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Scanning probe microscopy experiments in microgravity

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Abstract

The scanning probe microscopy setups are small, lightweight and do not require vacuum or high voltage supply. In addition, samples can be investigated directly without further preparation. Therefore, these techniques are well-suited for applications in space, in particular, for operation on the International Space Station (ISS) or for high resolution microscopy on planetary missions. A feasibility study for a scanning tunneling microscopy setup was carried out on a parabolic flight campaign in November 2001 in order to test the technical setup for microgravity applications. With a pocket-size design microscope, a graphite surface was imaged under ambient conditions. Atomic resolution was achieved although the quality of the images was inferior in comparison to laboratory conditions. Improvements for future scanning probe microscopy experiments in microgravity are suggested.

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1. Introduction

The scanning tunneling microscope (STM) invented by Binnig et al. in 1982 [1] was the first of a variety of scanning probe techniques (SPM) developed during the last 20 years. These methods gave access to objects and phenomena on the nanometer scale and are the fundament of the fast growing field of nanotechnology. SPM techniques can be applied to non-organic as well as organic or biological materials, and are widely applied for micro- and nanoscale investigation and engineering in basic and applied sciences.

Scanning tunneling microscopy allows for atomic resolution imaging of surfaces and the observation of individual adsorbed molecules, as well as in situ studies of chemical reactions on an atomic scale [2,3].

In contrast to electron microscopy, the SPM setups are small and do not require vacuum or high voltage supply. Samples can be investigated directly without further preparation. Therefore, SPMs are well-suited for applications in space, in particular for operation on the International Space Station (ISS), or for high resolution microscopy on planetary missions [4].

For the operation of a scanning probe device in microgravity, the experimental setup must be adapted to the conditions on board, e.g. small space, mounting possibilities and power supply [5]. Also, experimental

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difficulties such as mechanical and electrical disturbances can be a serious problem.

A feasibility study of an STM experiment was carried out on a parabolic flight campaign in November 2001. A tunneling microscope was chosen, because the stiff construction is less sensitive to vibrations and large gravitational changes than a force microscope [6]. During the microgravity phases, STM measurements of a graphite surface in ambient conditions were successfully carried out.

2. Experimental

2.1. Flight conditions

Depending on the methods utilized for the creation of microgravity (parabolic flights, satellite missions or Spacelab/ISS), the requirements for an experiment are quite different. The environment of a microgravity experiment differs greatly from usual laboratory conditions. Space is limited, as well as preparation and operation time. Every part of the instrument must be securely affixed. In a parabolic flight campaign there is a lot of noise, vibration and electrical interference, and shielding is difficult due to the limited mass of the whole setup. Also, experimental procedures have to be carefully adapted to the strict time schedule of the microgravity campaign.

The parabolic flight campaign was run by the company Novespace on an Airbus A300 aircraft from Bordeaux, France, under the scientific supervision of the German Aerospace Center (DLR). During the three-flight campaign the aircraft executed 93 parabola manoeuvres with a period of 22 s of microgravity.

The experimental setup had to follow the guidelines for parabolic flight experiments [7]. Each part of the equipment had to be fixed in a frame. In addition, the whole setup had to be as light as possible. The setup in flight configuration is shown in Fig. 1. The access time to the experiment in normal gravity was limited to about 15 min before the commencement of the parabolas and occasional normal flight phases up to 8 min between the parabolas. Sample preparation and microscope adjustments had to be carried out on the ground before the flights.

As the plane was not heated during the night, the temperature difference between preparation and flight was about 10 K. In a parabolic flight manoeuvre the gravitation on board of the airplane initially went up to 1.6–1.8 g for about 20 s, then dropped to 10^{-2} g for 22 s. Afterwards it rose once more to 1.6–1.8 g before normal gravity was restored. A typical z acceleration curve is shown in Fig. 2. In addition to the gravitational changes, there were strong mechanical vibrations caused by the engines and movements of the other operators in the plane.

2.2. STM setup

For the STM experiment, a homebuilt setup [8] based on the pocket-size STM design invented by Binnig and co-workers [9] was used (Fig. 3). The tunneling tip is mounted horizontally and the sample vertically. Thus the system is relatively insensitive to an alternation of vertical mechanical stresses caused by gravitational changes. The distance between tip and sample is controlled by 2 μm screws, which allow a manual adjustment. The maximum scan range of the four-segment piezo tube is about 1 μm in all directions.

As scanning electronics a modular system of analog scan generation and signal processing was used [10]. The signals were converted to a S-VHS video signal and recorded on a video tape. Although this technique has only an 8-bit resolution, it is easy to handle and robust. Due to the compact mounting of the experiment, the high voltage amplifiers showed a strong temperature-induced gain factor drift.

