ROLE OF OXIDATION IN MICRO-FRACTURE PROCESS OF AISI 316 STAINLESS STEEL IN HIGH TEMPERATURE LOW CYCLE FATIGUE

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ABSTRACT

An effect of oxidation on the initiation and early stage of propagation of fatigue crack was studied on AISI 316 stainless steel at the temperature of 700°C with 3 cpm in frequency. The parameter ρ "the crack density" (Kunio, Iwamoto and Kanazawa, 1969) was introduced to evaluate the characteristic feature of fracture process involving multiplication of microcracks at the early stage of fatigue in air and vacuum. The transition in crack growth mode from intergranular to transgranular was well characterized by this parameter. The role of oxidation in the transition of fracture mode towards depth direction was discussed based on the results of metallographic and fractographic analyses of the micro-fracture process depending on the environments imposed.

KEYWORDS

Fatigue; high temperature low cycle fatigue; oxidation; intergranular crack; crack density; transition point; crack propagation rate.

INTRODUCTION

Presently, it is commonly believed that the fatigue life in high temperature low cycle range in air is shorter than that in vacuum (Jacisin, 1967; Homma, 1975) except for a special case of Ni-base superalloy (Duquette and Gell, 1972) in which shorter fatigue life appears in vacuum compared with that in air. As a reason, it can be pointed out that the localized oxidation along grain boundary or grain boundary itself plays a significant role as a preferential site of crack initiation and propagation (Mcmahon, 1974), although many discussions regarding such an oxidation effect are made specifically from a qualitative viewpoint because of difficulty in grasping the complicated properties of cracks at the initiation and early stage of its propagation in the high temperature low cycle fatigue process (Coffin, 1973). Thus, it can be said that this essential problems have not been fully understood yet. The present study is intended to make clear the role of oxidation on the initiation and propagation of microcracks in the high temperature low cycle fatigue.

SPECIMEN AND EXPERIMENTAL PROCEDURE

The material used in this study is the austenitic stainless steel AISI 316. The chemical composition of this material is shown in Table 1. Fatigue specimens were machined into the shape and dimension as shown in Fig. 1 after applying a standard solution heat treatment at 1100°C for 1 hr. The surface microstructure after polishing and aqua regia etching is given in Fig. 2. The average grain size of the microstructure was obtained as 70 µm using an ASTM method (ASTM standards, 1980). Mechanical properties at room temperature of this material is shown in Table 2. The tension-compression low cycle fatigue test with 3 cpm in frequency was carried out on the above specimen in both air and vacuum under the diametral displacement control condition employing an servo-controlled fatigue testing machine (MTS). Test temperature was in the range from 650°C to 800°C in both environments. The vacuum was maintained as high as 10^{-5} torr during the fatigue test. The measurements of crack length were made by an optical microscope on the surface.

RESULTS AND DISCUSSION

Feature of Micro-fracture Process in Air and Vacuum

TABLE 1 Chemical Composition (wt %)

Material	C	Si	Mn	P	S	Ni	Cr	Mo
AISI 316	0.05	0.38	1.38	0.034	0.023	10.01	17.13	2.02

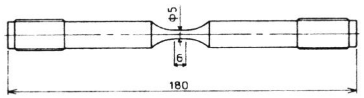


Fig. 1. Dimensions of fatigue specimen (mm).

TABLE 2 Mechanical Properties of Material at Room Temperature

0.2% Yield Strength MPa	Tensile Strength MPa	Elongation %	Reduction of Area %	Hardness Hv
274	628	62.0	71.2	156

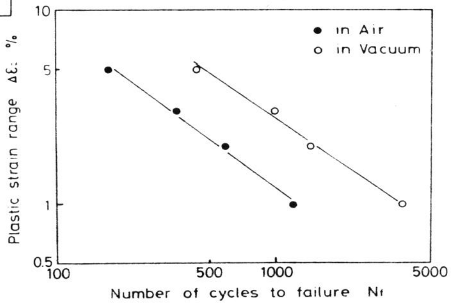


Fig. 3. Relationship between plastic strain range and cycles to failure in air and vacuum (700°C, $\nu = 3$ cpm).

