The influence of hydrogen on the very high cycle fatigue property and crack growth characteristics of bearing steels

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Abstract

In the present study, the growth rate of optically dark area (ODA) was investigated for two types of bearing steels using a fracture mechanics approach. Ultrasonic fatigue tests were performed for the specimens with hydrogen pre-charging followed by exposing in the atmosphere for a week in order to investigate the effect of hydrogen trapped by inclusions on the growth of ODA. Together with the constant amplitude loading test, two-step and repeated two-step variable amplitude loading tests were performed in order to determine the fatigue crack growth rate by beach marks. The results revealed that fatigue strengths of the both materials were reduced by the hydrogen pre-charging. The size of ODA increased with decreasing the stress amplitude at fracture so that the stress intensity factor range at the end of the ODA growth became a constant value ranging between 4.5 and 5.5 MPa \(\sqrt{a}\) independent of the materials and the fatigue lives. ODA formation behavior is enhanced by trapped hydrogen by inclusions, which reduces the fatigue strength in the very high cycle fatigue regime.

Keywords: Fatigue strength, Fatigue crack growth, Very high cycle regime, Optically dark area, Bearing steel

1. Introduction

Owing to the demand for longer fatigue lives in industrial equipment and components, very high cycle fatigue is becoming important and studied for a wide range of materials (Sakai et al. 2000, Shina et al. 2003, Ogawa et al., 2009). As high strength steels and titanium alloys exhibit interior originated fatigue fracture in the very high cycle regime, many research works has been done on the fracture mechanism. It has been clarified that most of the fatigue lives are spent for the formation of the specific region having characteristic fracture surface morphology, which is named optically dark area (ODA) by Murakami et al., (2000). They have been investigated growth of the ODA in relation to the effect of hydrogen trapped by nonmetallic inclusions (Murakami and Yamashita, 2014, Yamashita and Murakami, 2016). Remarkable characteristic of the ODA is that the similar fracture surface morphology can be observed for different material systems, i.e. high strength steels and titanium alloys, and that the morphology is found on the fracture surface tested in high vacuum environment (Oguma and Nakamura, 2013). Recent study by Yoshioka and Nakamura, (2016), using synchrotron radiation \(\mu\)CT imaging directly observed the initiation and growth of ODAs in Ti-6Al-4V alloy. This observation method enabled direct measurement of the growth rate of the ODA, which was between 10\(^{-14}\) and 10\(^{-11}\) m/cycles (Yoshinaka et al., 2017). One of the authors and Furuya have investigated beach mark method to measure the growth rate of ODA using variable amplitude loading by the ultrasonic fatigue test (Ishida et al., 2012, Ogawa et al., 2014, Furuya, 2015). The measured growth rates of ODA were between 10\(^{-14}\) and 10\(^{-13}\) m/cycles for bearing steel, SUJ2, and between 10\(^{-12}\) and 10\(^{-11}\) m/cycles for high strength steel, SCM440. As these growth rates are much less than lattice spacing per cycle, the ODA growth behavior may be discontinuous and sensitive to the microstructural factors.

In the present study, the growth rate of ODA was investigated for two types of bearing steels using a fracture
mechanics approach established by Ogawa et al., (2014). Ultrasonic fatigue tests were performed for the specimens with hydrogen pre-charging followed by exposing in the atmosphere for a week in order to investigate the effect of hydrogen trapped by inclusions on the growth of ODA. Together with the constant amplitude loading test, two-step and repeated two-step variable amplitude loading tests were performed in order to determine the fatigue crack growth rate by beach marks.

2. Materials and experimental procedures

The materials tested were high carbon chromium bearing steels, SUJ2 and 100Cr6, prescribed in the Japan Industrial Standard (JIS) and the Deutsche Industrie Normen (DIN), respectively, with similar chemical compositions and mechanical properties, which are presented in Table 1 and 2, respectively. Both materials were oil quenched from 850 °C for 50 min. and tempered at 180 °C for 240 min. The specimen geometry is shown in Fig. 1. The surfaces of the test specimens were polished with abrasive paper in axial direction with final grade #2000. Hydrogen was supplied to the specimens by cathode charging using a similar method to Hamada and Matsubara, (2006). The charging conditions are given in Table 3. H2SO4 and H3BO4 solutions were used for 100Cr6 and SUJ2, respectively. The charged specimens were polished again with the grade #1500 and kept in the atmosphere for more than a week in order to release the diffusible hydrogen and to investigate the effect of trapped hydrogen by inclusions (i.e. non-diffusible hydrogen) on the fatigue behavior. A week is enough long to release the diffusible hydrogen (Nakatani and Minoshima, 2009) and the non-diffusible hydrogen is in a stable condition (Nakatani and Minoshima, 2010). Finally, the specimens were polished again in axial direction with final grade #2000.

