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# Casimir force experiments with quartz tuning forks and an atomic force microscope (AFM)

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## Abstract

The aim of the measurement series is to study the Casimir force, specifically the effects of different materials and geometries. The art of measuring sub-nano Newton forces has been engineered to a great extent in the material sciences, especially for the atomic force microscope. In today's scanning microscope technologies there are several common methods used to measure sub-nano Newton forces. While the commercial atomic force microscopes (AFM) mostly work with soft silicon cantilevers, there are a large number of reports from university groups on the use of quartz tuning forks to get high resolution AFM pictures, to measure shear forces or to create new force sensors. The quartz tuning fork based force sensor has a number of advantages over the silicon cantilever, but also has some disadvantages. In this report the method based on quartz tuning forks is described with respect to their usability for Casimir force measurements and compared with other successful techniques. Furthermore, a design for Casimir force measurements that was set up in Berlin will be described and practical experimental aspects will be discussed. A status report on the Casimir experiments in Berlin will be given, including the experimental setup. In order to study the details of the Casimir effect the apparatus and active surfaces have to be improved further. The surfaces have to be flatter and cleaner. For better resolution, cantilevers and tuning forks with a low spring constant have to be employed.

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(Some figures in this article are in colour only in the electronic version)

## 1. Introduction

One of the most fascinating effects in quantum field theory is the Casimir effect, which leads from microscopic fluctuations to a macroscopic force [1]. In 1948 Hendrik Casimir came

to the conclusion that two parallel metal plates should experience an attractive force. Since there are less quantum states between the plates than there are outside the plates there should be a difference in photon pressure that leads to a force on the plates. The effect is also closely related to the van der Waals forces [2]. In fact Casimir found his result while working on discrepancies of van der Waals forces. After the theoretical prediction Sparnaay made the first measurements in 1958 and van Blokland in 1978. The early experiments had large uncertainties.

Up to the late 1990's the work on the Casimir effect was mostly theoretical [3]. The recent expansion of experimental work on the Casimir effect was started with the groundbreaking work of Lamoreaux [4] and Umar Mohideen [5]. The late experiments use state-of-the-art nanopositioning with piezo actuators. Umar Mohideen uses the well-known force measurement techniques that are used in atomic force microscopy. Another notable precision experiment is based on a microelectromechanical torsional oscillator (MTO) [6]. An overview of recent experimental developments can be found in Onofrio's publication [7].

The motivation for this work in setting up a Casimir force measuring apparatus lies in the fact that we still do not have sufficient experimental data on the influence of material and geometries, i.e. boundary conditions, on the energy of the vacuum state. So the shape of the objects bordering the space or being in the space in question is of interest. The aim of the author is not to conduct another set of precision Casimir experiments but to be able to pursue interesting questions on the nature of the Casimir force and the quantum fluctuation. In order to carry out own experiments a suitable experimental setup had to be developed. The content of this paper is largely the knowledge gained by developing the apparatus and exploring the different methods in order to be able to choose the suitable method. The pro and cons of the different ways to measure Casimir forces are discussed at the end of the paper. A focus in this paper is laid on the method to measure Casimir forces with quartz tuning forks, as this is the newest and most original way that was pursued to set up a Casimir experiment in Berlin. Since an AFM was acquired during the process, mainly to study and control the surface roughness, the ability to measure Casimir forces with quartz tuning forks is compared with the method of measuring the Casimir force with an AFM based on light silicon cantilevers with an attached polystyrene sphere, as developed by the Riverside group and Umar Mohideen. Since our results are not better than those published there, the interested reader is referred to the publications of the Riverside group. Details on the measuring technique and the interesting results obtained there can be found in [5, 8]. Another successful technique to measure Casimir forces with high precision is the MTO [6, 9]. Since the author had no access to an MTO and to apply it in his laboratory, the MTO approach was not further pursued. To give justice to this important method, it will be included in the final discussions and the comparison at the end of the paper.

