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Precision Casimir Force Measurements after *In Situ* Argon-Ion Beam Bombardment: Studying the Role of Electrostatic Patches

A Dissertation submitted in partial satisfaction of the requirements for the degree of

Doctor of Philosophy

in

Physics

by

Jun Xu

September 2017

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Precision Casimir Force Measurements after *In Situ* Argon-Ion Beam Bombardment: Studying the Role of Electrostatic Patches

by

Jun Xu

Doctor of Philosophy, Graduate Program in Physics
University of California, Riverside, September 2017
Dr. Umar Mohideen, Chairperson

The Casimir effect was first predicted by Hendrik Casimir in 1948. It was described as an attractive force between two neutral, parallel conducting plates placing in a vacuum. It is explained by the quantum field theory using the zero-point oscillations of quantized electromagnetic fields with the presence of boundaries. As a macroscopic phenomenon arises from the zero-point energy, it plays an important role in both fundamental physics and, micro- and nano-technology. The development of precision Casimir force measurements since 1997 allows a quantitative comparison between experiment and theory. Specifically, the comparison results help us to investigate the thermal Casimir effect and set constraints on the hypothetical non-Newtonian gravity.

In this work, we study the residual force induced by electrostatic patches in precision Casimir force measurements. We find that the residual force can be eliminated even prior to Ar ion cleaning. Compensation could be achieved only if the residual
potential for the two grounded surfaces did not vary with distance within the experimental error. This however happens for a small fraction of the measurements. For most surfaces the residual potential changed as a function of distance due to the electrostatic patches. The optimal Ar ion cleaning was found to be 500 V beam voltage, 80% focus voltage, 10 µA anode current, $2\times10^{-5}$ torr Argon pressure and 15 cm gun-sample separation. After 100 minutes Ar ion cleaning the residual potential decreased to $1.3 \pm 1.2$ mV. For cleaned surfaces the residual potential did not change as a function of distance allowing compensation of the electrostatic force. The Casimir force for cleaned surfaces matches the Lifshitz theory for Au where the energy loss from free electron scattering with zero point photons in the metal has been neglected.
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Chapter 1

Introduction
1.1 The Origin of the Casimir Effect

1.1.1 Prediction of the Casimir Effect

For two closely spaced neural polar or nonpolar atoms, the instantaneous dipole of one atom from quantum fluctuation will induce polarization in the other atom. The interaction between this temporary dipole and the induced dipole gives rise to an attractive force between two atoms, known as the van der Waals force. The electric field due to a dipole at distance $r$ away is proportional to $r^{-3}$, when $r$ is between a few angstroms and a few nanometers and the retardation effect is negligible. Thus the interaction energy between dipole and corresponding induced dipole is proportional to $r^{-6}$. In 1947, Casimir and Polder found that, when the separation gets large compared with the characteristic absorption wavelength, the finite speed of electric field needs to be considered. The influence of the retardation effect leads to the correction of interaction energy to $r^{-7}$ [2],

$$U = -\frac{23}{4\pi} \frac{\hbar c}{r} \frac{\alpha_1 \alpha_2}{r^7},$$

(1.1)

where $\alpha_1$ and $\alpha_2$ are the static polarizabilities. The dispersion force between two neutral atoms or a neutral atom and a perfectly conducting plane with a retardation effect is also called the Casimir-Polder force. One year later, in 1948, Casimir calculated the interaction energy between two parallel neutral perfectly conducting plates by studying the change of electromagnetic zero point energy and found the Casimir
effect [3]

\[ E = \frac{U}{A} = -\hbar c \frac{\pi^2}{720} \frac{1}{a^3}, \]  

(1.2)

where \( E \) is the interaction energy per unit area, \( U \) is the interaction energy, \( A \) is the area of the plates, \( a \) is the separation between two plates. It is understood that the presence of two metal plates allows only the virtual photons with certain wavelengths to exist between two plates and it results in a net energy difference with and without two plates.

1.1.2 Zero-point Energy and the Casimir Effect

Unlike the classical vacuum which is empty and contains no matter or energy, the QED vacuum is filled with the ground states of quantized electromagnetic fields. These quantized electromagnetic fields can be treated as harmonic oscillators. In their ground states, the harmonic oscillators keep moving about their equilibrium positions and have the nonzero lowest energies. This oscillation is also called as zero-point oscillation. The corresponding energy of all possible zero-point oscillations is the so-called zero-point energy. The QED vacuum contains infinite number of this harmonic oscillators leading to an infinite zero-point energy. Mathematically this energy emerges from the minimum energy determined by the Heisenberg’s uncertainty principle

\[ \Delta p \Delta x \geq \frac{\hbar}{2}. \]  

(1.3)
Consider the zero-point energy from all discrete energy levels of many quantum harmonic oscillators

\[ E = \sum_k \left( n_k + \frac{1}{2} \right) \hbar \omega_k \]  

(1.4)

where \( k \) is the wave factor, \( \omega_k \) is the angular frequency, and \( n_k = 0, 1, 2, \ldots \) is the energy level, and \( \hbar \) is the reduced Planck constant. For the ground state \( (n_k = 0) \), the energy is

\[ E = \sum_k \frac{1}{2} \hbar \omega_k. \]  

(1.5)

This is the zero-point energy predicted by quantum field theory.

In free vacuum, the half quanta energy in eqn (1.5) is obviously divergent. To avoid the problem of infinite energy, Casimir considered the configuration of two perfectly conducting parallel plates separated in a vacuum. With boundary conditions of two plates, some wavelengths are forbidden between two plates. It causes the zero-point energy with two plates to be less than the free vacuum energy. A net attractive force arises from this energy difference as illustrated in fig (1.1). Assume the length and width of two perfectly conducting parallel plates are both \( L \) and their separation is \( z \) as shown in fig (1.2).

The energy without the presence of two metal plates is equal to the energy of free
Figure 1.1: The presence of two parallel metal plates causes net attractive forces on both sides of the plates. (Adapted with permission from Emok (https://commons.wikimedia.org/wiki/File:Casimir_plates.svg), Casimir plates, https://creativecommons.org/licenses/by-sa/3.0/legalcode)
Figure 1.2: Two perfectly conducting parallel metal plates with length L and width L in vacuum form a narrow gap. The small separation between two plate is z. The wave factors of electromagnetic waves allowed in this narrow gap must be integer multiples of $\pi/z$, which causes a slight change of zero-point energy.
vacuum. It is

\[
U(d = \infty) = \frac{1}{2} \left( \sum_i \hbar \omega_i \right)_{d=\infty} = \hbar c \frac{L^2}{\pi^2} \int_0^\infty \int_0^\infty \sqrt{\left(k_z^2 + \chi^2\right)} \chi d\chi \left(\frac{z}{\pi} dk_z\right),
\]

(1.6)

where \(\omega = c\sqrt{k_x^2 + k_y^2 + k_z^2} = c\sqrt{\chi^2 + k_z^2}\), and \(k_z = \frac{n\pi}{z}\). With the presence of two parallel metal plate separated by distance \(z\), the energy will be

\[
U(d = z) = \frac{1}{2} \left( \sum_i \hbar \omega_i \right)_{d=z} = \hbar c \frac{L^2}{\pi^2} \sum_{\substack{n \geq 1}} \int_0^\infty \sqrt{\left(\left(\frac{n\pi}{z}\right)^2 + \chi^2\right)} \chi d\chi.
\]

(1.7)

To obtain a finite value from the subtraction of eqn (1.6) from eqn (1.7), a regulator \(e^{-t\omega}\) needs to be introduced for the \(\omega\) in both equations. The regulator vanishes when \(t \to 0\).

\[
\delta U(z) = U(d = z) - U(d = \infty)
\]

\[
= \lim_{t \to 0} \hbar c \frac{L^2}{\pi^2} \frac{\pi}{2} \left( \sum_{\substack{n \geq 1}} \int_0^\infty \sqrt{\left(\left(\frac{n\pi}{z}\right)^2 + \chi^2\right)} \chi e^{-tc\sqrt{\chi^2 + (\frac{n\pi}{z})^2}} d\chi \right) - \int_0^\infty \int_0^\infty \sqrt{\left(k_z^2 + \chi^2\right)} \chi e^{-tc\sqrt{\chi^2 + k_z^2}} d\chi \left(\frac{z}{\pi} dk_z\right)
\]

\[
= -\frac{\pi^2 \hbar c}{720 z^3} L^2
\]

(1.8)

The resulting Casimir force and Casimir pressure from this energy difference are

\[
F(z) = -\frac{\partial (\delta U(z))}{\partial z}
\]

\[
= -\frac{\pi^2 \hbar c}{240 z^4} L^2,
\]

(1.9)
\[ P(z) = -\frac{\pi^2 \hbar c}{240 z^4}. \]  

(1.10)

which only depends on the reduced Planck constant \( \hbar \), the speed of light \( c \), the separation distance \( z \), and the area of plate \( L^2 \). From this eqn (1.8), we can tell that the Casimir effect is a quantum phenomenon (\( \hbar \)) and also a relativistic effect (\( c \)). The negative sign means it is an attractive force for the geometry of two parallel plates. It is surprising that the force doesn’t depend on the charge of a single electron (\( e \)).

1.2 The Relation between the van der Waals Force and The Casimir Force

The attractive van der Waals force between two neutral atoms or molecules at the distance from a couple angstroms to a couple nanometers (larger than the dimensions of atoms or molecules and smaller than the characteristic absorption wavelength) is well known. It was first demonstrated by F. London [4, 5] in 1930 that the van der Waals force originates from the electrostatic interaction of two dipoles using second order perturbation theory. The Casimir-Polder force found in 1947 by H.B.G. Casimir and D. Polder was known as the van der Waals force with retardation effect. However, the Casimir force found one year later in eqn (1.9) is valid for all distance and won’t transform to non-relativistic van der Waals force at short separations. Using the van der Waals forces between individual microscopic bodies to calculate the similar force between macroscopic bodies will not be correct due to the non-additive field inter-
Figure 1.3: Illustration of van der Waals force, Casimir-Polder Force and Casimir Force. 

- **a**, A fluctuating dipole $P_1$ induces a fluctuating electromagnetic field, which induces a fluctuating dipole $P_2$ on a nearby particle, leading to van der Waals forces.

- **b**, when separation between particle gets large comparable to characteristic wavelength, the retardation effect plays an important role. If a third particle presents, multiple scattering leads to a breakdown of the pairwise force law.

- **c**, Casimir effect is a macroscopic effect between many quantum fluctuated dipoles. Adapted with permission from Macmillan Publishers Ltd: [Nature Photonics] (ref [6]), copyright (2011).

action of condensed matter. The presence of neighboring atoms to the interacting atoms will disturb the electromagnetic field through which the interaction is established. For sufficiently rarefied bodies, i.e., gases, it will not be practical to form a certain shape. It seems like the Casimir force is not related to the van der Waals force.

It wasn’t until 1956 that E.M. Lifshitz constructed a macroscopic theory to unify
the van der Waals force and the Casimir force [7] between arbitrary dissipative materials using fluctuation-dissipation theorem and proved that they have the same origin of quantum fluctuation. Since the interatomic distance is assumed to be smaller for the interaction distance, Lifshitz described the interaction through the fluctuating electromagnetic fields that are always present in the interior of any dissipative medium and extends beyond its boundaries. Lifshitz also pointed out that this does not vanish at zero temperature and it is associated with the zero-point electromagnetic fluctuations.

The Lifshitz theory starts from the Maxwell’s equation. The retardation effect is automatically taken into account for large distance compared to the characteristic absorption wavelength and is general for all materials. In this theory, material properties are presented by the frequency-dependent dielectric permittivity ($\epsilon(\omega)$), which is responsible for the reaction of material to external electromagnetic field. The van der Waals force and the Casimir force are just two limiting cases of the same phenomenon. At short distance, the effect of retardation is very small and can be neglected leading to the van der Waals force. At larger distance, the effect of relativistic retardation effect should be taken into account and leads to the Casimir force [8]. Since both the van der Waals force and the Casimir force arise from the field dispersion, they share the same name of dispersion force.

Now it is clear that eqn (1.9) will not transform to van der Waals force, because the assumption of ideal metal with infinite conductivity in Casimir’s original derivation.
The ideal metal performs like a perfect mirror and will reflect fluctuating electromagnetic waves of any frequency. For real material, the reflectivity is determined by frequency-dependent dielectric permittivity and magnetic permeability. Metals will be transparent to waves with frequency higher than the plasma frequency.

1.3 Application of the Casimir Effect

The Casimir effect is a multidisciplinary subject. As a quantum and relativistic phenomenon that arises from zero-point oscillations of quantized fields, the Casimir effect plays an important role in many fields, such as condensed matter physics, atomic and molecular physics, gravitation and cosmology, particle physics and mathematical physics [9]. In addition to theoretical aspects, the Casimir effect is important in micro-electromechanical systems (MEMS), nano-electromechanical systems (NEMS) and the semiconductor industry.

1.3.1 Application of the Casimir Effect in Fundamental Physics

In condensed matter physics, the Casimir effect leads to the investigation of multilayer thin films [10], critical systems [11], different boundary conditions [1, 12, 13], surface tension and latent heat [14], and effect of low temperature [15]. The Casimir force also depends on the different carrier concentrations [16, 17] and material properties [18].
In atomic and molecular physics, the Casimir effect improves our understanding of atom-atom and atom-wall interaction [2, 19]. Casimir-type effect is detected in the field of cavity quantum electrodynamics, where the spontaneous emission rates of atoms are subject to manipulation [20].

In gravitation and cosmology, the Casimir effect is involved in the problem of cosmological constant [21]. The non-trivial topology of space makes additional contributions to the cosmological constant. This cosmo-topological Casimir effect is investigated using zeta regularization in quantum field theory with boundary conditions [22].

In particle physics, the new light scalar particles predicted by many extensions to the Standard Model can solve the charge parity problem in strong interaction of quantum chromodynamics (QCD) [23]. The precision measurements of the Casimir force and van der Waals force can yield constraints on the hypothetical Yukawa-type interaction with nucleon and extra-dimensional physics [24–27].

In mathematical physics, to deal with the ultraviolet divergence in calculation of the Casimir effect, powerful regularization methods based on the zeta functions and the heat kernel expansion have been developed [28].
1.3.2 Application of the Casimir Effect in Micro- and Nano-technology

The Casimir effect dominates the interaction between macrobodies at separations from tens of nanometers to several micrometers. From eqn (1.9) we can know that, at separation of 10 nm, which is the scale of current computer microprocessors [29], the Casimir effect produces the equivalent of about 1 atmosphere of pressure. The Casimir effect between different micromachined components in integrated silicon chips has already been demonstrated by [30].

While the attractive Casimir force may cause an undesirable effect, like pull-in behavior [31], deflection, stiction [32], and adhesion [33] in micro- and nanoelectromechanical systems (MEMS and NEMS), a changed sign of the Casimir force from attractive to repulsive is possible by suitable choice of interacting materials [10] or by using non-conventional shapes of components [34] to build a virtually frictionless environment. It is also possible to exert control on its magnitude by optical modification of the charge carrier density with laser light [17] and with ultraviolet light [18]. Even the attractive Casimir force is found to have application to nonlinear Casimir oscillators in nanoscale position sensors [35] and space propulsion drive for interstellar flight [36].
1.4 Developments of Casimir Force Measurement

1.4.1 The First Casimir Force Measurement between Metallic Surfaces

The first experiment to measure the Casimir force between two parallel metal plates was reported by M.J. Sparnaay in 1958 [37]. The selection of metal plates instead of glass and quartz greatly reduced the impact of static charge and gel layer due to chemiabsorption. The experiment was based on a spring balance system at separations of 0.5 \( \mu \text{m} \) to 2 \( \mu \text{m} \). The attractive force was calculated by multiplication of the extension of the spring and the force constant of the balance system. The extension of the spring was measured through a capacitor formed between two plates with a distance resolution of 100 \( \text{Å} \). The force sensitivity of that balance system is \( 0.1 \sim 1 \times 10^{-8} \text{ N} \). Two plates were brought to contact at the start of experiment to minimize the potential difference on two plates. Three combinations of materials (aluminum-aluminum, chromium-chromium and chromium-steel) were used in this experiment. Attractive force was detected between chromium-chromium and chromium-steel and the force magnitude generally matched the prediction given by eqn (1.9) of two perfectly conducting plates with the error of around 100%. Only a repulsive force, though, was detected for aluminum-aluminum. The aluminum oxide layer was believed to be responsible for this repulsion force.

Sparnaay’s experiment qualitatively indicated the existence of Casimir effect. The
experimental error was too large to quantitatively compare the experiment result to theory due to the limitation of fabrication technique at that time and drawback of its experiment design. The large hysteresis from spring and knife edge contact made the determination of separation distance difficult. The surface roughness and dust with the size up to 0.2 µm contributed to the uncertainty of absolute separation at contact. The residual potential between two surfaces ranged from 5 to 25 mV. It drifted with time and was not compensated. Static charge might accumulate on the oxide layer on top of metal surfaces. The anharmonic oscillation of spring from surrounding mechanical noise greatly limited the force sensitivity of the system. The two plates were aligned parallel only by visual inspection. Regardless of these deficits, this experiment opened up the way for precision Casimir force measurement and pointed out the necessary improvements for any more accurate experiments in the future.