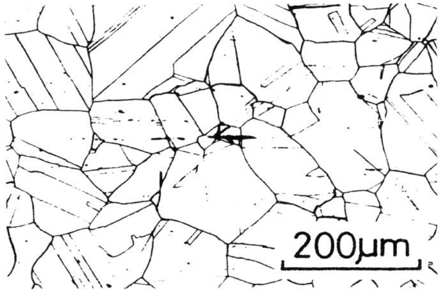
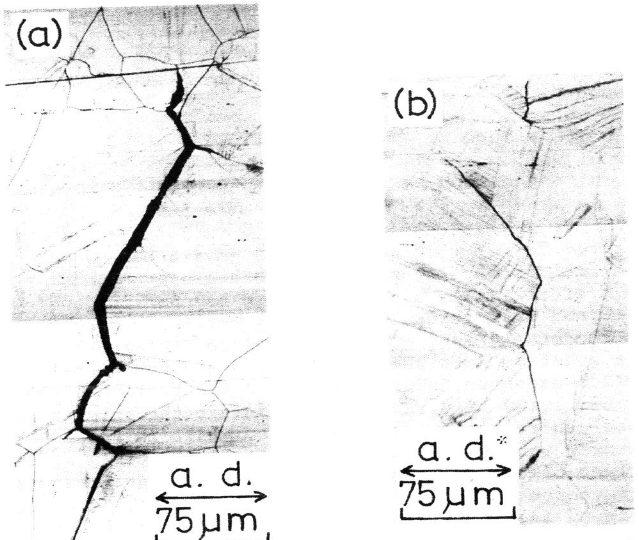
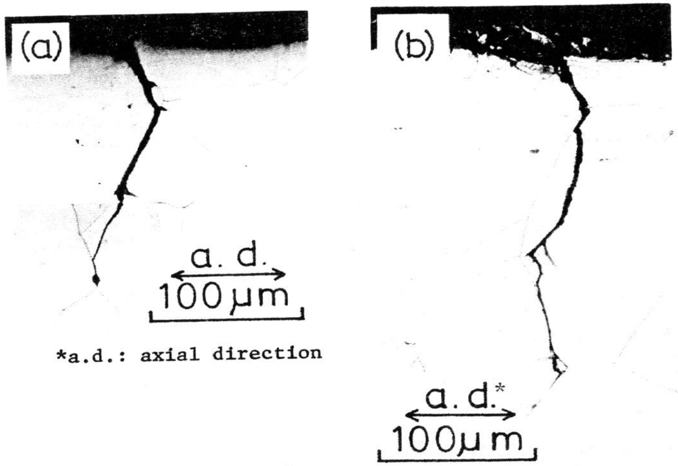


Fig. 2. Microstructure of material.



*a.d.: axial direction

Fig. 4. Surface microcracks on AISI 316 (700°C, $\Delta\varepsilon_p$=1%, $\nu = 3$ cpm).
(a) in air N = 400 (b) in vacuum (10^{-5} torr) N = 650



*a.d.: axial direction

Fig. 5. Longitudinal cross section of AISI 316 showing fatigue cracks (700°C, $\Delta\varepsilon_p$=1%, $\nu = 3$ cpm).
(a) in air N = 385 (b) in vacuum (10^{-5} torr) N = 975

Figure 3 shows a relationship between the plastic strain range and the fatigue life obtained under the experimental condition of 700°C and 3 cpm in both environments of air and vacuum. The number of strain cycles leading to failure in vacuum is three times larger than that in air. The difference between these fatigue lives could be ascribed to the environment to which the specimens were exposed, i.e., in vacuum and air, because other parameters had been kept constant during the fatigue tests. Then the crack growth behavior at the early stage of fatigue loading was examined at $\Delta\varepsilon_p = 1\%$ in both environments. The results show that intergranular cracking is the predominant mechanism of the initiation and early stage of propagation on the surface in both environments as shown in Fig. 4-(a),(b). However, in the depth direction away from the surface, a different feature of crack propagation was observed through the metallographic examination. Figure 5-(a),(b) shows the following difference of the feature of cracks in both environments. In the air, a wedge-shaped crack develops along the surface grain boundary and its initial growth occurs preferentially along the grain boundary expanding about two grain diameters and then turns into the transgranular mode of propagation. In vacuum, on the other hand, cracks take place preferentially in the surface grain boundary and turn into the grain within a depth of only one grain size below the surface as shown in Fig. 5-(b). A fractographic observation as shown in Fig. 6-(a),(b) also reveals that in air the intergranular facet is frequently observed on the fractured surface while in vacuum the striation pattern is clearly recognized in the vicinity of specimen surface with no appreciable trace of intergranular facet. On the fractured surface which is located at 1 mm depth below the specimen surface, a typical striation pattern, as shown in Fig. 7-(a),(b), is observed in both cases. These evidences indicate that although an essential feature of fatigue process would be basically the same for both environments, the difference in the behavior of crack growth near the surface is likely to exist at the early stage of crack growth. Then further study was made on this difference from a quantitative viewpoint in terms of the parameter "crack density ρ" (Kunio, Iwamoto and Kanazawa, 1969) which is related to the total crack length per unit area.

