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In-situ micro bend testing of SiC and the effects of Ga^+ ion damage

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Abstract. The Young's modulus of 6H single crystal silicon carbide (SiC) was tested with micro cantilevers that had a range of cross-sectional dimensions with surfaces cleaned under different accelerating voltages of Ga^+ beam. A clear size effect is seen with Young's modulus decreasing as the cross-sectional area reduces. One of the possible reasons for such size effect is the Ga^+ induced damage on all surfaces of the cantilever. Transmission electron microscopy (TEM) was used to analyse the degree of damage, and the measurements of damage is compared to predictions by SRIM irradiation simulation.

1. Introduction

Micro mechanical testing has been widely enployed for the characterisation of materials used for micro- and nano- electro-mechanical systems (MEMS/NEMS). Young's modulus size effect is of interest because it is an essential input for finite element method (FEM) modelling [1]. Focused ion beam (FIB) has the ability to machine a range of geometries for testing bulk mechanical properties along specific crystallographic orientations, or properties of interesting regions such as grain boundaries [2], [3]. However, many micro mechanical tests have failed to match theoretically predicted properties, such as Young's modulus and breaking strength, despite their size being sufficiently small to contain few or no dislocations or flaws [4], [5]. A likely factor is the changes introduced by ion milling. Such changes may include the creation of an amorphous layer causing discrepancies in the measurement of the geometry of micro mechanical samples and the introduction of surface dislocations, point defects, and/or residual stresses [6]. SiC has great potential for NEMS/MEMS, especially for uses at high temperatures or in harsh environments. Single crystal SiC typically deforms elastically in bending until failure, is electrically conductive, and can be milled well in the FIB, making SiC an ideal model material for exploring the size effect and surface damage by a Ga^+ ions.

2. Methods / materials

The micro cantilever beams were prepared using a FIB system (Nova 600 Nanolabs, FEI, USA) in a single crystal of 6H SiC (Marketech International, USA). To ensure the top face of the beams were orientated parallel to the 0001 plane a three stage milling technique was developed. Once milled to the required dimensions all faces were finished by a Ga^+ beam under three different accelerating voltages and currents: 30 keV and 0.3 nA followed by 10 keV and 50pA for 20s; 30 keV and 0.3 nA followed by 5keV and 70 pA for 30s. Three beams were prepared for each finishing conditions. Bending test was completed inside the FIB through loading the micro beam with a micro probe (FMT 120, Kleindiek

Nanotechnik, Germany). The Y stage of the microscope was indexed towards the indenter bending the cantilever. A video and time stamped images were recorded to link force and displacement.

To gain evidence of the surface modification by the Ga^+, a cross sectional lift-out foil was prepared for TEM analysis from a surface finished with Ga^+ beam under each of the three aforementioned finishing conditions. Microstructure characterisation of the cross-section was performed using a FEI Tecnai F20 G2 S-Twin field emission gun (FEG) scanning TEM (STEM). Selected area electron diffraction patterns and conventional high-resolution electron micrographs (HREM) of the SiC were recorded. Energy dispersive X-ray (EDX) spectroscopy line scans were conducted using an Oxford Instruments X-Max 80mm2 TLE detector.

The Stopping and Range of Ions in Solids (SRIM, 2008, USA) calculations were completed with full cascade simulations up to 250 ions [7]. SiC was modelled with a density of 3.16g/cm^3 and a compound coefficient of 1, Gallium was model with a mass of 68.93 amu and an accelerating voltages of 30 keV, 10 keV and 5 keV [7]. Modelling provide ion range, straggle, sputter yield and vacancies per ion at a range of incident angles.

3. Results and discussion

Figure 1 shows a typical bend test in progress. From these still images the displacement and contact point of the beam were measured.

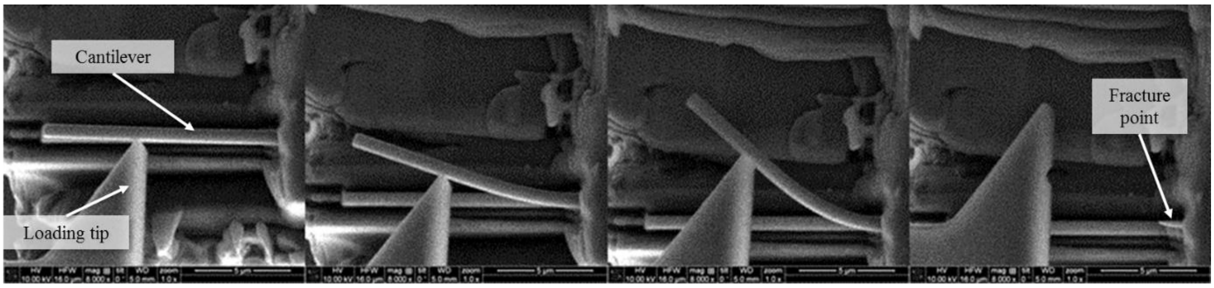

Figure 1. micro bend testing in progress left to right showing deformation to failure

From the force displacement data, small deflection beam equations were used to determine modulus and failure strength. Figure 2(a) summarizes the Young's modulus measurements obtained from SiC micro-cantilevers that have different cross-sectional areas and have undergone different surface cleaning conditions.

