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1 Executive Summary

Introduction

Micro electromechanical systems (MEMS) are well-established in consumer products such as electronic displays, inkjet printers, car airbags and engine control systems, however there is increasing demand for more complexity, speed and performance combined with smaller size. Nano-Electro- Mechanical Systems (NEMS) is an emerging technology that can provide a potential solutions to a range of technological barriers for sectors including ICT, physical sensors and biological sensor applications.

The Problem

The trend towards ultra-miniaturisation in electronic components and circuits is mirrored by a similar trend in sensors and in the scale of measurement requirements. These trends pose extreme challenges and NEMS are an important part of the solution. Unlike electrical circuits, which are approaching some hard limits in size and frequency, NEMS devices have yet to be exploited at their limits of size or performance. They have the potential to offer measurement and sensor capabilities which exceed conventional electronic approaches for many applications.

If the metrology community does not anticipate and meet industries' metrological needs technological growth in Europe will be significantly limited. In the short term this project will yield highly innovative metrology with wide ranging applications. In the longer term major new security, biotechnology, IT and QIP (Quantum Information Processing) applications are expected, as well as enhanced quantum metrology. NEMS resonators and actuators will address several leading scientific challenges including 'nanotechnology, ultra-low-loss new materials, and metrology of single entities, leading in turn to future traceable measurement requirements in the bio, security, telecoms and sensing areas.

If Europe is to remain competitive in innovative industries with the USA and Asia it is crucial to address the challenges of measurement at the nanoscale. The project is very challenging; no single organisation can provide all the required capabilities at the leading edge, so collaboration with leading research academics and industrials is essential.

NEMS devices can detect single photons and single molecules; and general force, displacement, mass and voltage sensing at the nanoscale. However, there was no metrological framework for the robust assessment of NEMS prior to the start of this project. In order to exploit NEMS devices the limits of their performance needs to be measured and determined. Then new and improved NEMS devices can then be designed and fabricated using existing (aluminium nitride and silicon) and new materials, such as graphene and magnesium diboride (MgB2). Testing and validating the performance of the NEMS devices will also require new measurement methods.

Nanoscale devices, fabricated on silicon (Si) chips, will increasingly demand nanoscale on–chip measurement standards such as voltage and temperature. At the nanoscale, the definition of local temperature becomes unclear and understanding temperature is important as it is crucial to performance. In addition, the coupling between microwave and NEMS devices leads to the phenomenon of dynamic cooling, which needs to be investigated in order to achieve measurements 'beyond the classical' limits of thermal noise. SQUIDs are measurement devices that are very sensitive and used at low temperatures. New designs for the integration of SQUID-NEMS combinations need to be developed to enable the operation of NEMS resonators at very low temperatures close to the thermal equilibrium quantum limit (i.e. mK-temperatures).

The Solution

This project addressed the need for traceable measurement by developing novel high-frequency, high-performance NEMS devices, which were tested and validated using new specifically developed methods. Using the new measurement techniques it was possible to optimise fabrication techniques and new materials for NEMS devices. Arrays of NEMS devices were also tested. Dynamic cooling methods for measurements beyond the limit of thermal noise were also investigated, as well as the integration of NEMS with Superconductivity Quantum Interference Devices (SQUIDs).

Impact

The project addressed the development of sensors based on NEMS to provide traceable nanoscale metrology for the characterisation of materials, and other novel sensors at the micro/nano scales necessary for wider applications. An improved understanding of graphene mechanical resonators was achieved and novel excitation and readout methods for these promising nano-sensors were developed. A non-contacting method for the measurement of graphene sheet resistance was also developed and has attracted much industrial attention for measuring the electrical properties of other 2D materials as well as graphene. Semimetrics Ltd, a UK based SME that provides solutions for semiconductor measurement problems, is keen to develop and commercialise the non-contact method.

The novel nanoSQUID current sensors were developed by PTB and operated with modified SQUID electronics from project partner, MAG (Magnicon), a German supplier of high performance SQUID electronics. The low-noise nanoSQUID sensors/arrays and customised SQUID systems developed in this project are also being commercialised.

The enhanced sensor capabilities emerging from the project will have a positive impact on the development and deployment of NEMS-based sensors in areas such as security surveillance (through smaller and more sensitive sensors and detectors) and biosensors in the medical sector. NEMS sensors can also be used as ultra-sensitive mass detectors and NPL is already collaborating on a proposal to use NEMS biosensors to distinguish between the mass of different types of cancerous cells.

In the longer term the outputs of the MetNEMS project will provide improved ultra-sensitive, traceable measurements and traceable metrology for mass, force and displacement, temperature, single photon and single molecule measurement at the nanoscale, as well as the potential for on-chip ultra-stable miniaturised voltage references. The results of the MetNEMS project will support the development of metrology beyond the 'conventional classical limits' and will help address the implications of quantum measurement in nanoscale metrology.

2 Project context, rationale and objectives

The trend towards ultra-miniaturisation of electronic components and circuits is mirrored by a similar trend in sensors and in the scale of measurement requirements. However, these trends pose extreme challenges and Nano-Electro-Mechanical Systems (NEMS) are an important part of the solution. Unlike electrical circuits which are approaching hard limits in terms of their size and frequency, NEMS devices needed exploiting at the limits of their size or performance. Furthermore, NEMS devices promised measurement and sensor capabilities which exceed conventional electronic approaches in many areas of application.

Micro electromechanical systems (MEMS) are well established in consumer products and industrial demand for increased complexity, speed and performance is driving their size reduction towards NEMS. As a new key disruptive technology NEMS promised to provide solutions to industrial needs across a wide range of technological barriers and over a wide spread of sectors, from ICT through to physical sensors and biological sensor applications. However, as the dimensions of devices and structures are reduced, new technologies and approaches are required. These new technologies and approaches should both enable and demand innovative metrology, but prior to the NEW08 MetNEMS project there was almost no metrological activity to anticipate these impending metrological needs for NEMS exploitation.

It is crucial that Europe does not fall behind in the area of NEMS, given its enormous potential. Already advanced ultra-low temperature research has allowed one US lab (University of California, Santa Barbara (UCSB)) to achieve the quantum ground state of a NEMS resonator, which has the potential to provide quantum information processing capabilities way beyond present achievements and will enable beyond Uncertainty Principle metrology applications. Therefore, if Europe is to remain competitive with the USA and Asia in innovative industries it is vital that the challenges of measurement at the nanoscale are addressed, and the MetNEMS project supported this need. In the short term the MetNEMS project helped to yield highly innovative metrology with wide ranging applications and in the longer term major new security, biotechnology, IT and QIP (Quantum Information Processing) applications were expected from the MetNEMS project, as well as enhanced quantum metrology.

The MetNEMS project did great deal to meet the challenges outlined above, by focussing on developing novel high frequency, high performance nanoscale mechanical resonators and actuators, aimed at both metrology and industrial applications. This included the development of ultra-sensitive mass, force, displacement and temperature sensing at the nanoscale, single photon & single molecule measurement. In addition the project facilitated the production of ultra-stable and miniaturised voltage references.

NEMS developments both enable and demand innovative metrology and impact a wide range of sectors. Due to this, the development of NEMS resonators and actuators, by the the MetNEMS project, addressed several leading scientific challenges including 'beyond classical metrology', nanotechnology, ultra-low-loss new materials, and metrology of single entities; in turn leading to (future) traceable measurement requirements in the bio, security, telecoms and sensing sectors. An overriding objective of the MetNEMS project was to develop key capabilities in several NMIs in order to meet the needs of NEMS development. This included focusing on specifics, as there was a need to address the lack of traceable metrology in mass, force, displacement and temperature at the nanoscale. Improved measurement methods and traceability for dynamic measurements of MEMS/NEMS (i.e. structural shape of moving accessible and hidden surfaces) were also crucial for validating device modelling and to better understand operational behaviour.

The optimised use of NEMS has required development of advanced fabrication techniques and new materials. In order to meet this need the MetNEMS has extensively investigated a range of new materials such as graphene (a unique new 2D material with vast range of promising properties), Aluminium nitride (AlN; a new piezo electric material particularly suited to thin film device development as well as metrological applications) and Magnesium diboride (MgB_2; a high temperature conventional superconductor). These materials were used by the project in the development of high performance NEMS resonators. Novel microwave excitation and detection schemes and optical interferometric methods incorporating nanoscale metrology were also developed for the NEMS resonator excitation and read out. These NEMS resonators then fed into the metrology-based work in the project where the focus was on demonstrating traceable electromagnetic metrology and high precision measurements.

The aim of the MetNEMS project was to develop NEMS as high frequency resonators and actuators for future metrology, including single entity and traceable measurements, 'beyond classical limits'. From a metrological viewpoint the most relevant NEMS devices are high quality factor (Q) (that is low-loss) mechanical resonators since they combine the high accuracy of frequency measurement with the exquisite sensitivity of nanoscale sensors. The MetNEMS project had the following five specific objectives:

1. Optimised Novel Materials for High Performance NEMS:

To select and optimise materials such as AlN, silicon on insulator (SOI), low stress polysilicon, and new materials e.g. graphene for high performance NEMS.

The selection and optimisation of materials is critical to optimising performance with the resulting application potential of NEMS structures. The project will characterise, optimise and develop several materials including graphene and MgB_2.

2. Novel Methods for NEMS Resonator Excitation and Readout:

To develop novel methods for nano-electro-mechanical systems (NEMS) resonator excitation and readout to take account of the reducing size of NEMS devices, including metrology methods for validation of dynamic optical characterisation of samples. Developments for readouts should address piezo-resistive, capacitive and near-field microwave techniques.

The project will develop our innovative near-field microwave resonator excitation and readout technique. This can be used in parametric mode to enable *classical squeezing* of the measurement process. Stroboscopic scanning white light interferometry (SSWLI) is another promising method, to determine 4D (3D + time) characteristics of MEMS/NEMS devices. The main technological objective is to identify a method to determine the exact trajectory of motion of the moving element of a MEMS/NEMS oscillator.

3. NEMS for Traceable Electromagnetic Metrology and Precision Instrumentation:

To develop architectures for integration/readout of NEMS sensors, references and arrays.

Metrology methods that validate SSWLI measurements of NEMS/MEMS samples will be developed and incorporated into the SSWLI instrument, achieving traceable 4-dimensional measurements in activated NEMS systems. The near-field microwave interferometer method will be compared with stroboscopic optical interferometer for the first time, harnessing the power of metrology to determine the exact motion (trajectory with known uncertainty). An innovative absolute calibration technique for a photon number resolving detector will be applied, to measure the quantum efficiency of nano transition edge sensors.

4. NEMS sensors: Going beyond Classical Limits

To develop dynamic cooling methods to enable measurement 'beyond classical' thermal limits.

Dynamic cooling is a key innovation enabling measurement 'beyond classical' thermal and sensitivity limits. The project will develop a microwave based cooling technique using near-field methods and high Q NEMS resonators, demonstrating 'beyond classical measurement' for force and mass. For electrical metrology ‘beyond classical’ relates to the development of ultra-small scale stable on-chip voltage references, based on the pull-in effect and ultra-sensitive M/NEMS-based charge detectors, areas pioneered by project partner MIKES. At the nanoscale quantum Casimir forces must be considered when evaluating performance of these devices.

5. Fabrication and Optimisation of Cryogenic Superconducting Quantum Interference Devices (SQUID)-NEMS Combination close to Quantum Limits:

To fabricate and optimise cryogenic SQUID-NEMS combinations, enabling the operation of NEMS resonators at close to thermal equilibrium quantum limits (mK-temperatures).

