



## Cracking behavior and positron annihilation measurement during fatigue damage in austenitic stainless steel

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### ABSTRACT

Fatigued deformation in the type 316 stainless steel was evaluated using microscopic crack observation and a positron annihilation measurement. A few microcracks of several-10  $\mu\text{m}$  length were observed where the slip line density was high in a grain, at 20 % of the fatigue life. The subsequent crack growth was continued by both propagation and coalescence with next cracks. The normalized crack length, which does not have any influence of the main crack site or minimum crack length on the statistics analysis, can be one of the valid parameter for representation of fatigue damage. The positron annihilation lineshape parameter (S-parameter) increases up to 10 % of the fatigue life in fatigue deformation, and supplement to describe of the fatigue damage in the early fatigue stage.

### INTRODUCTION

Fatigue damage is well-known as one of the cause of failure of structural materials. The fatigue process of metals is divided into the following three stages: the stages of crack initiation, crack propagation and final failure. Understanding about fatigue damage has increased considerably recently, however, the crack initiation event is the least well understood of the various aspects of fatigue [1][2]. The conditions for the crack initiation and the rate of its propagation are strongly influenced by microstructure.

On the other hand, it is becoming desirable to establish a nondestructive residual life prediction method in connection with the plant life extension. The microscopic observation of the material surface, where micro cracks develop, reflect mechanism of failure directly, and has a possibility to apply as a nondestructive detection of fatigue damage[2]-[4]. Positron annihilation measurement is also one of the nondestructive techniques, which is said to be sensitive to the population of lattice defects induced by plastic deformation[5]-[7]. In this paper, material degradation during low cycle fatigue deformation in austenitic stainless steel

were evaluated using microscopic crack observation and a positron annihilation measurement. The relationship between a process of a low cycle fatigue damage in austenitic stainless steel and how to apply nondestructive techniques will be discussed.

### EXPERIMENTAL PROCEDURE

The chemical composition of the type 316 stainless steel is shown in Table 1. Fig. 1 shows the dimension of specimens used for the fatigue tests. The gauge sections of these specimens, which had two flat planes of 5 mm width and 20 mm length, were polished with emery papers, and some of them were electropolished to observe the slip lines and cracks.

Fatigue tests were performed by strain-controlled axial tension-compression cyclic deformation at room temperature with 0.6 % total strain range. Failed and terminated specimens were prepared as shown in Table 2. We defined the 100 % fatigue life,  $N_f$ , as the number of cycles at which the stress reduced by 25 %, and the fatigue life fraction,  $N/N_f$ , as the proportion to the number of cycles  $N$  to  $N_f$ . One sample was fatigued to  $N_f$ , and from time to time during the fatigue test, replicas were taken from the electropolished surface.

Slip line and crack observation were performed by optical microscope and laser microscope. A main crack and some major sub cracks were observed and measured their length at each fatigue life. To obtain statistics data, all cracks were observed and measured in two points of 4 mm x 4 mm square.

Positron annihilation measurement is based on the detection of annihilation gamma-ray energy through the combination of the electrons in a material with the positrons entering from a radioactive isotope. In damaged materials the gamma-ray lineshape changes narrower. S-parameter was used to evaluate the lineshape change. Fig. 2 shows the positron annihilation equipment used in this study. The S-parameter is given by the central region gamma-ray counts divided by the total counts (Fig.3). In damaged materials, the gamma-ray lineshape becomes narrow and the S-parameter increased. In this study,  $^{68}\text{Ge}$  was used for a positron source and these positrons penetrate in steel down to 250  $\mu\text{m}$  approximately.

Table 1. Chemical composition of the type 316 stainless steel

	C	Si	Mn	P	S	Cr	Ni	N	Mo
wt%	0.007	0.50	0.83	0.025	0.004	16.48	10.98	0.075	2.08

Table 2. Summary of fatigue tests

Total strain range		0.60 %
Temperature		RT
Terminated fatigue life $N/N_f$	for positron annihilation measurement	0.01, 0.1, 1, 3, 10, 20, 100 %
	for crack observation	0.01, 0.03, 0.1, 1, 3, 10, 20, 30, 40, 55, 70, 85, 100 %

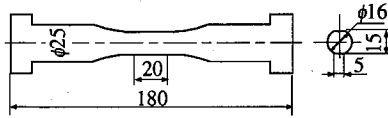


Fig. 1 Dimensions of the specimens.

