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SPECIMEN PREPARATION FOR TRANSVERSE MODULUS MEASUREMENT OF CARBON FIBRES USING FOCUSED ION BEAM

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ABSTRACT

Transverse Young's modulus of carbon fibres is an important material property for micromechanical modeling and design of carbon fibre reinforced composites. To accurately measure their transverse Young's modulus is of special importance for applications in novel multifunctional devices, such as structural composite batteries. However, experimental measurement of their transverse Young's modulus is still largely lacking due to experimental challenges. In this study, we successfully prepared high quality longitudinal cross sections from a commercial carbon fibre using precision ion milling in a combined focused ion beam and scanning electron microscope (FIB/SEM) instrument. These cross sections were then directly used in an atomic force microscope (AFM) and a nanoindentation equipment to measure the transverse Young's modulus. Here, the entire procedure is described in detail. In particular, the most critical aspects for specimen preparation are identified and discussed.

1 INTRODUCTION

Carbon fibres possess a unique combination of lightweight and excellent mechanical properties (high stiffness, high tensile strength). They are widely used to reinforce polymer matrix in aircraft, high-end sports equipment, and automobile [1,2]. Accurate values of their elastic constants are essential parameters for simulation-based prediction of mechanical properties via modeling, as well as designing of novel carbon fibre based composites. The properties related to the axial direction of the carbon fibres largely determine the mechanical behaviour of a composite. Additionally, they are relatively easy to obtain. Therefore, axial properties are carefully measured and well known. On the other hand, although transverse Young's modulus of carbon fibres is also an important material property for micromechanical modeling of carbon fibre reinforced composites, it is seldom reported, essentially due to experimental difficulties. Very often an approximate value of transverse modulus is used, deduced by indirectly measurement. For some novel multifunctional composites, an accurate value of transverse modulus is critical.

Recently, carbon fibres have been used in multifunctional composite devices, for instance structural composite batteries. These batteries integrate energy storage capabilities to structural components. They are regarded as a key enabling technology in realizing substantial weight savings on the system level for electrified transportation, particularly in electrified vehicles. Carbon fibres are widely used as reinforcements in polymer composites, while graphite powders are widely used as negative electrodes in batteries. Thus, using carbon fibres as negative electrodes, together with solid electrolyte and other components, one can build the so-called structural composite batteries [3]. Imagine the doors and hoods of an electric car also store energy!

A structural composite battery is rechargeable. Carbon fibres play dual roles here, reinforcements (as in carbon fibre composites) and negative electrodes (as in batteries); and the polymer matrix is also multifunctional, bonding the fibres together and transferring loads between them (as in carbon fibre

composites), and also functioning as electrolytes (as in batteries). When charging, Li ions can be inserted into the graphene layers and form intercalation compounds under low electrical potential. Given that transverse Young's modulus of graphite can increase up to three times after lithiation [4], a remarkable increase in transverse Young's modulus is also expected in carbon fibres. Thus, an accurate measurement of transverse Young's modulus is essential for designing structural composite batteries [5].

Direct measurement of transverse Young's modulus on a single carbon fibre is desirable [6]. Both nanoindentation [7,8] and atomic force microscope (AFM) are promising techniques. However, there are several challenges related to preparing specimens for nanoindentation and AFM measurement on the tiny carbon fibres ($\sim 5 \mu\text{m}$ in diameter). First, the surface must be parallel to the fibre axis. Otherwise, the measured results will be interfered by the axial modulus [8]. Second, the surface quality must be high, since rough and/or mechanically damaged surfaces can lead to misleading results. Particularly for AFM, which uses an indentation tip only a few tens of nanometers in size, with an indentation depth of only $\sim 10 \text{ nm}$ [9]. Thus, these requirements exclude the usage of mechanical polishing of carbon fibres embedded in polymer matrix.

In this study, we successfully used precision ion milling in a combined focused ion beam and scanning electron microscope (FIB/SEM) instrument to prepare longitudinal cross section on single carbon fibres, which were carefully mounted on a Si wafer.

2 MATERIALS AND EXPERIMENT

2.1 Materials

A commercially available polyacrylonitrile (PAN)-based carbon fibre, IMS65 (Toho Tenax), was selected for this study, due to its good mechanical properties (stiffness and strength) and excellent overall electrochemical capacities [10, 11].

