

# MECHANICAL PROPERTIES MEASUREMENT OF MICROSCALE MATERIALS FOR MEMS APPLICATION

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## ABSTRACT

The elemental size for future Micro/Nano Electro Mechanical Systems (MEMS/NEMS) devices is expected to be 1/1000 to 1/10000 that of traditional structures. This causes many difficulties, called "size effects". In order to understand and overcome the difficulties associated with these effects, mechanical testing machines and methods for micro-sized specimens with load resolutions the order of 100 $\mu$ N and a displacement resolution of 10nm have been developed. Micro-sized cantilever beam specimens (approximately 10 x 10 x 50 $\mu$ m) of Ni-11.5 mass%P amorphous alloy have been used to investigate the fracture behaviour on the micro-sized material in this work. This includes the calculation of fracture toughness values, examination of the effect of the presence of a fatigue precrack on fracture toughness and the effects of testing at elevated temperature. Fracture behaviour differs between specimens with and without a fatigue precrack. This indicates that even a notch with a root radius of 0.25 $\mu$ m cannot be regarded as a crack for these micro-sized specimens. Comparison of fracture toughness values for specimens with different crack growth directions (in-plane and out-of-plane directions) suggests that the Ni-P amorphous thin film exhibits anisotropic fracture behaviour.

## 1 INTRODUCTION

For future application in MEMS/NEMS devices, most materials require their structure to be designed on the nano-scale in order to provide the required physical and/or mechanical properties. The elemental size for MEMS devices is considered to be 1/1000 to 1/10000 that of traditional structures. This down sizing can cause many difficulties, known as "Size Effects", which are described as follows,

- [1] Preparation Process ( Layered production of films),
- [2] Handling and Fixing Methods (Too small for traditional machining and assembling methods),
- [3] Accuracy of Dimensions (Nano-meter accuracy required),
- [4] Accuracy of Measurement Method (Limit of measurement accuracy is 1/1000 that of the component size),
- [5] Surface Effect (High Surface/volume ratio),
- [6] Materials Strengthening Methods (New methods required for micro-sized materials) and
- [7] Mechanical Properties (Must be homogeneous, as in nano-crystalline or glassy materials).

In order to understand these size effects and to overcome the difficulties associated with them, it is essential to develop mechanical testing methods for micro-sized specimens as well as standardized evaluation methods for the design of durable and reliable MEMS devices. This means that the development of versatile testing machines to enable the mechanical testing of tensile, fracture toughness and fatigue properties of micro-sized materials and elements is essential for the development of MEMS devices. Previously nano-indentor type loading systems have been applied for the measurement of mechanical properties in thin film specimens, although the low stiffness of such systems has made them inadequate for applying cyclic loading. Versatile machines also require the combination of load resolutions in the order of 100 $\mu$ N with a displacement resolution of 10nm.

## 2 TESTING MACHINE FOR MICROSCALE MATERIALS

A mechanical testing machine has been developed for the evaluation of micro-sized materials. Figures 1 (a) and (b) respectively show (a) a photograph and (b) a block diagram of this machine, which consists of an actuator, a specimen holder, a strain gauge type load cell and a controller. In this case a TERFENOL-D (TbDyFe) magnetostrictive device is used as an actuator to impart displacements upto  $10\mu\text{m}$ , with an accuracy of  $5\text{nm}$  and a maximum cyclic response frequency of  $100\text{Hz}$ . The actuator is connected through a metal shaft to a diamond tip of  $5\mu\text{m}$  in radius, providing a high stiffness loading fixture. The displacement of the actuator is measured by a laser displacement meter to an accuracy of  $5\text{nm}$ , the displacement signal being used as feed back control.