The microscope was mounted on a stack of copper plates of different size. These provided an effective damping of vibrations, due to their different resonant frequencies [11]. Two aluminum plates were added to the original design in order to mount the stack to the metal case without losing a vibration insulation stage. The plates were separated by pieces of Viton elastomer. The STM body was enclosed in a metal case placed in a large plastic box lined with both a layer of stiff and a layer of flexible foam in order to mechanically separate it from the surroundings. The box was fixed to the frame with four cable springs (WR2-800-10EM, Enidine GmbH, Bad Bellingen, Germany) for additional mechanical decoupling in microgravity.

The tunneling tips were prepared by electrochemical etching of a tungsten wire in 2 M KOH with a voltage of 10 V ac at 230 Hz [12]. The surface of the

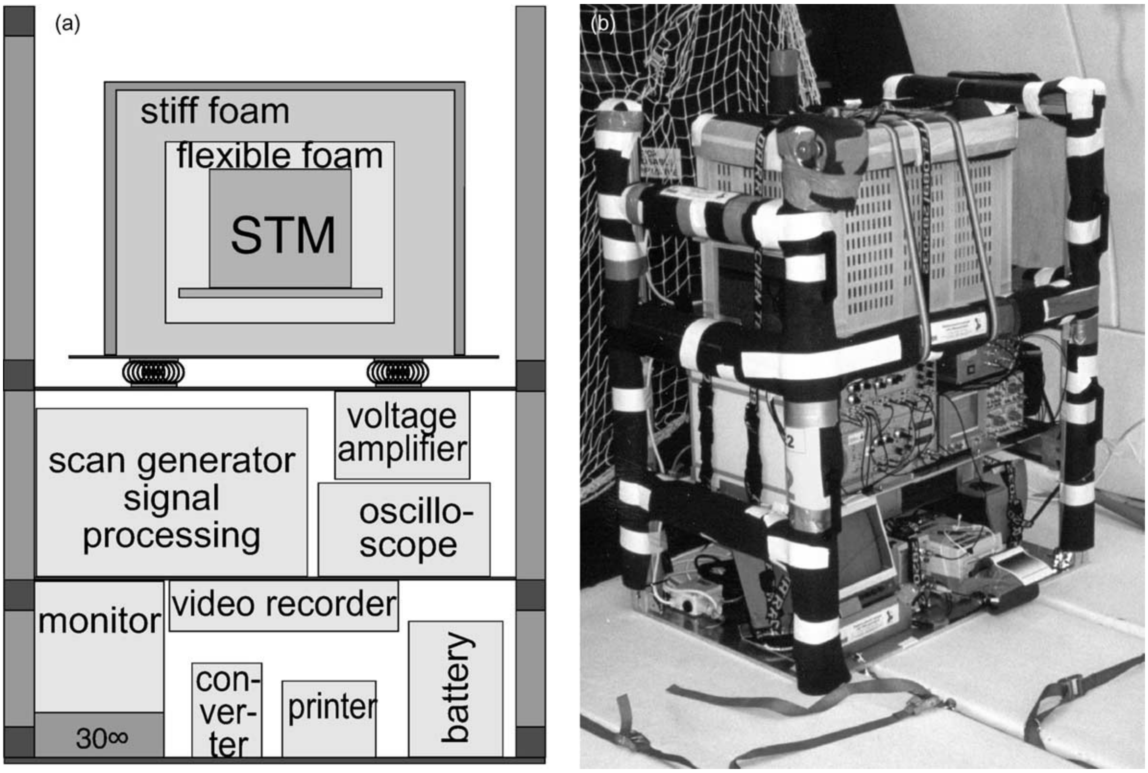

Fig. 1. The STM setup for the parabolic flight campaign: (a) schematic drawing. The microscope is placed in a box lined with two different layers of foam. The box is mounted on the frame by four damping elements. The scanning electronics and data acquisition devices are located below the microscope. The monitor is tilted by 30° for ergonomical reasons. (b) Photograph of the setup. The whole frame is covered with a foam layer to reduce the risk of injury during the flights.

