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# Weldability of Fe-36% Ni Alloy (Report I)<sup>†</sup> - Hot Cracking with Cross-Bead Test -

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#### Abstract

Behavior of weld hot crack in Fe-36%Ni alloy which is used as elements of membrane-type container for liquefied gas has been studied. Cracking test used has been cross-bead type in order to study the cracks occurring in previous welding pass reheated by the following welding pass or repair welding pass. Main conclusions obtained are as follows;

- 1) Weld metal reheated to the range of about 800 to 1300°C is susceptible to crack.
- 2) The crack occurs at migrated grain boundary after solidification.
- 3) The fractograph gives a brittle intergranular fracture.
- 4) These behaviors mean that the crack is not a liquation crack, but a ductility-dip crack.

KEY WORDS: (Hot Cracking) (Controlled Expansion Alloys) (Containers) (Fractography) (GTA Welding)

#### 1. Introduction

In recent years, increase in energy demand promotes the requirement of container for storage and transport of different liquefied gases such as LNG. Since the container must be made of materials with high toughness at extremely low temperature, austenitic stainless steel, 9% nickel steel, aluminum alloy are used. On the other hand, membrane-type container, standing on a different design art, is also a suitable one, and Fe-36%Ni alloy (Invar) is used as the elements, because its thermal expansion is very little.

Metallographically, Fe-36%Ni alloy has fully austenitic microstructure, and thus hot cracking is considered to be a major problem in welding. Therefore, its weld solidification crack has been exclusively studied so far<sup>1)-3</sup>. It is said, however, that following welding pass or repair welding often causes cracking in its surrounding previous welding pass. This means that the cracking is not solidification cracking, but liquation or ductility-dip cracking. The detail, however, has not been studied so far.

This paper describes the behavior of the cracking utilizing a cracking test of cross-bead type, and with metallographic and fractographic viewpoints.

## 2. Experimental Procedures

#### 2.1 Materials used

Thin sheets (1.5mm thickness) of two Fe-36%Ni alloys were used and their chemical compositions are shown in **Table 1**.

Table 1 Chemical compositions of materials used

Material	Composition (wt.%)										
	С	Si	Mn	P	S	Ni	Cr	0	N	Ca	
1	0.036	0.22	0.31	0.012	0.001	35.99	0.186	0.004	0.005	0.001	
2	0.028	0.20	0.37	0.002	0.005	36.29	0.006	0.005	0.005	0.001	

#### 2.2 Hot cracking test

Hot cracking test of cross-bead type, the detail of which is later described in Fig. 2, was used to study the behavior of cracking in a previous weld metal reheated by the following welding or repair welding pass. In this paper "previous weld metal" is hereafter named "first (welding) pass", and "following welding pass" or "repair welding pass" is named "second (welding) pass".

In this test both the first and second welding passes were bead-on-plate TIG welding without filler metal. The first welding pass was made on the specimen shown in Fig. 1 mainly in welding chamber filled with 1 atm argon

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gas and partly in the air without any backing material. Welding conditions were: arc length of 1.6mm (thus arc voltage of 7V), welding current of 40A, welding speed of 100mm/min, and bead width of about 4mm in both sides. The welded specimen was then cut with a shearing machine along the broken line in Fig. 1.

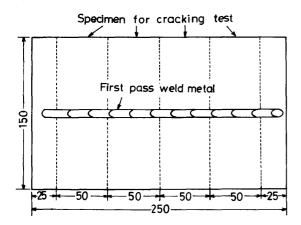
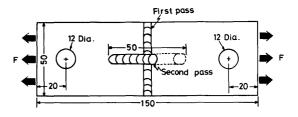


