Fretting Wear of a Bearing Steel in Hydrogen Gas Environment Containing a Trace of Water

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Fretting wear tests on a bearing steel under gross slip condition were conducted in hydrogen and nitrogen gas environments containing water at 2 to 70 ppm using a new gas-tight chamber. Wear in hydrogen and nitrogen is sensitive to the water content of the gases and it increases as the water content increases. Water in these environmental gases reduces the coefficient of friction during the early cycles of experiments. Furthermore, exposure of the test specimens to high pressure hydrogen (40 MPa, 373 K, 200 hours) before the experiments enhances wear. These findings are consistent with the findings obtained in the authors’ previous study, wherein the water content should have been higher and a comparison between hydrogen and nitrogen was not carried out because of insufficient control of water content. In this paper it is shown that wear in hydrogen is slightly larger than in nitrogen.

Keywords: hydrogen, fretting, gross slip condition, bearing steel, water content, electric resistance method

1. Introduction

For the development of the hydrogen society, the effects of hydrogen on the friction and wear properties of candidate materials for machine components should be made clear to ensure and improve the reliabilities of them. For this purpose, integrated tribological studies in hydrogen gas environments have been conducted1-3). The authors thought that fretting wear tests under gross slip condition were suitable to study the effects of hydrogen because of frequent exposure of fresh surfaces to surrounding gas and launched a study on the effect of hydrogen on fretting wear of some steels4).

The findings the authors obtained in the previous study5 were: (1) the amount of wear and the insulation voltage decreases with an increase in the flow rate of the environment gas, (2) exposure of test specimen to high pressure hydrogen gas (40 MPa, 373 K, 200 hours) before the fretting wear test increases wear. Concerning the first finding, it was assumed that impurities in the surrounding gas decrease with the flow rate. For the second finding, multiple experiments were required because of insufficient control of impurities in the surrounding gas, and a comparison between wear in hydrogen and nitrogen was not performed because wear seem to be sensitive to impurities.

Water is a major residual gas in evacuated vacuum chambers5,6). This water contaminates hydrogen gas. The effect of water on fretting wear was investigated over several tens percent of relative humidity. Feng7) found that the fretting wear rate of mild steel decreases with increasing humidity. Goto8) investigated the influence of humidity during fretting of a range of pure metals, such as Cu, Ag, Fe and Cr, and found that friction and wear have their maxima at some critical value around 5 to 20% humidity depending on the material.

Meanwhile ISO/TS 14687-2:20089) specifies that the maximum concentration of water and oxygen in hydrogen gas for road vehicles should be 5 ppm. Because 100 ppm corresponds to 0.43% relative humidity at 293 K, the water content in the gases used for fretting wear tests have been too high, even though high purity gas is contaminated after charging in vehicles. Fukuda10) found that the ppm order of water in...
the surrounding gas has significant effect on the wear of a bearing steel during sliding. Endo\textsuperscript{11) investigated the effect of water vapor on the fretting fatigue of an aluminum alloy in air and found that water vapor content of several tens ppm accelerates the initiation and propagation of cracks compared with several ppm of water. They also found that fatigue strength deteriorates with an increase in the water content from several ppm to several tens of ppm.

These findings suggest that trace amounts of water have a significant effect on fretting wear in hydrogen. The water content of the environmental gasses used in the authors’ previous fretting experiments was assumed to be about 100 ppm\textsuperscript{11)11)}. Therefore we conducted fretting wear tests in hydrogen with controlled water content of 2 to 70 ppm.

2. Experimental method

Fretting wear tests in gross slip condition were conducted with a new test apparatus build according to the basic design by Fukuda\textsuperscript{12)}. This apparatus and a schematic diagram of loading and oscillating mechanism are shown in Fig. 1 and Fig. 2. The contact configuration is a point contact between a ball specimen and a plate specimen as explained in the previous study\textsuperscript{5)}. Ball clamping and loading mechanisms were designed to measure the friction force. A ball specimen of 4 mm in diameter was clamped on an end of a vertical beam which was connected orthogonally to a horizontal beam. The friction force was measured with strain gages seated on the vertical beam which was connected orthogonally to a horizontal beam. The plate specimen had a width of 10 mm, a length of 35 mm, and a thickness of 5 mm. It was clamped on and oscillated by a piezoelectric actuator.

The clamping and oscillating mechanism of the specimens were placed in a highly gas-tight chamber. A normal load was applied to the specimens through the orthogonally-connected beams. The tail end of the beams was pushed up by an end of loading lever. The loading lever was supported by a fulcrum outside the chamber and dead weight was loaded on the other end. The loading lever was sealed by a metallic bellows which deflect to follow the tilting of the loading lever.

