

Small Punch Test for the Evaluation of Thermal Aging Embrittlement of CF8 Duplex Stainless Steel

*Jin Sik Cheon and In Sup Kim
Korea Advanced Institute of Science and Technology*

*Jae Gyoo Jang and Joon Gu Kim
Korea Institute of Nuclear Safety*

Abstract

Small punch test was performed on CF8 duplex stainless steel aged at 370 and 400°C up to 5,000 h to evaluate the degree of the thermal aging embrittlement. At room temperature, the SP load-displacement curve was in a similar shape to those of ferritic steels and had a good reproducibility in spite of two-phase structure. The aging heat treatment resulted in a slight increase of the yield strength. As test temperature was lowered, the SP load showed a sudden drop followed by serrations before the SP specimen was fractured, resulting from the cracking of ferrite phase. The extent of thermal embrittlement was assessed in terms of the SP energy. Aging treatment at higher temperature led to a larger shift in the transition temperature and the corresponding change in the fracture mode. The main cause of the degradation was the embrittlement of ferrite phase. Additionally the phase boundary separation profoundly contributed to the degradation of the specimen aged at 400°C.

1. Introduction

Duplex stainless steels are degraded because several phases are formed thermally in ferrite phase when aged in the range of 280 to 500°C[1-3]. There have been various attempts to evaluate the degree of thermal embrittlement. The evaluation methods of a component at a given time of service are based on measuring the variation of magnetic or chemical properties owing to the microstructural changes during thermal aging[4,5]. However the correlation and test condition are dependent on the material condition[5]. Another method, the measurement of the ferrite hardness does not reflect the effects of ferrite content and distribution and that the hardness continues to increase although the room-temperature Charpy energy shows full embrittlement after a certain period[2].

Small Punch(SP) test was recently developed for testing irradiated materials and has been reported to achieve good results at evaluating mechanical properties in steels[6-9]. In the present work, the thermal aging embrittlement of duplex stainless steel was assessed by employing the SP test.

2. Experimental Procedure

The chemical composition of ASME SA351 CF8 duplex stainless steel is shown in Table 1. The ferrite content of unaged material slightly decreased from 13% during aging. Fig. 1 shows the microstructure of duplex stainless steel in which ferrite phase (black color) is embedded in austenite phase (gray color). The spacing of ferrite phase is nearly even but the orientation is not uniform. The Vickers microhardness of the ferrite phase is listed in Table 2 for each aging condition.

The dimension of SP specimen was 10 mm by 10 mm by 0.5 mm. SP jig consists of a puncher of 2.4 mm diameter, a ball, upper die, and lower die. The diameter of SP steel ball was 2.4 mm and hole diameter of lower die was 4 mm. SP specimen was fixed by four clamping screws between upper and lower dies. The temperature was controlled within $\pm 2^\circ\text{C}$ during the tests.

3. Results and Discussion

3.1. SP load-displacement curve of duplex stainless steel

Fig. 2(a) shows the SP load-displacement curves obtained at room temperature for one unaged and two aged conditions of CF8 duplex stainless steel. The overall shape of curves at room temperature is like that obtained from the SP test on ferritic steels. From the good coincidence of five tests shown in Fig. 2(b), one can say that the SP test for duplex stainless steel has a good reproducibility comparable to those of ferritic steels. With increasing the degree of aging, the SP load tended to increase slightly at the same displacement, which may be attributed to the hardening of ferrite phase due to aging. The yield strength was calculated by using the load at the breakaway from the initial linearity of load-displacement curve as shown in Fig. 2. The load was selected the value at $t/100$ offset from the tangent of initial slope, where t is the specimen thickness because the curve exhibited a gradual transition. For the 0.5 mm thick SP specimen, it was correlated with the yield strength by the following equation[6],

$$\sigma_{ys} = 2.63 \times \text{Load at the breakaway.}$$

The calculated yield strength is given in Table 2. The aging treatment did not result in a pronounced increase of the yield strength. Although the microhardness of the ferrite was increased about twice on the specimen aged at 400°C for 5,000 h, the effect of the aging on the SP load-displacement curve and the yield strength at room temperature was minor. It is thought that the small hardening in the load response was attained due to the small content of ferrite phase and the constant microhardness of austenite phase ($\sim 200 \text{ H}_v$).

The variation of the SP load-displacement curve for the unaged specimen with temperature is illustrated in Fig. 3. Above -150°C , the SP load exhibited a smooth decrease until the SP load dropped abruptly when the specimen was broken. In comparison, below -150°C , a sudden load drop initially occurred and the load-displacement curve exhibited repeated rise and drop of load which persisted to the fracture of the SP specimen. Fig. 4 is the scanning electron micrographs of the unaged specimen after the SP test at -175°C was interrupted before the initial load drop. The figures reveal that several cracks were preferentially build up in the ferrite phase. Immediately the cracks are arrested by ductile

austenite. As further deformation proceeds, the cracks are combined each other by tearing of austenite phase. It is believed that this process results in the initial load drop. The serration seems to be caused by the repeat of it. Similar results were obtained in the set of load-displacement curves for aged conditions except that with further aging treatment the serrated curve developed at higher test temperature.