An Effect of Oxidation on Early Stage of Crack Propagation

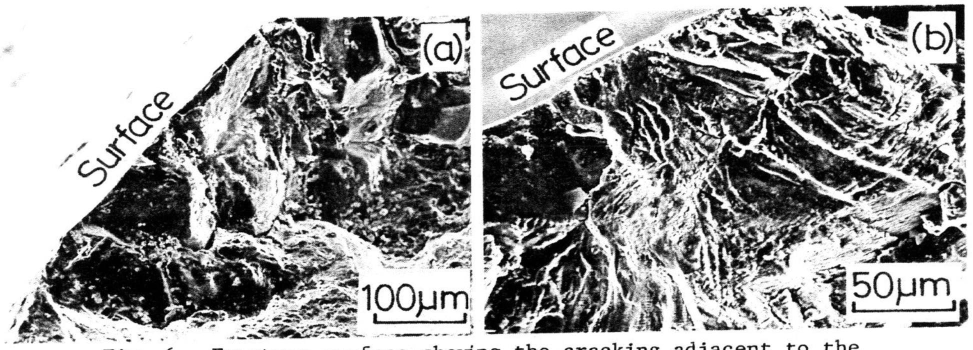


Fig. 6. Fracture surface showing the cracking adjacent to the specimen surface, SEM (700°C, $\Delta\varepsilon_p$=1%, ν= 3 cpm).
(a) in air (b) in vacuum (10^{-5} torr)

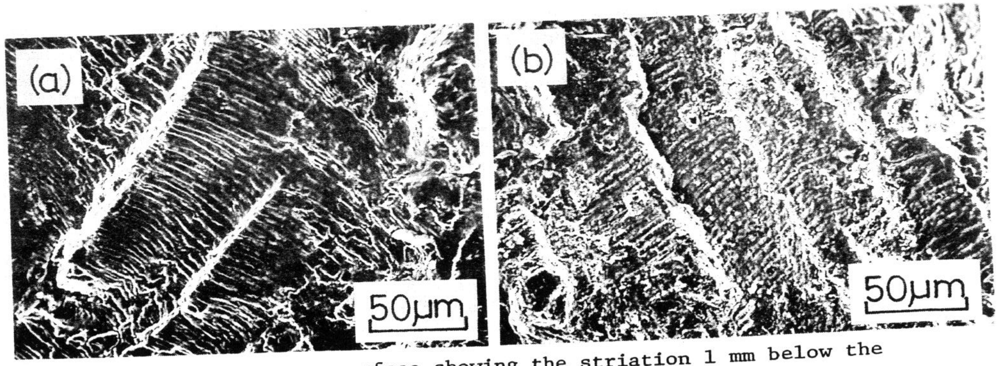
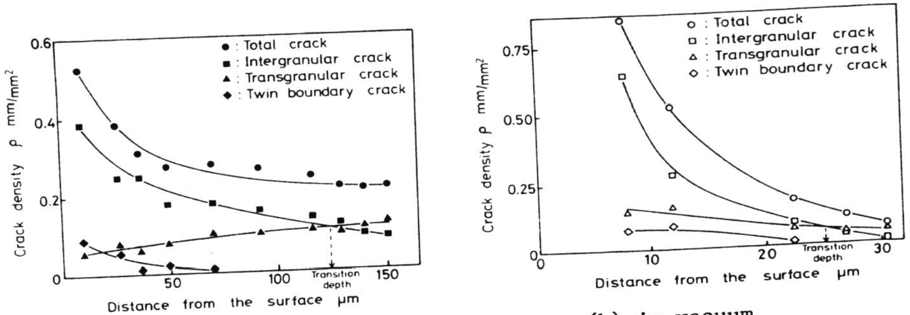


Fig. 7. Fracture surface showing the striation 1 mm below the surface, SEM (700°C, $\Delta\varepsilon_p$=1%, ν= 3 cpm).
(a) in air (b) in vacuum (10^{-5} torr)



(a) in air (b) in vacuum

Fig. 8. Change of the crack density of inter- and trans-granular mode of crack towards the depth direction (700°C, ν = 3 cpm).

Figure 8-(a),(b) gives a variation of crack density regarding to the inter- and trans-granular cracks along the depth direction in both environments. From these figures, the intergranular selectivity of crack path is lost gradually in air, while in vacuum it is lost rapidly with respect to the depth direction. No appreciable difference was observed for twin boundary cracking in both environments. In order to evaluate this transitional behavior from intergranular cracking to transgranular one in the depth direction, the depth at which both intergranular and transgranular crack density distribution curves intersect was defined as a "transition point". This may stand for some indications how hard the oxidation attack is. The depth at this transition point for present materials was obtained from Fig. 8 as 125 μm in the air and 25 μm in the vacuum respectively. The depth of this transition point would be closely related to the localized oxidation along the grain boundary leading to the intergranular cracking. If the environment is on the way to more severe condition for oxidation, the corresponding depth to the transition point should be increased with the severity of oxidation. Then the test temperature was varied from 650°C to 800°C, while keeping the plastic strain range and loading frequency constant. The results are given in Fig. 9. It is recognized from this figure that the transition occurs at deeper depth with an increase in temperature in air. In vacuum, on the other hand, the transi-