1.4.2 Modern Casimir Force Measurements

The first modern Casimir force measurement was performed by S.K. Lamoreaux in 1997 between an optical flat quartz with the diameter of 0.5 cm and a spherical lens with the radius of curvature of 11.3 ± 0.1 cm using a torsional pendulum [38]. Both surfaces were coated with 0.5 µm Cu and, on top of it, 0.5 µm Au. Based on the experience gained from older experiments, including Sparnaay’s experiment, the surfaces were in the form of a flat plate and a sphere to avoid the issue of non-parallelism in the conventional plate-plate configuration. The force for curved geometry was obtained
by the application of the proximity force approximation (PFA) [39].

A vacuum of \(10^{-4}\) torr was maintained to eliminate viscous effects. The plate was mounted on one end of a pendulum. The distance between the lens and plate was changed using the lead zirconate titanate (PZT) from 10 \(\mu\)m to 0.6 \(\mu\)m at 16 steps. The relative displacement of PZT was calibrated by a laser interferometer to 0.01 \(\mu\)m accuracy. The unbalance of pendulum due to attractive force between sphere and plate was detected by a phase-sensitive detector (PSD), which in turn provided a small correction voltage to the dual capacitor located on the other end of the pendulum to keep its angle fixed. A shockingly large residual potential \((V_0)\) of 430 mV was observed between the grounded lens and plate. To compensate this residual potential and calculate the closest separation \(a_0\) from the fitting of capacitance to its theoretical form, different voltages were applied between the lens and plate. The total force was fitted to this function:

\[
F^m(i) = F^T_c(a_i + a_0) + \frac{\beta}{a_i + a_0} + b,
\]

where \(F^m(i)\) is the measured force at the \(i\)th step, \(F^T_c(a_i + a_0)\) is the theoretical Casimir force, \(a_i\) is the displacement of PZT, \(a_0\) is the absolute lens-plate separation, \(\frac{\beta}{a_i + a_0}\) is the electrostatic force, \(\beta = \pi \epsilon_0 RV^2\), and \(b\) is a constant. Agreement between experiment and theory with thermal correction at the level of 5% was reported. The thermal Casimir effect was not observed in this experiment due to its limited accuracy.

Another independent demonstration of the Casimir effect with higher accuracy using the atomic force microscope (AFM) was conducted by U. Mohideen and A. Roy
in 1998 [1]. A standard AFM was used in this experiment as shown in fig (1.4). This experiment demonstrated the corrections of finite conductivity and surface roughness to the theory for the first time. The force measurement was performed at separations from 0.1 to 0.9 µm with the uncertainty of 1%. A good agreement of experiment and theory was found within experimental error.

The force measurements were based on Hooke’s law: \( F = k\delta z \), where \( k \) is the force constant and \( \delta z \) is the small deflection of the free end of the cantilever. The electrostatic force between a charged sphere and plate is given as [40]

\[
F = 2\pi\varepsilon_0(V_1 - V_2)^2 \sum_{n=1}^{\infty} \text{csch} n\alpha (\coth \alpha - n \coth n\alpha).
\]

Here \( V_1 \) is the applied voltage on the plate and \( V_2 \) is the residual potential between the grounded sphere and plate. \( \alpha \) is defined as \( \cosh^{-1}(1 + d/R) \), where \( R \) is the radius of the sphere and \( d \) is the separation distance between the sphere and the plate. The residual potential \( V_2 \) was measured as 29 mV. The absolute distance and spring constant were calculated using Hooke’s law and eqn (1.4.2). A root mean square of average deviation of 1.6 pN between theory and experiment was achieved, which corresponds to a 1% deviation at the smallest separation.

Since then, many other experiments have been conducted to measure the Casimir effect, like the Casimir force gradient measurement [41], the lateral Casimir force measurement [42,43], the Casimir force measurements using micro-mechanical torsion oscillator (MTO) [12], the thermal Casimir force measurement [44], and the Casimir
Figure 1.4: Schematic diagram of AFM setup for Casimir force measurement. The force probe had a polystyrene sphere attached to the free end of a tipless micro-cantilever. Both sphere and plate were coated with a metal film. The deflection of cantilever was detected by the PSD measuring the position of the laser beam bouncing off the back side of cantilever. Voltages applied to the piezo resulted in the displacement of the plate corresponding to the sphere. The experiment was performed at the vacuum pressure of 50 mTorr and at the room temperature. Adapted with permission from American Physical Society (ref [1]), copyright (1998).
force gradient measurement at low temperature [15].

1.5 Motivation of Research

1.5.1 Key Factors in Precision Casimir Force Measurements

The purpose of Casimir force experiment is to measure the Casimir force (or force gradient) as a function of separation, $F_c(z)$, and compare it to the theory. There are several key factors in any precision Casimir force measurement:

1. A high force sensitivity measurement system,

2. Low theoretical uncertainty due to material properties,

3. Precise and reproducible measurements of the separation between two objects,

4. Accurate and independent measurements of the electrostatic force.

In Casimir force measurement, a high force sensitivity system usually means probe with high quality factor (Q), high signal-to-noise ratio (SNR), small bandwidth, high vacuum, low mechanical, optical and electrical noise, stable temperature, small force constant, etc.

The theoretical uncertainty may come from a lack of the complete characterization of the optical properties, theoretical models applied, and application of approximations and assumptions. Also geometry information about two objects including
radius, roughness, and parallelism may lead to uncertainties in the calculation of theoretical Casimir force.

The measurement of separation between two surfaces is usually split into two parts: the relative displacement and the closest distance. Due to the surface roughness and spike, which is usually at the order of nanometers, two surfaces get into contact with each other at a nonzero separation. This makes the direct measurement of the closest distance difficult. In most precise Casimir force measurements, the closest distance is calculated as a parameter in the fitting of the capacitance between two objects to its theoretical form. The theoretical distance dependence of the electrostatic force between the sphere-plate configuration can be derived using the image charge method [40] with the assumptions of two equipotential and perfectly smooth surfaces. The movement of plate is usually controlled by applying voltages to piezoelectrics. Real-time calibration of piezo movement can be performed with laser based interferometer with a resolution of sub-nanometer.

For separations in the range of sub-micron and micron meters, the electrostatic force can be at the same order of the Casimir force, or even larger, depending on the potential difference between two objects. The electrostatic force needs to be subtracted from the measured total force to achieve the Casimir force. Moreover, by making fittings of the electrostatic force to its theoretical equation, some important parameters, including the closest distance and the force constant of the probe, are determined. So a proper measurement of the electrostatic force is vital to the accuracy
of the Casimir force measurement.

1.5.2 the Residual Potential and Electrostatic Patches in the Casimir Force Measurements

A residual potential ranging from ten millivolts to hundreds of millivolts between two grounded metallic surfaces in Casimir force measurements is usually observed [1, 3, 15, 38, 41, 45]. Similar effects were found in a measurement on a pair of metallic plates employed in the Laser Interferometer Gravitational Wave Observatory (LIGO) project [46]. It may come from several sources:

- the potential difference on the metallic contacts along the electric circuit between two surfaces,
- the different work functions of different crystallographic surface orientations,
- the surface oxide layer, contamination, and adsorbates.

By applying the voltage with the same magnitude and an opposite sign to the residual potential, the long range electrostatic force can be compensated. Since both the electrostatic force and the Casimir force are attractive, the compensation of electrostatic force leads to the minimum force at certain separation. This minimum force is regarded as the Casimir force.

The important assumption here is that all electrostatic force can be compensated by applying some DC voltage to one surface while grounding the other one. This
assumption is questionable [47–50], because the surface of any real conductor is not equipotential. This spatial variations in surface potential create small or large electrostatic patches and the potential difference between different electrostatic patches result in dipole-dipole interaction between two surfaces.

When two bodies of different work functions are brought into contact, the chemical potential (or Fermi level) difference will drive electrons from one body to the other until the chemical potentials of both sides equalize [51]. This transfer of electrons causes a charge separation and forms a net dipole distribution at the interface. The potential difference arises from this dipole is equal to the work function difference of two bodies. The length scale of this patch effect is expected to be the typical size of surface crystallites.

The dipoles induced by the metallic contacts along the electric circuit are usually far from the interaction area between two surfaces. Thus, they will cause the residual potential, but no electrostatic patches and can be completely compensated by applying DC voltage.

However, the varying work functions of different crystallographic surface orientations will create electrostatic patches on two surfaces. If the crystallites of different orientations are randomly distributed and their sizes are much smaller than the interaction area and the closest separation between two surfaces, the residual potential and the force induced by small electrostatic patches should be close to zero.

The surface oxide layer, contaminations and adsorbates create a surface dipole
layer on two surfaces and may cause both large residual potential and electrostatic patches. Use of chemically unreactive noble metals, like Au and Cu, can avoid the problem of oxide layer films. Surface contamination comes from dust, strains, human skin oils, and cleaning solvent residues. Careful precleaning procedure and ultraviolet-ozone (UV/Ozone) treatment can remove a variety of contaminants [52]. The adsorption of atoms, ions, and molecules to a metal surface can be classified as physisorption and chemisorption. Chemisorption creates a chemical covalent or ionic bonding and is stronger than the physisorption. In ambient environment, it is long known that a 'gel' adsorption layer containing water and hydrocarbons can form on material surfaces. The application of noble metals can reduce the problem of chemisorption. Physisorption is a result of the van der Waals force and can not be avoided. It reported that an Au surface was covered by 2∼3 monolayers of adsorbates after baking [53]. Even in ultrahigh vacuum (UHV), an adsorption film can grow on metal surface [54]. The adsorption films create a surface dipole layer on top of metal surface, which may alter both the residual potential and the electrostatic patches.

Depending on their sizes and potential variations, the electrostatic patches may result in residual force that can not be compensated and cause systematic errors in the precision Casimir force measurements. The purpose of the present research is to better understand the origin of residual potential and the role of electrostatic patches in the precision Casimir force measurements using in situ Argon-ion beam bombardment.
1.6 Structure of the Dissertation

In chapter 2, the Lifshitz theory for both van der Waals force and Casimir force will be introduced. For real metals, the frequency-dependent dielectric permittivity function and Casimir energy corrections due to finite conductivity, non-zero temperature, and surface roughness will be discussed. At the end of this chapter, the Proximity Force Approximation for the sphere-plate configuration will be presented.

Our measurement techniques are based on AFM. So, in chapter 3, we will review the operation principle of commercial AFM and its application for imaging and force measurements. It is followed by the calibration method for piezo and force probe. Then, the application of AFM in biophysics will be discussed.

In chapter 4, our experimental setup, including the vacuum system, fiber-optic interferometer, positioner and piezo actuators, frequency modulation techniques, and the Argon-ion gun will be presented in details. The experimental method for cantilever modification will be introduced.

Chapter 5 presents our experimental procedures to determine Casimir pressure and experiment results. The measurements of frequency shift, calibration of piezo movement and drift, and measurements of long range electrostatic force will be discussed. The measured residual force induced by electrostatic patches will be compared to its theoretical expectation. The relation between distance dependence of the residual potential and the residual force will be studied. The role of electrostatic patches on dirty surfaces and clean surfaces will be discussed with Argon-ion cleaning.
Chapter 2

The Casimir Force between Real Materials

2.1 The Lifshitz Theory between Plane Dielectrics

An ideal metal has infinity conductivity and zero skin depth. It can reflect electromagnetic fields of all frequencies with 100% reflectivity. However, for any real material, it becomes transparent at high enough frequency. Thus, the electrical, optical and mechanical properties of real materials need to be considered. So the result of eqn (1.9) is limited to ideal metal and can not be applied to any real material. A unified theory of both the Van der Waals force and the Casimir force between separated planar parallel dielectrics in thermal equilibrium was developed by Lifshitz [7, 55, 56] in 1956. There many different derivations of the Lifshitz formula for the Casimir force
Figure 2.1: Two identical parallel dielectric semispaces separated by a vacuum gap of width $a$. The frequency-dependent dielectric permittivity of two homogeneous semispaces are both $\varepsilon(\omega)$. The spatial dispersion is neglected. The dielectric permittivity of vacuum is one.

The derivation given below was first formulated by van Kampen [57] and later elaborated by others [58–62].

### 2.1.1 The Lifshitz Formula at Zero Temperature

First, consider the configuration of two identical parallel dielectric semispaces separated by a vacuum gap of width $a$ as shown in fig (2.1). The Maxwell equations without charge or current for nonmagnetic materials are:
\[ \nabla \cdot \mathbf{D}(t, r) = 0, \]
\[ \nabla \times \mathbf{E}(t, r) + \frac{1}{c} \frac{\partial \mathbf{B}(t, r)}{\partial t} = 0, \]
\[ \nabla \times \mathbf{B}(t, r) - \frac{1}{c} \frac{\partial \mathbf{D}(t, r)}{\partial t} = 0, \]
\[ \nabla \cdot \mathbf{B}(t, r) = 0, \quad (2.1) \]

where \( \mathbf{D} \) is the electric displacement, \( \mathbf{E} \) is the electric field, and \( \mathbf{B} \) is the magnetic induction. Both the electromagnetic field inside material (\(|z| \geq a/2\)) and outside (\(|z| < a/2\)) are the products of Maxwell’s equations (2.1). At the interface, the continuity boundary conditions of classical electromagnetic waves should be satisfied:

\[ E_{1t}(t, r) = E_{2t}(t, r), \quad D_{1n}(t, r) = D_{2n}(t, r), \]
\[ B_{1n}(t, r) = B_{2n}(t, r), \quad B_{1t}(t, r) = B_{2t}(t, r), \quad (2.2) \]

where \( n \) means the normal component to the boundary plane directed inside the dielectric and \( t \) in subscript refers to the tangential component in the (x,y) plane. The \( t \) as a variable is the time and \( r \) is the position. The dielectric displacement in frequency space is given by

\[ \mathbf{D}(\omega, r) = \varepsilon(\omega, r) \mathbf{E}(\omega, r). \quad (2.3) \]

The frequency-dependent dielectric permittivity in our configuration is

\[ \varepsilon(\omega, r) = \begin{cases} \varepsilon(\omega), & |z| \geq a/2, \\ 1, & |z| < a/2. \end{cases} \]

Consider a monochromatic electromagnetic field

\[ \mathbf{E}(t, r) = \mathbf{E}(r)e^{-i\omega t}, \quad \mathbf{B}(t, r) = \mathbf{B}(r)e^{-i\omega t}. \quad (2.4) \]
The electric displacement in a dielectric material will be

\[ D(r) = \varepsilon(\omega)E(r). \]  

(2.5)

Substitute eqn (2.4) and eqn (2.5) into eqn (2.1), the Maxwell’s equations take this form

\[
\begin{align*}
\nabla \cdot E(r) &= 0, \\
\nabla \times E(r) - i\frac{\omega}{c}B(r) &= 0, \\
\nabla \times B(r) + i\varepsilon(\omega)\frac{\omega}{c}E(r) &= 0, \\
\n\nabla \cdot B(r) &= 0.
\end{align*}
\]  

(2.6)

The second order equations can be derived as

\[
\begin{align*}
\nabla^2 E(r) + \varepsilon(\omega)\frac{\omega^2}{c^2}E(r) &= 0, \\
\nabla^2 B(r) + \varepsilon(\omega)\frac{\omega^2}{c^2}B(r) &= 0.
\end{align*}
\]  

(2.7)

The vacuum energy of the electromagnetic fields in thermal equilibrium confined in the vacuum gap shown in fig (2.1) at zero temperature is

\[
U(a) = \frac{\hbar}{4\pi} \int_0^\infty k_\perp dk_\perp \sum_n \left( \omega_{k_\perp,n}^{\text{TM}} + \omega_{k_\perp,n}^{\text{TE}} \right) S.
\]  

(2.8)

Here \( k_\perp = \sqrt{k_x^2 + k_y^2} \). \( \omega_{k_\perp,n}^{\text{TM,TE}} \) are photon eigenfrequencies of transverse magnetic (TM) mode and transverse electric (TE) mode, which can be solved from eqn (2.7). \( S \) is the interaction area.

This vacuum energy given by eqn (2.8) is infinite. The finite Casimir energy per unit area of the boundary planes is obtained by the subtraction of the free space
vacuum energy from $U(d = a)$ as Casimir did in his first derivation for ideal metals [3]:

$$E(a) = \frac{U(d = a)}{S} - \frac{U(d \to \infty)}{S}$$

$$= \frac{\hbar}{4\pi^2} \int_0^\infty k_\perp dk_\perp \int_0^\infty d\xi \left\{ \ln[1 - r_{TM}^2(i\xi, k_\perp)e^{-2qa}] + \ln[1 - r_{TE}^2(i\xi, k_\perp)e^{-2qa}] \right\},$$

(2.9)

where

$$\xi = -i\omega,$$  

(2.10)

$$q^2 = q^2(i\xi, k_\perp) = k_\perp^2 + \frac{\xi^2}{c^2},$$  

(2.11)

$$k^2 = k^2(i\xi, k_\perp) = k_\perp^2 + \frac{\varepsilon(i\xi)}{c^2} \frac{\xi^2}{c^2}. $$

(2.12)

Here $\xi$ is the imaginary frequency. The Casimir energy here is achieved by integrating contributions for all imaginary frequencies instead of all real frequencies. They are mathematically equivalent. At imaginary frequencies, the integral is smooth and exponentially decaying, while it is oscillatory at real frequencies [6]. This makes the presentation of eqn (2.9) more convenient for numerical computations [9]. $r_{TE}(i\xi, k_\perp)$ and $r_{TM}(i\xi, k_\perp)$ are the Fresnel reflection coefficients for TM and TE waves. They take this form:

$$r_{TM}(i\xi, k_\perp) = \frac{\varepsilon(i\xi)q(i\xi, k_\perp) - k(i\xi, k_\perp)}{\varepsilon(i\xi)q(i\xi, k_\perp) + k(i\xi, k_\perp)},$$

(2.13)

$$r_{TE}(i\xi, k_\perp) = \frac{q(i\xi, k_\perp) - k(i\xi, k_\perp)}{q(i\xi, k_\perp) + k(i\xi, k_\perp)}. $$

(2.14)

They coincide with the reflections coefficient for real electromagnetic waves in vacuum.

