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Novel diamond/sapphire probes for scanning probe microscopy applications

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Abstract. Novel diamond/sapphire probes are introduced for different scanning probe microscopy applications: from nanoindentation to high-resolution imaging of soft samples. These probes have sharp diamond tips with apex size below 10 nm and their stiffness/resonant frequency and tip shape can be adjusted for customer needs. Several examples of imaging with novel probes are given. Probes with conducting diamond tips exhibited high-sensitivity in electric force microscopy study.

1. Introduction

In the development of scanning probe microscopy (SPM) the availability of sharp probes has been always one of the crucial factors. In the pioneering SPM method - scanning tunneling microscopy (STM), conducting probes, which are used for detection of tunneling current, are made either by electrochemical etching of metal wire or even by a more simple mechanical sharpening (e.g. cutting wire with scissors). Though the overall shape of the electrochemically-etched and mechanically sharpened probes is quite different, the probes of both kinds have been successfully applied for atomic-scale imaging of flat conducting and semiconducting surfaces of various inorganic and organic crystals. This is a consequence of the fact that only most advanced atoms of the probe are involved in electron transfer process between the probe apex and a sample. In atomic force microscopy (AFM), which was introduced for imaging of samples independent of their conductivity, the probe consists of a cantilever with a sharp probe attached to one of its ends whereas the other end is fixed to a large substrate that allows handling the probe. In AFM, the tip interactions with a sample cause the cantilever displacement or perturbation of its oscillation. The cantilever geometry and material it is made off define its stiffness; a tip shape indicates what type of corrugations it can profile and AFM resolution on flat surfaces depends on a size of tip apex.

Initially the AFM probes were manually made off a piece of metallic foil and a diamond shard, which was glued to the foil to become a tip. The introduction of microfabrication of AFM probes was important development in making this method a routine technique. First, probes for contact AFM measurements were made of Si_3N_4, [1] and, later, etching of Si wafers using appropriate masks was used for batch manufacturing of Si probes [2]. At present, etched Si probes are the largest volume AFM probes employed for imaging in tapping mode. This mode is most applicable to a broad variety of samples: from soft biological objects to semiconductor structures and ceramics. Typically, the cantilevers of Si probes have following dimensions (width

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– 30-40 um, length –125 – 400 um, thickness 2-7 um) and their stiffness varies in the 0.1 –50 N/m range. The geometry of the Si tips is pyramidal (angles?) and the tip apex below 10 nm.

In addition to the mass-produced probes, there is a room for specialized AFM probes. Probes with high aspect ratio shapes and special tip geometry are needed for evaluation of critical dimensions objects and undercut profiles in semiconducting applications. Long tips are required for examination of industrial surface structures with corrugations in tens of micron that became possible with scanners having large Z-range. The probes with hard tips of different shapes are required for nanoindentation measurements some of them made using AFM microscopes. This paper describes novel probes consisting of sapphire cantilevers and diamond tips. The primary reason for making diamond tips is high mechanical strength. High thermal conductivity and chemical resistivity of diamond are also useful properties for AFM probes. The diamond tips with electrical conductivity arising form doping can be used for STM and for electric AFM modes.

A combination of a diamond tip with a sapphire cantilever is rational from different viewpoints. Sapphire has high-mechanical strength that allows an attachment of massive probes, and its high temperature resistivity tolerates strong metal bonding of a diamond tip to a cantilever. The sapphire cantilevers being custom made can have different shapes. The cantilevers with semispherical cross-section have definite advantage compared with flat Si cantilevers because they allow avoiding optical interference that is harmful for AFM measurements.

2. Description and characterization of the novel probes

A general view of the probes is shown in Figure. 1. A manufacturing of a diamond\sapphire probes includes (a) a polishing of a sapphire wire to a beam with a semispherical profile and its bonding to a substrate; (b) a polishing of a diamond crystal into a triangular or conical shape (height 50-100 um); (c) a metal bonding of a tip to a beam; (d) a final sharpening of a tip apex that makes it below 10 nm at the end. Metal bonding of the cantilever/substrate and cantilever/tip junctions is made at ~ 1200°C. It also leads to a metal coating of the cantilever that improves its reflectivity and makes electric contact to a conducting diamond tip (see below).

