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Characterization of Micro-mechanical Properties of AlON Ceramic by Cantilever Bending Test

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Abstract

The AlON transparent ceramic cantilever beams containing twin and grain boundaries were fabricated by focused ion beam (FIB) technique. The deformation behaviors were investigated by measurement of the load and displacement dependence of cantilever beams from micro bending tests. Young's modulus of AlON transparent ceramics was calculated from load and displacement curves, the results of which were consistent with results of previous works. Moreover, the bonding strengths of twin lamella boundary, twin boundary and normal grain boundary in AlON transparent ceramics were 5.00 GPa, 5.05 GPa and 4.81 GPa respectively.

KEYWORDS: AlON, Cantilever, Grain boundaries, Mechanical properties

1. Introduction

In recent years, AlON transparent ceramics have received great attention, due to their combination of the excellent mechanical and optical performance. Generally, micro-mechanical properties of AlON ceramics were studied from calculations and simulations, experimental works have rarely been carried out to verify theoretical values for strength and Young's modulus. McCauley[1] experimentally determined the Young's modulus of polycrystalline cubic aluminum oxynitride spinel by using pulse superposition interferometry approach. Hartnett[2] employed the equibiaxial flexure testing in the investigation of the elastic properties of AlON. Recently, two kinds of twin structures with different microstructural feature were observed by Wang[3]. However, no experimental research on the micro-mechanical properties of AlON transparent ceramics has been conducted to determine key properties such as Young's modulus and fracture strength. These properties may elucidate the role of twin boundary and grain boundary on the mechanical performance of AlON transparent ceramics.

Micro-mechanical testing has been widely adopted to characterize the mechanical properties of advanced materials especially in the field of thin film on-chip test[4-7]. Young's modulus and fracture strength can be determined by micro bending test which were considered to be the key parameters affecting the bending behaviors strongly. Hommel[8] quantitatively investigated the mechanical properties of thin polycrystalline Cu films on deformable substrates. Euan Boyd[9] determined Young's modulus of single and multilayer SiC cantilevers by comparing stylus and resonant methods.

In this work, a focused ion beam (FIB) approach was employed to fabricate AlON cantilevers containing different grain boundary structures. The Young's modulus of AlON transparent ceramics was measured from load-displacement curves by micro-mechanical testing. The bonding strengths of ordinary grain boundary and twin boundary in AlON ceramics were studied simultaneously.

2. Experimental procedures

The transparent polycrystalline AlON ceramics were prepared by pressureless sintering with sintering additives of Y_2O_3 and MgO added, starting from synthetic sub-micron γ-AlON powders. Then as-sintered AlON ceramics were finely polished with diamond suspension from a grit size of 9 μm to 1 μm (Struers, Cleveland, USA). Subsequently, a carbon layer with the thickness of ~20 nm was deposited on the as-polished surface to enhance the electrical conductivity of the sample surface.

The micro cantilever beams were fabricated in focused ion beam (FIB) system (Helios Ge PFIB CXe, FEI, USA) equipped with back-scattered electron

(BSE) detector and electron back-scattered diffraction (EBSD) detector. The main process diagrams are displayed in Fig.1 and described as follow. The AlON grains of interest were selected in BSE mode, and then coated with a platinum layer with a thickness of 10 μm on the surface. Then staircase cuts at a voltage of 30 keV and beam current of 20 nA were carried out around at selected regions at a tilt angle of 52° and then symmetric cuts were conducted at the bottom of the region at 0°. As shown in Fig.1 (a) and (b), the chunk waiting to liftout was finished. Subsequently, the chunk was transferred to a silicon wafer by using a micromanipulator, see Fig.1 (c). The grain and twin boundary of interest were marked by cutting shallow lines using Xe^+ ion beam. After being cut with successively lower currents (1 nA to 100 pA), the micro cantilever beam with a width and height of ~1 μm to 2 μm (Fig.1 (d)) was fully prepared. In this way, three micro cantilever beams containing normal grain boundary, twin boundary and twin lamella structure were fabricated. It is worth pointing out that the normal grain boundary and twin boundary with different features were positioned at ~1um away from the fixed end of cantilever beams. Such positioning of grain boundaries of AlON specimens would enable the cantilever to generate the relative high stress concentration during bending testing and guarantee the grain boundary and twin boundary play a critical role in the cantilever cracking. Micro bend testing of the cantilever beams was accomplished in the FIB by employing a force sensing probe (FMT 120, Kleindiek Nanotechnik, Germany).