Note that the vacuum energy here arises from imaginary photons rather than real
ones. Thus, $k_\perp$ and $\xi$ are two independent variables.

The Casimir pressure can be obtained by taking derivative of eqn (2.9):

$$P(a) = -\frac{\hbar}{2\pi^2} \int_0^\infty k_\perp dk_\perp \int_0^\infty d\xi q \left\{ \left[ r_{TM}^{-2}(i\xi, k_\perp) e^{2qa} - 1 \right]^{-1} \right. 
+ \left[ r_{TE}^{-2}(i\xi, k_\perp) e^{2qa} - 1 \right]^{-1} \}. \quad (2.15)$$

At small separation limit of $a \ll \lambda_0$, where $\lambda_0$ is characteristic absorption wavelength, the Casimir pressure in eqn (2.15) is

$$P(a) = -H \frac{\lambda_0}{6\pi a^3}, \quad (2.16)$$

$$H = \frac{3\hbar}{8\pi} \int_0^\infty d\xi \int_0^\infty y^2 dy \left\{ \left[ \frac{\varepsilon(i\xi) + 1}{\varepsilon(i\xi) - 1} \right]^2 e^y - 1 \right\}^{-1}. \quad (2.17)$$

Here $y = 2aq$ and $H$ is the Hamaker constant. This is the non-retarded Van der Waals pressure in the configuration of two semispaces. The speed of light, $c$, is not involved.

At large separation limit of $a \gg \lambda_0$, the Casimir pressure is

$$P(a) = -\frac{\pi^2 \hbar c}{240a^4} \Psi(\varepsilon_0), \quad (2.18)$$

$$\Psi(\varepsilon_0) = \frac{15}{2\pi^4} \int_0^\infty d\zeta \int_\zeta^\infty y^2 dy \left\{ \left[ \frac{\varepsilon_0 y + \sqrt{y^2 + \zeta^2(\varepsilon_0 - 1)}}{\varepsilon_0 y - \sqrt{y^2 + \zeta^2(\varepsilon_0 - 1)}} \right]^2 e^y - 1 \right\}^{-1} 
+ \left[ \left( \frac{y + \sqrt{y^2 + \zeta^2(\varepsilon_0 - 1)}}{y - \sqrt{y^2 + \zeta^2(\varepsilon_0 - 1)}} \right)^2 e^y - 1 \right\}^{-1}. \quad (2.19)$$

where $\zeta = \xi/\omega_c$. The pressure here includes the speed of light and the retardation effect. The distance dependence is $a^{-4}$, instead of $a^{-3}$. For ideal metals, $\varepsilon_0 \rightarrow \infty$ and $\Psi(\varepsilon_0) \rightarrow 0$, the pressure coincides with the pressure between ideal metals given by eqn (1.10).
2.1.2 The Lifshitz Formula at Nonzero Temperature

At zero temperature, the Casimir force results from the zero-point energy of electromagnetic field modes in the presence of boundaries. At finite temperature, both thermal fluctuations and quantum fluctuations contribute to the Casimir effect.

Consider the same configuration in fig (2.1) at finite temperature in thermal equilibrium with the environment. The frequency-dependent dielectric permittivity of a dielectric material can be expressed as

\[
\varepsilon(\omega) = 1 + \sum_{j=1}^{K} \frac{g_i}{\omega_j^2 - \omega^2 - i\gamma_j \omega},
\]

(2.20)

where the \(\omega_j \neq 0\) are the oscillator frequencies of electrons, the \(g_i\) are the oscillator strengths, the \(\gamma_j\) are the damping parameters, and \(K\) is the number of oscillators.

At nonzero temperature, the free energy instead of electromagnetic energy should be considered. By applying the dielectric permittivity in eqn (2.20), the Casimir free energy is given by

\[
\mathcal{F}(a, T) = \frac{k_B T}{2\pi} \sum_{l=0}^{\infty} \int_{0}^{\infty} k_{\perp} dk_{\perp} \left\{ \ln\left[1 - r_{TM}^2(i\xi_l, k_{\perp})e^{-2qa}\right]
+ \ln\left[1 - r_{TE}^2(i\xi_l, k_{\perp})e^{-2qa}\right] \right\},
\]

(2.21)

where \(l = 0, \pm 1, \pm 2, \ldots\). The prime indicates a factor of 1/2 to be multiplied for \(l = 0\). The \(\xi_l\) are the discrete Matsubara frequencies:

\[
\xi_l = 2\pi \frac{k_B T}{\hbar} l.
\]

(2.22)
Notice that the difference between eqn (2.9) and eqn (2.21) is the substitution:

\[
\frac{\hbar}{2\pi} \int_0^\infty d\xi \rightarrow k_B T \sum_{l=0}^\infty \tag{2.23}
\]

Using the same substitution, the Casimir pressure at nonzero temperature can be obtained from eqn (2.15):

\[
P(a, T) = -\frac{k_B T}{\pi} \sum_{l=0}^\infty q_l k_{\perp} d k_{\perp} \left\{ \left[ r_{TM}^{-2}(i\xi, k_{\perp}) e^{2aq_l} - 1 \right]^{-1} + \left[ r_{TE}^{-2}(i\xi, k_{\perp}) e^{2aq_l} - 1 \right]^{-1} \right\}. \tag{2.24}
\]

The reflection coefficients at the Matsubara frequencies are

\[
r_{TM}(i\xi_l, k_{\perp}) = \frac{\varepsilon_l q_l - k_l}{\varepsilon_l q_l + k_l}, \quad r_{TE}(i\xi_l, k_{\perp}) = \frac{q_l - k_l}{q_l + k_l}, \tag{2.25}
\]

where

\[
q_l^2 = k_{\perp}^2 + \xi_l^2 c^2, \quad k_l^2 = k_{\perp}^2 + \varepsilon_l \xi_l^2 c^2, \quad \varepsilon_l = \varepsilon(i\xi_l). \tag{2.26}
\]

## 2.2 The Lifshitz Theory between Real Metals

### 2.2.1 The Dielectric Permittivity of Metals

The dielectric permittivity given by eqn (2.20) for nonpolar dielectric materials only includes the driving, damping and restoring forces on core electrons. The number of conduction electrons in metals is usually much less than core electrons. But the core electrons are tightly attached to the heavy metallic ions and the conduction electrons
are non-localized and free to move around. Therefore the conduction electrons are more important for the electrical conductivity of metals.

When the frequency of electromagnetic field is relatively high, the free electron model is a good approximation. The second-order wave equation for electric field takes this form,

\[-\nabla^2 E = \frac{\omega^2}{c^2} (1 - \frac{\omega_p^2}{\omega^2}) E,\]  \hspace{1cm} (2.27)

where \(\omega_p\) is the plasma frequency. It is given by

\[\omega_p = \sqrt{\frac{4\pi n_e e^2}{m^*}},\]  \hspace{1cm} (2.28)

where \(n_e\) is the number density of conduction electrons, \(m^*\) the effective mass of electrons. When the frequency of electromagnetic waves is higher than plasma frequency, the reflection coefficients of metals rapidly decrease and metals become transparent.

The dielectric permittivity for plasma model is

\[\varepsilon_p(\omega) = 1 - \frac{\omega_p^2}{\omega^2}.\]  \hspace{1cm} (2.29)

It provides a good approximation for distances larger than the plasma wavelength \(\lambda_p\) [64]. For Au, \(\lambda_p\) is 137 nm [65].

When the frequency of electromagnetic field falls in the region from quasistatic frequencies to infrared frequencies, the relaxation processes of electrons become important. The Drude model of dielectric permittivity derived from classical electron
gas is applicable for this region,

\[ \varepsilon_D(\omega) = 1 - \frac{\omega_p^2}{\omega(\omega + i\gamma)}, \]  

(2.30)

where \( \gamma \) is the relaxation frequency that reflects the damping effect. The relaxation frequency is related to the static electrical conductivity by

\[ \sigma_0 = \frac{\omega_p^2}{4\pi\gamma}. \]  

(2.31)

When \( \omega \gg \gamma \), \( i\gamma \) is negligible to \( \omega \) and dielectric permittivity of the Drude model in eqn (2.30) transforms to the Plasma model in eqn (2.29).

However the application of the Drude model to the Lifshitz formula leads to a negative Casimir entropy at zero temperature for perfect crystal lattices and violates the third law of thermodynamics [66–68]. This is because the relaxation term in the Drude model results in a drift current of conduction electrons at nonzero temperature and dissipation of energy due to Joule heating, which is inconsistent with the thermal equilibrium assumption in Lifshitz theory. The displacement current in the Plasma model will not cause Joule heating and therefore will not violate the Nernst heat theorem.

Compared to the dielectric permittivity of dielectric materials in eqn (2.20), both the Plasma model and Drude model do not take into account the interband transitions of core electrons.

One approach to solve this problem is using tabulated optical data for complex index of refraction \( n(\omega) = n_1(\omega) + in_2(\omega) \) [65] to calculate the dielectric permittivity.
along the imaginary frequency axis \(\varepsilon(i\xi)\) from zero to infinity. First the imaginary part of the dielectric permittivity can be calculated using \(n_1(\omega)\) and \(n_2(\omega)\):

\[
\text{Im}\ \varepsilon_\omega = 2n_1(\omega)n_2(\omega).
\]  

(2.32)

Then apply the Kramers-Kronig relation to obtain the dielectric permittivity along the imaginary frequency axis:

\[
\varepsilon(i\xi) = 1 + \frac{2}{\pi} \int_0^\infty \frac{\omega \text{Im}\varepsilon(\omega)}{\omega^2 + \xi^2} d\omega.
\]  

(2.33)

The tabulated data were measured from 0.125 eV to \(10^4\) eV [65]. The most important contribution to the Lifshitz formula comes at \(2a\gamma \sim 1\), where the characteristic frequency of the Casimir effect is \(\omega_c = c/2a\). At the separation of sub-micron meters, the character frequency is at the order of 0.1 eV for Au, the usual metal applied for Casimir force measurements. To compute highly accurate values of dielectric permittivity, the frequencies ranging from \(10^{-3}\omega_c\) to \(10\omega_c\) are needed. To obtain the dielectric permittivity at frequencies lower than 0.125 eV, it is necessary to extrapolate the tabulated optical data [69]. The Drude model has the \(\omega^{-1}\) dependence at low frequency as expected for metals. The imaginary part of the Drude dielectric permittivity according to eqn (2.30) is

\[
\text{Im}\varepsilon_D(\omega) = \frac{\omega_p^2\gamma}{\omega(\omega^2 + \gamma^2)},
\]  

(2.34)

where \(\omega_p = 9.0\) eV and \(\gamma = 0.035\) eV for Au.

Another approach to obtain the dielectric permittivity at both short and long separation distances and is consistent with thermodynamics is the generalized plasma-
like model [70]. It takes into account the interband transitions of core electrons in addition to the plasma model and neglects the energy dissipation caused by relaxation process. Its dielectric permittivity is

$$\varepsilon_{gp}(i\xi) = 1 + \frac{\omega_p^2}{\xi^2} + \sum_{j=1}^{K} \frac{g_j}{\omega_j^2 + \xi^2 + \gamma_j \xi},$$

(2.35)

where $$\omega_j$$, $$\gamma_j$$ and $$g_j$$ are parameters determined from a fit to the available optical data. $$K$$ is the number of oscillators used for the fitting.

### 2.2.2 Finite Conductivity Correction of Real Metals

In contrast to dielectric materials, which have zero conductivity at zero temperature, and ideal metals, which have infinite conductivity at any temperature, real metals have finite conductivity at zero temperature due to the presence of conduction electrons. The Casimir energy and pressure of ideal metals with infinite conductivity between two parallel plates are given by eqn (2.9) and eqn (2.15). The finite conductivity of real metals leads to a nonzero skin depth, which can be described with dielectric permittivity functions. Using the same way of applying dielectric permittivity to reflection coefficients of dielectric materials, the effect of finite conductivity of real metals can be investigated with Lifshitz theory.

The simplest configuration of two planar parallel surfaces in fig (2.1) is again considered, except that two plates thicker than the skin depth of metals instead of semispaces are necessary. The spatial dispersion of metals is again neglected, which means the dielectric permittivity only depends on the frequency.
For Casimir force measurement at the separation distances from 0.1 \( \mu m \) to 1 \( \mu m \), the main contributing frequencies close to the characteristic frequency lie in the region of visible light and infrared optics, where the Plasma model is a good approximation. The thermal Casimir effect is no more than a few percent below 1 \( \mu m \). By substituting the dielectric permittivity of the Plasma mode into the reflection coefficients eqn (2.13, 2.14) and the Lifshitz eqn (2.9) and expanding it up to the fourth order of \( \delta_0/a \), the Casimir energy with finite conductivity correction is given by [66]

\[
E_{p}(a) = -\frac{\pi^2 \hbar c}{720a^3} \left[ 1 - 4\frac{\delta_0}{a} + \frac{72}{5} \left( \frac{\delta_0}{a} \right)^2 
\right.
\left.
- \frac{320}{7} \left( 1 - \frac{\pi^2}{210} \right) \left( \frac{\delta_0}{a} \right)^3 + \frac{400}{3} \left( 1 - \frac{163\pi^2}{7350} \right) \left( \frac{\delta_0}{a} \right)^4 \right],
\] 

(2.36)

Here \( \delta_0 \) is the skin depth due to finite conductivity. It takes the form of

\[
\delta_0 = \frac{\lambda_p}{2\pi},
\]

(2.37)

where \( \lambda_p \) is the plasma wavelength. For Au, \( \lambda_p \) is 137 nm. Thus,

\[
\frac{\delta_0}{a} \frac{\lambda_p}{2\pi a} \ll 1
\]

(2.38)

for the separations between 0.1 \( \mu m \) and 1 \( \mu m \).

The corresponding Casimir pressure with finite conductivity correction is

\[
P_{p}(a) = -\frac{\pi^2 \hbar c}{240a^4} \left[ 1 - \frac{16}{3} \frac{\delta_0}{a} + 24 \left( \frac{\delta_0}{a} \right)^2 
\right.
\left.
- \frac{640}{7} \left( 1 - \frac{\pi^2}{210} \right) \left( \frac{\delta_0}{a} \right)^3 + \frac{2800}{9} \left( 1 - \frac{163\pi^2}{7350} \right) \left( \frac{\delta_0}{a} \right)^4 \right].
\]

(2.39)
Notice the first term in eqn (2.36) and eqn (2.39) are the Casimir energy and Casimir pressure for ideal metals, respectively. At small separations, the finite conductivity correction is important. At 200 nm, the finite conductivity correction for Au is around 58%. At large separations, the real metal acts like the ideal metal and finite conductivity doesn’t make any contribution.

2.2.3 Temperature Correction of Real Metals

The Casimir free energy and Casimir pressure for real material at nonzero temperature is given by eqn (2.21) and eqn (2.24). For ideal metals with infinite dielectric permittivity, according to the Schwinger’s prescription [71], the limit $\varepsilon \to \infty$ should be taken before $\xi \to 0$ when calculate the zero frequency term using eqn (2.21) and eqn (2.24). This process results in the following reflection coefficient for ideal metals:

$$r_{TM}(i\xi_l, k_{\perp}) = 1, \quad r_{TE}(i\xi_l, k_{\perp}) = -1. \quad (2.40)$$

Substitute them into eqn (2.21) and eqn (2.24), the Casimir free energy and Casimir pressure between two parallel planar ideal metals is

$$F(a, T) = \frac{k_B T}{\pi} \sum_{l=0}^{\infty} \int_{0}^{\infty} k_{\perp} dk_{\perp} \ln \left(1 - e^{-2a q_l}\right), \quad (2.41)$$

$$P(a, T) = -\frac{2k_B T}{\pi} \sum_{l=0}^{\infty} \int_{0}^{\infty} k_{\perp} dk_{\perp} \frac{q_l}{e^{2a q_l} - 1}. \quad (2.42)$$
The following forms for Casimir free energy and Casimir pressure can be obtained by calculating the integrals [72, 73]:

\[
F(a, T) = E(a, 0) \left\{ 1 + \frac{45}{\pi^3} \sum_{l=1}^{\infty} \left[ \frac{\coth(\pi tl)}{t^3 l^3} + \frac{\pi}{t^2 l^2 \sinh^2(\pi tl)} \right] - \frac{1}{t^4} \right\} \quad (2.43)
\]

\[
P(a, T) = P(a, 0) \left\{ 1 + \frac{30}{\pi^4} \sum_{l=1}^{\infty} \left[ \frac{1}{t^4 l^4} - \frac{\pi^3 \cosh(\pi tl)}{tl \sinh^3(\pi tl)} \right] \right\} \quad (2.44)
\]

where \( t = \frac{T_{\text{eff}}}{T} \) and \( k_B T_{\text{eff}} = \frac{\hbar c}{2a} \). Here \( E(a, 0) \) and \( P(a, 0) \) are the Casimir energies between two parallel ideal metals given by eqn (1.8) and eqn (1.10), respectively. At zero temperature, \( t \to \infty \), eqn (2.43) and eqn (2.44) are equal to eqn (1.8) and eqn (1.10).

