QUARTZ CRYSTAL RESONATOR AS A SENSOR IN SCANNING PROBE MICROSCOPY

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***Abstract*—A brief review of the Scanning Probe Microscopy (SPM) on basis of a Quartz Crystal Resonator (QCR) is done. A look on principles of design and development of electronic interface circuits for QCR based SPM is given. A simple and versatile interface for a QCR based SPM developed is presented and some applications are shown.**

***Index Terms*—Quartz crystal sensor, Scanning Probe Microscopy, interface electronic,**

I. INTRODUCTION

Despite of a wide choice of industrial cantilever based force sensors in scanning probe microscopy (SPM), complementary approach such as symmetrical quartz crystal resonators (QCR) e.g. tuning fork (TF), trident tuning fork and needle quartz as a platform are of great interest. QCR belong to the most frequently used sensors in engineering because of their low internal energy dissipation, high temperature as well as mechanical and chemical stability, and relatively high piezo-constants. Recently, various QCR such as tuning fork (TF), trident TF and needle quartz were introduced as a platform for an attached sensor in different SPM techniques such as acoustic, force, optical near-field and magnetic force microscopy (see review articles [1], [2] and literature therein). A high quality factor, even in air, and high spring constants are preconditions for resolving short-range forces and stable operation in force microscopy. Further, very low energy dissipation at low excitation amplitudes, the possibility to maintain a sub-nanometer tip-sample gap without repulsive mechanical contact (snap-in) and low dielectric losses even at high frequencies are crucial factors in specific cases e.g. in optical near-field, scanning microwave or capacitance microscopy.

A high performance electronic interface is a precondition for the realization of QCR advantages in measurements. A modern interface electronic should deliver fast and precise QCR oscillation amplitude and frequency (and/or phase) data in order to perform stable distance control and investigate dissipative and conservative tip-sample forces. Design of suitable interface circuits for QCR requires special circuit configurations, which are not to be obtained by simply modifying standard applications. Miniaturization and component selection are also crucial for minimizing parasitic effects. Here we compare and discuss various QCR excitation modes and signal processing used in SPM applications. Further, we briefly discuss the perspective of new piezo materials (langatat and langasit) as quartz competitor in SPM.

Finally, we present characteristic experimental data obtained by using QCR based sensor in SPM on various samples.

II. SCHEMATICS FOR SIGNAL PROCESSING AND DISTANCE CONTROL

Precision distance control with stable sub-nanometer resolution is a need in many SPM techniques: Scanning Near-Field Microscopy (SNOM), Scanning Capacitance Microscopy, Magnetic Force Microscopy, Scanning Microwave Microscopy, etc. (anywhere the interaction is characterized by long range signals $1/d$ or $1/d^2$, where d is the tip-surface distance).

The short-range non-contact mechanical force distance control is preferred over other methods [2]. There are mainly two alternative sensor platforms used in SPM: the cantilever and the QCR. The QCR (a watch 32kHz TF) was first introduced as platform in SNOM in 1995 (K. Karrai and R. D. Grober [3]).

The trend in SPM interface electronics in the last years goes toward self-oscillation schemes (always operating on the resonance frequency). The simpler variant with one amplitude feedback loop and only amplitude modulation/demodulation (AM) circuit (e.g. [4], [5]) is a good choice for distance control on samples characterized through dissipation and stiffness values lying in the not-extreme range. More versatile and now most used in distance control and precision tip-sample interaction measurements is a self-oscillation scheme with amplitude and frequency feedback loops (introduced in 1991 Ref. [6], see also [7], [8]). In AFM a technique known as Q-control [9], [10], is also widely used. Here the positive feedback in force signal is adjusted between zero and one, to manipulate the effective Q-factor. Note this technique is very similar to a method in radio known as regenerative receiver. Later this technique was found less useful in communication, and now can be found only in amateur radio.

Fig.1 shows the typical “workhorse” schema in QCR based SPM with amplitude and frequency feedback loops [11]. The goal of the electronic is to drive the QCR platform at its resonant frequency with constant amplitude and to measure the frequency shift of the QCR or/and amplitude control signal changes induced by the force interaction.

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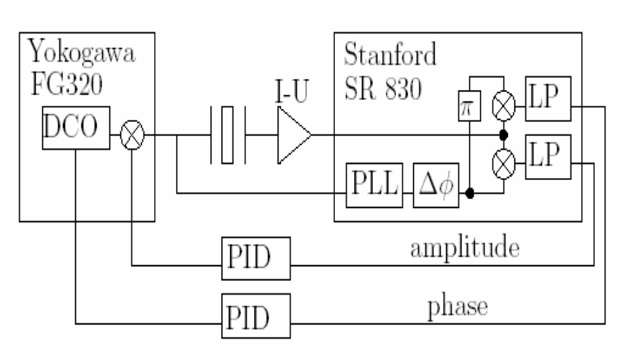

Fig. 1. Typical structure of an experimental setup in SPM technique.

