Fatigue Crack Growth Behavior of Micro-Sized Specimens Prepared from an Electroless Plated Ni-P Amorphous Alloy Thin Film

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Fatigue crack growth tests have been performed for micro-sized Ni-P amorphous alloy specimens to investigate the size effects on fatigue crack growth behavior of such micro-sized specimens. Two types of cantilever beam type micro-sized specimens with different breadth (B10 × W12 × L50 μm² and B30 × W12 × L50 μm²) were prepared from an electroless plated Ni-P amorphous alloy thin film by focused ion beam machining. Notches with a depth of 3 μm were introduced in the specimens. Fatigue crack growth tests were performed using a newly developed fatigue testing machine for micro-sized specimens in air at room temperature under constant load range and stress ratios of 0.1, 0.3 and 0.5. Striations were observed on the fatigue fracture surfaces and fatigue crack propagation rates were estimated by a careful measurement of the striation spacings. The fatigue crack growth rates at stress ratios of 0.3 and 0.5 were higher than that at 0.1. This suggests that crack closure may occur even in such micro-sized specimens. The fatigue crack growth resistance is also dependent on the specimen breadth. Shear lips which correspond to plane stress dominated region and a flat fatigue surface which corresponds to plane strain region are clearly observed on the fatigue surfaces, and the size of the plane strain region is different between the specimens with different breadth. This difference in stress state ahead of the crack may affect the crack growth behavior. The results obtained in this investigation are the first measurements of fatigue crack growth properties for micro-sized specimens and provide important information on reliability of actual micro systems and microelectromechanical devices.

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1. Introduction

Micro-sized machines and microelectromechanical systems (MEMS) are expected to be applied to bio-medical and micro-photonics devices such as micro-catheters for brain surgery and optical switches for electro-optical communications. The size of the components used in such MEMS devices is considered to be in the order of microns, and the mechanical properties of such micro-sized materials are considered to be different from those of bulk (ordinary sized) materials, as surface effects on the deformation mechanisms are prominent. Therefore, the evaluation of mechanical properties including elastic modulus, tensile strength, fracture toughness and fatigue properties are essential for practical applications of such MEMS devices.

There have been several studies which investigate the mechanical properties of small sized materials.¹⁻⁵ In particular, fatigue properties such as fatigue life and fatigue crack growth properties of micro-sized materials are extremely important to enable reliable design of actual MEMS devices, since the many micro-sized moving components are involved in such devices and the components are subjected to cyclic loading as they move. For example, the components used in micro-optical mirrors and switches are considered to experience extremely high number of cyclic load (over 10⁶ cycles). These micro components are usually prepared from thin films on substrates by surface micromachining technique, so fatigue tests have been attempted for such thin films on substrates and fatigue life of such materials have been evaluated.⁶⁻⁸ However, to date there has been an absence of fatigue data available for designing reliable and log-term durable MEMS devices. Particularly, there have been no data for fatigue crack growth properties which are important for damage tolerant design of actual devices. This is due to the lack of suitable fatigue testing equipment for micro-sized materials. In our previous investigation, we have designed a new fatigue testing machine for micro-sized specimens,⁹,¹⁰ and fatigue life curves have been obtained for micro-sized Ni-P amorphous alloys.¹¹ Amorphous alloy thin films deposited on substrates by sputtering or plating techniques are considered to be potential candidate materials for MEMS devices of their isotropic mechanical properties and high corrosion resistance. Therefore, it is important to clarify the fatigue properties of amorphous thin films. However, the fatigue properties of micro-sized amorphous specimens have not yet been studied except our previous investigation on the fatigue life of a micro-sized Ni-P amorphous alloy.¹¹ In the present study, fatigue crack growth tests have been performed for micro-sized specimens prepared from an electroless deposited Ni-P amorphous alloy thin film and the size effects on the fatigue crack growth behavior of micro-sized specimens have been investigated.

