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Cryogenic Fracture Toughness Evaluation for Austenitic Stainless Steels by Means of Unloading Compliance Method

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Most research to date concerning the cryogenic toughness of austenitic stainless steels has concentrated on the base metal and weld metal in weldments. The most severe problem faced on the conventional austenitic stainless steel is the thermal aging degradation such as sensitization and carbide induced embrittlement. In this paper, we investigate the cryogenic toughness degradation which can be occurred for austenitic stainless in welding. The test materials are austenitic stainless JN1, JJ1 and JK2 steels, which are materials recently developed for use in nuclear fusion apparatus at cryogenic temperature. The small punch (SP) test was conducted to detect similar isothermally aging condition with material degradation occurred in service welding. The single-specimen unloading compliance method was used to determine toughness degradation caused by thermal aging for austenitic stainless steels. In addition, we have investigated size effect on fracture toughness by using 20% side-grooved 0.5TCT specimens.

Key Words : Cryogenic Fracture Toughness, Austenitic Stainless Steels, Thermal Aging Degradation, Small Punch Test, Nuclear Fusion Apparatus, Unloading Compliance Method, Size-Effect

1. Introduction

The advance of the thermonuclear fusion reactor ,demands superior mechanical properties comparing to the conventional austenitic stainless steels, especially in strength and toughness. (Mchenry and Reed, 1980)

Cryogenic fracture toughness is one of the most important properties for thermonuclear fusion reactor components, such as large superconducting magnet systems. One of the major applications of nitrogen-bearing stainless steels is for cryogenic service as a solid-solution strengthener. (Reed and Simmons, 1984)

A severe problem faced on conventional austenitic stainless steels is a thermal aging degradation such as a sensitization and a embrittlement (Simmons et al., 1994). It has been reported that the fracture toughness decreases because of the intergranular precipitation of carbides caused by long-term application at 550~650℃. The fabrication of the load-bearing members of large superconducting magnet support structures requires the welding of thick sections. Therefore, an evaluation of the mechanical properties for cryogenic structural materials must be conducted to establish the integrity and the confidence in a fusion reactor, especially the toughness degradation by sensitization. Cryogenic mechanical properties have been investegated for austenitic stainless steels, but these studies have focus on base metal and weld metal in weldments. (Ogawa, 1990 ; Shindo et al. 1995) There have been few reports on the effect of sensitization on the toughness degradation.

It has been proposed two testing method as

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toughness evaluation method for ductile high-toughness materials. One is a unloading compliance method recommended by ASTM E813-89. It has been reported that this test method is an useful technique to determine the fracture toughness at cryogenic temperature (Shindo et al., 1995; Kwon, 1998).

The other is a small punch(SP) testing method. The SP testing method to provide information about the mechanical properties of materials is very useful, simple and convenient. So, this method has been widely used to evaluate the fracture toughness of materials, especially metal as well as composite materials. (Lyu et al, 1990 ; Okuda et al, 1991)

The objectives in this research is to investigate the toughness degradation caused by sensitization for a new type of stainless steels which can be used as a cryogenic magnet support structures. The SP testing technique is applied to base metal, sensitized materials and weldments, which is high-nitrogen stainless steel JN1, to detect similar isothermally aging condition with service weldments. Finally, unloading compliance method is applied to evaluate toughness degradation for three types of austenitic stainless steels. Also, we intend to investigate size-effect on fracture toughness.

2. Experimental Procedure

2.1 Materials

The materials investigated in the present study are nitrogen-strengthened austenitic stainless steels anticipating as structural material in nuclear fusion apparatus at cryogenic temperatures. These materials are classified into high Cr-Ni JN1, JJ1 and high Mn JK2 steels. Solution-treatment temperatures are 1348K, 1323 to 1346K and 1253K, for JN1, JJ1 and JK2, respectively. Chemical compositions of the steels is listed in Table 1. Also, for investigating the toughness degradation by sensitization, forged plates were isothermally aged at 650°C for 5hrs.

Weldments on the 200mm-thick plate machined from the 250mm-thick JN1 plate were made by gas tungsten arc welding(GTAW) process. GTA welding was performed with a welding voltage of 10 to 11.5V and a welding current of 250 to 270A using Inconel 625 type filler metals. A 81.5 degree included angle double-V joint assembly was used with a root opening of 12mm. The chemical compositions in weldments is given in Table 2.