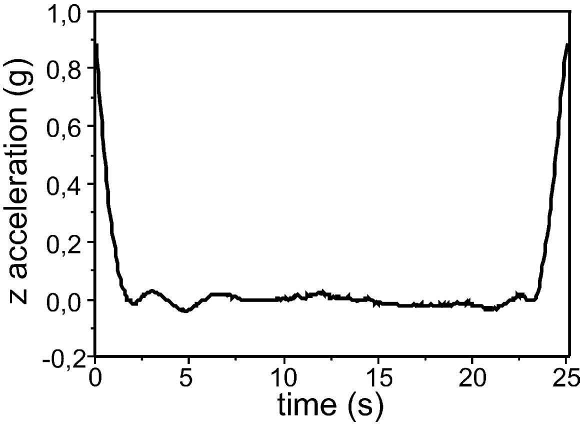

Fig. 2. Typical z acceleration curve measured in the cockpit of the aircraft during a parabola. The gravity drops within 2 s. At the beginning and the end of the microgravity phase there are some oscillations around the zero-g line. Sometimes negative accelerations occur (data: Novespace).

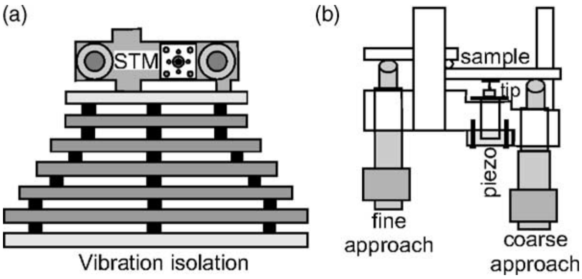

Fig. 3. Schematic drawing of the STM body (a) side view. The STM is placed on top of a stack of aluminum and copper plates of different sizes. (b) Top view of the STM body. The sample holder is pressed against an arm of the STM mount by 2 μm screws, allowing a well-controlled manual approach of the tip. The tip is mounted on a four-segment piezo tube scanner.

highly ordered pyrolytic graphite sample (HOPG) was prepared by cleaving with a strip of adhesive tape [13].

2.3. Experimental procedures

The manual tip approach was carried out before the flights. After the preparation procedure, the tip was retracted by contracting the piezo. During take-off, the piezo voltage was maintained by a battery system. Several manual readjustments of the tip–sample distance were necessary during the flights in order to compensate drifting.

The piezo performed a continuous scanning movement at 90 Hz. Measurements were made in a quasi-constant height mode using the tunneling current as