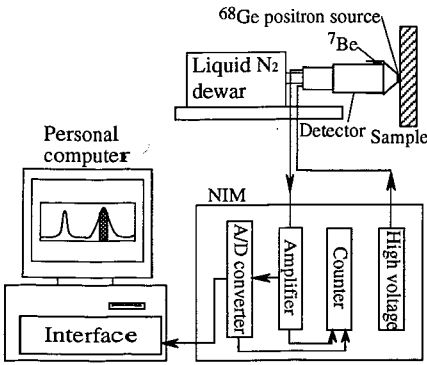


Fig. 2 Positron annihilation lineshape analysis system.

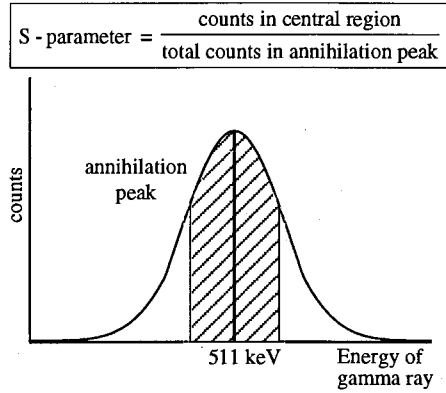


Fig. 3 Definition of S-parameter in positron annihilation lineshape analysis.

## RESULTS AND DISCUSSION

### Initiation of slip lines and cracks

Slip lines were seen even at 0.01 % of the fatigue life (3 cycles). The density of slip lines increased with the cyclic deformation. A few microcracks of several-10  $\mu\text{m}$  length were observed where the slip line density was high in a grain, at 20 % of the life. The direction of cracks was perpendicular to the stress axis, and was not parallel to that of slip lines.

### Growth of microcracks

An example of crack growth behavior is shown in Fig. 4. Identical cracks were observed and traced at each fatigue life, and the numbers on the cracks indicate their length in  $\mu\text{m}$ . Microcracks propagated themselves after the initiation. The subsequent crack growth was continued by both propagation and coalescence with next cracks. Furthermore, cracks over 100  $\mu\text{m}$  helped to initiate new cracks near the original cracks. It was caused by the stress or strain concentration near the edge of the cracks at crack propagation. The original cracks took in the new cracks and growth longer.

Fig. 5 shows the growth behavior of the main crack and the sub main cracks. The crack growth rate of the main crack was not high as compared with other cracks at the early stage of the fatigue. The main crack grew rapidly after 75 % of the fatigue life. Fig. 6 indicates the growth behavior of the main crack in detail. All cracks, which jointed the main cracks at the

final failure, were measured their length. Large number of cracks were observed and major ones are plotted. In this case, several cracks over 100  $\mu\text{m}$  coalesced and this crack happened to be the main crack. Therefore, crack growth rate over several-100  $\mu\text{m}$  was different in each crack. The sub main cracks did not have a chance to coalesce with other long cracks in Fig. 5.

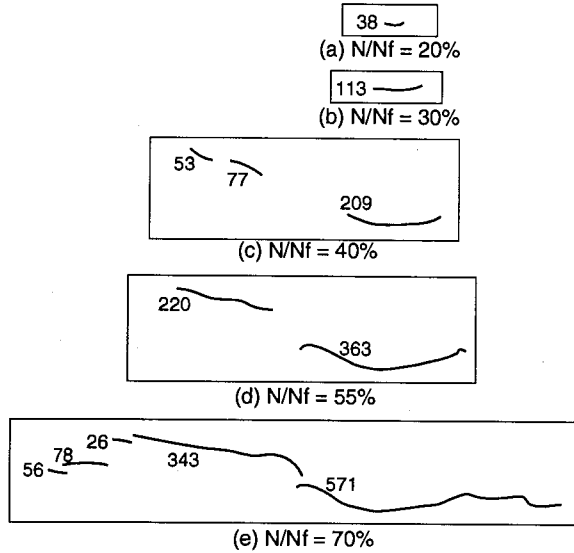


Fig. 4 An example of crack distribution at each fatigue life (The unit of the numbers is  $\mu\text{m}$ ).

