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Contact atomic force microscopy using piezoresistive cantilevers in load force modulation mode

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ABSTRACT

Scanning probe microscopy (SPM) encompasses several techniques for imaging of the physical and chemical material properties at nanoscale. The scanning process is based on the detection of the deflection of the cantilever, which is caused by near field interactions, while the tip runs over the sample's surface. The variety of deflection detection methods including optical, piezoresistive, piezoelectric technologies has been developed and applied depending on the measurement mode and measurement environment. There are many advantages (compactness, vacuum compatibility, etc.) of the piezoresistive detection method, which makes it very attractive for almost all SPM experiments. Due to the technological limitations the stiffness of the piezoresistive beams is usually higher than the stiffness of the cantilever detected using optical methods. This is the basic constraint for the application of the piezoresistive beams in contact mode (CM) atomic force microscopy (AFM) investigations performed at low load forces (usually less than 20 nN). Drift of the deflection signal, which is related to thermal fluctuations of the measurement setup, causes that the microscope controller compensates the fluctuations instead of compensating the strength of tip-surface interactions. Therefore, it is quite difficult to keep near field interaction precisely at the setpoint level during the whole scanning process. This can lead to either damage of the cantilever's tip and material surface or loosing the contact with the investigated sample and making the measurement unreliable.

For these reasons, load force modulation (LoFM) scanning mode, in which the interaction at the tip is precisely controlled at every point of the sample surface, is proposed to enable precise AFM surface investigations using the piezoresistive cantilevers. In this article we describe the developed measurement algorithm as well as proposed and introduced hardware and software solutions. The results of the experiments confirm strong reduction of the AFM entire setup drift. The results demonstrating contactless tip lateral movements are presented. It is common knowledge that such a scanning reduces tip wear.

1. Introduction

Atomic force microcopy (AFM) is a well known technology used in the nanoscale investigations of the physical and chemical surface properties. In the AFM technology the strength of the interaction between microprobe and surface is detected basing on the observation of the AFM cantilever's static or resonance deflection. The most popular method used in this task is the optical beam deflection (OBD) technology [1]. It provides high sensitivity but it is difficult to be applied in the vacuum and low temperature experiments, due to troublesome optics adjustment. Moreover in the basic OBD setup the detection of the microprobe-surface interaction is done, whereas the quantitative (in other words metrological) investigations are possible only when more complex calibration procedure is applied. From that point of view, solutions in which the AFM cantilevers integrate a deflection sensor are very useful, especially when the interactions should be measured metrologically. The cantilevers with integrated deflection sensors are also very suitable in applications, where the AFM technology is combined with another experimental techniques like scanning electron microscopy (SEM), optical microscopy (OM) or focused ion beam (FIB) solutions. In these applications quartz tuning forks (QTF) and piezoresistive cantilevers have been sometimes utilized [2,3].

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It has been already presented that the piezoresistive cantilevers are very versatile microprobe sensors used in different applications [4–12]. The main advantages of the piezoresistive technology is the possibility of the sensor calibration independently on the system, in which the probe is applied [13,10]. Moreover, various piezoresistive cantilever devices for almost every scanning probe microscopy (SPM) mode can be fabricated, which enabled investigations of not only mechanical, but electrical or thermal surface properties as well [14]. Finally, the integration with the piezoresistive beam makes it possible to increase the scanning speed and surface imaging [15]. In this way, it was possible to design and fabricate massively parallel systems, which may speed up the surface characterization to the level required by the industrial production processes. The high sensitivity of the piezoresistive probes was shown in many papers, in which images of atomic structures of various crystals observed in the contact and non contact AFM modes (CM AFM and NC AFM) were shown.

Tortonese et al. presented the CM AFM images of subnanometer images of graphite, boron nitride, molybden disulfide and tantalum diselenide crystals recorded with cantilevers of stiffness ranging from 5 to 100 N/m. No indication as to the load force was given and as no crystal edges were observed it can be assumed that the presented images correspond with the atomic corrugations rather than the true atomic topography [3]. Jumpertz et al. presented CM AFM images of highly orientated pyrolytic graphite (HOPG) but as very little information on the tip-sample interaction were given, they should be interpreted only as qualitative ones [16]. The true atomic resolution on silicon surface was shown for the first time by Giessibl et al. [17], who presented images recorded in the frequency modulation (FM) AFM mode in ultra high vacuum (UHV). Recently a report on application of the piezoresistive cantilevers in the investigations of biological samples was published in which high resolution images of biological samples recorded in amplitude modulation (AM) AFM mode were shown [18].

