Elastic double structure of amorphous carbon pillars grown by FIB-CVD

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A free standing 3D-submicron structure[1] of amorphous carbon can be grown by a focused ion beam (FIB) with a carbon precursor. The characteristic feature of the amorphous carbon pillars was the fact that the gallium primary ions condensed in a core region, but the outer shell of the structure only consisted of amorphous carbon[2] (as shown is Fig. 1). We previously reported that the Young's modulus of the FIB-CVD pillar varied from 80 to 600 GPa, depending on growth conditions such as growth speed, source-gas pressure, and accelerating voltage[3]. In the present study, we found that the FIB-CVD pillar, which had an apparent Young's modulus of about 100 GPa, formed an elastically double structure. And the core region of this structure coexists with gallium and had extremely high Young's modulus over 300 GPa, but its outer amorphous carbon shell had a very low Young's modulus of below 50 GPa as shown in the structural model in Fig. 2.

We measured the young's modulus on a series of tilted FIB-CVD pillars, as shown in Fig. 3. All the pillars were fabricated by 1 pA ion beam current at 30 keV acceleration voltage with phenanthrene ($C_{14}H_{10}$) source gas. Static point beam irradiation produced a perpendicular pillar, and beam scanning produced a tilted pillar with inclination angle θ to the scanning direction. Figure 4 shows the results of measured Young's modulus of the tilted pillar. Two methods were used to determine Young's modulus: calculation from pillar deflection against a standard AFM cantilever[4] (indicated by large circles) and derivation from the resonant vibration frequency of the pillar[2] (indicated by solid diamonds). Note that both measurement methods were confirmed to give basically the same results within the tolerance of the density estimation error. The width of the error bar thus suggests the case that gallium is not incorporated, and the upper limit indicates the case that all irradiated gallium atoms are trapped in the pillar. The important point is that Young's modulus increases with decreasing pillar inclination angle θ, but the pillar diameter decreases with decreasing θ. However, the size of the core region was not changed, since the core size was not determined by scanning speed but by ion penetration depth as a function of accelerating voltage. If we assume a simple model of the pillar as a double elastic cylinder, the mean Young's modulus can be written as $E = (E_C d_C^4 + E_S (d_S^4 - d_C^4)) / d_S^4$, where core radius is d_C, shell radius is d_S, Young's modulus of the core is E_C and Young modulus of the shell is E_S. The solid line for Young's modulus was calculated from the double structure model as shown by the dashed line in Fig. 4, under the assumption of Ec=300 GPa and Es=50 GPa. Even though the model is very simple, it explains the behavior of the Young's modulus of a tilted pillar well. Such low Young’s modulus for amorphous carbon at the outer shell agrees well with the results for a EB-CVD pillar[3].

We also confirmed that etching treatment of the outer shell of the pillar by oxygen plasma dramatically increased Young's modulus. The tested pillar initially had thickness of 105 nm. Oxygen plasma etching was performed, and finally a total of four minutes etching reduced the thickness to be 85 nm. These etching results are summarized in Fig. 5. Here, the as-grown FIB-CVD pillar had apparent Young's modulus (measured by the resonant method) of about 140 GPa. However, the reduction of the thickness dramatically increased Young's modulus up to 300 GPa despite etching depth of only 10 nm. The authors thus conclude that the measured data could be well explained by our elastic double-structure model, assuming core diameter of 80 nm with Young's modulus of 300 GPa.

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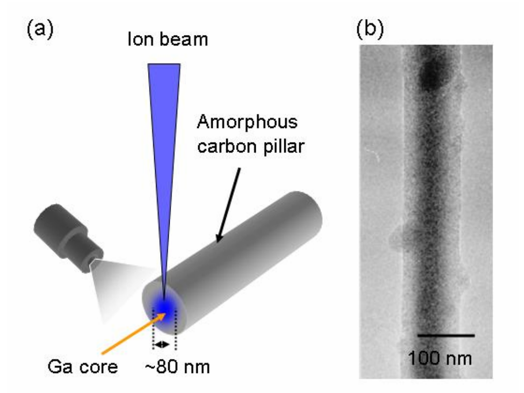

Fig. 1: Schematics of FIB-CVD and HR-TEM of pillar.

