Effect of vacuum environment on fatigue fracture surfaces of high strength steel

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1. Introduction

Recent studies have reported fatigue fractures in the very high cycle region, where the number of cycles is over $10^7$ cycles. These phenomena have been commonly observed in high strength materials such as high strength steel (Li, et al., 2015) and titanium alloys (Jha, et al., 2012) and are termed as Very High Cycle Fatigue (VHCF). In VHCF, fatigue fractures originate from inside materials (subsurface fracture) at stresses lower than the ordinary fatigue limit, which is defined as the fatigue limit of fractures originating from the surface of materials (surface fracture). However, the process of internal fatigue crack propagation is not yet fully understood, since their lengths are typically too short to be detected by conventional non-destructive inspection techniques.

It seems that internal cracks are shut off from air and propagate through a vacuum-like environment with negligible oxidation or gas absorption. On the basis of this concept, we conducted fatigue tests in vacuum environments on various high strength materials such as high strength steel SNCM439 (Shiina, et al., 2002), SCM435 (Nakamura and Oguma, 2011), and Ti-6Al-4V (Oguma and Nakamura, 2013). Our findings have revealed similarities in fracture surface features between subsurface fractures and surface fractures in vacuum, indicating that a vacuum-like environment inside internal cracks strongly affects the process of internal crack propagation. However, this consideration is based on only qualitative observations of fracture surfaces; quantitative assessment of the effects of...
environment on fracture surfaces is also needed.

In the present work, we investigated the effect of vacuum environment on fatigue fracture surfaces of SNCM439 quantitatively in order to clarify the effect of environment inside an internal crack on the crack propagation process. Uniaxial fatigue tests were performed in both air and vacuum environments and 3D fractography was then carried out to measure the surface roughness of fracture surfaces with 3D-SEM. The results were then used to clarify the process of internal crack propagation in terms of environmental effect focusing on air and vacuum.

2. Experimental Procedures

2.1 Experimental Material

The experimental material was high strength steel JIS-SNCM439. The chemical composition of the material is shown in Table 1. The supplied material was a 17-mm-diameter round bar heat treated by normalizing at 1133 K for 3.6 ks followed by air cooling, quenching at 1123 K for 3.6 ks followed by oil cooling, and tempering at 433 K for 7.2 ks followed by air cooling. The tempered martensite microstructure observed by SEM is shown in Fig. 1. The average prior γ grain size was 8.9 μm. Mechanical properties are listed in Table 2. The tensile strength was 2106 MPa and the Vickers hardness was 640 HV.

The material was lathe-turned into hourglass-type specimens having a parallel part of φ4.1×6mm. Specimen surfaces were ground with #120 to #2000 grit emery paper and then buff-polished with diamond abrasive. The diameter of the parallel part after polishing was φ4.0 mm.

Fig.1 Tempered martensite microstructure after heat treatment. Average prior γ grain size was 8.9 μm.

### Table 1  Chemical composition of experimental material SNCM439.  [mass\%]

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cu</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
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<td></td>
<td>0.40</td>
<td>0.22</td>
<td>0.78</td>
<td>0.022</td>
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<td>0.18</td>
<td>1.78</td>
<td>0.83</td>
<td>0.20</td>
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### Table 2  Mechanical properties of experimental material SNCM439.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
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<tr>
<td>Tensile strength [MPa]</td>
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</tr>
<tr>
<td>0.2% proof stress [MPa]</td>
<td>1368</td>
</tr>
<tr>
<td>Elongation [%]</td>
<td>11</td>
</tr>
<tr>
<td>Reduction of area [%]</td>
<td>46</td>
</tr>
<tr>
<td>Vickers hardness [HV]</td>
<td>640</td>
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</table>

2.2 Experimental Conditions

Uniaxial fatigue tests were carried out in both air and vacuum environments by using the ultra-high vacuum uniaxial fatigue testing machine shown in Fig. 2 (Nakamura, et al., 2010). The vacuum chamber of the machine is evacuated by using a dry scroll pump and a turbomolecular pump. The ultimate pressure in the chamber is 4.6×10⁻⁷Pa. The tests were carried out under sinusoidal waveform loading at a stress ratio of –1 and test frequencies of 30 or 60 Hz. The test pressure in the vacuum chamber during vacuum fatigue tests was 2×10⁻⁶ –1×10⁻⁵Pa.