Fig. 1 Specimen configuration for first welding pass

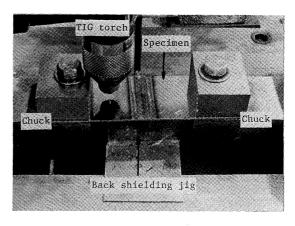
The principle of the hot cracking test is as follows (see Fig. 2): the second welding pass was made on the above cut specimen perpendicularly to the first pass under constant load (F) paralleling the second pass. The load was applied with a 10-ton TRC tensile testing machine placed horizontally, and was taken off within several seconds after the welding. The weld length was 50mm. The specimen was fixed with screw bolts to chucks of the machine as shown in Fig. 2 (b). General view of the specimen after the testing is shown in Fig. 2 (c). Figure 3 shows an example of loading curve during the testing. Selected loads were those that gave applied stresses of 1.3, 2.5, 5, 10 and 20kg/mm<sup>2</sup>, where the applied stress was obtained by dividing the load by the crosssectional area of specimen. In order to maintain constant load during testing, the chuck was moved to tensile direction. The average moving speed (i.e. cross-head speed) was approximately 1 x 10<sup>-3</sup> mm/sec per applied stress of  $1 \text{ kg/mm}^2$ .

Welding conditions of the second pass were: arc length of 1.5mm, welding current of 60A, welding speed of 100 mm/min, and back shielding argon gas of 20 /min from back shielding jig seen in Fig. 2 (b).

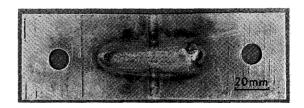
The specimen after the testing was metallographically polished and etched with 20% Nital, and cracked region and total crack length were measured with an optical microscope.



(a) principle of testing



(b) Close-up view around the specimen



(c) appearance of specimen after testing

Fig. 2 Explanation of cross-bead hot cracking test

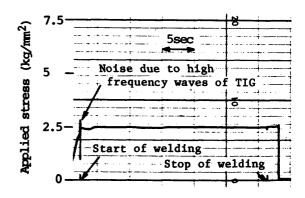


Fig. 3 Example of loading curve during testing

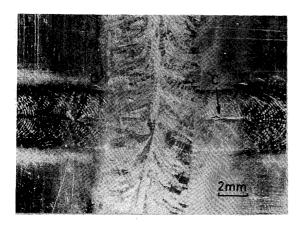
#### 2.3 Others

Cracked surface was observed in detail with a scanning electron microscope (SEM) and segregation of elements was measured with a wave-dispersive type electron probe micro analyzer (EPMA) attached to the SEM utilizing the specimen current of about  $10^{-8}$  A.

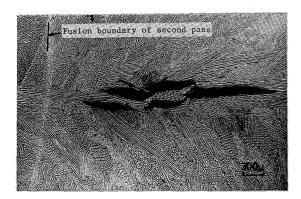
#### 3. Experimental Results and Discussion

#### 3.1 Cracking region, time and temperature

General example of weld bead after the testing is shown in Fig. 4, from which cracks are seen to occur at the weld center of the first pass at some distance from the fusion boundary of the second pass. At higher applied stress, for example  $20 \text{kg/mm}^2$ , the crack developed to liquation crack and further to solidification crack (see Fig. 5).



(a) as welded macrophotograph (C:crack)



(b) etched microphotograph

Fig. 4 General appearance of crack in low magnification

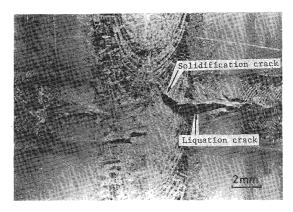


Fig. 5 As welded macrophotograph in a high applied stress (20kg/mm<sup>2</sup>)

Figure 6 shows the relation between applied stress and cracking region in the range of applied stress from 1.3 to 15kg/mm<sup>2</sup>. Solid mark gives the location of the nearest crack to the fusion boundary of second pass, and open mark does the location of the farthest crack. Therefore the crack occurred in the region between the solid and open marks. As regards abbreviations in Fig. 6, numerals 1 and 2 mean kinds of materials given in Table 1, letter A does that the first pass was done in the air, and CI does that the first pass was done in the chamber filled with inert gas (argon). In the data marked  $\triangle$  and  $\blacktriangle$ , the first pass was done with welding current of 70A. It is observed that the nearest crack situates at about 1 mm from the fusion boundary irrespective of material, applied stress, heat input, and welding atmosphere, thouth it somewhat approaches to the fusion boundary at 15 kg/mm<sup>2</sup>. The

