

# Nano-fabrication

*Nano-science has generated a lot of new knowledge about phenomena in the small world. It is expected that these phenomena contribute to mastering current and future industrial and societal challenges. One of the scenarios how this shall be implemented involves microfabricated instruments and tools for sensing and production. In both cases throughput considerations impose parallel operations of large amounts of instruments. In turn, this requires innovations in the field of micro-actuation, systems architecture and control, and micro-assembly. This article presents the case of the scanning force microscope that was developed for the Phoenix mission to Mars, addresses the challenges of up-scaling nano-manufacturing, and presents current and future research in Delft.*

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On May 25, 2008, the Phoenix Mission landed on Mars. On board was a scanning force microscope opening a new era, the microscopic investigation of the solar system. This original nano-scientific instrument, developed by a Swiss consortium which I led, enabled an unprecedented view on Martian soil and dust, and contributed to the understanding of the history of 'Water on Mars'. The challenges on the instrument were formidable and its successful operation was an important milestone for micro electro-mechanical systems (MEMS) in space. This example shows how scientific instrumentation enables new basic scientific research; and it also stands for what engineering research receives from science: exciting, stimulating first applications and knowledge for innovative building blocks for future technologies.

# and -manufacturing

## Life

Mars has played an important role in the history of science, the development of scientific knowledge and the search for extraterrestrial life. Liquid water is considered the single most important factor for the development of life. The presence of liquid water leaves traces, which (on earth) can be detected in the geomorphology of a landscape and in the structure of the soil. Hence, one way to assess whether liquid water has been present is to investigate the soil, its particle size distribution and particle texture. This requires microscopic experiments, for which the most recent Mars exploration, the Phoenix mission, had a special scientific instrument on board, the Microscopy and Electrochemistry and Conductivity Analyzer. MECA comprised an optical microscope and a scanning force microscope (SFM). The SFM was designed as a 'technology demonstrator' but turned out to provide 'real' results. Space applications are among the most demanding environments for any instrument. Therefore, the development of the Phoenix SFM may serve as an example for the fascinating engineering field of scientific instrumentation.

## The Mars SFM

The Swiss consortium led by the University of Neuchâtel designed the Mars SFM to measure the size, size distribution, texture and hardness of dust and soil particles on Mars. It was originally conceived as part of the Mars

Surveyor 2001 mission, which was cancelled, to explore hazards for future human explorers of the red planet. These threats include dust and soil particles, which were to be inspected by optical microscopy followed by SFM. Later, the SFM was included in the Phoenix mission [2], which was launched in 2007; see Figure 1. Now, the main objective was to investigate the structure of the soil and look for traces of liquid water.

## SFM basics

The SFM, often called atomic force microscope (AFM) [3], extended the abilities of the scanning tunneling microscope (STM) [4] to image also insulating material at atomic resolution. Both the SFM and the STM have a small sharp probing tip scanning very closely across the sample's surface. The distance between the tip and the sample surface is so small, that atomic range forces act between them, hence the name AFM. The tip is attached to the end of a cantilever spring in order to measure these forces. The force acting on the tip can then be determined by detecting the deflection of this cantilever, by means of either a laser beam deflection system, or a piezo-resistive strain gauge located on the cantilever, as implemented in the Mars SFM. During operation, the tip is raster scanned across the sample and the cantilever bending kept constant by means of controller electronics, which moves the sample closer to or further away from the tip to compensate for changes in the force. The servo signal is recorded and when plotted against the tip position, it represents an iso-interaction force contour, which closely represents the topography.

In order to reduce unwanted lateral sample-tip interactions, the SFM can be operated in the so-called dynamic mode, where the cantilever is set to vibrate at its resonance frequency. Any force gradient that the tip experiences detunes this resonance, which can be used as a signal for controlling the time-averaged separation between the sample and the tip. There are several variants to this principle, the Mars SFM employing the frequency modulation technique. In this implementation, the phase difference between the cantilever excitation and its vibration frequency is locked by a phase-locked loop. The control signal is used to approach or withdraw the sample, hence, suppressing the modulation of the frequency. Again, the servo signal represents the sample topography; see Figure 2.



