Transactions of the Materials Research Society of Japan 28 [3] 695-698 (2003)

Fracture Toughness Tests of Thin Films for MEMS Applications

Satoru Koyama, Katsuhisa Nakai, Kazuki Takashima*and Yakichi Higo*
Graduate student, Tokyo Institute of Technology, 4259, Nagatsuta-Cho, Midori-Ku, Yokohama, 226-8503, Japan
e-mail: skoyama@ames.pi.titech.ac.jp, knakai@ames.pi.titech.ac.jp
*P & I Lab., Tokyo Institute of Technology
e-mail: takashik@pi.titech.ac.jp, yhigo@pi.titech.ac.jp

Thin films prepared from smart materials are expected to be applied to MEMS devices. The evaluation of fracture toughness of micro components prepared from such thin films is thus important to design reliable and durable MEMS devices. In this investigation, we have developed a fracture toughness testing method for micro components used in MEMS devices. The material used was a Ni-P amorphous alloy thin film (12 μm in thickness). Cantilever beam type specimens with dimensions of 10 x 12 x 50 μm were prepared by focused ion beam machining, and a notch (6 μm in depth and a notch root radius of 0.5 μm) was introduced into the specimen. A fatigue pre-crack was also introduced ahead of the notch by a far-field cyclic compression technique. The introduction of fatigue pre-crack and fracture toughness tests were successfully carried out using a mechanical testing machine for micro-sized specimens. A valid K_{IC} value was not obtained as the criteria of plane strain requirements were not satisfied for this specimen size, but this technique is found to be a promising method for measuring fracture toughness of thin films for MEMS applications.
Key words: MEMS, Thin film, Fracture toughness, Fatigue pre-crack, Micro-sized specimen

1. INTRODUCTION

Micromachines and microelectromechanical systems (MEMS) are expected to be applied to micro-photonics devices such as optical switches for electro-optical communication and micro-medical devices such as stent and catheter for brain surgery. In such micro devices, components are often prepared from thin films and the size of component is considered to be in the order of microns. The mechanical properties of such micro-components are thus important for practical applications. These thin films are usually prepared by deposition or sputtering technique, and micro-sized defects are often introduced in the films during their processing. In ordinary-sized materials, micro-sized defects do not affect the strength, but in thin films, micro-sized defects greatly affect the strength. Therefore, the evaluation of fracture toughness of such thin films is essential to design reliable and durable MEMS devices.

For ordinary-sized specimens, fracture toughness values are measured according to the ASTM standard. However, for micro-sized specimens, there are no such standards, so the fracture toughness measurements have been carried out by their own methods [1, 2]. This indicates the comparison of fracture toughness value has been difficult. Therefore, the development of standardized fracture toughness testing method for MEMS devices is urgent.

To date, we have developed a mechanical testing machine that can apply both static and cyclic loads to micro-sized specimens [3], and bending fatigue crack growth and fracture toughness tests of micro-sized cantilever specimens have been examined [4-8]. As a result, we found that a fatigue pre-crack has to be introduced into a micro-sized specimen for measuring fracture toughness. However, a fatigue fracture of micro-sized specimen occurred within 1000 cycles after the fatigue crack started to grow. This indicates that the control of fatigue pre-crack length is extremely difficult by bending fatigue, and a fatigue pre-cracking technique suitable for micro-sized specimen is required.

In this study, we have developed a fracture toughness testing method, including introduction of fatigue pre-crack, for micro-sized specimens prepared from thin film.

2. EXPERIMENTAL PROCEDURE

2.1 Material

The material used in this study was a Ni-11.5 wt%P amorphous alloy thin film that was electroless-plated on an Al-4.5 wt%Mg alloy. As amorphous alloys have isotropic mechanical properties and high corrosion resistance, it is expected to be one of the candidate materials for MEMS/micromachines applications. This material has been used for hard disk substrates and is mass-produced with uniform quality, and the surface roughness is in the order of nano-meters. In addition, it has been confirmed that there are no surface defects in the material. From these features, this material is considered to be one of the suitable materials for the development of the methodology of micro-sized testing. This material was also used in our previous studies [4-8].

2.2 Specimen Preparation

A disk with a diameter of 3 mm was cut from the Ni-P/Al-Mg plate 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 micro-cantilever beam type specimen was prepared by focused ion beam machining as shown in

Fig. 1. The breadth (*B*), width (*W*) and length of specimen were 10 μm, 12 μm and 50 μm, respectively. This specimen size is equivalent to approximately 1/1000 of ordinary sized bending specimen. Notches with a depth of 3 μm and a root radius of 0.5 μm were introduced into the specimens by focused ion beam machining. The notch position was 10 μm from the fixed end of the specimen. The loading position was set at 30 μm from the notch as shown in Fig. 1.