The use of excessive and expensive commercial devices (here function generator FG320 and Lock-In amplifier SR830) does not solve problems with low-noise signal pre-amplification, parasitic capacitance compensation etc. The solution of these problems requires the development of more specific circuits, typically positioned in the close proximity of the sensor. A look on older related techniques such as microbalance technique or sonar could be helpful on this point. Especially the microbalance technique is very similar. In principle it differs only by measurement of equivalent inductance (*L*) changes instead of equivalent capacity (*C*) changes in QCR based SPM technique. The resistance (*R*) changes measurements important in both. Here the *RCL* describes mechanical properties of the QCR according to the Butterworth–Van Dyke Model (*L* corresponds to the mass, *C* - force gradient, *R* - damping) [12], [13].

All these techniques use similar signal processing which originates from communication technique. However, the progress, at least in the SPM technique, was only with large delay influenced from the communication technique (e.g. in the SPM the frequency modulation/demodulation was introduced only in 1991 [6], the phase modulation - in 2006 [14]) only.

On the other hand, in communication technology the signal modulation/demodulation (modem) using separation of the in-phase and the quadrature components (the I/Q modem) became generally accepted routine method. Now it is possible on the base of circuitry developed in radio-communication to create very simple, cheap and nearly optimal devices for the SPM. Furthermore, the physics in synchronous I/Q processing applied to SPM has some new aspects. AFM theory [15] shows that any frequency change in signal due to tip-sample interaction is entirely determined by conservative interaction force. The amplitude change depends on both dissipative and conservative forces (typically much more on dissipation). This means that the both I and Q components should be analyzed to investigate dissipative processes.

Fig. 2 shows the similarity of signal processing and the comparison of radio schemes and QCR based SPM. The carrier frequency is in both techniques modulated by applying of pressure (microphone in radio) or force (in SPM), and then demodulated for baseband processing. Shown is the simple synchronous I/Q processing schema. A more expensive schema with carrier frequency converting can be advantageous in some special situations (e.g. for very fast detection circuits).

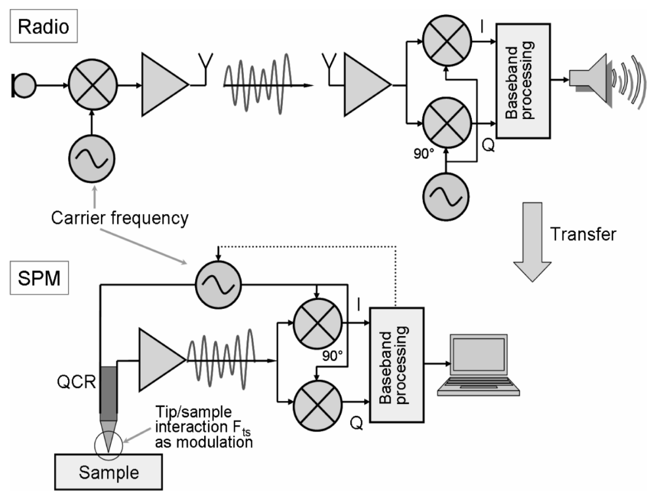

Fig. 2. Comparison of synchronous radio and SPM processing.

The application of synchronous I/Q modem in QCR based SPM is depicted in Fig. 3. Shown is the principle of separation in independent phase and amplitude loops (arrows in Fig.3).

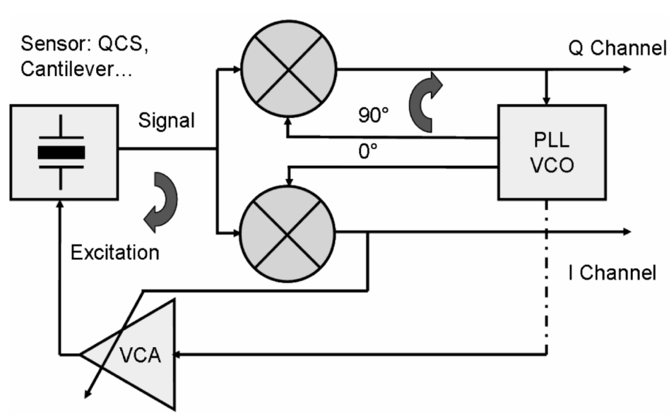

Fig. 3. The complex signal is separate processed in the amplitude and the phase loop

These considerations make plausible that a synchronous I/Q modem schema, where the Q channel signal detects changes in frequency, full separated from changes in the amplitude channel, is preferred to be used for distance control in SPM. Furthermore, in some situations (e.g. on surfaces with high dissipation) it would be of advantage to use both I and Q signals (appropriately weighted) to perform distance control.