Figure 1. Artist impression of Phoenix landed on Mars. (Picture: NASA/JPL-Caltech/Univ. of Arizona)

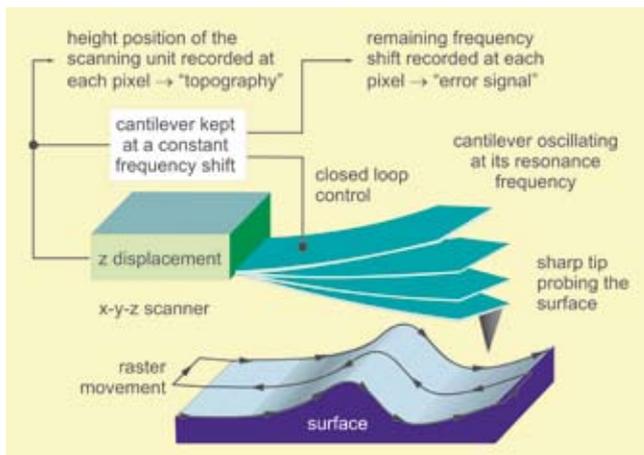


Figure 2. SFM working principle described for the dynamic mode with closed loop for constant force gradient imaging.

### Instrument design

The requirements for the Mars SFM differ in many aspects from those for a normal laboratory version. The obvious demands are low mass (sending one gram of payload to Mars costs about 10,000 US\$) and structural robustness, required by the rough conditions during journey, where shocks of up to 2,500 g, and strong vibrations ( $\sim 0.1 \text{ g}^2/\text{Hz}$ , from about 80 to 800 Hz) occur. These specifications can at least partially be addressed by reducing the size, because the relevant physical properties scale favorably. The second obvious condition was that there is no operator on Mars. Broken or contaminated tips must be exchanged by remote operations. The time delay of a few minutes for radio signals traveling from Mars to Earth, and the fact that satellite communication with the lander was restricted to only two or three times a day, required complete autonomy of the instrument. The tip exchange was implemented by an array of eight cantilevers, which can be cleaved off if no longer usable; see Figure 3. Additional design challenges were posed by the low atmospheric pressure on Mars and the nearly 100% absence of magnetic and atmospheric shielding of cosmic radiation.

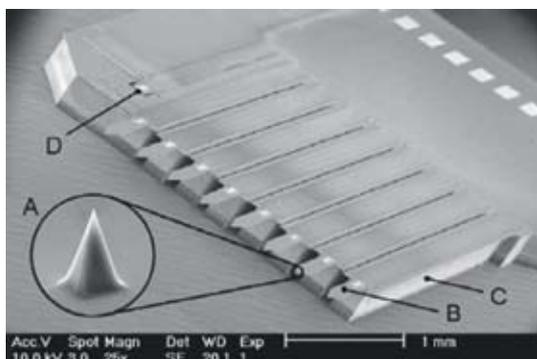


Figure 3. SFM chip of the MECA AFM [5].

- (A) The tip.
- (B) The cantilever with integrated, piezoresistive strain gauge.
- (C) Support beam, which can be cleaved off.
- (D) A reference resistor for a Wheatstone bridge.

### Measurements on Mars

The first successful measurement was performed on a calibration sample, in the less demanding static operation mode. Then, changing from static to dynamic mode proved to be much more demanding than anticipated. On Mars, the motors were powered for a much longer time than in the laboratory tests and therefore heated up more. This led to thermal drifts and false detection of sample-tip contact. Once the problem was identified, the software was re-coded and validated to drive the motors faster and to preheat the electronics longer. Finally, images of a calibration grid in dynamic mode could be measured: we had accomplished our ‘minimal success goal’, the demonstration of SFM measurements on another planet. Soon thereafter, we could also measure particles; see Figure 4. Images like these were used to draw up a particle size distribution (PSD). Comparing this PSD with terrestrial data, allowed estimating how long the investigated soil might have been exposed to liquid water. A maximum of about 20,000 to 40,000 years (within the last 500 million years) was deduced.

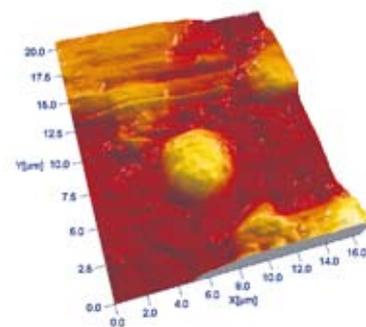


Figure 4. SFM image from Mars soil, showing individual particles. (Photo: NASA/JPL-Caltech/Univ. of Arizona/Univ. of Neuchâtel)

### Nano-engineering

This Mars SFM example shows how technology contributes to science. On the other hand, basic science generates knowledge that enables new technologies. In my opinion, the academic engineering community should take care of making this wealth of knowledge available for technology implementations. Nano-engineering as I will conduct it in my research in Delft is focusing on the new knowledge developed in the emerging domain of nano-sciences, where chemistry, biology and physics meet at the