2.3 Mechanical Testing Machine for Micro-sized Specimens

Introduction of fatigue pre-crack and fracture toughness tests were carried out using a mechanical testing machine for micro-sized specimens (MFT-2000), which we have developed in our precious investigation [3]. This mechanical testing machine can apply static and cyclic loading to micro-sized specimens. The specimen can be positioned with an accuracy of 0.1 μm. The displacement resolution and load resolution are 0.005 μm and 10 μN, respectively. The details of the testing machine are described in our previous paper [3].

2.4 Introduction of Fatigue Pre-crack

In this study, we have noted the far-field cyclic compression technique. This method has been applied to introduce a pre-crack into brittle materials including ceramics and intermetallics, and a fatigue crack will arrest completely after the crack distance exceeds residual tension stress field at the notch tip, which was formed by the application of compression load [9, 10].

Far-field cyclic compression load was applied to the specimen as schematically shown in Fig.2 (a). Cyclic compression was carried out under a maximum compressive load of -60mN, and a stress ratio of 10 at room temperature (20 °C) and in laboratory air. The cyclic frequency was set to be 10 Hz.

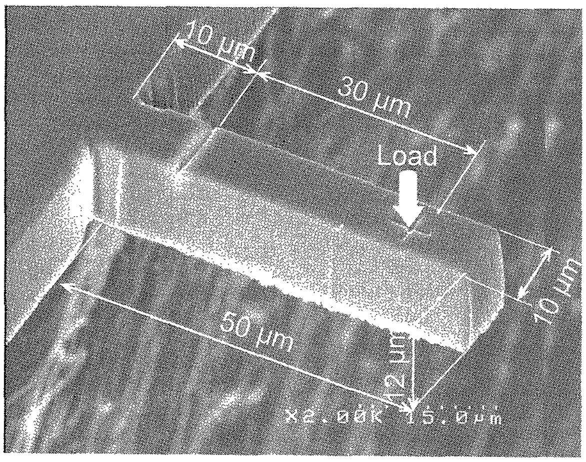

Fig. 1 Scanning electron micrograph of micro-sized cantilever beam specimen for fracture toughness test.

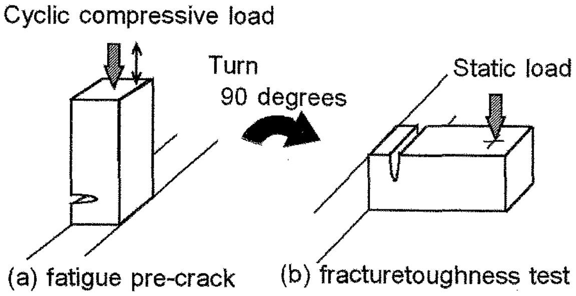

Fig. 2 (a) Introduction of fatigue pre-crack by far-field cyclic compression technique.
(b) Fracture toughness test by bending.

2.5 The Fracture Toughness Test

Fracture toughness tests were conducted as schematically shown in Fig. 2 (b). The specimen was turned 90° and static load was applied at the loading point shown in Fig. 1. The test was also carried out for specimen with a notch only. After fracture tests, fracture surfaces were examined using a scanning electron microscope (HITACHI-S4500).

3. RESULTS AND DISCUSSION

Figure 3 shows a scanning electron micrograph of specimen appearance after fracture test. A crack initiates from the notch root and this indicates the fracture test was successfully performed for such micro-sized specimens. Figures 4(a) and (b) show fracture surfaces of notched only and cyclically compressed (10000 cycles) specimens, respectively. For notch only specimen, vein pattern, which is often observed on a monotonic fracture surface in amorphous alloys, is observed near the notch root as shown in Fig. 4(a). In contrast, for the cyclically compressed specimen, a flat region is observed clearly between the notch and the vein pattern as shown in Fig. 4(b). In addition, markings aligned parallel to the crack growth direction are observed on the flat region. These markings are quite similar to the striations observed on the fatigue surface of the micro-sized specimens by cyclic bending tests [5, 6]. This indicates that a fatigue pre-crack was introduced successfully by far-field cyclic compression technique for micro-sized specimens. The observed fatigue pre-crack length is approximately 0.2 μm. This is approximately the same for the specimen cyclically compressed over 10000