To approach the thermal equilibrium quantum limit, operation of NEMS resonators at mK-temperature is needed. Readout techniques thus have to be compatible with this temperature range. Unlike other techniques SQUID-based readout is optimised in this temperature range and offers high flexibility and enables high sensitivity. The performance of the combined SQUID-NEMS systems will be optimised to approach quantum limits on measurement. Finally, SQUID-NEMS systems at mK will allow the investigation of temperature metrology at the nanoscale.

3 Research results

3.1 Objective 1. To select and optimise materials such as AlN, silicon on insulator (SOI), low stress polysilicon, and new materials e.g. graphene for high performance NEMS.

The selection and optimisation of materials is critical to optimising performance with the resulting application potential of NEMS structures. Therefore in this objective the project fabricated, characterised and optimised several materials which showed great promise for NEMS resonators and other nanoscale metrology, including graphene, AlN and MgB_2.

3.1.1 Graphene

A new material which has been thoroughly investigated in the course of this project is graphene, the single atomic layer self-supporting carbon structure. The project has demonstrated the extremely desirable properties of graphene for NEMS resonators, i.e. high strength, low mass density and low mechanical losses. Using the CVD growth processes, transfer and annealing processes developed at one of the project’s Researcher Excellence Grants (REG(IC)) the project was able to transfer high quality graphene onto suspension-support structures fabricated from silicon (Si) materials in particular silicon nitride (Si_3N_4) and silicon dioxide (SiO_2) on Si (Fig. 1). A procedure for this, was written by NPL and (REG(IC)), describing in detail the transfer process for removing the copper (Cu) backing from the CVD grown graphene, following spinning on Poly(methyl methacrylate (PMMA) and subsequent flotation onto any desired substrate (Fig. 2).

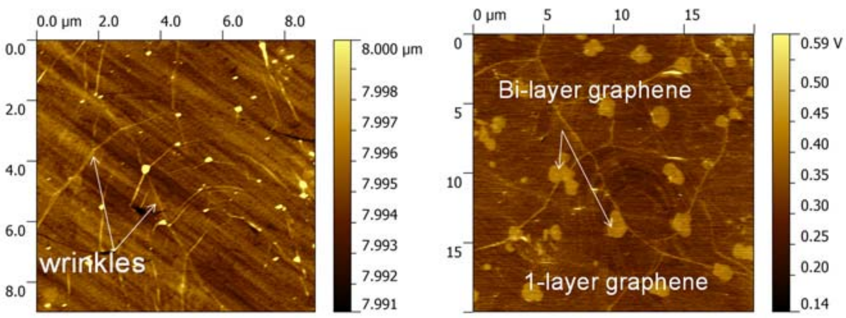


Fig 1. a) AFM (Atomic Force Microscopy) and b) SKPM (Scanning Kevin Probe Microscopy) images of CVD-grown graphene, showing topography changes and layer number changes respectively.

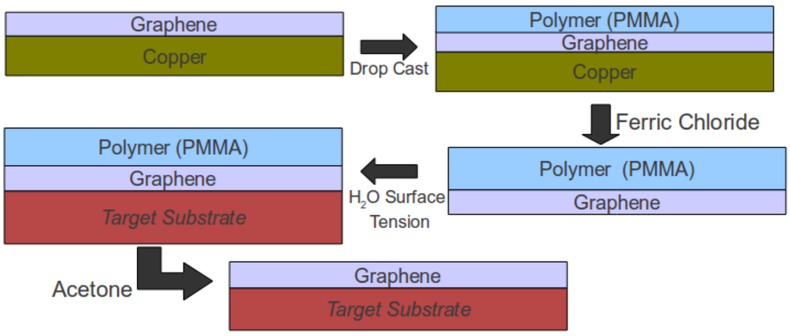


Fig 2. Wet transfer process for transferring the graphene target substrate.

In parallel with these experiments on graphene for NEMS resonators, NPL and (REG(IC) also pursued the development of a novel microwave based method for examining the electrical properties (and hence overall quality) of graphene films using a microwave dielectric resonator perturbation method (Fig. 3). Not only did this novel microwave based method prove useful in assessing CVD samples grown for use as NEMS resonators but it quickly became clear that this technique, being fast, non-invasive and non-contacting, had much wider applications such as with the electrical properties of the liquid exfoliation of graphene materials, sensor applications, CVD graphene quality control in a production facility etc.

A senior scientist, Prof. Yifang Chen from one of the project's collaborators Fudan University, Shanghai visited NPL twice during the project and gave talks on the state of the art fabrication facilities and research at Fudan University. NPL used Fudan University's expertise in electron beam lithography to help enable some of the more challenging NEMS fabrication processes during the MetNEMS project.

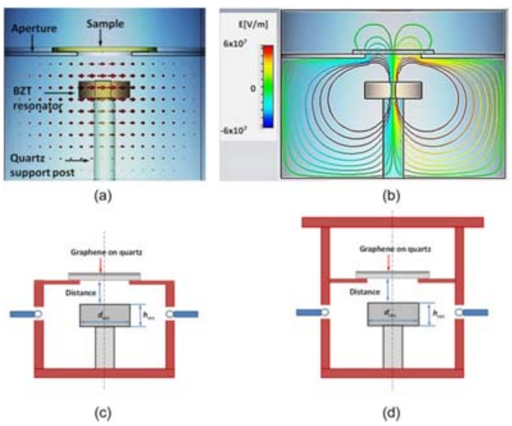


Fig. 3 Schematics of a) and b) electric field distribution in sapphire puck and surrounding housing, c) open resonator structure and d) closed housing structure (details see our published paper no. 21).

3.1.2 Piezoelectric Materials

The project also carried out work on a promising piezoelectric material, AlN. Several MEMS/NEMS resonators made from AlN on Si (grown and fabricated as part of a development project by US based foundry MEMSCAP were investigated by project partner NPL. Although the initial room temperature performance of these AlN on Si MEMS/NEMS resonators showed promise the piezo material was found to be unstable over relatively short periods therefore, it was decided by the project that graphene showed much greater promise and potential for mechanical resonators for real-world applications and metrology.

However, NPL made extensive tests on two further AlN on Si chips containing double clamped beams and drums using an automated probe station, in order to make current voltage characteristic measurements. But these measurements only confirmed the excessive leakage in the AlN on Si chips devices, which increased over time. Instead a Lead Zirconate Titanate (PZT) film on Si membrane was acquired by the project and tested using the microwave near-field device from section 3.2.1, at frequencies up to 600 kHz and an inverse piezo effect was demonstrated.

Substantial work on non-linear excitation of piezo MEMS/NEMS resonators was also undertaken by project partner NPL. This led to the production of a simple non-linear equation (the Duffing equation) to be fitted to this data and hence permitted accurate characterisation of the peizo MEMS/NEMS resonators.

3.1.3 New Superconductors and Processes

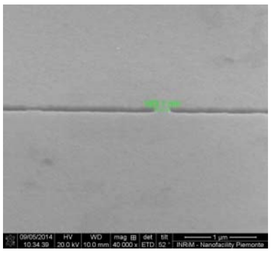
Project partner INRIM focussed its efforts in this objective on the fabrication of a first prototype cryogenic resonator based on superconducting magnesium diboride (MgB_2) nanowires (Fig. 4). The advantage of using a superconducting nanoresonator is that the applied excitation force is proportional to the r.f. current amplitude which can be maximised in a superconductor. In addition, the desirability of using MgB_2 resides in its rather high critical temperature (~40 K) compared to traditional superconductors that enables researchers to work with cheaper and more compact cryocoolers.

INRIM fabricated (Transition- Edge Sensors) (TES) devices based on Titanium (Ti) film on double side polished Si wafers covered on both sides by 500 nm thick Silicon Nitride (SiN) layer grown by low pressure chemical vapour deposition (LPCVD). Before deposition, the base pressure in the evaporation chamber was lower than $4x10^{-8}$ mbar for Ti films grown in UHV. The substrate temperature T_s, monitored by a thermocouple, was varied between 100°C and 250°C. The Ti films were prepared from a pure 99.99% bulk Ti (≥99.99%). The distance between crucible and substrate was 10 cm and the deposition rate was about 1.5 nm/sec. The fabrication process consisted of three steps; firstly a few nm of Ti were deposited on a SiN substrate in order to improve the adhesion of the gold layer (second step). The third/last step was the deposition of the superconducting Ti film. The TES devices were fabricated by e-beam lithography process and removal of the unwanted material was done via lift-off techniques. The superconducting wiring made from Al was defined by a lift-off technique combined with rf-sputtering of the superconducting films.

At PTB, a new Niobium thin film process was developed to enhance the quality of SQUID current sensors. The SQUID current sensors were processed on 3 inch substrates in a 10 layer process. The Niobium films were used for Nb/AlOx/Nb trilayers for forming Josephson Junctions and additional superconducting wiring layer. The Niobium films were dc-magnetron sputtered on oxidised silicon wafers in a high-vacuum sputter system with a base pressure of about 2 E^{-5} Pa. The Niobium target had a diameter of 153 mm and the films were deposited in Argon plasma of 0.5 Pa by a constant power of 40 W: this allowed the deposition rate to be decreased by a factor of 5.3 to about 1.2 nm/s. The Niobium thin films were patterned by reactive-ion etching with Argon and SF6 in a plasma etching system and the resist masks were subsequently exposed to UV-lithography.

Further to this, project partner PTB has developed and fabricated different types of novel nanoSQUID gradiometers and current sensors (SCS) with nanoscale Hafmium Titanium (HfTi) superconductor-normal conductor-superconductor (SNS) junctions.

The experience acquired in developing the novel materials above was subsequently used by the project to design and fabricate NEMS resonators, photon detectors and SQUID readout structures. Throughout the project a wide range of mechanical resonant structures were constructed including single clamped cantilevers, double-clamped beams and drum-head resonators. The linear dimensions of these NEMS structures ranged from 100 nm to 500 μm and the resonant frequencies ranged from a few kHz to hundreds of MHz.



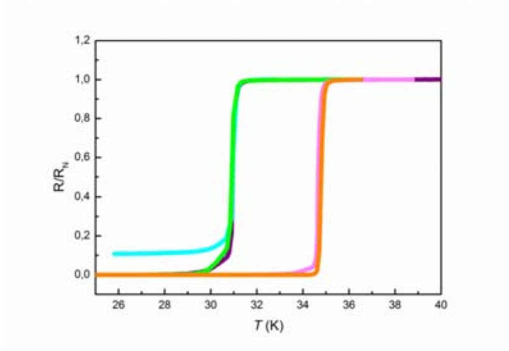


Fig. 4 a) SEM image of MgB_2 nanobridge nanostructured by focused ion beam, b) resistance vs temperature plots for two MgB_2 samples (A: 50 nm thick, Tc ~ 31 K, sample B: 55 nm thick, Tc ~34.5 K)

Conclusions

In summary the project met Objective 1. to select and optimise materials such as AlN, silicon on insulator (SOI), low stress polysilicon, and new materials e.g. graphene for high performance NEMS by:

- Producing a procedure describing the transfer process for removing the copper backing from grown CVD graphene.
- Developing a non-invasive, non-contacting and fast microwave method for measuring the graphene electrical properties.
- Using a microwave near-field system to measuring PZT film on Si membrane at frequencies up to 600 kHz.
- Obtaining non-linear behaviour and fitting by the Duffing equation for peizo MEMS/NEMS resonators.
- Developing and fabricating different types of novel nanoSQUID gradiometers and current sensors (SCS) with nanoscale HfTi SNS junctions.
- Fabrication of a first prototype cryogenic resonator based on superconducting MgB_2 nanowires.