Basing on our over two decade activities on the piezoresistive cantilever research, we feel obliged to note, that application of the piezoresistive cantilevers in the CM AFM is still a challenging task [19]. In contrast to the resonance AFM technologies in the CM AFM the interactions between the tip and the investigated surface can be determined with very high accuracy, which constitutes the biggest advantage of this technology. Moreover, if the load force acting at the tip is low, the obtained in this way measurement results reflect directly the surface properties. The reason, why the CM AFM investigations using piezoresistive cantilevers are quite seldom reported is the high stiffness of the piezoresistive cantilevers stemming from relatively huge thickness of the beam in which the piezoresistors are integrated. The surface imaging was usually done at load force bigger than 50 nN, which was limitation of many surface measurements. There a reports on piezorestive probes, whose stiffness less than 5 N/m [20,21]. However, it should be noted, that the described in these papers solutions do not integrate tips for topography surface scanning. In work [22] it has been shown that adding only of a conductive tip reduced deflection resolution to 2,4 nm (with force resolution ranging from 1,9 nN to 36 nN were probe stiffness was not reported). Moreover, in such a setup even the smallest thermal, mechanical or electrical drift of the piezoresistive cantilever and/or measurement head may lead to huge variation of the load force acting at the tip, which in turn can cause damage of the tip or sample surface modification.

Here, we present so called load force modulation (LoFM) mode in which the interaction at the tip is precisely controlled at every point at the sample surface. This is done by cyclic approach and retraction of the tip to and from the investigated sample. In the performed cycle the piezoresistive deflection detector output is analyzed and the feedback setpoint is calculated so that to maintain precisely the defined load force. In this way the surface may be scanned precisely in contact, even if the cantilevers are used with stiffness of around 100 N/m. The LoFM is based on the solution called Pulse Mode AFM proposed by Rosa-Zeiser et al [12]. In this setup the analog PID controller was driven by a sample and hold (SH) circuit responsible for measurements in and out of the tip-surface contact. More advanced system was proposed by Pablo et al. [23]. In the described architecture a digital signal processor (DSP) controller was applied to analyze the interaction between the tip and the investigated surface. In the mentioned above setups soft cantilevers with stiffness less than 5 N/m detected only by the OBD were utilized. The main purpose of the described experiments was to investigate the mechanical surface properties and record the force-distance curves.

In our setup we applied special algorithms to detect the tip surface distance by smart analysis of the tip-surface contact in the attraction and retraction phase. In this way we are able to distinguish between the snap-in and adhesion phenomena, whose dynamics differs dramatically. Moreover the applied piezoresistive cantilevers were calibrated basing on the thermomechanical noise analysis, which made it possible to quantify the load force and the tip deflection detection sensitivity [10,24]. In the LoFM technology the XY movements are done only when the piezoresistive cantilever is withdrawn. It is not an easy task for the piezoresistive cantilevers, due to thermal drift present in the signal. Digital analysis of the deflection signal is required in order to identify the "out of contact" state. In this way the tip wear is reduced and the load force is precisely controlled. In the developed scheme we are able to detect the load force in the range from 2 nN up to 100 nN with the resolution of 2.4 nN and the time period in which signals are analyzed at every point on the surface varies from 3 ms till 20 ms. The described resolution is limited by the range of observable deflections for a cantilever with stiffness equal to 75 N/m. The increase or decrease of the stiffness scales this range up or down.

The proposed technology is also suitable for the surface measurements performed using scanning thermal microscopy (SThM) and conductive atomic force microscopy (C AFM) modes. In both modes the LoFM technology offers the possibility to change and/or monitor the measurement conditions at the tip "in contact" and "out of contact" state. The constant load force maintenance is also a very important factor in this kind of measurements.

In order to implement the LoFM algorithms we designed a dedicated module, enabling to control the entire tip approach-retract process, signal acquisition and analysis and finally the load force definition. The core of the LoFM controller is formed by an ARM microprocessor, which ensures the needed software and hardware flexibility. The designed solution makes it also possible to scan the surface in the classical way when the proportional integral derivative (PID) controller defines the load force while sample scanning.

2. Methods

2.1. Piezoresistive cantilever

In our experiments we used cantilevers which integrate a piezoresistive Wheatstone bridge deflection sensor and a 4-wire electrically connected metallic tip - Fig. 1. The presented structure is foreseen to be applied in the SThM experiments, in which the thermal flux between the resistive tip and the surface is monitored [25,26]. The architecture of the cantilever is optimized so that the structure openings improve thermal and electrical shielding between the resistive probe, the tip wiring and the Wheatstone bridge deflection sensor. When the cantilever is bent the mechanical stress occurs in the whole cantilever volume, including the middle structure beam, in which the Wheatstone bridge is integrated. The signal U_D of the bridge diagonal voltage is proportional