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科学研究費助成事業　　研究成果報告書

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研究課題名（和文）サブナノメートルの分解能を実現する走査型熱顕微鏡の開発

研究課題名（英文）Development of Scanning Thermal Microscopy with sub-nanometer lateral resolution

研究代表者

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交付決定額（研究期間全体）：（直接経費）　15,300,000円、（間接経費）　4,590,000円

研究成果の概要（和文）：走査型熱顕微鏡（SThM）は、熱カンチレバーをプローブとした原子間力顕微鏡（AFM）の一種である。カンチレバーで発生した熱は、ナノスケールでの表面及び熱抵抗を観測、ならびに半導体基板のパターニングとデバイスファブリケーションのプロトタイプのための数ナノメートルハーフピッチのマスクの製造に使用することができる。
このプロジェクトの主な目標であった「ミリピードプロジェクト」用にIBMが開発した加熱カンチレバープローブ技術を我々の専門と組み合わせ、SThMの分解能の限界をプッシュする原子分解能AFMを開発することに成功した。

研究成果の概要（英文）：Scanning thermal microscopy (SThM) is a variant of atomic force microscopy (AFM) that uses a heat-sensing cantilever probe. The heat generated by this cantilever probe can be used to study the thermal resistivity of surfaces and devices at the nano scale, as well as for the fabrication of masks of a few nanometers half-pitch that can be used for the patterning semiconductor substrates and prototype device fabrication. The main goal of this project was to combine the heating cantilever probe technology developed by IBM for the "millipede project" with our expertise in the development of instrumentation for atomic resolution AFM to push the resolution limits of SThM. We reached our goal of developing a high-resolution SThM operative in UHV environment.

研究分野：複合新領域

科研費の分科・細目：ナノ・マイクロ科学　・　ナノ構造科学

キーワード：ナノプローブ　走査型熱顕微鏡　原子分解能原子間力顕微鏡

１．研究開始当初の背景

Scanning thermal microscopy (SThM) is a variant of atomic force microscopy (AFM) that uses a heat-sensing cantilever probe. The heat generated by this cantilever probe can be used to study the thermal resistivity of surfaces and devices at the nano scale, as well as for the fabrication of masks of a few nanometers half-pitch that can be used for the patterning semiconductor substrates and prototype device fabrication.

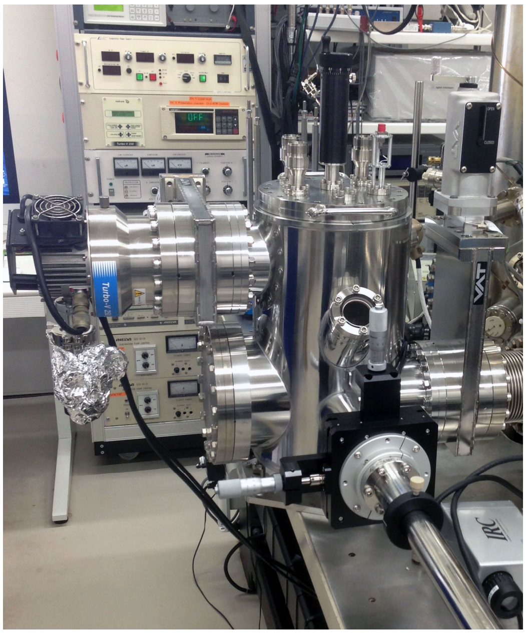
２．研究の目的

The main goal of this project was to combine the heating cantilever probe technology developed by IBM for the "millipede project" with our expertise in the development of instrumentation for atomic resolution AFM, and in particular, for the precise detection and regulation of the tip-surface interaction force.

The lateral resolution in SThM is determined by the geometry of the tip apex and the tip-surface area of contract. In atomic resolution AFM, we are able to control the tip-surface contact down to the formation of a single atomic bond between the foremost atom of the tip and the surface atoms. By applying the instrumentation techniques we currently use for atomic resolution AFM, we aimed to control the tip-surface contact with enough precision to perform SThM measurements with a lateral resolution below 1 nm (state-of-the-art SThM has a lateral resolution of ~25 nm), pushing in this way the resolution limits of SThM.