3.2 Objective 2. To develop novel methods for nano-electro-mechanical systems (NEMS) resonator excitation and readout.

This objective focussed on development of novel but quite distinct methods for NEMS resonator excitation and readout, in order to take account of the reducing size of NEMS devices. The work also included metrology methods for the validation of dynamic optical characterisation of samples and developments for readout methods addressed piezo-resistive, capacitive and near-field microwave techniques.

One aspect of this objective focussed on the development of an innovative near-field microwave resonator excitation and readout technique at project partner NPL. The second aspect focussed on an optical dynamic method i.e. stroboscopic scanning white light interferometry (SSWLI), which project partners, MIKES and UH developed, in order. The main technological objective for both aspects/methods was to determine 4D (3D + time) characteristics of MEMS/NEMS devices and hence determine the exact trajectory of motion of the moving element of a MEMS/NEMS oscillator and these two methods were compared in their capabilities for this.

3.2.1 Near-field microwave resonator excitation and readout technique for NEMS

NPL modelled, designed and constructed two complete near-field microwave resonator systems for use in the MetNEMS project. These near-field probe systems consisted of a high Q miniaturised quarter wavelength dielectrically loaded coaxial resonator with the open end of the centre conductor sharpened into a protruding tip. A microwave signal was fed to the quarter wave resonator from a tuneable high stability microwave source, via a circulator. The NEMS device which the microwave system interrogates was mounted on a low permittivity polystyrene-based structure and was attached by magnetic strips combining rigid fixture in order to provide ease of removal. The support structure was mounted on a three axis piezo scanner system so that the NEMS could be moved adjacent to the microwave tip and then scanned across the tip using scanners on the orthogonal axis. Optical microscopes allowed careful alignment of the near-field microwave resonator system and the entire system can be operated in air or in vacuum, or in any other mixture of gases.

When testing the near-field microwave resonator system the project demonstrated that the resonance properties which determine the microwave signal amplitude reflected from the quarter wave resonator via the circulator were very sensitive to the position of a conducting cantilever relative to the sharpened tip, with nanometre sensitivity.

It was important to show that the response of the near-field microwave system to motion of a NEMS resonator could be accurately modelled. Therefore the project developed a ‘quasi-lumped circuit’ analogue of the real system, incorporating finite element modelling of the electromagnetic fields around the tip, together with inductor, capacitor and resistor elements which modelled the remainder of the quarter wave resonator.

An initial aspect of this objective related to accurate measurement of the way in which the reflected signal amplitude varied with distance between mechanical resonator and sharpened microwave tip. NPL was able to do this and showed that the functional dependence could be accurately modelled in terms of the capacitance between the tip and resonator based on a calculation using analytic results for capacitance between a hemisphere and an infinite conducting plane.

At the start of the MetNEMS project readout using a microwave near-field technique had been previously demonstrated at NPL but it was only during this project that it was discovered that this same near-field system could also be used to excite movement of a NEMS system, through application of modulated radiation pressure arising from amplitude modulation of the microwave power applied. This achievement represented a major outcome and has the potential to be used in parametric mode to enable classical squeezing of the measurement process.

Using two phase locked r.f. generators NPL has also been able to produce a heterodyne detection system based on near-field microwave excitation and readout which is capable of detecting extremely small displacements of a mechanical resonator. A paper on this has been published which describes the predicted sensitivity of the method and this has attracted considerable interest within the measurement and sensor community. Furthermore a development of this technique, using modulated microwave excitation, has been demonstrated using two phase locked digital r.f synthesisers which may be applied to an external I-Q mixer to provide single sideband amplitude modulation up to 80MHz, adequate for all of our present mechanical resonator designs.

3.2.2 Dynamic optical technique (SSWLI) for MEMS/NEMS

Development of dynamic optical techniques for MEMS and NEMS metrology was another aspect of this objective and SSWLI was the promising method which MIKES and UH developed. UH modified their scanning white light interferometer (SWLI) for used with high and low frequency dynamic SSWLI measurements. The modified SSWLI was able to perform measurements of periodically moving samples at frequencies up to a few MHz and had LED light source and custom built electronics that allow short bright light pulses.

Further to this, MIKES and UH created calibration methods and transfer standards for the calibration of the SSWLI set-up. The developed calibration methods allowed characterisation of dynamic measurements of SSWLI which had not been achieved prior to the MetNEMS project. The characterisation methods also had smaller uncertainty levels than the repeatability of SSWLI height measurements at tested frequencies and amplitudes.

UH and MIKES characterised the SSWLI setup of UH for low and high frequency dynamic measurements and following this methods were developed to calibrate dynamic SSWLI measurements of vertical displacements up to kHz frequencies. This was the first time dynamic SSWLI measurements were characterised and led to the publication of 2 journal articles and several conference publications. The calibration methodology achieved 2-10 nm standard uncertainty for dynamic measurements, which is comparable to the repeatability of the SSWLI setup for static measurements. The technologies used by MIKES and UH could also be used to span frequencies from sub-kHz up to MHz range and for quasidynamic measurements a level of 1 nm / µm was achieved.

The characterised SSWLI setup featured a small negative amplification error in the quasi-dynamic measurements, which was an order of magnitude less than the accuracy achievable for static measurements using typical commercially available step height standards or accuracy promised by manufacturers of commercial SWLI setups. In addition, the dynamic characterisation measurements of the heights measured by SSWLI deviated from the SI-metre with a smaller expanded uncertainty of the characterisation than when the setup was used with short light duty cycle <0.5%.

Further to this UH modified their SSWLI setup for measuring *hidden surfaces* in a moving sample. The modified SSWLI setup featured an IR camera and a light source consisting of three LEDS with wavelengths 970 nm, 1020 nm and to 1050 nm. The LEDs provided stroboscopic measurement capability up to few MHz and a coherence length of <5 µm was possible with low interferogram side peaks.

UH in collaboration with MIKES have also tested their SSWLI setup for hidden surface measurements. The measured artefact consisted of a piezo transducer and silicon microchannel structure that is moved vertically by the piezo at sub kHz frequencies and amplitudes up to few µm (Fig. 5b). Measurement results were not affected by the moving sample and the measured channel depth and layer thickness were similar in all phases of movement. The measured thickness and depth was also similar to earlier measurements using Static SWLI and SEM.

3.2.3 Comparison of the near field microwave device and SSWLI setup

As discussed in section 3.1.2, methods that validate SSWLI measurements of NEMS/MEMS samples were developed, demonstrated and incorporated into the SSWLI setup, in order to achieve traceable 4-dimensional measurements in activated NEMS systems. Following this the near-field microwave interferometer method (see section 3.1.1.) was compared with the SSWLI setup in terms of its ability to determine the exact motion (trajectory with known uncertainty).

MIKES defined a protocol for the comparison of movement measurement of cantilever structures in collaboration with UH and NPL. Comparison measurements by UH on the SSWLI setup, NPL on the near field microwave device and MIKES using a laser interferometer were performed according to the protocol and the results analysed. By using MIKES's laser interferometer this allowed the comparison of the two novel methods (i.e. the near field microwave device and the SSWLI setup) to each other and to a mature traceable method for displacement measurement.

The comparison was made by measuring the movement of a quartz tuning fork (Fig.5a) oscillating at out of plane antiphase oscillation mode with a frequency of 10.7 kHz and peak to peak amplitudes from 300 nm to 900 nm. The comparison showed that the measurement results of the near field microwave device, SSWLI setup and laser interferometer were similar and within the uncertainty limits of the comparison (Fig.5c). The agreement of the measurement results within 10 % by both the near field microwave device and the SSWLI setup was obtained. Analysis of the results revealed which parts of the near field microwave device and SSWLI setup could potentially be improved. For SSWLI measurement repeatability was a dominant source of uncertainty with a standard error of ~3nm, whereas the motion of calibration scan was largest uncertainty contribution for the near field microwave device. Based on the results of the comparison, MIKES, UH and NPL have written a protocol for the comparison of movement measurement of cantilever NEMS structures.

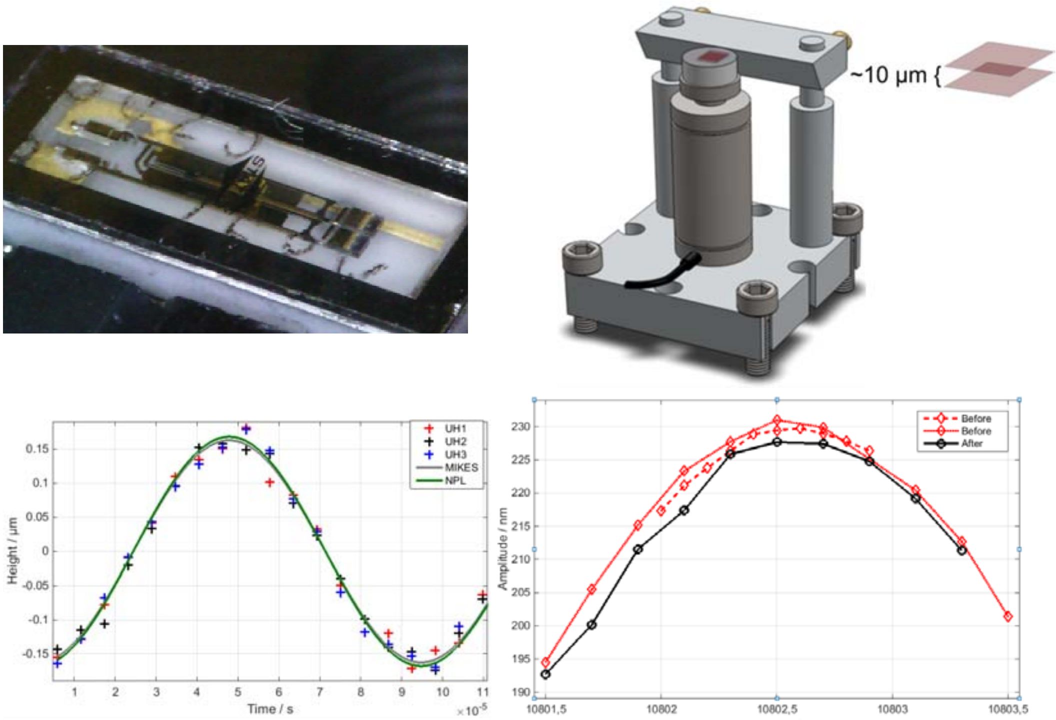


Fig 1. a) Image of a tuning fork artefact. b) System developed for testing novel SSWLIM method, allowing two moving surfaces to be measured simultaneously. c) Measurements at MIKES, UH and NPL, results show similar movement amplitude. d) Cantilever stayed unchanged during travel to NPL and back. Preliminary results from NPL showed measured amplitudes within 10 % from MIKES and UH.

