in HAZ. However, there are some difficulties to explain the practical failure analysis due to the absence of fracture mechanics concepts such as crack initiation, propagation and stress fields around crack tip. So, CTOD test is carried out to evaluate toughness of weldment in accordance with BS 5762(formulated in 1979)[4], BS 7448(1991)[5] or ASTM E1290(1989)[6]. This CTOD test based upon elasto-plastic fracture mechanics(EPFM) is more easily applied to weldments than other fracture test because the test has also the advantages of Charpy test, and unlike K_{IC} and J_{IC} test, there is no requirement of plain strain condition. But standardized CTOD test for the homogeneous materials has many problems in applying itself to the weldments that have metallurgical and mechanical characteristics. Although BS draft(1986)[7], ASTM draft(1991)[8] suggested CTOD test for weldment with K-groove or half-V-groove, CTOD test for weldments is not standardized, and especially for weldment with X-groove, there is no draft. So, in this study, modified CTOD test for X-grooved weldment was suggested as follows.

Mechanical notches for the tests were located at fusion line (F.L.), F.L. + 1mm, F.L. + 3mm, F.L. + 5mm, F.L. + 7mm, respectively. Figure 1 shows those locations.

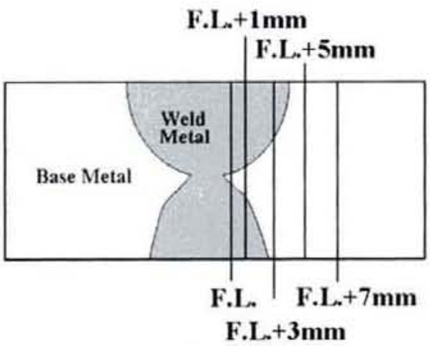

Figure 1. Notch locations of CTOD specimen with X-groove .

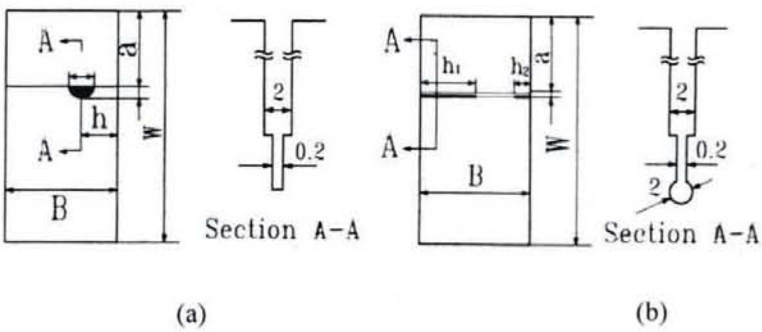

Figure 2. Methods of fatigue precracking by Kajimoto et al.[12] (a) partial arc notch and(b) drill hole notch .

Fatigue precracking

The first problem of CTOD test for thick weldment like this case, is the non-uniform shape of fatigue precrack through thickness due to the influence of welding residual stress. Unlike the situation of single-pass-welded thin plate, which has large residual stress along welding direction (σ_x) and small residual stress of transeverse direction (σ_y), multi-pass welded thick plate has distribution of welding residual stresses through thickness ($\sigma_x(Z)$).

To prevent this problem and get uniform precrack, many investigations have been previously performed. Dawes et al.[9] proposed the local compression method which was accepted by BS draft[7], ASTM draft[8] and IIW WG Guideline[10] and is most generally used. In this method, 1% compressive loading is applied before precracking to redistribute the welding residual stress mechanically. However, when using this method, more loads are needed for weldment with larger thickness and overestimation of toughness is possible because the compressive plastic stress exists. Like using Chevron notch,[11] other methods also failed to get the satisfactorily uniform precrack. On the other hand, Kajimoto et al.[12] proposed the other method in which partial arc notch was made at the region under relatively compressive residual stress and drill hole notch was made at the region under relatively tensile residual stress, as shown in Figure 2. They reported that the notches induced a uniform precrack successfully, and did not affect the material toughness. Now, in this study, Kajimoto's method was re-examined. They measured the residual stress by the method, as shown in Figure 3(a), using XRD. But measured results indicated the mixed stress of σ_y and σ_z, excluding the σ_x to the direction of precrack propagation. Therefore, we sectioned specimen and assessed the mixed stress of σ_x and σ_y with the arrow direction shown in Figure 3(b) using XRD. Since the value of σ_y is only 5-10 % of σ_x and uniform tensile residual stress at the center of the welded plate where specimens were taken, the measured results represented the relative stress distribution ($\sigma_x(Z)$) successfully. Also, unlike Kajimoto's way, we measured the stress after making of mechanical notch because stress state would be changed after notch making, and only the partial arc notch was used in this study because the drill hole notch blunted the precrack tip and thus, toughness could have been overestimated.