3D fractography was conducted by using 3D-SEM (KEYENCE, VE-9800) and surface roughness was measured on the 3D images of fracture surfaces. 3D images were created from two images at the same position: one taken by tilting the sample stage at 5 degrees and the other taken normally without tilting.
3. Results and Discussion

3.1 Fatigue Test Results

The results of the fatigue tests are shown in Fig. 3. The vertical axis and horizontal axis indicate the stress amplitude $\sigma_a$ and the number of cycles to failure $N_f$, respectively. Solid circles (●) and open circles (○) indicate fatigue lives of surface fractures and subsurface fractures in air, respectively. Solid triangles (▲) and open triangles (△) indicate surface fractures and subsurface fractures in vacuum, respectively. The solid line is a least square line of surface fractures in air and the alternate long and short dashed line is that of surface fractures in vacuum. The dashed line, a least square line of subsurface fractures, was calculated from all data of subsurface fractures without distinction of test atmospheres since fatigue lives of subsurface fractures were independent of test environments, as discussed later.

Surface fractures occurred at higher stress level and shorter life time. In comparison with the surface fractures in air, the number of cycles to failure in vacuum was greater at the same stress amplitude. This tendency was strongly marked at lower stress amplitudes. For example, the fatigue life in vacuum was about twice as long as that in air at $\sigma_a = 1150$ MPa, while their number of cycles to failure was approximately the same at $\sigma_a = 1400$ MPa. In contrast, the number of cycles to failure of subsurface fracture was not affected by the test environments. This tendency is similar to our previous work, which clarified that the surface finishing method had no effect on the fatigue lives of subsurface fractures (Shiina, et al., 2001).

In air environment, the number of cycles to failure dramatically increased when the fracture mode changed from surface to subsurface. Therefore, the fatigue limit of surface fracture in air should be defined as the average value of the minimum stress at which surface fracture occurred and the maximum stress at which subsurface fracture occurred. The calculated fatigue limit was 1038 MPa, indicated by the horizontal part of the solid line in Fig. 3. In contrast, in vacuum environment, no such horizontal part was obtained since the range of number of cycles to surface fracture and that to subsurface fracture overlapped each other around $N_f = 10^7$ as a result of improved fatigue lives of surface fracture.
3.2 Fracture Surface Analyses

Fracture surfaces were observed to determine the fracture origin. Fig. 4 (a), (b), and (c) shows the typical fatigue fracture surfaces of surface fracture in air, surface fracture in vacuum, and subsurface fracture, respectively. These specimens were fatigued at stress amplitudes of 1200 MPa, 1150 MPa, and 1125 MPa and the number of cycles to failure was $2.18 \times 10^4$, $8.71 \times 10^4$, and $7.31 \times 10^4$, respectively. These fracture surfaces were chosen so that the numbers of cycles to failure of these specimens were comparable.

The arrows in Fig. 4 indicate the fracture origin sites. Fig. 5 (a), (b), and (c) shows magnified views of the fracture origin sites of Fig. 4 (a), (b), and (c), respectively. In Fig. 5 (a), a semi-circular hollow was observed at the fracture origin site. This hollow was a falling trace of $\text{Al}_2\text{O}_3$ as a result of Energy Dispersive x-ray Spectroscopy (EDS) analysis. In contrast, $\text{Al}_2\text{O}_3$ remained at the fracture origin site in Fig. 5 (b) and (c). SEM observations and EDS revealed that fracture origins of all specimens were non-metallic inclusions and that there were three types of fracture origin inclusions: $\text{Al}_2\text{O}_3$, MnS, and TiN. However, no difference of fatigue life was observed among these three types.