The asymptotic behavior of the Casimir free energy and Casimir pressure can be studied at low temperature limit (or small separation distance), \( T \ll T_{\text{eff}} \), and high temperature limit (or large separation distance), \( T \gg T_{\text{eff}} \). At the separation distance of 1 \( \mu \text{m} \), the effective temperature is 1150 \( K \). At the room temperature of 300 \( K \), the low temperature limit is applicable at the separation of 1 \( \mu \text{m} \). The temperature correction for Casimir pressure calculated at the room temperature using eqn (2.44) is only 0.15%.

At low temperature limit (\( T \ll T_{\text{eff}} \)), the Casimir free energy and Casimir pressure are given by

\[
F(a, T) = E(a, 0) \left[ 1 + \frac{45G_R(3)}{\pi^3} \left( \frac{T}{T_{\text{eff}}} \right)^3 - \left( \frac{T}{T_{\text{eff}}} \right)^4 \right], \quad (2.45)
\]

\[
P(a, T) = P(a, 0) \left[ 1 + \frac{1}{3} \left( \frac{T}{T_{\text{eff}}} \right)^4 \right]. \quad (2.46)
\]
At high temperature limit \((T \gg T_{\text{eff}})\), the Casimir free energy and Casimir pressure are given by

\[
F(a, T) = -\frac{k_B T}{8\pi a^2} \zeta_R(3),
\]
(2.47)

\[
P(a, T) = -\frac{k_B T}{4\pi a^3} \zeta_R(3).
\]
(2.48)

For real metals, appropriate dielectric permittivity model needs to be selected. Substituting the Plasma dielectric permittivity eqn (2.29) into the reflection coefficients eqn (2.25), and the Casimir free energy and Casimir pressure eqn (2.21, 2.24), the Casimir free energy and Casimir pressure combined with temperature and finite conductivity corrections can be obtained using perturbation theory [74, 75].

At low temperature limit \((T \ll T_{\text{eff}})\), the Casimir free energy and Casimir pressure to the first order of \(\delta_0/a\) are

\[
F(a, T) = E(a, 0) \left\{ 1 + \frac{45\zeta_R(3)}{2\pi^3} \left[ \left( \frac{T}{T_{\text{eff}}} \right)^3 - \left( \frac{T}{T_{\text{eff}}} \right)^4 \right] \right. \\
-4 \frac{\delta_0}{a} \left[ 1 - \frac{45\zeta_R(3)}{2\pi^3} \left( \frac{T}{T_{\text{eff}}} \right)^3 + \left( \frac{T}{T_{\text{eff}}} \right)^4 \right] \right\}
\]
(2.49)

\[
P(a, T) = P(a, 0) \left\{ 1 + \frac{1}{3} \left( \frac{T}{T_{\text{eff}}} \right)^4 - \frac{16}{3} \frac{\delta_0}{a} \left[ 1 - \frac{45\zeta_R(3)}{8\pi^3} \left( \frac{T}{T_{\text{eff}}} \right)^3 \right] \right\}
\]
(2.50)

When \(\delta_0 \rightarrow 0\), eqn (2.49), (2.50) of real metals described with the Plasma model are equal to eqn (2.45), (2.46) of ideal metal at low temperature limit.

At high temperature limit, the Casimir free energy and Casimir pressure to the
first order of $\delta_0/a$ are

$$
F(a, T) = -\frac{k_B T}{8\pi a^2} \zeta_R(3) \left(1 - 2\frac{\delta_0}{a}\right), \quad (2.51)
$$

$$
P(a, T) = -\frac{k_B T}{4\pi a^3} \zeta_R(3) \left(1 - 3\frac{\delta_0}{a}\right). \quad (2.52)
$$

When $\delta_0 \to 0$, eqn (2.51), (2.52) of real metals described with the Plasma model are
equal to eqn (2.47), (2.48) of ideal metal at high temperature limit.

If the dielectric permittivity of the Drude model in eqn (2.30) or any other
dielectric permittivity with $\omega^{-1}$ dependence is used to characterize real metals, the
reflection coefficient of transverse electric mode, $r_{TE}(i\xi_l, k_\perp)$ is discontinuous at the
point of $\xi_l = 0, k_\perp = 0$. When $r_{TE}(i\xi_l, k_\perp)$ approaches $r_{TE}(0, 0)$ along any path except
$\xi_l = 0, r_{TE}(0, 0)$ is equal to $-1$. It coincides with the case of ideal metal in eqn (2.40).
When $r_{TE}(i\xi_l, k_\perp)$ approaches $r_{TE}(0, 0)$ along $\xi_l = 0$, $r_{TE}(0, 0)$ is equal to $0$. In this
case, the asymptotic expressions for the Casimir free energy and Casimir pressure at
large temperatures are

$$
F(a, T) = -\frac{k_B T}{16\pi a^2} \zeta_R(3), \quad (2.53)
$$

$$
P(a, T) = -\frac{k_B T}{8\pi a^3} \zeta_R(3). \quad (2.54)
$$

They are only one-half of ideal metals in eqn (2.47) and (2.48). A large thermal
correction is contributed from $r_{TE}$ at zero frequency. Thus, the application of the
Drude model in the zero frequency term of the Lifshitz formula is ambiguous and
different values of $r_{TE}$ were chosen [76–79].
2.2.4 Roughness Correction of Real Metals

The surfaces considered so far have perfect geometrical shapes. Small deviations always present on any real surfaces, especially polycrystalline surfaces. A rough surface might have different reflection coefficients of electromagnetic waves compared to a smooth surface. Different methods have been developed to investigate the roughness corrections to the Casimir effect, such as the perturbation theory for the Green’s function [80], the path-integral approach [81], and the pairwise summation method (PWS) [82]. The PWS method will be introduced below. It is applicable when the surface roughness amplitude is small and the correlation length is large, both compared to the separation distance.

The PWS calculates the Casimir energy by summation of the interatomic potentials over all atoms of two test bodies with a subsequent multiplicative renormalization [82],

\[ U(a) = -\frac{CN_1N_2}{K} \int_{V_1} dr_1 \int_{V_2} dr_2 |r_1 - r_2|^{-7}. \]  \hspace{1cm} (2.55)

Here \( N_1, N_2 \) are the number of atoms per unit volume of two test bodies, \( C \) is the constant of the retarded Van der Waals interatomic energy, \( K > 1 \) is a renormalization constant to take into account approximately the screening effects [83], and \( a \) is the separation distance between the test bodies.

Consider the configuration of two parallel plates separated by \( a \) in the direction of \( z \). The irregular surface roughness of two surfaces are described with stochastic
functions,

\[ z_1 = \delta_1 f_1(x, y), \]
\[ z_2 = a + \delta_2 f_2(x, y). \]

Here \( \delta_{1,2} \) are variances of surface roughness on two surfaces. The mean values of roughness are equal to zero, \( \langle \delta_i f_i(x, y) \rangle_i = 0, \ i = 1, 2 \). With the small roughness assumption, \( \delta_i/a \ll 1, \ i = 1, 2 \).

Assuming a normal distribution at each point of the surface, the Casimir pressure with partial accounting for the effects of nonadditivity is given by [82]

\[ P_R(a) = P(a) \left[ 1 + 10 \frac{\delta_1^2 + \delta_2^2}{a^2} + 105 \frac{(\delta_1^2 + \delta_2^2)^2}{a^4} \right], \]

(2.58)

where \( P_R(a) \) and \( P(a) \) are the Casimir pressure with and without roughness correction. Since the correlation length is large, \( P_R(a) \) only depends on \( \delta_1^2 + \delta_2^2 \). No correlation term is involved.

For the configuration of sphere-plate, the Casimir pressure is given by [84]

\[ P_{sp,R}(a) = P_{sp}(a) \left[ 1 + 6 \frac{\delta_1^2 + \delta_2^2}{a^2} + 455 \frac{(\delta_1^2 + \delta_2^2)^2}{a^4} \right]. \]

(2.59)

With a typical value of roughness amplitude of 2 nm, the roughness correction to the Casimir pressure at closest separation of 200 nm is around 0.12\%.
2.2.5 Proximity Force Approximation for Sphere-Plate Configuration

Based on the experience of older experiments, it is obvious that keeping two flat plates perfectly parallel is extremely difficult. Even a small tilt of one plate at close distance could cause large systematic error. To avoid the issue of parallelism in the conventional plate-plate geometry, the form of a flat plate and a curved surface was adopted in most modern Casimir force measurements. For sphere-plate geometry, the mode summation and subtraction of vacuum energy method will not be applicable due to the ultraviolet divergences. The proximity force approximation (PFA) [39] will be introduced in this section to connect the Casimir energy per unit area between two parallel plates with the Casimir force between a sphere and a plate.

The proximity energy $U_{sp}$ associated with a sphere with the radius of $R$ should be [39]:

$$U_{sp}(z) = \int \int E_{pp}(D)d\sigma + \text{corrections},$$

where $E_{pp}$ is the interaction energy per unit area of two parallel surfaces at the appropriate separation $D$. The integral is over the area of sphere interacting with the plate. When the curvature defining the gap is small, only the contribution of parallel parts, which is marked as red in fig (2.2), is important. The non-parallel corrections become negligible.

It can be easily derived that $D(\theta) = z + R(1 - \cos \theta)$ and $d\sigma = 2\pi R \sin \theta R d\theta$. 
Figure 2.2: Proximity Force Approximation (PFA) applied to sphere-plate configuration. Each small curved surface is replaced with parallel surface. The non-parallel contribution is neglected as the curvature of the sphere defining the gap is small.
Therefore the proximity energy will be

\[ U_{sp}(z) = 2\pi R \int_{z}^{z+R} E_{pp}(z + R - R \cos \theta) R \sin \theta d\theta \]

\[ = 2\pi R \int_{z}^{z+R} E_{pp}(D) dD. \quad (2.60) \]

The lower and upper limits are chosen to integral over the lower half of the sphere. Considering \( E_{pp}(D) \) decays with \( D^{-3} \) as shown in eqn (1.9), if \( R \gg z \), the upper limit \( z + R \) can be treated with infinite.

\[ U_{sp}(z) = 2\pi R \int_{z}^{\infty} E_{pp}(D) dD \quad (2.61) \]

So the force between sphere and plate will be

\[ F_{sp}(z) = -\frac{\partial U_{sp}}{\partial z} \]

\[ = 2\pi R E_{pp}(z). \quad (2.62) \]

Plug in the eqn (1.9) for \( E_{pp}(z) \). The Casimir force between the perfectly conducting sphere and plate at zero temperature is

\[ F_{sp}(z) = -\frac{\hbar \pi^3 R}{360 z^3}. \]

The deviation of PFA is less than 0.4\( z/R \) [85]. At a separation of 200 nm, for a sphere with the radius of 60\( \mu m \), 0.4\( z/R \) = 0.13\%. 

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Chapter 3

Atomic Force Microscope
3.1 Introduction

In 1982, the scanning tunneling microscope (STM) was first developed by Gerd Binning and Heinrich Rohrer [86] based on quantum tunneling effect. When a sharp and conducting tip is close enough (4-7 Å) to a sample with a bias voltage applied, a small tunneling current will be induced. By moving tip laterally across the sample, a topographical image of the sample surface can be achieved by monitoring the magnitude of tunneling current. STM has a remarkable 0.1 nm lateral resolution and 0.01 nm depth resolution.

Inspired by STM, Gerd Binnig, Calvin Quate and Christoph Gerber invented the atomic force microscope (AFM) 4 years later in 1986 [87]. Both STM and AFM belong to the family of scanning probe microscope (SPM). AFM takes topographic images and surface mechanical information with small force interaction between the probe and sample. This force is usually smaller than 100 nN. Such a small force will cause very little damage to the sample. The main advantage of AFM is that it has very high resolution. In force mode, it can detect force as small as $10^{-18}$ N. In image mode, it has about 3 Å resolution in horizontal plane and 1 Å in vertical direction. Unlike scanning electron microscope (SEM) [88] and STM, AFM can be applied to both conductors and insulators. AFM can also be operated in air, ultra high vacuum or liquid environments. AFM can also work as force spectroscopy. The main restrictions of AFM include the limited sample size, slow scanning rate, and high requirement of vibration control. AFM is now widely used as a common surface characterization
technique in the fields of semiconductor technology, solid state physics, biology and chemistry. Our experimental set up of precision Casimir force measurement is based on AFM. Thus, the detailed operation principle of AFM will be discussed in this chapter.

3.2 AFM Configuration and Operation Procedure

One typical AFM system is shown in fig (3.1). A red laser beam shoots from the top and is reflected off the back of cantilever. By analyzing the position of the laser on the split photodiode, the AFM controller detects the deflection and oscillation amplitude of cantilever and sends the feedback signal to XYZ scanner to maintain the average tip-sample separation constant. The scanner is made of piezoelectric material that allows an nm level control of sample position. Finally a topographic image is stored to the computer by AFM controller.

The basic operation procedure for AFM measurements is similar for different operation modes. First mount the probe into the cantilever holder and place it into the AFM header. Align the red laser with cantilever to maximize the SUM signal and adjust the position of photodiode to minimize both horizontal and vertical signal to almost zero. Tune the cantilever to its thermal resonance frequency and activate it to proper amplitude if operated in Tapping Mode. Bring the tip close to the sample within 1 mm and start automatic engaging process to find the appropriate separation
Figure 3.1: The schema of a typical AFM system. The deflection of cantilever is measured by detecting the position of laser beam reflected off the back side of cantilever using the position-sensitive photoreceiver. The tip-sample separation is controlled a XYZ scanner, made of a piezoelectric material. The AFM controller sends feedback signal through a proportional-integral-derivative loop to the scanner and saves topographic images or force curves to the computer.
between tip and sample. Finally, optimize the resolution of topographic images or force curves by adjusting the scanning parameters and feedback signals.

### 3.3 Operation Modes

Our AFM can be operated in Contact Mode and Tapping Mode for imaging measurements and in Force Curve Mode for surface mechanical properties measurements.

#### 3.3.1 Contact Mode and Tapping Mode

After the probe and sample are loaded into the AFM header and the laser is adjusted to the center of the split photodiode detector, either Tapping Mode or Contact Mode needs to be chosen to proceed. Fig (3.2) illustrates how these two operation modes work. ”A” and ”B” mean the voltage measured by upper and lower photodiodes.
In Contact Mode, AFM probe oscillates due to the its thermal noise resulting from environmental temperature. This thermal noise amplitude along with the distance resolution of splitted photodiode limits the vertical resolution of AFM. Before engaging process, a setpoint value is given to AFM Controller for feedback purpose. In Contact Mode, the deflection signal of cantilever \( \frac{A-B}{A+B} \), is output to AFM controller. After the deflection signal of cantilever matches the setpoint value, AFM stops the engaging process and starts to scan through sample surface. The scan size and scan rate is controlled by the X and Y voltage output to the piezoelectric scanner. By monitoring the deflection signal of cantilever and applying a Z voltage to the piezoelectric scanner, AFM maintains a constant vertical deflection of cantilever. The vertical distance moved by scanner is equal to the height of sample at each position \((x, y)\). This height of sample at each \((x, y)\) is recorded by the computer and displayed as a pseudocolor plot on the screen.

In Tapping mode, the cantilever is mechanically driven by a piezo at or close to its fundamental resonance frequency. The oscillation amplitude usually ranges from 20 nm to 100 nm. Instead of the reflection of cantilever in Contact Mode, the setpoint for Tapping Mode is usually Root Mean Square (RMS) of voltage signal measured by the split photodiode detector. It is proportional to the resonance amplitude of cantilever. The oscillating probe is brought into intermittent contact with sample. At the vertex of one tapping cycle, the probe feels a net long range attractive force dominated by Van der Waals force. At the bottom of one tapping cycle, the tip penetrates through
Figure 3.3: AFM images of CVD graphene with wrinkles and some double layer area taken in Tapping Mode.
the adsorbed fluid layer on sample surface. Its dynamics are controlled by a net short range repulsive force dominated by Pauli and ionic repulsion. This complex force interaction reduces resonance amplitude as tip-sample separation reduces \[89–94\]. In simple words, by maintaining a constant resonance amplitude in the cantilever, tip-sample separation is also kept constant. The AFM feedback loop applies Z voltage to the piezoelectric scanner to maintain the resonance amplitude. The process of data acquisition and visualization is the same as Contact Mode.

An AFM image of Chemical Vapor Deposition (CVD) grown single layer graphene taken with Tapping Mode is shown in fig (3.3). The size of image is 10 x 10 \( \mu m \). These straight lines are wrinkles formed during graphene growing. Their heights are between 1 and 4 nm. Some double layer area that are apparently higher than single layer can be observed.

The scanning rate of Tapping Mode is limited by the response time of amplitude change. For samples with large features and severe change in height, Contact Mode is a better choice than Tapping Mode. For most other cases, Tapping Mode is more popular, because Contact Mode exerts large lateral friction force on sample and these forces cause damages and distort features in the image.