Secondly, to determine the maximum compressive load for fatigue precracking, $P_{f,max}$, calculated in accordance with the following equation in the standard method, the average of yield strength and ultimate tensile strength, σ_Y is used.

$$P_{f,max} = 0.5(B(w-a)^2\sigma_Y/S) \quad (1)$$

where the other symbols are in accordance with ASTM E1290[6].

But in F.L.~F.L.+3mm, the weld metal with low strength and the base metal with high strength were mixed. To prevent rapid precrack propagation, relatively low strength of weld metal was used for $P_{f,max}$.

Calculation of CTOD

The CTOD values are calculated from the data measured by a CTOD test via the method as will be explained below. For the conventional calculation of CTOD for homogeneous

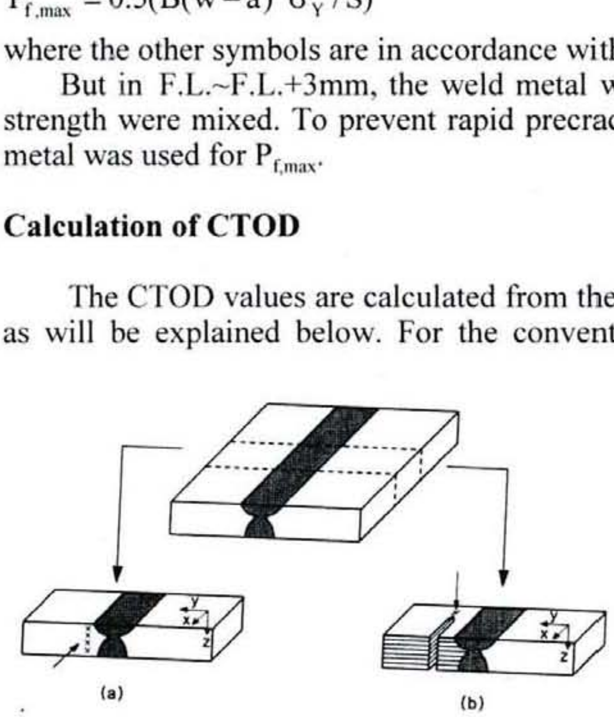

Figure 3. Measurement of welding residual stress through thickness. (a) Kajimoto's way and (b) method for this study.

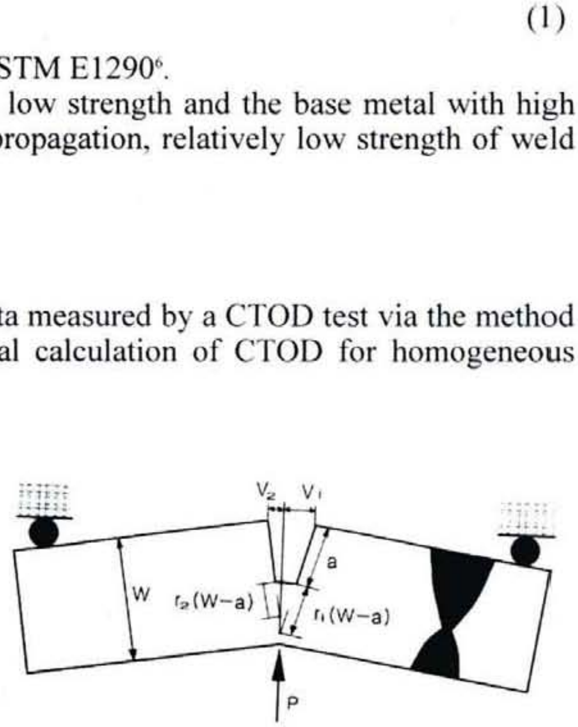

Figure 4. The modified Hinge model for the CTOD calculation of weldment with strength mismatch.