Reversed loading fatigue tests were performed at 20 kHz in laboratory air controlled at 24 °C with relative humidity of less than 40% using an ultrasonic fatigue testing machine in house, which consists of ultrasonic welder (BRANSON Co., CR-20), power supply (2000bcd) and our original software. In order to reduce the specimen heating by the ultrasonic oscillation, compressed cold air was blown on the specimen surface using vortex tube. Intermittent loading was conducted by monitoring the specimen temperature with a radiation thermometer. Typical loading condition under constant amplitude test is 120 ms loading with 360 ~ 1500 ms dwelling.

![Table 1 Chemical compositions [mass%].](image)

|          | C  | Si | Mn  | P  | S  | Ni | Cr  | Mo  | Cu  |
|----------|----|----|-----|----|----|----|-----|-----|-----|
| SUJ2     | 1.00 | 0.180 | 0.380 | 0.020 | 0.008 | 0.060 | 1.37 | 0.020 | 0.0900 |
| 100Cr6   | 0.936 | 0.237 | 0.329 | 0.0050 | 0.006 | 0.089 | 1.38 | 0.034 | 0.156 |
|          | Al | Ti [ppm] | O2 [ppm] | 0.0050 | 0.006 | 0.089 | 1.38 | 0.034 | 0.156 |
| SUJ2     | 0.011 | 15 | 5 | 0.011 | 0.01 | 1 | 0.001 | 0.9 |
| 100Cr6   | 0.028 | 20 | 3 | 0.011 | 0.01 | 1 | 0.001 | 0.9 |

![Table 2 Mechanical properties.](image)

|          | Young's Modulus [GPa] | Poisson's ratio | Vickers Hardness [HV] | Density [kg/m³] |
|----------|------------------------|----------------|-----------------------|-----------------|
| SUJ2     | 206                    | 0.28           | 816                   | 7830            |
| 100Cr6   | 206                    | 0.30           | 791                   | 7810            |

![Fig. 1 Specimen geometry of SUJ2 and 100Cr6 for the ultrasonic fatigue test. The resonant frequency of the longitudinal oscillation is 20 kHz.](image)
Together with the constant amplitude loading test, two-step and repeated two-step variable amplitude loading tests were performed. In the two-step tests, stress amplitude, \( \sigma_a = 1100 \) MPa (initial stress amplitude, \( \sigma_1 \)), was applied for the number of cycles of first step, \( N_1 = 1.0 \times 10^7 \) for SUJ2 and \( N_1 = 1.0 \times 10^5 \) or \( 1.0 \times 10^6 \) for 100Cr6, followed by the application of secondary stress amplitude, \( \sigma_2 = 1000 \sim 700 \) MPa, until fracture up to the cut off number of cycles of more than \( 3 \times 10^9 \) cycles. In the repeated two-step test for SUJ2, higher stress amplitude, \( \sigma_{H1} = 1092 \) MPa, and lower stress amplitude, \( \sigma_L = 841 \) MPa, were repeated until fracture. The number of cycles, \( N_i \), of the \( \sigma_0 \) and \( \sigma_L \) were \( N_{H1} = 1.0 \times 10^5 \) and \( N_{L} = 1.0 \times 10^4 \) cycles, respectively. Details of the test procedures are the same with our previous work by Ogawa et al., (2014).