Si wafer, 2 inches in diameter, was cut and cleaved to approximately $1.0 \times 0.5 \text{ cm}^2$ small pieces using a diamond scribe. The wafer pieces were used to support carbon fibres for the followed FIB/SEM cutting, and AFM and nanoindentation measurement.

2.2 Experiment

2.2.1 Preparation of carbon fibre

Individual IMS65 carbon fibres were cut, separated, and carefully fixed onto a small Si wafer piece using silver glue. In this procedure it is important to place the fibre as close to one of the edges of the wafer as possible. Each single fibre was managed to put within $500\text{--}1000 \mu\text{m}$ from the edge of the wafer. The wafer with a carbon fibre was then put onto an aluminum SEM stub using a double-sided carbon tape, and then fixed onto a sample holder with 45° pre-tilted angle.

2.2.2 FIB/SEM

An FEI Versa 3D FIB/SEM workstation was used to prepare cross sections on the carbon fibres. The angle between the FIB and SEM column is 52° . The workstation is equipped with a gas injection system, which enables electron-beam- and ion-beam-induced Pt deposition onto the sample. The deposited Pt layer has two functions: firstly, it protects the surface from damage induced by the high energy Ga ions; secondly, it adheres and fixes the fibre stably to the Si wafer for AFM and nanoindentation measurement.

The wafer with carbon fibre was put in the FIB/SEM instrument. Note that the edge with the fibre must be positioned toward the ion beam, and the fibre axis should be approximately perpendicular to the ion beam (Fig. 1). In order to properly fix the carbon fibre for AFM and nanoindentation testing, two Pt strips $20 \times 3 \times 1 \mu\text{m}^3$ (length, width and thickness) were deposited on both sides of the carbon

fibre and exactly above the contact line with the Si wafer. The Pt layer facing the ion beam also protects the surface of the carbon fibre from ion beam damage during ion milling.

After Pt deposition, the whole sample stage was tilted $\sim 7^\circ$ until the flat Si wafer surface (mounted on the 45° pre-tilted holder) just became a line, to ensure the ion beam looking parallel the Si wafer (Figure 1). Then at 30 kV a sequence of ion currents, 3 nA, 1 nA, 500 pA, and 100 pA, were used to remove material and polish the sample surface. Finally, the sample was tilted another 5° towards the ion beam. At 5 kV, a beam current of 28 pA was used to mill the entire sample surface for 30 s, to mitigate the damage on the surface area.

