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TEMPERATURE DEPENDENCE OF FRACTURE TOUGHNESS IN A MICRO-SIZED Ni-P AMORPHOUS ALLOY

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ABSTRACT

Temperature dependence of fracture toughness has been studied on micro-sized Ni-P amorphous alloy specimens. The material used in this study was a Ni-11.5wt%P amorphous thin film. The specimens were prepared in a cantilever beam type configuration of dimensions of $10\times12\times50$ μm^3 by focused ion beam machining. Fatigue pre-cracks were introduced into all the specimens. Temperature was controlled using a newly developed specimen holder from room temperature to 473 K. Compared with room temperature, fracture toughness was increased approximately 36 % at 373 K but decreased 16 % at 473 K. From the transmission electron microscope (TEM) observation, no precipitation of crystalline phase was observed in plastically deformed regions tested at 373 K. Differential scanning calorimetry (DSC) measurement also revealed that no crystallization occurred at a temperature range from 300 to 473 K. From these results it is considered that fracture toughness of an amorphous alloy may be changed due to short or medium range order structural relaxation caused by heating.

KEYWORDS

temperature dependence of fracture toughness, amorphous alloy, micro-sized material, structural relaxation

INTRODUCTION

Microelectromechanical systems (MEMS) are expected to be applied to many industrial fields such as biomedical, semiconductor, telecommunication, aerospace, etc [1]. The size of the components used in these devices will become less than a micron or sub-micron which is smaller than the grain size of conventional crystalline metals. The anisotropic mechanical properties are not negligible, which depends on crystallographic orientation. Furthermore, the mechanical properties of such micro-sized materials are considered to be different from those of bulk materials because of the surface effects. It is therefore unreliable to apply the mechanical properties measured using bulk materials, such as elastic constants, yield stress, fracture toughness, etc., to design the micro-sized machines or MEMS devices. Because of these requirements for micro-sized materials, direct measurements of mechanical properties on micro-sized materials are essential for practical applications of such MEMS devices.

Amorphous alloys have high strength and isotropic mechanical properties because of the absence of long range ordering [2]. Thus it is expected to be used as a structural material for micro machines or MEMS devices. Several mechanical properties such as fracture toughness [3], fatigue life and crack growth properties [4] and corrosion fatigue properties [5] of a micro-sized amorphous alloy have already measured by our group at room temperature. In particularly, fracture toughness is one of the most important parameters to design actual micro-sized machine or MEMS devices. The case of practical applications, however, temperature is considered to be increased. Amorphous alloys are in a thermally non-equilibrium and meta-stable state so that structural relaxation (including crystallization) may occur with increasing temperature [6-8]. Structural relaxation may change the fracture toughness of amorphous alloys. The dependence upon temperature of fracture toughness, however, has not been measured even though temperature in such micro machines may increase under practical applications. In this study, temperature dependence of fracture toughness on micro-sized amorphous alloy has been investigated with a temperature range from 300 K to 473 K. Furthermore, structural changes in plastically deformed regions were also investigated using a transmission electron microscope (TEM).

EXPERIMENTAL PROCEDURE

The mechanical testing machine used in this study was micro fatigue testing machine (MFT2000), which can apply static and cyclic loading. The specimen can be positioned with an accuracy of 0.1 μm using a precise X - Y stage. The load resolution is 10 μN, and the displacement resolution is 5.0 nm.

The material used in this study was a Ni-P amorphous thin film prepared by electro-less deposition on an Al-Mg based substrate. This material has been used for hard disk substrates. This material is mass-produced with uniform quality. The roughness of the surface is in the order of nano-meters. From these features, this material is considered to be one of the suitable materials for the testing of micro-sized material. Crystallization temperature of this material was 639.5 K, which was determined from a differential scanning calorimetry (DSC) measurement at a constant temperature increasing rate of 20 K/min.