3.2. Assessment of embrittlement of thermally aged duplex stainless steel

Based on the dependence of SP load-displacement curves on test temperature, the degree of thermal embrittlement was evaluated. The energy consumed during the test until the occurrence of the initial load drop was adopted to describe the temperature dependence of SP load-displacement curve.

The variation of the SP energy with temperature is plotted in Fig. 5 for the unaged and aged specimens. Fig. 5 shows that the SP energy increases slightly with decreasing temperature. This can be interpreted by the increase of the yield stress with decreasing temperature. It is also observed that as temperature decreases further, the SP energy shows a sudden decrease with a transition in the fracture behavior. The transition has been reported to be correlated with the ductile-to-brittle transition temperature of material[7-9]. It was also manifested by a large difference in plastic deformation at failure. Above the transition, SP specimen had a substantial deformation at failure. The difference in the transition temperature is clear between unaged and aged specimens as shown in Fig. 5. For the aged specimens, the shift in the transition temperature is larger in the specimen aged at higher temperature. At the constant aging temperatures, however, it is difficult to observe a distinct difference with aging time.

Scanning electron micrographs on the fracture surfaces of the SP specimens tested at room temperature are shown in Fig. 6. There was an obvious difference in the fracture surface, although aging heat-treatment led to a slight variation in the SP load-displacement curves at room temperature as seen in Fig. 2(a). The unaged specimen and the specimen aged at 370°C for 3,000 h consisted of ductile failure and dimpled ductile tearing on its fracture surface. The size of the dimples for the 370°C/3,000 h aged specimen was smaller than that of the unaged specimen and its depth was shallower. For the specimen aged at 400°C for 5,000 h, the fracture surface contained a significant amount of separation of the ferrite-austenite phase boundary with a little cleavage of ferrite phase as well as ductile failure.

When considering the preference of cracks at the ferrite phase, one can easily notice that the shift in the transition temperature is attributable to the hardening of ferrite phase. The embrittlement of ferrite phase is caused because dislocations are impeded by compositional fluctuations arising from spinodal decomposition and precipitation of the G phase[1-3]. In addition, carbide precipitation at the ferrite-austenite interface is considered to be a secondary mechanism of the degradation for 400°C aging. This was confirmed by observing the fractographic features of the SP specimens. The specimen aged at 400°C for 5,000 h exhibited different fracture mode distinguished from the ductile failure and dimpled ductile tearing appearing on the specimens aged below 400°C. The difference is clearly presented in Fig. 6. According to Chung[3], the precipitation of phase boundary carbide significantly occurred at 400 or 450°C for CF-8 heats containing carbon content more than

0.05 wt% and controlled the fracture mode. The carbon content of the present steel(0.057 wt%) explains the ferrite-austenite boundary separation took place in the 400°C aged specimen.

While the effect of aging temperature on the shift of the transition temperature was manifested, strict difference in proportion to aging time was not seen by means of the SP test method. This limitation, however, is not peculiar in SP test but also appeared in CVN test although the CVN test condition is much more severe than that of SP test[10]. Consequently, it is likely that the relatively small difference in aging time may not be identified by mechanical tests due to insufficient degree of microstructural change. Furthermore, it was reported that the embrittlement got saturated after aging for 2,500 h at 400°C [1].

4. Conclusions

- (1) At room temperature, the general trend and reproducibility of SP load-displacement curve were similar to those of ferritic steels. With increasing the degree of aging, the yield strength tended to increase a little. Decreasing test temperature led to a sudden drop and serrations in the SP load.
- (2) The variation of the SP energy with temperature exhibited a transition corresponding to the distinct difference in the plastic deformation at failure and fracture appearance. The transition temperature was shifted as a result of aging treatment, and the shift was larger in the specimen aged at higher temperature, which is in good agreement with the change in fracture mode.

Acknowledgment

The authors would like to thank Dr. Jun Sang Park for the provision of aged CF8 duplex steel.