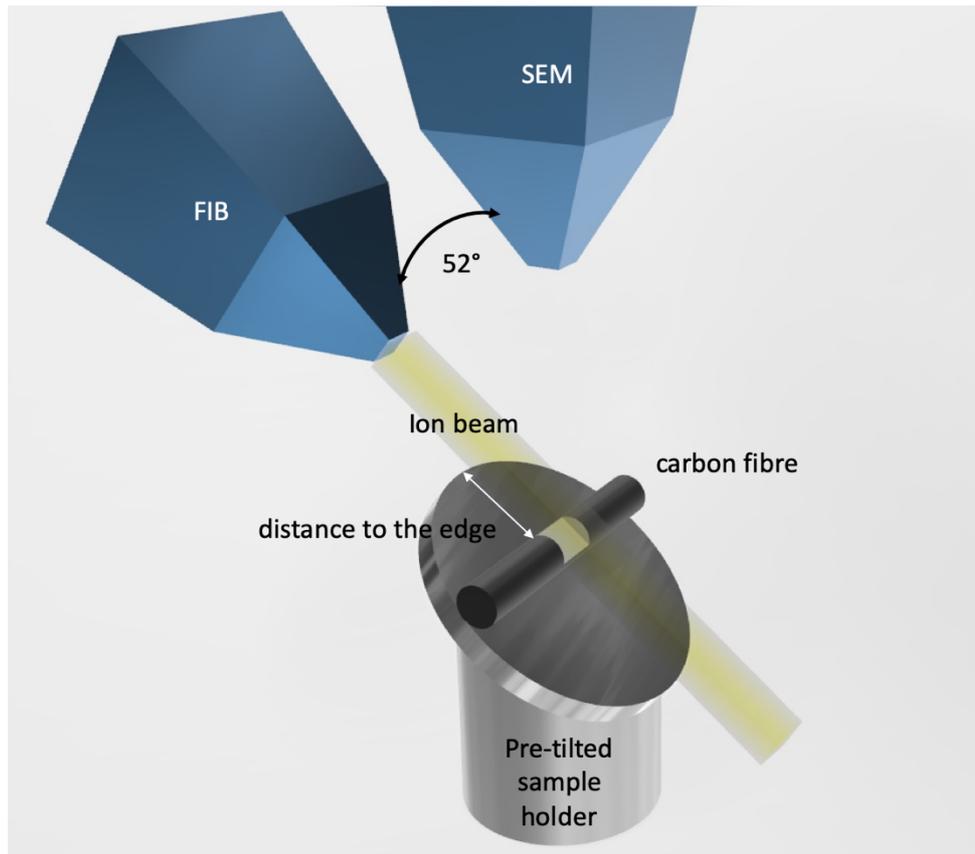


Figure 1: Schematic of positioning of carbon fibre during ion milling in the FIB/SEM sample chamber. Note that the fibre should be placed as close to the edge of the wafer as possible; the edge with the fibre must be positioned toward the ion beam; and the fibre axis should be approximately perpendicular to the ion beam.

3 RESULTS AND DISCUSSION

3.1 Fine polished and parallel cross sections

In order to obtain accurate measurement of transverse modulus of carbon fibres using AFM and nanoindentation, the cross-sectional surface must fulfil a few requirements:

- It must be flat with minimum roughness;
- It must be parallel to the fibre axis.

In the literature, mechanical sample preparation was used on carbon fibre bundles embedded in epoxy to obtain cross-sectional surface, particularly for nanoindentation testing [7,8]. Although cross sections from a large number of carbon fibres were obtained, a few severe challenges were reported. Firstly, the hardness of carbon fibres differs remarkably from that of the polymer matrix. Therefore, it is difficult to obtain good cross-sectional surfaces for both parts, which makes mechanical polishing

demanding. Very often scratches were found on the surface of carbon fibres. The local unevenness is detrimental for particularly AFM testing, where the diameter of the indentation tip is a few tens of nanometres, and the penetration depth is often ~ 10 nm. Secondly, it is impossible to control the angle between the surface and the axis of the carbon fibres using mechanical preparation. For instance, Csanádi et al. used mechanical polishing to prepare transversal cross section (perpendicular to fibre axis) for nanoindentation measurement. The authors then used SEM and AFM to measure the major and minor diameters of the ellipsoid cross section and calculated the inclination angle of the fibre. They reported significant deviation, about 20%, in the indentation modulus for inclined fibres of 14° [8]. Similar effect is expected on the longitudinal cross sections. Measurement on perfectly parallel cross section remarkably simplifies analysis of the indentation results.

Ion milling on a single fibre can overcome the above-mentioned weaknesses of mechanical sample preparation, and achieve smooth and parallel cross sections. Fine polished cross sections were successfully obtained in this study. One example is shown in Figure 2. In the SEM secondary electron image, the flat area with dark contrast is the longitudinal cross section (parallel to the fibre axis) of the carbon fibre. Two Pt layers with bright contrast lie on both sides of the fibre to fix the fibre and also provide stable support during indentation testing.

The FIB prepared cross sections of carbon fibre were used to measure transverse Young's modulus by AFM and nanoindentation. Consistent results were obtained using different indentation methods. Details are reported in [12] in the same proceeding.

3.2 Critical steps in the procedure

In the procedure, caution should be taken to

- position the fibre, and thus enabling ion milling on the fibre;
- mitigate ion damaging on the prepared cross-sectional surface.

For conventional FIB milling, the to-be-removed material lies on the surface or directly under the surface, and normally within $20\ \mu\text{m}$ in depth. In our case, we managed to put each single fibre within $500\text{--}1000\ \mu\text{m}$ from the edge of the Si wafer (Figure 1), which means the focused ion beam must travel closely along the wafer surface for $500\text{--}1000\ \mu\text{m}$ before it hits the fibre. Given that the diameter of the fibre is $5\ \mu\text{m}$, the fibre is placed $1000\ \mu\text{m}$ below the edge, and the ion beam size is $0.12\ \mu\text{m}$ at $30\ \text{kV}$ beam voltage and $3000\ \text{pA}$ beam current [13], a focused ion beam with a virtual convergence angle larger than $\sim 5\ \text{mrad}$ will interfere with the Si wafer. The longer the distance from the edge of the wafer to the fibre, and the larger the ion beam size (e.g. higher beam current), the severer the interference. Indeed, during ion milling we observed that the live image was much more severely distorted, when a high ion current (large aperture, large beam size, and large convergence angle) was used. To position the fibre as close to the edge as possible helps the following ion milling procedure remarkably.