3. Results and Discussion

Fig.2 displays the BSE images of the grains of interest and the regions of cantilevers liftout schematically depicted in the BSE images. Two types of twin structure in transparent AlON ceramics were observed in Fig.2 (a) and (c). From Fig.2 (a), there is a thin lamella with a thickness of ~3μm inside an AlON grain, which has already been verified as twin lamella in the previous work[3]. Common twin structure was shown in Fig.2 (c). The twin lamella, twin boundary and ordinary grain boundary were positioned at ~1um away from cantilever beam end, aiming to compare the fracture strength of two kinds of twin boundary, ordinary grain boundary and grains. As mentioned in the experimental section, micro bend testing of these cantilever beams was then carried out to obtain load and displacement synchronously.

Typical micro bend testing images are illustrated in Fig.3. The cantilever beams were fixed on a movable stage, then a micro force probe was inserted to the side of beams at a distance of 25 μm from the root of the beam (Fig.3 (a)). Thereafter the beam moves toward the tip of the probe with a constant speed of 0.5 μm/s the load at the tip was recorded simultaneously with displacement every half second until the beam was broken (Fig.3 (b-d)). Fig.3 (e) shows the image of the cantilever beam with maximum bending angle, which exhibited the bending state of cantilever close to fracturing. As shown in Fig.3 (f), the cantilever beam was broken at the position of ~ 1μm from the root. A similar phenomenon was observed in the other two cantilever beams, demonstrating that the fracture was more prone to occur at the grain boundary or twin boundary compared with grain even though highest stress was present on grains at the beam root.

From the still images in Fig.3, displacements at loading point of the beam were measured. Combining the recorded force, the dependence of load on as-measured displacement of AlON transparent ceramics cantilevers containing different grain boundary features were depicted in Fig.4. Obviously, linear relationships were observed in all three curves except for the red circled part in Fig.4 (b). Such an exception was generally attributed to the inevitable stick-slip occurred at the interface of probe and beam when the beam was bent at a large angle. According to the linear relationship observed, the bending of cantilever beams was believed to be elastic deformation.

The Young's modulus and fracture strength of AlON transparent ceramics specimens can be calculated by employing the Euler-Bernoulli beam theory[10], as expressed in Eq. (1) and (2).

$$D = \frac{F{l_0}^3}{3EI} \qquad (1)$$

where *D* is the displacement, *F* the force, l_0 the distance between the loading point and the fixed end, *E* the young's modulus and *I* the moment of inertia of the beam.

$$\sigma_{max} = \frac{F_{max}(l_0 - l_f)y_{max}}{I} \qquad (2)$$

where σ_{max} is the maximum normal stress at failure, F_{max} the maximum force when beam broken, l_f the distance between the fracture point and fixed end and y_{max} the maximum distance from neutral axis of beam.

For the beam with rectangular section, the *I* and y_{max} were given as

$$I = \frac{bh^3}{12} \qquad (3)$$

$$y_{max} = \frac{h}{2} \qquad (4)$$

where b is the width of beam, Thus Young's modulus, *E* and failure strength could be expressed as

$$\mathrm{E} = \frac{4{l_0}^3}{bh^3} \times \frac{F}{D} \qquad (5)$$

$$F_S = \frac{6F(l_0 - l_f)}{bh^2} \qquad (6)$$

where the $\frac{F}{D}$ is the slope of the load-displacement curve.

By using the equation (5) and (6), the Young's modulus and Fracture strength of three beams were calculated and compared with previous works, expressed in Table 1.

From the middle row of Table 1, it is found that the Young's modulus in our work was well corresponded with the previous experimental results and slightly lower than the calculated results. There is a small divergence in the Young's modulus in three beams which may be attributed to the size effect of beam[13-15]. Thus the cantilever beam test exhibited its reliable and effective in the determination of Young's modulus of AlON materials. As the three beams were broken at the position of grain or twin boundary, the fracture strength of the beams represent the bonding strength of different interfaces. Fracture strength of S_1, S_2 and S_3 represent the bonding strength of twin lamella boundary, twin boundary and grain boundary, which were 5.00GPa, 5.05GPa, 4.81GPa respectively. Such high fracture strengths which are significantly higher than the bulk AlON properties may be ascribed to the sufficiently small beam size. At the small scale, the influence of unfavorable factors such as flaws and dislocations on fracture strength can be neglected. Moreover, the presence of more twin and grain boundaries can also reduce the fracture strength of bulk AlON ceramics.