3.3.2 Force Curve Mode

The mechanical properties of sample surface can be measured using AFM in Force Curve Mode. A force curve graph between a probe and a sample is shown in fig (3.4).
Figure 3.4: A Standard Force Curve taken by AFM in Force Curve Mode. The vertical axis is the force magnitude. The horizontal axis is the separation distance. At large distance, there is no force between the tip and sample. At separations between contact point and 10 nm, the attractive Van der Waals force is dominant. After contact, the repulsive force is dominant.
Tip approaches, contacts and indents sample from left to right in the figure.

The force curve can be divided into three regions. In region (a), the tip is far away (>10 nm) from the sample. At this distance, all force interaction is almost equal to zero. Thus, the force curve is flat. In region (b), the tip-sample separation distance is below 10 nm. In this region, attractive van der Waals force dominates. This force can be as large as 1 nN. For a cantilever with the spring constant of 0.1 N/m, it can cause a bending distance of 10 nm. An obvious curvature of the force curve can be observed in this region. Region (c) is separated from region (b) by the contact point. The contact point can be understood as the separation between tip and sample is equal to the average intermolecular distance. After contact, the repulsive force due to Pauli and ionic repulsion starts to dominate. It causes both bending of the cantilever and indentation of the sample surface. The slope of curve depends on the spring constant of cantilever and sample surface.

The sample surface stiffness measurement can be conducted in ambient and liquid environments. After the engaging process, a zero voltage is applied to the probe piezo to stop its excitation. The operation mode of AFM is then switched from Tapping Mode to Force Curve Mode, as shown in fig (3.5).

According to the Newton’s Third Law, the force on cantilever and sample has the same magnitude:

\[ F_c = F_s, \]

where \( F_c \) is the magnitude of force applied on the cantilever and \( F_s \) is the magnitude
Figure 3.5: Force Curve Mode.

of force applied on sample surface. According to Hooke’s Law, we have

\[ k_c z_c = k_s z_s \]  \hspace{1cm} (3.2)

where \( k_c \) is the spring constant of cantilever, \( z_c \) is the bending distance of the free end of the cantilever, \( k_s \) is the spring constant of sample surface, and \( z_s \) is the indentation of sample surface.

Two force curves are shown in fig (3.6). Fig (3.6a) is a force curve on a glass plate. The vertical axis is tapping mode deflection voltage measured by PSD. The product of tapping mode deflection voltage (V) and deflection sensitivity (nm/V) gives the deflection of cantilever (nm). The horizontal axis is the voltage applied to scanner in Z direction. With the calibration of the AFM scanner, a calibration constant (nm/V)
(a) Force curve on a glass plate.

(b) Force curve on a soft sample.

Figure 3.6: Force curve measured with AFM.
can be achieved to convert this voltage (V) to separation distance (nm). The blue line stands for approaching and the red line stands for retracting. The small wave at large separations is a result of interference of laser reflected from the cantilever and glass. Fig (3.6b) is a force curve on a soft sample. As we can see, the slope of the force curve on glass is much larger than that on soft sample.

Suppose that the deflection sensitivity is $S_{def}$ (nm/V), the calibration constant of the scanner is $K_{piezo}$ (nm/V), the slope of force curve on the sample is $S_{sample}$ (V/V) and the movement of the scanner is $z_{piezo}$ (nm). The slope of force curve takes this form:

$$S_{sample} = \frac{z_c}{S_{def}} \frac{S_{def}}{z_{piezo}/K_{piezo}}.$$  \hspace{1cm} (3.3)

After contact the movement of the scanner is equal to the sum of the bending of cantilever and the indentation of sample

$$z_{piezo} = z_c + z_s.$$  \hspace{1cm} (3.4)

So according to eqn (3.3), we have

$$k_s = \frac{S_{def} S_{sample}}{K_{piezo} S_{def} S_{sample} - K_{piezo}} k_c.$$  \hspace{1cm} (3.5)

Notice the $S_{def}$ and $K_{piezo}$ in eqn (3.3) are constants of the AFM and are independent of samples. Eqn (3.5) has a simpler form as derived below.

The glass plate is much stiffer than cantilever and undergoes negligible indentation in the stiffness measurements:

$$z_{glass} = 0,$$  \hspace{1cm} (3.6)
\[ z_{\text{piezo}} = z_c. \]  

(3.7)

Then eqn (3.5) can be rewritten as

\[ S_{\text{glass}} = \frac{K_{\text{piezo}}}{S_{\text{def}}}. \]  

(3.8)

Finally, we have the final equation for spring constant of sample

\[ k_s = \frac{S_{\text{sample}}}{S_{\text{glass}} - S_{\text{sample}}} k_c. \]  

(3.9)

The slope of the force curve can be easily measured with Nanoscope analysis software. Measurements of deflection sensitivity and spring constant of cantilever will be discussed on the next two sections.

### 3.4 Calibration of AFM

#### 3.4.1 Calibration of the Piezoelectric Scanner

To extract physical meaning from topographic images and force curves, the voltages applied to the scanner and the difference voltages measured by the PSD need to be converted from voltage (V) to distance (nm).

AFM scanners are made from piezoelectric materials, which produce voltages in response to mechanical stress and create displacement when voltages are applied. The ceramic material, lead zirconate titanate (PZT), is widely used for piezoelectric
Figure 3.7: Calibration of Scanner. A pitch grating with known feature size as a reference provided with manufacturer is used for calibration. It contains uniformly distributed pits in X & Y directions and each pit has the same depth. The common pitch grating for calibration has $5 \times 5 \, \mu m$ size and 200 nm depth. Red squares are the uniformly distributed pits.
devices. The sensitivity of a piezo means its displacement responded to the applied voltage. It takes this form for Veeco AFM:

\[ K_{\text{piezo}} = \frac{z_{\text{piezo}}}{V_{\text{piezo}}} = lw d_{31}, \] (3.10)

where \( z_{\text{piezo}} \) is the displacement of piezo, \( V_{\text{piezo}} \) is the driving voltage, \( l \) and \( w \) are the length and width of piezo, and \( d_{31} \) is material property. The sensitivity of piezoelectric scanner decreases exponentially with operation time. It needs to be re-calibrated about every six months to ensure the precision and accuracy of images.

An AFM image of pitch grating is shown in fig (3.7). For calibration of X & Y, an automated calibration routine is provided by Veeco AFM. This routine captures multiple images at various scan sizes and settings. By measuring the separation between different pitches, the sensitivity for X & Y can be calculated. The height calibration can be calculated by the following formula

\[ \text{New Sensitivity} = \frac{\text{Old Sensitivity} \times \text{Actual Depth}}{\text{Measured Depth}}. \] (3.11)

The sensitivity of the piezo is not linear and has hysteresis as shown in fig (3.8). For a small range of voltage, it can be treated as linear. For a large range, it depends on the magnitude, frequency and polarity of applied voltages. Therefore, a real-time precise calibration of piezo will be necessary for our Casimir force measurements.
3.4.2 Deflection Sensitivity

Deflection sensitivity of the AFM, $S_{def}$, can be calculated from the slope of force curve on a glass plate or any stiff surface, $S_{glass}$. Once the sensitivity of piezoelectric scanner $K_{piezo}$ is known, $S_{def}$ can be calculated from eqn (3.8):

$$S_{def} = \frac{K_{piezo}}{S_{glass}}$$

(3.12)

3.5 AFM Probe

Proper selection of AFM probe is essential to achieve high resolution. AFM probe consists of a cantilever and a sharp tip integrated on the end of cantilever. They are usually made of silicon or silicon nitride. Silicon probes tend to be stiffer and
have larger spring constant, larger mechanical quality factor, and higher resonance frequency. Fig (3.9) shows two Scanning Electron Microscopy (SEM) images of AFM probes with different geometric shapes. Fig (3.9a) is a V-shaped probe and fig (3.9b) is a rectangle probe. The bright dot on the end of cantilever is a sharp pyramid tip. Fig (3.10) is a top-down SEM image of a sharp pyramid tip.

3.5.1 Cantilever

The cantilever greatly determines the spring constant, resonance frequency, and quality factor of the probe. Different cantilevers should be chosen depending on the operation modes and samples.

For Contact Mode and Force Curve Mode, soft cantilevers are preferred, which produce smaller lateral force and therefore create fewer damages to samples. Soft
cantilevers also cause significant deflection to small force and increase vertical resolution.

For Tapping Mode in air, a stiff rectangular cantilever with large spring constant and high resonance frequency is preferred. A stiff (∼50 N/m) probe can overcome the adhesive capillary force from sample surface. A rectangular cantilever has a higher quality factor (Q) comparing to a similar V-shaped cantilever.

Tapping Mode in liquid environment is necessary for imaging of biological samples. A typical choice of cantilever is a soft V-shaped silicon nitride cantilever with the spring constant of ∼0.1 N/m. V-shaped cantilever provides more lateral stability than rectangular cantilevers. A cantilever with 100 Q factor in air may only have
1~3 Q factor in liquid due to the hydrodynamic damping. This requires more effort and patience to adjust feedback parameters. Also a larger driving voltage is needed to achieve desired oscillation amplitude of cantilever. Due to the damping, a cantilever with 40 ~ 60 kHz resonance frequency in air has only around 10 kHz resonance frequency in liquid.

The back side of the cantilever is sometime coated with metal to increase the reflectivity. An alkali solution can dissolve the Al coating. Thus, Cr/Au coating is recommended for liquid measurements. One side effect of the coating layer is that it may cause the bending of cantilever due to the surface tension. Depending on the coating thickness and process, this bending angle is sometimes 10 degrees or even larger.

3.5.2 AFM Tip

Most AFM tips are made of silicon or silicon nitride. They can have 3-sided pyramid shape or 4-sided pyramid shape. Fig (3.10) is a 4-sided pyramid tip. The very end of most AFM tips contains more than one atom and can be modeled as a hemisphere, as shown in fig (3.11).

The tip’s radius of curvature is important because it arises lateral artifacts of AFM image. As shown in fig (3.12), when the size of sample is comparable to tip’s radius of curvature, artifacts are significant. Imaging some well calibrated particles can be helpful to determine the tip size.
The typical tip size is 5~10 nm for silicon tips and 20~60 nm for silicon nitride tips. Silicon tips are sometimes doped for static charge dissipation. To make the whole probe conductive and reduce adhesion force, the AFM tip can also be coated with a metal film. For measurements of surface mechanical properties where the lateral resolution is not important, colloidal AFM tips are used.

3.5.3 Spring Constant Calibration

There are different methods to calibrate the spring constant of cantilever, including dimensional analysis [95, 96], added mass [97], reference cantilever [98], and thermal noise analysis [99]. Each method has some deficits and may have 10~20% uncertainty.

Dimension Analysis

Dimension analysis gives theoretical value of the spring constant of cantilever based on its geometry shape and material properties. For a rectangular cantilever,
Figure 3.12: Artifacts in AFM imaging produced by large tip size. The blue lines stand for the AFM tip with the radius of curvature equal to R. The black spheres with radius of r are small samples. The red line is the actual track of AFM tip recorded by the computer.

the spring constant is given by

\[ k = \frac{E \cdot w}{4} \left( \frac{t}{l} \right)^3, \]  

(3.13)

where \( k \) is the spring constant, \( E \) is the Young’s modulus, \( w \) is the width, \( t \) is the thickness and \( l \) is the length, as shown in fig (3.13). The error of this method mainly comes from the determination of thickness, which usually has a very small value of around 1 \( \mu \)m to achieve high Q factor.

**Added Mass**

Added mass method measures change in resonance frequency by adding characterized mass to the end of cantilever. The fundamental resonance frequency of a
rectangular cantilever is given by

\[ v = \frac{\omega}{2\pi} = \frac{1}{2\pi} \sqrt{\frac{k}{M + m^*}}, \]  

(3.14)

\[ m^* \approx 0.24m_c, \]  

(3.15)

where \( m_c \) is the mass of cantilever, \( m^* \) is the effective mass of the rectangular cantilever, \( M \) is the calibrated added mass, and \( k \) is the spring constant of cantilever to be calibrated. The value of added mass, \( M \), and the position where the mass is added contribute to uncertainties of the spring constant. Also, the process of adding mass and removing mass may damage the cantilever.

**Reference Cantilever**

Reference cantilever method is shown in fig (3.14). This method is similar to the stiffness measurements in Force Curve Mode of AFM. Force Curve Mode measures the stiffness of sample with a calibrated cantilever. Reference cantilever method calibrates the cantilever with another reference cantilever. According to eqn (3.9),
we have

\[ k_c = \frac{S_{\text{glass}} - S_{\text{ref}}}{S_{\text{ref}}} k_{\text{ref}} \]  \hspace{1cm} (3.16)

where \( k_c \) is the spring constant of the test cantilever, \( k_{\text{ref}} \) is the spring constant of the reference cantilever, \( S_{\text{glass}} \) is the force curve slope of the test cantilever on a glass plate, and \( S_{\text{ref}} \) is the force curve slope of the test cantilever on the reference cantilever.

**Thermal Noise Analysis**

Thermal noise analysis was proposed in 1993 [100]. It is now the most popular method for the spring constant calibration of cantilevers. The end of the cantilever vibrates at its resonance frequency as a result of thermal noise. According to the equipartition theorem, in thermal equilibrium, the thermal energy stored in any free mode is equal to \( \frac{1}{2} k_B T \). When a cantilever is treated as a perfect spring, we have

\[ \frac{1}{2} k \langle z^2 \rangle = \frac{1}{2} k_B T, \]  \hspace{1cm} (3.17)
where $k$ is the spring constant of cantilever, $z$ is the thermal noise amplitude, $k_B$ is the Boltzmann constant, and $T$ is the environment temperature in kelvin. $\langle z^2 \rangle$ is the area under fundamental resonance peak in the power spectrum. Its value can be calculated by fitting the power spectrum to the Lorentzian distribution in air

$$A(\nu) = A_0 + \frac{C_1}{(\nu - \nu_0)^2 + C_2}, \quad (3.18)$$

or simple harmonic oscillator distribution in liquid

$$A(\nu) = A_0 + A_{DC} \frac{\nu^2}{\sqrt{(\nu^2 - \nu_0^2)^2 + \frac{\nu_0^2 \nu^2}{Q^2}}}, \quad (3.19)$$

where $A(\nu)$ is the resonance amplitude as a function of frequency, $A_0$ is the background noise, $C_1$ and $C_2$ are fitting parameters, $\nu_0$ is the fundamental resonance frequency, $A_{DC}$ is the DC amplitude, and $Q$ is the quality factor. Fig (3.15) shows the power spectrum of a stiff cantilever vibrating due to the thermal noise and its Lorentzian fitting.

To achieve higher precision of spring constant, higher order harmonics, deflection sensitivity correction [101], cantilever shape [102], and viscous resistance [103] need to be considered. Because of its convenience, thermal noise analysis can be performed with many AFM commercial softwares. The error of this method is around 5%~10%.
Figure 3.15: Power spectrum of a stiff cantilever driven with thermal energy in the air. Black line is the data collected by PSD. Red line is its corresponding Lorentzian fitting.
3.6 Application of AFM in Biophysics

Two advantages of AFM compared to STM and SEM is that it can measure surface mechanical properties in force curve mode and work in liquid environments. These two advantages make AFM widely used in biophysics. In this section, procedures for measurements of the mechanical properties of basement membrane (BM) using sharp tip and living cells using colloid tip will be introduced.

BM is a thin, fibrous, extracellular matrix of tissue. Its function is to maintain stable tissue borders. It can also communicate with cells and bind them to the underlying collagenous matrix. The mechanical properties of BM indicate the state of tissue [104–106].

BM and cells are grown on a glass coverslip. To measure the stiffness of BM, cells are decellularized [107] and only BMs are left on the glass coverslip. The glass coverslip is then fixed to the AFM sample holder with double sided tape. Special attention should be paid to avoid air bubbles. With the presence of air bubbles, quick drift of tip-sample separation is usually observed.

Fig (3.16) shows an AFM image of BM. It has a matrix shape. Imaging of BM in liquid is relatively difficult. Without knowing the exact landing position, a sharp tip can easily point to the hole of the matrix. So, instead of sharp tips, colloidal tips are suggested, as shown in fig (3.17). The colloidal probes have a round tip with a radius of 2~10 µm. For stiffness measurements of living cells, the colloid tips are required.
Figure 3.16: AFM image of basement membrane.

