Multi-functional MEMS/NEMS for Nanometrology Applications

Ling Hao, John Gallop, Mark Stewart, Kevin Lees and Jie Chen, *Member, IEEE*

***Abstract*—As micro- and nano-electromechanical systems (M/NEMS) enter more widely into sensing applications and precision nanometrology it is timely to consider the advantages to be gained from a excitation and readout system which may be tailored for a wide range of applications. In this paper we describe a system that we are developing which allows a combination of excitation and readout methods which impact on many potential applications.**

I. Introduction

Electro-mechanical resonators at the micro- or nano-scale (so called MEMS or NEMS resonators) are expected to have ever wider application in sensing, detection and accurate measurement as science and technology move towards ever smaller length scales [1-8]. In this paper we describe a flexible system which we have developed to investigate a range of methods to excite and read out different types of mechanical resonators, made of a range of materials, sizes and resonant frequencies. Our system is capable of characterising materials and providing an approach to traceable metrology at the nanoscale. We show that no one method is optimal and frequently a combination of methods provides higher sensitivity and a better approach to traceable measurements.

Here we outline how each individual method for NEMS excitation and readout has its own advantages and disadvantages and we show that in combination the techniques can be greater than the sum of the parts. By combination the optical and microwave methods, these will lead possible traceable measurements capability from micro to nanoscale.

II. Piezo Excitation & Optical Readout

The original NPL system was based on conventional piezo excitation and optical detection methods (see region inside the dashed line box in figure 1). This combination is similar to the techniques used in atomic force microscopy (

Manuscript received May 1, 2013. This work was funded by the UK NMS Programme and the EU EMRP Projects MetNEMS (NEW-08) and EMINDA. The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.

L. Hao is with the National Physical Laboratory, Teddington, TW11 0LW, UK (phone: +44-208-943-6296; fax: +44-208-614-0407; e-mail: ling.hao@npl.co.uk).

J. C. Gallop is with the National Physical Laboratory, Teddington, TW11 0LW, UK (e-mail: john.gallop@npl.co.uk).

M. Stewart is with the National Physical Laboratory, Teddington, TW11 0LW, UK (e-mail: mark.stewart@npl.co.uk)

K. Lees is with the National Physical Laboratory, Teddington, TW11 0LW, UK (e-mail: kevin.lees@npl.co.uk)

J. Chen is with School of Engineering and Design, Brunel University, Uxbridge UB8 3PH, UK (e-mail: jie.chen@brunel.ac.uk).

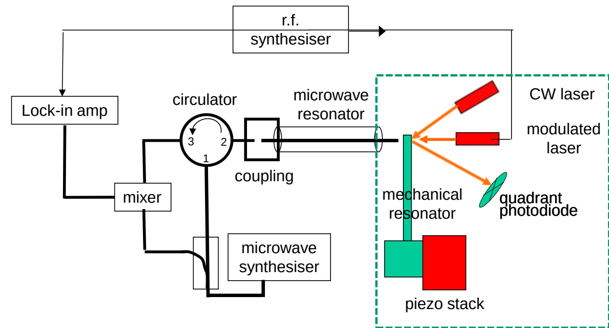


Fig. 1. Schematic of multi-functional mechanical resonator excitation and readout system.

(AFM) for exploring the resonant motion of mechanical resonators [9-12].

The simplest demonstration of the use of the system involves piezoelectric excitation of the mechanical cantilever using a piezo multi-layer stack or a ‘buzzer’ piezo driver. This is mounted on the same support as the cantilever and is driven by a modest a.c voltage (up to 10V peak to peak) applied at a variable frequency from a synthesized r.f. source (0 – 60MHz). A low power (3mW) diode laser beam is focused onto the mechanical cantilever resonator which must be reflecting of visible light) and the reflected beam is aligned on the centre of a quadrant photodiode. The difference voltage at the piezo driving frequency is measured with a lock-in amplifier and is proportional to the angular deflection of the spot on the cantilever on which the laser is focused. Fig. 2 shows a typical frequency sweep for a low resonant frequency cantilever.