An evacuating system with a scroll vacuum pump and a turbo molecular pump (TMP), as shown schematically in Fig. 3, was used to decrease the impurities in the chamber before the introduction of the environmental gas. After setting the ball and plate specimens, the air in the chamber was evacuated. The environmental gas was then fed continuously. The water content in the environmental gas was estimated from the chamber’s exhaust gas. This was measured by an electrostatic capacitance type dew point meter and was adjusted by varying the evacuation pressure (down to 1 \times 10^{-5} \text{ Pa}) and gas flow rate (50 - 1000 mL/min).

The experimental conditions are listed in Table 1. Hydrogen and nitrogen were used as environmental gases. The water content ranged from 1.5 to 70 ppm. In addition to the two gases, experiments in vacuum of the order of ten to the fourth power Pa and ten to the fifth power Pa were conducted for comparison. The specimen material for the ball and plate specimens was the bearing steel SUJ2 (JIS G 4805), which is equivalent to several tens of ppm.

![Fig. 1: The new test apparatus for fretting wear test in hydrogen gas environment](image1)

![Fig. 2: Loading and oscillating mechanism](image2)

![Fig. 3: Schematic diagram of the evacuation and gas supply system](image3)

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Test specimens and experimental conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Environment gas</strong></td>
<td>H\textsubscript{2}(5N), N\textsubscript{2}(5N) : atmospheric pressure</td>
</tr>
<tr>
<td></td>
<td>Vacuum : 8.6\times10^{-5} \text{ Pa}, 6.4\times10^{-4} \text{ Pa}</td>
</tr>
<tr>
<td><strong>Specimen configuration</strong></td>
<td>4 mm ball / plate (Ra 0.015 \text{ µm})</td>
</tr>
<tr>
<td><strong>Specimen material</strong></td>
<td>SUJ2 (Hv 865) / SUJ2 (Hv 695)</td>
</tr>
<tr>
<td><strong>Load (p\text{max} , a)</strong></td>
<td>5 N (1.45 \text{ GPa}, a=40.5 \text{ µm})</td>
</tr>
<tr>
<td><strong>Oscillation amplitude</strong></td>
<td>80 \text{µm}</td>
</tr>
<tr>
<td><strong>Oscillation frequency</strong></td>
<td>10 Hz</td>
</tr>
<tr>
<td><strong>Gas supply</strong></td>
<td>Continuous supply</td>
</tr>
<tr>
<td><strong>Total cycle number</strong></td>
<td>\text{10}^{10} (167 \text{ min.})</td>
</tr>
</tbody>
</table>

*SUJ2 \equiv \text{AISI 52100 bearing steel}


Table 2  Wear scars in hydrogen

<table>
<thead>
<tr>
<th>Water content</th>
<th>1.5ppm</th>
<th>11ppm</th>
<th>49ppm</th>
<th>70ppm</th>
<th>previous study* 100ppm (estimated)</th>
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<tbody>
<tr>
<td>ball sliding</td>
<td><img src="image1" alt="Image" /></td>
<td><img src="image2" alt="Image" /></td>
<td><img src="image3" alt="Image" /></td>
<td><img src="image4" alt="Image" /></td>
<td><img src="image5" alt="Image" /></td>
</tr>
<tr>
<td>plate sliding</td>
<td><img src="image6" alt="Image" /></td>
<td><img src="image7" alt="Image" /></td>
<td><img src="image8" alt="Image" /></td>
<td><img src="image9" alt="Image" /></td>
<td><img src="image10" alt="Image" /></td>
</tr>
</tbody>
</table>

* Load 1N

to AISI 52100. Before the experiments the test specimens were cleaned twice by ultrasonic cleansing with an organic solvent mixture of hexane 50% and acetone 50%. The treatment time was 10 minutes respectively.

The normal load applied was 5 N and the resultant Hertzian contact pressure and radius were 1.45 GPa and 40.5 μm, respectively. The applied load was 5 times of the previous experiments as a decrease in the wear was anticipated because of the reduced water content. The oscillation amplitude was 80 μm, which is two times longer than the diameter of Hertzian contact circle. The oscillation frequency was 10 Hz and the total oscillation number was 10⁵. The test duration was 167 min. All the experimental conditions except for the normal load were the same in the previous experiments.