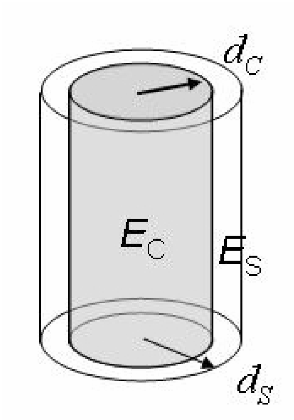

Fig. 2: Model of elastic double structure image for FIB-CVD pillar.

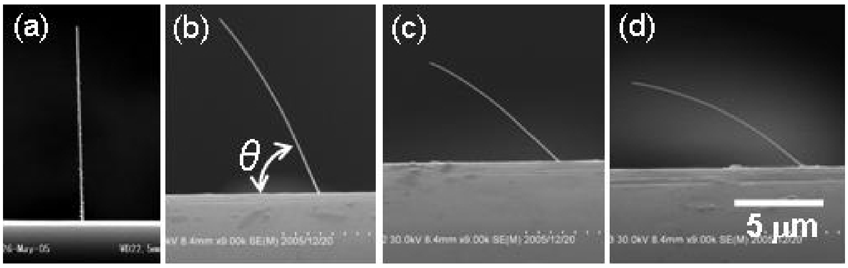

Fig. 3: SEM images of tilted pillar. (a) perpendicular, (b) θ=56°, (c)θ=45°, (d) θ=34°, inclination angle θ was defined as the angle between the substrate and pillar as shown in (b).

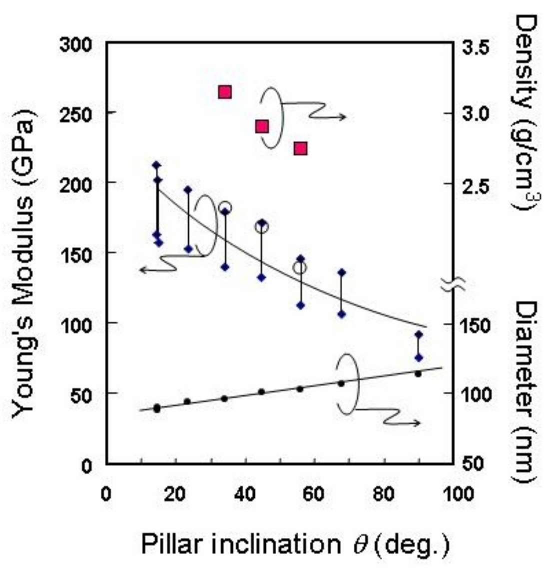

Fig. 4: Dependencies of Young's moduli, pillar diameter, and mean density on inclination angle.

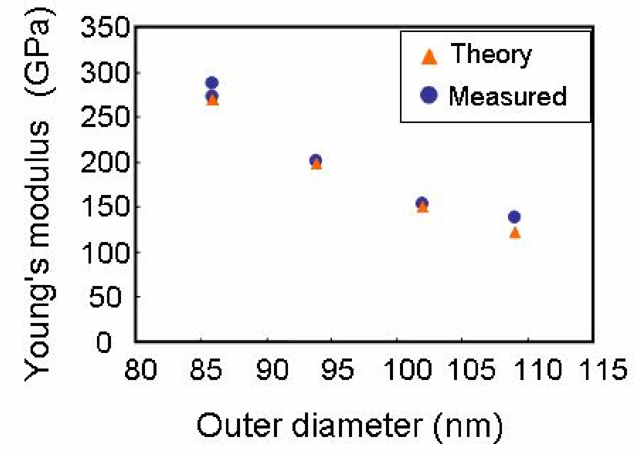

Fig. 5: Dependencies of Young's moduli on the reduction of the outer diameter by plasma etching.