PRECISION MEASUREMENT OF THE CASIMIR FORCE FOR Au USING A DYNAMIC AFM

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The gradient of the Casimir force between carefully cleaned Au surfaces of a sphere and a plate is measured using a dynamic atomic force microscope in the frequency modulation regime in high vacuum. The electrostatic calibration of the setup did not reveal any effect of patches or surface contaminants. The experimental data for the force gradient are found to be consistent with theory using the plasma model approach over the entire measurement range. The Drude model approach is excluded by the data at separations from 235 to 400 nm at a 67% confidence level.

Keywords: Casimir force; precision measurement; dynamic regime.

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1. Introduction

The Casimir effect¹ has become very important in modern physics. Not only is it the most accessible evidence of vacuum fluctuations in macroscopic systems but also has important applications in many areas such as condensed matter physics, atomic physics and cosmology. Because of its unique dependence on the separation, geometry, and material properties, the Casimir effect has potential applications in modern micro- or nanotechnology^{2–4} and in multidisciplinary areas.⁵ Therefore, theoretical activity in the analysis of the Casimir effect has dramatically increased in the last few years. To provide deeper insights, precise Casimir force measurements are necessary.

Recently, the atomic force microscope (AFM) technique has become the method of choice for precision measurements of the Casimir force.^{6–11} Two modes of AFM operation, static deflection and dynamic have been employed. In the static deflection mode, the force measurement results from the linear elastic response of the cantilever related to Hooke's law. The details of this mode of operation are discussed in Ref. 12. The advantages of the static deflection mode are the ease of use and the ability

to measure the force at short distances as close as 60 nm. Also the contact point between sphere and plate provides a reference for the corrections in separation due to drift and the precise determination of the separation distance.

The operation of the AFM technique in the dynamic mode^{13,14} for Casimir force measurement methodology is very promising due to its much greater sensitivity. The dynamic measurements can also be classified into three techniques: amplitude shift, phase shift and frequency shift. In the first the cantilever is driven at a fixed frequency close to its natural resonant frequency. The presence of a force gradient shifts the resonant frequency causing a change in the oscillation amplitude which is detected using a lock-in amplifier.¹⁰ In the phase shift method, the corresponding change in phase between drive and the cantilever is monitored also using a lock-in amplifier.⁹ In the frequency shift technique, a force gradient acting on the cantilever modifies the resonant frequency and the corresponding change in frequency is recorded using phase locked loop (PLL). In the first two techniques of phase and amplitude shifts, the range of changes is restricted to the frequency bandwidth of the cantilever oscillator. This limits both the precision and the separation distance ranges in Casimir force measurements. The frequency shift technique, which is also referred to as frequency modulation (FM) is free from such limitations and has been shown to lead to a factor of 10 in increased sensitivity as compared to the other two.¹⁵ Attonewton sensitivities have been reported with the FM technique using a combination of a fiber optic interferometer along with specially designed cantilevers in a vacuum environment.¹⁶

In this paper we report the measurement of the gradient of the Casimir force between an Au-coated plate and Au-coated sphere using the FM technique. The critical experimental and data analysis procedures for precision force measurements in the sphere-plate geometry using the dynamic mode are described. Our experimental results are in agreement with measurements performed using a micromachined oscillator.^{17,18} We compare our results with theoretical predictions on the basis of the Lifshitz theory. The data are shown to be consistent with the Lifshitz theory combined with the generalized plasma-like model^{5,12} over the entire measurement range from 235 to 1300 nm. In the region from 235 to 400 nm the Drude model approach (i.e., the Lifshitz theory using the tabulated optical data for Au extrapolated to lower frequencies by the Drude model) is excluded by the data at a 67% confidence level.

In Sec. 2 we discuss the experimental setup used and the measurement scheme. Section 3 contains information about the calibration procedures. Measurement results for the Casimir force and the comparison between experiment and theory are presented in Sec. 4. In Sec. 5 the reader will find our conclusions.

