Size effect of large deformable nanopillar by focused-ion-beam chemical vapor deposition

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Abstract. Nanoscopic fabrication technique has been achieved by the direct deposition methods using focused-ion-beam chemical vapor deposition (FIB-CVD). The nanopillar fabricated by FIB-CVD consists of an outer amorphous carbon ring and a inner gallium core. We developed the original double-cantilever (DC) bending test using two pillars rigidly connected by the exposure of a focused electron beam in a scanning electron microscope. The obtained deflection curves suggest that nanopillars have the size dependence to the mechanical response. The pillar with the diameter over 180 nm exhibits a wide region of stiffness weakening after linear response and then becomes extremely hardened at a large deflection. Thus, the pillar intrinsically possesses much more flexibility for bending without any fracturing. The accuracy of a DC testing is also discussed by estimating the bending rigidities of nanopillars, comparing to those obtained by resonance frequency tests.

1. Introduction
Focused ion beam-induced chemical vapor deposition (FIB-CVD) has distinctive advantages, such as much higher deposition rate, in the fabrication of complicated structures. Some delicate microstructures have been actually designed with this micro-beam technology [1,2]. They are composed of the deposited beam members made from amorphous carbon (a-C). Therefore, the overall structural characteristics are, in principle, determined by the mechanical behavior of the member. It has been reported that the a-C pillars have the cross section with a Ga core in nanosized diameter [3]. Thus, the mechanical response of the whole a-C pillar is closely related to the fundamental physical properties as density and Young’s modulus, and it has already been investigated by the dynamic resonance vibration and the quasi-static small bending with the piezoactuator [4-6]. We here propose the simple deflection test with a rigid connection of two pillars using a focused electron-beam (EB) in a scanning electron microscope (SEM), and develop the evaluation technique for not the infinitesimal deformation but the large deflection of a-C pillars, which intrinsically equip the remarkable large deformability [7]. In this paper, the bending tests of nanopillars under two conditions are conducted, the parameters of which are the cross-sectional diameter and longitudinal length of a-C pillars, and the size effect of the pillars for large deflection is discussed. In addition, the bending rigidities of pillars
are estimated by the results of proposed bending tests, and the accuracy of this test is evaluated by comparing the obtained bending rigidities with that by the resonance vibration tests.

2. Double cantilever test

Nanopillars are grown by a commercially FIB-CVD (SMI9200, Seiko Instruments, Inc.) with the Ga⁺ ion beam in the supplied phenanthrene vapor (C_{14}H_{10}) precursor gas. In this study, double-cantilever specimens for bending tests are assembled by connecting these two pillars with almost equal cross-sectional diameters (d) and equal longitudinal lengths (l), as shown in figure 1(a). We call it double-cantilever model (DC model). Nanopillars are joined at intersection point of two pillars by irradiation using a focused EB in a SEM (JEOL JSM5310). The residual hydrocarbons from the pump oil (so-called contamination) are absorbed on the pillars’ intersection in the process of decomposition by the EB [8,9].

In the DC model, the commercially available AFM cantilever, as shown in figure 1(a), is made of silicon nitride (SiN) coated with Au (BL-RC150VB, Olympus Corporation, Inc.), and it is used in the present bending tests with a spring constant of \( k = 0.006 \) N/m. The longitudinal length (L), with (b), and thickness (t) of the cantilever are 100(±10), 30(±2), and 0.18(±0.04) \( \mu m \), respectively. The flexural and tensile rigidities are assumed \( E_2I_2 = kL^3/3 = 2.0 \times 10^{-15} \) Nm² and \( E_2A_2 = 1.15 \) N using Young’s moduli of SiN and Au (234 GPa and 78 GPa).

We demonstrated large deflection analyses by applying a load to the above DC model, which encompasses the AFM cantilever specified to provide the measurable deflection. This structure seems to be framed rigidly because the connection is exposed to a focused EB for sufficiently long time. The large deflection tests are carried out to displace the AFM cantilever in a vertical direction. The deflections \( w_1 \) of the horizontal pillar and displacement \( w_2 \) of the AFM cantilever are then measured by in-situ observation using SEM. Figures 1(b) and (c) show SEM images of the bending test. The SEM image, as shown in figure 1(b), is the magnified view of a rigid joint, and the deflection \( w_1 \) can be measured. On the other hand, the SEM image, as shown in figure 1(c), is the overall DC testing view, and the deflection \( w_2 \) can be measured from this figure. The microstructure was deformed largely beyond a small bending by manipulating the cantilever. Subsequently, it has achieved the tension-dominated shape, in which two members have been linearly oriented (see figures 1(b) and (c)). It is noted that the joint connected by two pillars maintained a right angle even though the joint was subjected to large deflection.

![Figure 1](image)

**Figure 1.** (a) Schematic drawing of a DC bending test, (b), (c) SEM images show the magnified view of a rigid joint and the overall DC testing view.

3. Results and discussion

3.1. Size effect of pillar diameters

Figures 2 show three large deflection curves among three types of pillars (\( d = 160, 180 \) and 210 nm, and \( l = 15 \) \( \mu m \)), which are plotted as the relationship between the normalized deflection of \( w_1/l \) and the normalized one of \( w_2/L \). The experiments are conducted by two ways of bending to evaluate the
elasticity of a-C pillars, one of which is only loading (open symbols), and the other is loading and then unloading (solid symbols). The large deflection curves indicate the weakening of stiffness in the medium-deformation region after linear response, which is remarkably observed for the diameter over 180 nm (see figures 2(b) and (c)). Thus, the pillar has the size dependence that the bending rigidity tends to decrease after linear response as the diameter increases. At finitely deformation, all pillars become hardened due to the geometrical nonlinearity, because the nonlinear elastic of the pillar suggests that tensile rigidity of the pillar contributes more to the large deflection than the flexural rigidity.

