Mechatronic Demodulation of Self-Sensing Cantilever for DC-bias free AFM Imaging in Liquid

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***Abstract*—This paper presents the application of mechatronic demodulation for performing dynamic mode AFM measurements with self-sensing cantilevers in liquid. In the proposed approach the bridge circuits with the piezoresistive sensing elements are supplied by an ac voltage that corresponds to the cantilever oscillation frequency. In contrast to conventional read-out circuits, no dc voltages are applied to the piezoresistive elements. Therefore, electrochemical effects leading to a deterioration of the self-sensing cantilever are avoided. This is demonstrated by immersing a self-sensing cantilever in water and comparing microscope images for operation with dc and ac voltages. To verify the feasibility of using mechatronic demodulation for dynamic mode AFM imaging in water the topography of a calibration grating is measured.**

***Index Terms*—atomic force microscopy, self-sensing cantilever, demodulation, dc-bias free, piezoresistive detection**

I. Introduction

Atomic Force Microscopy (AFM) is widely used for the analysis of biological samples due to its unique capability of imaging topography and various other surface properties in aqueous solutions [1]. AFM measurements of biological samples are typically carried out by dynamic measurement modes, such as Amplitude Modulation AFM (AM-AFM), due to the low tip-sample interaction forces with the soft samples [2].

In AM-AFM the cantilever oscillation amplitude is kept constant by a feedback controller, which requires the measurement of the cantilever deflection. The commonly used optical beam deflection (OBD) method [3] for measuring the cantilever deflection has several important limitations for AFM imaging in liquids. It requires an optically transparent path for the laser light to pass from the source via the cantilever to the detector, which prevents AFM measurements in opaque liquids, such as blood or crude oil [4]. Additionally, the implementation of the OBD method can be tedious since it requires specifically designed holders and liquid cells which enable an immersion of cantilever and sample in liquid while additionally providing an optical path for the laser beam. The laser alignment process is also complicated by the fact that the beam typically has to pass through multiple air/liquid/glass interfaces.

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To overcome these limitations self-sensing cantilevers with integrated piezoresistive elements have been used for AFM measurements in liquid [5]. No external components are needed for the deflection measurement which makes the design of the cantilever holder and the liquid cell significantly easier and enables measurements in opaque liquids [4]. To detect small changes of the resistances due to the cantilever deflection, the sensing elements are typically connected in a bridge circuit supplied by a dc voltage. However, dc voltages in liquid excite unwanted electrochemical effects or bubble formation, which can damage the sample or the cantilever [6], [7]. A widely used method to avoid electrochemical corrosion of self-sensing cantilevers is covering the electrical connections by insulating coating [5], [8]. While it has been shown that suitable coatings can provide protection from the liquid environment for up to several hours, it is reported that variations of the deposited layer thickness have a significant impact and limit the repeatability of the fabricated probe [9]. Additionally, it is vital to coat all conductive parts which are immersed in liquid, which may include the connection of the probe to subsequent electronics by bond wires which are difficult to uniformly coat due to their complicated shape. As a result, the required passivation leads to a more cumbersome microfabrication process and to higher costs of self-sensing cantilevers. In summary, the commonly used deflection measurement electronics, consisting of sensing elements connected in a dc bridge circuit, can lead to unwanted electrochemical effects or the requirement for an insulating coating, thus limiting the wider applicability of self-sensing cantilevers for AFM imaging in liquid.

As shown in [10], [11], mechatronic demodulation enables a measurement of the oscillation of self-sensing cantilevers by connecting the piezoresistive elements in two independent bridge circuits supplied by in-phase and quadrature sinusoidal signals. Thus, the amplitude and phase of the cantilever oscillation can be measured without the application of a dc voltage. It is therefore expected that this method avoids the occurrence of unwanted electrochemical effects and enables robust operation of self-sensing cantilevers in liquid.

The contribution of this paper is the implementation of dc-bias free dynamic mode AFM imaging in liquid with self-sensing cantilevers. This is achieved by mechatronic demodulation, which applies an ac voltage to the piezoresistive sensing elements instead of the commonly used dc-bias. A commercially available self-sensing cantilever with piezoresis-

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tive sensing elements is immersed in water. Dc and ac voltages are applied to the piezoresistive elements and the effect of the voltages on the self-sensing cantilever is observed by a microscope. While the application of a dc voltage leads to a deterioration of the cantilever over time, no adverse effects can be observed when applying an ac voltage. To verify the applicability of the mechatronic demodulation method, AM-AFM topography measurements of a calibration grating in water are performed.