2. Experimental Procedure

2.1 Material and specimen preparation

The material used in this investigation was a Ni-11.5 mass%P amorphous thin film electroless plated on an Al-4.5 mass%Mg alloy. The thickness of the amorphous layer was 12 μm and that of the Al-4.5 mass%Mg alloy substrate was 0.79 mm, respectively. This material has been widely used for hard disk substrates. In micro-sized specimens, even small surface flaws may affect the mechanical properties. The surface roughness of this amorphous layer was less than one nm and hardly any defects are involved. Therefore, this material is considered to be one of the most suitable materials for establishing the methodol-
ogy of mechanical properties measurements for micro-sized specimens. A disk with a diameter of 3 mm was cut from the Ni-P/Al-Mg sheet by electro discharge machining. An amorphous layer was separated from the Al-Mg alloy substrate by dissolving the substrate with a NaOH aqueous solution. The amorphous thin film was fixed on a holder and two types of micro-cantilever beam specimens with dimensions of $B10 \times W12 \times L50 \mu m^2$ and $B30 \times W12 \times L50 \mu m^2$ were cut from the amorphous layer by focused ion beam machining. This specimen size is equivalent to approximately 1/1000 of ordinary sized bending specimens. Figures 1(a) and (b) show the specimens prepared by the above procedures. Notches with a depth of 3 µm were introduced into the specimens by focused ion beam machining. This notch depth is equivalent to $a/W = 0.25$, where $a$ is notch length and $W$ is specimen width. The width of the notch was 0.5 µm, and the notch radius is thus deduced to be 0.25 µm. The notch position was 10 µm away from the root of the specimen. The loading position is set at 40 µm from the root of the specimen.

### 2.2 Fatigue testing machine

Fatigue crack growth tests were carried out using a newly developed fatigue testing machine for micro-sized specimens. Figure 2 shows a block diagram of the fatigue testing machine. The testing machine consists of an actuator, a specimen holder, a load cell and a controller.

A magnetostrictive device is used as an actuator which imparts a small amount of displacement to a specimen. The magnetostrictive material used is TERFENOL-D (TbDyFe), which is able to produce displacements up to ±10 µm with an accuracy of 5 nm, and maximum response frequency of cyclic displacement is 100 Hz. So far, a nano-indentor type loading system has been applied for the measurement of mechanical properties in thin film specimens. The stiffness of such system is very low and is inadequate for applying cyclic loading. In this testing machine, the end of the actuator is connected to a metal shaft and a diamond tip of 5 µm in radius is attached to the other end of the shaft. This makes it possible to construct a high stiffness loading fixture. The displacement of the actuator is measured by a laser displacement meter with accuracy of 5 nm and the displacement signal is used as feedback control.

The micro-sized specimen is set in a specimen holder and the holder is placed on a load cell as shown in Fig. 2. This holder is interchangeable and is also used in the focused ion beam machine for machining the micro-sized specimens, in this fatigue testing machine and in a field emission-gun type scanning electron microscope. Therefore, machining, testing and observation processes are able to be performed without removing the specimen. This makes it easier to handle the micro-sized specimens. A CCD camera is set near the specimen holder to monitor the specimen appearance during fatigue testing.

Small amount of displacement is applied to the specimen through the diamond tip. The amount of load applied to the specimen is measured by a strain gauge type load cell with a load resolution of 10 µN that is set under the specimen. Load control is also an available option on this instrument. The horizontal location of the specimen stage and the load cell can be moved to adjust the loading position precisely by a
stepping motor at a translation resolution of 0.1 μm.
This testing machine is set up in a clean room with constant temperature and humidity to eliminate dust and the effect of temperature change during the measurement. The testing machine is also placed in a windscreen box to shield from the slight wind stream in the room. Further details of the testing machine are described in our previous papers.\textsuperscript{9,10}

2.3 Fatigue test
Fatigue crack growth tests were performed in air at room temperature. Fatigue tests were carried out at a frequency of 10 Hz and different stress ratios, \( R (R = P_{\text{min}}/P_{\text{max}}) \), where \( P_{\text{min}} \) is a minimum load and \( P_{\text{max}} \) is a maximum load applied over the fatigue cycle) of 0.1, 0.3 and 0.5 under constant load amplitude (\( \Delta P/2 \), where \( \Delta P = P_{\text{max}} - P_{\text{min}} \)) of 2 mN. This fatigue test condition was determined from our preliminary experiments. Although the crack length was not able to measure directly in this testing machine, the change in specimen compliance can be measured during fatigue tests. The initiation of crack growth was then determined by monitoring the specimen compliance. The fatigue surfaces after the tests were observed using a HITACHI S-4000 filed emission-gun type scanning electron microscope.