Obviously the bonding strength of cantilever beam with twin boundaries was higher than that of cantilever beam with grain boundary, which may ascribe to the lower surface energy of the {111} twin plane in cubic AlON spinel. In addition the almost identical bonding strength of two types of twin boundaries elucidates that the interface states of the two kinds of twin boundaries are similar even though the formation mechanism of two kinds of twins may be different.

4. Conclusion

The cantilever beams of AlON transparent ceramics with twin and grain boundary structures were fabricated by using FIB system. Linear load-displacement curves were obtained from the micro bending tests of three cantilever beams with different grain boundary features. The linear load displacement relationship represents the elastic deformation behavior during bending of AlON. The Young's modulus was calculated from the load-displacement curves, which reached 331.3 GPa, 326.8 GPa and 291.4 GPa respectively. The bonding strengths of twin lamella and twin boundary were 5.00 GPa and 5.05 GPa, showing no obvious discrepancy, whereas the bonding strength of the normal grain boundary attained 4.81GPa only.

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Fig.1 The process images of cantilever beam preparation of AlON transparent ceramics (a, b) the chunk waiting to liftout, (c) the chunk on Silicon wafer, (d) the finished cantilever beam.

Fig.2 The BSE images of the grains which contain (a) twin lamella, (c) binary twins and (e) ordinary grain boundary and (b, d, f) the cantilever beams liftout from the corresponding regions.

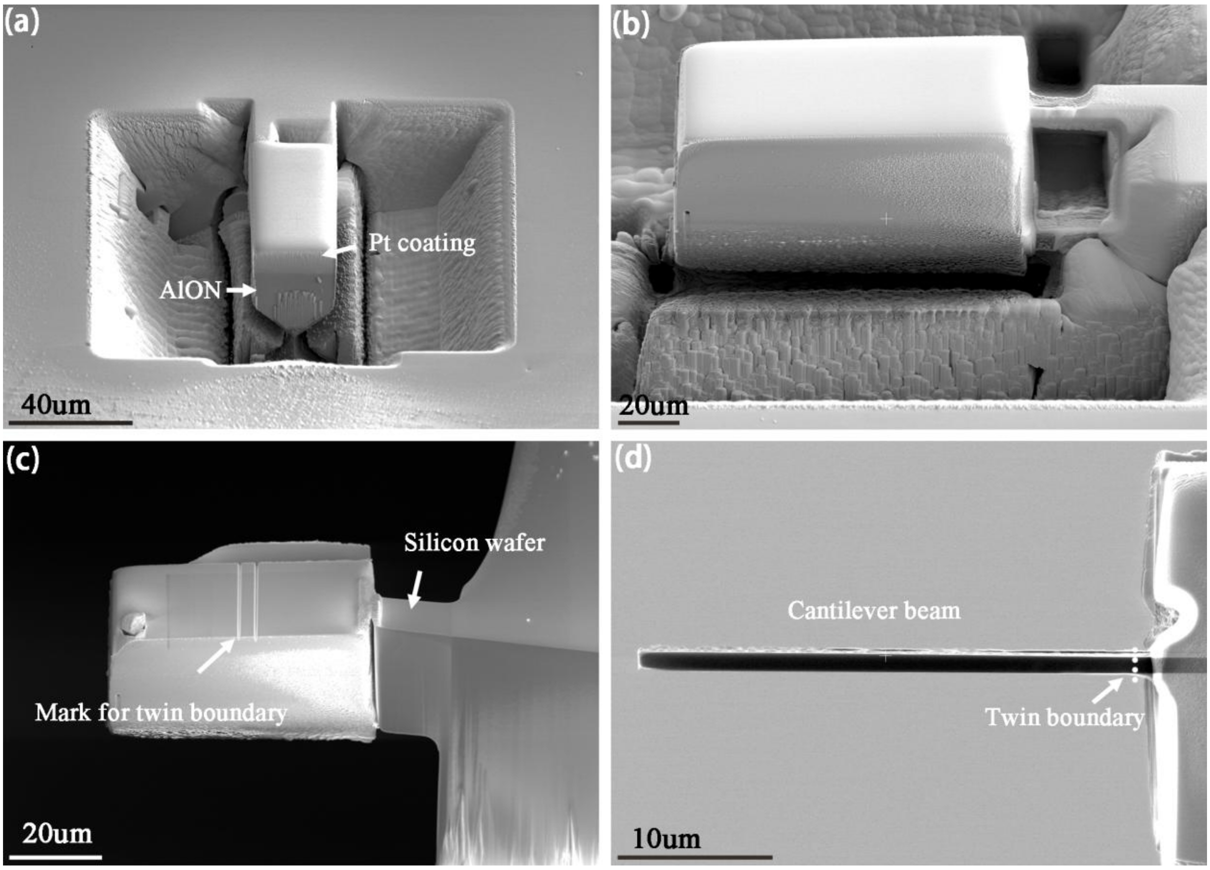
Fig.3 The process of micro bending test, showing deformation and fracture (a)initial state, (b-d) gradually bending, (e) critical bending before broken and (f) beam was broken.