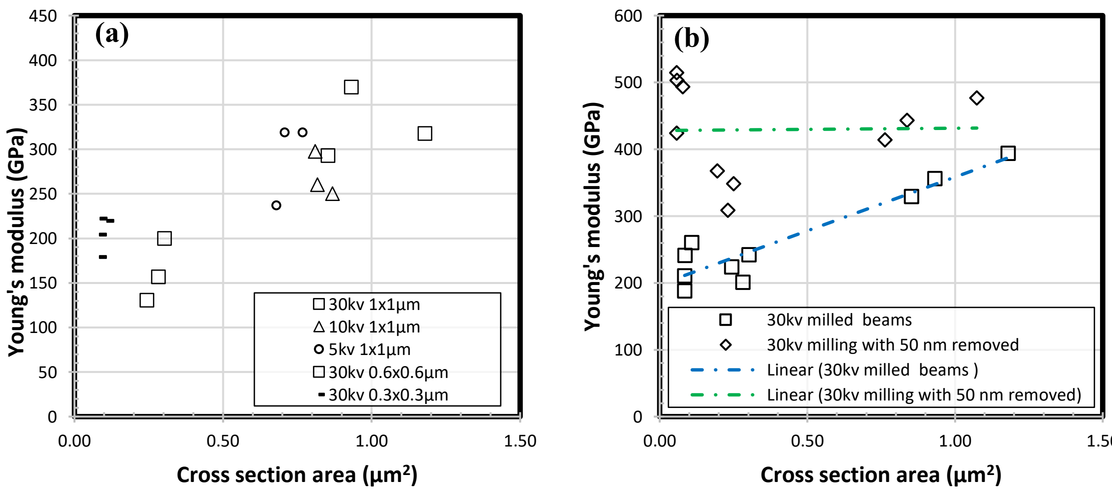

Figure 2. (a) micro bend test results modulus against cross section (b) adjusted micro bend tests data with original 30 kV data and beams with 50 nm removed from breadth and width

Young's modulus decreases approximately linearly as the cross-section area of a cantilever beam becomes smaller. The bulk modulus of SiC is cited as > 415 GPa [8]. Among our measurements, the highest Young's modulus, 393.98 GPa, was measured from beams with a cross-sectional of 1.18 μm^2, and the lowest, 187.84 GPa, from those with a cross sectional area of 0.086 μm^2. Lower accelerating voltage cleaning did not seem to have a strong impact on the size effect, as shown in Figure 2(a).

TEM analysis shows that the thickness of damaged layer by each cleaning routine was similar, 42-51 nm. The gallium ion beam produced an amorphous layer on the surface with a sharp transition between the damage layer and the bulk material, as shown in Figure 3. SRIM simulations predicted an ion penetration range of 19 nm at 30 keV (as shown in Figure 3(b)), nearly 3 times smaller than measured in the sample. EDX in STEM mode, shows the damaged layer contained around 1.5 At % Gallium at 21 nm depth from the surface, this matches well with the SRIM simulations.

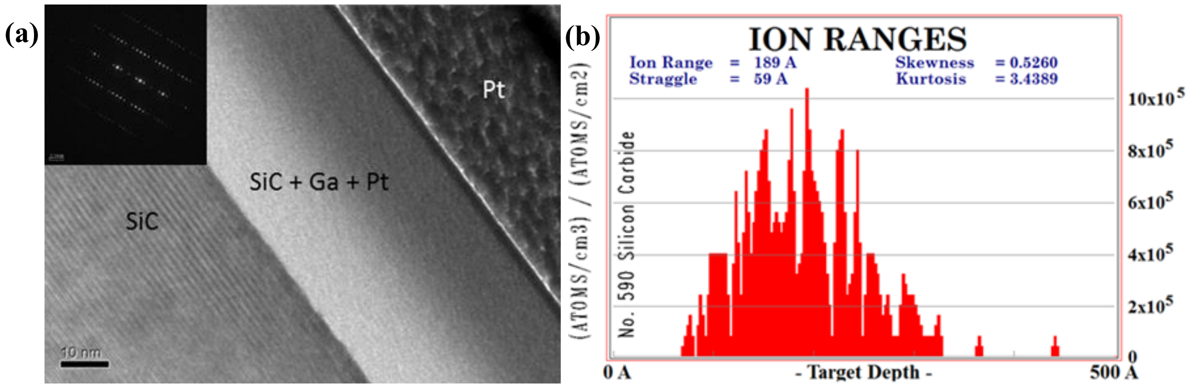

Figure 3 (a) shows HRTEM image of silicon carbide surface with the amorphous layer and platinum protective coating. (b) SRIM simulation of 30 kV gallium ion implantation distribution in silicon carbide

The mechanical performance of the damaged level is still under investigation, we believe that this layer likely contributes to the observed Young's modulus size effect. Other factors may contribute the size effect. Based on the TEM analysis, 50 nm was removed from the breadth and thickness of the micro beams and the modulus calculations were reprocessed to simulate the effects of the amorphous layer on the results of the micro bend test results. The effects of reprocessing the measurements from cantilevers with surfaces finished under 30 keV can be seen in Figure 2(b) where the modulus of the beams is increased closer to literature values. It was noted that cantilevers with a cross section of 0.3x0.3 μm exhibited plastic deformation at high loads. These beams give overestimated Young's modulus of the SiC, and the likely reason is due to the inability of the small deflection equations dealing with plastic deformation.

4. Conclusions

It was identified that a size effect is present in SiC when testing micro beams fabricated in the FIB. The source of the size effect may be from the amorphous zone developed on the surface of the material. Cleaning with low accelerating voltages did not significantly improve mechanical properties of the micro cantilevers. Based on SRIM calculations it is fair to assume that prolonged low voltage cleaning might have likely reduced the ion damage layer, however this strategy would also lead to the modification of the micro beams shape. Plasticity was observed in micro beams with cross sections below 0.3x0.3 μm, and the modulus of these micro beams was over estimated if the small elastic deflection equations are used. SRIM simulations did not accurately predict the extent of the damage in the SiC surface but were accurate in predicting the implantation depth of the gallium ions.

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