UFM **shakes out** the details at the **nanoscopic scale**

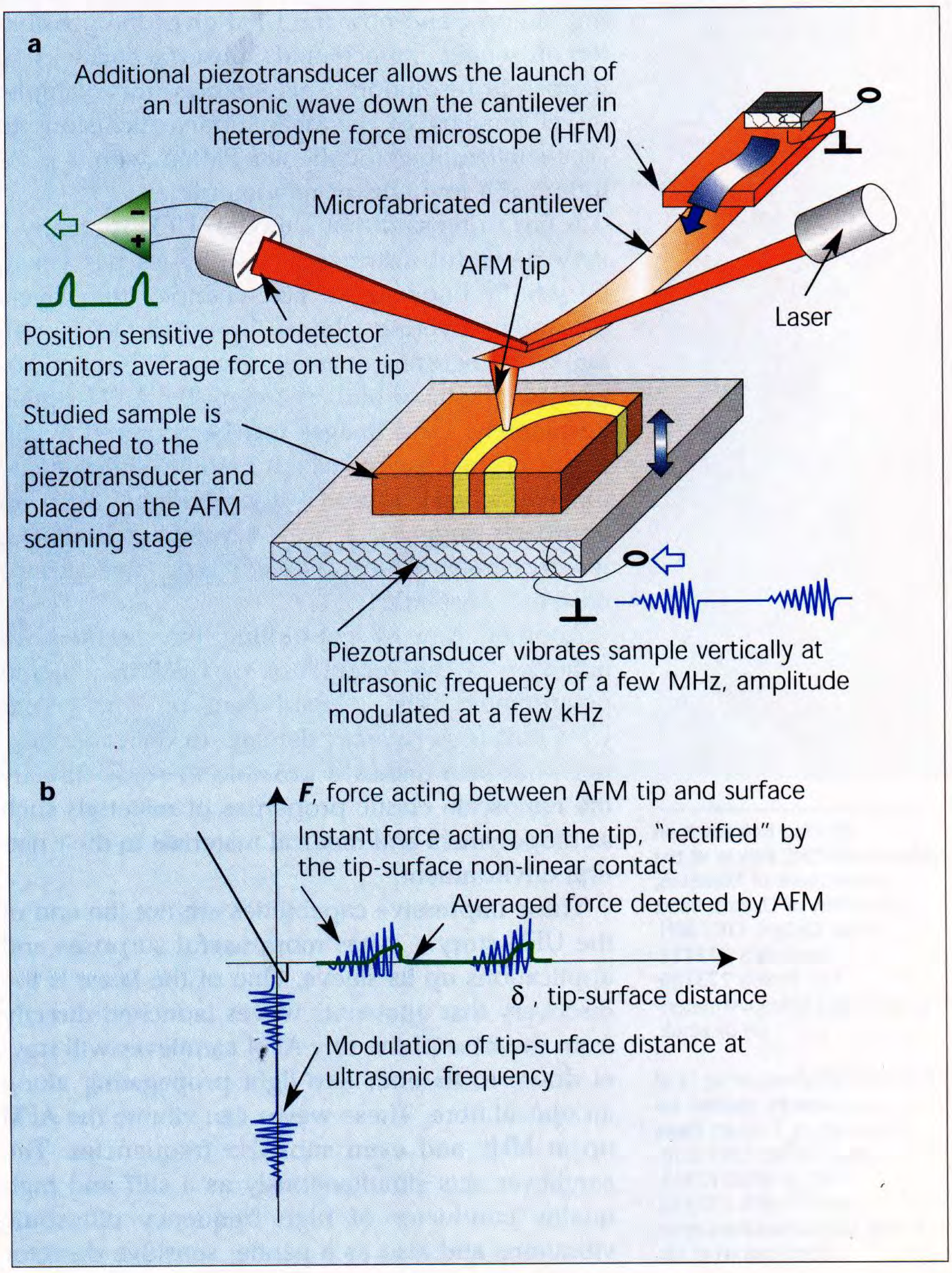
New microscopy techniques are giving researchers insight into the elastic properties of materials at nanoscale resolution, says Oleg Kolosov

The majority of modern advanced materials achieve their impressive performance by relying on clever design and a balanced set of properties of their mechanical microstructure. Stiff fibres in composites endure huge stresses and the matrix materials distribute the load effectively, yet the distinctive mechanical properties of sub-micrometre thin interfaces often determine the ultimate strength of the material. Even in the field of semiconductor quantum structures, the size, shape, and electronic properties of nanoscale germanium dots on silicon surfaces are determined by local elastic strain arising from the lattice mismatch. Material scientists dream of being able to see directly these nanometre scale elastic properties, just as they can image surface topography with the atomic force microsope (AFM) or view optical properties with the optical microscope. That dream is now being realised thanks to the ultrasonic force microscope and a family of related instruments that are being developed at the Department of Materials, University of Oxford, in collaboration with ISIS Innovation Ltd and with support from the Paul Instrument Fund and the EPSRC.