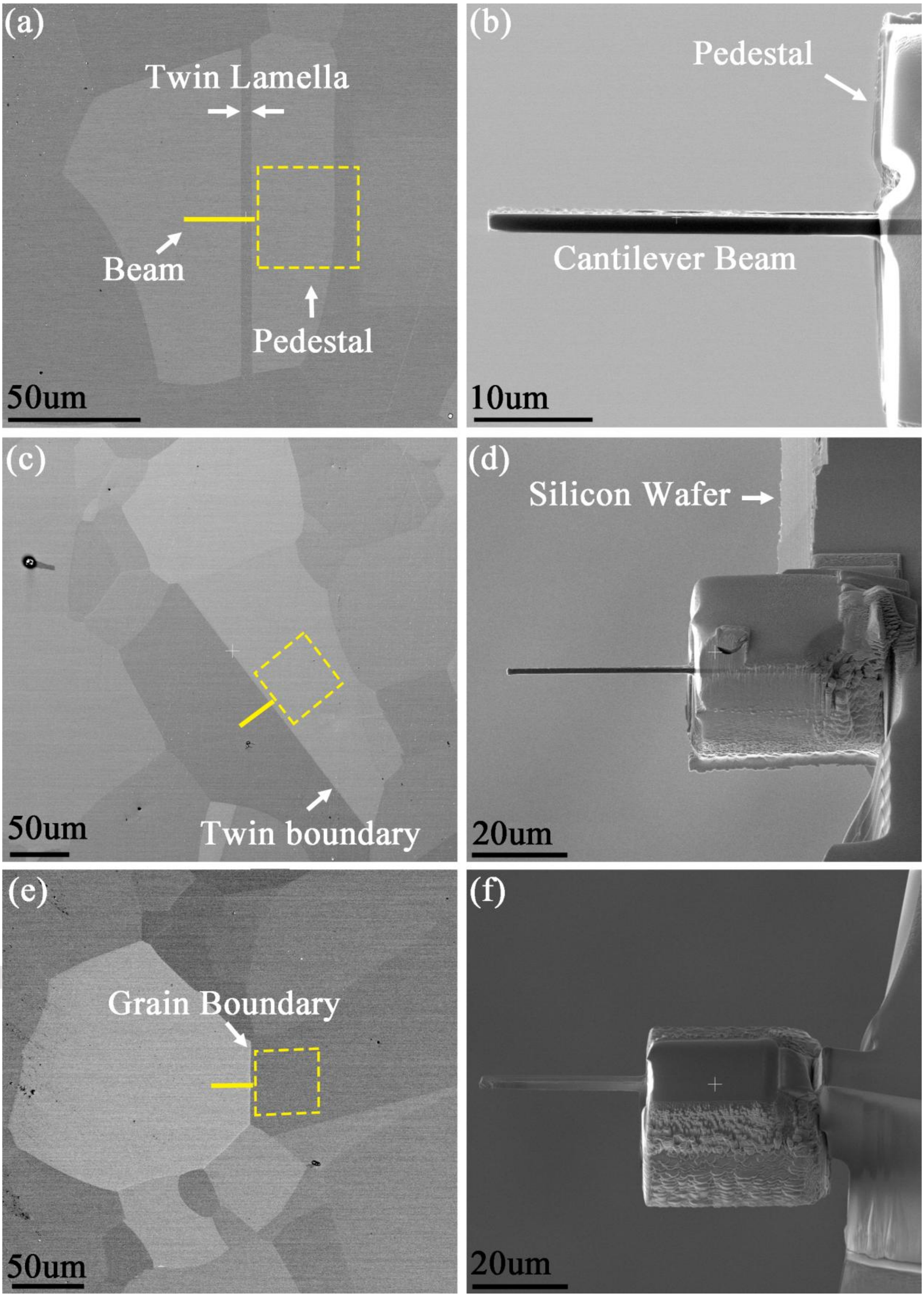
Fig.4 Load-displacement curves of cantilever beams containing (a) twin lamella, (b) twin boundary and (c) grain boundary.