![Table 3](image)

| Electrolysis Solution          | H₂SO₄ (0.05 mol/L) + CH₃N₃S (1.4g/L) | H₂BO₄ (31 g/L) + KCl (2.46 g/L) + CH₃N₃S (1.5g/L) |
|-------------------------------|--------------------------------------|--------------------------------------------------|
| Current Density               | 0.3 mA/cm²                           |                                                  |
| Charge Time                   | 20 h                                 |                                                  |

### 3. Results and discussion

#### 3.1 Fatigue strength

Figure 2 shows the fatigue life diagram under the constant amplitude and the two-step tests for SUJ2 (a) and 100Cr6 (b). The data for Lot 2 of SUJ2 are quoted from our previous work by Ogawa et al., (2014). Under the constant amplitude loading, the hydrogen charge reduced the fatigue strength for the both materials. A few specimens of SUJ2 were charged by a solution of H₂SO₄ instead of that of H₂BO₄, however, there was no difference in fatigue strength. For the hydrogen charged specimens, the fatigue strengths under constant amplitude loading of SUJ2 and 100Cr6 were approximately the same, while for the no charged specimens, those of SUJ2 are higher than those of 100Cr6. Under the two-step test, 100Cr6 was used to investigate the effects of test conditions of the \( N_i \) values and the period of hydrogen charging before or after the application of \( \sigma_1 \) on the fatigue strength. No significant influence was observed by the test conditions. For both materials with hydrogen charge, the number of cycles to failure, \( N_i \), increased with decreasing \( \sigma_0 \) over the test conditions.

![Figure 2](image)

**Fig. 2** Fatigue strength of SUJ2 (a) and 100Cr6 (b) showing the relationships of the number of cycles to failure, \( N_i \), and the stress amplitude, \( \sigma_0 \), for the constant amplitude loading test or the secondary stress amplitude, \( \sigma_2 \), for the two-step test. Note that the fatigue strength reduces by the hydrogen charge.
3.2 Fracture surface observation

Figure 3 presents the optical micrograph of fracture surface observed by scanning laser microscope (SLM, Lasertec Co., OPTELICS) for the hydrogen charged specimen tested under the lowest \( \sigma_2 \) for SUJ2. The fatigue fracture originated from the subsurface fish-eye with optically dark area (ODA) at the centre as shown in Fig. 3(a). Figure 3(b) is a magnified SLM micrograph, which demonstrates the ODA and the non-metallic inclusion at the centre. For some other specimens, however, the inclusion was unclear. The size of ODA was measured as a function of a square root of the area of ODA, \( \sqrt{\text{area}_{ODA}} \), and the corresponding stress intensity factor, \( \Delta K_{ODA} \), was calculated by the following equations, where Eq.(1) and Eq.(2) were for interior and surface origins, respectively.

\[
\Delta K_{ODA} = 0.5 \sigma_a \sqrt{\pi \sqrt{\text{area}_{ODA}}} \quad (1)
\]
\[
\Delta K_{ODA} = 0.65 \sigma_a \sqrt{\pi \sqrt{\text{area}_{ODA}}} \quad (2)
\]

Figure 4 shows the results of \( \sqrt{\text{area}_{ODA}} \) (a) and \( \Delta K_{ODA} \) (b) as a function of \( N_t \), where the ODAs are observed for all the test conditions. There are some exceptions, but the values of \( \Delta K_{ODA} \) were ranging between 4.5 and 5.5 MPa\( \sqrt{m} \) independent of the materials and the \( N_t \) values, as in our previous work (Ogawa et al., 2014), i.e. small crack problem is not significant in the present study. As \( \sqrt{\text{area}_{ODA}} \) of the hydrogen charged specimens became very large under the long life regime, fatigue strengths were reduced as demonstrated in Fig. 2.

Figure 5 presents the fracture surface of the hydrogen charged specimen tested under the repeated two-step test. Beach marks are clearly observed on the fish-eye, where the dark and bright fracture surfaces corresponded to the crack growth increments caused by \( \sigma_H \) and \( \sigma_L \), respectively. This behaviour was confirmed under a wide range of \( N_H/N_L \) conditions in our previous work (Ogawa et al., 2014). At the centre of the fish-eye, ODA and inclusion was observed, however, no beach mark was found in these areas even by an observation by atomic force microscope (AFM) with a much higher magnification than that of Fig. 5.