With view on commercialization, it is clear that the market demand relates to the complete integration of the different aspects of a SPM: sensor, detection, interface electronic, protocol analysis and data analysis. The development toward a "lab on a chip" device is obvious for the future efforts also in SPM techniques.

III. SCHEMATICS DEVELOPED

In order to supply our SPM developments (RF scanning noise microscope and SNOM) with precise distance control we developed interface circuits applicable to various QCR's. The first variant was a full analog circuit with a tunable VCXO, the second shown in Fig. 4. an analog-digital variant with DDS as frequency generator and an ATMEGA µP.

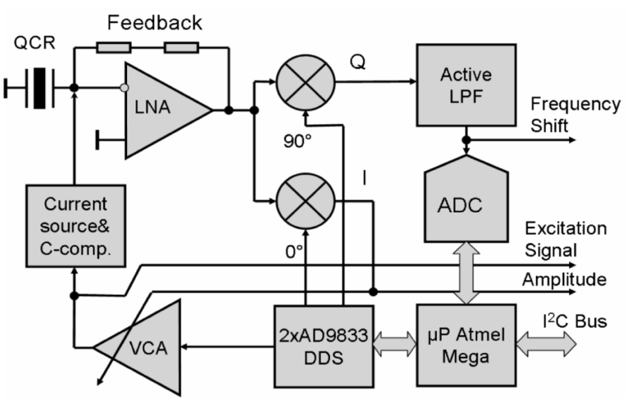

Fig. 4. Schematics of the interface electronic developed on base of µP ATMEGA. The current source is used to excite the QCR in constant excitation mode.

On the input we are used a wideband, low-noise JFET operational amplifiers OPA657 or AD8067, providing high performance with any QCR up to 10MHz resonance frequency. The need of a current source drive is discussed in [16], the capacity compensation importance – in [12]. Other main IC's: the ADC is an AD7680, VCA – AD 8337. In the Fig. 5 the device developed is shown.

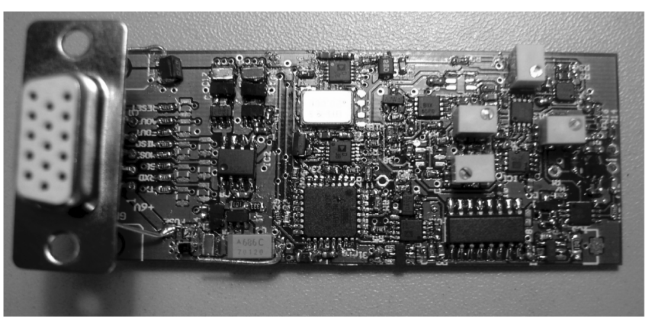

Fig.5. Interface electronic designed on a 4-layer printed board, dimensions 7cm x 3cm.

Fig. 6, 7 and 8 present the performance of our development on cleaved clean mica, obtained in ambient condition. As QCR is used a "needle quartz" with a radio frequency sensor attached.

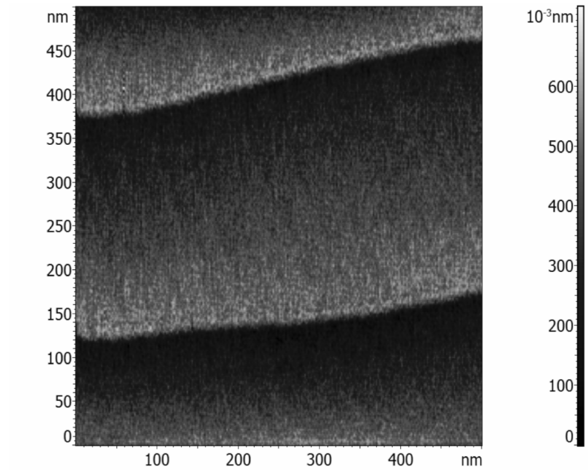

Fig. 6. Atomic steps on mica.