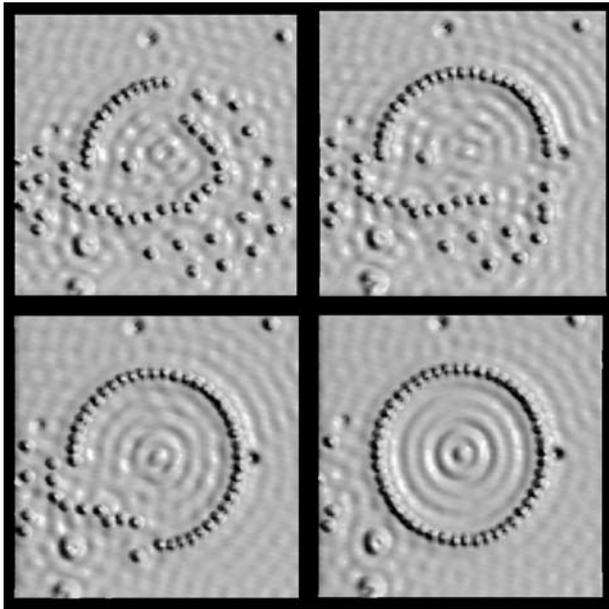


Figure 5. Arranging Fe atoms on a Cu(111) surface into a so-called quantum corral, at 4 K, using a STM [6].

nano-length scale. Note that this length scale is not the essential aspect in nano-science; rather it is a paradigm shift in the investigation. The individual, atomistic functional unit is addressed and its interaction with other units is experimentally interrogated. New scientific instruments were needed for that purpose, a prominent representative being the AFM.

### Scale-up

These instruments or nano-tools have to bridge a dimension gap of about six orders of magnitude, from the lower nanometer to the millimeter scale range. Micro electro-mechanical systems (MEMS) technology offers this interface between the nano- and the macro-world.

Widespread AFM use became only possible once MEMS technology was used to mass-produce miniaturized force sensors with reproducible and controllable properties. With them, scientific research on individual atoms and molecules exploded. People started thinking in molecules and atoms, moving them around (see Figure 5), probing them and measuring their optical spectrum. While scientifically highly relevant, this dealt with a technologically insignificant amount of units, e.g. a few atoms or molecules. One of the most prominent questions in nano-engineering, therefore, is that of scale-up.

Three approaches may tackle the problem of scale-up:

- “Self assembly”, through interactions between individual functional units, the target configuration being the one where an energy minimum is achieved.
- “DNA nano-technology”, inspired by the molecular recognition properties of DNA, viewed as mechanical building blocks of two- and three-dimensional structures.

- VLSI MEMS, Very Large Scale Integration of minute tools, nano-tools, that all work in parallel and at high speed.

### Challenges for MEMS scale-up

#### Microfabrication

Shrinkage of MEMS systems is limited by functional conditions. A related problem is associated with actuators. Since they are part of a MEMS, they contribute to its footprint, increasing the separation between the MEMS units. Therefore, high stroke- and force-to-volume ratios in a small envelope are required, an issue we address in our micro-actuation research. Moreover, not all functionalities can be implemented by silicon-based MEMS. Hence, heterogeneous systems and their integration will be needed.

#### Architecture and systems integration

Each MEMS must be supplied with energy, information, and raw materials. Most likely, the associated bus lines demand for a three-dimensional integration, comparable to the interconnect layers in micro-electronics. Contact- or wireless solutions, e.g. for powering, may also be implemented. Given the gigantic data volume that a VLSI MEMS can produce, analysis and processing have to be performed locally in such a system. Hence, each MEMS or at least a cluster of MEMS will need some ‘intelligence’.

#### Operations

VLSI MEMS-based manufacturing provides a generic solution, which requires a tight control of the tools for any specific assignment. This amounts to an enormous task for thousands of tools working in parallel. Some kind of local intelligence may be used to adapt control signals to local specificities, or to exploit the interaction between neighboring tools. One could even think of ‘programming’ the structure of the nano-product into the tool rather than into the individual elements of the product – a kind of “self assembly via the tool”.

### Current and near-future research

To address the above outlined challenges, several research activities have been started or existing ones were intensified by the Micro and Nano Engineering research group, in collaboration with other research groups in Delft.