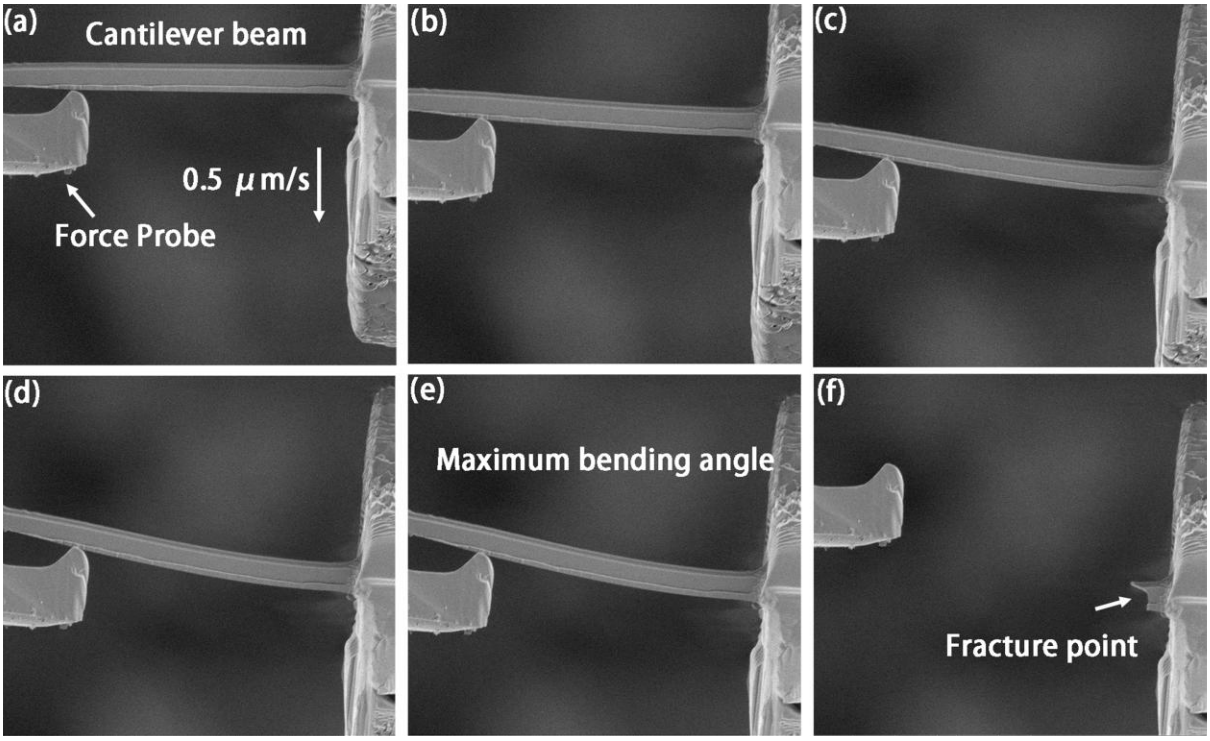
Table 1. The Young's modulus and fracture strength of AlON in present and previous works.

