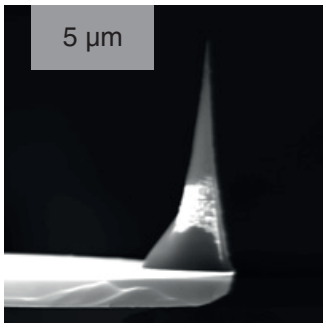
Mechanical properties in AFM

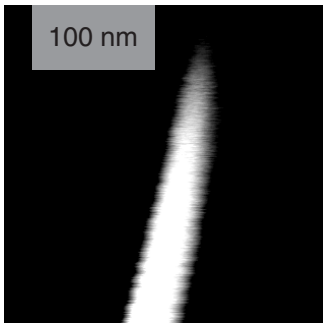
AFM measurements of mechanical properties of materials, e.g. hardness and elasticity of thin films, coatings and substrates, require probes of extra hardness. This is related not only to durability of the tip that is in tough contact with the surface. The question is how one can be sure that the measured value is the property of the surface and not the tip itself.

Traditionally, the use of AFM techniques for measuring mechanical properties, such as peak force, force modulation or nanoindentation, is limited to polymers, biomaterials and other soft samples as the hardness of Silicon tips is not too high. The useof the Single Crystal Diamond (SCD) as a probe material allows measuring the same for hard materials.

ART™ SCD tips are specially grown in CVD process and attached to silicon cantilevers for use in AFM. The probes have high aspect ratio and tip radius less than 10 nm.



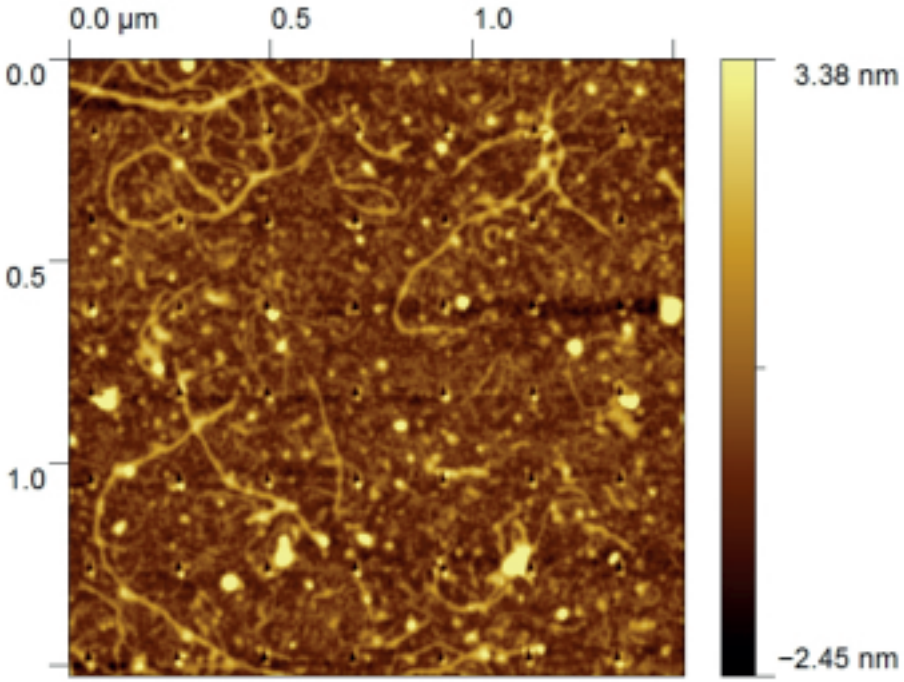
SEM image of the SCD probe tip.



SEM image of the SCD tip end.

Nanoindentation on Silicon by SCD probe

The useof ART™ SCD probe tips allows performing nanoindentation experiments on Silicon. The image below shows a small fragment of an array of 64000 indents made by a single SCD probe. Scan size is 1.5x1.5 um, height 6 nm. The scan is obtained by the same probe that made the indents.



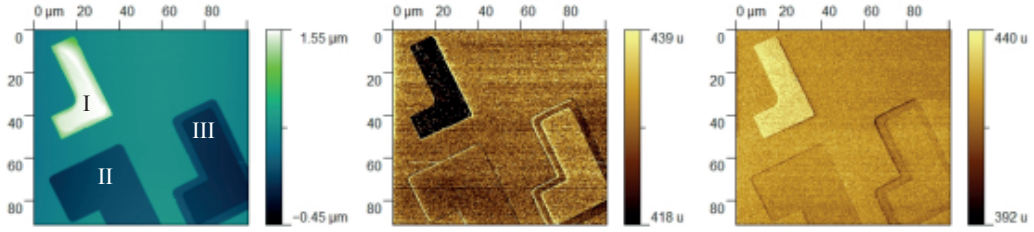
Fragment of an array of 4000 indents made using ART™ SCD probe. AFM image.