References

- [1] O.K. Chopra and A. Sather, NUREG/CR-5385, ANL, 1990.
- [2] J.J. Shiao, C.H. Tsai, J.J. Kai and J.H. Huang, 6th Int. Symp. on Environmental Degradation of Materials in Nuclear Power Systems, San Diego, CA, USA, August 1-5, 1993 (The Minerals, Metals & Materials Society, 1993).
- [3] H. M. Chung, Int. J. Pres. Ves. & Piping 50 (1992)179.
- [4] S. Evanson, M. Otaka and K. Hasegawa, J. Eng. Mat. & Tech. 114 (1992)41.
- [5] J.S. Park and Y.K. Yoon, Scripta Metall. 32 (1995)1163.
- [6] J.S. Cheon and I.S. Kim, J. Testing and Evaluation, To be published.
- [7] J.M. Baik, J. Kameda and O. Buck, ASTM-STP 888 (1986)92.
- [8] M. Suzuki, M. Eto, Y. Nishiyama, K. Fukaya and T. Isozaki, ASTM-STP 1204 (1993)217.
- [9] J. Kameda and O. Buck, Mater. Sci. & Eng. 83 (1986)29.
- [10] P. McConnell, W. Sheckherd and D. Norris, J. Mater. Eng. 11 (1989)227.

Table 1. Chemical composition(wt%) of CF8 duplex stainless steel.

C	Si	Mn	P	S	Ni	Cr	Mo
0.057	1.34	0.62	0.003	0.002	8.23	19.94	0.21

Table 2. Microhardness of ferrite phase and yield strength for various aging conditions.

Aging conditions	Unaged	370°C/1,500h	370°C/3,000h	400°C/2,500h	400°C/5,000h
Microhardness(H_{v50})	258±11	304±11	341±15	394±17	445±12
Yield strength[MPa]	316±10	356±26	337±9	360±18	376±26



Fig.1 Ferrite morphology of CF8 duplex stainless steel.

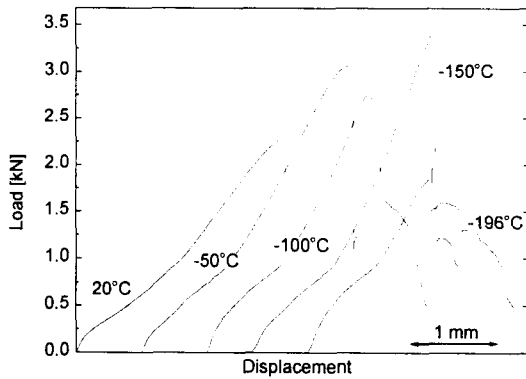
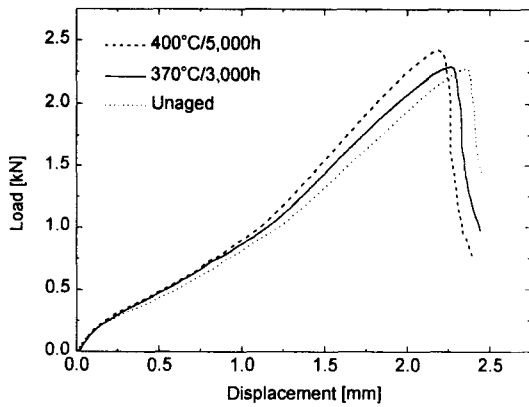
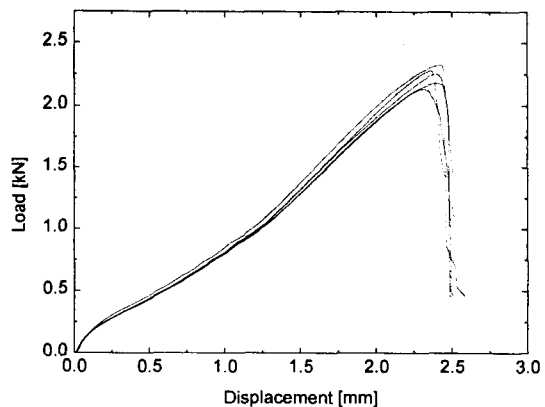


Fig. 3 Load versus displacement curves at various temperatures for unaged specimen.



(a)



(b)

Fig. 2 Load versus displacement curves at room temperatures; (a)three different conditions, (b) five unaged specimens.

