Effect of 475°C Embrittlement on

Cast Duplex Stainless Steel*

Fracture Resistance of

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Synopsis
Cast duplex stainless steel (CF3M) which contained 15 % ferrite was
aged at 500°C for 1 000 h, and fracture toughness tests were conducted
at various loading rates and temperatures. Fracture resistance o fthe aged
material reduced largely compared with that of unaged material. The
fracture resistance of the aged material had strain rate and temperature dipendence. Fracture morphology in the aged material also changed by loading rate and test temperature. The role of embrittled frerrite phase on toughness reduction ofduplex stainless steel have described qualitatively based on scanning electron microscope observations.

Key words: cast duplex stainless teel; 475°C embrittlement; fracture toughness; dynamic fracture; fractography.

I, Introduction

Cast duplex stainless steels are widely used for structural material of chemical plants because of their high resistance to stress corrosion and superior me-chanical properties. On the other hand, it is known that they are degraded by long term aging at high temperatures. One of the degradation characteristics of the cast duplex stainless steels is 475°C embrittle-ment, which means these steels have the highest em-brittlement around 475°C. This phenomenon is at-tributed to separation of the ferrite of the Fe-Cr alloys into an iron-rich a phase and a chromium-rich a' phase. Many researches have been conducted on 475°C embrittlement of duplex stainless steels. Solo-mon and Devinei~ reviewed the phenomena in thermo-dynamical and metallurgical aspects. Chang and Chopra2~ studied microstructural characteristics by TEM observations.

Fracture toughness behavior and the role of em-brittled ferrite phase on toughness reduction of em-brittled cast duplex stainless steel are also important for evaluating structural integrity.
Only Landerman and Bamford3~ have examined fracture toughness behavior of embrittled cast duplex stainless steels. They conducted fracture toughness tests using 427°C/3 000 h-aged materials under quasi-static loading condition. They found that although a significant reduction in Charpy absorbed energy

was observed, the fracture toughness properties (Jic) did not change extensively.
Loading conditions of real structure are not only

quasi-static, but also dynamic. So it is also necessary to evaluate the effect of loading rate on fracture re-sistance of cast duplex stainless steels. Furthermore, any study has not been reported on the role of the embrittled ferrite phase on toughness reduction of cast duplex stainless steels.
The main purposes of this paper are to clarify the fracture behavior of embrittled cast duplex stainless steels and to provide a qualitative description on the effect of embrittled ferrite phase.

II, Experimental Procedure

The material used was CF3M centrifugally cast stainless pipe. Its chemical compositions and me-chanical properties are shown in Table 1. Figure 1 shows microstructures of the material. The ferrite

phase is seen in an austenite matrix and the mean ferrite content, measured by Ferrite Scope (Fischer Type FE 8e 3), is 15.8 %. Charpy and fracture toughness tests were conducted on material embrittled by aging. Charpy tests were carried out for as-re-ceived material and after aging for 200, 410 and 1 000 h to evaluate the aging time effect. Fracture toughness tests data were obtained for specimens as-received and after aging for 1 000 h.
Fracture toughness tests were conducted both in

quasi-static and in dynamic loading conditions. Quasi-static fracture toughness tests were conducted following the Jic test recommendation of The Japan Society of Mechanical Engineers (JSME).4) The specimen configuration for three point bending was 10 mm thick, 100 mm wide and 20 mm high. These specimens were machined with cracks in the circum-ferential direction. Load was applied by a screw drive test machine. Test temperature was 0°C. Crack advance during the test (4a) was marked by a heat tint method and measured as a 7 point average. Dynamic fracture toughness was measured by the

Table 1. Chemical composition (wt%) and mechanical properties.

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dynamic bend initiation technique using a drop tow-er.5> The arrangement of the drop tower and stop-block is schematically shown in Fig. 2. A hardened-steel deflection stop was used to stop the falling tup. Several amounts of deflection were obtained by chang-ing the height of the specimen support stage using different kinds of spacers. Load was measured by a strain gage mounted on the top tensile surface of the specimen. The strain gauge was located at enough distance from the cracked region where large plastic deformation occurred, and load-strain gage output relation remained linear after crack propagation. Ap-plied load-strain gage output relation was calibrated beforehand.
Load-time relations were memorized in a wave

memory and output on an X-Y recorder after testing. The values of J were calculated from the load-time

trace, using the method of Ref. 5). Test tempera-tures were 0 and 180°C. 4a was measured with the

same procedure as that of the quasi-static test.