3.3 Fatigue crack growth rate

Fatigue crack growth characteristics are presented in Fig. 6, where the fatigue crack growth rate, \( da/dN \), is determined by the beach marks shown in Fig. 5 for the stress intensity factor range, \( \Delta K \), of larger than 6 MPa\( \sqrt{m} \). As a comparison, Fig. 6 also presents the growth characteristic of a long fatigue crack obtained by a CT specimen at a stress ratio, \( R \), of 0.1 (Nishizawa and Ogawa, 2005). The values of \( da/dN \) for \( \Delta K \) of smaller than 6 MPa\( \sqrt{m} \) are obtained by ODA sizes under the two-step tests according to the method proposed in our previous work (Ogawa et al., 2014). The ODA growth rates

![Fig. 3 Optically dark area (ODA) observed by laser microscope with low (a) and high (b) magnifications for the H2 charged specimen tested under \( \sigma_2 = 750 \) MPa of the two-step variable amplitude loading test broken at \( N_t = 2.0 \times 10^9 \) cycles.](image-url)
in the two-step tests could be estimated making some assumptions, as explained in the following: in the present study, the first step of $\sigma_1$ was applied up to a number of cycles that was somewhat smaller than the number of cycles to failure obtained in the constant amplitude test given in Fig. 2. At this point, we assumed, an ODA of similar size as those observed in the constant amplitude tests at $\sigma_1$ had been formed at a constant growth rate. In the subsequent second step, after applying a second defined stress $\sigma_2$, this ODA then grew up to the size observed at fracture. In other words, we considered the ODA at $\sigma_1$ as a pre-crack, which grew at the cyclic stress amplitude during the second step without any effects of stress history. Fig. 6 shows the $da/dN$ values calculated by the ODA growth increment divided by the number of cycles to failure during the second step, in which the number of cycles to form a fish-eye was ignored owing to their much smaller values. The values of $\Delta K$ were determined by the Eq. (2) with the $\sigma_2$ of the second step together with the average of initial and final sizes of the ODA. As can be seen, the growth rate of an ODA is hundred to ten thousand times smaller than that of a fish-eye. This great difference in the growth rates seems to be the most probable reason for the apparent change in the fracture surface morphology.

Figure 7 represents the relationships between the growth rate of ODA and the secondary stress amplitude, $\sigma_2$. The ODA growth rate increases with increasing $\sigma_2$ for each specimen, while it is assumed to be constant under the cyclic stress amplitude. Yoshinaka et al., (2017), observed that the growth rate of ODA increased with increasing $\Delta K$ in Ti-6Al-4V alloy using synchrotron radiation μCT imaging. These results suggest that the growth rate of ODA increased with increasing the crack driving forces of $\Delta K$ as well as $\sigma_2$ as commonly observed for the fatigue crack growth experiment based on the fracture mechanics. However, it is not possible to discuss which one is the controlling parameter of the growth rate of ODA based on the present test results. Corresponding to Fig. 2 for both SUJ2 and 100Cr6, ODA grew under the lower $\sigma_2$ for the hydrogen charged specimens than those for the no charged ones. In our previous work (Ogawa et al., 2014), it was estimated that the ODA morphology was formed by repeated fracture surface contact in a vacuum, i.e. in the interior of material, and was not determined by its growing mechanism which, however, caused a disintegration the original microstructural features. The present study indicates that this behavior is enhanced by trapped hydrogen by inclusions, which reduces the fatigue strength in the very high cycle fatigue regime. It has been demonstrated that the trapped hydrogen is released from the trap site by fatigue loading and that the hydrogen becomes diffusible and is supplied.

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Fig. 4  Size of ODA, $\sqrt{\text{area}_{ODA}}$, (a) and the corresponding stress intensity factor, $\Delta K_{ODA}$, (b) as a function of the number of cycles to failure, $N_t$, where the latter tend to be constant.
to the crack tip (Nakatani and Minoshima, 2010). Similar mechanism can be estimated for the growth of ODA.

4. Conclusions

In the present study, ultrasonic fatigue tests were performed for two types of bearing steels, SUJ2 and 100Cr6, with or without hydrogen charge. The results obtained are summarized as follows.
Fig. 7 Relationship between $da/dN$ and $\sigma$ determined from radii of ODAs. The $da/dN$ tends to increase with increasing $\sigma$, which suggest that the stress amplitude is the controlling parameter of the FCG of ODA.

(1) Fatigue strengths of the both materials reduced by the hydrogen pre-charging followed by exposing in the atmosphere for a week. This behavior is attributed to the effect of hydrogen trapped by inclusions.

(2) Size of ODA increased with decreasing the stress amplitude at fracture so that the stress intensity factor range at the end of the ODA growth became a constant value ranging between 4.5 and 5.5 MPa$\sqrt{m}$ independent of the materials and the fatigue lives.

(3) ODA formation behavior is enhanced by trapped hydrogen by inclusions, which reduces the fatigue strength in the very high cycle fatigue regime.

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