In order to test a micro-sized specimen it is set in a specimen holder, which is then located onto the load cell, as shown in Figure 1 (b). This holder is interchangeable between a focused ion beam scanning machine (for machining of the specimens), this testing machine and a field emission-gun type scanning electron microscope, therefore allowing machining, testing and observation processes to be completed without handling the specimen directly. 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  $0.1\mu\text{m}$ . A CCD camera monitors the specimen during testing. As displacements are applied to the specimen through the diamond tip the load being applied is measured by the load cell, to a load resolution of  $10\mu\text{N}$ . Load control is also an available option on this instrument. This testing machine is set up in a windscreen box located in a clean room with constant temperature and humidity. This is in order to eliminate dust, temperature change and slight wind streams within the room during testing. Further details of this testing machine are described in references by Takashima [1] and Higo [2]. Fracture surfaces after the tests were observed using a HITACHI S-4000 field emission-gun type scanning electron microscope.

## 3 EXPERIMENTAL PROCEDURE

### 3.1 Material

The material used in this investigation was a Ni-11.5 mass%P amorphous alloy thin film electroless deposited on an Al-4.5 mass%Mg alloy. The thickness of the amorphous layer was

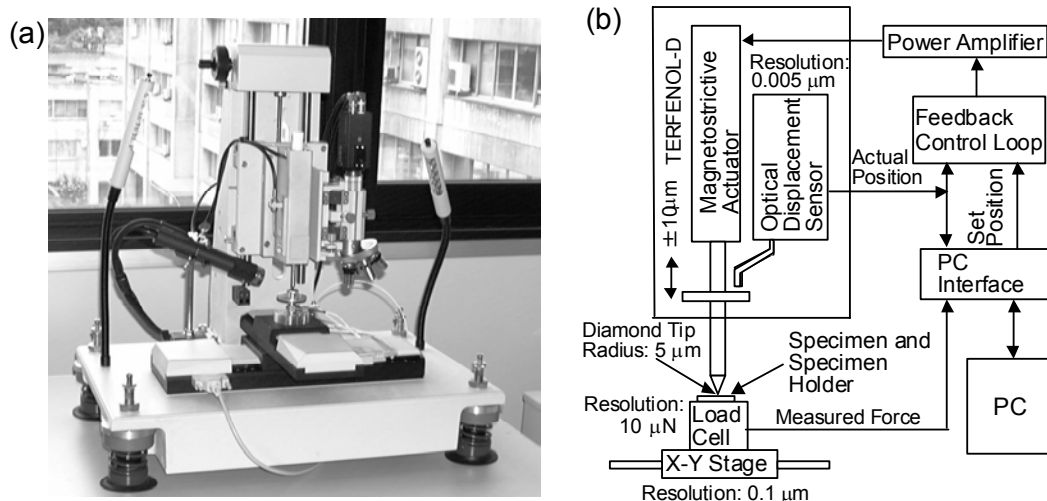


Figure 1: Photograph (a) and block diagram (b) of the fatigue testing machine for micro-sized specimens used in this investigation.

12 $\mu\text{m}$  whilst that of the Al-4.5 mass%Mg alloy substrate was 0.79mm. A disk with a diameter of 3mm was cut from the Ni-P/Al-Mg sheet by electro discharge machining and the layers separated by dissolving the substrate in a NaOH aqueous solution.

### 3.2 Specimen Preparation

Two types of micro-sized cantilever beam specimens with different crack orientations were prepared in order to allow investigation of the anisotropic fracture behavior. These are referred to as “out-of-plane type specimen” and “in-plane type specimen” as schematically shown in Figures 2 (a) and (b), respectively. The crack will propagate parallel to the deposition direction in the out-of-plane type specimen but perpendicular to the deposition direction in the in-plane type specimen. Figures 3 (a) and (b) show scanning electron micrographs of the specimens. The breadth of the out-of-plane type specimen,  $B$ , is  $\sim 12\mu\text{m}$ , the distance from the loading point to the notch position,  $L$ ,  $\sim 30\mu\text{m}$ , and the specimen width,  $W$ ,  $\sim 10\mu\text{m}$ . The size of the in-plane type specimen is approximately the same size;  $B \sim 10\mu\text{m}$ ,  $L \sim 30\mu\text{m}$  and  $W \sim 10\mu\text{m}$ . All specimens were prepared by focused ion beam machining. Notches with depth of equivalent to  $a/W = 0.25$  ( $a$ : notch length) were introduced into the top of specimens, 10  $\mu\text{m}$  from the fixed end of the specimen, as shown in Figure 3. A notch width of  $0.5\mu\text{m}$  is used to provide a notch radius of  $0.25\mu\text{m}$ . These specimens