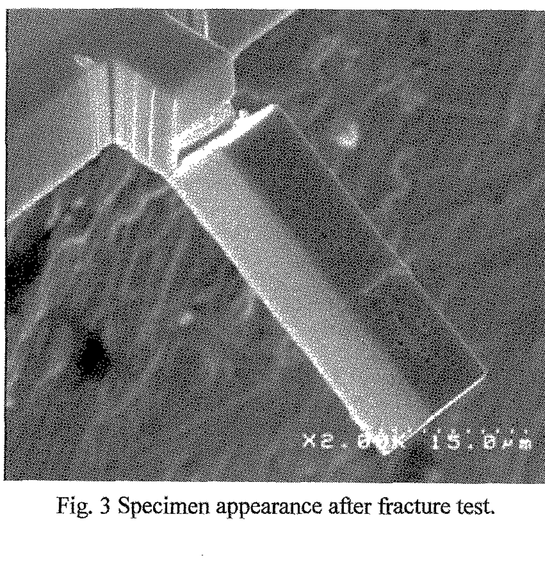

Fig. 3 Specimen appearance after fracture test.

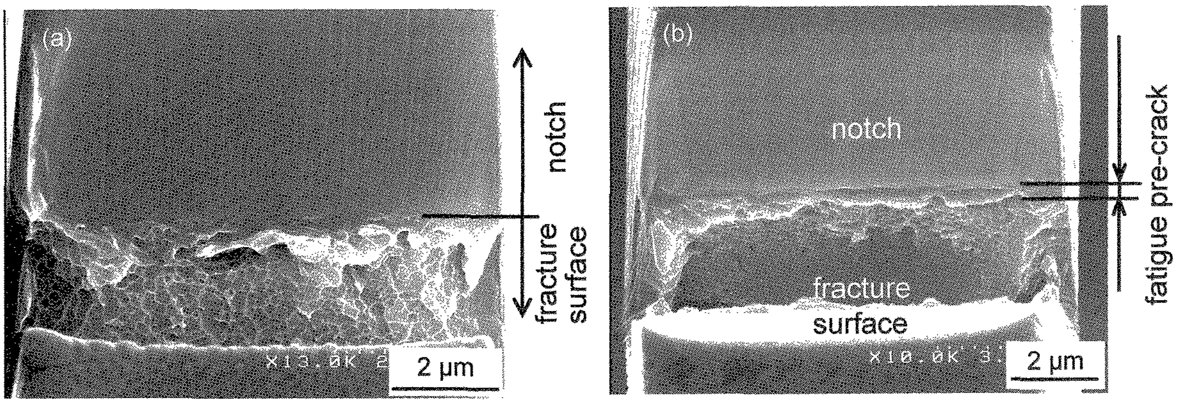

Fig. 4 Fracture surfaces after fracture toughness tests. (a) Specimen with a notch only. (b) Far-field cyclic compressed specimen (10000 cycles).

cycles. This suggests that the crack arrests after 10000 cycles. The fatigue crack length under far field cyclic compression (a_f) has been estimated to be equivalent to the following equation [9].

$$a_f \approx \frac{1}{2\pi}\left(\frac{K_{max}}{\sigma_{ys}}\right)^2 \tag{1}$$

where, K_{max} is a maximum stress intensity at the crack tip and σ_{ys} is a yield stress of the material (1.8 GPa for Ni-P amorphous alloy). This value is calculated to be 0.23 μm in this case. This is consistent with fatigue pre-crack length observed on the fracture surface (Fig. 4(b)).

Figure 5 shows typical load-displacement curves for the specimen with a notch only and the specimen after 10000 cyclic compression. The fracture load of the cyclically compressed specimen is lower than that of the notch only specimen. This result is the same as those obtained for the specimens with fatigue pre-cracking by bending fatigue [7, 8].

As crack opening displacement was not able to be measured for these specimens, the crack initiation load

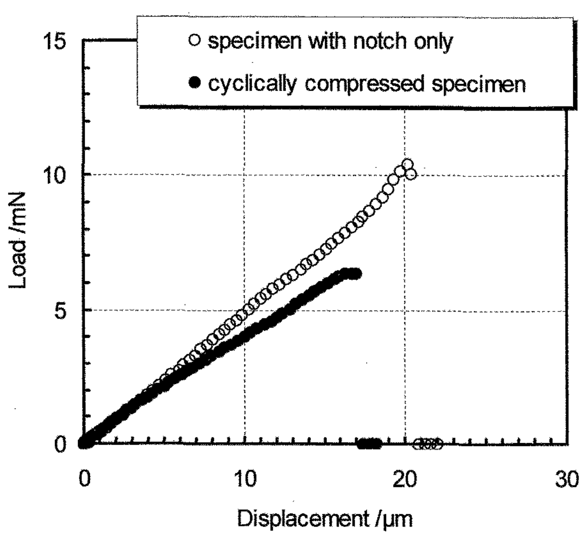

Fig.5 Load-displacement curves for micro-sized specimens with a notch only and with a fatigue pre-crack introduced by cyclic compression.