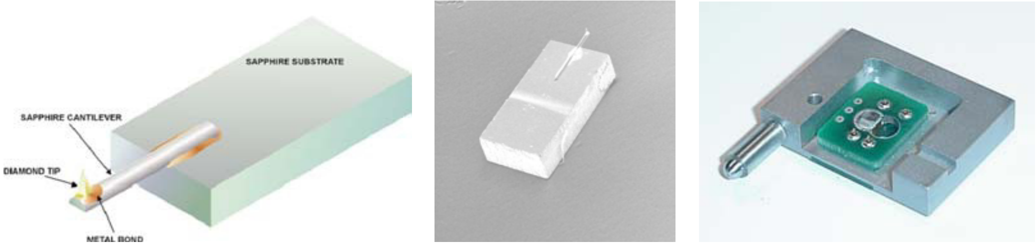

Figure. 1 (a) A sketch of a diamond/sapphire probe. (b) A photograph of a diamond/sapphire probe attached to a sapphire substrate. (c) A module with a diamond/sapphire probe for MultiMode™ and Dimension™ types of scanning probe microscopes (Veeco Instruments).

SEM and TEM micrographs of two diamond/sapphire probes with tips of different shapes are shown in figure 2. One of the probes has a tip with a shape of trigonal pyramid; the other exhibits a conical shape. A half angle between 13 and 7 degrees is common for these tips. Their height is in the 50-100 nm range and sharpness of an apex reaches several nm.

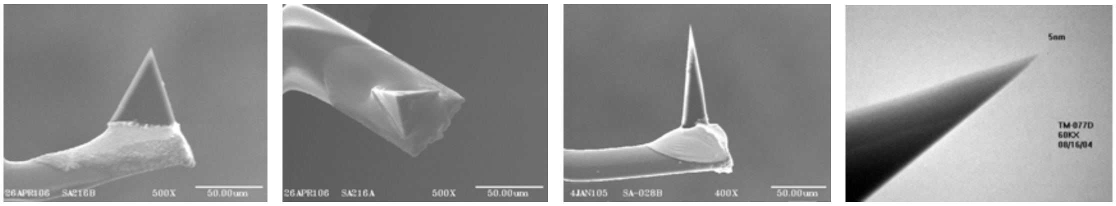

Figure. 2 (a) – (c) SEM micrographs of the diamond tips of different geometry. (d) TEM micrograph of a 5-nm apex of the diamond tip.

In addition to the tip shape, stiffness of the probe is the important parameter. Because the novel probes are custom-made, their stiffness and resonance frequency can be easily adjusted by changing geometry of the cantilever. The graphs, which relate these parameters with the cantilevers' length and thickness, are shown in figure 3. The presented data were obtained from the pertinent mathematical calculations, verified with a hundred actual cantilever measurements. The resonant frequency of a probe may vary about 15% from the graph because of variations on the actual size and weight of the diamond and bonding metal. The value of the stiffness predicted by the graph may vary about 5%.

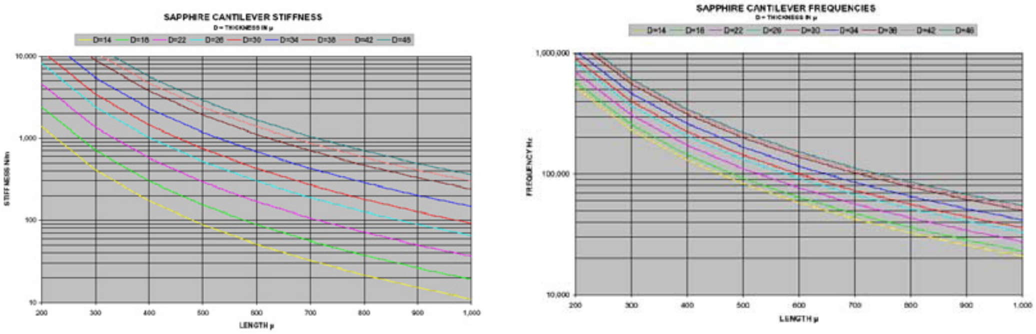

Figure 3. Relationships between the probe stiffness (left) and resonance frequency (right) and the length and thickness of the cantilevers with a semispherical cross-section.