The ultrasonic force microscope (UFM) marries acoustic microscopy (and its sensitivity to elastic properties) to atomic force microscopy, which gives nanoscale resolution imaging of various materials. In UFM, a high frequency ultrasonic vibration with a frequency of a few MHz is applied to a sample, forcing it to elastically 'indent' itself against a static AFM tip positioned at the end of force sensitive cantilever, which is very stiff at MHz frequencies, figure 1a. The contact between the AFM tip and the sample, which is only a few nanometres in size, serves as a mechanical diode, detecting the amplitude-modulated ultrasonic vibrations in the kHz frequency range. The way the system works is analogous to a crystal radio, figure 1b. UFM takes advantage of the intrinsic dynamic properties of the AFM cantilever – it is extremely stiff at the indentation frequency (being 10^2-10^4 times more stiff than at the lower modulation frequency) and it has outstanding force sensitivity at the modulation frequency.

As a result of these features, the elastic properties of many materials – from soft polymers to hard ceramics – are shown in remarkable contrast by UFM, figure 2. The AFM topographic image in figure 2 shows an interface as a jagged trench dividing a silicon carbide fibre (A) and a mullite (Al_2O_3-SiO_x) matrix (B). Much greater detail is shown in the elasticity image, figure 2b, which reveals a detailed interface structure. A relatively soft intermediate concentric carbon rich layer (C) and a softer reaction layer (D) separate the much stiffer regions of SiC and mullite. When applied to semiconductor low-dimensional structures such as quantum wells and quantum dots, this material-dependent elastic contrast makes it possible to differentiate between areas of different material composition (for example, image the percentage of Ge in the Si_xGe_{1-x} compound) and also to

Figure 1
(a) Design of the ultrasonic force microscope (b) Principle of nonlinear detection of ultrasonic vibration in UFM