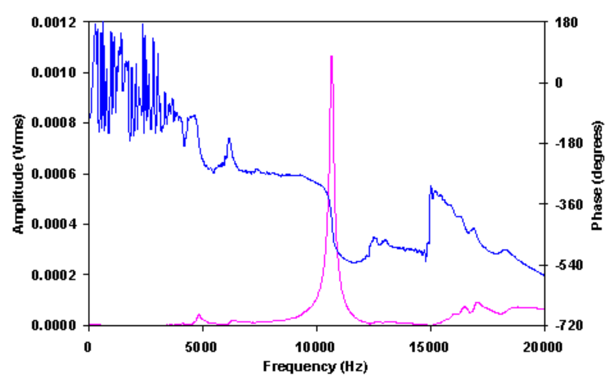
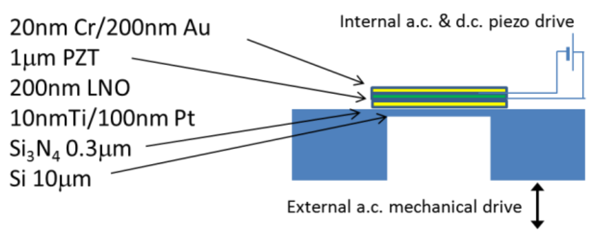
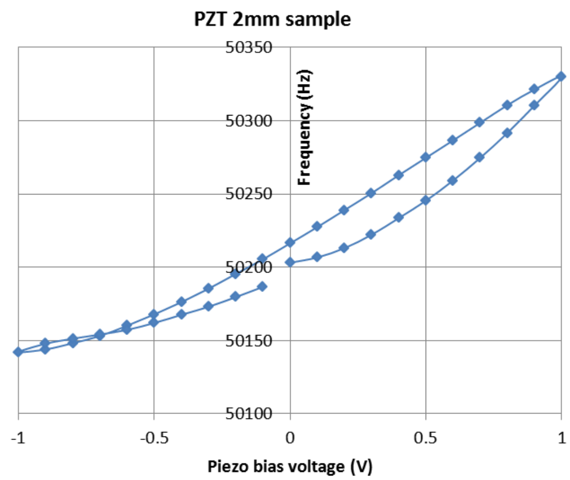


Fig.2. Optical readout of mechanical cantilever displacement as a function of piezo-driven frequency. Note the cantilever resonance at 10.5 kHz.

Apart from their use in excitation piezo materials may be *characterised* using the system we have developed. For example a piezo-active material, Lead zirconate titanate (PZT), deposited in a 1µm thick layer on a 10µm thick Si substrate with thin film metal electrodes on either side (see Fig. 3a). Fig. 3b shows the shift in resonance frequency, detected with a microwave readout technique (described in section IV). Note that very small applied voltage ~ 1 V produce easily measurable frequency shifts and hysteretic behavior is clear.



a)



b)

Fig. 3 a) schematic of PZT layer deposited on 10µm thick Si disc. b) Measurement of disc's resonant frequency as a function of applied voltage between upper and lower electrodes.

III. Optical Excitation as an Approach to Traceable Spring Constant Measurement

Increasingly, mechanical resonators are needed to make measurements of the forces involved in molecular motors or interactions between single bio-molecules. These are of great interest as a route towards improved understanding of fundamental biological processes. The forces are at the limit of what may be measured with scanned probe microscope techniques, and are typically in the range up to 1-100 piconewtons. It is very important to develop reliable measurement techniques in this critical area of study, which is attracting growing attention. There exist a number of problems in making *reliable and traceable measurements* of forces in the region below a few tens of nanonewtons. These arise from difficulties in characterising the spring constant *k* of scanned probe cantilevers. Cantilever force constant calibrations are traditionally made either through dimensional measurement methods or by measurement of thermal noise excitation amplitude which requires careful elimination of extraneous noise and interference as well as a knowledge of the cantilever temperature under operating conditions. (A summary of the strengths and, more significantly, the limitations of the major methods currently employed may be found in [9]).

We have proposed, and demonstrate here, a novel calibration method based on optical radiation pressure which we believe can eliminate many of these difficulties. In addition the method is relatively easily implemented on most existing AFM systems. Below the basis of the method is described, along with experimental results in air and vacuum. In particular, we show how thermal effects may be differentiated from that of radiation pressure. Requirements for extension of the method to liquid environments are also being considered.