3. Results and Discussion

3.1 Fatigue crack growth behavior
Figure 3 shows a scanning electron micrograph of the specimen with a breadth, \( B \), of 10 μm after a fatigue crack growth test at a stress ratio of 0.5. A fatigue crack initiates from the notch root. The crack did not start to grow immediately after applying cyclic load and the crack started to grow after approximately 20000 cycles (this was confirmed by a compliance change of the specimen during fatigue test). This indicates that even the notch with root radius of only 0.25 μm is not regarded as a natural crack for micro-sized specimens. Figure 4 shows a high magnification of the center region of the fracture surface in Fig. 3. The upper part of Fig. 4 is a notch and the bottom region is a final fracture surface featured by a vein pattern which is observed on monotonic fracture surface in amorphous alloys.\textsuperscript{12} The region between the notch and the final fractured region is thus a fatigue surface. The fatigue surface is relatively flat and very fine equispaced markings are clearly observed on the fatigue surface. The spacing between these markings is approximately 30 nm at near the notch and 80 nm at near the final fractured region, and increased with the crack extension. It is not certain whether these markings correspond to striations, but these markings are aligned perpendicular to the crack growth direction and were not observed on the fracture surface by static bending tests,\textsuperscript{13} so these markings are deduced to be striations. Such striations have also been observed on fatigue surfaces of metallic glass bulk specimens.\textsuperscript{14} The formation of striations suggests that the crack has propagated by cyclic plastic deformation at the crack tip (i.e., blunting and resharpening of crack tip). Actually, shear bands which are considered to be formed by plastic deformation were observed on the side surface of the specimen near the crack tip. Consequently, the fatigue crack growth seems to be based on cyclic plastic deformation at the crack tip even in micro-sized amorphous alloys.

3.2 Effect of stress ratio on fatigue crack growth
If the spacing between the striations on the fatigue surface is assumed to be equivalent to the fatigue crack growth rate for the specimens, fatigue crack growth resistance curves can be obtained from the measurement of the striation spacings. Careful measurements of the striation spacings were made and fatigue crack growth rates (\( da/dN \)) as a function of applied stress intensity factor range (\( \Delta K = K_{\text{max}} - K_{\text{min}} \)) were obtained. Stress intensity factor (\( K \)) is calculated from the following equation for a single edge notched cantilever beam specimen\textsuperscript{15}:

\[
K_1 = \sigma_0\sqrt{a} f(a/W)
\]

where \( a \) is a crack length, \( W \) is a specimen width and \( f(a/W) \) is a dimensionless polynomial function and is given by

\[
f(a/W) = 1.112 + 1.40(a/W) + 7.33(a/W)^2
\]
and \( \sigma_0 \) is expressed as

\[
\sigma_0 = \frac{6PL}{W^2B}
\]

where, \( P \) is an applied load, \( L \) is a distance from the notch to the loading position (30 \( \mu \)m for the specimens in this investigation) and \( B \) is a specimen breadth. The crack length, \( a \), is measured from scanning electron micrographs of the fatigue surfaces.

Figure 5 shows the fatigue crack growth resistance curves for the specimens with a \( B \) of 10 \( \mu \)m at stress ratios of 0.1, 0.3 and 0.5. As once a crack started to grow, the specimen failed after only several thousand cycles for the micro-sized specimens, so \( \Delta K_0 \) (a stress intensity range at which a crack starts to grow) was not able to be determined. Also, the number of data points is only three for one specimen, but this is due to the difficulty of the measurement of striation spacings since the spacing is only between 20–70 nm. It is not certain whether a Paris-Erdogan relationship \((\,d\sigma/dN = A\Delta K^m,\) where \( A \) and \( m \) are material constants) is applicable for these data, but the fatigue crack growth rates at stress ratios of 0.3 and 0.5 are higher than that at 0.1 at a given value of \( \Delta K \). Generally, a decrease in fatigue crack growth rate at a low stress ratio can be explained by crack closure effects for ordinary sized specimens. This suggests that crack closure effects may occur even in such micro-sized specimens and may affect the fatigue crack growth behavior. The fatigue surface is relatively flat as shown in Fig. 4, so this crack closure is deduced to be a plasticity-induced crack closure.

### 3.3 Effect of specimen breadth on fatigue crack growth

In order to investigate the effect of stress state ahead of the crack tip on the fatigue crack growth behavior of micro-sized specimens, fatigue crack growth tests for specimens with different breadth \((B)\) were carried out. Figure 6 shows a fatigue crack growth resistance curves at a stress ratio of 0.5, at which no closure effect is assumed, for the specimens with \( B \) of 10 and 30 \( \mu \)m, respectively. The fatigue crack growth rate with \( B \) of 10 \( \mu \)m is higher than that with \( B \) of 30 \( \mu \)m.