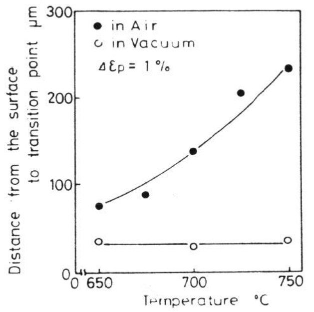


Fig. 9. Temperature dependency of the transition depth of crack from intergranular mode to transgranular one (ν = 3 cpm).

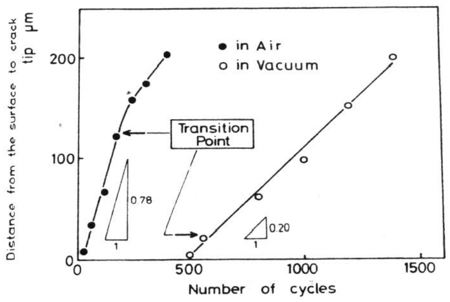


Fig. 10. Crack initiation and growth behavior towards depth direction (700°C, $\Delta\varepsilon_p$=1%, ν= 3 cpm).

tion point appears almost the same depth irrespective of the test temperatures. As a result, oxidation gives a promoting effect of the intergranular cracking mode in the fatigue process.

An Effect of Oxidation on The Crack Propagation Accompanying Striation

The crack propagation rates before and after the transition point were measured in order to examine the cause for the difference of fatigue life between both environments. Figure 10 shows the relationship between the maximum crack length in depth direction and the number of plastic strain cycles. The maximum crack length was obtained from the successive removal of surface layer until the trace of crack vanished. It can be observed from Fig. 10 that the number of cycles to crack initiation in the air is overwhelmingly smaller than that in vacuum, that is, N=20 in air and N=480 in vacuum. This would be due to the localized oxidation (Mcmahon , 1974) at the grain boundary in air. Moreover, the average initial crack propagation rate before the transition point was obtained as 0.78 μm/cycle in air and 0.20 μm/cycle in vacuum respectively. This means that the transgranular crack which propagates forming striations has slow propagation rate as compared with the case where the crack propagates preferentially along grain boundary. Similar result has been reported for A 286 by Coffin (Coffin, 1972). Regarding the crack propagation rate after the transition point, little difference is recognized from the comparison of the crack propagation rate in Fig. 10 and also from the comparison of striation spacings at the same crack depth, i.e., 1.5 mm below the surface, in both environments, that is, 6.8 μm/cycle in air and 4.5 μm/cycle in vacuum. This implies that the oxidation products on the crack surface may not affect the crack growth behavior after the transition point. The true reason for this has not been obtained at this moment. However, the following possible reasons might be given to the interpretation of this result. (1) A crack closing effect due to oxidation of the crack surface takes no part in the crack propagation in the present fatigue experiment, where the stress intensity is considerably higher than the ΔK_{th} of the material. The decrease in crack opening ratio U due to oxidation of the crack surface has been observed to occur only near the ΔK_{th} level (Hasegawa, 1982). (2) No net effect of the oxidation appears in the crack growth behavior, because the decrease in the intrinsic resistance for crack growth (e.g. decrease in surface energy (Smith, Shahinian and Achter, 1969)) due to oxidation masks the mechanical effect which suppress the crack growth owing to the oxide induced closure effect (Suresh, Zamiski and Ritchie, 1981). Consequently, it may be concluded that the effect of oxidation has a predominant role on the early stage of crack propagation in the high temperature low cycle fatigue behavior.

CONCLUSIONS

The low cycle fatigue tests of AISI 316 stainless steel were carried out in air and vacuum at elevated temperatures in order to study the effect of oxidation on the initiation and propagation from the mechanical and metallurgical viewpoints. The results obtained are summarized as follows:

(1) Transition phenomenon of crack mode from intergranular to transgranular one was characterized by the parameter "crack density ρ" in both vacuum and air.

(2) A severity of oxidation attack on the early stage of crack propagation can be evaluated from the depth of transition point obtained from the distribution curves of microcrack density under both environments. The transition point depth is defined by the distance from the specimen surface to the point at which the intergranular crack preferentially propagating inwards along the grain boundary turns into the transgranular one.

(3) As for the crack propagation rate prior to reaching the transition point, the oxidation gives a promoting effect of the intergranular cracking mode in the fracture process resulting in the high propagation rate in air compared with that in vacuum.

(4) Discussion has also been made on the effect of oxidation on the crack propagation accompanying the striation pattern.

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