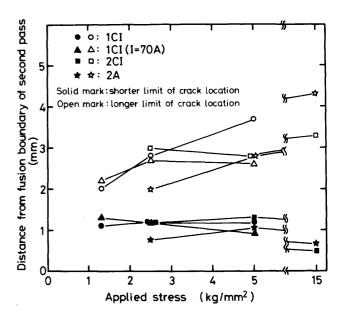


Fig. 6 Relation between applied stress and cracking location

farthest crack situates at about 2 to 3 mm from the fusion boundary, and a tendency is observed that the increase in applied stress enlarges the distance.

Distribution of peak temperature during the second welding pass measured with CA thermocouple (0.3mm dia.) and lacquer sensitive to temperature, "Tempilaq", is given in Fig. 7. Combination of Fig. 6 and 7 means that the cracking occurred in the region where the peak temperature was within about 800 to 1300°C.

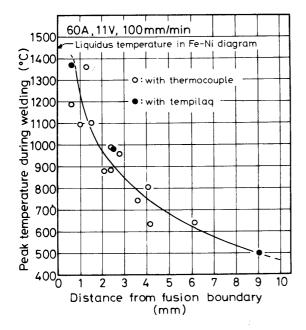


Fig. 7 Distribution of peak temperature in first pass during welding of second pass

Then, Fig. 8 shows an example of sequence of video pictures during the cracking test which was recorded using a TV camera with a telephoto lens and a video tape recorder. The cracking occurred (Fig. 8 (b)) at 6 sec after the time  $t_c$  at which the tungsten electrode passed the center of the first pass weld metal (Fig. 8 (a)). After that, the crack somewhat further developed (Fig. 8 (c) and (d)). Many repeat tests gave the result that the cracking occurred at  $5 \pm 2$  sec after  $t_c$ . Figure 9 gives examples of thermal cycle curves at 1 and 2.8mm from the fusion boundary, and means that the temperature at  $5 \pm 2$  sec after  $t_c$  was near or somewhat lower than the peak temperature.

Therefore, the cracking occurs in the first pass weld metal which is reheated to the range of 800 to 1300°C, about the time when the peak temperature is reached. The liquidus temperature of Fe-36%Ni alloy is about 1450°C in Fe-Ni phase diagram<sup>4</sup>), and the nominal solidus temperature is 1425°C<sup>5</sup>). Therefore it is considered that the crack is not a liquation crack but a ductility-dip crack.

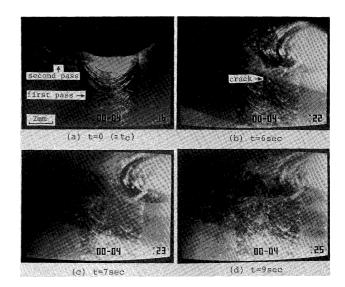


Fig. 8 Example of sequence of video pictures during cracking test (t<sub>C</sub> is the time when tungsten electrode passed the center of first pass.)

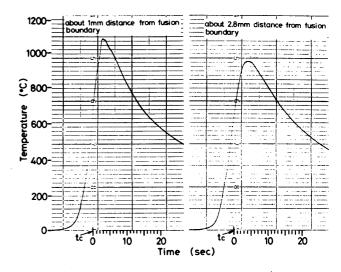
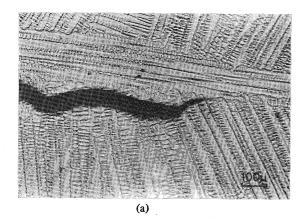
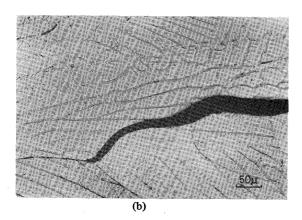