Micro-assembly, for example, is used to assemble the VLSI MEMS out of different components, or for replacing

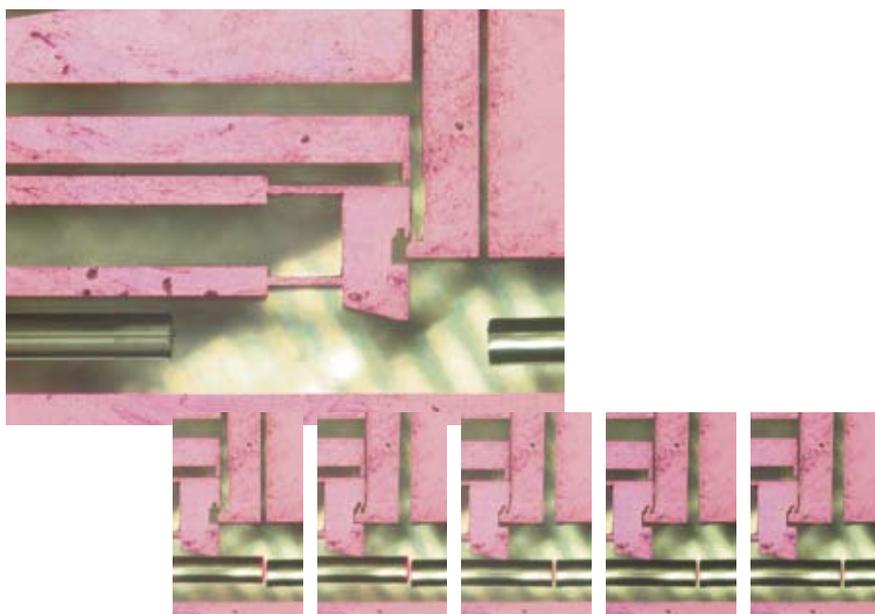


Figure 6. ID fiber positioning and clamping sequence, from (left) fabrication position, to (right) maintaining the fiber in position without using power.

defective parts. Also, many production processes in nano-fabrication will directly rely on assembly steps. One could, for example, think of integrating carbon nano-tubes as electro-mechanical elements in a sensor. A central question concerns the fundamental steps of the assembly process and how they can be modeled. What key characteristics make a process suitable for industrial applications? At the same time, different steps in the assembly chain are being experimentally investigated, for example the displacement of an object over large distances with high final placement precision. Various driving forces can be applied for that purpose. For instance, mechanical vibrations combined with local electrostatic chucking, or asymmetric air cushions again combined with electrostatic or magnetic chucking. One of the current topics in research on micro-assembly is concerned with the clamping of an optical fiber; see Figure 6.

A new activity on micro-actuation was started, aimed at realizing a two-dimensional high-precision conveyor

system. Such a device should be capable of positioning several micro-scale objects at the same time to different locations within a limited but macroscopic area. Currently, the interaction between the object and the positioning system, by means of Van der Waals forces, is investigated.

The nano-tools that are being used limit their interaction with the object by means of a mechanical constriction. This constriction could be considered as “materialized focus”. This focus could be a simple cone, like in the case of an AFM tip, or it could be a tipped heat, current, or light source, or a measuring tool. Also the opposite of a tip, a small hole in a membrane can be used to localize the interaction. Such ‘nano-pores’ were employed to study e.g. DNA molecules. Combination of these two elements, by machining a small hole at the end of a hollow tip and cantilever, results in a ‘nano-pipette’; see Figure 7. It can be used to precisely deposit or take-up small amounts of liquids.

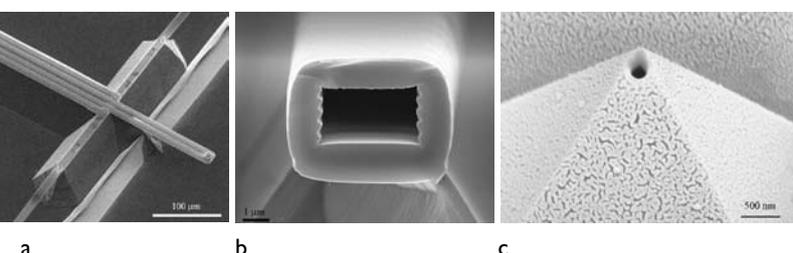


Figure 7. A nano-pipette.

- (a) The hollow cantilever and tip are essential parts of this fluidic system.
- (b) A cross-section through the channel.
- (c) At the end of the tip, a small pore is drilled by means of a focused ion beam such that the hole is slightly off-axis and the tip apex stays intact.

### In conclusion

When developing a new technology, scientific knowledge forms the repertory of building blocks. Investigating and establishing these building blocks for future technologies is an important part of academic technology research. In this endeavor, scientific instrumentation plays a key role: it is the natural link to scientific research and stimulates the dialogue among the sister disciplines. As an example, microfabricated elements and instruments were essential in the advancement of nano-science. If similar means, but now in the form of nano-tools, shall be used also in production, then micro-engineering has to overcome formidable challenges. Some of them I like to address together with my team. Is there a market need for such nano-technology? That is the wrong question! There is

never a need for a specific technology. The market needs solutions for certain problems – engineers may find them in knowledge developed in nano-science. Enabling this is the mission of nano-engineering!

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