A. Radiation Pressure as a Traceable Force Standard

When an incident flux of $\boldsymbol{n}$ particles per second each with momentum $\boldsymbol{p}$ is reflected without loss from a surface, the force $\boldsymbol{F}$ exerted on the surface is simply given by the rate of change of momentum:

$$\boldsymbol{F} = \frac{d\boldsymbol{P}}{dt} = 2\boldsymbol{np} \qquad (1)$$

Of course it is not just particles with rest mass which possess momentum. Massless photons of frequency ν and energy $\boldsymbol{h}\nu$ each possess momentum $\boldsymbol{h}\nu/\boldsymbol{c}$ (where c is the velocity of light in vacuum) and so, if totally reflected at normal incidence from a surface, there is a change of momentum of $2\boldsymbol{h}\nu/\boldsymbol{c}$ for each particle. The intensity of a beam of $\boldsymbol{n}$ such particles per second is $\boldsymbol{E}$ (where $\boldsymbol{E}=\boldsymbol{nh}\nu$so that the force applied by such a light beam is

$$\boldsymbol{F} = \frac{2\boldsymbol{E}}{\boldsymbol{c}} \qquad (2)$$

Note that for a total incident intensity of 1 W the force applied is only around 6 nN. A laser of this intensity would probably heat the cantilever unacceptably since, in the visible region of the spectrum, the reflectivity of even noble metals such as gold and silver is only of order 75-95 %. The additional power absorbed in the cantilever would raise its temperature dramatically since although cantilevers are made of high thermal conductivity materials (such as single crystal Si) their geometrical shape (long with small cross sectional area) does not favour efficient heat transfer to the clamped end of the cantilever.

It is likely the very low value of the ratio of force to intensity (arising from the extreme velocity of light) that has limited this method to date. However, this low ratio is very desirable for force measurements in the piconewton range. In this case milliwatt intensity levels are required, a power level available from a typical diode laser used to read out the deflection of the cantilever in a conventional atomic force microscope.

B. Imperfect Reflection and Non-Normal Incidence

In a real experimental situation the cantilever surface will not be perfectly reflecting but will have a reflection coefficient R (<1). In addition the light beam may be incident at angle θ with respect to the normal to the reflecting surface of the cantilever. In these cases the above expression for the force normal to the cantilever is modified to become:

$$\boldsymbol{F} = \frac{\boldsymbol{E}}{\boldsymbol{c}}\boldsymbol{(1+R)\cos\theta} \qquad (3)$$

A further complication may arise if the incident laser beam is not completely intercepted by the reflecting cantilever surface. If the density of incident laser power orthogonal to the propagation direction is *P(x,y)* the total force exerted on the cantilever becomes

$$\boldsymbol{F} = \frac{1+\boldsymbol{R}}{\boldsymbol{c}}\boldsymbol{\cos\theta}\iint \boldsymbol{P(x,y)dxdy} \qquad (4)$$

where the integral is over the entire reflecting surface of the cantilever and we have assumed the reflection coefficient and incident angle do not vary across it.

C. Experimental Realisation

To demonstrate the feasibility of our proposed calibration technique a cantilever test structure has been constructed (see Fig. 4). This consists of a mounting plate which attaches to an optical bench and a stable magnetic mount for any standard scanned probe cantilever. Two separate low power laser diodes (wavelength 633nm, with output power up to 3mW) are mounted on the same bench and may be focussed on the back-side of the free end of the cantilever by means of mirrors and adjustable lenses. The light from one laser reflected from the cantilever is incident on a quadrant photodiode, the position of which may be adjusted by a manually adjusted x-y translation stage. The power output of both laser diodes may be modulated by applied periodic signals.

The simple system has been further improved so that cantilever, lasers and photodiode are enclosed in a vacuum chamber which may be evacuated or filled with a variable pressure of any available gas. The vacuum environment has allowed measurements to be done in the low viscous damping regime (with pressures as low as 10^{-5} mbar) where the cantilever mechanical Q can be as high as 10^{5}. It is then easy to measure the thermally-generated vibrational noise amplitude.