As shown in Fig. 5, bright stripe patterns were frequently observed in the vicinity of the origin of surface fractures in air, while no characteristic patterns were seen in the same regions of surface fractures in vacuum and subsurface fractures. Additionally, as shown in Fig. 5 (a), fracture surface morphology of surface fracture in air was edgy and exhibited asperity. In contrast, that of surface fracture in vacuum and subsurface fracture was smooth and flat as they had been squashed by contacts of the crack surfaces. Increasing tendencies of the roughness with crack propagation were recognized in all fracture surfaces. Fig. 6 (a), (b), and (c) shows the regions far away from the fracture origins corresponding to the same value of stress intensity factor range $\Delta K$ (calculated from Eqs. 1 and 2, discussed in 3.3). Rough morphology was commonly observed in all fracture surfaces and there were no major differences between test atmospheres and fracture modes. No striation pattern was detected in any of the fracture surfaces.

In subsurface fractures, many researchers have reported a unique fracture surface consisting of a fine concavo-convex pattern around the origin (Murakami, et al., 1999). This type of fracture surface is termed an Optically Dark Area (ODA). In the present work, however, no ODA was observed around the origin of subsurface fracture, including in Fig. 4 (c). Although several different models have been proposed for the formation of ODA (Murakami, et al., 1999, Sakai, et al., 2006, Shiozawa, et al., 2006, Nakamura, et al., 2010), it is commonly pointed out that a great number of cycles is crucial to obtain this peculiar fracture surface. It seems that the fatigue lives of the subsurface fracture were too short to form an ODA in the present work.
(a) Surface fracture in air environment. Stress amplitude was 1200 MPa and number of cycles to failure was 2.18×10⁴.

(b) Surface fracture in vacuum environment. Stress amplitude was 1150 MPa and number of cycles to failure was 8.71×10⁴.

(c) Subsurface fracture in vacuum environment. Stress amplitude was 1125 MPa, and number of cycles to failure was 7.31×10⁴.

Fig.4 Fracture surfaces.

(a) Fracture origin site of the specimen shown in Fig. 4 (a). A falling trace of the inclusion was observed.

(b) Fracture origin site of the specimen shown in Fig. 4 (b).

(c) Fracture origin site of the specimen shown in Fig. 4 (c).

Fig.5 Fracture origin site of specimen shown in Fig. 4. EDS analysis revealed that these fracture origin inclusions were Al₂O₃.

(a) Fracture surface region far away from the origin of the specimen shown in Fig. 4 (a).

(b) Fracture surface region far away from the origin of the specimen shown in Fig. 4 (b).

(c) Fracture surface region far away from the origin of the specimen shown in Fig. 4 (c).

Fig.6 Fracture surface region far away from the origin, corresponding to ΔK=20MPa√m. There were no major differences between test atmospheres and fracture modes.

3.3 Results of Fracture Surface Roughness Measurements

3D fractography was carried out on the fracture surfaces shown in Fig. 4. The fracture surface properties were quantitatively evaluated to estimate the effect of environment on fatigue crack propagation. Fracture surface roughness (arithmetic mean roughness) \( R_a \) was measured at various points and the relationship with stress intensity factor range \( \Delta K \) was investigated. \( \Delta K \) was calculated using Eq. 1 for surface fracture (Nishitani, et al., 1984) and Eq. 2 for
subsurface fracture (Sneddon, 1946). $F$ is a coefficient value and $a$ is the length from the origin in these equations. The crack shape was assumed to be semi-circular for surface fracture and to be circular for subsurface fracture.

$$\Delta K = F \Delta \sigma \sqrt{\pi a} \quad \text{(1)}$$

$$\Delta K = 2\Delta \sigma \frac{a}{\pi} \quad \text{(2)}$$

Fracture surface roughness $R_a$ was obtained from the fracture surface regions corresponding to $\Delta K=5$–$30\text{MPa}\sqrt{\text{m}}$. $R_a$ was calculated as the average value of the seven arithmetic mean roughnesses on seven roughness curves along the crack propagation direction at each $\Delta K$. Sampling length was 15.2 µm and the interval between each roughness curve was 2.5 µm.