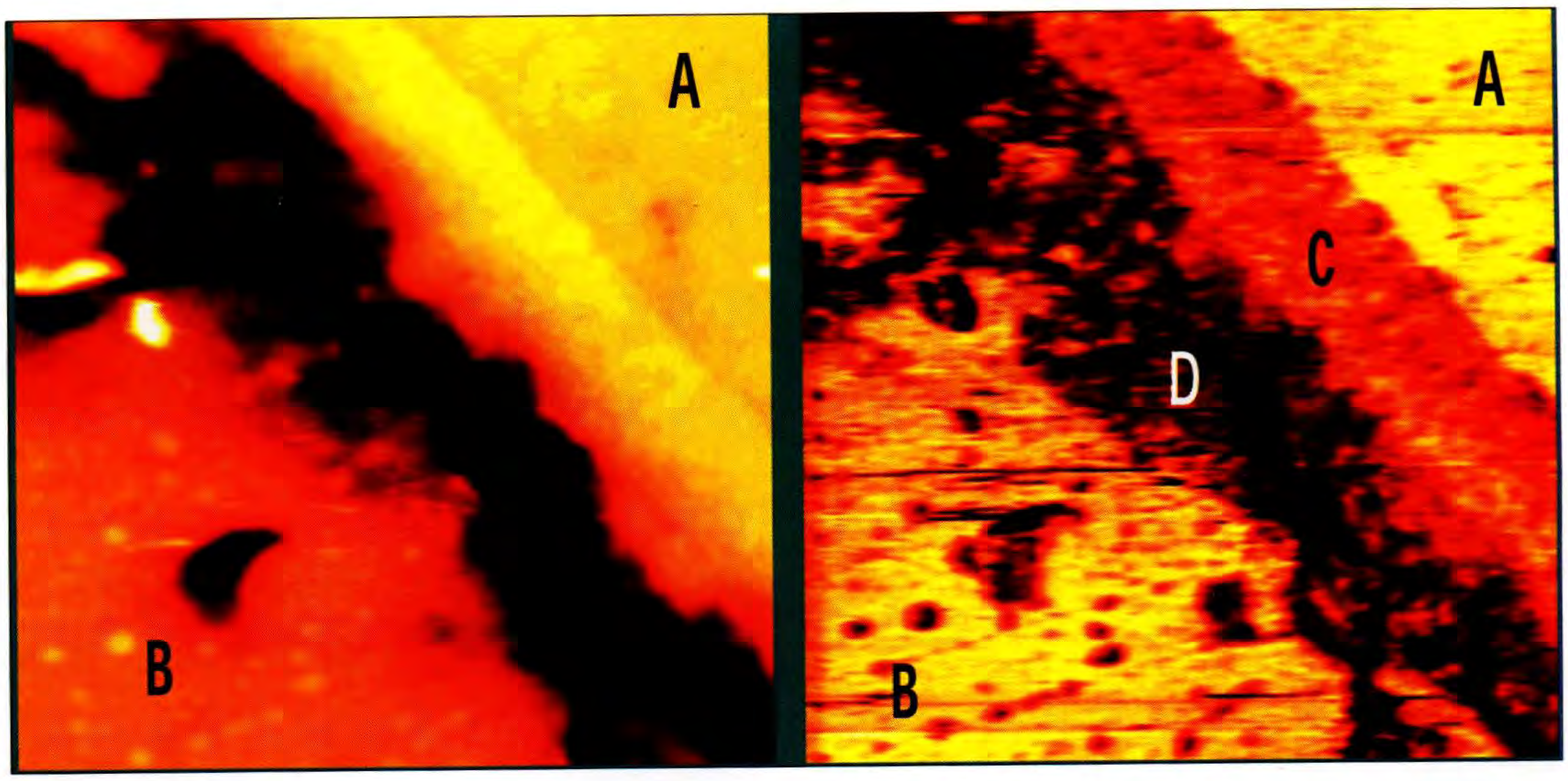


Figure 2
Topographic AFM image (left) and elasticity UFM image (right) of a silicon carbide ceramic fibre. The AFM image shows the interface as a jagged trench dividing a SiC fibre (A) and a mullite (Al_2O_3-SiO_x) matrix (B). The UFM image reveals the detailed structure of this interface, consisting of the relatively soft intermediate concentric carbon rich layer (C) and a softer reaction layer (D) separating much stiffer regions of SiC and mullite. Images courtesy of Franco Dinelli.

highlight growth defects or detect residual polishing damage. Defect free areas of such nanostructures were used to test the lateral resolution of UFM to local elasticity – finer than 4nm – and its sensitivity to differences in elastic moduli – better than 0.5%.

Another useful feature of UFM is its ability to detect cracks and delaminations. This is based on the fact that cracks interrupt the propagation of acoustic waves. The principle is used widely in ultrasonic nondestructive testing and acoustic microscopy, and now the UFM gives the possibility of seeing surface and subsurface cracks at nanoscale resolution. This enables, for example, direct imaging of the near-surface inclusions in core-shell rubber toughened PMMA with a resolution of a few tens of nanometres.

It has to be said that although UFM is clearly a very powerful instrument, its physics has yet to be wholly understood. For example, thin layers of water covering almost every surface in an ambient environment act at ultrasonic frequencies as a tough sticky film, changing the UFM signal. Fortunately, UFM images can be acquired at different normal forces, which helps to differentiate the elastic and adhesive contributions. Such an approach suggests a way towards quantitative measurements of nanoscale elastic and surface adhesive properties.

Another way of controlling the problem of adhesion is the recent use of UFM in a liquid environment. The development of 'underwater UFM' not only reduces damage to delicate materials, but also makes it possible to study directly the nanoscale elastic properties of materials such as biopolymers and medical materials in their natural environment.

These impressive capabilities are not the end of the UFM story – it has more useful surprises and applications up its sleeve. One of the latest is the discovery that ultrasonic waves launched directly from the base of the tiny AFM cantilever will travel down to its end, like light propagating along an optical fibre. These waves can vibrate the AFM tip at MHz and even sub-GHz frequencies. The cantilever acts simultaneously as a stiff and high quality conductor of high frequency ultrasonic vibrations and also as a gentle, sensitive detector of the nonlinear ultrasonic force at the sample modulation frequency of a few kHz. This is of enormous benefit – now, even bulk samples of materials can be easily studied by UFM as access is required from one side only of the sample.

Another intriguing possibility with this development is combining ultrasonic vibrations of the sample and the cantilever. When such vibrations happen at adjacent frequencies, the AFM tip detects the oscillating force at the difference frequency, much like a heterodyne radio receiver – which prompted the name for the new instrument, the heterodyne force microscope, or HFM. Just as modern heterodyne radios are far superior to crystal detectors, the HFM has superior sensitivity to small vibrations than UFM.

Even more significantly, it picks up information about the phase of high frequency vibrations. Such phase information is directly related to the time dependent processes of tip-surface force interaction. This opens up the new realm, which is just beginning to be explored, of observing the mechanical structure of materials with both nanometre lateral resolution and also nanosecond time sensitivity.

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Further reading

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