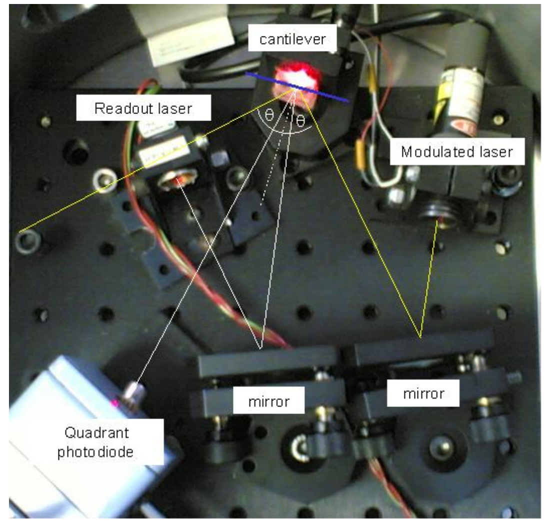


Fig 4. Image of laser excitation and readout system with cantilever mount on an optical table within the vacuum system.

D. Deconvolving Photo-Thermal and Radiation Pressure Effects

An important improvement over earlier investigation which we have demonstrated is the deconvolution of *thermomechanical* and *radiation pressure* responses of a hybrid cantilever. This thermal response is well known in inhomogeneous cantilevers. For example transparent Si_3N_4 cantilevers are coated with a thin film of Au to provide a highly reflecting surface but this means that the cantilever can act as a bi-metallic strip when its temperature changes since the thermal expansions of the two materials are different. When illuminated by even a low power laser the resultant heating produced can cause deflection of the cantilever tip which is orders of magnitude greater than the deflection brought about by radiation pressure. We have been able to accurately deconvolve the two effects by realising that the thermal response has a time constant which is relatively long for typical cantilevers. Then, by sweeping the frequency at which the incident laser beam is modulated, the low frequency regime is dominated by photothermal effects, whereas the high frequency regime is only sensitive to radiation pressure. Fig. 5 shows a plot of the cantilever amplitude response as a function of the modulation frequency in air. At low frequencies (< 1 kHz) photothermal effects dominate and can be larger in amplitude than true radiation pressure. However the thermal time constant of a typical cantilever will be longer than 1ms so for higher frequencies the cantilever acts a low pass filter, damping out the photothermal response.

E. Analysis of Results

At low applied laser modulation frequencies the cantilever is deflected but the amplitude of the laser-driven oscillation is reduced rapidly as the frequency rises, with a response

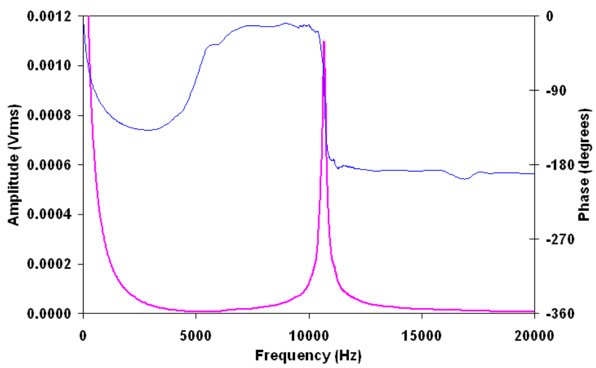


Fig. 5. Optical readout of laser excitation of cantilever. The sharp resonance at 10.5kHz is driven by radiation pressure from the modulated laser whereas the rise in amplitude at low frequency is due to the photo- thermal effects.

with the characteristic of a simple low pass filter. At higher frequencies the response amplitude shows a sharp resonance around 10 kHz. We also measure the phase of the cantilever response with respect to that of the laser modulation and this is also in excellent agreement with the modelled behaviour. For a cantilever in vacuum a single thermal time constant based on the specific heat and thermal conductivity of the cantilever materials accurately predicts the observed low frequency behaviour. In air or liquid the thermal behaviour is more complex and additional time constants will be needed for accurate modelling.