III. Results

1. Change in Charpy Absorbed Energy by Aging
Figure 3 shows the effect of aging time and test temperature on Charpy absorbed energy (ECV) of the material. In the as-received condition, ECv had a high value over a wide temperature range and there was a ductile-brittle transition around -100°C. For

aged samples, the Charpy absorbed energy decreased

significantly from the as-received state. It increased slightly, as test temperature increased.

2. Change in Fracture Resistance by Aging
Figure 4 shows the J-4a curve for as-received and aged materials under quasi-static loading condition at 0°C. The former showed high toughness and crack initiation point on the J-4a relation could not be decided. From SEM observations, crack propaga-tion was evident on the upper three data points. Based on these results, a blunting line and a resistance curve were derived as shown in Fig. 4. Fracture toughness (JIC) and crack growth resistance (dJlda) of as-received material were obtained as 750 kN/m and 980 MN/ m2, respectively. For this JIC value, the specimens used do not satisfy the size requirement of the JIC test method.4~ On the other hand, the J-4a relation of aged material was lowered significantly as compared with that of as-received material. JIC and dJf da of the aged material obtained were 150 kNf m and 314 MN/m2, respectively. For these values, the present specimens almost satisfy the size requirement of f10 test method. Table 2 shows the reduction rate

of Ecv, JIC and dJ/da of the present results compared with those of Landerman and Barmford3~ at room

temperature. The latter used CF8M which con-tained 18 % ferrite. The subscripts " u " and " a " denote unaged and aged, respectively. In the pres-

Fig. 1. Optical microstructure.

Fig. 2. Schematic diagram
rangement.

of the drop tower stop-block ar-

Fig. 3. Charpy test results.

Fig. 4. J-d a curve (quasi-static 1oading condition).

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ent results, both JIC and ECV were reduced significant-ly by aging, but Landerman and Bamford found the reduction of JIC was not as significant as that of ECM. As mentioned earlier, JIC of as-received material in the present study was invalid. But the JIC value of Landerman and Bamford for as-received material was

valid because they have used a large size specimen

(4TCT). It has been reported that JIC decreased with increasing specimen size.6~ This may be one of the reasons for the descrepancy of the toughness re-duction characteristic between the results of Lander-

man and Bamford and the present results. So, the significant reduction of JIC by aging in the present results might be apparent phenomena. On the other hand, dJ/da reduced significantly in both studied and was almost proportional to the ECV reduction, though the size requirement of dJ/da is not clear.7~ It seems that the embrittled ferrite phase mainly affects crack

growth resistance of in cast duplex stainless steel. Figure 5 shows the loading rate dependence of the

J-4a relation for aged materials at 0°C. The crack initiation and growth resistance under dynamic load-ing condition lowered largely as compared with those under the quasi-static loading condition. Figure 6 shows temperature dependence of dynamic fracture toughness (Jia) of aged material. Jid of aged mate-rial tended to increase as test temperature rose. The loading rate and temperature ranges may not wide enough, but it is clear that the fracture resistance of aged material depended both on loading rate and test temperature. The data obtained are summarized in Table 3.

Iv. Discussion

The main cause of degradation of duplex stainless steel by aging below 500°C is thought to be 475°C embrittlement of the ferrite phase. But no report could be found on the role of embrittled ferrite phase on the reduction of the crack growth resistance. Here, a qualitative explanation was made on the effect of embrittled ferrite phase on fracture toughness reduc-tion based on SEM observations.

Figure 7 shows a typical fracture surface of as-re-ceived material under quasi-static loading condition at 0°C. Elongated dimples are mainly observed. The same fracture surface was observed on as-received

material under dynamic loading condition. Figure 8 shows typical fracture surface of aged material under

quasi-static loading condition at 0°C. Although the dimple pattern was observed, it was equiaxed dim-

ples. Figure 9 shows typical fracture surfaces of aged material under dynamic loading condition at 0°C. Fracture surface morphorogy was somewhat different from that under the quasi-static loading condition at 0°C; a crevasse-like directional fracture surface (Fig. 9(a)), a quasi-cleavage-like fracture surface (Fig. 9(b)) and an inter-granular fracture surface (Fig. 9(c)). Figure 10 shows the fracture surface of aged material under dynamic loading condition at 180°C. Equi-axed dimples are seen as it was observed on the frac-ture surface of aged materials under quasi-static load-ing condition at 0°C. Figure 11 shows the fracture surface of aged material under the quasi-static loading condition at -196°C. The same brittle fracture sur-

face was observed on aged material under the dynamic loading condition at 0°C. From the fractographic observations mentioned above, it was found that either a higher loading late or lower test temperature caused more brittle fracture morphology in the aged

Table 2. Comparison of test data.

