Atomic Force Microscopy of Nanomaterials

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Personal Details
After completing my Diploma in Instrumentation and Applied Physics in the Institute of Technology in Tallaght, I decided to complete my degree and enrol in the Degree in Applied Science course in DIT, taking the Physics and Physics Technology options. My main areas of interests include Nuclear Physics, EM and Lasers, Spectroscopy and Systems modelling. Before starting my final year I worked in the FOCAS research institute at DIT as a research assistant. My hobbies include drawing, comics, reading, football and socialising.

Project Summary
The aims of this project were to familiarise myself with the operation of the nanosurf© easyScan E-AFM (atomic force microscope) and use it to view a range of different nanoscopic materials. This firstly involved the calibration of the system with a supplied well-characterised specimen. Once this was completed the material samples were studied, which consisted of nanotubes (single and multi-walled) and gaps in deposited aluminium.

The AFM operates by scanning a tip, attached to a cantilever, across the surface of the samples. Local forces cause a deflection in the cantilever, which obeys Hooke’s Law. This is measured by reflecting a laser off the back of the cantilever and onto a 4-quadrant photodetector. This AFM operates in the contact mode, which means the tip actually stays in contact with the surface. The tip is scanned across the surface at a constant average height.

The samples used in this project were made using a number of techniques. This includes evaporation, using the Edwards Evaporator Auto 306, and drop casting. The evaporation was used to deposit aluminium onto different substrate with a Mylar fibre across them to create a gap in the surface. The drop casting was used to place nanotubes onto the surface of silicon and glass.

The E-AFM was set up as in Figure 1, with the SPM (scanning probe microscopy) electronics connected to the AFM drive and then to the scan head. This was all connected to a host computer via a RS232 cable. Once this was done the calibration sample was placed under the scan head and the approach started. Firstly, a coarse manual approach by hand using the 3 screws on top of the scan head; secondly, a fine approach by linear motor using the down button in the easyScan software until the tip was a fraction of a millimetre above the surface. Finally an automatic final approach using the approach button in the easyScan software. The calibration sample was then run.

The microstructure sample offered a sample with known dimensions against which the system could be calibrated. This sample has a XY periodicity of 10µm. This sample measured a length 8.35µm in X and 8.41µm in Y. This resulted in calibration errors of 16.5% in X and 15.9% in Y. This data was used to recalibrate the system throughout the project.

Figure 1. Nanosurf easyScan E-AFM system with (a) host computer with RS232 connection, (b) the AFM drive electronics, (c) the scan head and sample stage.
The first sample viewed was an aluminium deposit on different substrates with parts obscured by a Mylar fibre. This was to view the degree of creep across the Mylar fibre. In this case 3 different substrates were used, glass, silicon, mica. The percentage creep evident in the glass sample was approximately 60 – 77%. In the silicon sample the percentage creep was approximately 65 – 75%. While in the mica samples the creep was 70 – 81%. All three samples have a large percentage creep although some of the variance may be a result of necking in the fibre as it was placed across the samples. Clearly though, the mica sample had the highest amount of creep.

Figure 2 shows an image of the aluminium on glass sample.

The next samples viewed were Single-Walled Carbon Nanotubes (SWNT). These samples had been treated using different solvents, toluene, dichloroethane and DMF. The purpose of this was to judge the effects of the solvents on the formation of bundles of nanotubes. The results showed that the toluene treated samples were larger than the pure but that the other samples were all smaller than the toluene. This suggested that the toluene caused the bundles to bind together to form larger bundles. The only problem in this was the inability to accurately determine if the images being viewed were actually nanotubes because of low resolution in the microscope.

The final images viewed were Multi-Walled Carbon Nanotubes (MWNT). As before with the SWNT these samples appeared as bundles when viewed. This meant that they would also be affected by a lack in resolution. The “clouds” observed had a size of around 2 – 3µm. This would suggest that, since MWNT had a size of approximately 10 – 100nm, the clouds possibly contained hundreds of individual nanotubes.

Figure 3. Image of a hair at a scan range of 16.9µm, a Z-range of 10µm and a time/line of 2s.