# **SQUID** Activities at PTB: Status 2008

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*Abstract* – The purpose of this article is to overview the current status of R&D into SQUID technology and applications at the German National Metrology Institute, PTB. We summarize the recent progress in SQUID electronics and sensors. Subsequently, we succinctly describe the status in SQUID applications emphasized at PTB. These are: biomagnetism, cryogenic radiation detector readout, metrology, nondestructive evaluation (NDE), low-field nuclear magnetic resonance (NMR) and NMR at very low temperatures.

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### I. INTRODUCTION

The German National Metrology Institute uses the acronym PTB (Physikalisch-Technische Bundesanstalt). It provides scientific and technical services in the broadly defined field of metrology. The PTB operations are located in Braunschweig and Berlin. Two PTB groups, both located in the Berlin Institute, are involved in SQUID technology and a variety of SQUID applications. These groups are: the Department 7.5 "Cryo- and Vacuum Physics", and the Department 8.2 "Biosignals". Over the years, beginning with early 1980s, PTB has been one of the main European contributors to SQUID technology and applications, especially to biomagnetism. In much of this work, a unique tool has been the world's most effective magnetically shielded room (MSR). The purpose of this article is to overview the status of the SQUID effort at PTB by the end of 2008. It has been prepared jointly by leaders of the two groups identified above.

### II. SQUID SENSOR DEVELOPMENT AND SQUID- RELATED TECHNIQUES AT PTB

We have been developing both, low- $T_c$  and high- $T_c$  SQUID sensors (LT and HT SQUIDs, respectively). This effort includes the development of integrated sensor chips, sensor packaging and read-out electronics. In 2007 PTB decided to desist from fabricating HT SQUID chips, but in the framework of cooperation with other institutes (*e.g.*, the Korean metrology

institute, KRISS) the work on applications of HT SQUIDs has been continued on a small scale.

The purpose of SQUID development at PTB is to provide the community with highly sensitive SQUID sensors and sensor systems and related equipment for metrology, basic research, and selected industrial applications. At present, our SQUIDs are mainly used for the development of novel medical diagnostic techniques and as extremely sensitive amplifiers, *e.g.*, for the read-out of cryogenic radiation detectors. In particular, during the last few years the still growing interest in robust and easy-to-use SQUID amplifiers, which can be operated in the temperature range below 1 K and down to 10 mK, has stimulated intensive work on complex devices with several hundreds of SQUIDs and auxiliary components on chip. Standard sensor designs and parameters have been engineered. Beginning with such more complex devices in 2002, the 6<sup>th</sup> generation "C6" of the PTB sensor family is currently under development.

The sensors and electronics developed at PTB have been made commercially available by the German company Magnicon GbR (see <u>www.magnicon.com</u>), which has opened a new branch in Berlin in November 2007. It is located on PTB premises, with access to the PTB infrastructure. The ESNF published in 2007 the paper <u>ST-2</u> on high-performance dc SQUID sensors and electronics, written jointly by PTB and Magnicon authors.

#### **III.SQUID ELECTRONICS**

We have developed general purpose dc SQUID electronics with up to 6 MHz bandwidth that can be used for the read-out of LT SQUIDs, and because of its bias reversal option for HT SQUIDs as well. The unique integrated ultra-low-noise current source makes possible the compensation of external magnetic fields. This is especially useful in our patented sensor package that includes a compact field compensation coil system [1–3].

In 2005, novel ultra-fast, user friendly, fully computer controlled, direct-coupled SQUID electronics, the XXF-1, was developed in cooperation with Magnicon. These electronics feature open-loop bandwidth of 50MHz and flux-locked loop (FLL) bandwidth of 20MHz. The parallel combination of a fast ac amplifier and a high-quality dc amplifier guarantees excellent performance from dc to rf frequencies. It is especially useful in combination with a number of SQUID-based current sensors developed at PTB during recent years. This sensor family includes single SQUID devices, SQUID arrays and two-stage SQUID devices. The XXF electronics supports the easy operation of these sensors, which will be described in more detail below. Of course, these electronics can be used also to operate other dc SQUIDs. The low electronics current noise allows for low-noise operation of SQUID series arrays. Additional integrated current sources provide, e.g., bias current and calibration pulses for TES (transition edge sensor) applications as well as bias and feedback currents for two-stage SQUID operation. The state-of-the-art control software "SQUIDViewer" provides convenient control of these electronics [4, 5]. Figure 1 of the ESNF paper ST-2 shows a photo of XXF-1 FLL board and also the complete electronics system.