Fig. 5. Effect of loading rate on J-4 a curve of as-received and 500°C/ 1000 h-aged material.

Fig. 6. Temperature
toughness.

dependence of dynamic fracture

Table 3. Summary of test data.

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material.
The change in fracture morphology related closely to the fracture behavior, that is, as more brittle frac-ture surface appeared, the fracture resistance was re-duced. This phenomenon was similar to the cleav-age-fibrous transition of ferric steels.
Figure 12 shows SEM micrographs of a polished surface of as-received and aged materials after elec-trochemical etching by oxalic acid. The ferrite phase of the aged material was corroded easely as compared with that of the unaged one. The reason for selective corrosion of the aged ferrite phase might be that the

iron-rich a phase caused by aging corrodes preferen-
tially. Utilizing this reduction of corrosion resistance in aged ferrite phase, the ferrite phase on the fracture

Fig. 7. SEM fractographs of as-received material
quasi-static loading condition (0°C).

under

Fig. 8. SEM fractographs of aged material
static loading condition (0°C).

under quasi-

Fig. 9. Typical SEM fractographs of aged material under dynamic loading condition (0°C)

Fig. 10. SEM fractograph of aged ma-terial under dynamic loading
condition (180°C).

Fig. 11. SEM fractograph of aged ma-terial under quasi-static load-
ing condition (-196°C).

Fig. 12. SEM observations
after etching.

of poi fished surface

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surface of aged materials was identified.
Figure 13 shows the brittle fracture surfaces shown in Fig. 9 after etching. In the crevasse-like direc-tional fracture surface (Fig. 9(a)), the longitudinal direction of the corroded ferrite phase coincided with that of ` crevasse'. The quasi-cleavage like fracture surface (Fig. 9(b)) and intergranular fracture surface

(Fig. 9(c)) were also corroded by etching. From these results, it was found that in the embrittled duplex stainless steel crack propagates through the embrittled ferrite phase at high loading rate and at low temperature.
On the other hand, Fig. 14 shows the dimple frac-ture surface of aged material after etching. Em-brittled ferrite phase existed on the bottom of the dimples. From this observation, the degradation of embrittled duplex stainless steels under quasi-static loading or high temperature condition was related to acceleration of void nucleation and growth by em-brittled ferrite phase which worked as source of voids. Figure 15 shows a schematic illustration of the crack path of embrittled cast duplex stainless steels.
The fracture behavior of embrittled cast duplex stainless steel is similar to the cleavage-fibrous transi-tion of ferric steels. So, a similar explanation is ap-plicable to fracture behavior of embrittled cast duplex stainless steel.8> Fracture of embrittled ferrite phase would be stress controlled rather than stain controlled.

And it would also have large temperature and loading rate dependence.

Under low temperature or high loading rate condi-tions, fracture stress of the ferrite phase would rela-tively low. Thus, the embrittled ferrite phase could crack at places other than the crack plane on which the principal stress is a maximum and cracks could propagate along the ferrite phase. On the other hand, under high temperature or low loading rate conditions, fracture stress of ferrite phase was relative-ly high and embrittled ferrite phase cracked mainly on the crack plane. As a consequence, the void nucleation and coalescence mechanism would domi-nant.

V. Conclusion

Charpy and fracture toughness tests on austenite-ferrite cast duplex stainless steels, embrittled by aging

Fig. 15. Schematic illustration of fracture
duplex stainless steel.

of embrittled

Fig. 13. Typical SEM fractographs of age d materials under dynamic 1oadin g condition (0°C, after etching).

Fig. 14.
Typical SEM
under dynamic
etching).

fractographs of aged materials
loading condition (180°C, after

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at 500°C, were conducted. The results are summa-rized as follows.

(1) Fracture resistance of the material reduced by aging at 500°C for 1 000 h compared with that of unaged material. There was a region where the frac-ture resistance of aged material largely depended on loading rate and test temperature.

(2) From fractographic observations, it was found that either higher loading rate or lower test tempera-ture caused more brittle fracture morphology in the aged material. This change in fracture morphology related well to the fracture toughness behavior.

(3) From the examination of electrochemically etched fracture surface, it was found that at low tem-

perature or high loading rate conditions, the tough-ness reduction was related to selective crack propaga-tion along the degraded ferrite phase, and at high temperature or low loading rate conditions, the tough-ness reduction was related to enhancement of void

nucleation and growth in ferrite phase which worked as a source of voids.

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