3D nanomechanical imaging of layered nanocomposite structures

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Nanocomposites are used in a range of applications, but have been shown to be particularly effective for mechanical functions [1]. However, this mechanical function is critically dependant on the structural composition of the nanocomposite material.

Phase contrast Atomic Force Microscopy (AFM) is an imaging mode of amplitude modulated AFM, whereby the AFM cantilever is driven at its resonant frequency and makes periodic contact with the sample surface. The amplitude of the cantilever oscillation is damped by its interaction with the sample and a predefined set-point amplitude is maintained by a feedback loop that alters the separation of the cantilever and sample as it is scanned across the surface. The phase lag between the driving oscillation and the response of the cantilever whilst scanning the sample surface is recorded, along with the standard AFM topographical data. Phase contrast AFM has been used to evaluate changes in surface mechanical behaviour with high spatial resolution [2], and attempts have been made in the literature to model the dynamics of the AFM cantilever in order to quantify the mechanical data obtained [3]. AFM is therefore effective at mapping the mechanical properties at a composite surface, but is hindered by being surface specific and by providing convoluted data due to topographical effects on the phase signal.

Focussed Ion Beam (FIB) microscopy is a developing technique for nanomachining and the preparation of microscopy samples. FIB has been successfully used for patterning of semiconductor materials [4] and preparation of thin samples with parallel sides for TEM analysis [5], and has been integrated into Small Dual Beam (SDB) systems for 3D imaging [6].

The combination of exposing composite surfaces using FIB sectioning and imaging the resulting surfaces with phase contrast AFM will therefore present the opportunity to evaluate the mechanical property distribution in a composite material.

The aims of this work are therefore to demonstrate the effectiveness of utilising FIB preparation with the mechanical imaging capabilities of phase contrast AFM.

The material examined in this study is a laminated polymer tape (polypropylene/elastomer) with a layer thickness ranging from 50 nm to 1 µm. Samples of the nanocomposite tape were first freeze fractured under LN₂ before being coated in gold and mounted within the chamber of a SDB. Cross-sections were milled from the edge of the sample using the FIB, as shown in figure 1b. The newly exposed cross-section was then imaged using AFM, as shown in figure 1c. This sequence of milling and imaging was then repeated to build a 3D mechanical dataset.

A phase image of an exposed composite surface is shown in figure 2b, which clearly shows the layered structure corresponding to the different material composition within the nanocomposite that is not apparent in the topographical data presented in figure 2a.

A series of 2D phase images collected during the progressive removal of material by the FIB is shown in figure 3. A clear correlation is observed between layered features from one slice to the next, while particulate features appear in only one or two adjacent slices. Thus, a complete mechanical evaluation of the nanocomposite structure in 3 dimensions has been achieved.

References

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Figure 1. A schematic representation of the slice and view process. a) Shows a block of layered nanocomposite. b) The FIB is used to mill out a cross-section through the composite block. c) The AFM is used to image the exposed surface.

Figure 2. a) AFM topography image of a layered nanocomposite cross-section. b) Corresponding AFM phase contrast image showing the layered composition of the sample.

Figure 3. Phase contrast AFM images of sequential cross-sections through a composite surface.