The amorphous thin film was separated from a substrate using an NaOH aqueous solution. This material was cut mechanically into a semicicular disk with a diameter of 3.0 mm. Micro-sized cantilever beam type specimens were prepared near the straight edge of the semicircular disk using a focused ion beam (FIB) machine. Figure 1 shows a scanning electron microscope (SEM) image of the specimen observed from the thickness direction (*B*) of the specimen. The dimensions of the specimens were 50 (*l*)×10 (*W*)× 12 (*B*) μm^3. A notch was introduced into each specimen by FIB machining with a notch tip radius of 0.25 μm. The distances of loading point and notch point were 30 μm (*L*) and 10 μm from the root of the specimen, respectively. According to our previous study [9], it is necessary to introduce a fatigue pre-crack for fracture toughness measurements because the notch tip radius (=0.25 μm) is not sufficiently small. It is therefore fatigue pre-crack was introduced into all the specimens, using an MFT2000 at a constant ΔK of 2.0 MPa m and a cyclic frequency of 10 Hz.

Fracture toughness, K, of the cantilever beam type specimen can be calculated from the following equation.

$$K = \frac{6PL}{W^2 B}\sqrt{\pi a}\, F(a/W), \qquad (a/W < 0.6) \tag{1}$$

where,

$$F(a/W) = 1.22 - 1.40(a/W) + 7.33(a/W)^2 - 13.08(a/W)^3 + 14.0(a/W)^4 \quad (2)$$

In eq. (1), *a* represents the crack length and *P* represents the load at which crack started to propagate.

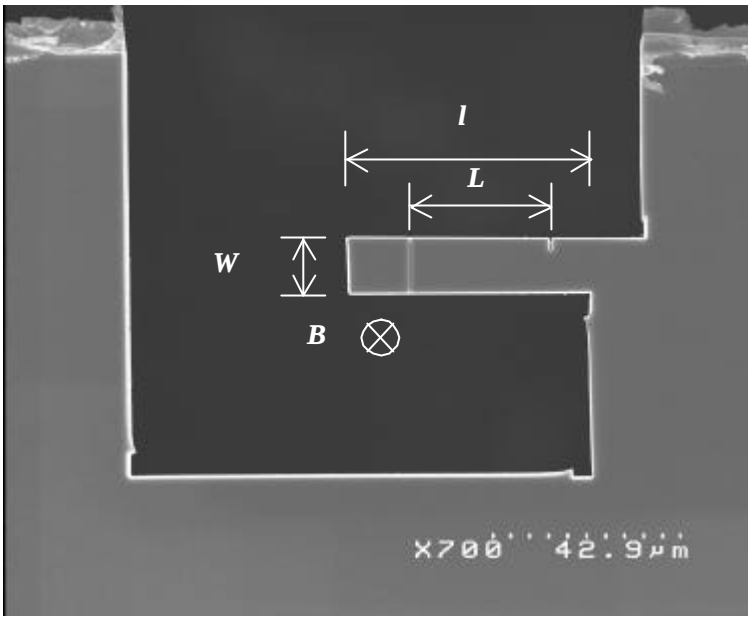

Figure 1 SEM image of a micro-sized cantilever beam type specimen prepared by FIB machining observed from the thickness direction of the specimen.

Fracture toughness measurements were also performed using the MFT2000 in laboratory air. Bending stresses were applied at a loading point with a constant displacement rate of 22 μm/min. The temperature of the specimen was controlled using newly developed special heating equipment installed in the specimen holder of the MFT2000. This heater can control the temperature of the specimen up to 773 K. After the fracture toughness measurements, scanning electron microscope (SEM) and transmission electron microscope (TEM) observations were performed to investigate the shape, fracture surface and structural changes of the specimens.

RESULTS AND DISCUSSION

Figure 2 shows a SEM image of a specimen after the fracture toughness measurement at a temperature of 473 K. It was confirmed from this figure that specimen was failed at the notch position. It was confirmed that other specimens were also failed at the notch position. Figure 3 shows a fracture surface of the specimen tested at 473 K. In front of the notch introduced by FIB machining, flat fracture surface was observed. From the high magnification observation, the formation of striations was observed in front of the notch. The formation of striations was also observed in other specimens. These results indicate that fatigue pre-cracks were introduced into all the specimens.

From the SEM observation a vein pattern [10], a typical fracture surface of amorphous alloys, was clearly observed in the fracture surfaces of all specimens. Moreover, on both sides of the fracture surfaces, the formation of shear lips were observed. This result indicates that the plane strain dominant region existed even in the micro-sized specimen.