Fig. 9 Thermal cycle curve in first pass during welding of second pass

#### 3.2 Metallography and fractography of crack

In Fig. 4 (b) which is a microphotograph in a low magnification, the cracks seem to occur at columnar grain boundaries at the solidification. The cracks observed in a higher magnification shown in Fig. 10, however, occur at migrated grain boundaries after solidification, and thus have no relation to columnar grain boundaries at solidification. This also confirms that the crack is ductility-dip crack, because liquation crack must easily occur at grain boundaties at solidification due to their much segregation causing partial melting. The reasons why the cracks seem





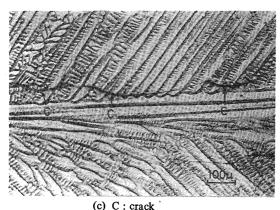


Fig.10 Relation between crack and microstructure

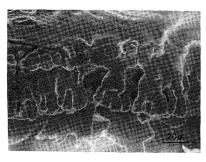
to occur at grain boundaries at solidification in low magnification are that the extent of grain boundary migration is not large, and that the migrated grain boundary is roughly parallel to the columnar grain boundary at solidification. It is considered that the migration occurred during the cooling after the solidification of the first pass and during the reheating by the second pass.

The authors have already revealed detailed features of fractographs of solidification cracks of several materials<sup>6</sup>)-8), and also have shown an example of fractograph of a liquation crack<sup>9</sup>). So, in the following, the fractograph of the crack discussed in this paper is compared with fractographs of solidification and liquation cracks.

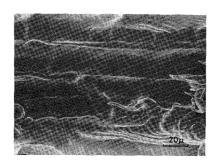
Both solidification and liquation cracks, as already mentioned in Fig. 5, occurred in a high applied stress. Fractographs of the solidification crack are shown in Fig. 11. Feature of cellular dendrites is seen clearly in Fig. 11 (a) and obscurely in Fig. 11 (b). Flatness are noticeable in Fig. 11 (c), although several protrusions which seem to be made by tearing of liquid droplets are seen. According to classification system<sup>6)-8)</sup> of fractographs of solidification crack, features of Fig. 11 (a), (b) and (c) correspond to Types D, D-F and F respectively.

A fractograph of the liquation crack is shown in Fig.12, which also gives a rounded feature affected by liquid phase on the cracked surface.

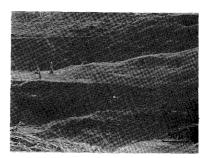
Now, fractographs of the crack discussed in this paper are shown in Fig. 13. Observation of Fig. 13(a) gives a feature of brittle intergranular fracture with sharp edges in contrast with Fig. 11 and Fig. 12. Moreover, observation in a higher magnification, Fig. 13 (b) and (c), ofen shows fine ruggedness and dimples or voids. These features resemble those of stress-relief cracking and temper embrittlement, and thus suggests that segregation of one or few elements to the migrating grain boundaries or the subsequent precipitation during thermal cycle of the first and second passes weakens the cohesive strength of grain boundary and causes the crack.



(a) Type D



(b) Type D-F



(c) Type F

Fig.11 Fractographs of solidification crack

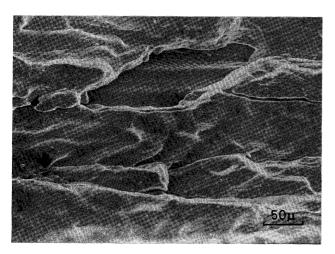
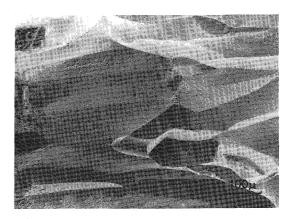