Mechanics of a semiconductor structure

ART™ SCD probe tips make indents in Silicon and metals easily, which allows measuring local hardness of semiconductor structures. The series of scans below exhibit structure that has a metal area (I) and two openings (II) and (III), the opening (III) has two levels. Topography (A) and the slope of loading (B) and unloading (C) curves were measured in one pass.

The metal area is distinct on both loading and unloading maps, the slope of the curve is lower when loading and higher when unloading, which speaks of a higher plasticity of material in the area in comparison to Silicon. The values of the force-curve slopes in the openings are also different, which may be an evidence of doping presence or coating. This way the SCD tip shows differences in mechanical properties of the materials.

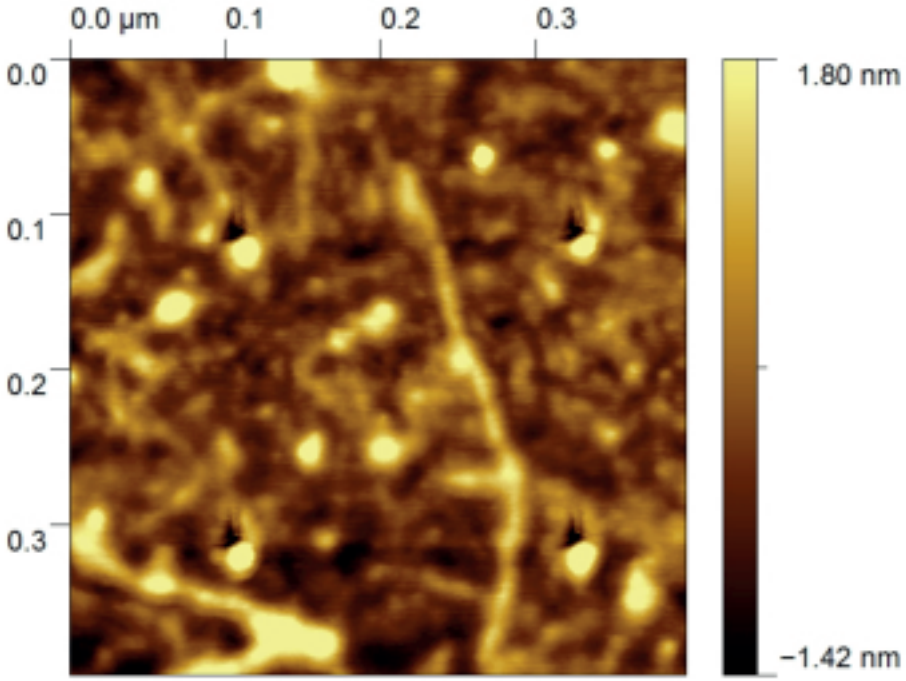
The size of the scans is 256x256 points, which is equal to 65 000 of indents.



(A) Topography (B) Loading map (C) Unloading map

AFM images of semiconductor structure made using SCD made using ART™ SCD probe.

After the force spectroscopy mapping experiment, the scan was zoomed in to see the traces the tip left on surface when loading. Image below shows a 400x400 nm fragment of the array of indents made by the SCD probe. Scan height is 3 nm.



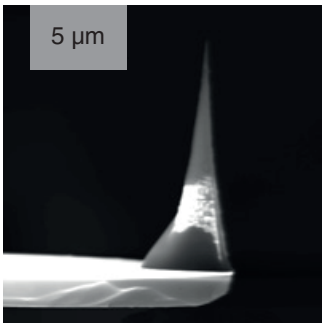
Close view of indents made using ART™ SCD probe. AFM image.

The scan is obtained by the same probe that made the indents. Note that the tip is still sharp, resolving surface features less than 10 nm in size.

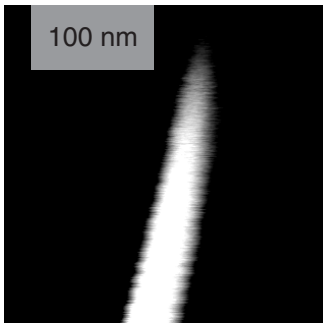
SCD Probe

ART™ tips are specially grown in CVD process and attached to silicon cantilevers for use in AFM. The probes have high aspect ratio and small tip radius.

