원자현미경에서 굽힘 실험을 통한 나노허니컴 구조물의 영률 측정

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Young's modulus measurements of nanohoneycomb structures by flexural test in atomic force microscope

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Abstract

원자현미경을 이용하여 나노허니컴 구조물의 굽힘 탄성계수를 측정하였다. 나노허니컴 구조물의 단면적은 기공들의 배열 때문에 위치마다 다르게 되고, 이로 인해 관성 영역 모멘트는 상수값으로 계산되지 않는다. 본 연구에서는 나노허니컴 구조물의 단위 면적 내 관성 영역 모멘트의 평균값을 벌크 구조의 나노허니컴 구조물의 영률로 가정하였다. 단위 면적 내 관성 영역 모멘트의 평균값과 나노허니컴 구조물의 기공률 사이에 관계식이 유도되었다. 기공의 직경이 31 nm인 양극 산화 알루미늄 필름이 나노허니컴 구조물로 제작되었다. 양극 산화 알루미늄의 영률이 원자현미경을 이용한 굽힘 실험으로 측정되었으며, 나노 인장시험기의 인장 실험 결과와 비교되었다.

Key Words: Flexural Modulus, Nanohoneycomb, Anodic Aluminum Oxide

1. Introduction

Honeycomb cell structures in nano/micrometer scale have fabricated in various methods [1-3]. Honeycomb structures with the pores of a nanometer size are referred to nanohoneycomb structures. Anodic Aluminium Oxide(AAO) films attract favorable attention as nanohoneycomb structures due to its very simple fabrication process, high aspect ratio, self-ordered hexagonal pore structure, and simple control of pore dimensions. Then nanohoneycomb structures such as AAO films can be used more widely than honeycomb cell structure of macroscale in the new fields of magnetic storage, solar cells, carbon nanotubes, catalysts, and metal nanowires. In all the applications, the mechanical properties of nanohoneycomb structures are of prime importance.

This study obtains the Young's modulus of nanohoneycomb structures from flexural test in AFM by determining the area moment of inertia for the nanohoneycomb structure. the Young's modulus measured from the flexural test is compared with that measured from tensile tests in Nano-UTM.

2. Theory

Consider the rectangular nanohoneycomb beam structure as shown in Fig. 1(a). In the top section view of Fig. 1(b), the unit cell and the unit area can be considered because the pores in nanohoneycomb structure are arrayed regularly. The porosity of the nanohoneycomb structure is calculated from the geometry of the unit cell as follows;

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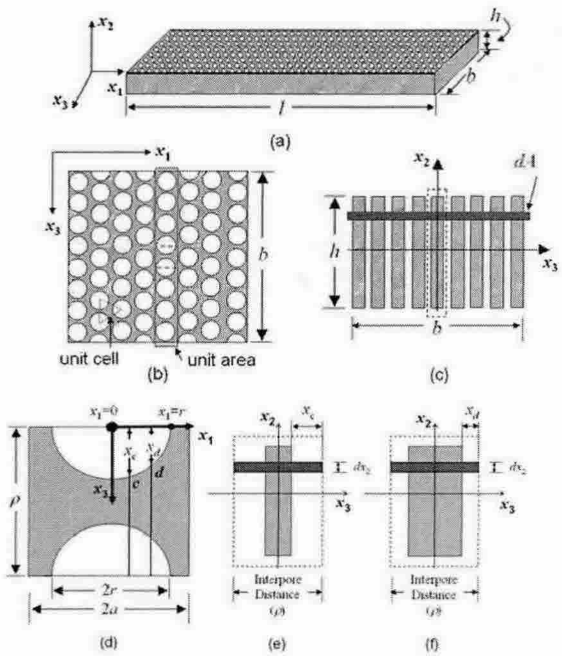


Fig. 1 Nanohoneycomb structure. (a) Rectangu--lar nanohoneycomb beam structure. (b) The top section view and (c) cross section view of the beam structure. (d) Magnified view of the dotted area. (e) Cross sections at the line c and (f) cross sections at the line d.

$$P=\frac{\pi r^2/2}{\sqrt{3}\rho^2/4}=\frac{2\pi}{\sqrt{3}}\left(\frac{r}{\rho}\right)^2 \qquad (1)$$

where r is the pore radius, and is the interpore distance. Fig. 1(c) shows the cross section view for an arbitrary position on the pore in the nano-honeycomb structure. The cross section area is varied along the position on the pore as shown in Figs. 1(e) and (f) which show the cross sections at the line "c" and the line "d" on the pore in Fig. 1(d). Since the magnitude of x_c or x_d can be determined from the position of x_1 in Fig. 1(d), the cross section area in the unit area is obtained as following;

$$A=\begin{cases}\dfrac{bh}{\rho}(\rho-2\sqrt{r^2-x_1^2}) & (-r\le x_1\le r)\\ bh & (-a\le x_1\le -r \ or \ r\le x_1\le a)\end{cases} \qquad (2)$$