was not able to be determined. The maximum load was then assumed to be a crack initiation load and this load was used to calculate fracture toughness value. The fracture toughness value was calculated using the following equations for stress intensity (K) for notched cantilever beam [11],

$$K \approx \frac{6PL}{W^2B}\sqrt{\pi a}F_1(a/W) \tag{2}$$

where

$$F_1 = 1.22 - 1.40 + (a/W) + 7.33(a/W)^2 - 13.08(a/W)^3 + 14.0(a/W)^4. \tag{3}$$

In equation (2), a, P and L are total crack length, crack initiation load and distance between the loading point and notch position, respectively. The calculated provisional fracture toughness value (K_Q) for the specimen with a fatigue pre-crack is 5.4 MPam$^{1/2}$ and this is approximately the same as the fatigue pre-cracked specimen by bending fatigue (4.8 MPam$^{1/2}$) [12]. This indicates that the technique developed in this investigation is very effective for measuring fracture toughness for micro-sized specimens. However, this value is not a valid plane strain fracture toughness value (K_{IC}), since the criteria of plane strain requirements ($a,W\text{-}a,B>2.5(K_Q/\sigma_{ys})^2$) were not satisfied for this specimen size. As the plane strain requirements are determined by K and σ_{ys}, it is difficult for micro-sized specimens to satisfy these requirements. Consequently, other criterion such as J-integral might be required to evaluate fracture toughness of such micro-sized specimens.

4. CONCLUSION

Fracture toughness tests were performed on micro-sized Ni-P amorphous alloy specimens. The far-field cyclic compression technique was applied for introducing a fatigue pre-crack into micro-sized specimen, and fatigue pre-crack was successfully introduced into micro-sized specimen using this technique. The calculated provisional fracture toughness K_Q for the specimen with a fatigue pre-crack by cyclic compression was approximately the same for the specimen with a fatigue pre-crack introduced by bending fatigue. This suggests that the fracture toughness measurement method developed in this investigation is promising for measuring fracture toughness of thin films for MEMS applications.

REFERENCES

[1] H. Kahn, N. Tayebi, R. Ballarini, R. L. Mullen and A. H. Heuer, *R. Soc. Lond. A*, **455**, 3807-3823 (1999).

[2] T. Tuchiya and J. Sakata, *ASTM STP*, **1413**, 214-228 (2001).

[3] Y. Higo, K. Takashima, M. Shimojo, S. Sugiura, B. Pfister and M. V. Swain, *Mat. Res. Soc. Symp. Proc.*, **605**, 241-246 (2000).

[4] S. Maekawa, K. Takashima, M. Shimojo, Y. Higo, S. Sugiura, B. Pfister and M. V. Swain, *Jpn. J. Appl. Phys.*, **38**, 7194-7198 (1999).

[5] K. Takashima, Y. Higo, S. Sugiura and M. Shimojo, *Mat. Trans.*, **42**, 68-73 (2001).

[6] K. Takashima, M. Shimojo, Y. Higo and M. V. Swain, *ASTM STP*, **1413**, 52-61 (2001).

[7] Y. Ichikawa, S. Maekawa, K. Takashima, M. Shimojo, Y. Higo and M. V. Swain, *MRS Symp. Proc.*, **605**, 273-278 (2000).

[8] K. Takashima, M. Shimojo, Y. Higo and M. V. Swain, *ASTM STP*, **1413**, 72-81 (2001).

[9] C. N. Reid, K. Williams and R. Hermann, *Fat. Eng. Mat. Struc.*, **1**, 267-270 (1979).

[10] S. Suresh and J. R. Brockenbrough, *Acta Metal.*, **36**, 6, 1455-1470 (1988).

[11] T. Kunio, H. Nakazawa, I. Hayashi and H. Okamura, "Fracture mechanics: Experimental technique", Asakura-shoten, Tokyo (1984) p.241, (in Japanese).

[12] K. Nakai, S. Koyama, K. Takashima and Y. Higo, *Proc. 6th FENDT '02*, 481-486 (2002).

(Received March 3, 2003; Accepted April 2, 2003)