AFM experiments with the diamond/sapphire probes

The use of diamond probes for nanoindentation is well established yet most of these probes are stiff that limits imaging to hard samples. This limitation is substantially removed with the diamond/sapphire probes as demonstrated in figure 4. The AFM images in tapping mode show traces of molecular-scale order at the *bc* plane of the polydiacetylene crystal, single DNA molecules, and microphase separation pattern of SBS triblock copolymer. The probes used in these studies have stiffness ~ 4 N/m. The spatial resolution of these images is comparable with that obtained with most sharp Si probes.

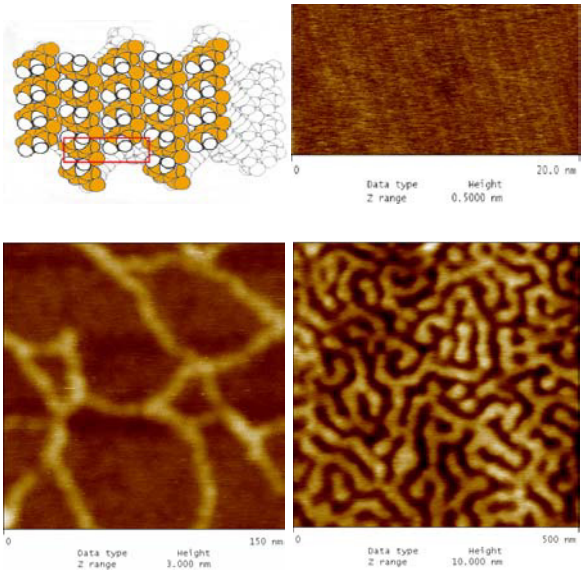

Figure 4. Top, left – a sketch of the crystallographic structure of the *bc*-surface of polydiacetylene crystal. Top, right - AFM image of this surface in tapping mode. Bottom – AFM images of double-stranded DNA (left) and surface of polystyrene-polybutadiene-polystyrene triblock copolymer.

It is worth to note that the limitations of uniform polishing of sapphire cantilever do not allow the preparation of the probes softer than 1N/m, whose length is not extreme for commercial scanning probe microscopes. On the other hand, there are no practical restrictions for making stiff probes.

Imaging with Conducting Probes

A use of conducting diamond, which can be naturally or specially doped for this purpose, offer unique capabilities for novel probes. One of the conducting probes is shown in figure 5, where it is making contact with a droplet of Hg for measurements of its conductivity.

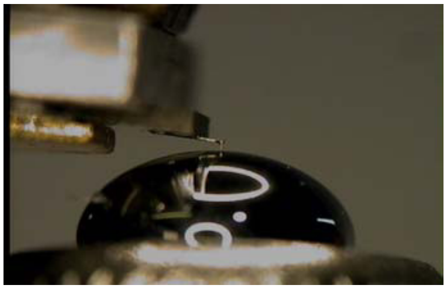

Figure 5. Optical photograph of the diamond/sapphire probe making contact with a Hg droplet.

Despite the fact that resistivity of the naturally-doped diamond probes was high (R~5.KΩ) they have been successfully applied for electric force microscopy (EFM) studies of polymer samples loaded with carbon black. A comparison of the electric force response of the regular Si probes, which were coated with a conducting layer, and conducting diamond probes showed that the latter are over 3 times more sensitive as judged by the phase changes in figures 6a-b. This effect is most likely related to the fact that the long diamond tip limits a reduction of the cantilever Q-factor close to the surface as it happened for the probes with shorter tips.