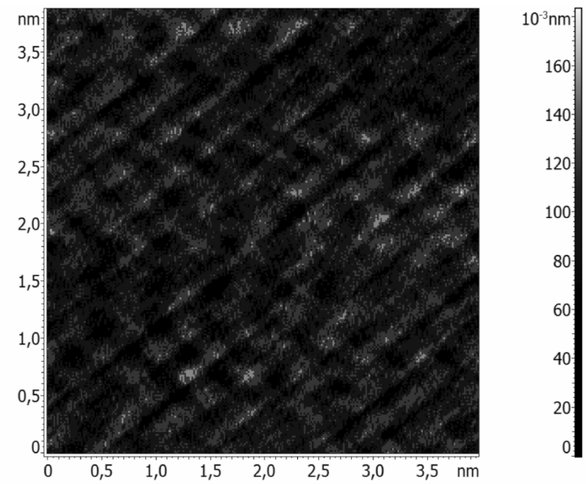

Fig. 7. Atomic resolution on mica

The approaching (red) and retracting (blue) curves in Fig. 8 corresponds to the frequency shift signal versus distance to the surface. The region where the tip is experiencing attractive or repulsive forces lies near 1nm. The hysteresis is due to the thin water film on surfaces at ambient conditions.

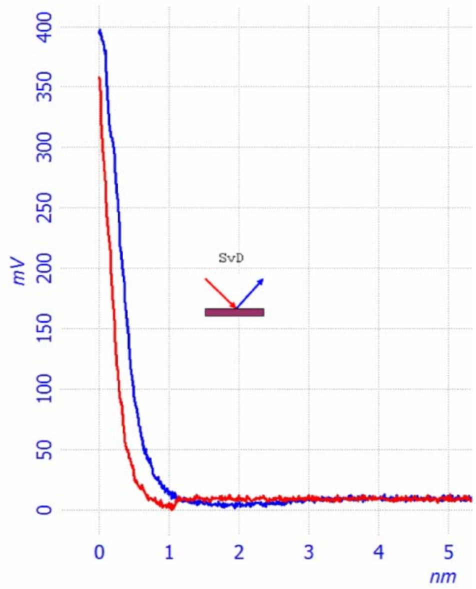

Fig.8. Signal versus distance curves on mica. Red line corresponds to the approach of the tip to the surface, the blue – to the retracting.

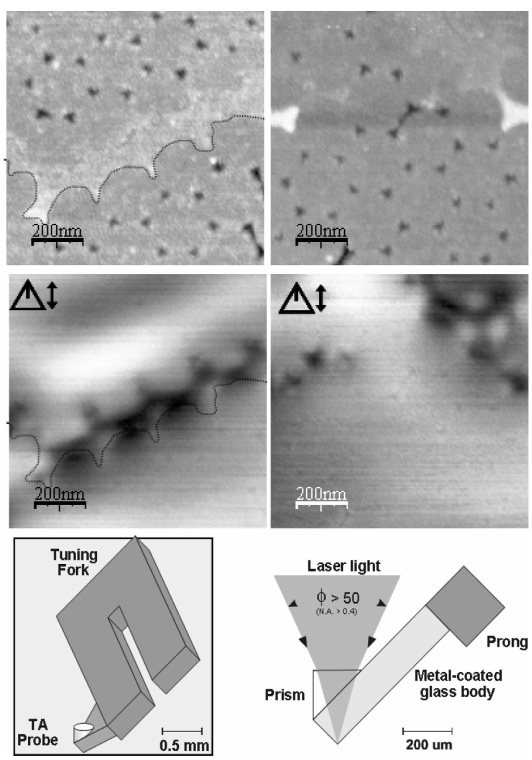

Fig. 9. Topography and optical images on special structures for plasmonic excitation (in cooperation with T. Maletzky).

The application as distance control in SNOM technique is demonstrated in Fig.9. The top line of the image is the topography of the metalized SNOM pattern, in the middle – optically detected plasmon waves and on the bottom - the SNOM tip attached to a 32kHz TF.

The quartz has a relatively low electromechanical constant and is very stiff. Therefore, measurements on soft materials may damage the surface. Desirable were new materials with higher electromechanical constant and lower stiffness. Single crystals of compounds with Ca3Ga2Ge4O14 (CGG)-type structure (langasite, langanite, langatate) that are promising new materials for surface-acoustic-wave (SAW) devices, perhaps can be candidate. However, low Q and low temperature stability of resonators made of these materials limited up to day the applications.

IV. Conclusion

Interface electronic and principles of QCR based SPM techniques briefly discussed in comparison with related techniques. A realization of interface electronic designed on a small and cheap board located near the QCR in presented. Some application of our development demonstrates performance comparable with best commercial SPM. New sensor materials briefly discussed.

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