The probe is highly resistant to wear, which is useful when fast scanning speed is needed, or when the surface contains sharp and rigid edges. Other applications are nanoindention, scratching and nanolithography experiments.



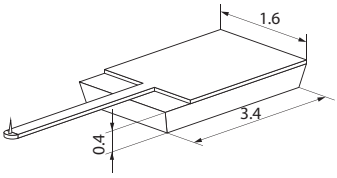
SEM image of the SCD probe tip.



SEM image of the SCD tip end.

Tip material	Single Crystal Diamond (SCD)
Tip aspect ratio	about 5:1
Tip radius	5-10 nm

Cantilevers



ART™ diamond probes are glued onto rectangular (diving-board) silicon etched cantilevers. The range of spring constants and resonant frequencies of cantilevers available covers the Contact mode, Force Modulation, Non-Contact and Tapping mode. Cantilever backside is coated by Aluminium.

The chip holder size is 1.6 mm x 3.4 mm x 0.4 mm.

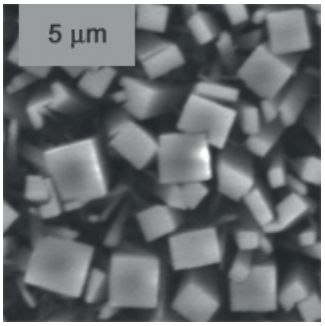
Part Number	Resonant Frequency, kHz	Spring Constant, N/m	AFM mode
D10	**10**	0.15	Contact mode
D80	**80**	3.5	Tapping mode. Force modulation. Contact mode.
D160	**160**	5	Tapping mode. Contact mode on hard surfaces.
D300	**300**	40	Tapping mode. Non-contact mode. Contact mode on hard surfaces. Nanoindentation. Force nanolithography.

Note: The glue used to attach the tip to the cantilever is not conducting, so the probe is not applicable for conductive AFM measurements. Values for resonant frequencies and spring constants are typical.

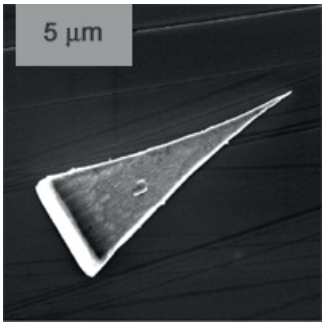
bAtch™ gRowth & aTtachment

ART™ stands for bAtch gRowth and aTtachment technology. ART™ probe for AFM consists of two parts that are manufactured separately: a cantilever on a chip-holder and a tip. The tips grow in batch in a specially designed process and then glued onto the cantilevers using a micromanipulation equipment and procedure.

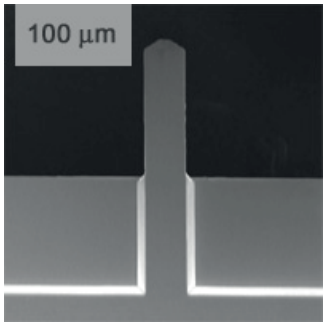
ART™ technique provides highly reproducible production at reasonable costs. Images below illustrate some of the key stages of the technology.



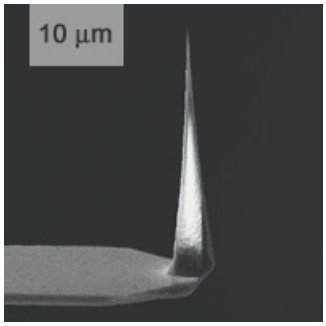
Film of diamond crystals. SEM image.



Single crystal diamond tip of the needed size and shape separated from others. SEM image.



Tipless silicon cantilever. SEM image.



SCD tip mounted on silicon cantilever. SEM image.

Diamond Tips

Diamond is a very promising material for making AFM tips because of its durability, hardness, outstanding chemical stability, high temperature conductivity and potential ability to conduct electric current. Besides application in AFM as a probes or indentors, the diamond tips can also be used as nanosized temperature sensors and X-ray detectors.

The tips are monocrystal diamond pyramids with the {001} facet in the basis having a controllable shape along the <001> axis.

Attachment

Attachment consists in positioning and gluing a the micro-sized object on a cantilever with high precision. This manipulation technique can be used to attach not only diamond tips and not only on silicon cantilevers. Our experience shows that other objects like carbon fibers or micro-sized particles can be handled the same way. For AFM, the objects can also be glued to silicon nitride cantilevers, piezo cantilevers or tuning forks.

Contact us if you have an idea how the diamond tips or micromanipulation technique can be used to make something special for your research.

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