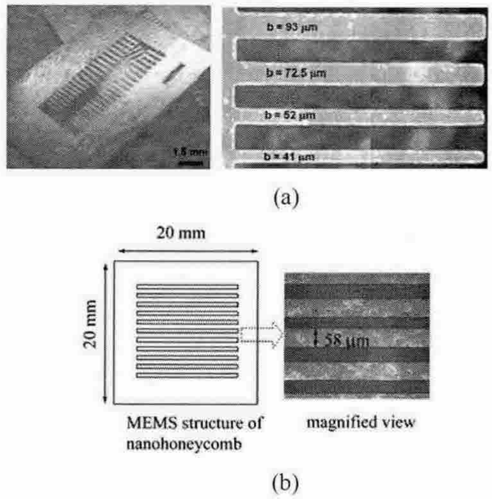


Fig. 2 (a) Bending specimens and (b) tensile specimens fabricated by UV lithographic process.

where $2a=\sqrt{3}/2\rho$ because $2a$ is the same with the height of the triangular unit cell. From the definition, the area moment of inertia of the nanohoneycomb structure for the arbitrary position x_1 can be calculated by

$$I_{33}=\begin{cases}\displaystyle\int_{-h/2}^{h/2} x_2^2\frac{b}{\rho}(\rho-2\sqrt{r^2-x_1^2})dx_2 & (-r\le x_1\le r)\\ \dfrac{bh^3}{12} & (-a\le x_1\le -r \ or \ r\le x_1\le a)\end{cases} \qquad (3)$$

This study assumes the area moment of inertia of bulky nanohoneycomb structures to be the mean value of the area moment of inertia in the unit area. The mean value is then calculated as follows;

By combining Eq. (1) and Eq. (3), the area moment of inertia of nanohoneycomb structures can be determined from

$$\overline{I}_{33}=\frac{bh^3}{12}\times(1-P) \qquad (4)$$

3. Experiments

AAO films are fabricated as nanohoneycomb structures. Experimental details of the fabrication of highly ordered AAO films in 0.3M oxalic acid have been reported elsewhere [4, 5]. The specimens for the bending tests in AFM and the tensile test in Nano-UTM were fabricated by UV lithographic process and wet-etching in a 5 wt% H_2PO_4 solution at 20C for 8h [6]. Fig. 2 shows the specimens for bending tests and tensile tests.

During the bending tests, the PZT scanner moves up and down, and the photodetector measures the vertical deflection, D_A, of the AFM cantilever. The vertical deflection, D_B, of the nanohoneycomb beam can be then calculated by $(D_s - D_a)$ where D_s is the vertical movement of the PZT scanner from the contact position of the AFM cantilever and the

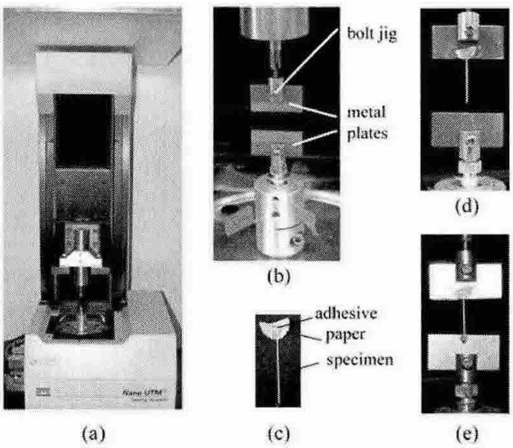
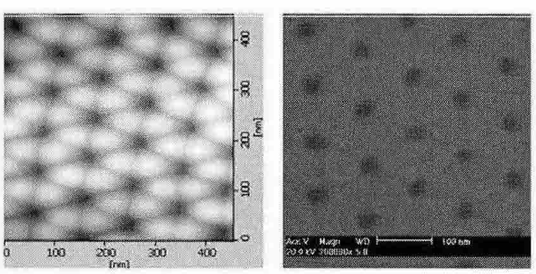


Fig. 3 Mounting procedure of nanohoneycomb specimen to the Nano-UTM for the tensile test.
(a) Nano-UTM for the tensile test (b) metal plate fastened on the bolt jigs. (c) nanohoneycomb specimen attached on the piece of paper by an adhesive. (d) nanohoneycomb specimen attached on the upper metal plate. (e) nanohoneycomb structure mounted on the metal plate gripper after aligning.