While designing the measuring apparatus for the Casimir force measurements, it was brought to the attention of the author by solid state physicist Bert Rähler that there is an alternative force measuring technique in experimental atomic force microscopy utilizing commercially available quartz crystal frequency standards [10]. A survey of the current literature on force measuring techniques made it clear that the quartz tuning fork usually employed in the watch industry for time measurements is widely used to measure sub-nanonewton and piconewton forces. The quartz tuning fork is a highly developed and thoroughly engineered precision instrument. The watch industry produces billions of these tuning forks each year. The most used frequency is  $2^{15} = 32\,768$  Hz. To achieve quality factors of  $\sim 10^5$  the crystal orientation and manufacturing process need to be of high standard. The quartz tuning fork is currently used for measuring shear forces in scanning near field optical microscopy (SNOM) [11] and to measure normal forces in magnetic force microscopy

[12], in atomic point contact measurements [13] and for seismometers and micro gyros as accelerometers [14]. The large amount of available literature on these topics is very helpful in designing the apparatus for Casimir force measurements. Grober *et al* discuss the fundamental limits of force detection using quartz tuning forks [15]. Grober found that at room temperature, atmospheric pressure and normal air gas composition, the noise level of his tuning fork was  $0.62 \text{ pN Hz}^{-1/2}$  and shows a root-mean square motion of  $0.32 \text{ pm}$ . Karrai has also published a review paper about the basic operation of a quartz tuning fork as a shear and friction force sensor, which covers most of the essential topics such as oscillation model, spring constant, signal detection and noise [16]. Giessibl, who is credited to be the first to show atomic resolution with a noncontact AFM, has published on the understanding of the physical relationship between the frequency shift of the tuning fork and the applied force [17]. The conversion of the frequency shift signal into a force value is of high importance in the application of quartz tuning forks in Casimir force measurements.

Rensen *et al* compared the resolution of force sensing with quartz tuning forks and light silicon cantilevers [18]. The motivation for setting up the quartz tuning fork was that the resolution of the tuning fork is higher compared with silicon cantilevers. In addition, the tuning fork is self-sensing and the measuring only needs electronic parts and is supposed to be more robust and cost effective than the optical laser reflection used in the light silicon cantilever setup. The tuning fork is also very suitable for low temperature force measurements because of the low energy dissipation of the fork, as low as picowatts, and the removal of optical dealignment problems on cooling down [19].

Hence, it seems promising to use quartz tuning forks for Casimir measurements as they are inexpensive, self-sensing, have no need for laser and optics, are easy to operate in vacuum and are robust and stiff, while being sensitive enough for Casimir forces.

## 2. Force measurement with quartz tuning forks

The force measurement with quartz tuning forks is based on the frequency shift that occurs if a force is applied to the tuning fork when it is operated close to its resonance frequency. The piezoelectric effect of the quartz crystal yields an electric signal proportional to the deformation. The tuning fork can be driven by applying an ac voltage directly to the electrodes of the tuning fork. A simple model for the tuning fork is a driven harmonic oscillator. The response to a driving frequency is well known. The amplitude and phase response are shown in figure 1.

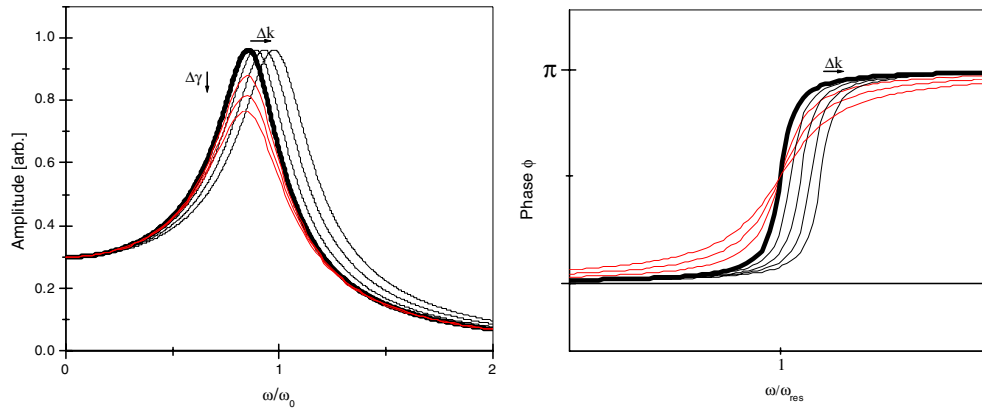
In our case the external force has to be included in the equation. The external force can be treated as a change of the spring constant of the system. The result is shown in figure 1. The resonance frequency is shifted when an external force is applied. It can also be suitable to track the phase difference between the driving force and the system because at the resonance frequency the gradient of the phase  $\phi$  is much larger than the gradient of the amplitude.