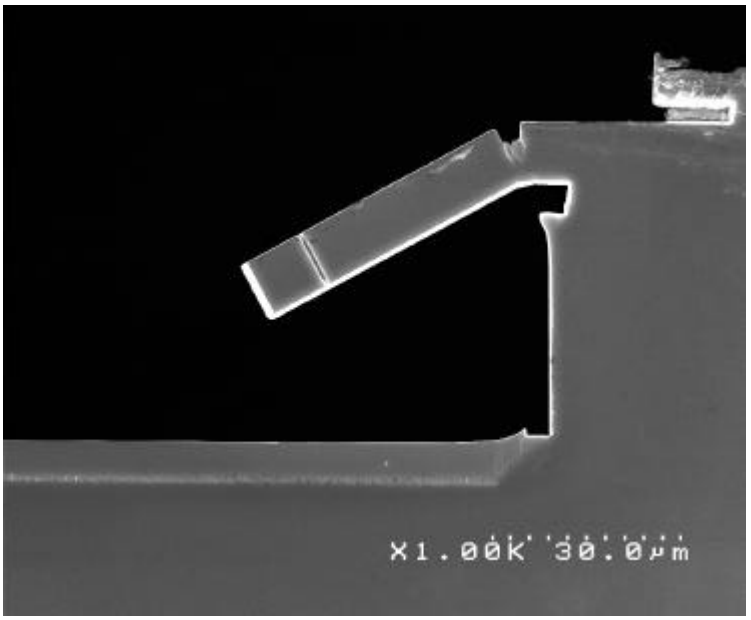

Figure 2 SEM image of micro-sized cantilever beam type specimen after the fracture toughness test observed from the thickness direction of the specimen.

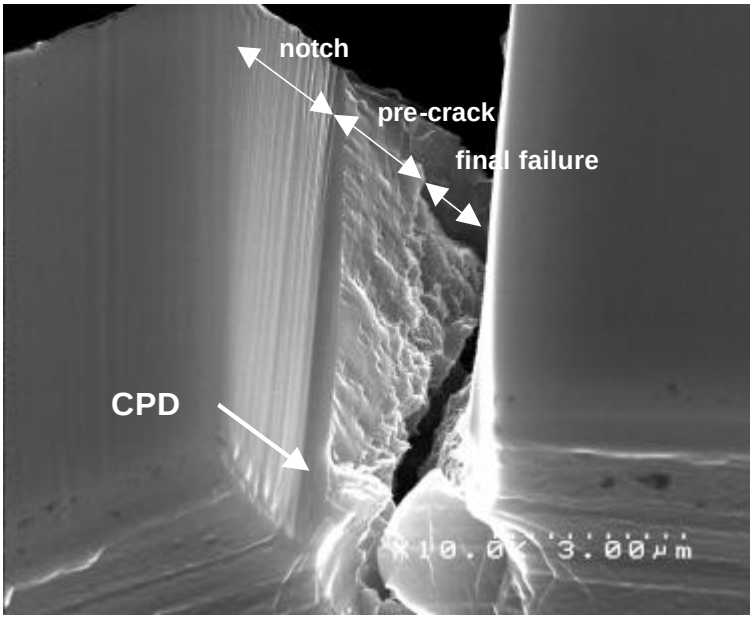

Figure 3 SEM image of fracture surface of the specimen tested at 473 K. Formation of fatigue pre-crack was confirmed in front of the notch tip. CPD in the figure indicates the crack propagation direction.

Figure 4 shows the load and displacement curves obtained at temperatures of 373 and 473 K. The maximum bending load (P_{max}) was 7.82 and 6.06 mN for 373 and 473 K, respectively. Correct value of *P* at which crack started to propagate could not be determined. Thus, the maximum load values in load and displacement curves were employed for the fracture toughness calculation.