In these experiments, the linear and large (except ultimate) deflection behaviors of pillars conform well to the fitted curves of third-order polynomials: Solid lines are fitted by all experimental data. In addition, the dashed lines are the linear elastic solutions obtained by these fitted curves, and each $\alpha^*$ is the gradient of this line. The linear elastic properties of nanopillars are discussed later.

Figure 2. Relationship between normalized deflection of the cantilever and one of the pillar for some diameters (160, 180, and 210 nm). The open symbol represents an experimental value for only loading, and the solid one represents the value of a loading and unloading test. The solid curve here is the cubic polynomial through the origin, fitted by all experimental data. The dashed line is the linear elastic solution obtained by the fitted curve, and $\alpha^*$ is the gradient of this line.

3.2. Size effect of pillar length
We next conduct the DC test by applying the different lengths of pillars with same diameter. Figure 3 shows three large deflection curves among three types of pillars \((d=220 \text{ nm}, \text{ and } l=15, 20 \text{ and } 25 \mu \text{m})\), and it indicates that nanopillars with short lengths exhibit the same weakening of stiffness as shown in figures 2(b) and (c).

Figure 3. Relationship between normalized deflection of cantilever and one of pillars \((d=220 \text{ nm and } l=15, 20, 25 \mu \text{m})\). The solid curve is the cubic polynomial through the origin, fitted by experimental data. The dashed line is the linear elastic solution.

All these results indicate that nanopillars with the diameter within a range of 180–220 nm have loss of stiffness for large deflection, which depends on a ratio of diameter \((d)\) to length \((l)\). This is partly because the internal structure of the a-C pillar changes due to the atomistic deformation manner with covalent interaction based on \(sp^2\)-\(sp^3\) bonding. It is sure that the change in a binding mode influence the local density of the pillar, and it express the material nonlinearity in the process of deformation.

3.3. Estimations of the linear elastic properties of nanopillars

When the DC model deforms infinitesimally, the analytical solution derived from the elementary beam theory can afford two Young’s moduli \(E_1\) and \(E_2\): \(E_1\) is Young’s modulus of the a-C pillar and \(E_2\) is that of the AFM cantilever. The parameter \(E^*\), which is defined by \(E^*=E_1I_1/E_2I_2\), can be derived from the elementary beam theory, as follows:

\[
E^* = Z + \frac{1}{2} L^* \alpha^*, \quad Z = \frac{1}{16} \left[ 10 L^* - 3 L'^2 + (3 L'^2 - 14 L'^2) \alpha^* \right],
\]

where \(L^*=l/L\), and \(\alpha^*\) is equal to \(w_2/w_1 L\) and represents the slope in the infinitesimal region of large deflection curve, as shown in figure 2 and figure 3. The flexural rigidity \(E_2I_2\) of the commercial AFM cantilever used in the DC model is the known value. Therefore, the flexural rigidity \(E_1I_1\) of a-C pillars can be obtained from equation (1). Figure 4 compares the obtained flexural rigidities from the present bending tests with those from the previous resonant vibration testing [7]. The solid circle corresponds the bending rigidity derived from the resonance frequency \(f\), as follows:

\[
f = \frac{\gamma_n^2 d}{2 \pi f^2 \sqrt{E_1 / 16 \rho}},
\]

where \(\gamma_n\) is the coefficient of the lowest resonance mode. The details of equation (2) are described in reference [7]. These flexural rigidities for samples with diameters 160 to 220 nm agree well with the predicted value of resonant vibration frequency. Therefore, the proposed DC testing seems to have high accuracy for large deformation region.
4. Conclusions
We have proposed the experimental technique called double cantilever test to measure large deflection of the pillar manufactured by FIB-CVD. Two identical pillars with the same diameter and the same length are rigidly jointed at the interconnected point by the focused electron beam. Results from the proposed testing verified the size dependence of the deposited pillar to the mechanical response. The pillar exhibits weakening of stiffness in the medium deformation region after linear response and this material nonlinearity depends on a ratio of pillar’s diameter to length. Some considerable factors of the stiffness weakening are the properly atomistic deformation manner with covalent interaction based on sp2-sp3 bonding and also the density of the pillar.

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References
[1] Matsui S, Kaito K, Fujita J, Komura M, Kanda K and Haruyama Y 2000 J. Vac. Sci. Technol. B18 3168
[2] Matsui S 2007 Nucl. Instrum. Methods Phys. Res. B257 758
[3] Morita T, Kometani R, Watanabe K., Kanda K, Hoshino T, Kondo K, Kaito T, Ichihashi T, Fujita J, Ishida M, Ochiai Y, Tajima T and Matsui S 2003 J. Vac. Sci. Technol. B21 2737
[4] Fujita J, Ishida M, Sakamoto T, Ochiai Y, Kaito T and Matsui S 2001 J. Vac. Sci. Technol. B19 2834
[5] Ishida M, Fujita J, Sakamoto T, and Ochiai Y 2002 J. Vac. Sci. Technol. B20 2784
[6] Fujita J, Ishida M, Ichihashi T, Ochiai Y, Kaito T and Matsui S 2003 Nucl. Instrum. Methods Phys. Res. B206 472
[7] Shibutani Y and Yoshioka T 2008 J. Vac. Sci. Technol. B26 201
[8] Koops H W, Kretz J, Rudolph M., Weber M, Dahm G and Lee L 1994 Jpn. J. Appl. Phys. 33 7099
[9] Nishio M, Sawaya S, Akita S and Nakayama Y 2005 J. Vac. Sci. Technol. B23 1975