Figure 3.17: Colloidal AFM Probe.
As mentioned before, to achieve significant bending of cantilever and indentation of sample, the spring constant of probe should be close to the stiffness of sample. The spring constant of BMs and cells are usually around 0.01~0.1 N/m. So silicon nitride V-shaped probes with the spring constant of 0.1 N/m are preferred. For our Veeco AFM, the range of deflection voltage measured by PSD is 5 V. With a typical deflection sensitivity of 36 V/nm, 2 V of deflection voltage corresponds to 7.2 nN for cantilevers with the spring constant of 0.1 N/m. This small force can minimize the physical damage caused to BMs and cells. For soft samples with the spring constant of 0.01 N/m, 7.2 nN causes 720 nm indentation. This indicates the minimum thickness of soft samples should be at least several μms.
Chapter 4

Experimental Setup
4.1 Introduction

The precision Casimir force measurements are conducted between an Au coated sphere and plate, as shown in fig (4.1). The dynamic Casimir force gradient measurement system is modified from a standard AFM. It operates in ultrahigh vacuum (UHV) to achieve a high Q-factor force sensor. The force sensor consists of a micro-cantilever and an Au coated sphere attached to the free end of cantilever. An fiber-optic interferometer is aligned perpendicularly with the back of the cantilever using an XYZ stage. The interference signal reflected off the fiber end and the back of cantilever is converted to AC and DC electrical signals through amplifier, photodiode and filters. A phase-locked loop (PLL) detects the phase difference between the cantilever oscillation and the driving force, measures the resonance frequency shift of cantilever, and provides new driving force to the cantilever. The resonance frequency shift of cantilever is proportional to the interaction force gradient between the sphere and plate and is recorded by the computer. The proportional-integral-derivative (PID) loop maintains the separation of fiber-cantilever gap constant by applying feedback voltages to piezo 2. The plate is supported by a long piezo tube. During the measurements, the plate first moves towards the sphere from a couple microns away, stops at a few hundred nanometers, and moves further from the sphere. This is accomplished by supplying a triangle voltage to the piezo tube at a low frequency. The displacement of the plate is calibrated in real time by fitting the interference fringes to its theoretical equation. To determine the closest sphere-plate separation distance, the
residual potential between the grounded sphere and plate, and the spring constant of the cantilever, different DC voltages are applied to the plate. An Argon-ion gun is mounted on the side of main chamber to remove the sample surface contaminants and adsorbates. The whole stainless steel chamber is supported by an air-damped optical table to reduce vibration noise from the environment. A dry scroll pump and a turbo-molecular pump brings the pressure from the atmosphere to UHV. Then an ion pump is started to maintain the vacuum and the scroll pump and molecular-turbo pump are turned off to further reduce vibration noise. We use the LabVIEW program for data acquisition, instrumental control, and automation. An AFM Nanoscope program is also used to control the movement of the piezo tube and data acquisition.

4.2 Vacuum System

A high vacuum environment is necessary to perform precision Casimir force measurements. In a vacuum environment, the Q factor of the cantilever increases without damping of air. Also, the air creates a thick layer of surface adsorbates on sample surface resulting in temporal variation of the residual potential between the sphere and plate [46].

In our experiments, the measurements are performed in a six-way stainless steel vacuum chamber with CF flanges. To direct the laser into the vacuum, two home-made optical fiber feedthroughs are connected to the main chamber. The buffer
Figure 4.1: The Experimental Setup for Precision Casimir Force Measurements with Argon-ion cleaning.
stripped bare fiber passes through a small hole in the middle of a 2.75 inches O.D. blank CF flange. The CF flange is sonicated with hot acetone to remove debris before use. The hole is then sealed with vacuum Torr Seal that can be used at pressures of $10^{-9}$ torr.

The electrical feedthrough uses subminiature type-D connectors on a CF flange. It has 25 gold plated pins that are hermetically sealed and electrically insulated in a stainless steel shell using glass ceramic bonding technology. A standard off-the-shelf serial cable connector is designed for air side connections. The vacuum side connections use an UHV compatible connectors and kapton insulated ribbon cable.

A differentially pumped direct drive multi-motion feedthrough is installed on the vacuum chamber. It provides rotary and linear motion to coarsely control the vertical position of the plate.

The chamber is directly connected to a Varian V-301 turbo-molecular pump backed by a Varian SH-100 dry scroll vacuum pump. After all screws are tightened, the scroll pump is switched on. It takes about 20 minutes to bring the pressure of chamber at the atmosphere pressure to $10^{-2}$ torr. A thermal conductivity gauge is used to monitor this low vacuum. After that, the turbo pump is turned on to achieve higher vacuum. Without baking, the vacuum will stabilize at $2 \times 10^{-7}$ torr, which is measured by an ion gauge. To pump out the water vapor adsorbed to the chamber walls, the bake-out procedure is necessary. An ultimate pressure of $10^{-9}$ torr can be achieved for our chamber. Then the pumping is taken over by the ion pump. The
scroll pump and turbo pump are turned off to reduce mechanical noise.

4.3 Fiber-Optic Interferometer

The fiber-optic interferometer is based on the optical interference in the cavity formed between the cleaved end of a single-mode optical fiber and the back side of cantilever [108]. The phenomenon of light interference has been known for centuries. The invention of the laser in 1960 provides high intensity light with both spatial coherence and temporal coherence properties. The development of the photodiode allows the detection of an optical signal at a low noise level. Finally the single mode optical fiber made it possible to guide light in a flexible fiber optic waveguide and realize a fiber optic interferometer [109]. The fiber-optic interferometer has very high sensitivity and is suitable for both DC and AC signal measurements. Since the optical single mode fiber is made of silica and has a typical diameter of 125 µm, it is mechanically robust and very compact.

Our measurement system contains two fiber-optic interferometers. The two interference cavities are shown in fig (4.2). The single mode fiber (SMF) for the fiber-optic interferometer on top of the cantilever is mounted on a coarse XYZ positioner. The fiber is positioned manually outside the chamber to make alignment with the cantilever. Due to the refractive index difference of silica and vacuum, 4% of the incident light is reflected at the cleaved end of SMF according to the Fresnel equations. The
other 96% of the light exits the fiber and is scattered by the cantilever. Part of the light is reflected back into the fiber. The total reflected optical power depends on the phase difference between two light beams. As the cantilever oscillates, the oscillation frequency, amplitude, and phase can be measured. The fiber-optic interferometer also has good stability in low frequency, which enables it to measure the displacement of the Au plate and the bending of the cantilever.

The whole fiber-optic interferometer is shown in fig (4.3). The laser emitted by a semiconductor laser diode is coupled into the input 1 of the 2×2 fused fiber optic coupler from Thorlabs. The 50:50 directional coupler splits the incident light equally by power to outputs 2 and 3. The light from output 2 is directed into vacuum by
a homemade optical feedthrough. The optical feedthrough is sealed by Torr Seal epoxy, which is good for pressure down to $10^{-9}$ torr. The reflected interference light from the cantilever passes back to the coupler end 2. Half of its power is directed by the coupler to end 4. The optical signal is then converted to voltage signal by a low-noise InGaAs fiber-optic photoreceiver. The fiber from end 3 is submerged in index-matching liquid to prevent reflection of light at the cleaved end.

The thermal peak of the cantilever as measured by the fiber-optic interferometer is shown in fig (4.4). To reduce the thermal noise from the heating problem, the laser diode for cantilever has an infrared wavelength of 1550 nm. It is controlled by a Newport LDC-3724B laser diode controller, which provides high stability and low noise current source with a thermoelectric cooler module. For the interferometer above the plate, a short wavelength of 520 nm is adopted in order to have more interference fringes and better calibrate the movement of the plate. The 520 nm
laser is produced by a fiber-coupled Fabry-Perot (FP) benchtop laser source from Thorlabs. The integrated thermoelectric cooler (TEC) stabilizes the temperature to within 0.005 °C. The FP laser diode is pigtailed to a single mode fiber that is terminated at an FC/PC bulkhead connector on the front panel. An angled fiber ferrule at the internal laser launch point is utilized to minimize reflections back into the laser diode and improve the stability of the laser diode’s output.

4.4 Coarse Positioner and Piezo Actuator

Similar to a commercial AFM, to optimize the signal-to-noise ratio, the laser must be aligned with cantilever. The cantilever is mounted firmly on the holder by a clip to avoid any extra vibration noise. The alignment of fiber to cantilever is controlled by an XYZ stage. This is completed outside the chamber with the help of a 5× video camera. The vertical separation between fiber end and cantilever is around 20~50 μm. The best alignment is achieved when the laser reflection power is maximized and an oscillation peak of the cantilever due to thermal noise shows up on an SR770 FFT spectrum analyzer. For the fiber above the plate, since the sample plate has a diameter of 10 mm, there is no need to align it.

After fiber alignment, the cantilever and plate are loaded into the chamber. At the moment of pumping down, the sphere and plate are separated by at least 350 μm (the length of cantilever) to avoid crash of cantilever due to the vibration noise. The
Figure 4.4: Thermal peak of a modified cantilever measured by the fiber-optic interferometer. The black line is the power density spectrum of the cantilever. The red line is the Lorentzian fitting. The resonant frequency of cantilever is 4431 Hz. The Q-factor is 1758.
linear-rotary motion feedthrough accessible outside the vacuum makes it possible to coarsely position the plate to within 1 \( \mu \text{m} \) from the cantilever. The fine position of the plate is controlled by the piezo tube made by Veeco E Scanner.

In the dynamic force measurements, the cantilever is actuated at its resonance frequency by a miniature ceramic encapsulated multilayer piezo actuator. It is made from a ceramic material, which optimally combines the properties of stiffness, capacitance, displacement, temperature stability, and lifetime. It accomplishes a sub-nanometer resolution in positioning. The lack of polymer insulation minimizes its outgassing rate. It can be baked up to 150 \(^{\circ}\)C due to its high Curie temperature.

The attractive force between the sphere and the plate causes bending of the cantilever. To compensate for this bending and keep the size of the interference cavity constant, a second piezo is stacked on top of the cantilever. The DC signal from the fiber optic interferometer is used for a PID loop to control the relative position of the cantilever to the fiber end.

### 4.5 Frequency Modulation Mode

In the Contact Mode of a commercial AFM, the force interaction between the tip and the sample surface is measured by monitoring the static deflection of the cantilever. The tip-sample separation and their first order interaction force is described by Hooke’s law: \( F = -kz \). Here \( F \) is the interaction force, \( k \) is the spring constant of cantilever, and \( z \) is the tip-sample separation. Since the cantilever is statically
mounted, this measurement is also called the static force measurement.

Typical Tapping Mode operation is carried out with constant excitation frequency, which is close or equal to the natural resonance frequency of the cantilever. The change of amplitude is used for topographical feedback. Thus it is called the amplitude modulation detection, as shown in fig (4.5). It has several drawbacks. First the response time of amplitude is related to the Q factor by \( t_{\text{response}} = Q/2f_0 \). Here \( t_{\text{response}} \) is the response time of the amplitude. When the Q factor is very high, the response time increases, which limits the maximum scan speed. When the frequency shift is too large, the amplitude change saturates. The amplitude change depends on both the tip sample interaction and the Q factor of the cantilever, which leads to systematic errors.

In dynamic force measurement, the cantilever is treated as a damped harmonic oscillator. With the presence of an external interaction force, the equation of motion for a damped harmonic oscillator is given by

\[
m \frac{d^2 z}{dt^2} + \frac{m \omega_0}{Q} \frac{dz}{dt} + k(z - z_0) = F_{\text{int}}(z).
\] (4.1)

Here \( \omega_0 \) is the undamped angular frequency, \( k = m \omega_0^2 \) is the spring constant of the cantilever, and \( F_{\text{int}}(z) \) is the interaction force between the sphere and plate. With a small resonance amplitude, the interaction force can be Taylor expanded at the rest position \( z_0 \) to the first order using

\[
F_{\text{int}}(z) = F_{\text{int}}(z_0) + (z - z_0) \left( \frac{\partial F_{\text{int}}}{\partial z} \right)_{z = z_0}.
\] (4.2)
Figure 4.5: Amplitude modulation detection. The black dashed line is the free resonant peak of cantilever. The red solid line is the shifted resonant peak of cantilever with the presence of external force. The cantilever is activated at the constant frequency ($f_0$). The external force results in change of a frequency ($\delta f$), amplitude ($\delta A$), and phase.
Figure 4.6: The block diagram of the Constant Amplitude mode of frequency modulation detection. $\delta f$ is the frequency shift of cantilever due to the presence of a force gradient.

Substituting eqn (4.2) into eqn (4.1), the equation of motion will be

$$m \frac{d^2z}{dt^2} + \frac{m\omega_0}{Q} \frac{dz}{dt} + \left( k - \left( \frac{\partial F_{\text{int}}}{\partial z} \right)_{z=z_0} \right) (z - z_0) = F_{\text{int}}(z_0). \quad (4.3)$$

Thus the new resonance angular frequency will be

$$\omega = \omega_0 \sqrt{1 - \frac{1}{k} \left( \frac{\partial F_{\text{int}}}{\partial z} \right)_{z=z_0}}. \quad (4.4)$$

Given $\left( \frac{\partial F_{\text{int}}}{\partial z} \right)_{z=z_0} \ll k$, the resonant angular frequency shift is

$$\delta \omega = \omega - \omega_0 = -\frac{\omega_0}{2k} \left( \frac{\partial F_{\text{int}}}{\partial z} \right)_{z=z_0}. \quad (4.5)$$

If the driving force has a sinusoidal form, the equation of motion has the following form:

$$m \frac{d^2z}{dt^2} + \frac{m\omega_0}{Q} \frac{dz}{dt} + k(z - z_0) = F_0 \sin(\omega t), \quad (4.6)$$
where \( F_0 \) is the driving force and \( \omega \) is the driving angular frequency.

The steady-state solution is proportional to the driving force with a induced phase shift of \( \phi \):

\[
z(t) = z_0 + A(\omega) \sin(\omega t + \phi), \tag{4.7}
\]

where

\[
A(\omega) = \frac{F_0/m\omega_0^2}{\sqrt{(1 - (\omega/\omega_0))^2 + (\omega/(\omega_0 Q))^2}} \tag{4.8}
\]

is the oscillation amplitude and

\[
\phi = \arctan\left(\frac{\omega_0}{(\omega^2 - \omega_0^2) Q}\right) + n\pi \tag{4.9}
\]

is the phase of the oscillation relative to the driving force.

To track the frequency shift, the Frequency Modulation (FM) Mode is used in our experiment. With the FM mode, the oscillation frequency of the cantilever is always adjusted to the new resonance frequency. The phase response of the cantilever instead of the amplitude is used to determine the cantilever’s resonance frequency. According to eqn (4.9), the phase response at the resonant frequency is always 90° independent of the Q factor. Moreover, the phase shift is instantaneous and doesn’t have a response time. The control system using phase response is called the phase-lock loop (PLL).

We use the easyPLL plus system to control the resonance of cantilever with FM mode. The easyPLL plus system contains a controller and a detector. The controller consists of an analog phase shifter, an amplitude measurement, and an amplitude
controller. The detector consists of a voltage controller digital oscillator, a phase detector, and a proportional-integral controller. The setup for PLL controlled resonance with constant amplitude is shown in fig (4.6). The oscillation of the cantilever is measured by the fiber-optic interferometer and converted to electrical signal by the photoreceiver. The voltage controller digital oscillator supplies a reference excitation signal for the cantilever. The phase detector measures the phase difference between input signal and the excitation output. The PI-controller changes the excitation frequency until the phase difference is equal to the phase shift set. The optimum phase shift can be determined from the maximum oscillation amplitude with a constant excitation. The minimum PLL frequency lock range is ±183 Hz and its corresponding
4.6 Argon Ion Gun

The schema for a backfilled Argon ion gun is shown in fig (4.8). An Argon ion gun generates an energetic inert argon ion beam for sputter-etching and cleaning solid surfaces. It is mounted to the main chamber with a 2.75-inch CF flange. To reduce its outgassing rate, the ion gun is wrapped with heat tape and baked to 100 °C for 24 hours to reduce its outgassing rate. The chamber is filled with Argon gas to pressure lower than $5 \times 10^{-5}$ torr. The Argon atoms are ionized by electron impact produced by filaments within the filament ionization chamber. These filaments are arranged off-axis to minimize impurity content of the Argon ion beam. The current of the filaments is slowly increased by adjusting the filament adjust dial on the front panel of the ion gun controller. The filament current has a limit of 2.5 A. Operating at large filament current will reduce its lifetime. Ionized Argon ions are then extracted out from the ionization chamber, accelerated through the focus lens, and directed to the sphere and the plate. The focus adjust dial on the front panel allows the beam size to be varied. Its nominal value is 4. Once the optimum value is set, it can be locked in place and there is no need to change it during the experiment. The beam voltage can be set from 500 V to 2000 V in 500 volt increments. Setting the beam voltage to 2000 V has a faster sputter rate, but also increases the mixing of elements in samples. Thus, the beam voltage of 500 V is used in our experiments. The current measurement resolution of frequency shift is 5 mHz. Its bandwidth is 50 Hz.
Figure 4.8: The Argon ion gun setup.

of Argon ions leaving the ion source ranges from 0 $\mu$A to 10 $\mu$A depending on the setting of ion gun controller. A built-in timer is available on the front panel of the ion gun controller, which allows for repeatable sputtering. In our setup, the ion gun is mounted at 15 cm away from the sphere and plate. The sphere and plate is separated about 1 cm during Argon ion bombardment to best clean the sphere.

4.7 Cantilever Modification

The rectangle tipless cantilever chosen from this experiment is made of silicon. Compared to silicon nitride, silicon cantilevers usually have lower energy dissipation and higher Q factor. The silicon cantilever is n-doped with a resistance from 0.01 to 0.05 Ohm-cm. Since an oxide layer up to 20 nm grows on a silicon surface in an ambient environment, both sides of the cantilever are coated with a 30 nm Au film and a 20 nm Cr sublayer to ensure the good conductance of cantilever. The coating
layer may cause a bending angle of less than $3^\circ$. The cantilever has a 350 µm length, 32.5 µm width and 1 µm thickness. It has a typical spring constant between 0.003 N/m and 0.13 N/m. Its resonance frequency is around 10 kHz. The added mass from the sphere usually decreases its resonance frequency to around 4 kHz. A small spring constant is preferred to achieve high force sensitivity. A high resonance frequency can make the cantilever oscillate at frequencies with low $1/f$ background noise.