Fig. 7 (a), (b), and (c) shows the relationships between $R_a$ and $\Delta K$ of the fracture surface shown in Fig. 4 (a) (surface fracture in air), (b) (surface fracture in vacuum), and (c) (subsurface fracture), respectively. The error bars in Fig. 7 indicate the maximum and minimum values at each $\Delta K$. Increasing trends of $R_a$ along with increasing $\Delta K$ were observed for all cases. $R_a$ of surface fracture in air environment varied more widely than others.

To compare the effect of environment on $R_a$, all data shown in Fig. 7 (a)–(c) are replotted in Fig. 8. Solid circles (●), solid triangles (▲), and open triangles (△) represent the $R_a$ of the fracture surfaces shown in Fig. 4 (a), (b), and (c), respectively. The solid line, the dash-dot line, and the dashed line were obtained by the least squares method.

$R_a$ of surface fracture in air is significantly greater than that in vacuum at lower $\Delta K$. For example, $R_a$ in air is more than twice as great as that in vacuum at $\Delta K=6\text{MPa}\sqrt{\text{m}}$. In contrast, at higher $\Delta K$, these $R_a$ are comparable with each other. As mentioned in 3.2, in the vicinity of the origin corresponding to lower $\Delta K$, edgy patterns were observed on the fracture surface of surface fracture in air while smooth morphology was observed on that in vacuum. There were also subtle differences between surface fracture in air and in vacuum far away from the origin corresponding to higher $\Delta K$. These qualitative properties of fracture surfaces are in good agreement with the measurement results of $R_a$, which indicates that fracture surface roughness can be used as a measure of the effect of environment on fracture surface. Additionally, several studies have reported that the small crack propagation process corresponding to low $\Delta K$ is more sensitive to the effects of environment (Duquette and Gell, 1971, Suresh, et al., 1981), and this tendency agrees with the present work.

The distribution trends of $R_a$ of subsurface fracture agreed quite well with that of surface fracture in vacuum, including the low $\Delta K$ region. This measurement result is consistent with the qualitative observation results of fracture surface shown in Fig. 4 (b) and (c) and indicates the similarity of fracture surface between surface fracture in vacuum and subsurface fracture quantitatively. These results show that the effects of vacuum environment and environment inside internal crack on fatigue crack propagation are almost the same. This leads us to conclude that the behavior of internal crack propagation can probably be estimated from surface crack propagation in vacuum environment.

(a) Surface fracture in air environment.
(b) Surface fracture in vacuum environment.
(c) Subsurface fracture.

Fig. 7 Relationship between fracture surface roughness and stress intensity factor range.
4. Conclusion

Uniaxial fatigue tests were carried out on high strength steel SNCM439 in air and vacuum to estimate the effect of the vacuum-like environment on the internal crack propagation process in very high cycle fatigue. The fracture surface roughness was measured by 3D fractography and the fracture surface properties were quantitatively compared. The main findings of this study are as follows:

1) In vacuum environment, the number of cycles to failure was higher than that in air environment.
2) The fracture surface roughness of surface fracture in air was larger than that in vacuum. This trend was strongly marked at lower $\Delta K$.
3) The relationship between $R_a$ and $\Delta K$ of subsurface fracture agreed quite well with that of surface fracture in vacuum. This indicates that the effects of vacuum environment and environment inside internal crack on fatigue crack propagation are almost the same.
4) The finding in 3) implies that the internal crack propagation process can be simulated by the surface crack propagation process in vacuum environment.

References

Nishitani, H. and Chen, D., Stress intensity factor for a semi-elliptic surface crack in a shaft under tension, Transactions