The shift of the resonance frequency  $f_0$  is related to the derivative of the force on the tuning fork.

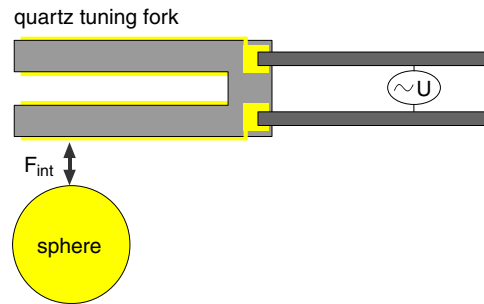
$$\Delta f \approx \frac{f_0}{2k} \frac{\partial F}{\partial x}. \quad (1)$$

Much more detail can be found in the AFM literature under the keyword 'dynamic mode theory'. An important value of the resonance is the quality  $Q$ . The quality of a resonance is known as the frequency  $f$  divided by the full width at half maximum (FWHM).

$$Q = f/\text{FWHM}. \quad (2)$$



**Figure 1.** Amplitude and phase of a driven harmonic oscillator with the effect of rising spring constant  $k$  and damping  $\gamma$  (red curves).



**Figure 2.** Casimir force measurement with a quartz tuning fork.  $U$  is the driving ac voltage and  $F_{int}$  is the Casimir force.

The physical amplitude of the tuning fork displacement has to be small in relation to the desired position resolution. We need a quasi-static regime in order to make Casimir measurements, as the position resolution should be of a few nm. The displacement of the tuning fork arms has been studied by several authors. If the driving voltage is sufficiently small, the displacement is less than a few nm and can be as low as pm [12, 20].

A simple formula for Casimir force in the flat sphere geometry (figure 2) is

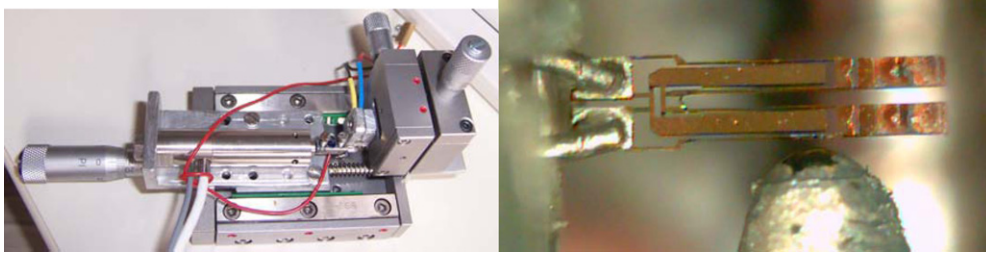
$$F = -\frac{\pi^3 \hbar c}{360} \frac{R}{x^3} \tag{3}$$

where  $R$  is the radius of the sphere,  $x$  is the distance between the flat surface of the tuning fork and the sphere,  $c$  is the speed of light in vacuum and  $\hbar$  is the Planck's constant. If we use formula (1) with the force relationship (3), we find the expected frequency shift

$$\Delta f \approx \frac{3 f_0 \pi^3 \hbar c}{2k} \frac{R}{360 x^4}. \tag{4}$$

The spring constant  $k$  can be calculated by measuring the physical dimensions of the tuning fork. The spring constant is given by