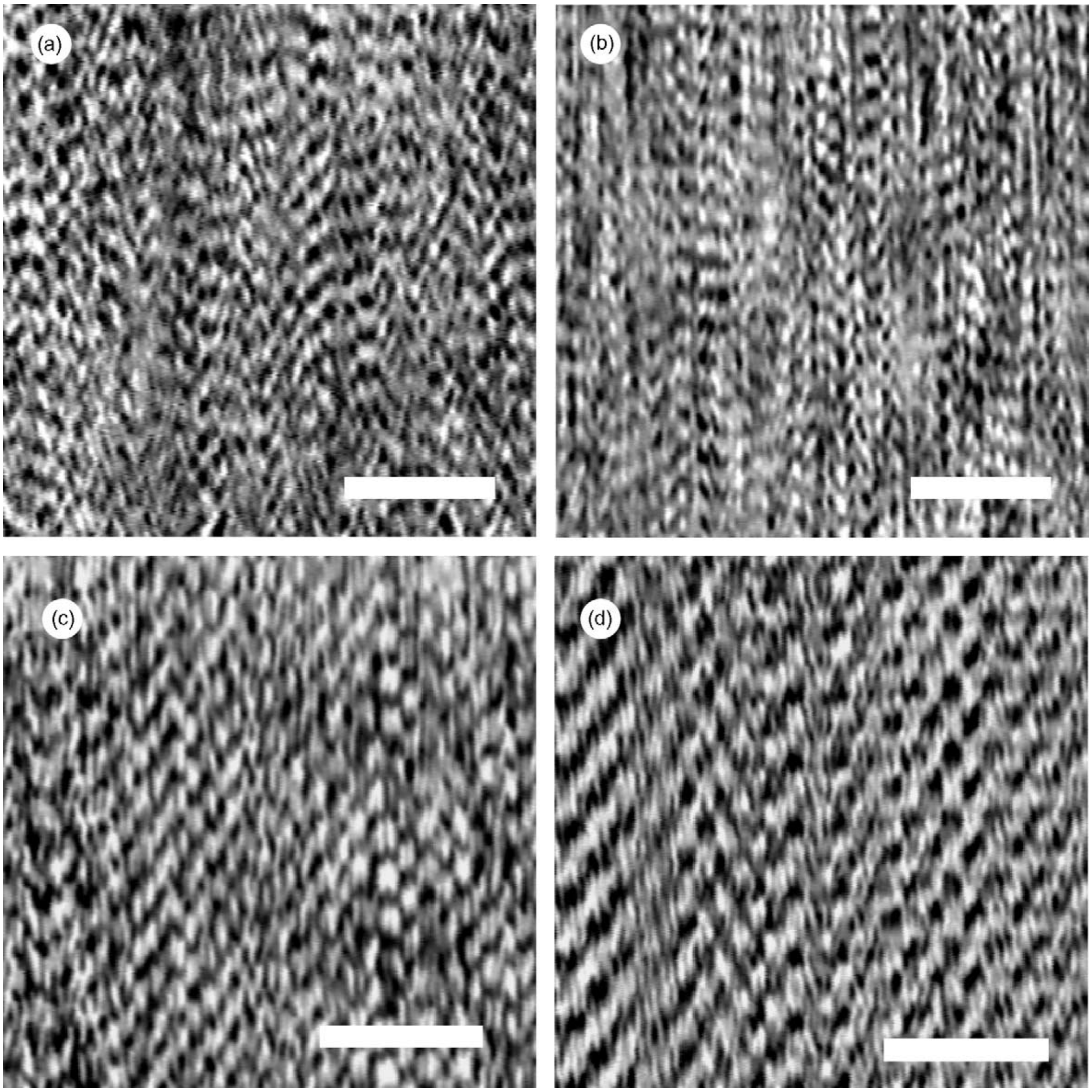

Fig. 4. STM images of a graphite surface recorded under different gravitational conditions. All images were measured in constant current mode: (a) microgravity (scale bar 1 nm), (b) 1.6–1.8 g (scale bar 1 nm), (c) at normal g in the plane on the ground (scale bar 1 nm) and (d) at normal *g* in a container hall on the airport (scale bar 0.6 nm). In the images made on the ground much less disturbance is visible.

the image signal, which allows for faster scanning than in constant current mode.

At the beginning of the microgravity phases the tip was approached to the surface. Half a scan was necessary for optimizing the system. During one period of microgravity it was possible to complete 2–3 images. It was also possible to record images in the supergravity phase. For each approach the imaging conditions were different, making it necessary to adjust the tunneling voltage and feedback parameters.

3. Results

The experimental conditions in the aircraft were not well-suited for a high resolution imaging method such as STM. In particular, mechanical vibrations and electrical noise disturb the measurements. The short periods of microgravity are an additional obstacle for a technique which requires the optimization of various experimental parameters such as scan speed and tunneling current. The optimal scanning parameters changed with the level of gravitation and thus were different for every microgravity phase. This was probably caused to alterations of the tip caused by small collisions. After optimization only about 15 s remained in a microgravity phase.

Several images of the graphite surface made under different conditions are shown in Fig. 4. It was possible to image the atomic features of the graphite surface in microgravity (Fig. 4a) although disturbances caused by electrical and mechanical noise are clearly visible in the images as well as in those from a supergravity phase (Fig. 4b). By alteration of the scan size it was proven that the features visible in the images correspond to the atomic structure of the graphite surface. The image in Fig. 4c was recorded in the aircraft on the ground with the engines not running. The mechanical noise visible here was transmitted via the floor.

The image shown in Fig. 4d was taken with the instrument placed in the ground laboratory, where the floor was much more stable than in the aircraft.

The vibration insulation was effective in the microgravity phases, but it could not dampen out all mechanical disturbances. In normal gravity it was insufficient to filter out both the impact sounds of experimentalists and crew and the noise of the engines. For future parabolic flight experiments the vibration insulation has to be improved to also function during normal gravity. For further improvement a galvanic separation of the electronic system and an enhanced electrical shielding are necessary. The STM measurement itself would profit from more rapid feedback electronics thus allowing a higher scan rate, and also from vibration compensation based on an integrated capacitive vibration sensor [14].

4. Conclusions

An ISS microscope setup could be used for quick and direct examination of materials and surfaces produced in experiments on board, as well as for in situ studies of, e.g. biological samples and crystal growth. A combined setup of an atomic force microscope and a scanning tunneling microscope would offer a multitude of experimental possibilities.

For SPM applications in space missions a variety of long run improvements are necessary. The conditions for operating a scanning probe microscope would be completely different to those of a parabolic flight campaign. The instruments have to work reliably over a long period of time. In case of an application on board of the ISS, all necessary standard operational work has to be done by the crew. Here the operators have no longtime experience with the microscope, and operating time is limited. An automatic approach of the tip combined with an in situ control of the tip–sample distance would simplify the preparation of the experiments. In the experimental setup of a space application it would be necessary to implement remote control.

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