Customized silicon cantilevers for Casimir force experiments using focused ion beam milling

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Abstract. Higher sensitivity cantilevers will lead to exploration of new phenomena in the Casimir effect. We have used focused ion beam milling to reduce the width of a commercial single crystal, rectangular-shaped silicon cantilevers with a massive Cr/Au-coated–hollow sphere attached at their free end. Theoretically these milled and modified cantilevers should have better Casimir force sensitivity than their non-milled counterparts. In this preliminary report however only 1 out of 4 modified cantilevers were found to have a higher force sensitivity. Future studies will be needed to determine the general applicability of focused ion beam milling for force sensitivity improvements in comparison to the complete nanofabrication of cantilevers.

1. Introduction
Many aspects of the Casimir effect [1] such as the temperature and shape dependence remain to be experimentally explored [2]. In the standard Casimir effect, the force between two ideal metal plates parallel to each other is given by:

\[ F_{\text{min}} = \left( \frac{\pi^2 \hbar c}{240} \right) \frac{1}{d^4} \]  

(1)

This effect plays an important role in many areas of fundamental and applied physics [2]. It has found applications in grand unification theories were non-Newtonian forces are predicted at distances where the Casimir force is currently being measured [2]. In fact, the Casimir force has already been used as a constraint for these predicted forces [2, 3]. Theorists have also predicted that the Casimir phenomenon is responsible for repulsive forces in non-parallel-plate geometries, such as an ideal spherical metal shell [4]. Technologically, the continuous miniaturization of micro electro mechanical systems (MEMS) has decreased their characteristic lengths to submicrometer distances. In this region, the Casimir force is the dominant forces for neutral devices and thus could be very detrimental to their

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function due to the resultant friction, dissipation and adhesion issues [5]. The Casimir effect has also been measured using the MEMS technique [6].

In the last ten years, various experimental techniques have been utilized to precisely measure this effect in different materials, geometries and conditions [See Ref. 2 for a list of previous measurements]. Most experiments measure the Casimir force using the sphere-plate geometry because of the difficulty to precisely align surfaces in parallel-plate experiments [3,6,7] (note that there has been one recent measurement using parallel surfaces [8]). For the sphere-plate geometry, the proximity force approximation is used to obtain the Casimir force, and thus places ultimate limits on its precision. However, these limits are beyond the sensitivity of the measurements to date. We pioneered the technique of measuring the Casimir force using an atomic force microscope (AFM) in which the cantilever has a metal-coated polystyrene sphere attached to its free end [7]. These spheres are perfectly smooth, and have much less than 10^{-3} deviation from sphericity. We have used static AFM techniques to measure the mentioned force between the sphere and a metal coated plate [7]. The dynamical AFM (DAFM) technique which measures the force gradient between the sphere and substrate is expected to be more sensitive [9-13]. More sensitivity implies higher precision in the Casimir force measurements at the explored sphere to plate distances. In addition, the increased sensitivity will allow us to investigate separation distances usually inaccessible with the static AFM technique. Thus, it might be possible to investigate the thermal corrections to the Lifshitz theory of the Casimir effect which are a large fraction of the total force at sphere plate separations exceeding one micron.

Static AFM and DAFM techniques use cantilevers as probes for interaction forces. According to theoretical models, rectangular cantilevers which are thin, long, and narrow will produce highly sensitive force probes [9,10]. Many experimental groups have utilized this recipe to measure the spin of small ensembles of electrons or nuclei [12]. Researchers have also used these types of cantilevers to make noncontact friction measurements [11], and non-Newtonian force measurements [13]. These experiments were mostly done with cantilevers fabricated in a traditional nanofabrication facility. The force resolution obtained with these cantilevers surpasses by orders of magnitude the resolution currently obtained in Casimir force experiments [6-8]. However, these cantilevers need specialized steps such as critical point drying, etching, annealing etc. to achieve their sensitivity. These steps might not be compatible with the configuration of cantilevers necessary for Casimir force measurements. As mentioned above, to measure the Casimir force we use the sphere-plate geometry, where we attach a Cr/Au-coated sphere to a single crystal silicon (Si) cantilever, and measure the force between the sphere and a Cr/Au plate. The sensitive cantilevers, used by others [10-13] might not be suitable for our experiment, because the metal coated sphere is large and massive. The cantilevers might break or the internal stresses induced will decrease force sensitivity. They might also be difficult to align with our detection system or reduce the signal to noise ratio because of their excessive curvature on attachment of the sphere. A simpler and reliable way to achieve both the above unprecedented force sensitivities and meet the configuration requirements of Casimir force measurements, would be by sculpting the cantilever with a focused ion beam (FIB) milling technique [14]. By performing the sculpting after attachment of the sphere, we should be able avoid distortions of the cantilever due to the presence of the large sphere. Traditionally, the FIB technique has been used for precise alteration or manufacture of small structures [14]. Recently, researchers have also used them to modify larger devices such as cantilevers [14,15], utilizing a larger flux (current) of positive Gallium ions (Ga+) than the ones used for small structures. The larger currents lead to higher rates of etching. It appears that the FIB might be ideally suited for the fabrication of the specialized cantilevers necessary for Casimir force measurements.

