High-Temperature Fatigue Crack Propagation Property of Mod.9Cr-1Mo Steel under Vacuum and Air Conditions*

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Microcrack propagation behavior of Mod.9Cr-1Mo steel under high-temperature fatigue in vacuum and air conditions were periodically observed. Microcracks propagated in the direction normal to stress axis in vacuum while the microcracks propagated in the maximum shear direction in air condition. It was found that propagation rate in air was faster than that in vacuum until the crack grew 1 mm length due to oxidation effect which was insignificant for the crack larger than 1 mm. Acceleration of the crack propagation rate in the compression hold test was supposed to be caused by accumulation of tensile strain in the center of the specimen due to off-balance of strain distribution between tension and compression side.

Key Words: Crack Propagation, Fatigue at Elevated Temperature, J-Integral, Mod.9Cr-1Mo Steel, Oxidation Effect, Vacuum, Strain Hold, Continuous Observation

1. Introduction

As Mod.9Cr-1Mo steel has superior high-temperature strength property, it is used for high-temperature components in power plants. Creep-fatigue life property and evaluation methods for the steel have been intensively investigated. Until now, it was reported that fatigue life reduction under compression hold was much larger than that under tensile hold which was different from austenitic stainless steels\(^{(3)-(5)}\). Some studies suggested that life reduction under compression hold would be caused by the effect of mean stress yielding during tests\(^{(4)}\) or acceleration of crack initiation and propagation rates due to oxidation effect\(^{(5)}\). On the other hand, the authors showed that life reduction could not be interpreted by oxidation effect because it happened even in vacuum condition\(^{(6)}\). The reason of this feature is still controversial and uncertain.

In this study, high-temperature fatigue tests with and without strain hold were carried out in vacuum and air environments and microcrack initiation and propagation behaviors, which dominate low-cycle fatigue life, was observed periodically. Influence of environment on microcrack propagation and the reason of life reduction in compression hold were discussed.

2. Test Procedure

2.1 Material

Material used in this study was Mod.9Cr-1Mo steel. Chemical composition and mechanical property are shown in Table 1. The material was treated by quenching and tempering followed by post welding heat treatment, 740°C and 504 min. Microstructure of the material is temper martensite and mean grain diameter is about 10 µm.

2.2 Specimen and testing machine

A specimen geometry, which is solid bar with 10 mm diameter and 10 mm gage length, is shown in Fig. 1. In order to observe crack initiation and propagation behaviors easily, a small notch of 0.1 mm depth was introduced by electric discharge processing in the
center of gage length. A test equipment is an electromechanical high-temperature fatigue testing machine with an induction heating device. The maximum load is 98 kN and maximum operating temperature is 1000°C. The testing machine used for tests in vacuum has a vacuum chamber and a turbo molecular exhaustion device. Degree of vacuum is about 10^-4 MPa.

2.3 Test condition

Test temperature was 550°C and strain waveforms are triangular, 10min compression hold and 60 minutes tension hold with strain range of 1.0%, strain rate of 0.1%/sec. Each test was stopped at predetermined cycles to take replica film from the surface. Crack length on the film was measured by optical microscope and it was defined as the straight length connecting both edges of a crack along crack propagation direction.

3. Test Results and Discussion

3.1 Crack initiation and propagation behavior

The crack initiation and propagation behaviors under the PP in vacuum and air environments observed by the optical microscope were shown in Fig.2. Micro cracks initiated from the notch in the direction normal to the stress axis both in vacuum and air conditions. Number of cycles to microcrack initiation from notch in vacuum was about 2000 cycles while that in air was about 300 cycles. Thus number of cycles to crack initiation in air condition reduced to 1/7 compared with that in vacuum condition. Duquett(4) was reported that the fatigue crack initiation period in air reduced compared to vacuum condition at room temperature due to the fact that the crack initiation was caused by larger irreversible slip in air than that in vacuum due to oxidation of the created surface by cross slip motion. It is easy to imagine that oxidation effect becomes much significant at high-temperature where cracking of oxidation film is likely to accelerate crack initiation at specimen surface. The microcrack grew with branching and opening in the same direction as initiation direction in vacuum condition whereas propagation direction in air inclined about 45° from the initiation direction with branching cracks. Thus crack propagation behavior is different between vacuum and air conditions even under the same temperature and loading conditions.