2.2 Specimens

SP specimens in the form of 10mm×10mm×0.5mmt plates, were supplied from the forged plates. Plate 1.2mm thick were produced by slicing and polished up to 0.5±0.05mm in thickness using emery paper. To investigate the decrease of mechanical property caused by thermal cycle in weldments, each specimen, whose middle point was located at 1.5mm, 3mm, 5mm from fusion line(F.L) in weldments, was precisely sliced. Each specimen is defined as HAZ1.5, HAZ3 and HAZ5, respectively. Figure 1 shows the location and designation of specimen machined from GTA

Table 1 Chemical compositions of JN1, JJ1 and JK2 forged plates (wt%)

Material	C	Si	Mn	P	S	Ni	Cr	Al	N	Mo
JN1 Forged plate	0.040	0.97	3.88	0.022	0.001	15.07	24.32	0.023	0.32	—
JJ1 Forged plate	0.025	0.48	10.13	0.021	0.0017	11.79	12.01	—	0.236	4.94
JK2 Forged plate	0.050	0.39	21.27	0.005	0.001	9.15	12.97	—	0.247	0.97

Table 2 Chemical compositions of weld metal for JN1 GTA welded joint (wt%)

Material	C	Si	Mn	P	S	Ni	Cr	N	Mo	Nb
Weld metal I-625^{R}	0.035	0.14	0.05	0.008	<0.001	21.6	24.32	0.029	9.0	3.55
Weld metal I-625^{R}M	0.008	0.10	0.15	0.001	0.0004	21.8	24.32	0.15	8.50	—

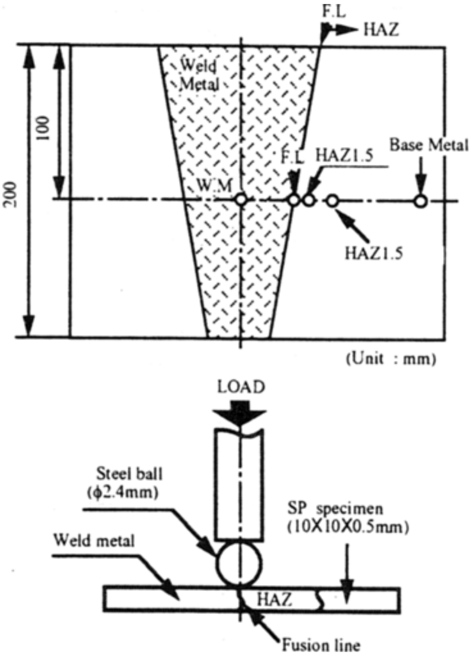
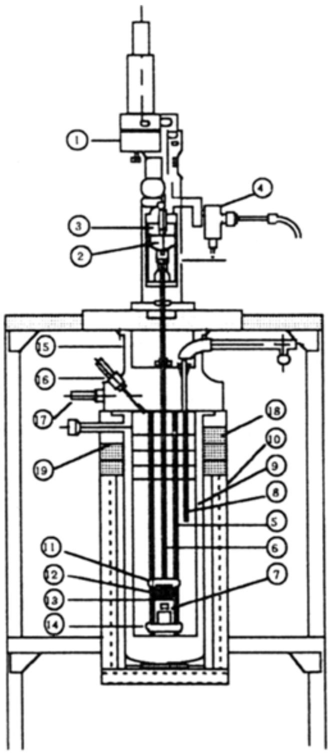


Fig. 1 Schematic illustration showing the extracted position in weldments and loading method for SP specime



(1) Loading unit, (2) Load cell, (3) Load cell adapter, (4) LVDT, (5) Load column, (6), (12) Pull load, (7) Puncher and dies for SP test, (8) Liquid helium transfer tube, (9) Liquid helium dewer, (10) Liquid nitrogen dewer, (11) Load plate, (13) Upper crosshead, (14) Lower crosshead, (15) Base frame, (16) Electrical fee-through, (17) Vacuum valve, (18) Styrofoam (exploded polystyrene plastic), (19) Radiation shield plates