Extremely fast FLL operation was recently demonstrated by D. Drung, who introduced a cooled FLL operated in liquid helium. This cold electronics consists of two transistor stages acting as a high-gain amplifier and buffer. A FLL bandwidth of 350 MHz could be achieved [6].

### **IV. SQUID SENSORS**

### A. LT SQUIDs

As mentioned above, we have developed a novel family of low-noise SQUIDs which cover a wide range of applications. These sensors are robust and easy to use without compromising noise performance. For the read-out of cryogenic detectors, series arrays of 16 SQUIDs with <3nH input inductance were designed which can be cooled down and operated in the Earth's magnetic field without magnetic shielding. A compact gradiometric design allows the chips to be mounted directly on a Cu block at the cold stage of a mK cryostat without degradation in noise. A current noise level of 9 pA/Hz<sup>1/2</sup> is achieved at 4.2 K, while at 300 mK a lower noise value of 5 pA/Hz<sup>1/2</sup> is obtained.

For applications requiring a larger input inductance of up to 2  $\mu$ H, integrated two-stage sensors were developed consisting of a single front-end SQUID with double-transformer coupling readout by a 16-SQUID array. These sensors are very convenient to use as their voltage-flux characteristic is essentially single-SQUID-like. Devices optimized for 4.2 K operation have a coupled energy resolution around 50 *h* (*h* is the Planck's constant). Resolution of 4 *h* could be demonstrated with heavily shunted devices at 300 mK. Other features (filters, input current limiters) are integrated for convenient use. The layout and basic circuit diagram of such integrated two-stage current sensor C4XL 116 is shown in Figure 3 of ESNF paper <u>ST-2</u>, so we don't reproduce it here.

Magnetic field sensing applications, *e.g.*, biomagnetic research, are addressed with PTBs standard SQUID multiloop magnetometers on 7.2 mm x 7.2 mm chips. These sensors have a white magnetic field noise of typically 1.2 fT/  $Hz^{1/2}$ . For applications where a smaller chip size is required, integrated miniature multiloop magnetometers were designed with maximized field resolution. For a 3 mm x 3 mm chip size, a noise level of 3.6 fT/  $Hz^{1/2}$  is obtained.

For novel diagnostic or spectroscopic techniques (magnetorelaxometry, low-field NMR and MRI) requiring a magnetization of the samples, SQUID sensors have been designed which can be used after exposure to prepolarizing magnetic fields of up to 3 mT (*e.g.*, sensor with white magnetic field noise of 0.6 fT/Hz<sup>1/2</sup>). These magnetometers and gradiometers are based on gradiometric SQUID current sensors with a wire-wound pick-up coil. Adjustable on-chip current limiters are available to avoid flux trapping in the devices caused by too large input currents [6].

In the framework of the joint research project "NanoSpin" funded by the European Commission within the scope of a metrology research program, PTB is developing, in collaboration with NPL, a nanoSQUID systems intended for the detection of single magnetic moments. In first joint experiments with such a system consisting of a nano-structured SQUID loop and a series SQUID array amplifier a white flux noise level of  $0.2 \ \mu \Phi_0/Hz^{1/2}$  ( $\Phi_0$  is the magnetic flux quantum) could be demonstrated which corresponds to a predicted spin sensitivity of ca. 2 units of electron spins per root hertz [7]. Figure 1 shows current-flux characteristics of a nanoSQUID from NPL read out with a 16 SQUID array current sensor of PTB. The current through the nano-SQUID is plotted as a function of the applied external magnetic field. The inset shows a SEM image of the NPL nanoSQUID.



**Fig. 1.** Current-flux characteristics of a nanoSQUID (NPL) read out with a 16 SQUID series array current sensor (PTB). The inset shows the SEM image of the nanoSQUID with nearly square loop size of 370 x 370 nm<sup>2</sup>.

#### B. Larger LT SQUID Circuits

Novel dc current sensors (SQUADs) have been developed consisting of an array of identical dc SQUIDs. The input current is coupled tightly but non-uniformly to the SQUID array elements resulting in a single-valued, non-periodic overall voltage response similar to Superconducting Quantum Interference Filters (SQIF) based on arrays of non-identical SQUIDs. These sensors are of interest in particular for metrological dc current measurements. A dc current resolution of < 1nA could be demonstrated [8].