Fig. 1 Specimen Geometry

Fig. 2 Crack initiation and propagation behavior from notch under PP waveform (top: vacuum, bottom: air)
Figure 3 shows crack propagation behavior under 10 minutes compression hold. A crack initiated from notch at 100 cycles in vacuum and slip bands are more visible than those in air. When it compares with the PP test result in vacuum, number of cycles to crack initiation reduced to 1/5. It suggests that producing of a crack plane is accelerated by irreversible deformation in asymmetric waveform greater than that in symmetric waveform. Although number of cycles to crack initiation was almost the same between in vacuum and in air conditions, propagation rate in vacuum is slower than that in air and ductile opening of the crack to the loading direction was occurred by elongating the notch after 1 000 cycles. On the other hand, number of cycles to crack initiation under compression hold in air was nearly equal to that in the PP. As same as observed in the PP, crack propagation direction changed to about 45° from the direction normal to stress axis. Crack opening displacement increase after 500 cycles due to accumulation of irreversible shear deformation to tensile direction in spite of compression hold. It is suggested that tensile residual strain accumulated in the center of the specimen due to concentration of plastic deformation during tensile loading after strain redistribution by creep deformation during compression hold period.

Figure 4 shows crack propagation behavior under 60 minutes tension hold. In the tension hold tests, the notch was elongated to the direction normal to stress axis. In this case, compressive strain was seemed to be accumulated in the center of the specimen. Crack initiation period in air was longer than that under compression hold. Crack propagation rate is also slower in the tension hold than that in the compression hold. The test in air was stopped at 2 000 cycles because the main crack initiated and propagated in a
different portion rather than the notch. It is considered that crack propagation resistance in tension hold is larger than that in compression hold due to residual compressive stress effect at the crack surface.

3.2 Crack propagation property

Crack growth curves in vacuum and air conditions are shown in Fig. 5. In the comparison of number of cycles to crack length in about 5 mm, the compression hold test is the shortest among the tests in air condition, and the PP and the tension hold tests are almost twice longer than the compression hold. In vacuum condition, the PP is twice longer than the compression hold and each test is longer than that in air condition under the same strain waveform. These tendencies agree with the difference of failure life under the creep-fatigue condition previously reported. The relationship between crack length and crack propagation rate is shown in Fig. 6. It can be seen clear difference of crack propagation property depending on environment and strain waveform. In the microcrack shorter than 1 mm in length, crack propagation rate in air condition is faster than that in vacuum condition in the comparison between the same strain waveform. However the difference of crack propagation rate became small for cracks larger than 1 mm. This fact suggests that oxidation effect on crack propagation rate is significant for the microcrack up to 1 mm in length but it becomes insignificant for larger cracks. Neumann carried out crack propagation tests on a copper and reported that although crack propagation rate accelerated by oxidation at low crack propagation rate region (below $10^{-3}$ mm/cycle), the oxidation effect disappeared beyond that crack propagation rate. In the comparison between waveforms, crack propagation rate under the compression hold is fastest both in vacuum and air conditions and the difference of propagation rate between the compression hold and the PP increase with increasing crack length due to accumulation of tensile strain at the center of the specimen. Acceleration of the crack propagation rate by creep during the tension hold could not be seen in air condition. Skelton addressed that crack propagation rate in the low cycle fatigue regime was expressed as

$$\frac{dl}{dn} = B l^m$$

(1)

where $l$ is crack length, $N$ is number of cycles, $B$ and $m$ are material constants, and acceleration of crack propagation rate by oxidation effect could be considered by changing the value of $B$. But the oxidation effect is not considered by changing the value of $B$ as obvious in Fig. 6. The value of $m$ took 0.65 for up to 1 mm in length and 1.5 for the crack length larger than 1 mm.