Fig. 2 Cryostat for SP test apparatus

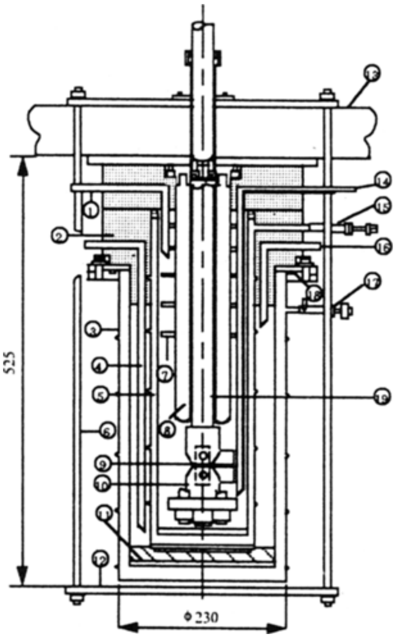
weldment. For the case of F.L specimen. SP test was conducted by locating F.L to middle of steel ball, as shown in Fig. 1.

The resulting specimens used in fracture toughness testing were finally machined into 0.5TCT geometry having the dimensions recommended by ASTM E813-87. All specimens are machined in the T-L orientation. These specimens were precracked with final ΔK of 20MPa • m$^{1/2}$ at room temperature. The ratio of initial crack length to specimen width, a/W, was 0.6 to 0.68. After precracking, all specimens were side grooved to 20% of gross thickness to investigate size-effect on fracture toughness. The fatigue precracking introduction was conducted prior to thermal aging treatment.

2.3 Cryogenic testing method

The graphic drawings of devices which is designed for SP and fracture toughness test at cryogenic temperature are showed in Fig. 2 and Fig. 3. The external (ϕ230mm), internal dewar (ϕ 160mm) of cryostat and liquid helium transfer tube are composed of double-walled vacuum-insulated structure. It makes holding 5×10^{-5} Torr of vacuum state to prevent boiling phenomenon of the liquid helium. In injection of the liquid helium during the experiment, 30mm thickness of thermal shield plate between plates was attached for preventing the heat input from the crosshead and for restraint of raising the evaporation helium. The copper plate with good thermal conductivity is used as materials of the thermal shield plate. Among the figures, the styrofoam with good thermal shield is used in the shadow part and isolates thermal with the outside as soon as possible.

As for the experiment at the temperature of the liquid helium, the specimen was fixed to Jig



(1) Gas helium vent, (2), (11) Styrofoam, (3) Outer dewer, (4) Liquid nitrogen fill, (5) Inner dewer, (6) Rod, (7) Thermal shield plate, (8) Load frame, (9) Clip gage, (10) CT specimen, (12) Base plate, (13) Cross head, (14) Liquid helium transfer tube, (15), (17) Vacuum valve, (16) GAS nitrogen vent, (18) O-ring, (19) Pull rod

Fig. 3 Cryostat for fracture toughness test

which is located on the internal dewar of the cryostat, and the liquid nitrogen is injected and pre-cooled about 30min. After the pre-cooling, liquid nitrogen of the internal dewar transfers to the external dewar. By flowing liquid helium continually in the internal dewar, the specimen cools to within 2K of the intended start temperature within approximately 2min. The liquid nitrogen is injected continuously in the external dewar for restraining the boiling of the liquid helium. The specimen temperature during the cooling was monitored in calibrations using carbon resistance thermocouple. All tests were carried out by using specimen completely immersed in the liquid helium.

All the SP tests were performed on a testing machine in the cryostat. It consists of the SP testing apparatus connected with Ti-6Al-4V alloy pull rods, a liquid nitrogen dewar, a liquid helium dewar with 120mm inside diameter, a liquid helium transfer tube and a loading unit with two-ton capacity, as shown in Fig. 2. The SP tests were started by pressing the steel ball against the specimen after the specimen position of the SP test system had been kept at a temperaturee of 4.2K for 30min. The tensile loading was converted into compressive force through the puncher at crosshesd speed of 0.2mm/min. The load versus LVDT deflection curve is drawn using an computer to display SP fracture behavior.