Fig.12 Fractograph of liquation crack

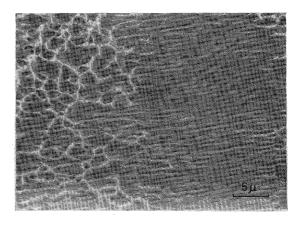
Then, Fig. 14 shows analysis results of O, N, Si, P, and S on the area of about  $60 \times 50\mu$  of the cracked surface utilizing EPMA, where the ordinate gives X-ray intensity, the abscissa does wave length of X-ray and the location of each characteristic X-ray (Ka) to be detected is also marked on the abscissa with an arrow. There is no obvious peak of characteristic X-ray in every figure. By the way, oxygen peak was easily detected from the cracked surface which was oxidized due to insufficient back shielding as seen in Fig. 15. Then, Fig. 16 shows line analysis results of O, S, Si, and Ni near the tip of the crack on a polished surface, from which no segregation at the crack tip is detected. These results mean that there is not so much segregation as to be detectable with EPMA. As already mentioned, the fractograph of the crack discussed in this paper resembles those of stress-relief cracking and temper embrittlement. Since grain boundary segregation causing stress-relief cracking and temper embrittlement is only detected with AES, AES analysis should be planned to this crack.

### 3.3 Discussion

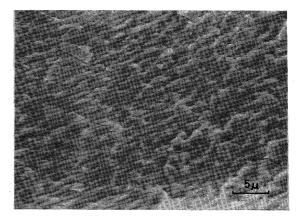
In comparison with the cracking in the first pass weld metal, cracking test was done without the first pass in order to obtain the crack sensitivity of heat-affected zone of base metal. The result showed that even applied stress of  $20 \text{kg/mm}^2$  cannot cause the cracking. Thus it is considered that absorbtion of minor impurity elements during melting and solidification, microsegregation due to solidification, coarsening and columnar characteristic of grains, grain boundary segregation of the impurity elements during the reheating by the second pass, and the consequent weakening of grain boundary strength are related to the cracking in the first pass weld metal. That the thermal cycle after solidification and during reheating is very rapid, and that grain boundary migration occurs



(a) low magnification



(b) high magnification



(c) high magnification

Fig.13 Fractographs of ductility-dip crack

during the thermal cycle may suggest that the harmful elements causing the cracking have high diffusivity such as interstitial elements.

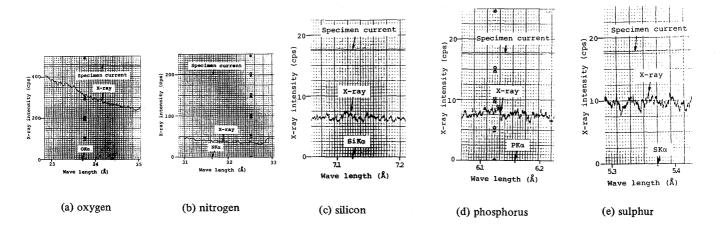


Fig.14 Analyzing results of impurities on the surface of ductility-dip crack

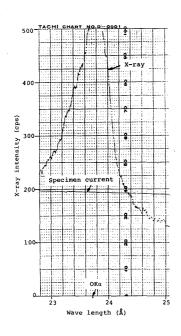
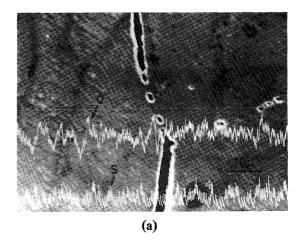


Fig.15 Oxygen peak on the surface of ductility-dip crack which was oxidized due to insufficient back shielding



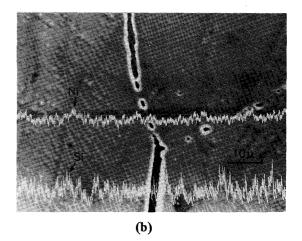


Fig.16 Line analysis of (a) oxygen and sulphur, (b) silicon and nickel across the tip of ductility-dip crack

#### 4. Conclusions

Behavior of the crack occurring in the first pass by the reheating of second pass was studied utilizing hot cracking test of cross-bead type. Main conclusions obtained are as follows;

- 1) The first pass weld metal reheated to the range of about 800 to 1300°C by the second pass is susceptible to crack. The crack occurs about the time when the peak temperature of the thermal cycle is reached.
- 2) The crack does not occur at the grain boundary at the solidification, but at migrated grain boundary after solidification.
- 3) The fractograph of the crack gives a brittle intergranular feature, and thus is different from those of solidification and liquation cracks.
- 4) These behaviors mean that the crack is ductility-dip crack.

### Acknowledgement

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