Conclusions

In summary the project met Objective 2. to develop novel methods for nano-electro-mechanical systems (NEMS) resonator excitation and readout by:

- Developing a heterodyne detection system based on near-field microwave excitation and readout is capable of detecting extremely small displacements of a mechanical resonator (~50fm/Hz1/2).
- Publishing a paper on the predicted sensitivity of the method and this has attracted considerable interest within the measurement and sensor community
- Establishing the first steps towards traceable dynamic SSWLI measurements: methods for Characterisation of vertical axis of a SSWLI setup for Quasi-dynamic and dynamic measurement
- Demonstrating the dynamic measurement of hidden structures using SSWLI adapted for IR light.
- A comparison between the SSWLI setup and the near-field microwave device that showed that: vertical movement of the tuning fork at 10.7 kHz could be reliably measured using both methods
- A comparison between the SSWLI setup and the near-field microwave device that showed tha the Tuning fork is stable enough to use as transfer standard for comparison of measurements at different laboratories

3.3 Objective 3. To develop architectures for integration/readout of NEMS sensors, references and arrays.

3.3.1 Graphene array resonators

One of the main projected applications of NEMS resonators is as ultra-sensitive mass detectors with predicted sensitivities reaching to the attogram level even for room temperature operation. However, such performance levels requires both ultra-low mass resonators with high Q values and ultra-sensitive read-out mechanisms. Therefore NPL and REG(IC) concentrated on making use of single layer graphene mechanical drum resonator arrays (based on the work in section 3.1.1) coupled to near-field microwave readout methods (from section 3.2.1).

Five coupled resonator systems were built and demonstrated by NPL and REG(IC). Following the acquisition of Si on SiO_2 substrates containing a range of different structures the project was able to transfer graphene films to these structures. The graphene membranes did not show sagging around the edges of the support structure, as was observed for graphene on Si_3N_4 membranes so this was a promising result and may indicate more rigid anchorage, with the expectation of higher mechanical Q values.

The project also investigated two forms of graphene mechanical resonators based on the CVD graphene transfer process developed by REG(IC) (section 3.1.1). The first substrate was a thin (~100nm) Si_3N_4 membrane which was predrilled with an array of circular holes (0.25 to 3µm diameter). The second substrate consisted of a 300nm SiO_2 layer grown on a Si substrate. A large array of circular holes, square holes and linear trenches were patterned in the SiO2 layer by photolithography by the project's collaborator Wroclaw Technical University, Poland, leaving the underlying substrate untouched. Subsequently CVD graphene was then transferred onto these substrates/chips. For optimum mechanical resonator performance the PMMA/graphene stack was ironed out via heating the PMMA as the graphene repeats the topography of the Cu it was grown on, as this softens and expands PMMA allowing it to relax flat upon the substrate, and minimising any wrinkling of the graphene which can cause mechanical and electrical deficiencies. This heating process also serves as an annealing process in which the PMMA is removed via hydrogenolysis and depolymerisation. The conditions for annealing were 350ºC in a furnace for 10 hours at ≈ 2mbar with a hydrogen flow rate of 100 sccm (Standard Cubic Centimeters per Minute). Argon was emitted from the process as it caused a dilution of hydrogen, leading to a direct increase in polymer debris left on the surface of graphene due to a lack of hydrogenolysis of the PMMA and also polymer debris in the holes via a capillary effect from insufficiently diluted PMMA vapour.

Raman measurements showed that the graphene was almost entirely a single layer with low PMMA contamination and the project successfully demonstrated that circular holes up to 40 microns in diameter were covered with graphene, which is believed to be the largest single layer drum resonator prepared anywhere in the world (see also section 3.4.1). Furthermore the project made measurements on these graphene NEMS resonators, using piezo excitation and microwave readout (Fig 6). The graphene covered substrate was mounted on a piezo disc which was in turn mounted on the Attocube 3-axis positioner. The substrate was not transparent so x-y alignment was more difficult. A second microscope was mounted to view the tip and sample transversely so that separate views of the x-y and x-z planes were available. With the tip positioned close to the 40 µm circular drums the first mechanical resonances from this system at 29.6 kHz were observed with a relatively high Q (in air) of 120 and 600 in vacuum. Non-linear Duffing excitation was also observed at high drive levels.

The areal density of single layer graphene is less than 1 mg/m^2. This means that the mass of a 10 µm diameter graphene drum resonator is around 50 femtogrammes. As the project had already observed resonant frequencies of such graphene drum resonstaors at frequencies up to 60 kHz and Q values of 500 at room temperature, it was possible to divide the Lorentzian lineshape of a resonance to at least 1 part in 10^3, i.e. for a Q value of 500 this implied that the fractional frequency change of 2 parts in 1 million should be readily attainable. As the frequency shift of the mechanical resonator is inversely proportional to the added mass this suggests that a performance level of 0.1 attogramme sensitivity should be achievable for the project's single layer graphene mechanical drum resonator.

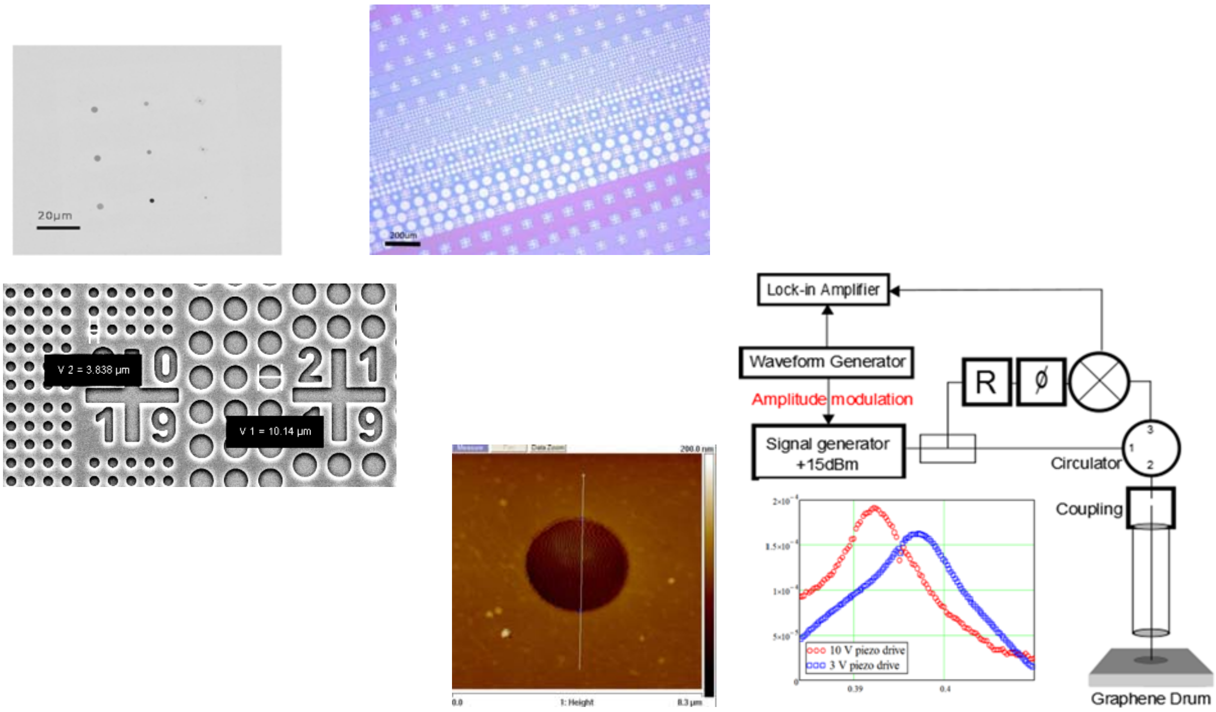


Fig. 6 a) SEM image of array of holes covered by graphene, b) Optical image of the fabricated substrate showing the array of features for graphene suspension c) SEM image of the same d) enlarged SEM image of single graphene drum resonator and e) schematic of microwave resonator-based excitation and read-out developed at NPL

3.3.2 Voltage references and voltage detector based MEMS/NEMS

MIKES worked on a MEMS/NEMS based stable voltage reference and a MEMS/NEMS-based ultra-sensitive voltage null detector/amplifier that could also be modified for charge detection. Finalisation of the designs for both was done partly in parallel with their actual construction.

The new MEMS/NEMS based stable voltage reference was produced by MIKES with the project's collaborator Aivon Oy. The stand-alone MEMS-based AC voltage reference with automatic tracking of the pull-in voltage was an improved version of an earlier MEMS-based AC voltage reference. The main improvements being that the MEMS component and most of the electronics were assembled into a hermetically sealed enclosure with active temperature control in order to remove the effects of varying ambient pressure and temperature, and that self-adjusting drive electronics were developed to keep the amplitude of drive current automatically at its correct pull-in value.

The stand-alone MEMS-based AC voltage reference with automatic tracking of the pull-in voltage was tested at 3.7 V voltage level and 100 kHz frequency. MIKES managed to solve most of the earlier drift problems for the voltage reference, however the stability of the measured output voltage was no better than ±5 ppm during two-weeks (see Fig 7), probably due to problems in the measurement instrumentation.

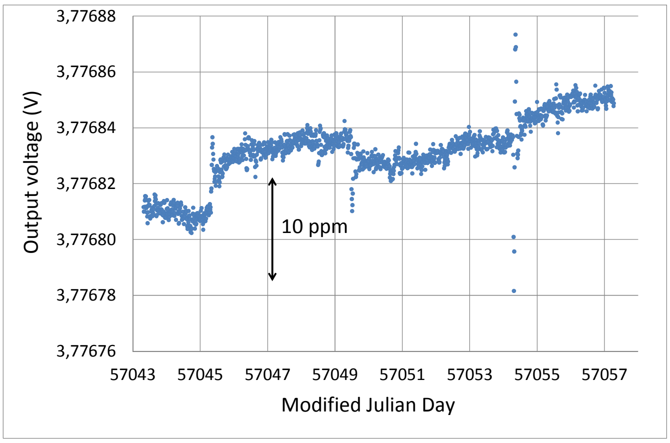


Fig. 7 Stability of MEMS based voltage reference over a period of 14 days.

The new MEMS/NEMS-based ultra-sensitive voltage null detector/amplifier was based on a novel solution proposed by MIKES. In this solution, an rf bridge-readout was used to monitor the MEMS capacitance and to provide feedback to maintain the device at the pull-in point in a microelectromechanical system. This then provided a large mechanical gain at low frequencies and thus eliminated the noise contribution of the readout electronics.

The MEMS/NEMS-based ultra-sensitive voltage null detector/amplifier was demonstrated by MIKES using a non-optimised MEMS device. The voltage noise had a clear minimum at the pull-in point where the relative deviation of the moving capacitor plate from the equilibrium position is $X/D_0 = 1/3$. Experimental results were in reasonably good agreement with theoretical predictions of the simple harmonic oscillator model and demonstrated that the noise level at pull-in could be decreased down to the estimated level of the thermal noise of the MEMS component. However, much better performance is expected with an optimised component at low temperatures.

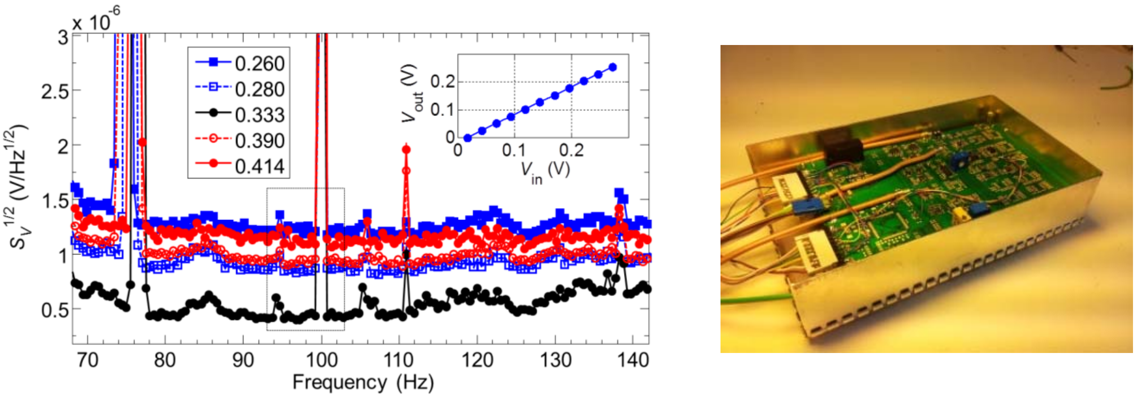


Fig. 8 Frequency dependent voltage noise spectral density from prototype MEMS based voltage detector.