To modify the cantilever to be suitable for our experiment, a hollow glass sphere is attached to the free end of the cantilever. Comparing to solid spheres, hollow spheres with less mass can retain much of the Q factor of cantilevers. Careful cleaning procedure is necessary to remove big pieces of debris on sphere surface. First, spheres are sonicated in 30% hydrogen peroxide solution ($\text{H}_2\text{O}_2$) for 20 minutes. Only spheres that float on the top are chosen for further cleaning. These spheres are washed with acetone, methanol/IPA and DI water in that order. Spheres are then transferred to unpolished rough silicon plates and dry out in a vacuum oven. A rough plate should be used here instead of a smooth surface. As shown in fig (4.9), when wet spheres dry on a smooth surface, many spheres gather together due to the electrostatic force. At the points of contact, dirty residues stick on the sphere surfaces. It is very hard to pick a single clean sphere. In the case of a rough surface, the friction will isolate each spheres. It also minimizes the contact of the spheres to the plate.

The next step is to pick an appropriate sphere and attach it to a tipless cantilever. The following process is done with the help of an optical microscope. A custom
Figure 4.9: Wet spheres on a smooth/rough surface.

A manipulator designed for this purpose is shown in fig (4.10). This manipulator can handle spheres with radius from 2 µm to 100 µm. Solid spheres with radius of 2 µm are useful for stiffness measurements in biophysics experiments. It is built by a heavy holder, a three direction manual translation stages, and two cleaved optical fibers. One optical fiber sticks a little electrical and thermal conductive silver epoxy to the end of cantilever. With too much epoxy, they may move to the back side of cantilever and lower the reflection of laser. The other optical fiber picks a sphere according to its smoothness on the surface, sphericity and radius. This optical fiber is carefully cleaned with Acetone, IPA and DI water and dried with Nitrogen gas before use. The top side of the sphere will be the effective interaction area with the plate in following force measurements. The optical fiber should avoid touching the top of sphere during the pick. For Casimir experiments, hollow glass spheres with radius between 40 µm and 60 µm are usually selected. Hollow glass spheres are used due to their reduced mass. A smaller sphere will fail the validation condition of
the PFA applied in our experiments. A larger sphere tends to have a rough surface with many defects. The smoothness of a sphere surface can be estimated from the reflection of light. A thinner sphere has a higher probability of being smoothness. If the time gap between the mixing of two epoxy precursors and putting the sphere on cantilever is larger than 10 minutes, the silver epoxy may have already hardened and the sphere will easily fall off from the cantilever. After the sphere is mounted on the cantilever, the radius of the sphere is measured using software from Nikon for preliminary analysis. To achieve a full cure of silver epoxy, the cantilever with sphere is heated up to 65 °C in a vacuum oven for 15 minutes.

The sphere-cantilever assembly is coated with a gold film at a vacuum of $10^{-6}$ torr using electron-beam physical vapor deposition. The electron beam is directed by magnetic field towards a crucible, which contains 99.999% Au source. The energy
of the electron beam causes the Au source to start evaporating. The cantilever is mounted on the top of Au source and rotated horizontally to create a uniform coating layer on the sphere surface. Only the end of the cantilever including the sphere is exposed to Au vapor in order to reduce the bending and preserve its Q-factor. The coating starts after the adsorbates layer on the Au source surface is completely eliminated and coating rate becomes stable at 2 Å/s. The Au vapors travel around 50 cm from the crucible to the target. The long path allows a sufficient diffusion of Au vapor before reaching the sphere’s surface. The final Au film should have a thickness of at least 100 nm. The Au film is characterized with the commercial AFM after the Casimir force measurements.

A 10 mm diameter uncoated fused silica window is used as the substrate for the plate. It is also coated with an Au film with electron-beam physical vapor deposition.

The modified cantilever and plate are then treated with UV/Ozone for 10 minutes to remove minor organic containments. Right after that, it will be mounted on the specially fabricated cantilever holder. After alignment with the 1550 nm laser interferometer and pumping out the air, it will be ready for measurements. A SEM image of an Au coated modified cantilever is shown in fig (4.11).
Figure 4.11: A SEM image of Au coated sphere on a tipless cantilever. Its diameter is $121.2 \pm 2.0 \, \mu m$. 
Chapter 5

Experiment Procedures and Results
5.1 Measurement of Frequency Shift

For an oscillating harmonic oscillator, such as the cantilever in our case, the resonance frequency shift due to an external force gradient has the following form:

\[
\delta f = -\frac{f_0}{2k} \frac{\partial F_{\text{int}}}{\partial z}.
\]  

(5.1)

Here \( \delta f \) is the frequency shift to its free resonance frequency, \( f_0 \) is the free resonance frequency of the cantilever, \( k \) is the spring constant, and \( \frac{\partial F_{\text{int}}}{\partial z} \) is the interaction force gradient between the sphere and plate.

The separation between the sphere and plate is controlled by the piezo tube. When the plate driven by the piezo tube approaches the sphere, the Casimir force gradient and electrostatic force gradient between the sphere and plate causes the frequency shift in the cantilever. Both the interaction force gradient and frequency shift increases as the plate gets closer to the sphere. The frequency shift is reflected in the interference signal recorded by fiber-optic interferometer and is tracked by the PLL. The \( \delta f \) are output by the PLL as voltage signals. They are converted to frequency using the ratio of 18.31 Hz/V when the lock range of \( \pm 183 \) Hz is chosen. Finally, the dependence of frequency shift on the voltage applied to the piezo tube is recorded by the computer.

Before the plate contacts the sphere, the voltage applied to the piezo tube decreases and the plate retracts from the plate. Only the frequency shift on the extension is used for analysis. The peak-to-peak measurement resolution of the PLL is 5
Figure 5.1: The frequency shift as a function of voltage applied to piezo tube. The plate is mounted on the piezo tube. Positive voltages extend the piezo tube to the sphere and negative voltages retract the piezo tube from the plate.
Figure 5.2: The noise level of frequency shift at large separation of sphere and plate.

Limited by the finite Q factor of the cantilever and the optical noise of the fiber-optic interferometer, the standard deviation of frequency shift noise level in our experiments is around 7.2 mHz.
5.2 Calibration of Piezo Movement

5.2.1 Displacement of Plate in a Single Scan

The frequency shift measured in fig (5.1) depends on the voltage applied to the piezo tube. To convert the applied voltages to displacements, it is necessary to calibrate the piezo tube. This is accomplished using the fiber-optic interferometer on top of the plate. The movement of plate changes the gap size formed by the cleaved fiber end and the plate surface leading to an interference fringe. Making a fitting of the interference fringe, the sensitivity coefficients up to high orders can be obtained.

The interference signal from the the photodiode is given by

\[ P = P_0(1 - V \cos\left(\frac{4\pi d}{\lambda}\right)), \]

where \( \lambda \) is the laser wavelength and \( d \) is the fiber-to-cantilever spacing. \( P_0 = \frac{P_{\text{max}} + P_{\text{min}}}{2} \) is the midpoint value. \( V \) is equal to \( \frac{P_{\text{max}} - P_{\text{min}}}{P_{\text{max}} + P_{\text{min}}} \). Expanding \( d \) as a function of \( V \), the following fitting function can be obtained

\[ P = P_0(1 - V \cos\left(\frac{4\pi}{\lambda}(C_1V + C_2V^2 + C_3V^3 + \phi))\right)). \]

It is necessary to make a fitting of the interference fringe to the third order in real-time, because the nonlinear piezoelectric coefficient coming from hysteresis depends on the magnitude, polarity, and frequency of applied voltage. Also the environment temperature will affect the movement of piezo. In our experiment, the piezo moves
Figure 5.3: The constructive and destructive interference fringe due to displacement of plate.
Figure 5.4: The quadrature points of fiber-cantilever spacing. They are the most sensitive positions, where the interference signal response is maximized.

about 9.86 V/nm. Given that the triangle voltage applied to the piezo tube has a peak-to-peak value of around 220 V, the measurement range is around 2.2 µm.

5.2.2 Quadrature Point of Fiber-Cantilever Spacing

A change of the fiber-cantilever spacing also leads to interference fringe as shown in fig (5.4). If the fiber-cantilever spacing is a multiple of λ/4, the interference signal
will be found on either $P_{\text{max}}$ (the mountain) or $P_{\text{min}}$ (the valley), where a small change of fiber-cantilever spacing results in no change of interference signal. Thus, to increase the sensitivity of our experiment, we need to avoid these blind positions and keep the fiber-cantilever spacing at quadrature points, like $\lambda/8$ and $3\lambda/8$. At quadrature, the interference signal response ($\Delta P$), for small changes in distance ($\Delta d$), is given by

$$\Delta P = P_0 \frac{4\pi V \Delta d}{\lambda}.$$  \hfill (5.4)

### 5.2.3 Deflection of Cantilever

As the plate gets close to the sphere, the attractive force causes static deflection of the cantilever and change in the sphere-plate separation. The deflection of the cantilever depends on the spring constant of the cantilever and the magnitude of the attractive force.

Since the frequency shift is a function of the force gradient, the bending of the cantilever can be derived by integrating the frequency shift. The force gradient as a function of the frequency shift given by eqn (5.1):

$$\frac{\partial F_{\text{int}}(z)}{\partial z} = -\frac{2k}{f_0} \delta f.$$  \hfill (5.5)

By integrating both ends of equation, the interaction force is

$$F_{\text{int}}(z) = -\frac{2k}{f_0} \int_{\infty}^{z} \delta f dz,$$

$$= -\frac{2k}{f_0} \int_{z_{\text{far}}}^{z} \delta f dz + F_{\text{Cas}}(z_{\text{far}}) + F_{\text{ele}}(z_{\text{far}}).$$  \hfill (5.7)
Here $z_{\text{far}}$ is the largest separation measured. It is around 2.2 $\mu$m. At this distance, the Casimir force is usually negligible. Thus, the interaction force is

$$F_{\text{int}}(z) = -\frac{2k}{f_0} \int_{z_{\text{far}}}^{z} \delta f dz + F_{\text{ele}}(z_{\text{far}}).$$

(5.8)

By dividing the spring constant of the cantilever on both ends of equation, the bending of the cantilever as a function of distance can be derived:

$$\delta d(z) = -\frac{2}{f_0} \int_{z_{\text{far}}}^{z} \delta f dz + \frac{F_{\text{ele}}(z_{\text{far}})}{k}.$$  

(5.9)

For a cantilever with a spring constant of 0.1 N/m, at a distance of 230 nm, the bending of the cantilever caused by the Casimir force and electrostatic force of 50 mV is about 0.25 nm.

To correct this deflection, another method uses the DC value of interference fringe as feedback signal for a PID loop to keep the cantilever-fiber spacing constant.

### 5.2.4 Drift of Piezo

To measure the force gradient as a function of distance, multiple scans with different sphere-plate voltages applied are conducted to calculate the spring constant of the cantilever and the closest sphere-plate separation. To compare the frequency shift from different scans, the mechanical drift of piezo tube and two piezo actuators needs to be compensated.

The drift shown in fig (5.6) is calculated from 10 scans of the frequency shift with
Figure 5.5: Horizontal shift of the frequency shift curves due to drift of the piezo tube and two piezo actuators. The same voltage between the sphere and plate is applied to all scans. The arrow shows the drift direction. The time interval between two continuous scans is 100 s. The drift distances are calculated at the same frequency shift curve of different scans. To reduce the random error due to the frequency shift noise, the final drift rate is calculated from the average drift distance of multiple frequency shifts.
Figure 5.6: Mechanical Drift of piezo tube and piezo actuators. Each data point comes from the horizontal position of one frequency shift scan. The slope of the linear fit is used for thermal drift correction.
the same voltage applied to the plate. The mechanical drift of piezo tube and piezo actuators causes horizontal shift of different scans. Each data point in fig (5.6) comes from the horizontal position of one scan. The rate of the mechanical drift depends on the environment temperature. In our experiment, it ranges from 0.002 nm/s to 0.02 nm/s.

5.3 Calibration of the Long Range Electrostatic Force

The frequency shift we have talked so far is due to both the Casimir force gradient and the electrostatic force gradient:

$$\delta f(z, V) = -\frac{f_0}{2k} \frac{\partial F_{\text{int}}(z, V)}{\partial z}$$  \hspace{1cm} (5.10)

$$= -\frac{f_0}{2k} \frac{\partial F_{\text{Cas}}(z)}{\partial z} - \frac{f_0}{2k} \frac{\partial F_{\text{ele}}(z, V)}{\partial z}$$  \hspace{1cm} (5.11)

$$= \pi C_0 P_{\text{Cas}}(z) - \frac{f_0}{2k} \frac{\partial F_{\text{ele}}(z, V)}{\partial z}. \hspace{1cm} (5.12)$$

Here the proximity force approximation is applied for sphere-plate geometry. $P_{\text{Cas}}$ is the Casimir pressure between the sphere and plate. $C_0$ is equal to $f_0R/k$. The frequency shift depends on both $z$ and $V$ as shown in fig (5.7).

The electrical capacitance in a sphere-plate configuration is given by [110]

$$C(z) = 4\pi \varepsilon_0 R \sinh \alpha \sum_{n=1}^{\infty} \frac{1}{\sinh(n\alpha)}. \hspace{1cm} (5.13)$$
Figure 5.7: The frequency shift as a function of sphere-plate separation and voltage.
Table 5.1: Constants for capacitance in a sphere-plate configuration.

<table>
<thead>
<tr>
<th>$k$</th>
<th>-1</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
</tr>
</thead>
<tbody>
<tr>
<td>$c_k$</td>
<td>0.5</td>
<td>-1.18260</td>
<td>22.2375</td>
<td>-571.366</td>
<td>9592.45</td>
<td>-90200.5</td>
<td>383084</td>
<td>-300357</td>
</tr>
</tbody>
</table>

where $\cosh \alpha = 1 + z/R$. In the limiting case of small separations, $z/R \ll 1$, an approximate expression for the capacitance is [111]

$$C(z) \approx 2\pi \epsilon_0 R \left( \frac{R}{z} + \ln 2 + \frac{23}{20} + \frac{\theta}{63} \right), \quad (5.14)$$

where $0 < \theta < 1$.

With a voltage $V$ applied to the plate, the electrostatic energy is given by

$$E_{ele}(z, V) = -\frac{1}{2} C(z)(V - V_0)^2 \quad (5.15)$$

$$= -2\pi \epsilon_0 R (V - V_0)^2 \sinh \alpha \sum_{n=1}^{\infty} \frac{1}{\sinh(n\alpha)}. \quad (5.16)$$

Here $V_0$ is the residual potential between the sphere and the plate. The electrostatic force is given by [40]

$$F_{ele}(z, V) = -\frac{\partial E_{ele}(z, V)}{\partial z} \quad (5.17)$$

$$= 2\pi \epsilon_0 (V - V_0)^2 \sum_{n=1}^{\infty} \text{csch} n\alpha (\coth \alpha - n \coth n\alpha). \quad (5.18)$$

A precise expansion for eqn (5.17) is given by [112]

$$F_{ele}(z, V) = -2\pi \epsilon_0 (V - V_0)^2 \sum_{k=-1}^{6} c_k \left( \frac{z}{R} \right)^k. \quad (5.19)$$

where $c_k$ are constants shown in table (5.1).

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Thus, the electrostatic force gradient is

$$\frac{\partial F_{\text{ele}}(z,V)}{\partial z} = -2\pi \epsilon_0 R (V - V_0)^2 \left( -\frac{c_{-1}}{z^2} + \sum_{k=1}^{6} \frac{k c_k}{R^2} \left( \frac{z}{R} \right)^{k-1} \right).$$ (5.20)

Substituting eqn (5.20) into eqn (5.12), we have

$$\delta f(z,V) = \pi C_0 P_{\text{Cas}}(z) + \beta(z)(V - V_0)^2,$$ (5.21)

where

$$\beta(z) = \pi \epsilon_0 C_0 \left( -\frac{c_{-1}}{z^2} + \sum_{k=1}^{6} \frac{k c_k}{R^2} \left( \frac{z}{R} \right)^{k-1} \right).$$ (5.22)

Here $\beta(z)$ is related to the second derivative of the sphere-plate capacitance. Therefore, the Casimir pressure is given by

$$P_{\text{Cas}}(z) = \frac{1}{\pi C_0} (\delta f(z,V) - \beta(z)(V - V_0)^2).$$ (5.23)

With the calibration of the piezo movement discussed in last section, we can plot the frequency shift as a function of voltage applied to the plate at every sphere-plate separation $z$. The position of the vertex of the parabola in fig (5.8) is $(V_0, \pi C_0 P_{\text{Cas}}(z))$, and the curvature is $\beta(z)$. By fitting the curvature to eqn (5.22), the value of $C_0$ and $z_0$ can be determined. Here $z_0$ is the closest separation of the sphere and plate. The Casimir pressure as a function of distance, $P_{\text{Cas}}(z)$ is then determined.
Figure 5.8: The frequency shift as a function of voltage applied to the plate at a given $z$. 
Figure 5.9: $\beta(z)$ as a function of $z_p$. Here $z_p = z - z_0$ is the displacement of the piezo tube, and $z_0$ is the closest sphere-plate separation. The values of $z_0$ and $C_0$ are determined from the fitting of the $\beta(z)$ curve to its theoretical equation (5.22). The distance independence of $z_0$ and $C_0$ are checked by varying the fitting end of the $\beta(z)$ curve.
5.4 Forces due to Spatial Variations of Surface Potential

The surfaces of real metals are not equipotential due to different crystallographic directions and surface adsorbates. The spatial variations of surface potential induce extra electrostatic interaction in addition to the basic long range electrostatic force from voltage applied to the plate. The magnitude of this additional force strongly depends on the size and size distribution of the electrical patches. As shown in fig (5.10), electrical fields produced by small patches are not able to reach the other surface. Only the patches that are large compared to the separations between two surfaces can induce force.