(a) (b)

Fig. 4 (a) AFM and (b) SEM image of nano-honeycomb structure with the pore diameter of 31 nm and the interpore distance of 100 nm.

nanohoneycomb beam. The load applied at the end of the nanohoneycomb beam is calculated by

$$F = k_b \cdot D_A \tag{5}$$

where k_b is the bending stiffness of the AFM cantilever. This study used a diamond coated AFM cantilever with the bending stiffness of 45 N/m and SPA 400 AFM fabricated by Seiko company. Fig. 3 shows the procedure of tensile tests in Nano-UTM.

4. Results

Fig. 4 shows the top section view of the

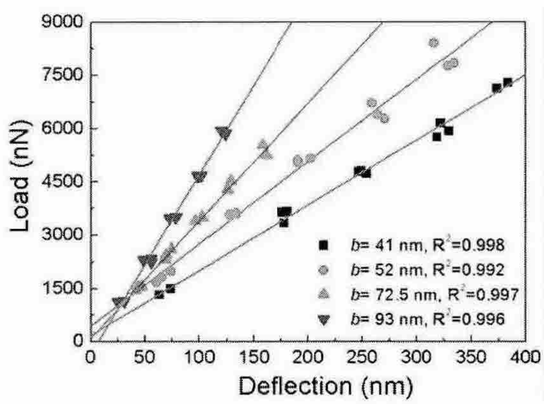


Fig. 5 Load-deflection curves measured from bending tests in AFM. *b* is the width of the rectangular nanohoneycomb beam structures.

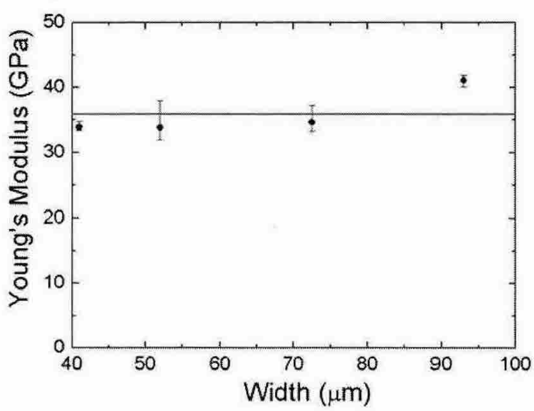


Fig. 6 Young's modulus of nanohoneycomb structures measured from bending tests. The mean value of the Young's moduli is 35.9 GPa.

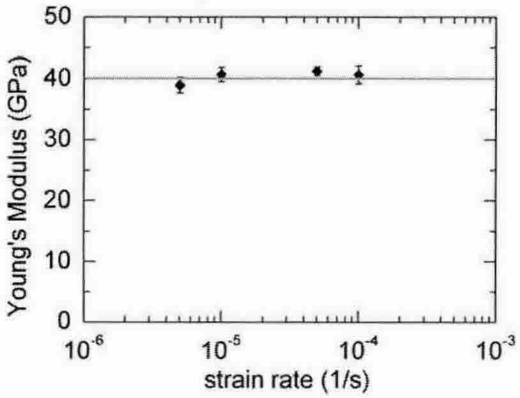


Fig. 8 Young's modulus of nanohoneycomb structures measured from tensile tests. The mean value of the Young's moduli is 40.0 GPa.

nanohoneycomb structure measured from AFM and SEM. The pore diameter of the nanohoneycomb structure was 31 nm, and the interpore distance was 100 nm, so that its porosity was 0.0872 from Eq. (1). The load-deflection curves of the rectangular nanohoneycomb beam structures were obtained from bending tests in AFM as shown in Fig. 5. The ratios between force and the displacement the specimens were obtained in Fig. 5, the Young's moduli in the transverse direction of hole thickness of the nanohoneycomb structure were determined in Fig. 6. There were no effects of the width on the Young's modulus. The mean value of the Young's moduli was 35.9 GPa. The Young's modulus measured using nano UTM is shown in Fig. 8. These values had no effect on the strain rate, and the mean value was 40.0 GPa. The results of bending tests and tensile tests showed good agreement within 10 % error, hence that it confirmed the applicability of Eq. (4) in the nanohoneycomb structures.

5. Conclusions

This study determined the Young's modulus of the nanohoneycomb structure. The area moment of inertia of the nanohoneycomb structure was assumed to be the mean value of the area moment of inertia along the position on the pore. The Young's moduli of the nanohoneycomb structures calculated from the bending tests in AFM and from the tensile tests in Nano-UTM showed good agreements within 10 % errors.

Acknowledgements

This work was supported by the National R&D Program (M10214000191-02B1500-02910) of the Korea Institute of Science and Technology Evaluation and Planning/Ministry of Science & Technology.

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