A report on the performance of the MEMS/NEMS-based prototypes was delivered to project partners at the beginning of March, 2015. As the final activity, MIKES designed and constructed a prototype DC voltage reference based on actuating a moving-plate MEMS capacitor with a square-wave signal which kept the MEMS capacitor at pull-in point by controlling the drive amplitude (Fig.8). Proof-of-principle experiments demonstrated operation of the device, but the drift of output voltage was too large for practical applications.

3.3.3 Nano-TESs detector

INRIM, PTB and NPL worked together on developing traceable TES detectors for single photon detection. TES detectors are superconducting devices operated in the temperature region between the normal and the superconducting state with the capability to discriminate the number of photons in a light pulse. Nano-TES detectors (Fig. 9) were designed, fabricated and characterised by the project. The main advantage with these small nano-TES detectors was that their reduced heat capacity allowed them to work at higher temperatures (400 mK compared to 100-200 mK for TES detectors) which increased the count rate up to 1 MHz, with negligible dark counts. Moreover the application of an innovative absolute calibration technique for a photon number resolving detector allowed measurement of the quantum efficiency of nano transition edge sensors.

PTB and INRIM also worked together on the application of a dc-SQUID developed at PTB with superconducting TES detectors developed at INRIM, in order to improve the measurement capability of single photon detectors in terms of timing jitter reduction.

Further to this a cryogen free single photon detector system based on TES detectors was developed for cutting-edge measurements in new quantum technologies fields.

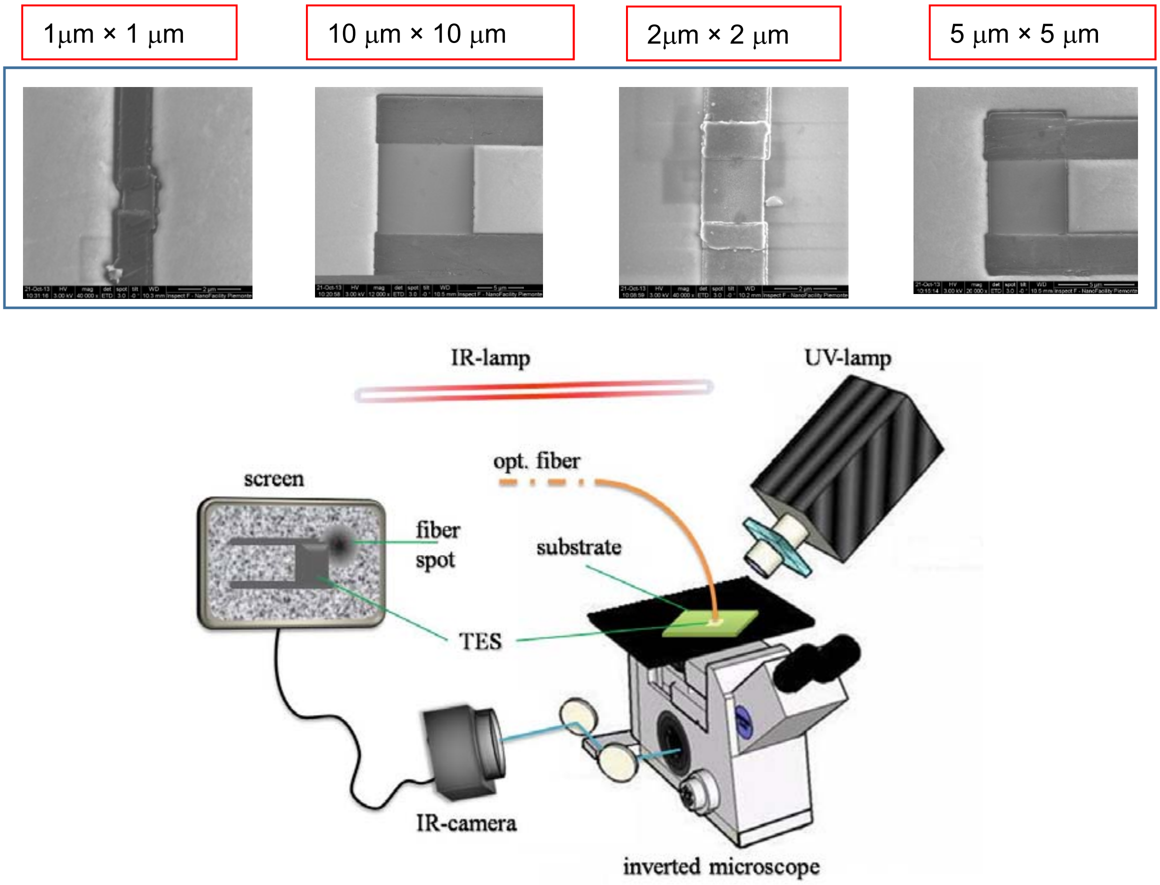


Fig. 9. SEM images of TES devices with active area ranging between 1 μm^2 and 100 μm^2. In figure (left side) is reported the together with schematic of alignment of optical fibre for coupling photon signals to the TES. the fiber is glued with UV curing on top of the detector checking the position in transmitted light with an inverted microscope chip.On the right side of the figure there is a sample holder with fiber glued on the detector.

3.3.4 NanoSQUID gradiometers and current sensors

For the readout of nanoSQUIDs, PTB developed SQUID current sensors which could be operated with a modified Magnicon SQUID electronics. PTB has also developed and fabricated different types of novel nanoSQUID gradiometers (Fig 10) and current sensors (SCS) with nanoscale HfTi SNS junctions. PTB's SQUID amplifiers for the read-out of nanoSQUIDs and superconducting photon detectors were produced with project partner Mag and can be operated with modified Magnicon SQUID electronics, with the aim of making them suitable for end-users and improving currently commercially available SQUID sensors. Mag is Magnicon Gmbh an engineering company and leading supplier of high performance SQUID electronics, low-noise SQUID sensors and customised SQUID systems and therefore has the necessary experience for this technology transfer and commercialisation of the project' results.

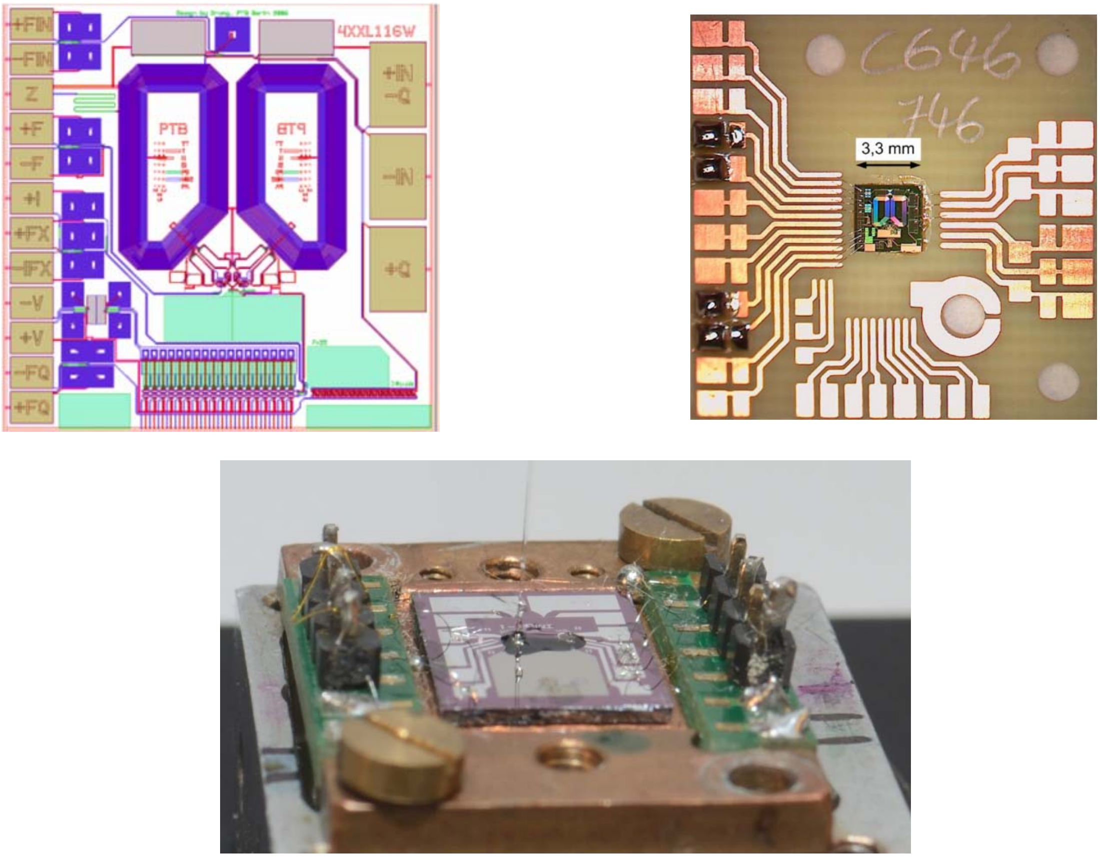


Fig. 10 Diagram of PTB gradiometer design (left) with image of device on chip (right) and chip installed in chip holder (below).

Conclusions

In summary the project met Objective 3. to develop architectures for integration/readout of NEMS sensors, references and arrays by:

- Fabricating several graphene array resonators were fabricated.
- Using the project's near-field microwave device to observe graphene resonant frequencies up to 60 kHz and Q values of 500 at room temperature using.
- Using the performance achieved, especially in Q value, to predict that ~0.1 attogramme sensitivity is possible for the project's single layer graphene mechanical drum resonator.
- Designing and evaluating two prototype devices:
  1. A MEMS/NEMS based stable voltage reference
  2. A MEMS/NEMS-based ultra-sensitive null detector/amplifier
- Designed and constructing a prototype DC voltage reference based on actuating a moving-plate MEMS capacitor with a square-wave signal.
- Publishing a paper on the MEMS-based null detector in Sensors and Actuators A.
- Designing, fabricating and characterising nano-TES detectors. An innovative absolute calibration technique for a photon number resolving detector allowed measurement of the quantum efficiency of nano transition edge sensors.
- Demonstrating single photon counting with a photon number resolution at a counting rate up to 1 MHz, with negligible dark counts

- Developing a cryogen free single photon detector system based on TES detectors for cutting-edge measurements in new quantum technologies fields
- Developing and fabricating different types of novel nanoSQUID gradiometers and current sensors (SCS) with nanoscale HfTi SNS junctions. For the readout of nanoSQUIDs, PTB developed SQUID current sensors which can be operated with modified Magnicon SQUID electronics, which will be made commercially available to end-users.