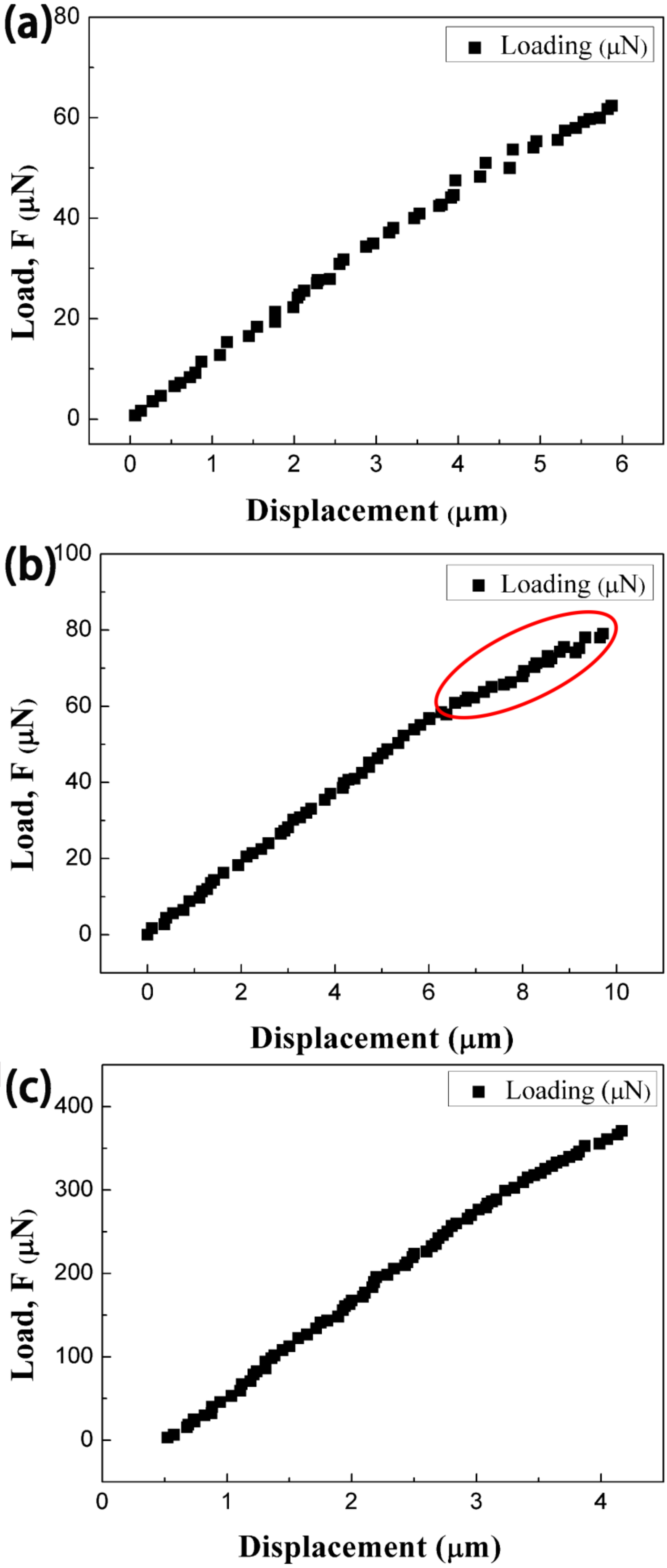
	Present Work			Mccauley JW[1]	Hartnett TM[2]	Wang Y[11]	Okeke OU[12]
	S_1	S_2	S_3				
Young's modulus (GPa)	331.3	326.8	291.4	306.96^e, 315.5^e, 320.3^e	321^e	347.3^c	340^c
Fracture strength (GPa)	5.00	5.05	4.81	-	-	-	-

e: experiment, c: calculated


(a)
Pt coating
AlON
40um
(b)
20um
(c)
Silicon wafer
Mark for twin boundary
20um
(d)
Cantilever beam
Twin boundary
10um


(a)
Twin Lamella
Beam
Pedestal
50um
(b)
Pedestal
Cantilever Beam
10um
(c)
50um
Twin boundary
(d)
Silicon Wafer
20um
(e)
Grain Boundary
50um
(f)
20um


(a)
Cantilever beam
0.5 μm/s
Force Probe
(b)
(c)
(d)
(e)
Maximum bending angle
(f)
Fracture point


(a)
Loading (μN)
Load, F (μN)
Displacement (μm)
(b)
Loading (μN)
Load, F (μN)
Displacement (μm)
(c)
Loading (μN)
Load, F (μN)
Displacement (μm)