Fracture toughness tests are conducted on a screw-driven electromechanical test machine at a constant cross head speed of 0.1mm/min (stroke control). These tests are performed with the specimen and Shepic clip gage completly submerged in liquid helium. The detailed test procedures for toughness testing method have been described in elsewhere. (Kwon, 1998)

2.4 Fracture toughness

The J-integral is calculated on the basis of the unloading compliance method recommended by ASTM E813-89. The blunting line is given as

$$J = 2\sigma_y \triangle a \qquad (1)$$

$$\sigma_y = (\sigma_{y.s} + \sigma_{t.s})/2 \text{ : flow stress}$$

Where $\sigma_{y.s}$ is a 0.2% yield stress and $\sigma_{t.s}$ is a maximum tensional strength. The R-curve is obtained from the logarithm curve after using regression line points located between 0.15~1.5mm on the graph. And, R-curve obtained from regression of points between 0.15~1.5mm by logarithm curve on the graphs. Also, specimen thickness, B or ligament length, b is given as

$$B_N,\ b > 25(J_Q/\sigma_y) \qquad (2)$$

where J_Q is J-integral value obtained from the intersection of the blunting line with 0.2mm offset line. b is W-a_0. If it is not satisfied Eq. (2), then J_Q is not J_{IC}. And, the following equation for points on the graph is given

$$B_N,\ b > 15(J/\sigma_y) \qquad (3)$$

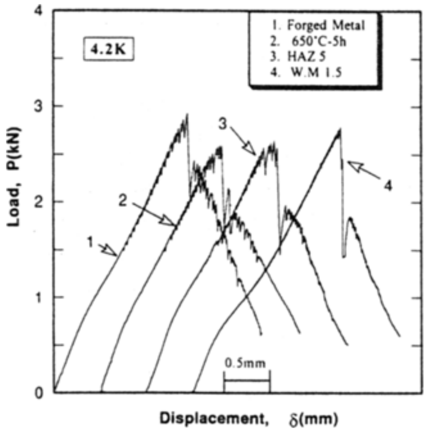


Fig. 4 Load-displacement curves obtained from specimens for the forged, thermal aged steels and weldments tested at 4. 2K

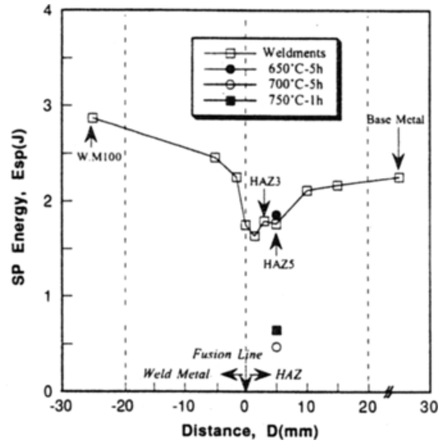


Fig. 5 SP energy obtained from specimens of the HAZs and weld metal near the fusion line in GTA weldments of JN1 steel

3. Results and Discussion

3.1 SP test

The fracture behavior in the SP testing for the austenitic alloys having different low-temperature fracture toughness is well defined in the applied central load(P) versus deflectrion(δ) curve, as well as base metal and weldments. Figure 4 presents a typical example of the P-δ curves obtained from SP tests at 4.2K. It is found that the P-δ curves are approximately linear at first, and that its curves show serrations behavior before maximun load(P_{max}). It has been reported that serrations occurred due to the istability of plastic deformation. (Madhava and Armstrong. 1973) It implies that all materials maintains the excellent mechanical properties at cryogenic temperature.

Table 3 Mechanical properties of the steels obtained from the tensional tests

Material	4K			
	σ_{ys} (MPa)	σ_{UTS} (MPa)	Elongation (%)	Reduction of area(%)
JN1 Steel	1349	1690	31	42
650℃-5h	1318	1671	13	—
JJ1 Steel	1169	1591	39	44
650℃-5h	1162	1517	17	—
JK2 Steel	1216	1576	44	50
650℃-5h	1188	1534	21	—

The mechanical properties of each material were investigated by SP energy defined as area under the P-δ curve. Because SP energy is very useful parameter to evaluate the fracture characteristics. (Lyu et al, 1990) The SP energy acquired on specimens extracted in weldments tested at 4.2K is shown in Fig. 5. The solid and open circles show the SP energy for therml aged specimens. The SP energy behaviors for the F.L, HAZ1.5 and HAZ5 specimens extracted from the weldments showed fairly low value compared with the weld metal and the forged metal. It is said that the HAZs near to the fusion line has material deterioration by the welding thermal cycle. Notice that the SP energy for HAZs in weldments was comparable to or slightly lower than that of thermal aging treated specimen for 5hrs at 650℃, as shown in Fig. 5. It is obviously indicated that the HAZs has material degradation equivalent to the condition of sensitization for 5hrs at 650℃. Based on the results above, we have choosen the thermal aging condition at 650℃-5h to investigate the toughness degradation by unloading compliance method for JN1, JJ1 and JK2 steels.