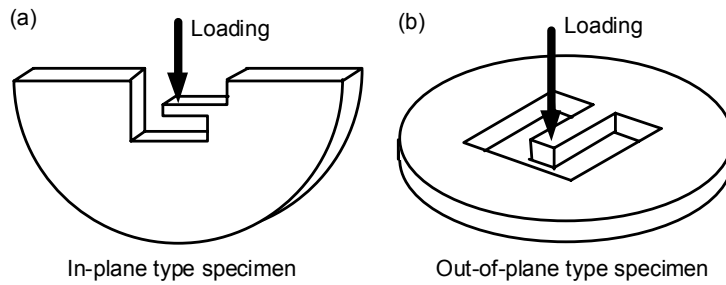


Figure 2: Two types of specimen orientations. (a) loading direction perpendicular to the deposition direction and (b) loading direction parallel to the deposition direction.

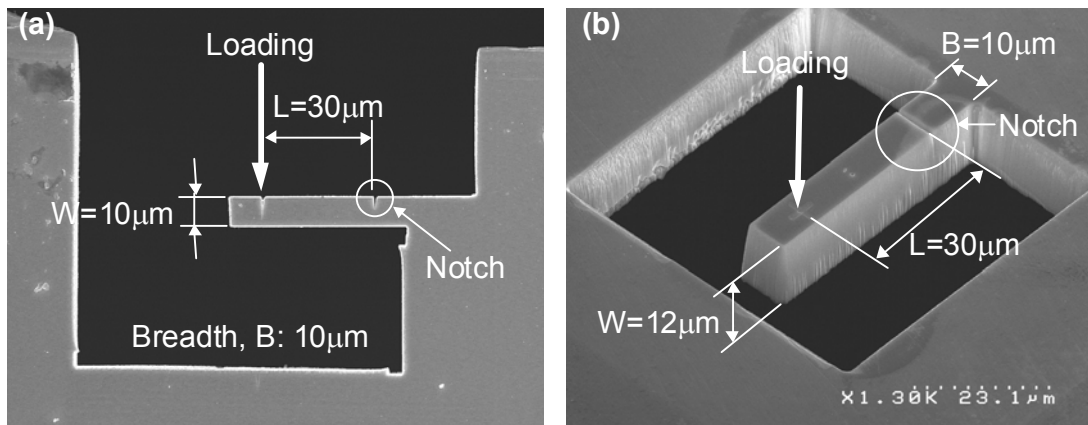


Figure 3: Scanning electron micrographs of micro-sized specimens produced by focussed ion beam (FIB) machining. (a) in-plane type specimen and (b) out-of-plane type specimen. Notches were also introduced by FIB.

were then fatigue precracked under a constant load amplitude in the testing machine in order to provide a total (notch and precrack) equivalent to  $a/W \sim 0.5$ . Samples were also tested with a notch length equivalent to  $a/W \sim 0.5$  and no precrack in order to investigate the effect of introducing a fatigue pre-crack on fracture behaviour of micro-sized specimens.

### 3.3 High Temperature Testing

Fracture toughness testing upon identical specimens was also completed at elevated temperatures in order to evaluate the effect of temperature upon fracture performance. This was completed upon the same testing machine previously used with the addition of a small ceramic heater, capable of generating a maximum temperature of 573K. This allowed the performance of fracture toughness tests at 373 K and 473 K for comparison with previous room temperature tests.