$$k = \frac{E}{4} W \left( \frac{T}{L} \right)^3 \tag{5}$$



**Figure 3.** The holding structure for the tuning fork (left) and the ball and tuning fork in close up (right).

where  $L$  is the length,  $W$  is the width,  $T$  is the thickness of the tuning fork and  $E$  is the Young modulus [8]. For the quartz crystal  $E$  is

$$E = 7.87 \times 10^{10} \text{ N m}^{-2}. \quad (6)$$

The sensitivity of our tuning fork measuring system is governed by the sensitivity of the frequency measurement. In our case a change of 1.5 mHz can be detected. With formula (1) we can calculate the corresponding force gradient and force sensitivity.

$$\frac{\partial F}{\partial x} = \frac{\Delta f}{f_0} 2k. \quad (7)$$

A typical value for  $k$  is  $2500 \text{ N m}^{-1}$  and frequency  $f_0$  of the used tuning forks is 32.8 kHz. The sensitivity of the force gradient is then  $0.2 \text{ pN nm}^{-1}$ . With a position sensitivity of 1 nm this would convert to a force sensitivity of 0.2 pN. A higher frequency would lead to a higher sensitivity. There are tuning forks available with a frequency of 100 kHz, but their spring constant is around  $100 \text{ kN m}^{-1}$ . Therefore, the sensitivity with 100 kHz tuning forks is lower than those of the 32 kHz tuning forks with selected low spring constants.

### 3. Experimental setup

#### 3.1. Quartz tuning fork

The basic setup for the tuning fork consists of the holding structure with  $xyz$  micrometer screws and a piezo actuator from PI seen in figure 3 left side. The tuning fork and the ball can be seen in closeup in figure 3 right side.

The tuning fork is driven by a function generator (Agilent 33220). The driving voltage is constant and the lowest possible strength of 10 mV is used. Often attenuators from 10 to 39 dB are used to drive the tuning fork with minimal voltage in order to keep the deflection amplitude of the tuning fork as low as possible. The voltage is applied between ground and the electrode of the tuning fork. The other electrode is connected to the current input of Standard Research SR 510 lock-in amplifier, as shown in figure 4. The current input is a virtual ground. The grounds of the function generator and the lock-in amplifier are connected. The lock-in is read out with a Keithley 2000 6.5 digit digital multimeter. A PC with a Lab view program is used to read out the current value from the lock-in via the Keithley. The program also scans through the frequency in a preset band in order to record resonance curves as shown in figures 5 and 6. The time constant of the lock-in was varied between 100 ms and 3 ms. The current range was typically 100 nA, depending on the attenuation of the driving signal and the air damping by the atmospheric pressure. In order to measure the frequency shift, a PC was

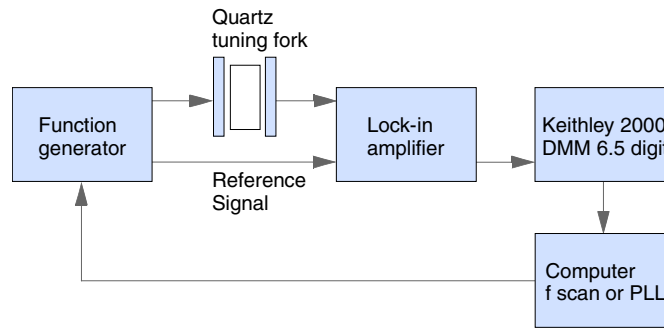


Figure 4. Electrical setup to measure the tuning fork resonance.