SQUID amplifiers are a proper choice for the read-out of low-impedance cryogenic radiation detectors. Large-format arrays of such sensors require multiplexing techniques. A novel SQUID multiplexer (SQMUX) for time-division multiplexing (TDM) has been introduced recently in [9]. The novel aspect of this device are its superconducting-to-normal (conducting) switches (SN-switches) which are used to activate and deactivate individual readout channels in a reliable and low-loss manner. Series arrays of 40 SQUIDs serve as SN-switches. This new SQMUX scheme reduces complexity and improves reliability for SQUID-based TDM [9]. Figure 2 shows the layout (a), and the basic circuit diagram (b) of the time-division multiplexer chip C5X416FLM. Circuit details are omitted for clarity. Up to four TES sensors can be connected to the chip using the input pads on the right side. The four pads on the upper left side are used to connect the FLL electronics and the lower left pads are required for addressing the four channels. For frequency division multiplexing see Section V.E.



**Fig. 2**. Layout (a) and basic circuit diagram (b) of the integrated time division SQUID multiplexer chip C5X416FLM. The chip size is 3 x 3 mm<sup>2</sup>. Circuit details are omitted for clarity.

# V. SQUID APPLICATIONS

### A. Novel Biomedical Diagnostic Techniques

### Multichannel Systems

A number of multichannel SQUID systems are operated at PTB in magnetically shielded rooms. In the currently world's best shielded room, BMSR-2, which contains seven mu-metal and one aluminum shielding layer, and also a feedback-controlled active field compensation, a 304 channel vector-magnetometer is operated. In this device, 19 modules are equipped with 16 SQUIDs each mounted in different orientation, so that the vector of the magnetic field can be recorded at various positions. A unique capability of this experimental system is the registration of magnetic fields generated by infra-slow brain activity [10, 11].

# Special Cryostats with Internal Superconducting Shield

For investigating the heart activity of small animals (e.g., mice) SQUID cryostats with a horizontal warm bore have been developed in cooperation with the Kanazawa Institute of Technology, Japan, and the Japanese company Fujihira. In order to assure operation of the SQUID system in an unshielded laboratory environment, the warm bore is surrounded by a superconducting shield. SQUIDs are mounted around the warm bore, but within the superconducting

shield. This cryostat concept is intended for use also in magnetorelaxometry experiments (see below) [12]. Figure 3 shows the external view of an 18 channel magnetometer scanner for small animals. The liquid helium cryostat has a horizontal warm bore of 110 mm in diameter. The 18 SQUIDs are arranged in a circular loop around the warm bore and are very effectively shielded by a superconducting niobium cylinder.



Fig. 3. Small animal scanner with horizontal warm bore.

# Magnetorelaxometry (MRX)

In the MRX method, SQUIDs are used for the measurement of the magnetization decay after a short polarization field was applied to magnetic nanoparticles (MNP). Such nanoparticles are promising new agents in cancer therapy. The *in-vivo* application of MNP requires a reliable measurement technique that makes possible the localization and quantification of particle agglomerations. PTB has developed magnetorelaxometry as a tool that provides information on the biodistribution of MNP. In addition, MRX is suitable for quality control of MNP preparations [13-15]. A newly developed system employs the cryostat with internal superconducting magnetic shield mentioned in the previous subsection. The system is equipped with SQUID magnetometers consisting of a wire-wound pick-up coil and SQUID current sensors with high immunity against interfering magnetic fields, which are described in Section IV. These sensors detect sample magnetization and a MRX signal within the magnetically shielded warm bore of the cryostat. This setup gives a very sensitive and compact MRX measuring system in comparison to the initial system that has to be operated in a heavily magnetically shielded room.

### Low-Field NMR (LF-NMR)

Recently, it was pointed out that nuclear magnetic resonance (NMR) at fields around a few microtesla offers a variety of new applications both for spectroscopy and medical imaging (MRI). SQUIDs have a much better sensitivity than induction coils for NMR signals at Lar-

mor frequencies below a few kilohertz. We have developed a SQUID based LF-NMR spectrometer with outstanding resolution and sensitivity, which can be used for the investigation of heteronuclear J-coupling and <sup>1</sup>H relaxometry at low frequencies. Using the 304-channel vector magnetometer, the spatiotemporal information of the recorded signals can be utilized for magnetic resonance imaging in low fields (LF-MRI). LF-MRI may enable simultaneous anatomical and functional imaging of the human brain [16-19]. Furthermore, PTB SQUDs are used for low-field NMR at Royal Holloway University of London in the framework of a collaboration [20]. Figure 4 shows a sketch of single-channel SQUID detection setup, and a photo of such a setup operated in BMSR-2.