## 4 RESULTS AND DISCUSSION

### 4.1 Effect of Fatigue Pre-crack on Fracture Behaviour

Figure 4 (a) shows typical load - displacement curves for in-plane notched specimens with and without a fatigue pre-crack. The notch only specimen can be seen to have fractured in a brittle manner, while the specimen with a fatigue pre-crack has fractured in a ductile manner. The maximum load of the specimen with a notch only is approximately twice as that of the specimen with a fatigue pre-crack. This may be due to differences in stress concentration at the crack tip with that at a fatigue crack tip being larger than that at a notch tip. This indicates that even the notch with a root radius of  $0.25\mu\text{m}$  cannot be regarded as a crack for these micro-sized specimens. In addition, the ion implantation caused by focused ion beam machining of the notch may have altered the mechanical properties around the notch tip in this case. As the depth of ion implantation area is estimated to be less than  $1\mu\text{m}$ , the influence of ion implantation can be negated by introducing a fatigue pre-crack of more than  $1\mu\text{m}$  in length. It is therefore essential to use a precrack in the evaluation of micro-sized fracture toughness of this Ni-P material.

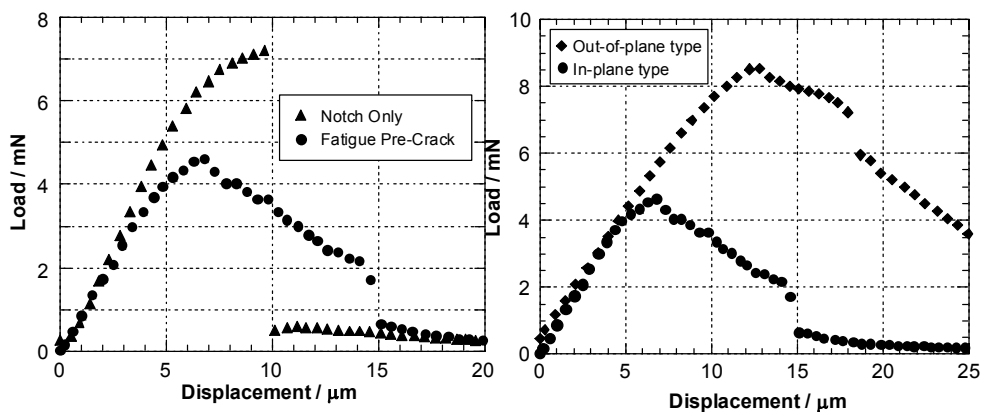


Figure 4: Load-Displacement curves for micro-sized specimens (a) with and without notches and (b) with different fatigue precrack orientations.

#### 4.2 Effect of Crack Orientation on Fracture Behaviour

Figure 4 (b) shows typical load-displacement curves from the fracture testing of in-plane and out-of-plane precracked specimens. The maximum load of the out-of-plane type specimen is higher than that of in-plane type specimen in spite of the size of specimen and the length of fatigue pre-crack being approximately the same. Fracture tests were carried out for five specimens of each type of specimen, and comparable results were obtained. This suggests that the electroless deposited Ni-P amorphous thin film exhibits anisotropic fracture behaviour.

#### 4.3 Fracture Toughness Measurement

As crack opening displacement could not be measured for these micro-sized specimens, the crack initiation load could not be determined. The maximum load was therefore assumed to be the crack initiation load for the purposes of calculating fracture toughness values. Stress intensity factor,  $K$ , is calculated from the equation for a single edge notched cantilever beam specimen, according to Okamura [3].