Pt layers are essential to provide very stable support to the fibre during the indentation testing and make solid contact between the fibre and the Si wafer. They also mitigate the negative influence on the roughness of the cross section by the rough surface of the carbon fibre (Figure 2), and thus ensuring a very smooth ion milled surface. It was well established that a rough and heterogenous surface that faces the ion beam often introduces severe "curtaining", strips along the ion milling direction, on the milled surface [14].

Ion beam induced surface damages, e.g. Ga-ion implantation and amorphous layer formation, are likely to affect the measurement results, in particular using AFM, due to the low penetration depth of the AFM cantilever tip into the prepared surface of the carbon fibre during the measurement. To this end, specimens with different ion milling parameters were prepared to study the potential influence. It was found that final cleaning using a low ion voltage can largely reduce the surface damages to a few nanometres. Detailed study on the depth of surface damages using AFM and nanoindentation was reported in another article in the same proceeding [12].

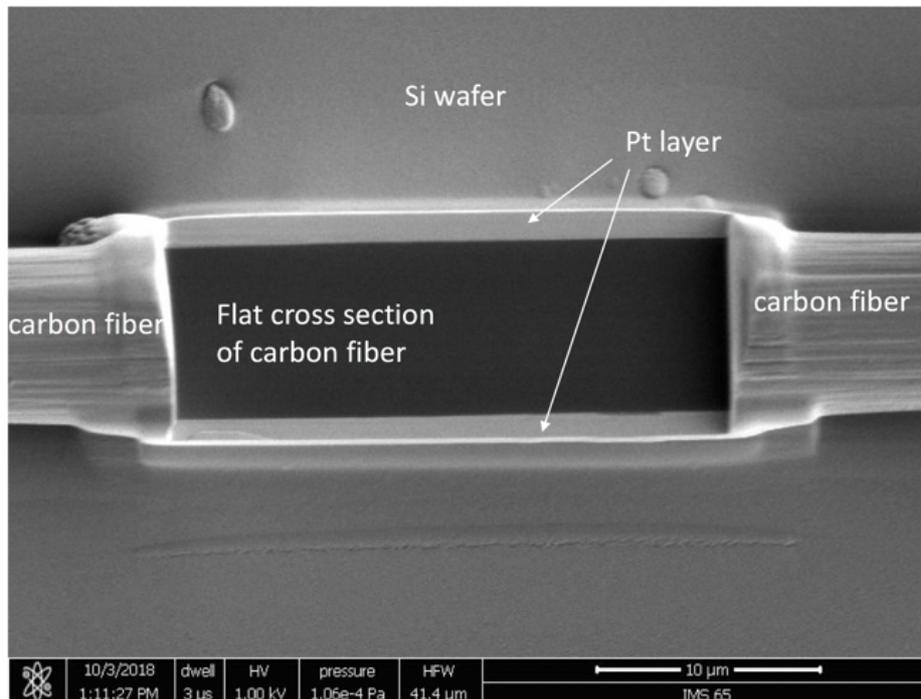


Figure 2: SEM secondary electron micrograph of a FIB prepared longitudinal cross section surface for atomic force microscope and nanoindentation measurement. The flat area with dark contrast is the cross section of the carbon fibre. Two Pt layers with bright contrast lie on both sides of the fibre to fix the fibre and also provide stable support during indentation testing. Note the high roughness of the original carbon fibre surface.

4 SUMMARY

In this study, we successfully prepared high quality longitudinal cross sections on a commercial carbon fibre using precision ion milling in a FIB/SEM instrument. The surface is smooth and parallel to the fibre axis, perfectly suitable for AFM and nanoindentation measurement of transverse Young's modulus. The most critical aspects for the specimen preparation are:

- to position the carbon fibre as close to the Si wafer edge as possible; and
- to final clean the surface with a low ion beam voltage to mitigate ion damage.

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