In this study, the insulating voltage between the ball and plate specimens was measured by an electric resistance method as a characteristic index for the metallic contact. The parallel resistance and the voltage for complete separation were 1.11 kΩ and 150 mV, respectively.

3. Experimental results and discussion

3.1. Appearance of wear scars

Laser microscope images of wear scars on intact ball and plate specimens fretted in hydrogen gases with different water content are shown in Table 2. Because the wear scars in plate specimens are oval and the size of the wear scars in the direction of relative motion are larger than those of ball specimens, it is thought that gross slip occurred.

As for the effect of water content, wear scars are larger in high water content conditions than those in low water content conditions. The shapes of the wear scars also vary with water content while the wear scar boundaries in low water content conditions are irregular and it seems that severe adhesion occurred. In high water content conditions, the boundaries are smooth and the wear scars consist of dark and bright zones. These zones on one friction pair are in the relation of negative and positive; on the ball specimens the central area are

Table 3  Wear scars in nitrogen

<table>
<thead>
<tr>
<th>Water content</th>
<th>3.8ppm</th>
<th>50ppm</th>
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<tr>
<td>ball sliding</td>
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<tr>
<td>plate sliding</td>
<td><img src="image13" alt="Image" /></td>
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</table>

Table 4  Wear scars in vacuum

<table>
<thead>
<tr>
<th>Degree of vacuum</th>
<th>8.6 x 10⁻⁹ Pa</th>
<th>6.4 x 10⁻⁹ Pa</th>
</tr>
</thead>
<tbody>
<tr>
<td>ball sliding</td>
<td><img src="image15" alt="Image" /></td>
<td><img src="image16" alt="Image" /></td>
</tr>
<tr>
<td>plate sliding</td>
<td><img src="image17" alt="Image" /></td>
<td><img src="image18" alt="Image" /></td>
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</tbody>
</table>

Table 5  Wear scars of specimens exposed to high pressure hydrogen

<table>
<thead>
<tr>
<th>Water content</th>
<th>1.9ppm</th>
<th>3.4ppm</th>
</tr>
</thead>
<tbody>
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<td>ball sliding</td>
<td><img src="image19" alt="Image" /></td>
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</tr>
<tr>
<td>plate sliding</td>
<td><img src="image21" alt="Image" /></td>
<td><img src="image22" alt="Image" /></td>
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</table>
dark and the annular zones surrounding them are bright, while on the plate specimens the core is bright and the peripheral areas are dark. This type of wear scarring was observed in the previous experiments, where the water content was assumed as around 100 ppm, and is shown in the table. As stated in the previous study\(^4\), these dark and bright areas had been observed by Kayaba and Iwabuchi\(^13\). They stated that the dark area is covered by black oxides, and the black oxides act as an abrasive but protects the surface on which the oxides are produced.

In Table 3 the wear scars in nitrogen are shown. The effect of water content on wear scar size and appearance are the same as in the case of hydrogen. They are irregular and small in low water content condition, and smooth and large in high water content condition. The wear scarring observed in vacuum is shown in Table 4. Their appearance is noticeably different as much larger and very heavy adhesion can be seen. It can be assumed from the comparison of wear scars in hydrogen, nitrogen and in vacuum that hydrogen and nitrogen reduce adhesion as reported by Mishina\(^14\).

Wear scars on specimens exposed to high pressure hydrogen of 40 MPa and 373 K for 200 hr before the experiments and fretted in hydrogen are shown in Table 5. Their appearances are similar to those of intact specimens shown in Table 2, but they are slightly larger.

3.2. Wear scar area and wear volume
In Fig. 4 the areas of the wear scars are shown. These measurements were performed using a function of the laser microscope. The wear scar areas on the plates are larger than those on balls because the width is the same and the length of wear scars in the direction of the relative motion is longer than that of ball specimens. The three areas are consistent so that if the area in a ball is large the other two areas, wear scar in plate and the total area, are large. Therefore, the evaluation of wear scar area can be carried out using any of these areas.

The effects of gas species and water content on the wear scar area are shown in Fig. 5. The wear scar area is the total area of those on the ball and the plate. The effects of gas species and water content are clear. The wear scar is larger in hydrogen than in nitrogen, and the area of the wear scar increases as the water content

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**Fig. 4** Effects of gas species and water content on the wear scar area (Figures in the graphs indicate water content or degree of vacuum)

**Fig. 5** Effect of gas species and water content on wear scar area

**Fig. 6** Relationship between wear scar area and wear volume
increases both in hydrogen and nitrogen. Furthermore, the exposure of specimens to high pressure hydrogen before the experiment increases wear. These findings about the effects of water content and exposure to high pressure hydrogen are consistent with those obtained in the previous study\cite{4}, which was conducted in higher water content of probably around 100 ppm.