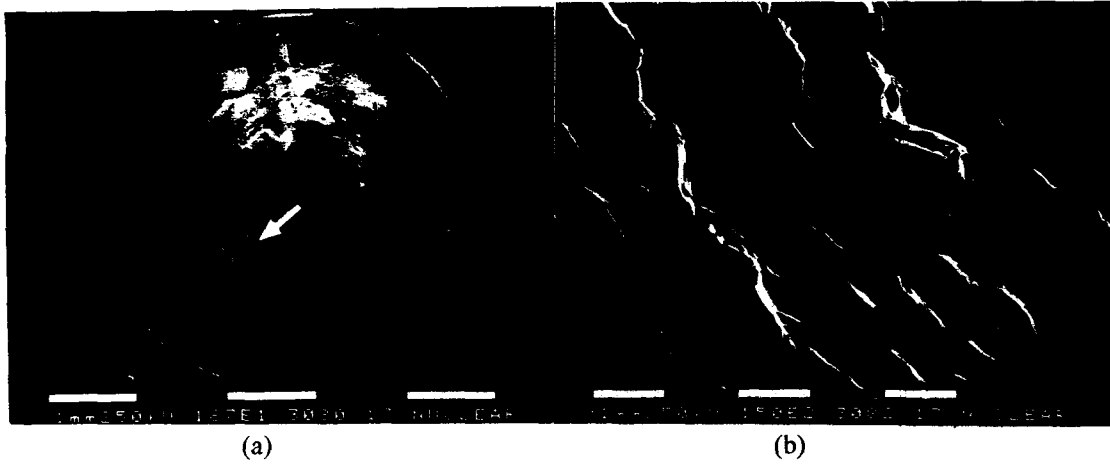


Fig. 4 Scanning electron micrographs of unaged specimen after the SP test at -175°C was interrupted; (a) overall appearance, (b) is higher magnification of the region indicated in (a).

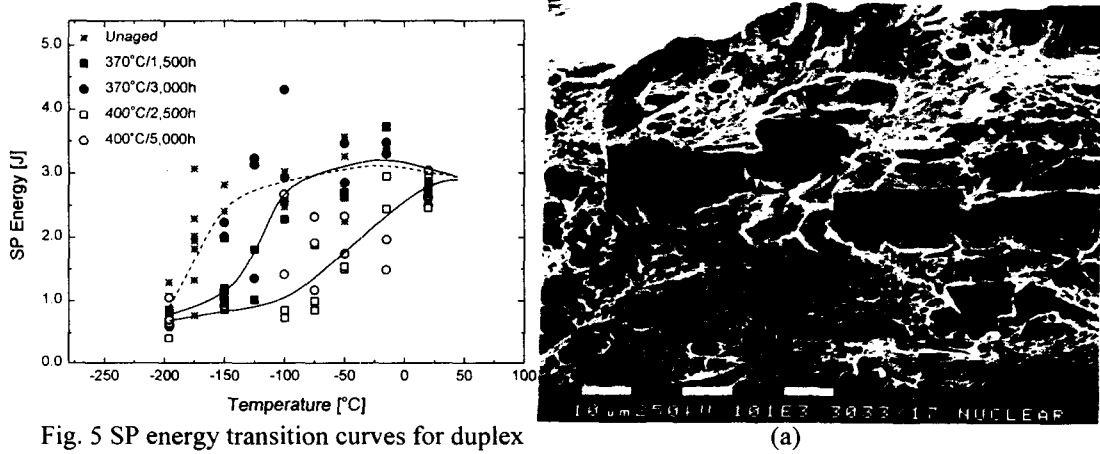


Fig. 5 SP energy transition curves for duplex stainless steel.

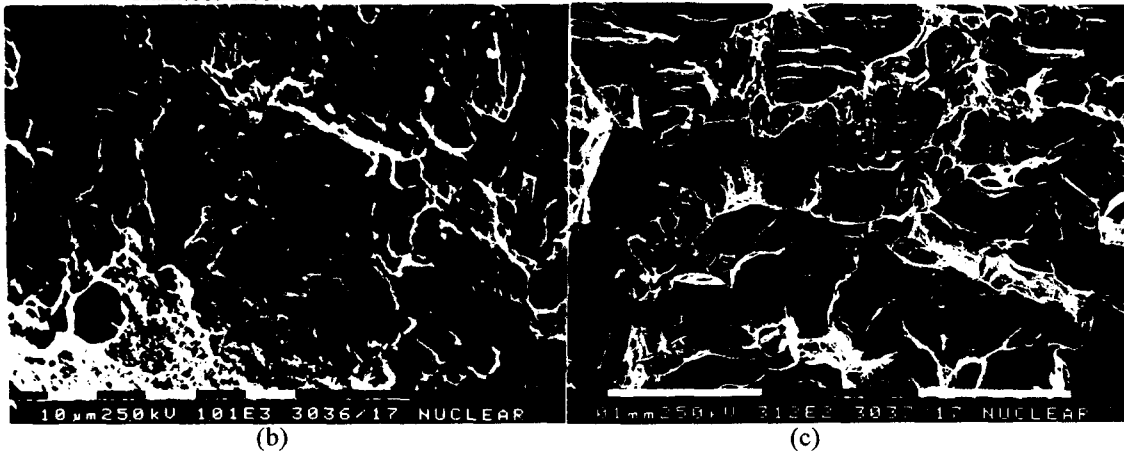


Fig. 6 Scanning electron micrographs of the fracture surface at room temperature; (a) unaged, (b) $370^{\circ}\text{C}/3,000\text{ h}$, (c) $400^{\circ}\text{C}/5,000\text{ h}$.