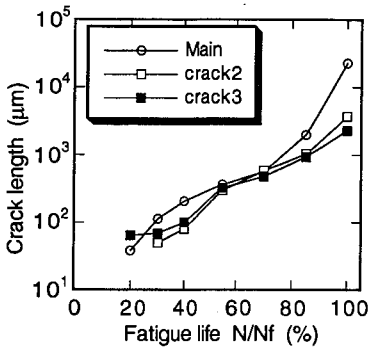


Fig. 5 Growth of the main and the sub main cracks.

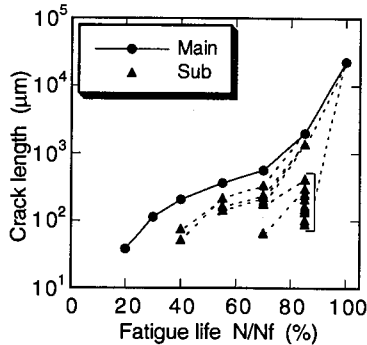


Fig. 6 Coalescence behavior of the main crack in detail.

*Microcrack distribution*

Fig. 7 and Fig. 8 illustrate change of the histograms of crack length against fatigue life. To clarify the effect of the exist of main or sub main crack, Fig. 7 shows the cracking behavior in the region including the main crack, and Fig. 8 shows that where there were far from not only

the main crack, but also sub main cracks. Though all cracks in area 4 mm x 4 mm were observed and measured, cracks less than 25  $\mu\text{m}$  were disregarded in this analysis.

The crack density, that is the number of cracks in unit area, increased with fatigue life in both two areas. However, the shape of the histograms were not similar after 55 % of the fatigue life. Comparative statistics plots of the crack density and normalized crack length, which means total crack length in the area divided by the area size, are shown in Fig. 9 and 10. Each plot was analyzed with both cracks over 25  $\mu\text{m}$  and cracks over 50  $\mu\text{m}$ . The change of the crack density in two areas shows similar behavior in data evaluated over 25  $\mu\text{m}$  crack, but different tendency in data of over 50  $\mu\text{m}$  crack. On the other hand, normalized crack length in two areas shows good agreement up to about 80 % of the fatigue life in both over 25  $\mu\text{m}$  and over 50  $\mu\text{m}$ .

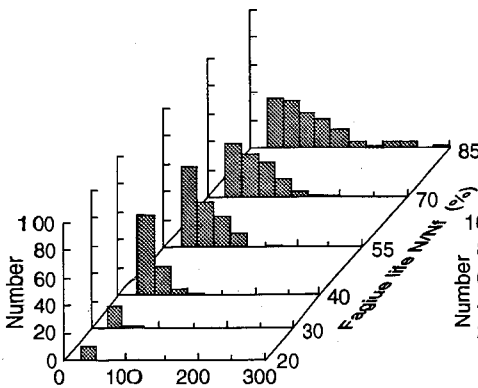


Fig. 7 Crack length distribution in the area far from the main crack.

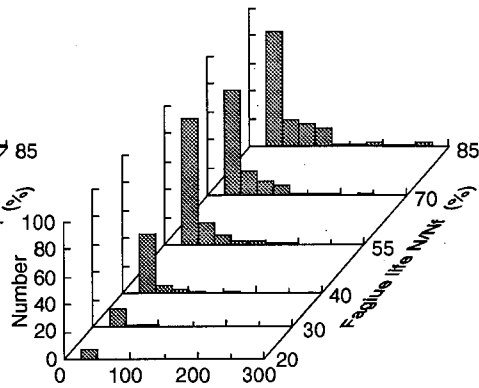


Fig. 8 Crack length distribution around the main crack.