**Fig. 4.** Low-field NMR setup. Left: sketch of single-channel SQUID detection of low-field Larmor precession. The polarization field generated magnetization along the y axis. After switching offthe the polarization field, sample magnetization begins to precess about the z axis of the detection field. Right: Photo of the LF-NMR setup operated in BMSR-2.

# B. Nondestructive Evaluation (NDE) by Laser SQUID microscopy

A novel technique for nondestructive evaluation of semiconductor samples combines laser illumination and magnetic field detection with SQUIDs. The sample is locally illuminated by a focused laser beam and the optically generated photocurrents are detected magnetically by a SQUID magnetometer. It has been demonstrated that this detection technique can be used for failure analysis in wafers, and devices such as photovoltaic cells. The spatial resolution of this technique is determined by the size of the laser spot (typically in the range of 5 to20  $\mu$ m), so relatively large SQUID magnetometers can be used. For the evaluation of special defects in photovoltaic samples or semiconductor circuits it could be demonstrated that alternatively to SQUIDs room temperature sensors (*e.g.*, induction coils) can also be utilized [21, 22].

# C. Nuclear Magnetic Resonance at Very Low Temperatures

SQUIDs are a powerful tool for detecting NMR signals of samples at very low temperatures, both in tuned and untuned spectrometer input circuits. Broadband and tuned NMR spectrometers in millikelvin-cryostats have been setup at Royal Holloway University of London utilizing PTB SQUID sensors. Especially the use of integrated two-stage SQUID amplifiers with a

single front end SQUID read out by a 16 SQUID array could improve the noise performance of the spectrometers. The additionally integrated 16 SQUID current limiter operates as a Q-spoiler which reduces the recovery time of the system from large current transients following removal of an NMR transmitter pulse. For the tuned system, a noise temperature of ca. 7 mK was measured with a frequency of 0.884 MHz [20,23-24].

### D. Metrological Applications

#### Noise thermometry

Noise thermometry employing SQUIDs is a well established method for absolute thermometry below 1 K. Its unique advantage is the large working range from a few K down to the submK range. The use of SOUID-based noise thermometers is still a domain of national standards laboratories, because of sophisticated design and measurement procedures. We have expended efforts to design novel SQUID-based noise thermometers for practical use in lowtemperature laboratories. Whereas integrated thin-film resistive SQUIDs seem to be limited to a temperature range down to about 100 mK [25], novel magnetic field fluctuation thermometers (MFFTs) which have been developed in close cooperation with the University of Heidelberg (group of Prof. Enss) are quite promising. The MFFT is a compact and easy-to-use thermometer for the temperature range of about 10 mK to 4K. It is based on the measurement of the thermal magnetic noise above the surface of a conductor. A statistical uncertainty of about 1 % can be achieved in a measurement time on the order of 20 seconds, which makes it attractive for many users of dilution refrigerators. The MFFT acts as a "semi-primary" noise thermometer, because one needs just a single reference temperature. Its high linearity and excellent resolution makes the MFFT a fast and practical alternative to existing thermometer solutions. Meanwhile it is available commercially (see www.magnicon.com) [26, 27].



**Fig. 5.** The magnetic field fluctuation thermometer (MFFT) mounted on the mixing chamber plate of a <sup>3</sup>He/4He dilution refrigerator. The insert shows the open MFFT with Cu block and the mounted radial SQUID gradiometer chip 3 x 3 mm<sup>2</sup>.

#### Cryogenic Current Comparators

For the maintenance and dissemination of the electrical units, such as the unit of the electrical resistance (Ohm), SQUIDs play an important role when comparing currents by using so called cryogenic current comparators (CCCs). These devices are of importance for electrical quantum metrology. Recently, an improved resistance calibration setup based on a CCC equipped with a state-of-the art PTB SQUID and a digital double current source has been constructed and commissioned [28].