II. Mechatronic Demodulation

Figure 1a shows the conventional method for measuring the oscillation of self-sensing cantilevers with piezoresistive elements. Two piezoresistive elements are connected in a half-bridge circuit supplied by a dc voltage U_0. The bridge output voltage is amplified and applied to a lock-in amplifier for demodulation to determine the amplitude A and phase ϕ of the cantilever oscillation at a given frequency f_{osc} (e.g. the resonance frequency of the cantilever). Depending on the measurement mode, the amplitude and/or phase is measured or used for feedback control during AFM imaging. A downside of the conventional method is the appearance of dc voltages directly on the piezoresistive elements as well as on the conductive traces for the electrical connection, which may start electrochemical processes during AFM imaging in liquid.

Figure 1b shows the working principle of mechatronic demodulation for self-sensing cantilevers. The piezoresistive elements are connected in two independent bridge circuits supplied by in-phase and quadrature sinusoidal signals with a frequency of f_{osc}. The multiplication of the bridge supply voltage with the variation of the resistance due to the cantilever oscillation leads to a demodulation at the output of the bridge circuit. The amplitude and phase of the cantilever oscillation can therefore be obtained without external demodulator. As described in [10], this simplified demodulation leads to a significant reduction of the required sampling frequency of the used digital hardware for dynamic mode AFM measurements. A major advantage of mechatronic demodulation with respect to the conventional method is that no dc voltages are present on the sensing elements or other conductive parts on the chip. Due to the high resonance frequencies of AFM cantilevers the frequency of the ac bridge supply voltages is in the order of tens of kHz to hundreds of kHz. It is therefore expected that the method can avoid unwanted electrochemical effects when performing dynamic mode AFM measurements with self-sensing cantilevers in liquid.

Before actual AFM measurements in liquid are performed, a self-sensing cantilever is immersed in water and the effect of applying dc and ac voltages to the sensing elements is investigated.

III. Self-Sensing Cantilever in water - DC vs. AC bridge supply

In this work, a self-sensing cantilever with two piezoresistive elements is used (PRSA-L70-F900-Si-PCB, SCLSensortech, Vienna, Austria). For the experiments in this Section

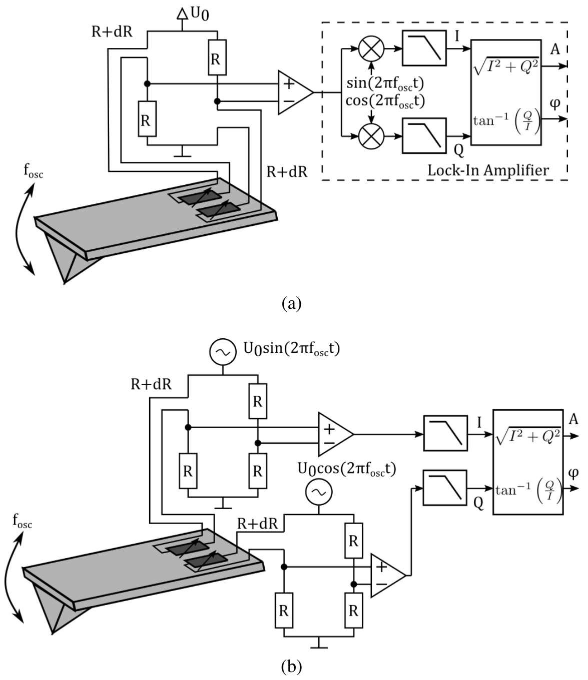

Fig. 1: (a) Deflection measurement by piezoresistive elements in dc bridge circuit and demodulation by lock-in amplifier. (b) Mechatronic demodulation using ac bridge circuits for direct measurement of oscillation amplitude and phase [10].

the cantilever is not mechanically excited or deflected. The piezoresistive elements are connected in a half bridge circuit as shown in Figure 1a. The two constant resistors are integrated on the chip in close proximity to the cantilever. For observing the self-sensing cantilever it is glued in a petri dish which is filled with tap water and placed under a microscope. A Function Generator (Keysight 33500B) is used to apply different voltages to the bridge circuit. Simultaneously, the bridge output voltage is recorded by a digital voltmeter (Keysight 34461A). Due to a small mismatch of the integrated half bridge circuit the manufacturer specifies a non-zero but constant bridge output offset voltage even without cantilever deflection. In a regular operation of the cantilevers an analog circuit would be used to subtract the offset from the bridge output and amplify the resulting voltage with a gain of 100. Here, the bridge output voltage is directly measured to detect potential changes of the electrical resistances during the experiment.