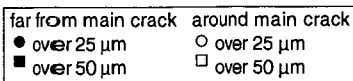
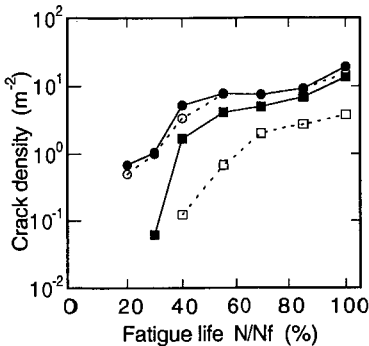


Fig. 9 Change of crack density in fatigue life.

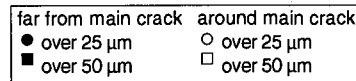
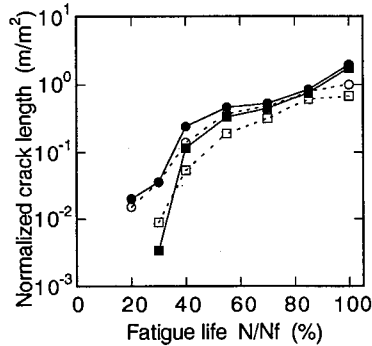


Fig. 10 Change of normalized crack length in fatigue life.

The reason for the dependency of the evaluated crack length is the different in crack propagation behavior between the main crack and other small cracks. Around the main crack, many microcracks initiated, and then the crack density increased rapidly, however there were few cracks over 50  $\mu\text{m}$ . The propagation of the main crack also had priority. This provides slow growth to other cracks. In the area not including large cracks, each crack, which did not have any interaction with other cracks, grew respectively and some of them grow into over 50  $\mu\text{m}$ . These can explain the crack density behavior, but are not effective to the normalized crack length.

It is difficult to observe and identify cracks less than 50  $\mu\text{m}$  even in laboratory examination. In this study, we used well polished specimens and high magnification observation and then we could find microcracks and measure their length. Cracks over 50  $\mu\text{m}$  can be found relatively easily. Regarding the convenient of experiment, longer length is preferable for requested minimum crack length. The normalized crack length, which does not have any influence of the main crack site or minimum crack length on the statistics analysis, can be one of the valid parameter for representation of fatigue damage[3].

#### *Positron annihilation*

Fig. 11 shows the results of the positron annihilation measurement. Change in S-parameter means S-parameter change from the as-received samples. S-parameter increased markedly up to 10 % of the fatigue life, while there were not any cracks on the surface. It is considered that positrons detect vacancy-like defects which are produced by dislocation movement and tangle[8]. It can be said that positron annihilation measurement is useful in the early stage of fatigue life, when there may be no cracks.

On the other hand, crack observation method can not detect the material degradation before crack initiation, of course. It is concluded that for the damage evaluation, it is necessary to combine these materials characterization methods compare with the objective material .

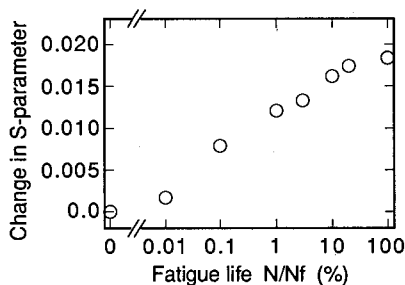


Fig. 11 Results of positron annihilation measurement.

#### CONCLUSION

Fatigue damage was induced in the type 316 stainless steel at room temperature. Material

degradation during low cycle fatigue deformation was evaluated using microscopic crack observation and a positron annihilation measurement. The following conclusions were reached.

- (1) It is concluded that the crack growth after initiation was continued by both propagation and coalescence with next cracks.
- (2) The normalized crack length, which does not have any influence of the main crack site or minimum crack length on the statistics analysis, can be one of the valid parameter for representation of fatigue damage.
- (3) The positron annihilation lineshape parameter (S-parameter) increases up to 10 % of the fatigue life in fatigue deformation, and supplement to describe of the fatigue damage in the early fatigue stage.

#### Reference

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