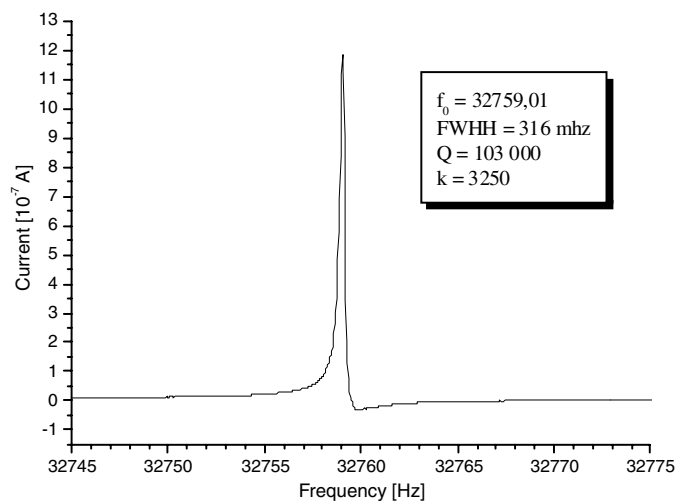


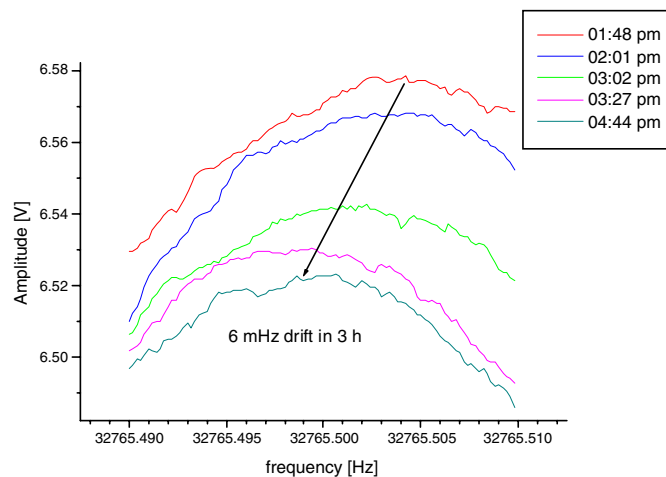
Figure 5. Resonance of the tuning fork in vacuum.

programmed to automatically set the frequency to a maximum amplitude. The speed of this algorithm has to be improved.

In figure 5 one resulting resonance of the tuning fork in  $2 \times 10^{-5}$  mbar vacuum can be seen. The quality is  $10^5$ .

In order to conduct Casimir force measurements the long-term stability is important. The drift of the resonance frequency was measured to 6 mHz in 3 h. The result is shown in figure 6. The drift was recorded in early summer. In winter the room temperature is not as stable, when running the system the drift can be considerably larger. The temperature dependence of the tuning fork is  $-0.038 \text{ ppm } ^\circ\text{C}^{-2}$  according to the data sheet.

The same 8 mm length, 3 mm diameter package cylinder contains a large variety of 32 768 Hz tuning forks. But the physical dimensions of the tuning fork vary from manufacturer to manufacturer and for the different product lines. The spring constant is connected to the physical dimensions as in formulae (5) and (6). The spring constant of used tuning forks varies from  $42 \text{ kN m}^{-1}$  to  $1200 \text{ N m}^{-1}$ . According to formula 1 the frequency shift is inversely proportional to the spring constant. The frequency is fixed and also the detectable frequency



**Figure 6.** Temperature stability of the tuning fork resonance.

shift is fixed; in our case it is about 1.5 mHz. For the Casimir measurements a metallic or metallized ball is approached to the side of the tuning fork as seen in figure 3 right side. As can be seen in formula (4) the frequency shift is proportional to the frequency and the radius of the sphere. So it is desirable for Casimir force measurements to use tuning forks with a low spring constant, practically 3000 to 1000  $\text{N m}^{-1}$ . Also, a sphere with a larger radius leads to a stronger force and a larger frequency shift at a given distance.