materials, in general, the combined model of strip yield model and hinge model is used in the following equation:

$$\delta = \frac{K^2(1-\nu^2)}{2\sigma_{YS}E} + \frac{r(w-a)}{r(w-a)+a}V \tag{2}$$

where the symbols are in accordance with ASTM E1290[6], and inner knife edge is assumed.

However, as shown in Figure 1, this test systems are mixed of the weld metal and the base metal of 9% Ni steel, especially, in the range of F.L.~F.L.+3mm. Thus, the determination of yield strength σ_{YS} in Eq. (2) is problematic due to the materials inhomogeneity. When the position of notch is located in the range of F.L.~F.L.+3mm, the CTOD can be obtained from Eq. (3) modified by applying the rule of mixture for yield strength. The fraction of the weld metal, X, decreases linearly with the position of the notch from fusion line.

$$\delta = \frac{K^2(1-\nu^2)}{2[\sigma_{YS,WM}X + \sigma_{YS,BM}(1-X)]\,E} + \frac{r(w-a)}{r(w-a)+a}V \tag{3}$$

where the yield strength of the weld metal is 450MPa at room temperature and assumed not changed although cooled to 111K because its crystal structure is FCC, whereas that of the base metal is 650MPa at room temperature and 910MPa at 111K.

By the way, in the range of F.L.+5mm~F.L.+7mm, since the notch position is only within the base metal region as shown in Figure 1, the effect of the materials inhomogeneity on σ_{YS} can be avoided. But, as elaborated before, the strength mismatch exists due to the presence of ductile weld metal (elongation: 43%) used for the improvement of weldability. This strength mismatch causes the asymmetry of plastic zone size around the notch tip. As a result, the crack mouth displacement becomes asymmetric, and so the rotational factor r is separated into r_1 at the right of the notch and r_2 at the left, as shown in Figure 4. Consequently, the CTOD is estimated in this case using the following modified hinge model suggested by FEM analysis of Toyada et al.[13] instead of Eq. (2).

$$\delta = \frac{K^2(1-\nu^2)}{2\sigma_{YS}E} + \left[\frac{r_1(w-a)}{r_1(w-a)+a}\frac{\alpha}{1+\alpha} + \frac{r_2(w-a)}{r_2(w-a)+a}\frac{1}{1+\alpha}\right]V \tag{4}$$

where α is V_1/V_2, as shown in Figure 4.

Examination of specimen after test

Another problem of strength mismatch is the possibility that the experimental results from HAZ with mechanical notch cannot yield the exact fracture toughness of the purposed region due to the asymmetry of plastic constraint on both sides of crack-tip by existence of strength distribution. For example, Minami et al.[14] studied strength mismatch effects of high strength steel when the toughness of CGHAZ was higher or lower than that of weld metal. They found that the crack propagation path and the fracture initiation point deviated from the

Table 1. Chemical compositions and mechanical properties of QLT-9% Ni steel

Chemical Compositions (wt%)						Mechanical Properties (at R.T.)		
C	Si	Mn	P	S	Ni	YP (MPa)	TS (MPa)	EL (%)
0.066	0.24	0.65	0.005	0.005	9.28	650	720	36.30

Table 2. Welding conditions used for this study

Welding method	Edge preparation	Multi-pass layer	Welding materials	Current (A)	Voltage (V)	Speed (mm/min)	Heat input (kJ/cm)
SMAW	X	6	Inconel type	100~120	25	50-70	28

purposed region for strong weld metal/soft base metal system. Thus, to evaluate the deviation of crack propagation by strength mismatch in this case of soft weld metal/strong base metal system, we had to examine the tested specimen in order to see that the crack propagated within the purposed region. The examinations after test were performed to check the notch location, crack path, and fracture initiation point by sectioning the sample, polishing and etching sequentially to evaluate whether they are within the expected region. The sectioning method was along the way proposed by API RP 2Z[15].