Figure 5 shows a scanning electron micrograph of fracture surface for an out-of-plane type specimen. Fine equispaced markings aligned perpendicular to the crack propagation direction are observed ahead of the notch. This kind of markings have also been observed on fatigue fracture surface of micro-sized Ni-P amorphous alloy specimens in previous research [8], and are considered to be striations. This indicates that a fatigue pre-crack was successfully introduced into these micro-sized specimens. Vein patterns which have been observed on fractured Ni-P amorphous alloys are visible ahead of the fatigue pre-cracked region. The total pre-crack length was therefore measured from scanning electron micrographs of the fracture surfaces prior to the calculation of a  $K$  value, as shown in Figure 5. The calculated provisional fracture toughness values ( $K_Q$ ) for the out-of-plane and in-plane specimens are 7.3 and 4.2 MPam<sup>1/2</sup>, respectively. These values are, however, not valid plane strain fracture toughness values ( $K_{IC}$ ), as the criteria of plane strain requirements ( $a, W-a, B > 2.5 (K_Q/\sigma_y)^2$ ) were not satisfied for this specimen size.

#### 4.4 Elevated Temperature Effects

Figure 6 shows the temperature dependence of fracture as plane strain requirement, are not satisfied for micro-sized specimens, fracture toughness values toughness values for the Ni-P

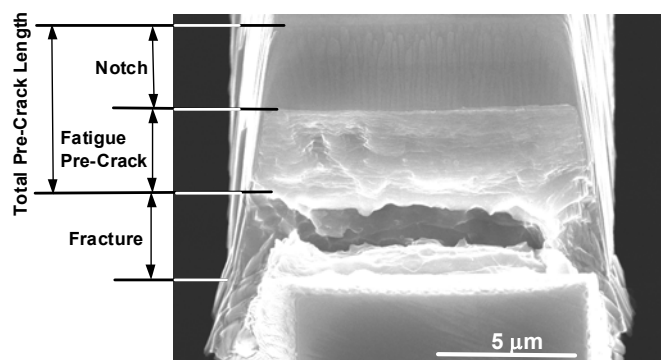


Figure 5: Scanning electron micrograph of fracture surface of out-of-plane type specimen.

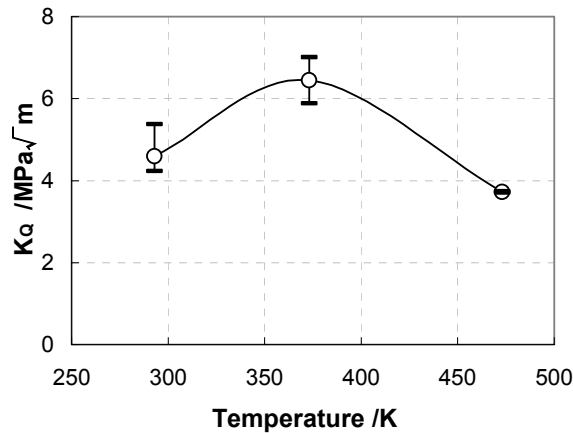


Figure 6: Temperature dependence of fracture toughness of micro-sized Ni-P amorphous alloy.

amorphous alloy specimens. In this investigation, plane strain fracture toughness ( $K_{IC}$ ) could not be obtained, are therefore indicated as  $K_Q$  in Figure 6. Values can be seen to have increased approximately 40 % at 373 K as well as decreasing approximately 19 % at 474 K compared to the room temperature value. As this amorphous material is in a thermally non-equilibrium state structural changes can effect the fracture toughness although the temperatures concerned are lower than the crystallization temperature ( $\sim 570$ K). One of these possible structural changes would be the formation of nano-sized crystals, leading to mechanical properties that are reported to depend on their size and volume fraction, see Inoue [4]. The enhancement of fracture toughness is therefore considered to be associated with the formation of nano-sized crystals with the decrease with further increased temperature being due to the growth of crystals. Further investigation is however required to confirm such a relationship.

## 5 CONCLUSIONS

It is hoped that the production of such devices will allow to compare material properties of different origin and the processing methods used to produce them. These methods can then be used to provide new materials and strengthening methods to further advance the capabilities of MEMS devices. The addition of this data to a mechanical properties database for the design and simulation of MEMS devices will be greatly beneficial to the development of MEMS devices.

## 6. REFERENCES

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