3.4 Objective 4. To develop dynamic cooling methods to enable measurement 'beyond classical' thermal limits.

3.4.1 Graphene NEMS Resonators

NPL, together with REG(IC), demonstrated effective transfer of single layer graphene grown by CVD on Cu onto a number of supporting substrates to Si_3N_4 membrane (section 3.3.1) and based on this successfully transferred CVD grown graphene to cover an array of micron size holes, pre-drilled by focussed ion beam (FIB), in a 200nm thick Si_3N_4 membrane. Measurements of piezo excited resonances at 440 kHz, 780 kHz and 1.04 MHz in this system were detected and analysed using the microwave near-field device from section 3.2.1. These resonances represent modes of one of the 3 μm diameter drums from which the project was able to deduce that there was little residual tension in the transferred film and the effective Young's modulus was around 150 GPa, lower than the best reported figure in current literature of 1 TPa. The Q values achieved with these mechanical modes were modest, in the range 70-100. This was initially believed to be due to remaining contamination of PMMA on the graphene, resulting from the transfer process from the original Cu substrate on which the graphene was grown, however there was also a suspicion that weak anchorage at the edge of the drums could have produced dissipation, with a resulting lowering of Q value.

Further work was carried out using improved CVD graphene, obtained from a stakeholder company Graphenea, which was shown to have much lower PMMA contamination. This improved CVD graphene material was transferred onto Si_3N_4 membranes drilled with FIB to give a range of hole sizes from 0.5 μm to 15 μm diameter. Resonances in the MHz range (up to 60 MHz) were observed using piezo excitation and microwave readout but, as with the earlier results, the Q values remained low (less than 100). Raman spectroscopy of the transferred material confirmed that it was almost entirely single layer graphene.

As a result of these measurements the project decided that the low Q value probably resulted from weak anchorage of the graphene layer to the perimeter region of the holes in the membrane so an alternative, thicker form of substrate was sought.

Arising from a meeting between NPL and a project collaborator Wroclaw Technical University, Poland a mutually beneficial collaboration was set up in which the Polish group provided NPL with a set of stiffer substrates consisting of SiO on Si which had been patterned an array of slots, square and circular depressions up to 300 nm deep. In return NPL and REG (IC) transferred graphene films on to substrates which were returned to Wroclaw Technical University for scanned probe measurements to be made. The Raman results on the graphene samples revealed that the CVD graphene was almost an entirely single layer with low PMMA contamination. In addition circular holes up to 40 μm in diameter were successfully covered with graphene, believed to be the largest single layer drum resonator prepared anywhere in the world at the time (see also section 3.3.1).

From these results, NPL demonstrated that the freely suspended graphene mechanical drum resonators (see section 3.3.1) showed non-linear behaviour as a function of increasing drive level, with the resonant frequency decreasing as a the drive amplitude increased. This information led to a better understanding of the nature of the importance of the graphene support regions around the resonator's perimeter.

3.4.2 Dynamic Cooling

Dynamic cooling is a key innovation enabling measurement 'beyond classical' thermal and sensitivity limits. NPL developed a microwave based cooling technique using near-field methods and high Q NEMS resonators, demonstrating 'beyond classical measurement' for force and mass.

During the project NPL focussed work on this objective on researching a novel excitation and readout technique based on near-field microwave resonance. Using this NPL was able to show that amplitude modulated microwave radiation emanating from a sharp tip at the open-end of a quarter wavelength dielectric resonator was capable of driving the oscillation of a mechanical resonator and that the same system was capable of reading out sub-picometre vibrations of the same.

NPL demonstrated that coupling between the near-field region of an open-ended microwave resonator and a conducting mechanical cantilever provided an effective means for both excitation and detection of the motion of the cantilever at the equilibrium thermal limit. Real-time measurement of the NEMS resonators were demonstrated i.e. without the need for any averaging (Fig. 11).

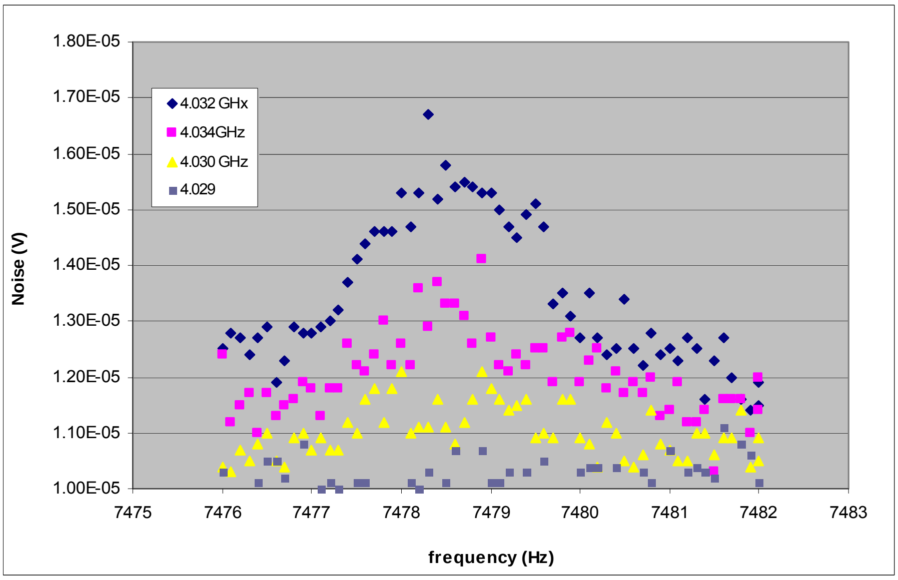


Fig.11 Thermal noise data from a Si_3N_4 cantilever resonance at 7.5 kHz, showing the reduction in noise amplitude (and thus of thermal noise in the oscillating mode) as the microwave pump frequency is tuned from 4.034 GHz to 4.029 GHz.

Conclusions

In summary the project met Objective 4. to develop dynamic cooling methods to enable measurement 'beyond classical' thermal limits by:

- Developing and demonstrating several different coupled microwave and mechanical resonator systems based on suspended graphene layers as well as suspended piezoelectric thin film resonators.
- Using AFM examination to show good coverage in general and that the graphene membranes did not show sagging around the edges of the support structure, as was observed for the graphene on Si_3N_4 membranes.
- Demonstrating NEMS development using suspended graphene layers and Q values of 600 at room temperature.
- Demonstrating passive mode cooling using detuned microwave excitation from 300 K to 60 K. The coupling constant between microwave and mechanical resonators was calculated and the demonstrated cooling was close to that predicted by sideband cooling theory. NPL has designed new tighter coupled structures which promise improved cooling in future.

3.5 *Objective 5. To fabricate and optimise cryogenic SQUID-NEMS combinations, enabling the operation of NEMS resonators at close to thermal equilibrium quantum limits (mK-temperatures).*

3.5.1 Novel SQUID Designs

During the project some of the very ambitious goals concerning SQUID designs and fabrication technology could not be achieved as expected (e.g. all Al technology). However other, novel (unforeseen) developments were used as an alternative.

For the readout of nanoSQUIDs, PTB developed SQUID current sensors which can be operated with modified Magnicon SQUID electronics (section 3.3.4). PTB has also developed and fabricated different types of nanoSQUID gradiometers and current sensors (SCS) with nanoscale HfTi SNS junctions (section 3.1.3). A novel technology based on EBL and chemical-mechanical polishing (CMP) was established at PTB to fabricate these nanoscaled devices with SNS junctions. However, in order to exploit this technology for NEMS readout complex nanoSQUIDs with gradiometric SQUID and feedback loops, gradiometric transformers and rf filters were designed and manufactured. A second wafer with an improved design including circuitry needed for the NEMS operation was also fabricated and the rate of yield of this second wafer was excellent. Due to the high potential of these devices with the second wafer, e.g. for metrological applications, PTB intends to continue with the development and fabrication of new much more complex sensors.

The loop dimension of the HfTi SNS junction containing nanoSQUIDs was about 1 µm x 1 µm and the lateral dimension of the junctions was about 200 nm × 200 nm, the barrier thickness was nominally 30 nm. This basic SQUID structure could also be used as a simple magnetometer. However, in order to make the HfTi SNS junction containing nanoSQUIDs suitable for a variety of applications, i.e. those which need a more complex design such as gradiometers, transformer coupled devices, more elaborated designs were developed and investigated. The project implemented and tested gradiometric nanoSQUID designs with integrated feedback loops, gradiometric transformers and auxiliary components as rf filters. The results of the tests represent a very important achievement for the project, as even with all the additional integrated components, a very low white flux noise level for the HfTi SNS junction containing nanoSQUID parallel gradiometers of about 115 $n\Phi_0/\sqrt{Hz}$ could be obtained. Due to the small junction size and the fully gradiometric design including the gradiometric transformers, the nonlinearity of these nanoSQUID devices was much lower than the nonlinearity of the devices fabricated in PTB’s standard Nb/AlOx/Nb process. The maximum operation field of the nanoSQUID devices was increased, too. Depending on the mode of operation and the type of transformers, magnetic fields of up to a few ten mT or a few hundred mT were applicable hence making the HfTi SNS junction containing nanoSQUID devices suitable for NEMS readout. In contrast to other groups which have reported similar high operation fields for simple SQUID designs, PTB’s HfTi SNS junction containing nanoSQUIDs were much more complex devices with the benefit of easier handling and increased robustness. Therefore, PTB’s nanoSQUIDs are more promising for future applications. Because of the high potential of these new HfTi SNS junction containing nanoSQUID devices PTB will continue developing this technology, with the aim of developing smaller junctions and more flexible device elements.

The high bandwidth measurements in a two-stage configuration, with a SQUID current sensor (SCS) used as cooled preamplifier, was investigated by the project. However the process was challenging as for each sensor type, the whole setup had to be optimised to make the bandwidth as large as possible, and in some cases, a return to a one-stage setup was needed in order to increase the bandwidth to the maximum, which gave the disadvantage of losing resolution in the amplitude. Each measurement also needed a modified setup and thus the high bandwidth measurements in a two-stage configuration, with a SQUID current sensor (SCS) used as cooled preamplifier were very time consuming.

NPL has also developed and fabricated Niobium (Nb) nanoSQUIDs using FIB. The improvements achieved by NPL with the Nb nanoSQUIDs include better matching of the critical currents of the two junctions of each SQUID, and improved yielding due to critical current modulation profiles as a function of the applied magnetic flux, which in turn led to better noise performance of the Nb nanoSQUID devices.

In addition to this, experiments were carried out at INRIM on a FIB-based structuring process for fabricating high quality MgB_2 nanowires. Following the FIB structuring at NPL the microbridge junctions imaged using scanning electron microscopy (SEM) showed excellent milling characteristics and thus the transition temperature of the MgB_2 nanowires was able to be measured at 35 K at NPL. INRIM also fabricated the first ever MgB_2 nanoSQUIDs using EBL for nanofabrication. The performance of these MgB_2 nanoSQUIDs devices was successfully measured at both PTB and NPL.

It is conceivable, that in the future, more complex devices can be developed based on the project's successfully established HfTi-junction technology, in order to overcome the current limitations in bandwidth or maximum operation field. Based on the project's experience with the HfTi SNS junction containing nanoSQUID gradiometers and current sensors (SCS) it is possible to imagine a really complex HfTi-sensor, which includes a sensitive frontend SQUID, a high SQUID gain and a high robustness in the magnetic excitation field. Such a potential sensor would also significantly increase practicability of SQUID current sensors.