In this article we compare and quantify the benefits of cantilever milling with respect to force sensitivity in the context of Casimir force experiments. In addition, we briefly discuss the role of the internal stresses induced when a massive sphere is attached to the cantilever’s free end. In our case, the cantilever milling will primarily decrease its mass and its width. The intrinsic thermomechanical
noise limits the cantilevers ultimate sensitivity [10], and provides us with the minimum force detectable:

\[ F_{\text{min}} = \left( \frac{2k k_b T B}{\pi Q f_0} \right)^{1/2} \]  

(2)

where \( k \) is the spring constant of the cantilever, \( f_0 \) its resonant frequency and \( Q \) is the quality factor, \( k_b \) is the Boltzmann constant, \( B \) is the bandwidth, and \( T \) is the temperature.

Equation (2) is expressed in terms of the cantilever’s dimensions and its intrinsic material parameters in the following way:

\[ F_{\text{min}} = 1.007 \left( \frac{w^2 l^2}{I Q} \right)^{1/2} (E \rho)^{1/4} (k_b T B)^{1/2} \]  

(3)

Equation (3) applies to rectangular cantilevers with thickness \( t \), width \( w \), and longitude \( l \). \( E \) is the Young’s modulus of the Si cantilever and \( \rho \) is its density. The following expressions are used in equation (3):

\[ f_0 = \frac{0.507}{\pi} \left( \frac{E}{\rho} \right)^{1/2} \frac{t}{l^2} \]  

(4)

and

\[ k = 0.25 E \frac{w^3 l^3}{l^3} \]  

(5)

The two previous equations are obtained for the first order resonance of an elastic beam fixed at a single end [16]. In our experiment, we have used FIB milling to reduce the width of a soft single crystal Si cantilever with a relatively massive metal coated sphere attached to its free end as shown in Figure 1. Using a network analyzer, we obtain the power spectral density of these modified cantilevers. From this power spectrum we measure their \( k \), \( Q \), and \( f_0 \). The values obtained permit us, in both cases, to calculate and compare the minimum force through the loss parameter, which is defined with the following expression:

\[ \gamma = \frac{k}{Q f_0} \]  

(6)

**Figure 1.** SEM images of the modified cantilevers. On the left, the rectangular cantilever with sphere attached before FIB milling. On the right is the same cantilever with sphere attached after FIB milling.
2. Instrument Design
We use a modified AFM capable of reaching ultra-high vacuum environments of $10^{-8}$ torr to measure the cantilevers intrinsic characteristics. The measurements reported here were taken at a pressure of about $10^{-6}$ Torr. The vacuum chamber that contains the AFM has been described previously [17]. This AFM apparatus utilizes a mix of commercial and lab fabricated components, e.g. a Nanoscope V controller for controlling the piezoelectric actuator, and an interferometer to measure the cantilever oscillations. The schematic diagram can be seen in figure 2. We have designed and built an AFM head capable of detecting the cantilever displacements through an all-fiber Rugar type interferometer [18]. This custom-built interferometer consists of a laser diode pigtail with a 1550 nm wavelength, an inline isolator, a 50X50 fiber coupler and two photodiodes. One photodiode, detects the interferometric signal, and is connected to a network analyzer. The other photodiode measures the laser power. Note that the top of the cantilever’s free end and the end of the optical fiber form the interferometric cavity. Hence only one leg of the fiber coupler needs to be introduced into the vacuum chamber; the rest of the interferometer’s components are outside the chamber and on an optical bench. Figure 3 has a schematic diagram of the all-fiber interferometer. It is worth mentioning that similar all-fiber interferometer techniques have been used to attain force sensitivities of the order of sub-attoNewtons [19].