EXPERIMENTAL PROCEDURE

Commercial 22mm-plates of 9% Ni steel with "QLT" heat treatment produced by Pohang Iron & Steel Co. were used in this study. Table 1 lists the chemical composition and mechanical properties of the used material. This steel was welded by the SMAW process under the same condition used for real welding of LNG storage tank . The welding condition is listed in Table 2.

Modified CTOD tests at room temperature, 173K and 111K were performed respectively, using 50ton-level dynamic universal test machine. Test specimens were taken from the 22mm-thick X–grooved welded joint. (transverse to the rolling direction)

All samples in this investigation were prepared for metallographic examination using standard techniques. For viewing under the optical microscope, 2% Nital was used as chemical etchant.

X-ray diffractometry was used to determine the amount of retained austenite and the welding residual stress distribution through thickness. The amount of retained austenite was estimated by comparing the integrated peak intensities of $(110)_\alpha$ and $(200)_\gamma$ plane, using CuK_α, while CrK_α and CrK_β for base metal and weld metal respectively, were used to evaluate the welding residual stress. The specimens for XRD were prepared through chemical thinning by solution of 10% HF + 90% H_2O_2 to prevent mechanical damages.

Finally, the fracture morphology of CTOD specimen was observed by Scanning Electron Microscope.

RESULTS AND DISCUSSIONS

Evaluation of Fracture Toughness through modified CTOD test

Figure 5 shows the measured results of residual stress distribution through thickness, $\sigma_x(Z)$, for the uniform fatigue precrack. At the middle of thickness, relatively large tensile

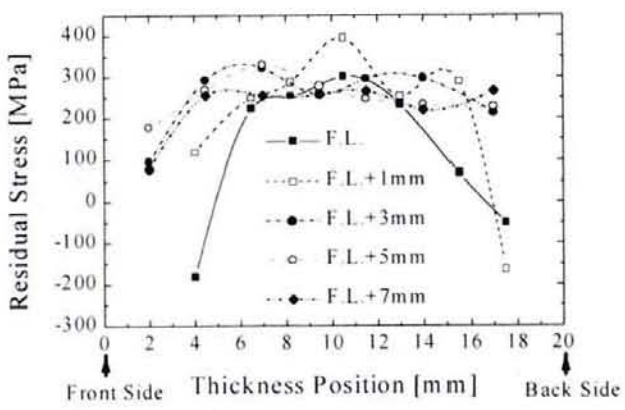

Figure 5. Results of welding residual stress measurements .

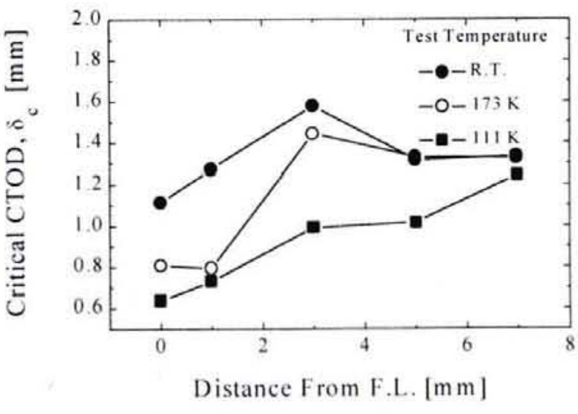

Figure 6. CTOD values at the various notch locations and the various temp.s .

stress existed, and thus, partial arc notches were made near the end of thickness. From this procedure, uniform fatigue precracks were obtained successfully (It is shown in Figure 9 for fractured surfaces).