3.2 Cryogenic fracture toughness

3.2.1 Tensile properties

The flow stresses are necessary to determine

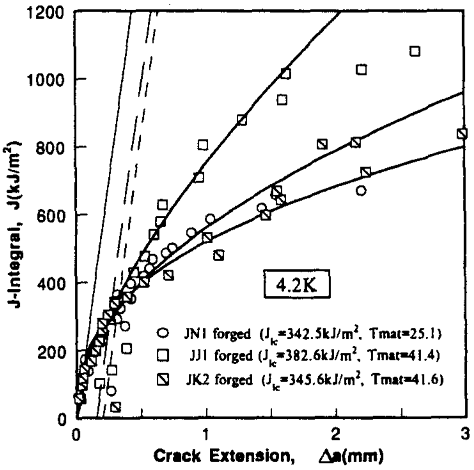


Fig. 6 J-R curves obtained from 0. 5TCT specimen of JN1, JJ1 and JK2 forged metals tested at 4.2K.

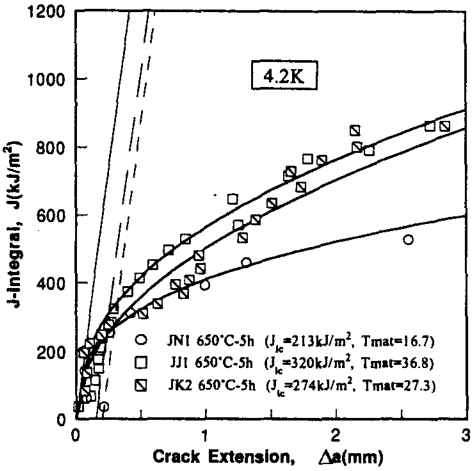


Fig. 7 J-R curves acquired on 0. 5TCT specimen of JN1, JJ1 and JK2 thermal aged metals tested at 4.2K

availability of the J_{IC}. Tensional tests were conducted at 4.2K using 28mm gage length and 5mm diameter specimens for the forged and the thermal aged JN1, JJ1 and JK2 steels. Material orientation for tension specimen loading axis was the same as that of the fracture toughness specimen. The test procedures and the cryostat for the low temperature tests were also simialr to that of the fracture toughness test.

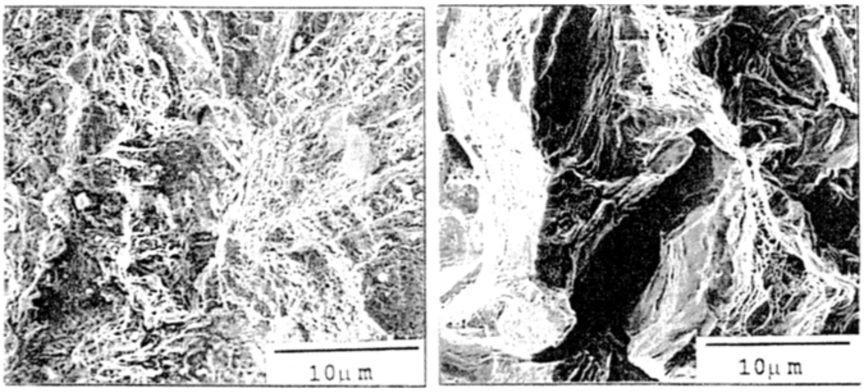
The yield strength σ_Y, the ultimate tensile strength σ_B, ultimate flow strength $\sigma_f = (\sigma_Y + \sigma_B)/2$, and Young's modulus E are listed in Table 3. The yield strength differences are due to the effect of nitrogen content on cryogenic strength. The yield strength dependence on nitrogen content is well-known. (Ogawa and Koseki, 1988 ; Reed and Simmon, 1984)