### E. Cryogenic Detector Readout

SQUID amplifiers are well suited for the readout of low-impedance cryogenic radiation detectors such as superconducting transition edge sensors (TES), magnetic microcalorimeters (MMC), and nanowire single-photon detectors. PTB is involved in a number of collaborations developing detector array systems requiring suitable SQUID amplifiers and multiplexers.

For future X-ray observatories, a demonstrator is under development, which has the acronym EURECA (European-JapanEse Calorimeter Array). This should be a 5 x 5 pixel imaging TES-based microcalorimeter array requiring SQUID-based frequency-domain-multiplexing. This array is to be operated in an adiabatic demagnetization refrigerator. For the frequencydomain multiplexed readout of TES a high linearity and thus a high system slew rate are required even at high frequencies. Typically a FLL bandwidth of 100 MHz is desirable if one wants to measure signals up to 10 MHz. With room-temperature FLL electronics, the bandwidth limit determined by the transmission line loop is about 10 MHz. The loop delay can be decreased considerably using a cold FLL built with appropriate semiconductor devices. However, the dissipated power of such circuits which is on the order of mW is not acceptable for multichannel systems and millikelvin cooling [6].

We developed novel SQUID current sensors, where large SQUID arrays with up to 640 low-inductance SQUIDs are utilized to provide negative feedback on the sensor chip. It can be operated close to the TES with acceptable power consumption. This technique called output current feedback (OCF) makes possible very high unity-gain frequencies of >100 MHz not limited by the transmission delay. This is a major improvement over previous local negative feedback schemes [29]. Figure 6 compares the standard direct-coupled FLL with OCF.



**Fig. 6.** Comparison of (a) the standard direct-coupled FLL and (b) SQUID linearization by OCF [29]. The whole SQUID circuit is represented by the usual single device symbol, with bias circuit omitted for clarity. The light blue area is at cryogenic temperature. The right lower corner shows the simplified equivalent circuit of the room-temperature SiGe amplifier.

Magnetic microcalorimeters are promising sensors for astronomical X-ray spectroscopy. PTB is collaborating with University of Heidelberg and NASA Goddard Space Flight Center to develop SQUID based readout for these sensors. Prototype arrays fabricated by NASA have been read out with our 2-stage SQUID amplifiers in first test experiments [30].

In collaboration with the German Aerospace Center (DLR) and the University of Karlsruhe, PTB is developing broadband SQUID amplifiers for the readout of NbN nano-wire single photon detectors. Recently, the operation of NbN detector cooled with a mechanical cryo-cooler down to 1.2 K has been successfully demonstrated. For visible light, the noise equivalent power (NEP) was better than  $10^{-18}$  W/Hz<sup>1/2</sup> at 4.2 K; spectral energy resolution of 1 eV at 6.5 K was achieved [31, 32].

### VI. SUMMARY and OUTLOOK

Due to the rapid progress in the current decade, the classical "cartwheel" SQUID magnetometers utilized in magnetically shielded systems, e.g., in biomagnetic MEG or MCG instrumentation, are no longer the working horse of PTB, even for bio-medical applications. More and more complex SQUID current sensors, which include SQUID array amplifiers with integrated auxiliary components such as filters and current limiters, offer unique performance for a variety of applications. In combination with wire-wound pickup coils they can be used for the novel biomedical applications such as low-field MRI where large pre-polarizing fields are required. Furthermore, they are very attractive as preamplifiers for low-impedance cryogenic radiation detectors and other highly sensitive low-temperature measurement techniques. As a matter of course, one sensor type cannot fit all these applications. Meanwhile, a large number of specialized SQUID current sensor designs have been developed and tested, reaching from chips with one active SQUID up to circuits with several hundreds of SQUIDs on a single chip. This complexity and the need for smaller linewidths is a challenge for our fabrication technology, which is currently the bottleneck for further improvements of the designs. Our patterning techniques (optical contact lithography, wet chemical etching and lift-off) currently limit the minimum linewidth to about 2.5  $\mu$ m. We are thus hoping to procure novel equipment for e-beam lithography and plasma etching in 2010. This should open the door for more elaborated sensor designs and hopefully higher fabrication yields.

According to PTBs technology transfer policy, the SQUID related devices, electronics, measurement systems, and techniques are subject to licensing or collaboration agreements [33]. Recently, such agreements have been concluded with Magnicon and a number of research groups.

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