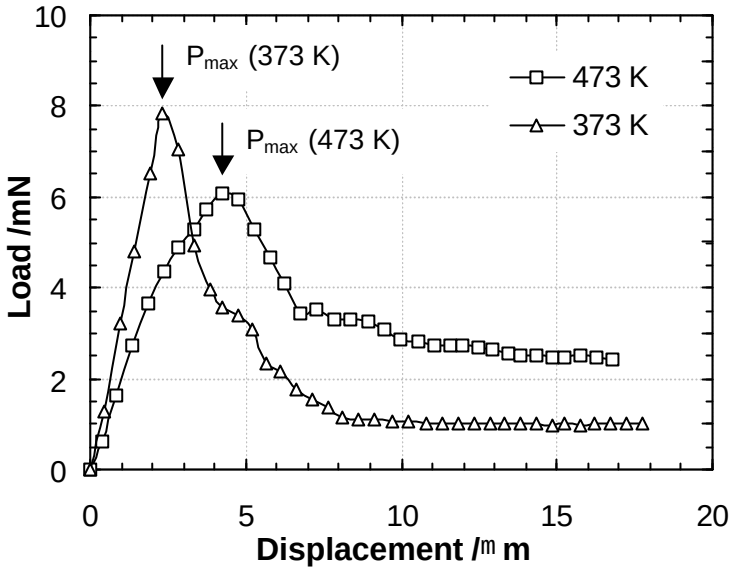

Figure 4 Load and displacement curve obtained at temperatures of 373 and 473 K. The maximum load values were employed for the calculation of fracture toughness.

Temperature dependence of fracture toughness calculated from eq. (1) is shown in fig. 5. In this figure, fracture toughness at room temperature is plotted [3] for the comparision. Measured fracture toughness at a room temperature was 4.33 MPa m. The measured fracture toughness at 373 K was 5.89 MPa m, which is approximately 36 % higher than that at room temperature. On the contrary, at 473 K, fracture toughness was only 3.71 MPa m, which is approximately 14 % smaller than that at room temperature. In the case of this amorphous alloy, fracture toughness does not increase with increasing temperature.

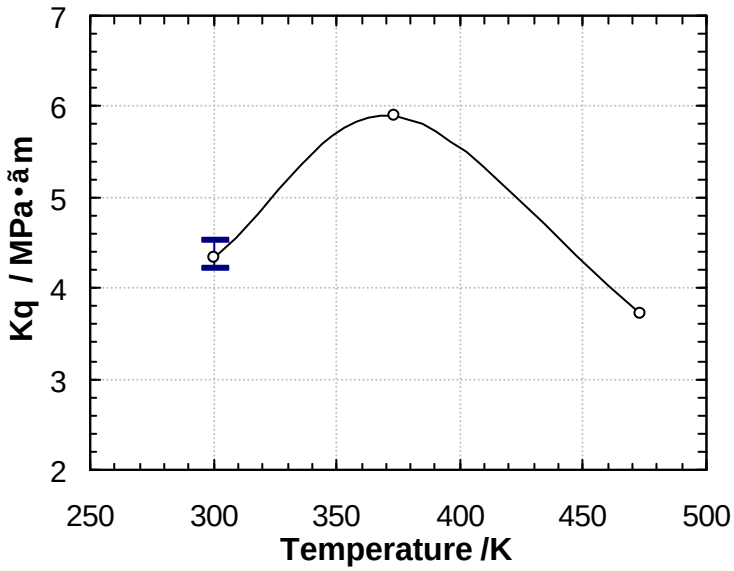

Figure 5 Temperature dependence of fracture toughness calculated from the eq. (1).

To investigate the reason for the abnormal temperature dependence of fracture toughness, TEM observation was performed on the plastic deformed region tested at 373 K. However, no precipitation of crystalline phase was observed. According to the DSC measurement, no heat flow was observed from 300 to 473 K. This result indicates that only the short or medium range structural relaxation was occurred in this temperature range. From these results it is considered that fracture toughness of an amorphous alloy may be changed due to short or medium range order structural relaxation caused by heating.

CONCLUSIONS

Temperature dependence of fracture toughness on a micro-sized amorphous alloy has been investigated at a temperature range from 300 to 473 K. Compared with room temperature, fracture toughness increased approximately 36 % at 373 K, but decreased approximately 14 % at 473 K. The reason for the abnormal temperature dependence of fracture toughness is considered to be caused by the structural relaxation of the amorphous alloy.

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