３．研究の方法

The development of a SThM with a lateral resolution below 1 nm requires operation of the microscope in an ultrahigh vacuum (UHV) environment to prevent the otherwise ubiquitous presence of a surface water layer that would yield to the formation of a water meniscus between the tip and the surface when bringing them into close proximity. Instabilities associated with the formation and rupture of a water meniscus would prevent a precise control of the tip-surface interaction force, and therefore hampers the ultimate control over the tip-surface contact area pursued in this project. Moreover, the presence of gas or a water meniscus between the probe and the sample results in an increase of the heat conduction cross-section with a consequent reduction of the lateral resolution in thermal measurements. To



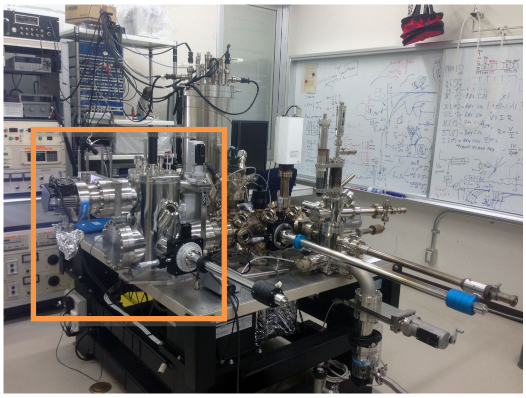
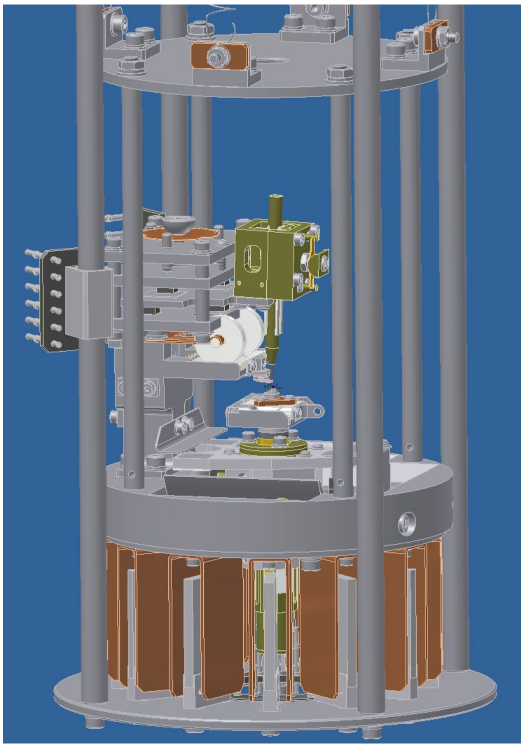


Figure 1: Detail of the UHV chamber accommodating the SThM microscope (upper panel), and the full UHV system (lower panel) in which the SThM has been attached. The orange square highlights the position of the UHV chamber hosting the SThM.

this end, we have designed a small UHV chamber that accommodates the high-resolution SThM prototype (Fig. 1). This set-up enables quick access to the microscope and the rapid execution of experiments in UHV by baking over night. This small UHV chamber has been coupled to a bigger UHV system (Fig. 1) containing instrumentation for preparing atomically clean probes and surfaces, and an operative low-temperature AFM.