3.2.2 J-R curves

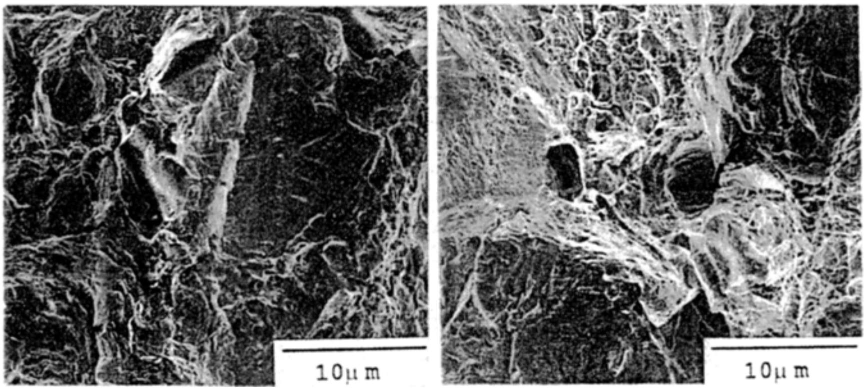
The J-R curves for the forged steels tested at 4.2K are shown in Fig. 6. The J-R curve from each specimens has effective points which are satisfied with the condition of Eq. (4) between 0.15～1.5mm off-set lines of blunting curve. The value from the intersection points between the blunting curve and the 0.2mm off-set lines are 342.5kJ/m^2 for JN1, 382.6kJ/m^2 for JJ1 and 345.6kJ/m^2 for JK2, respectively. These values are satisfied with the condition of the Eq. (3), so it is valid J_{IC} value. The J_{IC} values acquired on the JN1 and JJ1 steels are almost equal to 341.8kJ/m^2 and 366. 1kJ/m^2, the values proposed by Horiguchi et al. for 1TCT specimens (Horiguchi et al., 1995 ; Shindo et al., 1995). Also, these values are almost equal to those obtained by multi-specimen method for JN1 and JJ1 steels. (Nakajima et al., 1988, Yoshida et al, 1988). It can be concluded that the fracture toughness values are independent on size-effect for 20% side-grooved 0.5TCT specimen that the unloading compliance method using single specimen is very useful technique to evaluate fracture toughness at cryogenic temperature.

Figure 7 shows the J-R curves for the isothermally aged specimens at 650℃ for 5hr. The points on the curve is satisfied with ASTM standard, so it is effective J_{IC}. The J_{IC} values obtained from the intersecting point between blunting line and 0. 2mm off set line are 213kJ/m^2 for JN1, 320kJ/m^2 for JJ1, 274kJ/m^2 for JK2, respectively. The fracture toughness was degraded with thermal aging treatment.

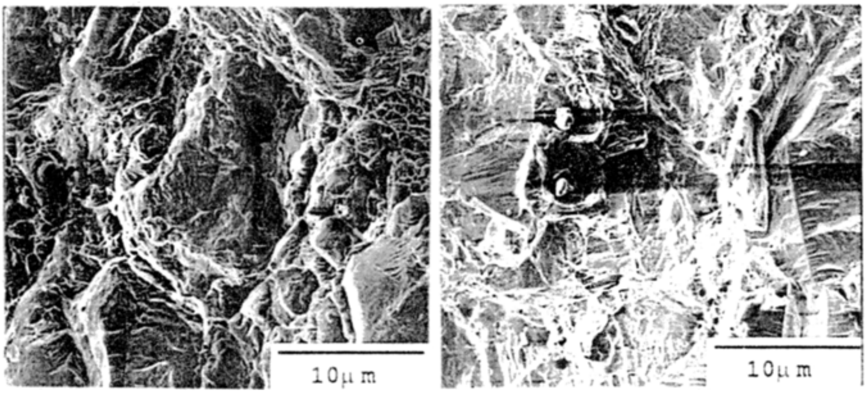
Comparing the fracture toughness values acquired on each materials, J_{IC} values of the forged and isothermally aged specimens for JJ1 steel exhibit higher than those obtained from other steels. The degradation of the fracture toughness caused by thermal aging treatment are 62kJ/m^2 for JJ1 steel, while the value are 129kJ/



(a) JN1 B.M (b) JN1 650°C-5h



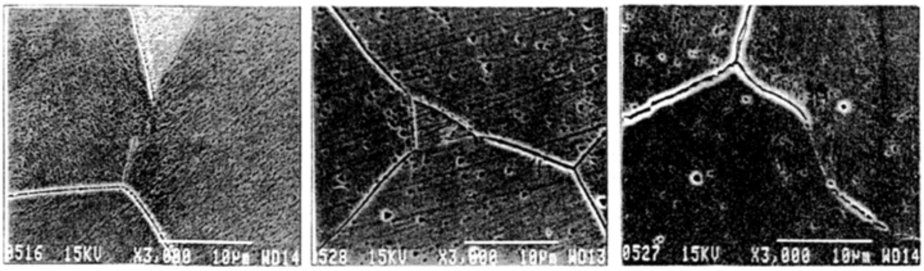
(c) JJ1 B.M (d) JJ1 650°C-5h.