### 3.2. Quartz tuning fork built into a commercial AFM

In order to take advantage of the fully engineered positioning and vibrational damping of the commercial AFM, the tuning fork was built into this system. The AFM consists of a head, a body, the control electronics and a PC. The head usually includes the silicon cantilever, the laser and the photodiodes as well as  $x$ - $y$  positioning for the laser and the photodiodes. The body includes the  $xyz$  piezo scanner and three stepper motors for the coarse approach. The tuning fork either replaced the silicon cantilever or was glued onto the AFM-specimen disk. In the first case, the ball is either glued onto a silicon cantilever or a stiff wire or substrate. When the sphere is glued to the cantilever, the force can be measured with the cantilever and the reflected laser, while simultaneously the frequency shift can be recorded. This configuration allows us to calibrate the tuning fork measuring process.

Electrically the tuning fork is driven by the AFM built in lock-in. One leg of the tuning fork is connected to the driving voltage, the other is connected via a current-to-voltage preamplifier to the lock-in, replacing the signal from the photodiodes.

The software of the AFM allows us to make approach curves. For this, the cantilever tip or the sphere, is approached from the current position towards the surface of the substrate by the set distance and back. Both directions can be recorded. The software allows us to record the phase shift during the approach curve. As can be seen in figure 1, the gradient of the phase angle is maximal at the resonance frequency. So the phase is very sensitive to a shift in resonance frequency. From the difference in phase, the frequency shift can be calculated.



### 3.3. Sample preparation and evaporation

Great care has to be taken that the surface roughness is less than 30 nm. The surfaces have to be clean and are then evaporated with empirically optimized parameters. The gold is evaporated on a tungsten wire in vacuum. The quality of the surfaces is checked with the AFM. The surface roughness is determined from the surface relief pictures taken with the AFM

## 4. Status of the experiments

So far the physical structure is complete and the driving circuitry has been implemented. To improve the positioning, vibrational damping and the software, the tuning fork force sensor has been integrated into the commercial AFM. This setup provides  $xyz$  positioning, stepper motors for gross approach, a camera for gross positioning and dynamic mode software to record approach curves. Some adjustments in the software still have to be done. The circuit described in section 3.1 still needs a faster PLL software to track the resonance frequency faster.

For the case of the tuning fork, the main improvement that needs to be done is to lower the surface roughness of the sphere and the flat surface. It is likely that a flat piece of material or a sphere needs to be glued to the side of the tuning fork in order to achieve the needed roughness. The side of the tuning fork that needs to be used to have the configuration as shown in figure 3 seems to have 70–100 nm ridges due to the chemical etching or laser manufacturing process. If these ridges are directly under the sphere we do not have a flat sphere configuration desired for the Casimir regime. One solution would be to glue a small flat piece or even the entire sphere to the tuning fork. Nearly all reported [11–21] measurements with tuning forks use modified forks. Usually some material is glued to the tuning fork in order to build the desired sensor. Only Morville *et al* pressed a glass fibre against the tuning fork without gluing in order to study different quality factors and to optimize the  $Q$ -factor [21]. Morville also surveyed the literature and found that the  $Q$ -factor of modified tuning forks reported by different researchers range from several hundred to 9000.  $Q$ -factors of unmodified tuning forks range from  $10^4$  to  $10^5$ . In early tests the author found that the  $Q$ -factor of a tuning fork with a small flat piece glued upon drops from 40 000 to 7700. The resonance frequency was shifted from 32 765 Hz to 32 600 Hz. This is still a high  $Q$  and is promising for precision measurements. Attaching 200  $\mu\text{m}$  polystyrene spheres lowered the  $Q$  to 880. To keep the symmetry of the tuning fork, identical mass should be attached to both tines, one piece acting as a counter weight. If the symmetry cannot be kept, it is better to glue down one tine to a solid material and to use only the free tine, because the tuning fork configuration has high  $Q$  only if there is a high degree of symmetry. All reported measurements showed that tuning forks with attachments make sensitive force sensors. Careful assembly of the sensor is necessary to keep the quality factor close to  $10^4$ . The sensitivity is at present governed by the least detectable frequency shift or phase difference. Since the detection is not at the fundamental limit, but connected to the circuitry and the stability of the reference signal, the sensitivity is not directly affected by a lower  $Q$ . In contrast a lower  $Q$  has the advantage to increase the frequency shift range detectable by the change in phase at the free-resonance frequency. This increases the maximum force still detectable before the phase angle flattens out into 0 or  $\pi$  (figure 1). Taking the approach curve with a bare tuning fork having a  $Q$  of  $10^5$  at the resonance frequency and recording the phase angle give a very sudden response and the frequency shift is limited to 100 mHz before the phase angle reaches 0.