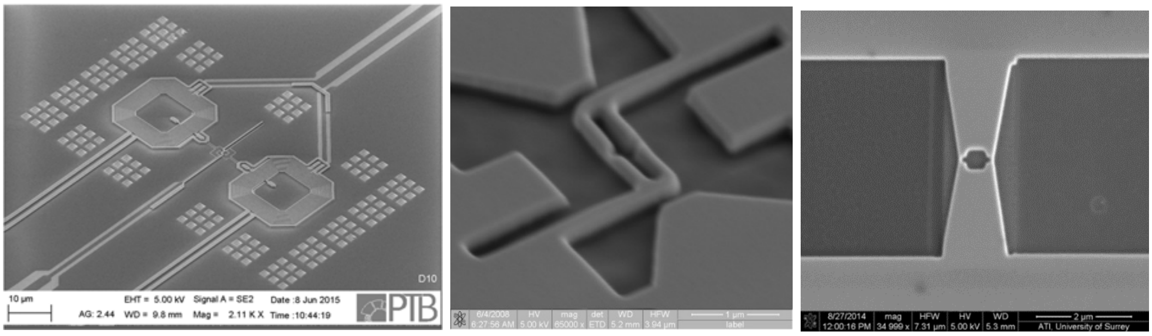


Fig. 12 SEM images of a variety of nanoSQUID based on HfTi SNS junctions (left), niobium microbridges (centre) and MgB_2 microbridges (right).

3.5.2 SQUID-NEMS Combinations

NEMS and SQUID combinations were jointly designed by PTB and NPL and then fabricated using the nano-manipulation techniques at NPL (Fig 13). At least three distinct designs for different applications were investigated. The first design was a paddle shaped NEMS resonator, fabricated from Gold (Au) coated Si. With this design resonances at frequencies up to 7MHz were observed at an operating temperature of around 7 K. The second design used a true nanoscale mechanical resonator made from an Au coated Si_3N_4 double clamped beam coupled to a slot-shaped Nb nanoSQUID. Both the first and second designs involved nanomanipulation to assemble the SQUID and NEMS resonator together. The third design was an integrated planar system fabricated around a Si_3N_4 window using an Nb SQUID and Nb coated mechanical resonator.

As part of PTB and NPL's SQUID-NEMS combinations, time consuming fabrication steps were necessary although it may be possible to eliminate these steps in future developments, such as adaption of the 4" stress-less NPL wafer to the PTB 3" process by using laser cutting. The production of freestanding nanoSQUID-NEMS combinations was also quite challenging and thus a fabrication process that did not damage the membranes was developed by the project. This technique included:

1. Sandwich preparation
2. Reduction of the thickness of the wafer
3. Chip separation

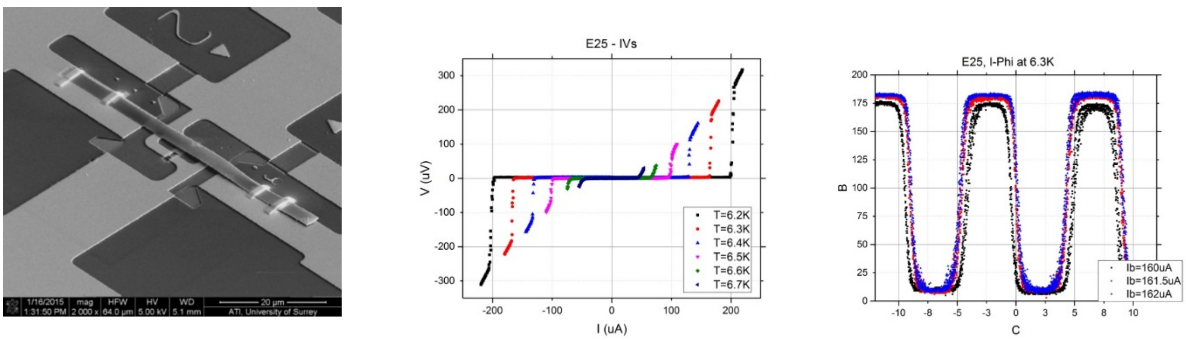


Fig. 13. SEM image of combined Si_3N_4 NEMS resonator with Nb slotted SQUID (left), I-V characteristics of the same device at varying temperatures for the same device (centre) and voltage-flux response (right).

The project developed measuring probe dip sticks for nanoSQUID operation and characterising with both a temperature range and a magnetic excitation field adapted to the sensors of project partners NPL and INRIM.

In order to measure SQUID sensor operation in an extended temperature range of 1.7 K to 60 K and in magnetic fields of up to 0.5 T a new probe dip stick for an evaporation cryostat at PTB was set up and implemented. The setup was used for example to characterise the MgB_2 nanoSQUIDs developed at INRIM (section 3.5.1).

A probe dip stick for operation in a Helium (He) storage can, was also designed for the characterisation of NEMS-nanoSQUID/combinations in the temperature range 4.2 K to at least 10 K and in magnetic fields of up to at least 100 mT, and was used for joint measurements by PTB and NPL. Together with readout electronics from partner Mag, this setup made the measurements at NPL easier and faster, and saves He compared with the existing measuring setup.

These two probe dip stick set-ups were used by the project to characterise more than 30 nanoSQUID sensors in a magnetic excitation field, mostly in a two-stage configuration with a SQUID current sensor used as a cooled preamplifier. The probe dip sticks are suitable for several types of SQUID sensors, for a one-stage as well as a two-stage operation, and will be used for both, sensor characterisation and operation (fig.14). Taking into account the experience gained by the project from these probe dip sticks, an improved version for SQUID sensor characterisation and operation was designed and is currently being manufactured at PTB

3.5.3. Millikelvin SQUID and NEMS Experiments

To approach the thermal equilibrium quantum limit, the operation of NEMS resonators at mK-temperatures is needed. Therefore, readout techniques must be developed that are compatible with this temperature range. Unlike other techniques SQUID-based readout is optimised in this temperature range and offers high flexibility and enables high sensitivity. Thus the project investigated whether the performance of the combined SQUID-NEMS systems (from section 3.5.2) could be optimised to approach quantum limits on measurement.

SQUID-NEMS systems at mK temperatures will also allow the investigation of temperature metrology at the nanoscale. Therefore, (REG)RHUL investigated the use of SQUID NEMS combinations for noise thermometry (fig. 14). However the results showed, that there are major problems regarding back action from the SQUID room temperature electronics to the NEMS and currently it is not clear whether this problem can be overcome with the currently available technology.

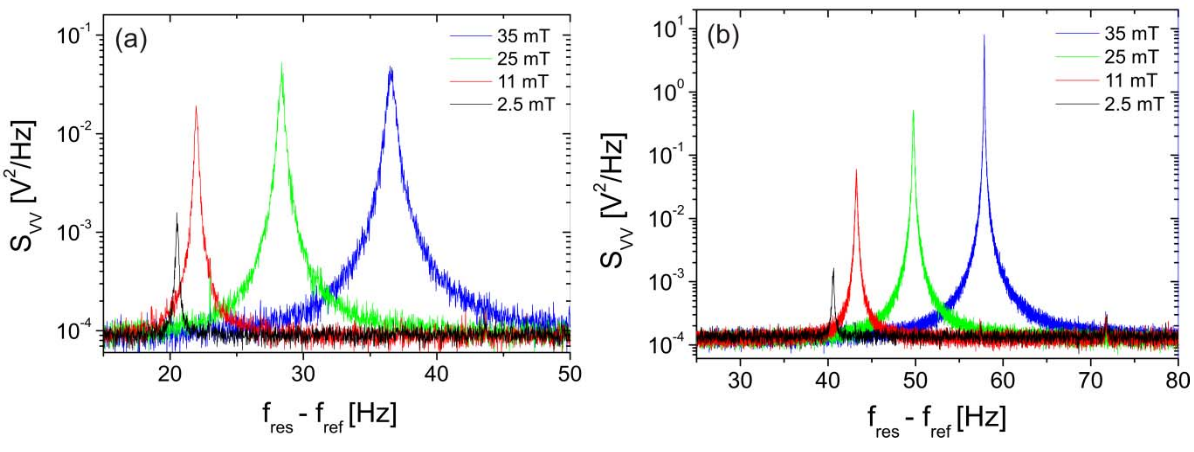


Fig. 14. Noise spectrum of the resonator at 100 mK with the SQUID locked on (a) the negative slope of the V-Φ curve (W) and (b) the positive slope.

Internationally, there is a serious demand on standardisation of cryogenic devices. The results of this project are subject to be implemented in the currently running standardisation activities of IEEE and ICE TC90.

Conclusions

In summary the project met Objective 5. to fabricate and optimise cryogenic SQUID-NEMS combinations, enabling the operation of NEMS resonators at close to thermal equilibrium quantum limits (mK-temperatures) by:

- Developing several novel HfTi-nanoSQUID gradiometers and current sensors which were intended to be used for investigation of nanosamples of magnetic material (e.g. magnetic nanoparticles) or NEMS readout.
- Establishing a new junction technology employing electron-beam lithography and a chemical mechanical polishing process for the fabrication of the HfTi-SQUID sensors. Since design optimisation, the yield is excellent.
- Fabricating the first MgB_2 nanoSQUIDs.
- Investigating high bandwidth readout. The results showed that with the currently available device technology such a high bandwidth operation in FLL mode cannot be achieved. However, this situation is subject to change in the near future, when novel more complex HfTi-SQUID current sensors are available and the devices developed in this project are an initial step towards this.
- Developing a new technology for producing freestanding nanoSQUID/NEMS combinations on membranes.
- Developing and building measuring probe dip sticks for nanoSQUID operation and characterising the nanoSQUIDs with both a temperature range and a magnetic excitation field adapted to the sensors of project partners NPL and INRIM.
- Using the probe sticks to characterise more than 30 nanoSQUID sensors in a magnetic excitation field, mostly in a two-stage configuration with a SQUID current sensor used as a cooled preamplifier.
- Investigating SQUID NEMS combinations for noise thermometry.

4 Actual and potential impact

To aims of the MetNEMS project, were to develop NEMS as high frequency resonators, sensors and actuators for future metrology, including single entity and traceable measurements, 'beyond classical limits'. Over its three year lifetime the project made very good scientific progress and achieved all of its objectives. A large number of papers and presentations have been produced during the project, totalling 35 papers and 92 presentations. Further to this, several international institutes have shown interest in the outcomes of this project and a number of small companies (e.g. Semimetrics, Magnicon and Entropy) have become involved in future research and development based on the outcomes of the project's objectives.

The outputs of the project provided greatly improved ultra-sensitive measurements and traceable metrology for mass, force and displacement, temperature, single photon & single molecule measurement at the nanoscale. On-chip ultra-stable miniaturised voltage references demonstrated. In addition a number of dramatic and disruptive methods have been developed which took metrology at the nanoscale into a regime beyond conventional classical limits and addressed the implications of quantum measurement on precise nanoscale metrology.

The MetNEMS project also supported the evolving sensor and metrology needs of a broad stakeholder community. At least four combined NEMS stakeholder and metrology end-user workshops were given by the project at NPL, PTB and INRIM and this allowed the project consortium to disseminate the project's outputs to the widest possible target user community. In addition to this, the project's large number of presentations at workshops and national and international conferences, as well as published papers in peer-reviewed journals have also enabled the project to disseminate its results beyond the metrological community.

In the short term, the MetNEMS project has led to the development of more precise measurements of physical parameters such as mass, force and displacement, through novel NEMS manipulation techniques. Dynamic cooling (also known as sideband cooling) using microwaves has been demonstrated and the project has shown how this will allow progress beyond classical thermal limits. Further to this, the inclusion of mechanical resonator control through ‘microwave gating’ of the NEMS resonator has been demonstrated by the project, a technique which will allow different physical parameter measurements to be optimised for near-simultaneous measurements of more than one parameter. Traceable electromagnetic methods were also developed by different project partners and have been compared and found to be compatible. In terms of cryogenics, novel nanoSQUID devices of five different types have been fabricated by the MetNEMS project and their performance assessed for NEMS readout suitability. The same superconducting electronic techniques have also been researched to assess their applicability for the development of absolute calibration technique based on parametric down conversion using nano transition edge sensors.