The results of modified CTOD test for the weld HAZ of QLT-processed 9% Ni Steel are shown in Figure 6 and compared with the results from standard CTOD calculation in Figure 7. Fracture toughness of weld HAZ for QLT-9% Ni steel increased with increasing distance from fusion line. It was clear from these results that the fraction of ductile weld metal, unexpectedly, didn't affect the toughness. From the results, it was also observed that critical CTOD value decreased with decreasing test temperature, i.e., from room temperature to 111K. But, in F.L.~F.L.+3mm, degree of toughness drop was larger than that in F.L.+5mm ~ F.L.+7mm. It is discussed with microstructure analysis in the next section.

After the tests, the crack propagation path and the fracture initiation point were examined. Although crack propagation path showed some deviation to the ductile weld metal, it was not serious. But, in F.L.+3mm, degree of deviation was larger than that of other region, and thus, it resulted in overestimation of critical CTOD as shown in Figure 6. In other regions, results were accepted without doubt.

Fractographs of CTOD specimens are shown in Figure 8 for the test at 111K. In this figure, increase of ductile mode is consistent with CTOD result. In F.L.+5mm and +7mm, the CTOD fracture surfaces indicated that failure occurred by the mixed mode of microvoid coalescence and ductile tearing. As the evaluated region approached the fusion line from base metal, the fraction of secondary cracking occurred normal to the fracture surface increased. In F.L., surface showed mixed mode of transgranular quasi-cleavage and microvoid coalescence. Dimpled areas indicating a ductile mode of failure were localized. Figure 9 shows macroscopic observation of CTOD tested surface. As shown in Figure 9, with the increase of distance from fusion line, shear lip of surface indicating ductile failure increased.

Microstructure analysis

Figure 10 shows optical micrographs for the example of the microstructural change in HAZ for QLT-processed 9% Ni steel. The microstructure of base metal consists mostly of tempered martensite with almost 5-10 vol. % of the retained austenite. The extensive banding present in Figure 10, which was the result of segregation during original solidification. As expected, the fraction of coarse-grained region within HAZ decreased away from fusion line. The schematic diagram of these results is shown in Figure 11. The fraction of coarse-grained region within HAZ means L_{CGHAZ}/L_{HAZ} in Figure 11.

Results of the X-ray diffraction measurements to evaluate the amount of retained austenite are given in Figure 12. The averaged amount of retained austenite also increases with increasing distance from fusion line. These results are well consistent with toughness

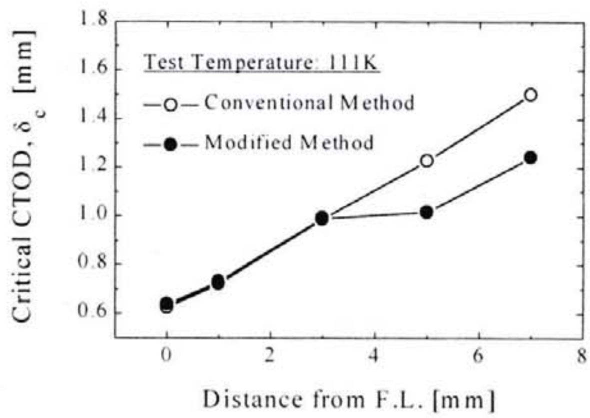

Figure 7. Comparison of the modified method with the conventional method .

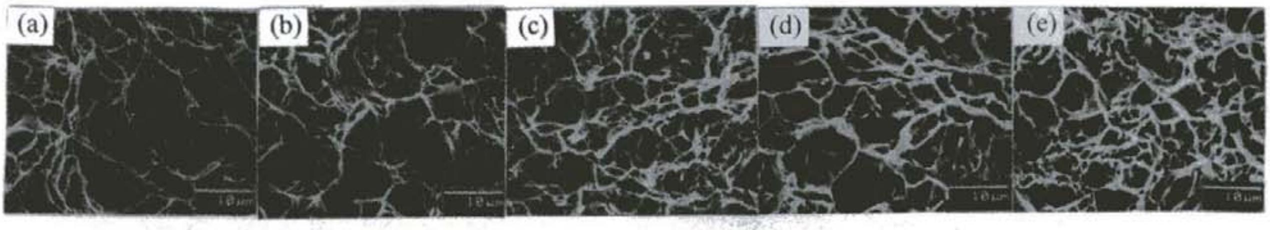

Figure 8. SEM fractographs of CTOD specimens at 110K: (a) F.L., (b) F.L.+1mm, (c) F.L.+3mm, (d) F.L.+5mm, and (e) F.L.+7mm .