2. Experiment Setup and Measurement Scheme

The scheme of the experimental setup for FM measurements of the Casimir pressure is presented in Fig. 1. The detection system consists of a cantilever attached to the sphere, piezoelectric actuator, fiber interferometer, light source and PLL circuit.

Fig. 1. Layout of the vacuum FM-AFM setup used in precision dynamic measurements of the gradient of the Casimir force.

The vacuum system as shown consists of a chamber body, pumps, valves, gauges and different kinds of vacuum feed-throughs.

The main vacuum chamber is an 8" six-way stainless steel cross. The chamber is evacuated by a turbo-pump (Varian Inc., V-301) followed by an oil-free dry scroll pump (Leybold Vac., SC-15D). The main vacuum chamber is mounted on a 8" ion pump (Varian Inc., Diode). The chamber is separated from the turbo pump by gate valve (6" viton o-ring sealed gate with stainless steel construction) which can be closed to isolate the turbo pump from the chamber, which is necessary when only the ion pump is to be used. The turbo pump can achieve a vacuum down to 2×10^{-7} Torr. After use of the turbo, the ion pump can achieve a vacuum of 10^{-9} Torr. Note that the chamber is supported on a damped optical table, designed to have a large mass to reduce the mechanical noise. The low vacuum pressure (less than 10^{-3} Torr) is measured with a thermal-conductivity gauge (Duniway Stockroom Co., DST-531) and an ionization gauge (MDC Co., p/n 432026) connected to main vacuum chamber for measuring high vacuums of 10^{-9} Torr. For the electrical connections to elements inside the vacuum chamber we used D-type subminiature connectors offering UHV feed-through with 25 pins which are hermetically sealed and electrically insulated using glass ceramic bonding. For optical connections we used a home-built optical fiber feed-through made on a CF flange welded with a clean stainless tube. A cladding-stripped 1550 nm SM28 fiber is inserted through the steel tube and sealed using Varian vacuum Torr seal.

Two fiber optic interferometers were used. One interferometer monitors the cantilever oscillation. The second records the displacement of the Au plate mounted on the AFM piezo. For constructing the interferometer, we used a 1550 nm (Thorlabs Inc., 1550B-HP) single mode fiber which has extremely low bending loss and low splice loss compared to the standard SMF-28 1550nm fiber. A super luminescent

diode (Covega Co., SLD-1108) with a wavelength of 1550 nm, serves as the light source for the cantilever frequency measurement interferometer. The coherence length of the diode was 66 μm . A short coherence length is necessary to avoid noise from spurious interferences from unwanted reflections. An optical isolator with FC-APC connectors joins the diode to a 50/50 directional coupler. We used the typical fused-tapered bi-conic coupler at 1550 nm wavelength with return-loss of -55 dB relative to the input power. To reduce the signal power attenuation, we avoid bulkhead connectors which usually have ~ 0.3 dB power loss. A fiber coupled laser source (Thorlabs Inc., S1FC635) with a wavelength of 635 nm was used for Au plate displacement interferometer. In our experimental setup, we used a commercial anodized black xyz-stage to move the fiber end vertically above and close to the cantilever. But the anodizing (and lubricants) may significantly increase out gassing rates because of its porous structure. Therefore special treatments were done before placing the xyz-stage into the high vacuum chamber. The xyz-stage was first disassembled and the parts scrubbed with strong solution of detergent in an ultrasonic cleaner. They were then rinsed with very hot water. Next they were immersed in a 10% solution of sodium hydroxide (NaOH) saturated with common salt (NaCl) at 80°C. The parts were then polished in a conventional wheel polishing machine. They were then immersed in 10% solution of hydrochloric acid to obtain a bright finish. This was then followed by rinsing in DI water and reassembly of the xyz stage using powder-free disposable plastic gloves. Finally the xyz stage was rinsed with acetone followed by ethyl alcohol before insertion into the vacuum chamber.