For longer term metrological applications the MetNEMS project expects the development of dynamic cooling of nanoscale mechanical resonators, further enhanced by thermodynamic cooling, to go beyond classical limits and to explore the quantum measurement regime. For example, the MetNEMS project demonstrated sideband cooling using near-field microwave probe coupled to a high Q NEMS resonator to confirm ‘beyond classical measurement’ capability for force and mass. The project’s extension to the cryogenic temperature range builds on recent developments of nano- and micro-scale SQUIDs; (the most sensitive measurement devices available for a vast range of physical parameters), which could be integrated to read out the displacement of mechanical NEMS systems, yielding higher sensitivities than are available at room temperature, and allowing access to the quantum ground state of mechanical resonators with frequencies in the MHz range. Techniques for developing the SQUID/NEMS coupled system to cool the mechanical system into its ground state and to control the coupling between them could also enable non-classical measurement protocols to be implemented, to explore the regime beyond the Heisenberg Uncertainty limit.

In the long term the greatly enhanced sensor capabilities emerging from the MetNEMS project will positively impact European society, via improvements in health care and health monitoring. Similarly the development and deployment of NEMS-based sensors will positively impact security issues, and could contribute to crime reduction and social stability. Better metrology at the nanoscale will also support industrial growth in high technology areas which will in turn support the European economy.

4.1 Impact in the Short Term

The project’s work on microwave resonator detection and excitation of mechanical resonators is an important outcome for the metrology of mechanical systems. The ability to scale down the excitation device towards the nanoscale and well below the scale of visible light wavelengths adds another tool to the nanometrologist’s kit for NEMS applications.

Microwave measurements on graphene were begun at NPL as a part of this project. One of the breakthroughs of the project was the realisation that the high 2D conductance of graphene means that it is well matched to the impedance of free space. This has confirmed that the use of graphene for NEMS resonators and provides evidence that its properties make it perfectly matched to a microwave excitation/interrogation approach.

One of the development highlights within the project was the joint development by NPL and REG(IC) of a microwave resonator method for measuring the electrical properties of graphene samples in a fast, non-invasive way. This led to a number of publications and more recently has led to a patent being filed by NPL for a novel microwave method for measuring the conductivity, mobility and carrier density of graphene and other thin semiconducting films. The commercial exploitation of these techniques are currently being explored with a number of companies and organisations.

The traceable characterisation of dynamic SSWLI measurements made by the project, has brought SSWLI to the same level of acceptance in industry as static SWLI measurements. This is important as the use of SSWLI in the characterisation of NEMS can make NDT (Non Destructive Test) of NEMS and MEMS easier especially in situations where rapid measurements of relatively large areas is important, e.g. measuring several samples on same chip simultaneously. The project’s work on the characterisation methods also led to developments by project partner MIKES on laser interferometer measurements, such that samples with higher oscillation frequency can now be measured and methods which cancel out periodic nonlinearity from laser interferometer measurements have been further developed.

The project’s comparison between measurement methods for SSWLI (at UH) and a near field microwave device developed by NPL, has increased confidence in using both for measuring nm to µm class displacements. The comparison also highlighted what could be improved in the measurement methods. Furthermore, the methods developed by the project for SSWLI and the near field microwave device could be used to characterise other instruments such as scanned probe microscopes, used for measuring samples with movement at similar frequencies and amplitudes.

Another significant finding for the project, was its demonstration that a self-oscillating NEMS resonator was capable of detecting an applied force of less than 1 pN. The system developed by the project for this, did not use expensive microwave digital equipment but merely used cheap microwave amplifier and filter. Thus this cheap and simple system could form the basis of an ultra-sensitive force sensor for applications in bioscience and medicine, for example being used to detect binding events between single pathogenic bio-molecules and functionalised surfaces on a NEMS resonator.

The most important output from project partner PTB was the development and fabrication of novel complex nanoSQUID gradiometers and current sensors (SCS) with nanoscale HfTi SNS junctions. The white flux noise of the nanoSQUID parallel gradiometers is the best result achieved at PTB and one of the best worldwide. The nonlinearity of the nanoSQUID gradiometers with gradiometric coupling transformers was also much lower than the nonlinearity of the devices fabricated in PTB's standard Nb/AlOx/Nb process, and the maximum field of operation of the new nanoSQUID devices was also increased. Depending on the mode of operation and the type of transformers, magnetic fields of up to a few ten mT or a few hundred mT were applicable and made the nanoSQUID devices suitable for NEMS readout. In contrast to other groups which have reported similar high fields of operation for simple SQUID designs, PTB's nanoSQUIDs devices were much more complex but with the benefit of easier handling and greater robustness. Therefore, they are much more promising in terms of future applications. Future developments of the nanoSQUID devices, by PTB will aim for smaller junctions and flexible device elements to meet more experimental requirements, (e.g. to overcome the current limitations in bandwidth) and to enable new user-oriented projects/science, (e.g. of material characterisation of small particles at low temperatures, or magnetic microscopy).

For the readout of nanoSQUIDs, PTB developed SQUID current sensors which can be operated with modified Magnicon SQUID electronics, from project partner MAG. The SQUID current sensors devices were tested in combination with several nanoSQUIDs and are now commercially available from MAG (Magnicon GmBH), as an attractive tool for nanoSQUID development and application.

In addition to this, project partner PTB developed expertise in the field of two-stage SQUID readout, in particular in the combination of a two-stage configuration combined with the bias reversal mode. This is a useful setup for investigating the noise contribution in SNS-junctions, and thus may be become a useful tool for the researchers developing nanoSQUIDs with SNS-junctions.

4.2 Longer Term Metrological Impact

NPL's demonstration of sideband cooling of a mechanical resonator using a detuned microwave resonator was a solid technical achievement or the MetNEMS project and can be expected to have a big impact on metrology in the coming years. Although the amount of sideband cooling produced from a room temperature starting point is at present only by a factor of five, the method may be improved to provide several orders of magnitude reduction in the effective noise temperature of a mode of the mechanical resonator. The requirement to achieve this greater cooling is that stronger coupling between microwave and mechanical resonators is needed and as such the research done in this project has provided clear routes for increasing coupling. One potential route for NPL to stronger coupling will focus on using graphene drum resonator with a conducting ground plane behind it, separated by a fraction of a micrometre. The graphene and ground plane will form a parallel plate capacitor whose capacitance is very sensitive to movement of the graphene. Since the parallel plate capacitor also forms a part of the microwave resonator, movement of the graphene drum will cause significant changes in the resonant frequency of the microwave system. Frequency changes can be readily measured with accurately therefore such as system will provide a highly sensitive and strongly coupled resonator system, ideal for measurements of force, mass and displacement at the nanoscale. Looking further ahead, such systems may also form the basis for true quantum metrology, based on manipulation of the ground and low order excited states of the mechanical resonator by means of complex microwave pulse sequences.

In order to extend this technology to the growing application area of quantum information processing it will be advantageous to operate the coupled resonator system at cryogenic temperatures. The work of REG(RHUL) in cooling a NEMS resonator to mK temperature range and observing the noise in the system has shown how this area can be opened up across a wide range of applications, from metrology at room temperature to entangled quantum systems for quantum metrology in cryogenic applications.

4.3 Dissemination Activities

At the start of the MetNEMS project some 25 stakeholders were identified as having an interest in the outcomes of the project, but by the end of the project this number had grown to 67, showing the relevance of the topics covered by the project and the successful promotion of the project's results.

At least seven separate inputs to standardisation bodies have been made during the project, including EURAMET TC-L Technical Committee for Length, the IEC TC90 committee on Superconductivity - WG 14 Superconducting electronic devices, CIPM CCEM Consultative Committee for Electricity and Magnetism and the EURAMET TC-EM Technical Committee for Electricity and Magnetism.

The increase in stakeholder interest was partly driven by the exposure of the project's results through the 92 presentations by project members at conferences over the duration of the project. These included a high proportion of oral presentations, many of them invited contributions at international conferences such as GrapChina 2014, Superconducting Quantum Devices workshop at Oxford University,2014 and KKNM, Poland 2014Poster presentations also attracted much attention, due to the novelty of the work and the quality of the results presented.

A more focussed form of dissemination arose from the project's end-user workshops which were organised at regular intervals throughout the project and included:

1. The first workshop entitled '*Physics and Metrology at Low Temperature*', was held 13 - 14 December 2012 at PTB (Berlin) and included presentations from project partners PTB, NPL and MAG. The audience of more than 50 people was predominantly from the wider scientific community (e.g. higher education, public research organisations and some SMEs).
2. A second workshop extending superconductivity applications towards small scale metrology entitled '*Superconductivity devices and applications*' on 22 November 2013 at NPL in Teddington (UK). 70 people attended and 5 companies took exhibition stands. Four of the project partners made presentations NPL, PTB, INRIM, REG(RHUL), along with 6 other oral presentations and more than 20 posters.
3. The third workshop was held at PTB (Berlin) and entitled '*Metrology with and for NEMS* on 20 May 2014. Project partners PTB, NPL, INRIM, MIKES, UH, REG(RHUL) and MAG presented their results at this meeting which took place during the last 15 months of the project. Predominantly the attendees came from the wider scientific community (e.g. higher education, public research organisations) but with a focus on both mechanical nanosystems and cryogenic NEMS.
4. The fourth workshop was held at INRIM (Turin) and entitled '*Novel Materials and Devices for NEMS*' on 26 February 2015 and included presentations from project partners PTB, NPL and MAG. The audience of more than 50 people was predominantly from the wider scientific community (e.g. higher education, public research organisations and some SMEs) with a focus on both nanosystems and cryogenic devices.
5. The fifth and final workshop was the dissemination meeting for the project's results. It was held at PTB (Berlin) on 18 June 2015, and entitled '*Metrology with and for NEMS and Superconducting Sensors'*. All project partners presented their work. In addition to academic and research institute attendees an increasing number of companies, particularly SMEs, were present in the audience which was of 90 people.

The overall success of the workshops can be seen by the steadily increasing size of the audience. A number of companies were encouraged to take exhibition space and sponsor these workshops for example through the sponsorship of prizes for the best posters etc.

4.4 Training within the Consortium

The high degree of collaboration between partners in the project has been reflected in the number of 'formal' training activities undertaken between partners with common interests in order to expand their capabilities or transfer knowledge. Below is a list of the formal training activities, however there were many more informal training activities and visits to other partners' premises for discussions:

1. Training in SDHLI measurements 3 January -31 March 2013 was held at MIKES for researchers from UH and REG(UH).
2. Two training activities in SSWLI by REG(UH) 2 April - 31 June 2013 and 1 September -30 November 2013 were held at MIKES for researchers from MIKES.
3. A training course entitled 'Readout of nanoSQUIDs' using a 2-stage configuration 24 - 27 May 2014 was held at PTB for researchers from PTB and NPL.
4. Further training in SSWLI by REG(UH) 7 April - 30 May 2014 was held at MIKES for researchers from ETLA, UH and REG(UH) and MIKES.
5. Training in the transfer of CVD grown grapheme onto a variety of surfaces 29 September 2014 was held by REG(IC) for researchers from IC and NPL.
6. A joint training meeting 'exploring best practice for MgB_2 Nano SQUID characterisation' 3 - 10 June 2015 was held at NPL for researchers from INRIM and NPL.

5 Website address and contact details

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6 List of publications

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