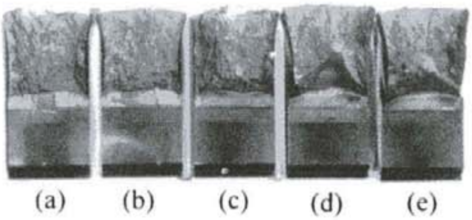

Figure 9. Macro-views of fractured specimens (a) F.L., (b) F.L.+1mm, (c) F.L.+3mm, (d) F.L.+5mm, and (e) F.L.+7mm .

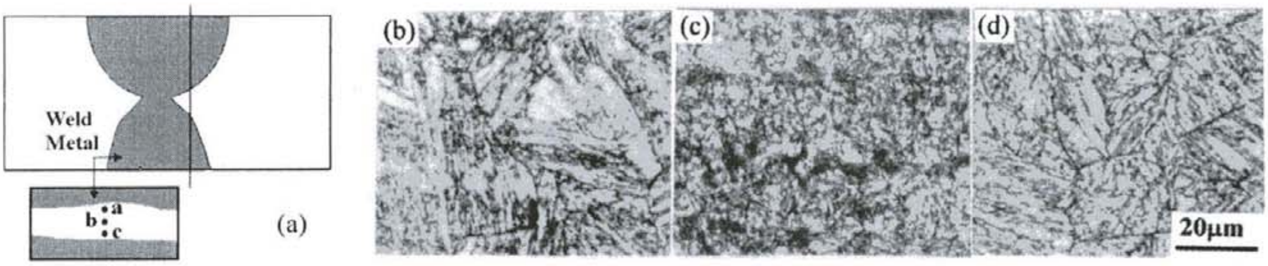

Figure 10. Examples of optical micrographs for the change of microstructure in HAZ (F.L.+1mm):(a) location of detection point, (b) a point, (c) b point, and (d) c point .

variations in HAZ as shown in Figure 6. These were results from the relation between the formation of retained austenite and the fraction of coarse-grained region. In coarse-grained region, the site for $\alpha \rightarrow \gamma$ reverse transformation by the subsequent thermal cycle during welding, decreased, and then, the formation of austenite decreased, as well, compared to the fine-grained region. Thus, near the fusion line with larger fraction of coarse-grained region, the amount of retained austenite was very small and fracture toughness was lower than that of other regions. Consequently, in QLT-9% Ni steel weldment with X-groove, the primary factor

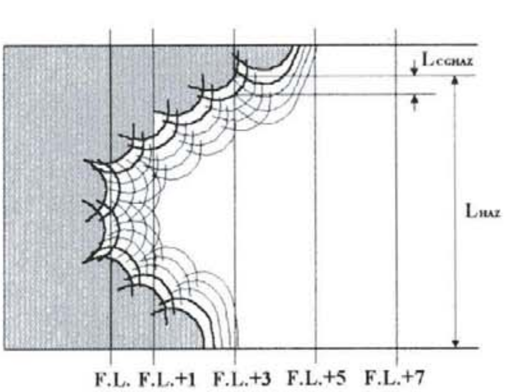

Figure 11. Schematic diagram for the change of coarse-grained region fraction .