The interferometer system used for measuring the frequency shift consists of the following parts. The output of the directional couplers is measured with InGaAs photodetectors. A low noise photodetector and amplifier system was fabricated for the cantilever interferometer. To avoid any potential errors or noise from the divider or balancing system, the InGaAs diode was coupled to an OPA627 low noise operational amplifier (very high input impedance $\sim 10^{13} \Omega$) as a trans-impedance amplifier. The output of the interference signal was fed into a band-pass filter (SRS Inc., SR650, with the range set between 1.20–1.85 kHz) cascaded by low (SRS Inc., SR965, 260 Hz) and high (SRS Inc., SR965, 1 kHz) pass filter to cut off unwanted frequency bands. The high-pass filter helped us to remove the noise in the excitation signal from FM controller and the low-pass filter removed $1/f$ and environmental noise. For frequency demodulation we use a PLL from Nano-Surf. Co. (Easy-PLL Plus). The PLL frequency demodulator system combines a controller module and detector module to measure the force gradient induced resonant frequency shift. The output of the high pass filter is fed to the PLL for the frequency demodulation. The output of the PLL is connected to a piezo (PI, PL033.31) which drives the cantilever at its resonant frequency with a constant amplitude. The oscillation amplitude of the cantilever was fixed at < 10 nm for all separations. The output of the low pass filter is used to form a closed loop PID controller and to maintain constant separation distance between the fiber end and the cantilever during

FM detection. The distance is kept constant using a piezo actuator with the PID controller.

The force gradient was measured between an Au coated sphere and Au plate. As in all previous experiments the sphere was attached to the cantilever. In difference with previous experiments, to achieve high resonance frequencies (and therefore low noise) hollow glass microspheres (3M Scotchlite) and stiffer rectangular Si cantilevers (μ masch, model CSC12/tipless/no Al) were used. The Si cantilevers are conductive, which is necessary for electrical contact to the sphere. The surfaces of the hollow glass spheres are smooth as they are made from liquid phase. We follow a special procedure for cleaning the spheres before attachment to the cantilever. The sphere cleaning procedure removes organic contaminants and debris from the surface. The first step in the cleaning process is to make a 10 ml solution of ethanol into which the spheres are deposited. This solution is thoroughly mixed in a vortex mixer for about 1 minute. Then, using a pipette, the alcohol is extracted from the solution, leaving the spheres in the vial. To remove strongly attached adsorbates and debris, a 10 ml solution of Hydrogen peroxide (H_2O_2) is added to the vial. A vortex mixer is again used to mix for about 2 min. The O_2 gas released from the solution removes any attached debris from the spheres. Next, the H_2O_2 is extracted with a pipette, and the spheres are immersed in 10 ml of ethanol again. To completely separate the debris from the spheres, we centrifuge the alcohol/sphere solution, for 10 minutes. The large radius, clean hollow spheres float to the top of the mixture and the debris sediment to the bottom. We use a pipette to remove the spheres at the top and place them in a pyrex petri dish to dry. The dried spheres are then mounted on the conductive Si cantilevers using Ag epoxy. The cantilever-sphere system and a 10 mm in diameter sapphire plate (Edmund Optics Inc.) are coated with Au in an oil-free thermal evaporator described elsewhere.¹⁹ The sphere cantilever system is rotated to get a uniform coating. In contrast to previous experiments only the tip of the cantilever with the sphere is coated with Au. Complete coating of the cantilever leads to large decrease in its oscillation Q-factor and loss of sensitivity. The thickness of the Au coating was measured to be 280 ± 1 nm using an AFM. The radius of the sphere was measured to be $R = 41.3 \pm 0.2 \mu\text{m}$ using a SEM.

The cantilever with the sphere was placed in a specially fabricated holder containing two piezos (first connected to the PID loop and the second to the PLL). The Au plate was mounted on top of a tube piezo from a commercial AFM (Veeco, AFM-3) capable of traveling a distance of $2.3 \mu\text{m}$. Ohmic contacts were made to the Au plate through a $1 \text{ k}\Omega$ resistor. To minimize electrical ground loops all the electrical ground connections were unified to the AFM ground. The calibration of the tube piezo was done using the second interferometer and is described in previous work.²⁰ To change the sphere plate distance using the tube piezo and avoid piezo drift and creep, a continuous 0.01 Hz triangular voltage signal was applied to the piezo actuator. The chamber was evacuated using the turbo pump to a pressure of 2×10^{-7} Torr. Next, the ion pump was turned on and the turbo pump was isolated

from the chamber by closing the 6" gate valve. When chamber reaches 3×10^{-8} Torr the experiment started.