The heating probe cantilevers developed by IBM have a design optimized for heat transmission and sensing: the tip is very short (~500 nm); it is located relatively far from the cantilever free end; and the cantilever is hundreds of micron wide. These restrictions compelled us to design a microscope prototype for exclusively using these heating probe cantilevers. Apart from the standard scan capabilities, the microscope prototype we designed has a special positioning system that allows



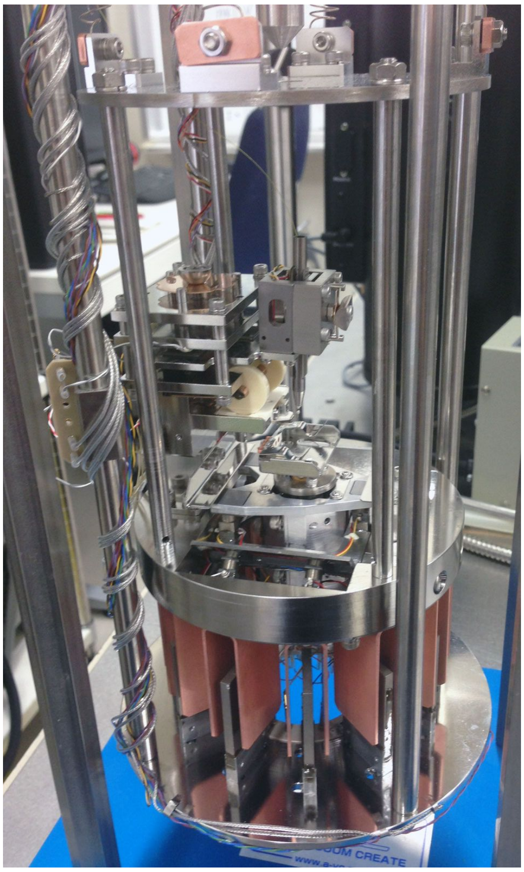


Figure 2: Design of the SThM (upper panel), and real SThM microscope constructed upon this design.

us to tilt the sample for an extremely well alignment of the plane of the sample surface with the cantilever chip (Figs. 2 and 3).

The microscope is in compliance with the high
degree of rigidity and stability required for atomic resolution experiments. It also

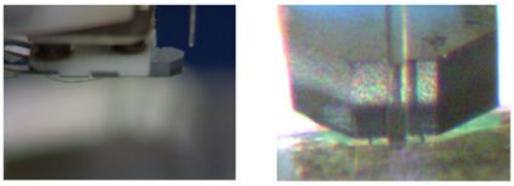


Figure 3: Detail of the alignment of the fiber of optical interferometer on the heat-sensing IBM cantilever probe: left, side view of the cantilever while approaching the optical fiber; right, front view of the scanning probe close to the sample surface surface.

has optical accessibility to positioning the probe over any desired location on the sample surface, and sample holders with multiple contacts to allow in situ operation of electronic devices for the characterization of the heat transfer during device operation conditions.

The detection of the cantilever defection and dynamics is based on the variation of the high-resolution optical-fiber-based interferometer that we use for atomic resolution AFM. The microscope is equipped with an additional set of piezoelectric motors for positioning the optical fiber over any location on the cantilever (Fig. 3).

４．研究成果

We reached our goal of developing a high-resolution SThM operative in UHV environment.

One of the main difficulties we faced is the appropriate design and construction of the required linear motors to properly operate the microscope in UHV conditions. Our SThM has seven axes of linear motion driven by homemade piezoelectric positioners: X, Y and Z motion of the sample; X, Y and Z motion of the optical fiber; and a tilt motion of the sample for perfect alignment of the plane of the cantilever chip with the sample surface. To build all these homemade piezoelectric positioners for a reliable operation took considerable time and effort of this project.

We also implemented the thermal calibration of the heating cantilever probes designed by IBM for the "millipede" project as well as the thermal detection mode, which is done by recording the current versus voltage characteristics of the heating probe cantilever (Fig. 4).