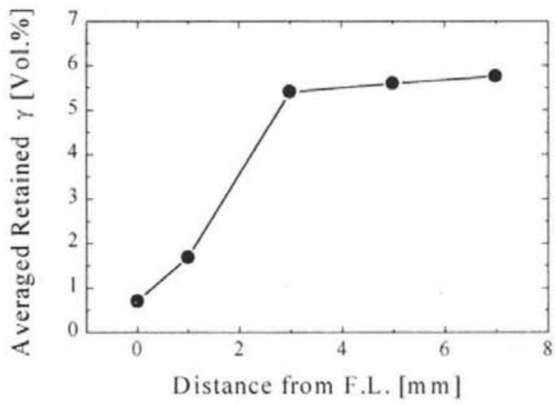

Figure 12. Change of averaged retained γ at the various locations (at room temp.) .

affecting fracture toughness is the fraction of the weakest coarse-grained region within HAZ, i.e., L_{CGHAZ}/L_{HAZ}, not the fraction of weld metal.

On the other hand, it is thought that in F.L.~F.L.+3mm, i.e., the mixed zone of weld metal/base metal, the distribution of alloying element would be also changed due to diffusion during the more complex thermal cycle than in F.L.+5mm and F.L.+7mm. So, the stability of retained austenite in F.L.~F.L.+3mm decreased at cryogenic temperature and retained austenite was transformed to martensite. Based on this reason, the toughness drop with decreasing test temperature from room temperature to 111K in F.L.~F.L.+3mm was larger than that in F.L.+5mm and F.L.+7mm. Now, the thermal stability of retained austenite and the thermal cycles in weldment with X-groove is going to be studied using AES, EDS and thermal cycle simulator.

CONCLUSIONS

Based on the concept of fitness-for-purpose, the cryogenic toughness of X-grooved weld HAZ for QLT- 9% Ni steel was evaluated. The primary studies of this investigation were:

1. Modified CTOD test method was proposed for the evaluation of HAZ toughness in X-grooved weldment, and then using this method the fracture toughness of actual weld HAZ was successfully evaluated.
2. As the evaluated region approached the fusion line from base metal the HAZ toughness of QLT-9% Ni steel decreased, which is confirmed by the decreasing area of ductile failure mode on the fracture surface.
3. The decrease in toughness of weld HAZ seems to be partly caused by reduction of the retained austenite content, resulting from the decrease in nucleation site for $\alpha \rightarrow \gamma$ reverse transformation due to the increasing fraction of coarse-grained region within HAZ. Therefore, in this steel weldment with X-groove, the primary factor affecting fracture toughness is the fraction of coarse-grained region within HAZ.
4. Due to the poor stability of retained austenite in the mixed zone of weld metal/base metal, toughness drop in F.L ~ F.L.+3mm was larger than that in F.L.+5mm and F.L.+7mm with decreasing test temperature.

ACKNOWLEDGMENT

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REFERENCES

1. H.T. Tamura, G. Onzawa, and S. Uematsu, *J. Japanese Welding Soc.*, 49:854 (1980)
2. E.F. Nippes and J.P. Balaguer, *Welding Journal*, 65:237-s (1986)
3. Consortium of five Japanese company, *GRI report*, GRI_86-0007 (1986)
4. British Standard BS 5762 (1979)
5. British Standard BS 7448 (1991)
6. ASTM Standard E1290 (1989)
7. S.J. Squrrel, H.G. Pisarski, and M.G. Dawes, BSIISM/4/4, *Working Party Report* (1986)
8. ASTM E24 Committee, Draft ASTM Test Standard for Fracture Toughness Testing of Weldments, ASTM E24. 06.05. (1991)
9. M. Toyoda, IIW Doc., X-1217-91 (1991)
10. H.G. Pisarski and M.G. Dawes, Measurement of COD in Weldment with Particular Reference to Offshore Structures, 111 (1980)
11. ASTM standard E1304 (1989)
12. K. Kajimoto, M. Tani, and N. Ikutoh, *Quartely J. Japanese Welding Soc.*, 4:182 (1983)
13. K. Arimochi, M. Nakanishi, S. Satoh, F. Minami, M. Toyoda and K. Satoh, *J. Japanese Welding Soc.*, 52:148 (1983)
14. F. Minami, M. Toyoda, C. Thaulow and M. hauge, *Quarterly J. of Japanese Welding Soc.*, 13:508 (1995)
15. API RP 2Z, "Recommended Practice for Preproduction Qualification for Steel Plates for Offshore Structures" (1987)