For sphere-plate configuration, the effective interaction area has distance depen-
(a) Effective interaction area at large sphere-plate separations.

(b) Effective interaction area at small sphere-plate separations.

Figure 5.11: Effective interaction area between sphere-plate changes with separations.

dence:

\[ A_{\text{eff}} = 2\pi Rz. \quad (5.24) \]

When the sphere-plate separation decreases from 2 \( \mu \text{m} \) to 0.2 \( \mu \text{m} \), the effective area \( A_{\text{eff}} \) decreases from \((27 \ \mu\text{m})^2\) to \((9 \ \mu\text{m})^2\). The change of effective area will then lead to a distance dependence of the measured residual potential when large patches and varying potentials are present on sample surfaces.

To study the magnitude of the patch effects, the patches on a conducting surface can be modeled as a layer of varying dipole moments overlying to a perfectly conducting surface, as shown in fig (5.12).

Assume the Fourier coefficients uniformly lie in two-dimensional \( k \) space with \( k_{\text{min}} < k < k_{\text{max}} \) and that spatial distributions of potentials are random and uncorre-
Figure 5.12: Two grounded perfectly conducting surfaces each with a dipole layer. Layer 1 and 4 are two perfectly conducting equipotential surfaces. The spatial variations of surface potential on real surfaces are modeled as layer 2 and 3, which contain varying dipole moments. Adapted with permission from American Physical Society (ref [47]), copyright (2003).
lated. Here \( k = 2\pi/\lambda \), \( \lambda \) is the size of a patch. Then the force per unit area between two parallel plates is [47]:

\[
P_p(z) = -\frac{2\epsilon_0\sigma_v^2}{k_{\text{max}}^2 - k_{\text{min}}^2} \int_{k_{\text{min}}}^{k_{\text{max}}} \frac{k^3}{\sinh^2 kz} dk.
\]

(5.25)

Here \( \sigma_v^2 \) is the variance of surface potentials, given by [47]

\[
\sigma_v^2 = \frac{1}{S} \int \int V^2(r) dr.
\]

(5.26)

For Au, the work functions for different crystallographic surface orientations \(< 100 \), \(< 110 \) and \(< 111 \) are 5.47, 5.37 and 5.31 eV, respectively. Assuming equal areas of three crystallographic planes, the variance is approximately \((81 \text{ mV})^2\).

The distance dependence of pressure due to patch effect is only determined by the integral part of eqn (5.25), that is primarily determined by \( k_{\text{min}} \) and \( k_{\text{max}} \). Specifically, \( \sinh kz \) approaches to zero when \( kz \ll 1 \) and the effect patch forces rapidly increases.

## 5.5 Measurement of the Casimir Pressure before Argon Ion Cleaning

### 5.5.1 Temporal Dependence of Residual Potential

In order to study the effect of electrostatic patches induced by surface adsorbates, the cantilever and plate were intentionally exposed to \(10^{-1}\) torr. Only the dry scroll pump was running to maintain the vacuum. After 24 hours, the turbo pump and the
Figure 5.13: Residual potential vs time at a pressure of $10^{-8}$ torr.
ion pump were turned on to bring the pressure of the chamber down to $10^{-8}$ torr. The residual potential at the separation of 250 nm as a function of time was measured. From the fig (5.13), the variation of residual potential in 10000 s at the pressure of $10^{-8}$ torr is around 1 mV. Over a larger time span, the variation of residual potential can be $\pm$ 2 mV.

### 5.5.2 Casimir Pressure with the Distance Independent Residual Potential

With surface adsorbates on sample surfaces, it is still possible to get distance independent residual potential as shown in fig (5.15). As we discussed in the last section, when the residual potentials are independent of distance, the electrical fields induced by patch effects are averaged out and cause no additional force within the experimental resolution.

The measurements were conducted after the vacuum pressure got stable. The free resonance frequency of the cantilever is 4427.59 Hz and its radius is 60.6 $\mu$m. A total of 21 scans of the frequency shift were conducted. The first 11 scans are applied with voltages between -66 mV to 134 mV at a step of 20 mV. The next 10 scans are applied with the same voltage of 34 mV, from which the drift rate was found to be $0.00231 \pm 0.00017$ nm/s, as shown in fig (5.14). The triangle voltage applied to the piezo tube was 220 mV, which corresponds to a displacement of 2170 nm. The residual potential was measured by fitting the frequency shift from the first 11 scans.
Figure 5.14: Drift rate of sphere-plate separation. The drift rate is found to be $0.00231 \pm 0.00017$ nm/s. The positive sign means the sphere-plate separation decreases during measurements.

to a parabola. It was found to be $34.97$ mV and is distance independent as shown in fig (5.15).

The closest sphere-plate separation calculated by fitting the $\beta(z)$ curve to its theoretical expectation is $214.1 \pm 0.4$ nm and the constant $C_0$ is $1.124 \pm 0.005$ Hz \cdot m$^2$/N. The Casimir pressure as a function of distance was obtained by subtracting the electrostatic force gradient from the total force gradient:

$$P_{\text{Cas}}(z) = \frac{1}{\pi C_0} (\delta f(z, V) - \beta(z)(V - V_0)^2).$$  \hspace{1cm} (5.27)
From each scan, a Casimir pressure curve can be obtained. The averaged Casimir pressure of 21 scans is shown in fig (5.16) and fig (5.17). The error bars are plotted at 67% confidence level. Our data fit well with the plasma model and the Drude model is excluded at separations from 235 nm to 500 nm.

The Casimir pressure at large distance was used to find the standard deviation of the background noise to be around 0.5 mPa. The systematic error of our experiment, shown in fig (5.19) is determined by using the equation

$$\delta P = \sqrt{\left(\frac{\delta C_0}{C_0}\right)^2 + \left(\frac{\delta f}{f}\right)^2}.$$  

(5.28)
Figure 5.16: The Casimir pressure curve with distance independent residual potential before Argon ion cleaning from 235 nm to 350 nm. The red and green lines are the theoretical Casimir pressure using the Plasma model and the Drude model, respectively.
Figure 5.17: The Casimir pressure curve with distance independent residual potential before Argon ion cleaning from 350 nm to 500 nm. The red and green lines are the theoretical Casimir pressure using the Plasma model and the Drude model, respectively.
Figure 5.18: The Casimir pressure at large distance. The standard deviation of the random noise is found to be 0.5 mPa.
Figure 5.19: Systematic error of Casimir pressure measurements. At large separations, the systematic error is constant at 2.1 mPa. It increases at close distance.
5.5.3 Casimir Pressure with the Distance Dependent Residual Potential

When the sample surfaces are covered by many surface adsorbates, most of the time (13 data sets out of 14), the residual potential has measurable distance dependence, as shown in fig (5.20). The measurements were made on slightly different locations. Note, when the residual potential has distance dependence, the measured residual potential is actually the minimizing potential, which might be slightly different from the actual residual potential. Additional pressure due to potential variations is expected. The long range electrostatic pressure is subtracted from the total pres-
Figure 5.21: Casimir pressure and residual electrostatic pressure due to Large electrostatic patches.
sure by fitting the total frequency shift, $\delta f(z, V)$, to different applied voltages. The vertex of the fitting is the sum of Casimir pressure and electrostatic patch pressure. As shown in fig (5.21), when the residual potential is tilted, the contribution of electrostatic patch effect can be around 10% of Casimir pressure. The electrostatic patch pressure is obtained by subtracting the theoretical Casimir pressure, both the Plasma model and the Drude model.

In our experiments, we found the magnitude of electrostatic patch pressure depends on the distance dependence of residual potential, as shown in fig (5.22) with the Plasma model used and in fig (5.23) with the Drude model used. The figure is plotted in log scale to find the distance dependence of electrostatic patch pressure. Here the plasma model is used for the Lifshitz theory. The distance dependences of the residual potential are different of different colored electrostatic patch pressures. Their slopes range from 2.5 to 4.5 $\mu$V/nm. The distance dependence of electrostatic patch pressures in this figure are similar, and depend only on the choice of $k_{\text{min}}$ and $k_{\text{max}}$ according to eqn (5.25). Thus, it is a reasonable assumption that the distribution of electrostatic patch sizes is similar. The two black dashed lines are calculated using eqn (5.25). The small limit and large limit of electrostatic patch sizes are 0.2 $\mu$m and 5 $\mu$m for both black dashed lines. This results in $k_{\text{max}} = 2\pi/(0.2 \mu m)$ and $k_{\text{min}} = 2\pi/(5 \mu m)$. The upper black dashed line has the larger potential variation, $\sigma_v^2$, equal to $(130 \, mV)^2$, and the lower line has the smaller potential variation of
Figure 5.22: The residual pressure with the use of the Plasma model. The colored solid lines are electrostatic patch pressure obtained by subtracting the Plasma model from the experimental pressure curves with different slopes of residual potential. Two dashed black lines are calculated using theoretical eqn (2.29). $k_{\text{min}} = 2\pi/(5 \, \mu m)$, $k_{\text{max}} = 2\pi/(0.2 \, \mu m)$ are used for both black dashed lines. The upper black dashed line has a higher potential variation of $\sigma_v^2 = (130 \, mV)^2$ and the lower one has $\sigma_v^2 = (40 \, mV)^2$. 
Figure 5.23: The residual pressure with the use of the Drude model. The colored solid lines are electrostatic patch pressure obtained by subtracting the Drude model from the experimental pressure curves with different slopes of residual potential. Two dashed black lines are calculated using theoretical eqn (2.29). $k_{\text{min}} = 2\pi/(5 \, \mu\text{m})$, $k_{\text{max}} = 2\pi/(0.2 \, \mu\text{m})$ are used for both black dashed lines. The upper black dashed line has a higher potential variation of $\sigma^2_v = (130 \, \text{mV})^2$ and the lower one has $\sigma^2_v = (40 \, \text{mV})^2$. 
Figure 5.24: The residual pressure divided by square of the residual potential slope as a function of distance.

\((40 \text{ mV})^2\). The scale of both black dashed lines is around \(1/z^{4.1}\).

Since the magnitude of the electrostatic patch pressure is related to the slope of the residual potential, we divided each electrostatic patch pressure by the square of the slope of residual potential. This results in fig (5.24). The lines almost collapse to one. Thus, we conclude that the square of the slope of residual potential is related to the variance of the surface electrostatic patches.
5.6 Measurement of Casimir Pressure with Argon Ion Cleaning

One source of residual potential is the surface adsorbates. Thus, removal of surface adsorbates should decrease the magnitude of the residual potential. We use an Argon ion gun to clean the surfaces of the sphere and plate. The energetic Argon ion beam accelerated and directed to sample surfaces. The surface adsorbates attached on sample surfaces are removed by the heavy Argon atoms.

The Argon ion gun was operated at a pressure of $2 \times 10^{-5}$ torr. The filament
Figure 5.26: Distance independent residual potential after Argon ion cleaning.
current is 2.1 A. The focus setting is 4. The Argon gas beam was accelerated to 500 eV. The Argon ion current leaving the anode is 10 µA. As shown in fig (5.25), the residual potential decreases exponentially with Argon ion cleaning. It takes about 90 minutes to reduce the residual potential from 30 mV to almost 0 mV. It indicates that the surface adsorbates are the main source of residual potential.

After the Argon ion cleaning, there is a much higher chance, around 50%, to get the residual potential that is independent of distance, as shown in fig (5.26). The voltages applied to the plate ranged from -47 mV to 53 mV at steps of 10 mV for
the first 11 scans and 3 mV for the second 10 scans. The average residual potential was measured to be \(1.3 \pm 1.2\) mV. The drift rate was found to be \(-0.00536 \pm 0.00038\) nm/s, as shown in fig (??). The averaged Casimir pressure of 21 scans is shown in fig (5.27) at distance from 235 nm to 350 nm and fig (5.29) at distance from 350 nm to 550 nm.

A typical AFM image of our Au coated plate is shown in fig(5.30). The patch size ranges from 10 nm to 150 nm. The potential variance for Au surfaces with uniformly distributed three crystallographic orientations is \((81 \ mV)^2\). The patch pressure calculated using these parameters is shown in fig (5.31). As we can see
Figure 5.29: Casimir Pressure at distance from 350 nm to 550 nm after Argon ion cleaning.
Figure 5.30: An AFM image of Au coated plate using electrical beam evaporation.

From the figure, the patch pressure produced by clean Au surfaces are negligible at separations from 200 nm to 400 nm.
Figure 5.31: Comparison of theoretical difference, experimental error and patch pressure by clean Au surfaces. The dashed line is the difference between Plasma model and Drude model at 300 K. The solid line is our experimental error. The dotted line is the patch pressure produced by clean Au surfaces.
Chapter 6

Conclusion and Future Work
6.1 Conclusion

We have investigated the role of electrostatic patches in the precision Casimir force measurement in the sphere-plate configuration. We first exposed the sphere and plate in low vacuum for 24 hours. Due to the surface adsorbates on Au surface, the residual potential is around 31 mV. The surface adsorbates will also change the size distribution and potential variation of electrostatic patches. For a clean surface, the electrostatic patches will correspond to the grain size of the polycrystal Au surface and originate from the different work functions of the Au crystal orientations. The role of the electrostatic patches can be identified through the distance dependence of the applied potential required to minimize the electrostatic force from equipotential surfaces. When the residual potential has a distance dependence, we find the residual electrostatic force cannot be fully compensated by applying voltage to the plate. It is because the presence of large electrostatic patches within the effective interaction area between the sphere and plate. Even without Argon ion cleaning, for a small fraction of experiments, the residual potential has no distance dependence within our experimental error. After sufficient Argon ion cleaning, the residual potential is reduced to 1.3 ± 1.2 mV and no distance dependence of minimizing potential required to compensate the electrostatic force is found within our experimental error. With the distance independence residual potential, the experimental Casimir pressure is in a good agreement with the generalized plasma model applied to the Lifshitz theory from 235 nm to 450 nm. The electrostatic patch pressure of clean Au surfaces based on
grain size and work function differences calculated using theoretical equation from [47] is found to be negligible within our experimental error.

6.2 Future Work

In dynamic force measurement, the force gradient due to the resonance frequency shift has this form:

\[
\frac{\partial F(z)}{\partial z} = -2k \frac{\delta f}{f_0}. \tag{6.1}
\]

For an undamped harmonic oscillator, we have

\[
f_0 = \frac{1}{2\pi} \sqrt{\frac{k}{m}}. \tag{6.2}
\]

Thus, we have

\[
\frac{\partial F(z)}{\partial z} = -4\pi \sqrt{mk} \delta f. \tag{6.3}
\]

Therefore, to improve our sensitivity, small spring constant, light sphere, and high frequency shift resolution is preferred.

For a rectangle cantilever, the spring constant is given by

\[
k = \frac{E \cdot w (t/l)^3}{4}, \tag{6.4}
\]

where \(E\) is the Young’s modulus, \(w\) is the width, \(t\) is the thickness and \(l\) is the length. Reduce the thickness of silicon cantilever might be worth trying.
However, with a small spring constant, the resonance frequency also decreases. Considering the background $1/f$ noise, a cantilever with the resonance frequency between 500 Hz and 1.5 kHz may be the best choice.

In our experiment, the resolution of $\delta f$ is mainly limited by the fiber-optic interferometer. The interference signal response of fiber-optic interferometer to the oscillation of cantilever is given by

$$\Delta P = P_0 \frac{4\pi V \Delta d}{\lambda},$$

where $P_0 = (P_{\text{max}} + P_{\text{min}})/2$ is the midpoint value. $V$ is equal to $(P_{\text{max}} - P_{\text{min}})/(P_{\text{max}} + P_{\text{min}})$. Using a grin fiber lens or closer fiber-cantilever spacing will lead to larger $V$ and increase our resolution in $\delta f$. 
Bibliography

[1] U. Mohideen and A. Roy, “Precision measurement of the casimir force from 0.1 to 0.9 \( \mu \) m,” *Physical Review Letters*, vol. 81, no. 21, p. 4549, 1998.


Appendix A

Stiffness Measurement with AFM

AFM combines the advantages of high force sensitivity and the ability of being operated in liquid environment. It provides a unique window to study the surface mechanical properties of biological samples, which play important roles in many biological processes. We use a commercial AFM to study the stiffness of basement membrane and living cells in phosphate-buffered saline (PBS) buffer [106, 118, 119]. Changes in stiffness of basement membrane and living cells are used to evaluate the status of cells. Instead of sharp tips, colloidal probes consisting a solid silica sphere of 5 µm on the end of cantilever are used. The AFM is operated in Force Curve Mode with a maximum applied force of 8 nN. This is kept very brief as it is outside the main subject of the thesis.