First, a dc voltage of $U_0 = 2\,\mathrm{V}$ is applied, which is the nominal supply voltage for the self-sensing cantilever. Figure 2a shows microscope images of the cantilever. The left image shows the cantilever shortly after switching on the voltage. The remaining images are recorded in intervals of 30 minutes as denoted on top of the images. The comparison shows changes on the cantilever as well as on the chip over time. Most noticeable is the bubble on the cantilever, which appears immediately after switching on the voltage and increases in

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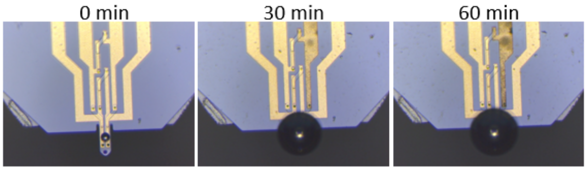
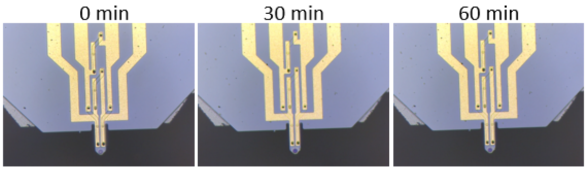


(a)



(b)

Fig. 2: Microscope images of self-sensing cantilever operated in tap water with (a) dc and (b) ac bridge supply voltages.

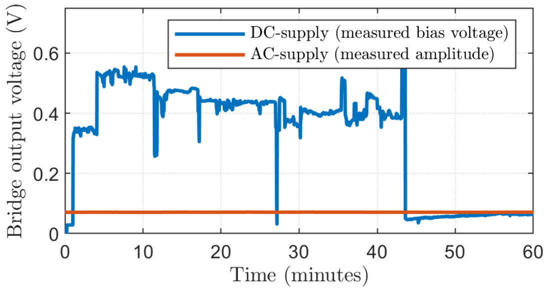
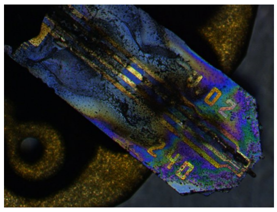


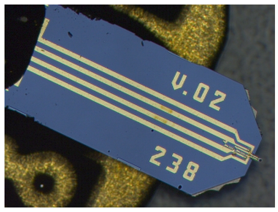
Fig. 3: Bridge output over time for dc and ac supply voltages.

size over time. Additionally, after 30 minutes it can be seen that one of the conductive traces appears significantly darker, which is believed to be due to corrosion. Figure 3 shows the measured bridge output voltage during the experiment. Variations of up to hundreds of millivolt can be observed. Given that this voltage is amplified by a factor of 100 during normal operation, this variation by far exceeds any potential detection of cantilever deflections and would therefore make AFM imaging impossible.

It is emphasized that neither the bubble formation nor the large variation of the output voltage are directly reproducible. This is found by repeating the experiment multiple times by subsequent cleaning of the cantilever with isopropanol and re-immersion in water. The depicted images and measurements are those obtained during the first 60 minutes of immersing a new cantilever in water. Bubbles rarely occur directly on the cantilever but rather on other parts of the conductive traces on the chip. While the output voltage repeatedly shows large variations, periods with relatively small variations are observed as well (see for example the last 15 minutes in Figure 3). It is therefore expected that the operation of the sensing elements in a dc bridge circuit may be possible in general, but the mentioned effects probably lead to unexpected



(a)



(b)

Fig. 4: Microscope image of the self-sensing cantilever after several hours of applying (a) dc and (b) ac supply voltages.

behaviour during AFM imaging.

Figure 2b shows microscope images of the cantilever when an ac voltage with an amplitude of $U_0 = 2\,\mathrm{V}$ and a frequency of $f_{osc} = 39\,\mathrm{kHz}$ is applied to the cantilever. The frequency is selected since it is the resonance frequency of the cantilever used for AFM imaging in Section V. The microscope images show that no bubbles are forming and no accumulation of particles or corrosion of the conductive traces is observed over time. Figure 3 show the measured amplitude of the bridge output voltage. The amplitude is constant which indicates that the electrical impedances of the sensing elements are constant during the experiment. The results are reproducible and remain unchanged for multiple repetitions of the experiment.