Crack propagation rate of Mod.9Cr-1Mo steel in air and vacuum conditions was correlated with cyclic $J$ integral range in Fig. 7. Cyclic $J$ integral range for a surface crack was calculated by:

$$AJ = \frac{\Delta \sigma \pi}{E} + f(n) \Delta \sigma \Delta \varepsilon_p a_{eq}$$

(2)

where $\Delta \sigma$ is stress range, $\Delta \varepsilon_p$ is plastic strain range, $E$ is elastic modules and $f(n)$ is given by Ref. (7),

$$f(n) = 3.85(n-1)/\sqrt{n} + \pi/n$$

(3)

where $n$ is inverse of work hardening exponent and takes 11.1 for this material. $a_{eq}$ is equivalent crack length given by

$$a_{eq} = Ma$$

(4)

where $M$ is correction factor for the stress intensity factor of a finite length crack subjected to stress $\sigma$. Although validation of this equation assured only for linear elastic regime, cyclic $J$ integral range for the surface crack was calculated with the assumption that the equation could be applied for plastic regime. The
crack shape was assumed to be half circle and \( M \) was derived from the table obtained by Raju-Newman. Data band obtained from high temperature crack propagation tests on low alloy steels with circular notch specimens was also indicated in the Fig. 7. The crack propagation data of the PP tests both in air and vacuum for the crack length larger than 1 mm located near the lower band and slope of the propagation curves is similar to other materials. Crack propagation rate in the compression hold was faster than that in the PP. Crack propagation rate for the crack length up to 1 mm in air was faster than that in vacuum due to oxidation effect. The same tendency can be seen in the relation between crack propagation rate and stress intensity factor on 316 stainless steel obtained from crack propagation tests under air and vacuum conditions by Sadananda.

4. Discussion

4.1 Crack propagation mechanism

Although as microscopic failure and deformation at the crack tip were not observed in this study, quantitative difference of crack propagation mechanism between in vacuum and in air environments can not be clarified, crack propagation mechanism in both environments will be discussed based on macroscopic observation results of the crack initiation and propagation behaviors and previous studies concerning the environmental effect on the crack propagation property.

Some studies on fatigue microcrack propagation in air environment have been reported. Neumann performed constant load amplitude fatigue tests at room temperature on the copper single crystal. He indicated that a shear type stage I crack initiated in air changed to opening mode in subsequent vacuum environment and the mode I crack propagation was explained by the macro slip model in which a crack grows by separation of slip planes and cross slip at conjugation slip planes. Kikukawa et al. studied deformation and growth mechanisms at the crack tip in vacuum by conducting fatigue tests at room temperature in a scanning electron microscope on 3% silicon iron and showed that crack propagation was caused by slip deformation at the crack tip depending on crack opening displacement and slip direction, and crack propagation rate in vacuum environment was slower than that in air due to rebonding at the crack tip. Pellox indicated that fatigue crack of the aluminum alloy in vacuum grew by repetition of reversible slip and rebonding at the crack tip and crack extension during one cycle in vacuum was smaller than that in air where a crack grew by repetition of irreversible slip. McEvilly carried out crack propagation tests on 304 and 316 stainless steel in vacuum and air environments at room temperature and reported that the crack tip blunting was easy to occur in vacuum environment, whereas the crack tip was relatively sharp and propagation rate was faster in air environment because of local strain concentration at the crack plane due to the irreversible slip.