The total force F_{tot} (electrostatic F_{el} and Casimir F_{Cas}) between the Au sphere and the plate was measured as a function of sphere-plate separation distance. To make measurements of the electrostatic force, the Au coated plate was connected to voltage supply (Agilent Inc., 33220A) operating with $1 \mu\text{V}$ resolution. 12 different voltages V_i in the range from -87.4 to 32.6 mV were applied to the Au plate, while the sphere remained grounded. Starting at the maximum separation, the plate was moved towards the sphere and the corresponding frequency shift was recorded at every 0.14 nm . The sphere-plate force acting on the cantilever causes a change in the resonance frequency given by

$$\Delta\omega = -\frac{\omega_0}{2k} \frac{\partial F_{\text{tot}}(a)}{\partial a}, \quad F_{\text{tot}}(a) = F_{\text{el}}(a) + F_{\text{Cas}}(a), \quad F_{\text{el}}(a) = X(a, R)(V_i - V_0)^2, \quad (1)$$

where $\omega_0 = 2\pi \times 1.604 \text{ kHz}$ and k are the resonance frequency and the spring constant of the cantilever, respectively, $X(a, R)$ is the known function of the absolute separation $a = z_{\text{piezo}} + z_0$, z_{piezo} is the plate movement due to the piezo and z_0 is the point of closest approach between the Au sphere and Au plate (note that in this experiment the separation at the closest approach is much larger than the separation on contact of the two surfaces). V_0 is the residual potential difference, which can be present due to different connections to ground or work functions of the sphere and plate material from adsorbates on the interacting surfaces (see Sec. 3). The frequency signal is measured by the FM detector. Unlike static measurements,^{6,8,12} in the dynamic measurement technique the total force gradient $\partial F_{\text{tot}}/\partial a$ is measured from the change in the resonant frequency $\Delta\omega$ given by

$$\Delta\omega = -\frac{\omega_0}{2k} \left(\frac{\partial F_{\text{el}}}{\partial a} + \frac{\partial F_{\text{Cas}}}{\partial a} \right) = -\beta(V_i - V_0)^2 - C \frac{\partial F_{\text{Cas}}}{\partial a}, \quad (2)$$

where $C \equiv C(k, \omega_0) = \omega_0/(2k)$, $\beta \equiv \beta(a, z_0, C, R) = C \partial X(a, R)/\partial a$. The first term is associated with the gradient of electrostatic force, corresponding to a constant voltage V_i applied to the plate (the sphere remains grounded). Equation (2) allows determination of all parameters of the setup by the application of different voltages to the plate. This is called electrostatic calibration. After the calibration is completed, the same Eq. (2) is used to find the gradient of the Casimir force from the measured frequency shift.

3. Calibration Procedure

The electrostatic force gradient and therefore the frequency shift (2) has a parabolic dependence on the voltage V_i applied to the plate. From Eq. (2), the maximum in the frequency shift corresponds to V_0 . The curvature of the parabola which includes the spatial dependence of the electrostatic force and the cantilever parameters is related to β and depends on z_0 and C . Thus, these parameters can be extracted

Fig. 2. (a) The drift in experimental curves for the frequency shift as a function of sphere-plate separation change for 8 repetitions of the same applied voltage to the plate. (b) The change in the plate position at one frequency shift signal value as a function of time.

from this dependence using χ^2 -fitting procedure. In order to test for systematic errors in the fitting parameters, the fitting is repeated at different distance ranges.