A simple analysis using the experimental data shown in Fig. 5 requires an intensity measurement of the modulating laser and the assumption that around 90% of the incident light is reflected from the cantilever. When the drive frequency $\omega = \omega_0$, the spring constant, k, can be calculated from the formula:

$$k = \frac{FQ}{|z|} \qquad (5)$$

where F is the radiation pressure force,

$$F = \frac{2P_R}{c} - \frac{P_A}{c} = 11.9\,pN \qquad (6)$$

Since the incident laser power is P_R = 2.00 mW, the absorbed power P_A = 0.11 mW and the quality factor, $Q = 67$, we only require to measure the amplitude of cantilever oscillations on resonance. This is derived from simple geometry and the measured quadrant photodiode output voltage change for a vertical displacement of the reflected laser beam. We find $|z|$ = 2.66nm, finally yielding a spring constant value for k = 0.30 ±0.03 N/m. This lies well within manufacturer's range of values for this type of cantilever, k of 0.07 to 0.4 N/m.

To use the radiation pressure method in its simplest form it is necessary that the laser spot size is considerably smaller than the cantilever width, to ensure that the entire available laser power is reflected from the cantilever. In many situations this will not be achieved. Recent work has shown that the impact of a laser spot considerably wider than the cantilever can be quite precisely modelled, allowing cantilever calibration to be achieved under these conditions.

IV. Microwave Homodyne Excitation and Detection System

More recently several groups have applied microwave methods to readout resonator motion in NEMS [10-13]. Microwave measurement using high Q resonators becomes attractive due to the high sensitivity of frequency measurement and the very low phase noise from synthesized microwave sources, in contrast with optical systems. Figure 1 shows schematic of multi-functional cantilever resonator excitation and readout system.

Elsewhere [11] we have described our novel method for excitation of mechanical resonators using amplitude modulated microwave radiation. This is based on a quarter wave microwave coaxial resonator with the open end connected to a sharp tip, to produce a very localized intense microwave field in a very limited region close to the tip. The spatial range of this high field region is on the order of the radius of curvature of the tip, which can be, comparable to the smallest cantilever dimensions. This has proved the most simple to use and convenient excitation method, carrying no penalties of spurious resonance excitation nor requiring ultra-precise 3D positioning. The approach of the microwave near-field tip towards the resonator can be sensitively detected by monitoring the reflection dip of the microwave signal returned from the circulator (see Fig. 1) as the microwave frequency is swept through resonance. By using external I-Q mixers it is possible to extend the microwave drive signal to hundreds of MHz, far higher than can be achieved with piezo or optical methods. Microwave readout is achieved by mixing down the reflected signal from the circulator to the r.f. AM frequency using a mixer. It is possible to maximise the sensitivity of the readout by bringing the near-field tip as close as possible to the mechanical resonator while also adjusting the phase and amplitude of the local oscillator input to the mixer so that the IF output is optimised. In this way the thermal noise oscillations of resonators at all length scales can be observed.

A. *Microwave Cooling of mechanical Noise*

Elsewhere we have reported sideband cooling of the effective temperature of a mechanical resonator at a frequency of 250 kHz using a microwave source, red detuned from the dielectric resonator centre frequency [11]. More recently we have demonstrated that this technique can also be applied to even lower frequency resonators, in this case a 7 kHz Au-coated Si_3N_4 ultra-soft mechanical resonator.

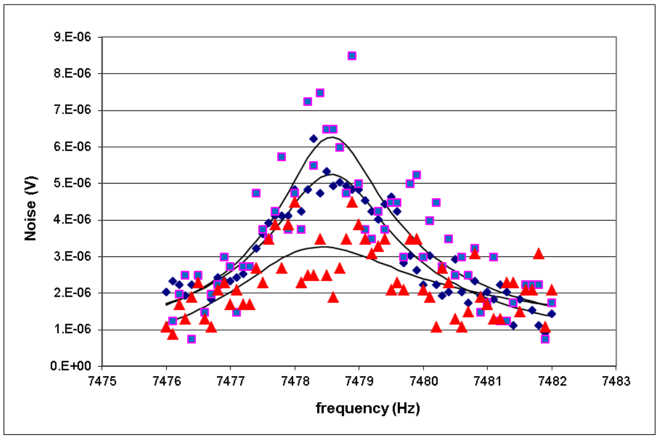


Fig. 6. Demonstration of sideband cooling of soft mechanical resonator. The noise analysis shows that the linewidth for red-detuned microwave drive (red triangles) is broader than for blue-detuned microwave drive (blue squares). The area under the Lorentzian is smaller for red-detuning, indicating mechanical resonator cooling.