Figures 7(a) and (b) show scanning electron micrographs of fatigue fracture surfaces of the specimens with different breadth. Slant fractured regions which appear to be shear lips are observed near the side surface of the specimens, and the width of the region near final fracture, where \( K_{\text{max}} \) corresponds to approximately 5 MPa\(\sqrt{m}\), is approximately 2 \( \mu \)m and this size seems to be the same for both the specimens. If these are shear lips, these areas should be plane stress dominated regions. The width of shear lip is comparable to the size of plane stress plastic zone, \( r_s \) \((r_s = (K_{\text{max}}/\sigma_y)^2/\pi, \) where \( \sigma_y \) is a yield stress of the specimen\). The calculated value of \( r_s \) \((i.e., \) shear lip width\) at \( K_{\text{max}} \) of 5 MPa\(\sqrt{m}\) is 2.4 \( \mu \)m and this size is approximately same as the slant fractured region in Figs. 7(a) and (b) \((\) the value of \( \sigma_y \) = 1.8 GPa in Ni–24 at\%P was quoted\) in this calculation). Therefore, these slant fractured zones are plane stress dominated region and the flat region corresponds to plane strain dominated one. It is very interesting that there exists plane strain region even in such micro-sized specimens. The effect of specimen thickness on fatigue crack growth behavior has been also observed for ordinary sized specimens. Thus, a decrease in crack growth rate with increase in specimen breadth may be due to the difference in stress state ahead of the crack.

### 3.4 Size effect on fatigue crack growth

As shown in Fig. 5, the effect of stress ratio on fatigue crack growth, which is deduced to be associated with a crack closure effect, is observed even for micro-sized specimens. The length of crack extended by fatigue loading in the micro-sized specimens used in this investigation was only 2–3 \( \mu \)m as shown in Fig. 4. Generally, crack closure effects are less pronounced for short cracks with length of less than 100 \( \mu \)m.
and almost no closure effects are observed for extremely short cracks with length of in the order of microns. However, these observations have been obtained for short cracks in ordinary-sized specimens. In contrast, the size of the specimen used in this investigation is three dimensionally small, so the normalized crack length is sufficiently long compared to the specimen size (actually, $a/W$ is approximately over 0.5 at final fracture). Therefore, the crack length of 2–3 μm should be regarded as a long crack for this size of specimen, so the closure effects are deduced to be pronounced even for micro-sized specimens.

The final fatigue fracture occurred at the crack growth rate less than $10^{-7}$ m/cycles. This crack growth rate is much lower compared to that of ordinary-sized specimens. This means that once a fatigue crack starts to grow then the fatigue fracture occurs only after several thousands cycles (this was confirmed in the actual fatigue crack growth tests for the micro-sized specimens in this investigation). Therefore, the fatigue life of micro-sized specimens is dominated by a crack initiation. This also suggests that even micro-sized surface flaw may be an initiation site of fatigue crack growth and this will shorten the fatigue life of micro-sized specimens.

Plane stress and plane strain dominated regions were clearly observed on the fatigue surfaces of the micro-sized specimens as shown in Fig. 7, and their sizes were consistent with those estimated by fracture mechanics calculations. This indicates that fracture mechanics is still valid for such micro-sized specimens, and that a damage tolerant design is applicable for actual micro-sized machine and MEMS devices.

In this investigation, the fatigue crack growth properties for micro-sized specimen was able to be measured for the first time. However, the detail of size effects on fatigue crack growth behavior is still unclear. Further investigation is required to quantify the size effect on fatigue mechanisms in micro-sized specimens.

4. Conclusions

Fatigue crack growth tests have been performed for micro-sized cantilever beam type specimens prepared from an electroless plated Ni–P amorphous alloy thin film to investigate the size effects on fatigue crack growth behavior of such micro-sized specimens.

Stiations were observed on the fatigue fracture surfaces and fatigue crack propagation rates were estimated by a careful measurement of the striation spacing. The fatigue crack growth rates at stress ratios of 0.3 and 0.5 were higher than that at 0.1. This suggests that crack closure may occur even in such micro-sized specimens. The fatigue crack growth resistance is also dependent on the specimen breadth. Shear lips which correspond to plane stress dominated region and flat fatigue surface which correspond to plane strain region are clearly observed on the fatigue surfaces, and the size of the plane strain region is different in those specimens. This difference in stress state ahead of the crack may affect the crack growth behavior. The results obtained in this investigation are the first measurements of fatigue crack growth properties for micro-sized specimens and provide basic guidelines for design of actual micro-sized machine and MEMS devices.

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