At first step, 12 different voltages V_i were applied to the plate and the corresponding frequency shift due to the total force gradient was measured. Then, we subtracted any drift of the frequency shift. For separations larger than $2\ \mu\text{m}$, the total force between the sphere and plate is below the instrumental sensitivity. At these separations, the noise is far greater than the signal and in the absence of systematic errors the signal should average to zero. The correction due to the drift in sphere-plate separation was next measured. The effect of the drift can be observed in Fig. 2(a), where 8 repeated measurements of the frequency shift signal $S_{\Delta\omega}$ as a function of the sphere-plate separation change Δz are shown for some applied voltage to the plate. Drift causes the separation to increase by around 1 nm in 700 s, where 100 s correspond to the time taken to make the one measurement [note that positional precision much better than 1 nm is achieved in this experiment as observed in Fig. 2(a)]. To calculate the drift rate, the change in position at one frequency shift signal is plotted as a function of time as shown in Fig. 2(b). This was repeated for 15 different signals and the average drift rate was found to be 0.002 nm/s. The separation distances in all measurements and between subsequent measurements were corrected for this drift rate.

After applying the drift correction the residual potential V_0 between the sphere and plate was found. The frequency shift signals at every 1 nm separation are found by interpolation (note that data acquisition is done every 0.14 nm). From the parabolic dependence of the electric force gradient shown in Eq. (2), V_0 can be identified at the position of the parabola maxima. The frequency shift signal was plotted as a function of the applied voltages V_i at every separation and the corresponding V_0 and curvature of the parabola β were found. This V_0 is shown in Fig. 3 as a function of separation distance. The mean value is $V_0 = -27.4\ \text{mV}$ and can be observed to be independent of separation distance.

The observed independence of V_0 on a should be considered in connection with the existence of patch potentials which arise due to work function inhomogeneities

Fig. 3. The residual electrostatic potential between Au-coated sphere and plate as a function of separation.

resulting from the different crystal orientations of the polycrystalline sample²¹ and chemical or physical adsorbates.²² One must make sure that patch potentials lead to only a negligibly small contribution to the total measured force. To address this problem, we considered an Au plate and a sphere with a stationary random distribution of the patch potentials and numerically simulated the electrostatic force for the cases of large and small patches. It was shown that in both cases the residual potential V_0 between the sphere and the plate depends heavily on the separation distance for patch sizes of order or larger than the separation distance. Keeping in mind that in our experiment $V_0 = \text{const}$ (see Fig. 3), this can be considered as an indirect confirmation of the fact that the interacting regions of the surfaces used are free from work function inhomogeneities which may have a pronounced effect on the values of the total force measured.

The next step was to determine the separation distance at closest approach z_0 and the coefficient C in Eq. (2). These parameters associated with the cantilever can be found from the dependence of the parabola curvature β on distance a . The corresponding theoretical expression for parabola curvature was fit to β as function of the separation distance. A least χ^2 -procedure was used in the fitting and the best values of z_0 and $C = \omega_0/(2k)$ were obtained. The fitting procedure was repeated by keeping the start point fixed at the closest separation, while the end point z_{end} counted from the closest separation was varied from 750 nm to 50 nm. In Fig. 4(a),

Fig. 4. The dependences of (a) the closest sphere-plate separation and (b) the coefficient C in Eq. (2) on the end point of the fit.

the z_0 so determined is shown as a function of the end point used in the fit. The values of z_0 are seen to be independent of the end point position indicating the absence of systematic errors resulting from z_{piezo} calibration, mechanical drift etc. in the experiment. Similarly value of the coefficient C , and, thus, k , can be also extracted by fitting the β -curve as a function of separation [see Fig. 4(b)]. The independence on separation again indicates the absence of systematic errors. The mean values obtained were $z_0 = 195.9 \pm 0.4 \text{ nm}$ and $C = 68.3 \pm 0.1 \text{ kHz m/N}$, respectively. After the determination of z_0 the absolute sphere plate separation can be found. Using C the frequency shift signal can be converted to force gradient values.