A very low roughness of the employed surfaces is necessary to measure the Casimir force. With the tuning fork sensors so far achieved in our work, that have a  $k$  of 2000–3000  $\text{N m}^{-1}$ ,

**Table 1.** Advantages and disadvantages of silicon cantilevers, micromechanical oscillators (MTO) and quartz tuning forks.

• Silicon cantilever	• MTO	• Quartz tuning fork
– Advantages:	– Advantages	– Advantages:
• Absolute force	• High sensitivity	• Self-sensing
• Commercially available	• Static mode with direct force	• Only electrical equipment needed
– Disadvantages:		
• Optical measuring system	• Dynamic mode with high Q and high accuracy	• Easy to operate
		• Low cost
• Low stiffness	• Only electrical force measurement	• High stiffness
• Catch in / sticking	• Small amplitude of oscillation	• Small amplitude of oscillation
• Adhesion	• Smooth surfaces	• Robust
• Fragile	– Disadvantages	• Low energy dissipation
	• Low spring constant	– Disadvantages:
		• Proportional to force derivative
		• Oscillating sensor

a frequency resolution of 1.5 mHz and a sphere with a 100  $\mu\text{m}$  radius, the distance between the flat and the sphere has to be less than 220 nm in order to measure the Casimir force. In order to take advantage of the high stiffness and the slower jump to contact of the tuning fork, the surfaces have to have a roughness of less than 10 nm. The other way to take advantage of the fact that the measurable force can be larger compared to low  $k$  configurations with light silicon cantilevers, is to use larger spheres. In formula (3) we can see that the force is proportional to the radius of the sphere. A factor of 5 seems possible, but the main problem will be the alignment of the flat surface to the larger sphere. The distance between the sphere and the flat surface still need to be less than the distance to the sides of the flat piece, in order to stay in the Casimir regime.

## 5. Conclusions and outlook

Silicon cantilevers and quartz tuning forks are both promising for measuring the Casimir force. The silicon cantilevers are more suited for absolute force measurements, while the tuning forks show very good sensitivity and are promising for detecting changes in the Casimir force. Great care has to be taken to measure the Casimir force with both systems. Careful and precise sensor preparation, smooth and clean surfaces and careful alignment of surfaces are necessary to keep the sensitivity up. In table 1 the main advantages and disadvantages of the light silicon cantilever AFM, the quartz tuning fork setup and the micromechanical oscillator (MTO) [9] are summarized. For completeness also the key features of the MTO are included

An important conclusion is that for the AFM and the tuning fork a very low  $k$  is desired in order to measure Casimir forces. In the case of the tuning fork thin and small tuning forks are selected, because they have a low  $k$  of 1000 to 3000  $\text{N m}^{-1}$ .

So far the surface roughness was too large to observe the Casimir force with sufficient resolution. In order to overcome this, the setup is currently being improved by a plasma surface cleaning option and a faster frequency tracing software. Furthermore, it will be necessary to glue a flat piece of material or metallized micro sphere to the side of the tuning fork, in order to have appropriate roughness conditions for the Casimir regime.

In the case of the silicon cantilever, the spring constant needs to be low, best 0.01  $\text{N m}^{-1}$  to get a better resolution. At the same time the reflected laser signal should be high, best

3000 mV or more to overcome the inherent noise problem of the system. With these improvements both systems should be capable of measuring and resolving Casimir forces with the desired specifications.

Once these minor improvements are implemented, the planned series of experiments for investigating the influence of real material and surrounding geometries can start.

### Acknowledgment

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