For a further analysis of the impact of the different supply voltages, the cantilevers are maintained in water with active supply voltages for several hours. The cantilevers are then taken out of the water and blow dried by beakers. Figure 4a and Figure 4b show the resulting microscope image after the experiment with dc and ac voltages, respectively.

The microscope images and the measured bridge voltage show that the application of a dc voltage leads to a significant deterioration when the self-sensing cantilever is operated in water. In contrast, no deterioration is observed when applying an ac voltage. It is therefore expected that the mechatronic demodulation method enables robust dynamic mode AFM imaging of self-sensing cantilevers in water.

IV. Experimental Implementation

The self-sensing cantilever and its connector are glued on a dither piezo which is mounted on a commercial AFM scanner (Dimension, Bruker, Billerica, USA). Figure 5 shows an image of the experimental setup, with the commercial AFM scanner in the center and the flat ribbon cable connecting the cantilever connector to a PCB on the right side of the image. The sample and the self-sensing cantilever are immersed in a petri dish filled water during AFM imaging.

For the implementation of the mechatronic demodulation method the piezoresistive elements are implemented in two separate quarter bridge circuits as shown in Figure 1b. The bridge circuits, differential amplifiers and analog low-pass filters are implemented on a custom-built PCB [10]. The filter outputs are applied to a RedPitaya FPGA board where the mathematical operations for calculating the oscillation

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Fig. 5: Image of experimental setup for implementation of AM-AFM imaging with mechatronic demodulation in water.

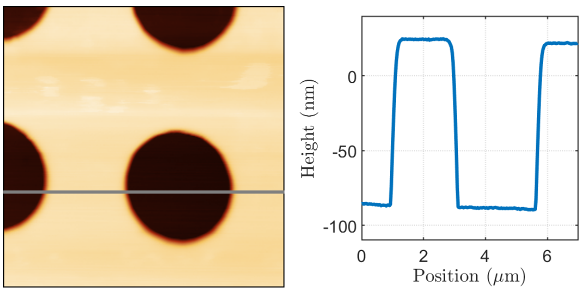
amplitude are implemented. The resulting amplitude is applied to the AFM system via a signal access module.

V. AFM Imaging in Water

For a verification of the feasibility of mechatronic demodulation for AFM imaging in water AM-AFM topography measurements on a calibration grating (HS-100MG) are performed. The grating consists of circular pits with a depth of 113 nm. The used cantilever has a resonance frequency of 39 kHz in water, which is determined by applying a frequency sweep to the dither piezo. For the measurements in water the cantilever is excited by the dither piezo at 39 kHz.

Figure 6a shows the measured topography. A cross section of a single scan line indicated by the grey horizontal line is shown in Figure 6b. The pits on the calibration grating can be clearly resolved with a height resolution < 0.5 nm (measured standard deviations of the flat parts within and between the pits of the cross section). The results verify the feasibility of using mechatronic demodulation for performing dc-bias free AM-AFM measurements with self-sensing cantilevers in water.

In summary, dynamic mode AFM imaging with self-sensing cantilevers in liquid is successfully demonstrated using mechatronic demodulation, thus enabling a dc-bias free deflection measurement and avoiding unwanted electrochemical effects.



(a) (b)

Fig. 6: AM-AFM measurement of calibration grating (HS-100MG) in water using mechatronic demodulation. (a) Topography image and (b) cross-section. The scan size is 7 μm × 7 μm and the scan rate is 1 Hz.

VI. Conclusion

The oscillation amplitude of a self-sensing cantilever is measured by mechatronic demodulation which enables dc-bias free dynamic mode AFM imaging in liquid. In conventional read-out circuits for self-sensing cantilevers, the piezoresistive elements are connected in dc bridge circuits for measuring the deflection, which is applied to an external demodulator for determining the oscillation amplitude and phase. It is demonstrated that the dc voltage leads to a deterioration of the cantilever when it is immersed in water. Mechatronic demodulation enables a direct measurement of the oscillation amplitude and phase by connecting the piezoresistive elements in ac bridge circuits operated at the oscillation frequency. With this method, no deterioration of the cantilever is observed when it is immersed in water. The feasibility of using mechatronic demodulation for AFM imaging in water is confirmed by AM-AFM topography measurements on a calibration grating.

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