The measurement of the area of wear scar is convenient for comparison of wear in various gas environments, because it can be performed manually. However, wear should be evaluated intrinsically by weight change or by wear volume. Wear volume is essential for the estimation of the specific wear rate. Except for small amplitude, ranges from $10^{-9}$ to $10^{-7}$ mm$^2$/N. Iwabuchi\cite{16} also obtained values around 10$^{-8}$ mm$^2$/N for steel in air. The values obtained in this study are about a hundredth of these values. This is attributed to the low oxygen content in the environmental gases.

3.3. Coefficient of friction

The coefficient of friction during the fretting tests is shown in Fig. 7. In vacuum (Fig. 7(a)), the coefficient of friction is 0.4 and is almost constant during the experiment. Its average value is the highest and this high adhesion results in the largest wear scars, as shown in Table 4. This coefficient of friction is smaller than that expected from an investigation on the fretting wear of carbon steel in vacuum by Iwabuchi\cite{17}, in which a value of 3 was obtained at $10^{-3}$ Pa. However, Ramalhoa\cite{18} found a coefficient of friction of approximately 0.5 at $10^{-6}$ Pa for a bearing steel. Therefore, the value of 0.4 obtained in this study is not extraordinarily low.

In the case of hydrogen and for the intact specimens (Fig. 7(b)) with a low water content of 1.5 and 1.6 ppm, the initial coefficient of friction is as high as 0.5, and it gradually decreases to 0.3 at 20 $\times$ 10$^5$ cycles. After that an almost constant value is observed. At higher water content of 49 ppm and 70 ppm the coefficient friction remains 0.3 throughout. This suggests that the water in the environmental gas reduces the initial adhesion. The high adhesion in the early stage probably causes the irregular boundaries and the small area wear scars at the low water content, as shown in Table 2. The initial high adhesion at low water content is also observed in nitrogen, as shown in Fig. 7(c). Therefore, water decreases the coefficient of friction during the early cycles of experiments and increases the wear. On the other hand the exposure to high pressure hydrogen on

![Coefficient of friction during fretting tests (figures in the graphs indicate water content or degree of vacuum)](image-url)
the coefficient of friction is unclear (Fig. 7(d)), but wear decreased slightly as stated above.

3.4. Comparison with the previous study

In Fig. 8, a comparison of wear scar areas between the previous and current experiments are shown. The load was 1 N and water content was assumed to be about 100 ppm in the previous study. In the present study the load was 5 N. The wear scar areas in the present study both in hydrogen and nitrogen at the water content of several tens ppm are obviously larger than that in the previous experiment[5] corresponding to the increased load. However, the effect of higher load was compensated for by the low water content of several ppm. The effect of water content is larger than the effect of load or gas species. The impurities content difference is supported by the insulation voltage, as shown in Fig. 9; the voltage is quite low in the present study.

The effect of water content was the focus of this study. However, oxygen also can be dominant in fretting wear, and the oxygen content might have varied with the water content. As for the oxygen content in the environmental gas, Fukuda[6] found for the similar test apparatus that oxygen content is almost constant and about 0.8 ppm for the water content of less than 60 ppm. Therefore, in this study oxygen content can be assumed almost constant, and it is though that the effect of water content could be observed.

4. Conclusions

Fretting wear tests of a bearing steel under gross slip condition were conducted in hydrogen and nitrogen gas environments using a new test apparatus, in which the water content of the environment gases can be measured and controlled. The water content was ranged from 2 to 70 ppm considering that the ISO/TS for the hydrogen fuel specifies the maximum water content as 5 ppm. Findings obtained are as follows;

(1) Using the new test apparatus, the effects of the water content in the environmental gases and exposure of specimens to high pressure hydrogen were observed clearly. Also the comparison of wear between hydrogen and nitrogen could be carried out.

(2) Wear in hydrogen and nitrogen is sensitive to the water content of the gases and it increases as the water content increases over the water content range intended.

(3) Exposure of specimens to high pressure hydrogen increases wear.

(4) Wear in hydrogen is slightly larger than in nitrogen.

(5) Water in hydrogen and nitrogen reduced the coefficient of friction during the early cycles of experiment.

(6) These two findings (2) and (3) are consistent with the findings obtained in previous experiments which were conducted in the higher water content of around 100 ppm.

5. Acknowledgement

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6. References