Considering above discussions, crack growth mechanism in vacuum seems to be explained by the separation of slip planes and the cross slip although difficulty of the cross slip and contribution of the separation of slip planes to the crack growth depends on the stacking fault energy of the material. The rebonding at the crack plane occurred under compression loading in vacuum environment in all materials. In the air environment, the difference of the crack growth mechanism is identified as irreversible slip and obstruction of the rebonding by oxidation layer at the crack tip, though the mechanism of crack extension by the slip at the crack plane is the same as that in the vacuum environment. Because of the fact shown in Fig. 2 that the crack propagated in the direction normal to stress axis in the vacuum condition and many slip bands indicating slip direction could be seen, the fatigue crack growth mechanism of the steel in vacuum at high-temperature is seemed to be the same as above mentioned mechanism. On the other hand, the microcracks in air condition propagated on the maximum shear plane macroscopically. It would be supposed that the separation of the main slip plane caused by irreversible slip in the motion of the main slip system at the crack tip is easy to occur and the motion of the conjugation slip system is prevented due to significant oxidation effect.
The crack growth mechanism of the Mod.9Cr-1Mo steel at high-temperature fatigue condition in air and vacuum environments might be drawn schematically as in Fig. 8. In vacuum, the crack plane extends by the separation of the main slip plane and the conjugation slip plane under the tensile loading, then the crack closure and rebonding occur during the compressive loading as shown in Fig. 8(a). The opening of the crack plane and the separation of the slip plane repeat at following cycles. In air, a new crack plane is created by the separation of the slip plane due to the motion of the main slip system under the tensile loading. The amount of the separation in air may be larger than that in vacuum because of brittleness of the slip plane due to invasion of the oxygen from the crack tip. Although the main slip system move reverse direction, irreversible cracking remains by the oxidation. Then the crack extends in the vicinity of the maximum shear direction by repetition of the separation of the main slip plane and the oxidation. Actually, the crack growth mechanism in air could be more complicated combining the opening mode contributed by the separation of the main slip and the conjugation slip planes, and the shear mode mainly controlled by the separation of the main slip plane.

4.2 Strain hold effect on propagation rate

Failure life reduction under the compression hold might be caused by the fact that crack propagation rate under the compression hold was faster than that under the PP as shown in Fig. 5. On the other hand, crack propagation rate under the 60 minutes tension hold was almost the same as that under the PP. These features would be related to the deformation property of this steel. Although quantitative strain condition in the center of the specimen was not measured during the tests, strain accumulation with cycles under each waveform was measured from the replica films as initial length in 0.5 mm across the notch along the specimen axis. Change in the strain accumulation with number of cycles was shown in Fig. 9. Although the effect of crack opening on measurement accuracy of strain is not neglected for larger cracks, it can be seen qualitatively that accumulated strain under the compression hold increase with number of cycles both in air and vacuum environments, whereas the strain gradually accumulates in compressive direction under the tension hold and no significant change occurs under the PP. Conway et. al.\textsuperscript{19} carried out the strain hold test under radial strain control at 538°C with an hourglass type specimen on zirconium-copper alloy, and reported that the failure specimen under the tension hold showed necking and that under the compression hold showed bulging. Coffin\textsuperscript{16} explained such deformation instability in high-temperature
fatigue by the relation between strain distribution during loading stage and creep strain redistribution during hold period. In this study, although significant strain distribution as observed by Conway et al. did not occur because of axial strain control, strain accumulation to the tensile direction under the compression hold was considered to be occurred by the reason that strain at center of the specimen at maximum tensile strain might be larger than redistributed strain at the same portion during the maximum compression strain hold. Within the results of this study, the effect of environment on strain accumulation was not observed. Larger crack opening displacement in vacuum than that in air was caused by the difference of number of cycles at the same crack length. Based on the crack extension mechanism shown in Fig. 8, it should be considered that increase of tensile strain under the compression hold causes increase of the crack opening displacement accelerating the crack propagation rate, and decrease of the crack opening displacement under the tension hold suppresses crack propagation rate. Though creep strain at the crack tip during tension hold must be rate acceleration factor, it is interpreted that rate acceleration effect by creep was canceled by the compressive strain accumulation.

5. Conclusions

Crack propagation tests on Mod.9Cr–1Mo steel at high-temperature were carried out in vacuum and air conditions. The main results obtained in this study are summarized as follows.

1) The difference of the crack propagation behavior between in vacuum and in air environments was clearly observed. The fatigue crack propagated by the opening mode in vacuum and by shear mode in air regardless of the strain waveform.

2) The main reason of the acceleration of the fatigue crack propagation rate under the compression hold was found to be accumulation of tensile strain in the center of the specimen.

3) The crack propagation rate in the air was faster than that in vacuum for the crack up to 1 mm in length due to the influence of oxidation. But the difference became small for the crack larger than that.

References