The data is shown in Fig. 6. Note that the red points indicate the frequency spectrum of thermal noise from the cantilever when it is irradiated with microwaves detuned by 2 MHz below its resonant frequency. The black points are when it is tuned to resonance and the blue points indicate blue detuning by 2MHz. The Lorentzian fits to the data show a broader linewidth but lower amplitude for red de-tuning than no detuning whereas blue detuning shows the opposite. The area under the Lorentzian curve is proportional to effective temperature, showing that this is decreased for red-detuning (cooling) and increased for blue detuning (heating). This sideband cooling technique, when applied at low temperatures, has recently led to the achievement of the quantum mechanical ground state of a mechanical resonator [14-20].

V. Comparison of Excitation & Readout Methods

Here we briefly summarise our experience with this multifunctional system. Piezo excitation is simple and cheap but is not selective so that a wide range of mechanical resonances in support structures or wiring may be observed, with similar frequencies and Q values to that expected of the M/NEMS resonator. In addition it is not realistic to achieve broadband piezo excitation above a frequency of a few MHz. Its limitations are apparent from the noisy low frequency traces both in amplitude and phase which are shown in Fig. 2. A further advantage that both optical and near-field microwave excitation possess is that an excitation force may be localized at a chosen point on the mechanical resonator (within the limits set by the diffraction effects for optical wavefronts or the near-field spatial scale for microwaves). In terms of sensitivity the microwave readout is at least as good as the optical readout, considering signal to noise ratios at low frequencies. Microwave readout comes into its own for higher frequency mechanical resonators such as graphene, which are quite lossy at microwave frequencies but highly transparent to light. Thus the material will not give an adequate reflected optical signal.

Thermal noise is easily visible using microwave and optical readout methods, at least for N/MEMS resonator greater than one micrometre in length. Resonator stiffening from microwave power and laser illumination are detectable and may be utilised as a tuning function. The result of this is that it is very easy to overdrive soft cantilevers so that the coupled microwave and mechanical system becomes non-linear. Again this feature may be turned to an advantage since non-linear behaviour may allow parametric amplification and bistable operation. Microwave excitation is suitable for resonators of micrometre sized or smaller. It can be used at high frequencies, up to hundreds of MHz, although it is difficult to ensure that the force applied is traceable. Optical excitation can be made into a traceable method for spring constant calibration, provided care is taken to understand any associated thermo-mechanical forces and to allow for them. It is also necessary to have a good knowledge of the reflectivity achievable from the cantilever material at the wavelength of the optical excitation.

VI. Conclusions

We have described in some detail a multi-purpose micro- and nano-electromehcnaical system resonator measurement facility. This combines several different excitation methods and readout mechanisms. We compare and contrast the different techniques and demonstrate how they may best be used to complement one another.

Acknowledgment

We thank Dr. Julia Davies (Imperial College London, formerly of NPL) who carried out much of the optical spring constant calibration measurements and Dr. Tobias Lindstrom (NPL) for their helpful technical discussions and input.

References

[1] K. L. Ekinci and M L Roukes, “Nanoelectromechanical systems,” *Rev. Sci. Instrum.* vol.76 061101, 2005.
[2] T. J. Kippenberg and K. J. Vahala “Cavity Optomechanics: Back-Action at the Mesoscale”,*Science* vol. 321 1172-4, 2008.
[3] S. Etaki, M. Poot, I. Mahboob, K. Onomitsu, H. Yamaguchi, and H. S. J. van der Zant, “Motion detection of a micromechanical resonator embedded in a d.c. SQUID,” *Nat. Phys.* vol.4, pp785-788, August 2008.
[4] L. Hao, J. C. Gallop, D. Cox, E. J. Romans, J. C. Macfarlane and J. Chen, “Focussed Ion Beam NanoSQUIDs as Novel NEMS Resonator Readouts,” *IEEE Trans. Appl. Supercond.*, vol.19, no.3, p693-696, 2009.
[5] Mo Li, H.X. Tang, M.L. Roukes, “Ultra-sensitive NEMS-based cantilevers for sensing, scanned probe and very high-frequency applications,” *Nat. Nanotechnol.* vol. 2, pp114–120, Feb. 2007.
[6] M Blencowe Quantum electromechanical systems Physics Reports 395 2004 159-222
[7] T.P. Burg, M. Godin, S.M. Knudsen, W. Shen, G. Carlson, J.S. Foster, K. Babcock, S.R. Manalis, “Weighing of biomolecules single cells