After building the microscope, checking the correct operation the seven axes of linear motion, and testing the microscope functions, we performed proof-of-concept experiments on a (111) surface of a diamond single crystal. The reason why we used a

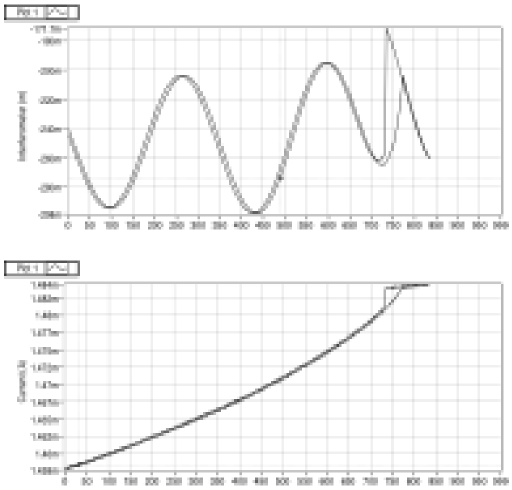


Figure 4: Optical (upper panel) and thermal (lower panel) signals recorded during the approach of a heating cantilever probe towards the surface.

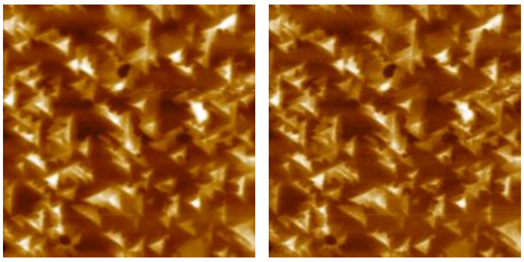


Figure 5: Topographic images of the Diamond (111) surface recorded (a) thermally by the heated scanning probe cantilever and (b) Optically by the Fiber interferometer.

diamond substrate for the experiments is because diamond it is a material with one of the highest thermal conductivity. We wanted to probe possible local variations of the thermal conductivity imposed by low dimensional structures at the surface, in particular, at small surface terraces and atomic steps of the diamond single crystal.

We established and demonstrated simultaneous thermal and optical operation of the microscope by successfully imaging atomic steps of the diamond (111) surface by both optical and thermal topography detection (Fig. 5). Although the heating probes designed by IBM for the “millipede” project are not designed for optical detection, the high-resolution specifications of our homemade optical interferometer made it possible to operate these cantilevers in standard AFM mode, measuring the topography of the sample through the vertical displacement of the cantilever when working in contact mode, or by detecting the dynamic response of the cantilever when measuring in dynamic mode. Figure 4 depicts the optical (upper panel) and thermal (lower panel) signatures of the heating probe upon approaching towards a diamond (111) surface. It is possible to recognize the landing of the tip on the sample surface by the change in thermal resistivity at the heating probe, that occurs simultaneously with the appearance of a discontinuity in the sinusoidal pattern of the optical interferometer signal.

We were able to record images, simultaneously in optical (Fig. 5 left panel) and thermal (Fig. 5 right panel) mode. The excellent agreement between these two imaging channels illustrates the successful construction and operation of the microscope. While the signal to noise ration of the optical channel is slightly better than those of the thermal channel, both imaging modes are capable to image atomic steps of the diamond (111) surface. This result is very promising towards achieving the anticipated atomic lateral resolution.

５．主な発表論文等

（研究代表者、研究分担者及び連携研究者には下線）

〔雑誌論文〕（計　１　件）

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①Oleksandr Stetsovych, Milica Todorović, Tomoko Shimizu, César Moreno, Rubén Pérez and Oscar Custance, “Characterization of pentacene on TiO_2 (101) anatase surface by cantilever-based intra-molecular atomic force microscopy imaging”, 1st KANSAI Nanoscience and Nanotechnology International Symposium, February 3-4, 2014, Osaka, Japan.

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③ Oscar Custance, “Clarifying atomic contrast of the TiO2(101) anatase surface by means of bimodal atomic force microscopy / spectroscopy and simultaneous scanning tunneling microscopy”, Materials Research Society Spring Meeting, April 1-5, 2013, San

Francisco, USA.
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〔図書〕（計 0 件）

〔産業財産権〕
○出願状況（計 0 件）

○取得状況（計 0 件）

〔その他〕
ホームページ等

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