4. Measurement Results for the Casimir Force and Comparison Between Experiment and Theory

Now the values of all parameters in Eq. (2) are determined and the gradient of the Casimir force is found from the measured frequency shift

$$\frac{\partial F_{\text{Cas}}}{\partial a} = -\frac{\Delta\omega}{C} - \frac{\partial X}{\partial a}(V_i - V_0)^2. \quad (3)$$

The obtained mean values of the gradient of the Casimir force over 48 repetitions (4 measurement sets with 12 applied voltages each) are shown in Fig. 5(a,b) as crosses. The arms of the crosses indicate the total experimental errors in the measurement of separations (0.4 nm) and gradients of the Casimir force determined at a 67% confidence level. The error in the gradient is a combination of the separation-independent random error equal to 0.00011 pN/nm and the systematic error. The latter is determined by the resolution error, calibration error and the error in the gradient of electrostatic force subtracted in Eq. (3). For example, at $a = 235 \text{ nm}$ the total error in the gradient of the Casimir force is equal to 0.00067 pN/nm. It

Fig. 5. The gradients of the measured Casimir force as a function of separation are shown as crosses. The theoretical predictions from the Drude model approach are plotted as the grey curves. The predictions of the plasma model approach are plotted (a) by the black curve and (b) as the white curve in an inset.

decreases to 0.00053 pN/nm at $a = 455 \text{ nm}$ and remains constant at larger separations.

It is instructive to compare our experimental results with the Casimir pressures measured by means of a micromachined oscillator.^{17,18} To obtain the Casimir pressures, the gradient of the Casimir force should be divided by $-2\pi R$. Then we arrive to the important conclusion that the mean Casimir pressures obtained by us are in complete agreement with the mean pressures measured in Refs. 17, 18 in the limits of experimental errors. For example, at separations of 250 and 300 nm the magnitudes of our pressures are equal to $225.93 \pm 2.4 \text{ mPa}$ and $114.89 \pm 2.2 \text{ mPa}$, respectively, whereas the respective results of Refs. 17, 18 are $225.16 \pm 1.65 \text{ mPa}$ and $114.82 \pm 0.82 \text{ mPa}$ (note that all errors are indicated at a 67% confidence level).

The experimental results for the gradient of the Casimir force were compared with computations performed using the Lifshitz theory at room temperature combined with the proximity force approximation (PFA). Recently it was shown^{23,24} that the PFA is readily applicable in the sphere-plate geometry at small a/R and that the exact theory leads to even slightly smaller magnitudes of the gradient of the Casimir force than the PFA. The results of computations using the Drude model approach (i.e., the tabulated optical data²⁵ for Au extrapolated to lower frequencies by the imaginary part of the Drude model permittivity with the plasma frequency $\omega_p = 9.0 \text{ eV}$ and relaxation parameter $\gamma = 0.035 \text{ eV}$) are shown by the grey curves in Fig. 5(a,b). The computational results using the generalized plasma-like model with 6 oscillators^{5,12} are shown by the black curve in Fig. 5(a) and by the white curve in an inset to Fig. 5(b). The surface roughness was investigated with the AFM and taken into account using the multiplicative approach. Keeping in mind that the r.m.s. roughness on the sphere and the plate was measured to be of 2.0 and 1.8 nm, respectively, this approach at $a \geq 235 \text{ nm}$ is appropriate.⁵ The roughness correction contributes only 0.13% of the force gradient at the shortest separation.

As can be seen in Fig. 5(a,b), the Drude model approach is excluded by the data over the separation region from 235 to 400 nm. The plasma model approach is consistent with the data over the entire measurement range.

5. Conclusions

To conclude, we have measured the gradient of the Casimir force between an Au sphere and an Au plate using a dynamic AFM operated in the frequency modulation regime. Our results appear to be in complete agreement with the Casimir pressures measured previously by means of a micromachined oscillator. No anomalous behavior of sphere-plate residual potential was observed. The experimental data for the force gradient are found to be consistent with theoretical predictions using the generalized plasma-like model over the entire measurement range. Unique curvature of the force gradient-distance curve, as predicted by the plasma model, was verified. Our data exclude the theoretical prediction of the Drude model approach over the separation region from 235 to 400 nm at a 67% confidence level.

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