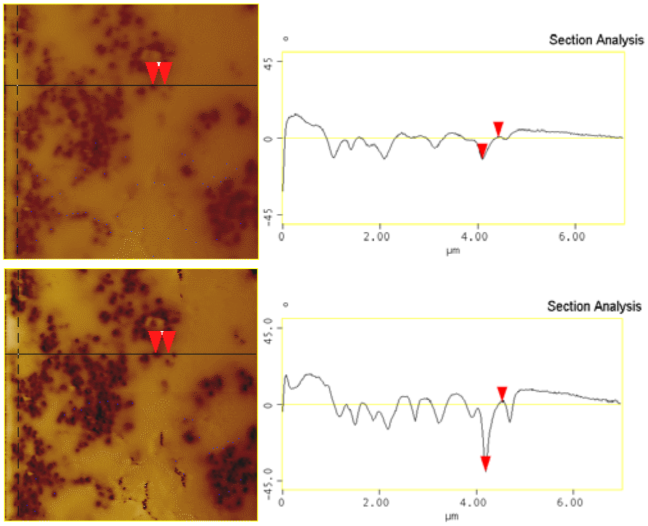

Figure 6. Left - The EFM phase images of thermoplastic vulcanizates recorded with a regular conducting Si probe (top) and a conducting diamond probe (bottom). The images were obtained at bias voltage of 10 V and lift height of 20 nm. Right - the cross-section profiles (right) along the directions shown with solid black lines in the images. Red arrows indicated the larger phase shift at the same locations when the conducting diamond probe was applied.

Tip Wear

Durability of AFM probes is the important practical issue of this technique. Our experience in AFM of various samples and with different probes suggests that a special care (a gentle tip engagement, a low-force operation, a use of soft cantilevers) is needed for preserving sharp asperities of the tip apex with diameter smaller than 10 nm. This is difficult in imaging of hard materials that causes a tip damage of Si probes. The apex of diamond tips might also be modified when used on surfaces such as porous Al - the standard for tip shape evaluation [3]. This is evident TEM and AFM experiments performed with a probe, which has stiffness 4 N/m and a diamond tip with initial apex ~ 6 nm, Figure 7a. After two AFM scans on porous Al, the probe apex became ~ 10 nm, Figure 7b. This change is also noticed by comparing the tip reconstruction made from 1st and 2nd AFM images on the Al sample. These images and reconstructed tip shapes are shown in Figure 8. The tip diameters estimated from the images were 11 nm and 14 nm, respectively. The diamond tips with diameter over 10 nm are stable and have longer lifetime.

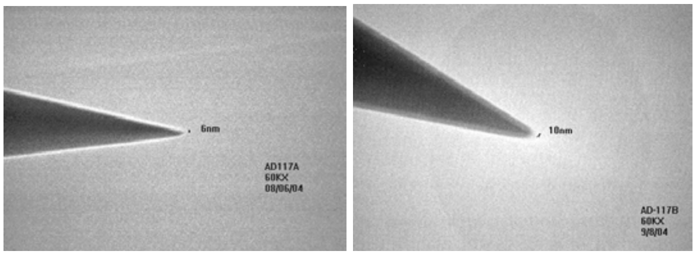

Figure 7. TEM micrographs of a fresh diamond tip before (left) & after 2 AFM scans on Al.

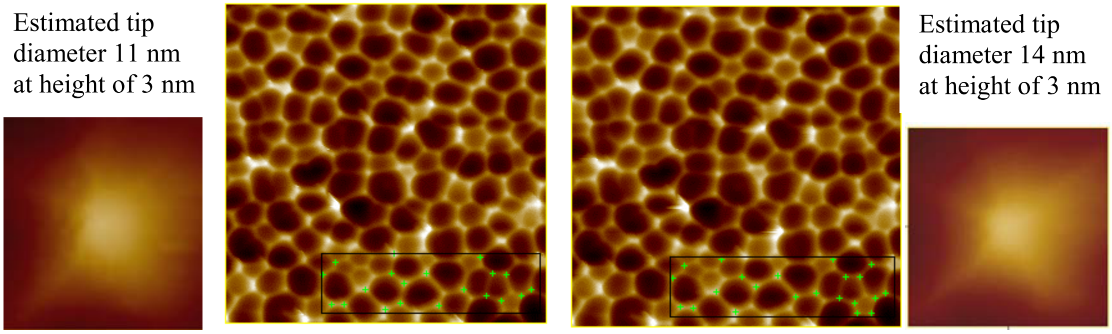

Figure 8. Two consequent AFM images obtained with a diamond probe and the results of tip shape reconstruction (Nanoscope software) from these images. Green dots on the images indicate sharp locations used in the reconstruction.

Summary

We have described novel diamond/sapphire AFM probes that can be successfully manufactured and applied in a number of different applications including high-resolution imaging of soft samples. Remarkable features of new probes are extremely sharp tip apex and custom-design that satisfies needs of various tip shapes and cantilever stiffness/resonant frequency. The use of tip made of conducting diamond offers unique capabilities for imaging in electric modes and STM.

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