![Figure 2](image)

Figure 2. Schematic diagram of the complete setup used to measure the cantilever characteristics. The vacuum chamber and the all-fiber interferometer are on an optical bench.

3. Experimental procedure
Four Si cantilevers were first modified by attaching a Cr/Au coated sphere. Next, the resonance characteristics of the cantilevers with the spheres attached were measured in high-vacuum. Then the width was reduced by FIB milling and their resonance characteristics were measured again in the same high vacuum. The cantilever’s power spectral density served two purposes: (i) obtain $k$ at a temperature $T$ and (ii) to obtain the $Q$. Assuming the cantilever behaves like a harmonic oscillator with one degree of freedom, we use the equipartition theorem to obtain the spring constant $k$ of the cantilever, using the following expression [10]:

$$\frac{1}{2}k(x_{rms})^2 = \frac{1}{2}k_BT$$

(7)

Here, $x_{rms}$ is the root mean square amplitude of the cantilever tip. For a temperature $T$ we integrate the square of the cantilever power spectral density to obtain the $(x_{rms})^2$ value. Eq. (7) can be used to obtain the spring constant $k$. Note that $x_{rms}$ is measured when the interferometric cavity is set at a quadrature point of the interference fringes. The quadrature point location, assures us that change in
the interferometric signal due to a change in the cantilevers position with respect to the end of the optical fiber is linear. This is shown in the following expression

\[ x_c = \frac{\lambda}{2\pi V_{pp}} V_{Det} \]  

(8)

Here \( \lambda \) corresponds to the laser wavelength and \( V_{pp} \) is the peak-to-peak voltage corresponding to the amplitude of the cavity interferometer fringes. \( V_{Det} \) is the photodetector’s signal and \( x_c \) is the relative cantilever displacement with respect to the end of the optical fibers. Equation (8) is obtained assuming a single reflection in the interferometric cavity and assuming a small cavity finesse. To modify the cavity length a piezoelectric actuator is attached to the cantilever holder. This piezoelectric allows us to keep the cavity length at a quadrature point. The calibration measurements used to achieve the quadrature point has been described elsewhere [17].

For one of the ends of the interferometric cavity we use a commercially available single crystal Si cantilevers with a \( k = 0.01 \text{ N/m} \) to \( 0.1 \text{ N/m} \). The reported estimated dimensions for these cantilevers are: Length \( (l) = 350 \mu\text{m} \), width \( (w) = 30 \mu\text{m} \), and the thickness \( (t) = 1 \mu\text{m} \) [20]. We attached a hollow borosilicate sphere of around 100 nm diameter with silver (Ag) epoxy to the cantilever’s free end. Using a thermal evaporator we coat the sphere with a 10 nm adhesion layer of Cr and subsequently with a 300 nm layer of Au. The cantilever with the sphere attached is shown in Figure 1. During metal coating, the modified cantilever is rotated for coating uniformity. Care is taken so that only the sphere and the cantilever’s free end are coated. A more detailed coating process for a triangular cantilever can be found elsewhere [17]. The process for the rectangular Si cantilever is essentially the same. The particular choice of coating position serves two purposes: increases the cantilever’s reflectivity at infrared frequencies, while maintaining the exceptional mechanical properties of the Si cantilever.

As soon as the modified cantilever has been coated it is set on the AFM and the vacuum chamber is evacuated. Calibration of the piezoelectric actuator is done. Next, using a spectrum analyzer we obtain the thermomechanical noise response of the cantilever. The data is averaged 10 times. This response is fit to a Lorentzian and analyzed to obtain the cantilever’s spring constant \( k \) using eq. (7), its resonant frequency \( f_0 \) and its quality factor \( Q = f_0 / \Delta f \). The uncertainty in these 2 values is around 60 mHz. After this the cantilever with the sphere is taken out and the FIB milling is done. For FIB milling [21] a 4nA current of 30 keV accelerated Ga+ ions is used. The right panel in Figure 1 shows the modified cantilever after this procedure. Finally, the milled, modified cantilever is inserted into the vacuum chamber, the piezoelectric actuator is calibrated again, and the measurement of the cantilever’s \( k, f_0 \) and \( Q \) is repeated.