(e) JK2 B.M (f) JK2 650°C-5h

Fig. 8 SEM photographs showing fracture surface transition between each of materials according to heat treatment obtained from 0.5TCT specimens fractured at 4.2K

m^2 for JN1 and 71kJ/m^2 for JK2 steel. From the above results, we know that JN1 steel reveals the most severe degradation of the fracture toughness value than other steels by sensitization. Meanwhile forged and isothermally aged specimen for JJ1 steel exhibits higher toughness than other steels and lower toughness degradation with thermal aging treatment. It implies that JJ1 steel can



(a) JN1 650°C-5h (b) JJ1 650°C-5h (c) JK1 650°C-5h

Fig. 9 SEM photographs observed from the heat treated materials of JN1, JJ1 and JK2 steel after etched using vilella solution

be maintained superior cryogenic fracture toughness compared to other steels when the same welding conditions are applied to JN1, JJ1 and JK2 steels.

3.2.3 Observation of the fracture surface

Fig. 8 shows the fracture surface of forged and isothermally aged steels at 650℃ for 5hrs in the 4.2K fracture toughness test. All fracture surfaces for forged steels show ductile dimples. It is indicated that forged steels are maintained excellent fracture toughness at cryogenic temperature. For the isothermally aged specimens, JJ1 and JK2 steels show the appearance of dimple fracture over nearly the entire surface, meanwhile JN1 steel which has the most degraded fracture toughness value shows slight local intergranular in addition to dimples. We can expect that JN1 steel is the most sensitized than other steels and that the fracture appearance after crack initiation is related to the fracture toughness value. It is suggested that the further study is necessary to analysis the correlation between ductile fracture resistance modulus, T_{mat} and fracture toughness value, J_{IC}.

3.3 Microstructure observation

To find out the cause of the toughness degradation occurred in thermal aged specimens, we have investigate intergranular precipitates by using vilella solution. Scanning Electron Microscope (SEM) micrographs are shown in Fig. 9. From this figure, microstructure of all samples shows an austenitic matrix with spheroidal precipitates formed mainly on austenitic grain boundaries. It has been reported that precipitates of this type are observed in either JN1 and JJ1 steel (Saucedo et al., 1996 ; Kwon and Chung, 1998). They suggested that spheroidal precipitates were $M_{23}C_6$ and M_2N by using x-ray diffraction analysis. It is important to mention that no precipitation are found in solution treated samples by optical and SEM observation. We can expect that these precipitates deteriorate the fracture toughness by decreasing the cohesive strength in grain boundaries. More extensive study, however, will be required in order to understand the toughness degradation caused by sensitization for newly developed austenitic stainless steels.

4. Conclusions

This paper aims to evaluate toughness degradation caused by sensitization for a new type of stainless steels which can be used as a cryogenic magnet support structures. In addition, we have investigated side-groove dependence on fracture toughness. In order to these purpose, we are conducted SP testing method using miniaturized specimen and unloading compliance method. The results obtained in the present study are summarized as follows.

(1) It was proved that the fracture characteristics for the HAZs in GTA weldments are similar to those of thermal aged material for 5hrs. at 650℃ by SP testing method.

(2) The fracture toughness are independent on size-effect for 20% side-grooved 0.5TCT specimen and the unloading compliance method using single specimen is very useful technique to evalu-

ate fracture toughness at cryogenic temperature.

(3) JN1 steel reveals the most severe decrease of the fracture toughness than other steels with sensitization.

(4) JJ1 steel exhibits superior cryogenic fracture toughness compared to other steels for forged and thermal aged specimen.

(5) It is proposed that the precipitates such as $M_{23}C_6$ and M_2N deteriorate the fracture toughness by reducing cohesive strength in grain boundaries for thermally aged materials.

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