and single nanoparticles in fluid," *Nature* vol. 446, pp1066–1069, 2007.

[8] L. Hao, D.C Cox, J.C. Gallop, J. Chen, S. Rozhko, A. Blois and Ed. Romans, 'Coupled NanoSQUIDs and Nano-Electromechanical Systems (NEMS) Resonators', *IEEE Trans. Appl. Supercond.*, vol.23, no.3, 1800304, 2013.

[9] N A Burnham, X Chen, C S Hodges, G A Matei, E J Thoreson, C J Roberts, M C Davies and S J B Tendler, "Comparison of calibration methods for atomic-force microscopy cantilevers", *Nanotechnology,* vol. 14 pp. 1-6, 2003

[10] T. Faust, P. Krenn, S. Manus, J. P. Kotthaus and E. M. Weig, ''Microwave cavity-enhanced transduction for plug and play nanomechanics at room temperature'', *Nature Communications*, vol.3, p1723, Feb. 2012.

[11] L. Hao, J.C. Gallop and D.C. Cox, "Excitation detection and passive cooling of a micromechanical cantilever using near-field of a microwave resonator," *Appl. Phys. Lett.* vol.95, 113501, September, 2009.

[12] L. Hao, S. Goniszewski, J. Chen and J. Gallop, "Microwave excitation and readout of nano- and micron scale cantilevers," *Applied Surface Science,* vol. 258, issue6, p2192-2195, 2012.

[13] L. Hao, J. Gallop and J. Chen, "Near-field Microwave Excitation and Detection of NEMS Resonators", 2012 12th IEEE International Conference on Nanotechnology (IEEE-NANO), 6321922, 2012. DOI: 10.1109/NANO.2012.6321922

[14] M. Aspelmeyer and K. Schwab, "Focus on Mechanical Systems at the Quantum Limit," *New J. Phys.* vol.10, 095001, September, 2008.

[15] A. Schliesser, O. Arcizet, R. Rivière, G. Anetsberger, T.J. Kippenberg, "Resolved sideband cooling and position measurement of a micromechanical oscillator close to the Heisenberg uncertainty limit," *Nat. Phys.* vol.4, pp415–419, 2009.

[16] T. Rocheleau, T. Ndukum, C. Macklin, J.B. Hertzberg, A.A. Clerk, K.C. Schwab, "Preparation and detection of a mechanical resonator near the ground state of motion," *Nature* vol.463, pp72–75, 2010..

[17] A. Naik, O. Buu, M. D. LaHaye, A. D. Armour, A. A. Clerk, M. P. Blencowe and K. C. Schwab, "Cooling a nanomechanical resonator with quantum back-action," *Nature* vol.443, pp193-196 , September 2006.

[18] C. A. Regal, J. D. Teufel and K. W. Lehnert, "Measuring nanomechanical motion with a microwave cavity interferometer," *Nature Phys.* vol.4, pp555–560, 2008.

[19] J.D. Teufel, Dale Li, M. S. Allman, K. Cicak, A. J. Sirois, J. D. Whittaker & R. W. Simmonds, "Circuit cavity electromechanics in the strong-coupling regime", *Nature,* vol. 471, 204-8, 2011

[20] J. D. Teufel, T. Donner, Dale Li, J. W. Harlow, M. S. Allman, K. Cicak, A. J. Sirois, J. D. Whittaker, K. W. Lehnert & R. W. Simmonds "Sideband cooling of micromechanical motion to the quantum ground state", *Nature*, vol. 475 pp.359-63, 2011