Figure 3. Schematic diagram of the experimental setup used to measure the cantilever’s characteristics. The layout of the all-fiber interferometer is also shown.
4. Modified cantilever characteristics before and after the Ion milling process

The cantilever dimensions before and after ion milling are shown in Table 1. A scanning electron microscope (SEM) was used to measure the cantilever dimensions as well as the radii of the coated hollow spheres. To compare and obtain accurate values of the dimensions we use a diffraction grating standard. The length of the cantilever in Table 1 corresponds to the distance between the beams clamped-end to its tip. Although in theory the effective length of the cantilever should include radius of the attached sphere. Note that after the milling procedure two out of four cantilevers have two different widths. This is due to the nonuniformity of the ion milling process. The opposite ends, one near the tip close to the sphere and the other near the substrate are different and shown in Table 1. The consequence of these kinds of discrepancies will be important when comparing theoretical calculations of the intrinsic cantilever characteristics for these non-prismatic beams with the measured values for these cantilevers. One more effect that should be accounted for in these calculations is the pseudo trapezoidal shape of the cantilever cross-section after milling. This shape is produced because of the Gaussian shape of the focused ion beam. This could be avoided if the cantilever milling is done with a smaller ion current.

Table 1. Cantilever dimensions before and after FIB milling.

| Cantilever | Width (μm) Before | Width (μm) After | Length (μm) | Sphere diameter (μm) |
|------------|------------------|-----------------|-------------|---------------------|
| #4         | 35.3             | 3.8/2.4         | 296.6       | 80.5                |
| #3         | 35.8             | 3.0             | 300.8       | 83.0                |
| #2         | 31.6             | 4.1/2.7         | 299.7       | 77.2                |
| #1         | 33.0             | 3.2             | 291.3       | 84.0                |

Care was taken to pick cantilevers with similar characteristics. However intrinsic cantilever discrepancies, different masses of the attached spheres, and the amount of Ag epoxy used, have produced noticeable differences in their characteristics. The characteristic values for the four cantilevers: spring constant $k$, the resonant frequency $f_0$, and the quality factor $Q$ are shown in Table 2. To easily compare these measured values, Table 2 is arranged, so the before and after values are next to each other. The most important values are the $Q$’s. These values changed in a fairly unexpected fashion. Previous research has found that the internal structure of single crystal cantilevers is modified by the Ga+ ions during ion milling [15]; thereby affecting the $Q$. We believe that in our case this modification should have been minimal since we etched away most of the region interacting with the ion beam. However, the scattered Ga+ ions still lead to radiation damage in the silicon, leading to a decrease in the $Q$, just as we have observed in our cantilevers. As the $Q$ measurement is done in vacuum, the viscous drag due to the air is negligible in our experiments and only the internal stresses of the cantilever determine the quality factor $Q$ [22]. Nevertheless since the ratio of the volume to surface area changed with the milling process, surface effects might play a larger role on the measured $Q$ for these cantilevers. For example the cantilever $Q$ might have decreased because the cantilevers’ surface was contaminated or because the cantilever surface was oxidized by the formation of SiO$_2$, similar to what other researchers have reported [22]. However, this alone cannot explain the decrease in $Q$ observed here, which is much larger than that reported by other researchers for contamination and oxidation. Wet chemical etching of the oxide layer or contaminants cannot be done because of possible damage to the attached sphere. Not only do the cantilevers $Q$ have valuable information; their $k$ suggests that the modified cantilevers Young’s modulus can be treated as those of regular unmodified ones. Using (5) and the cantilevers dimensions from Table 1 the calculated spring constant
\( k \) is listed in table 2. The measured \( k \) values closely match the theoretical values for cantilevers without an attached sphere. This is true even though the values of the measured \( k \) have changed by an order of magnitude, from the change in the cantilever width.

### Table 2. Cantilevers’ intrinsic characteristics before and after FIB milling.

|        | \( f_0 \) (Hz)
|--------|-----------------|
|        | Before | After |
| Cantilever #4 | 5433.3 | 2269.2 |
| Cantilever #3  | 5704.9 | 2265.4 |
| Cantilever #2  | 5994.7 | 2520.8 |
| Cantilever #1  | 5426.0 | 1984.0 |

|        | \( k \) (N/m)
|--------|-----------------|
|        | Before | After |
| Cantilever #4 | 0.3029 | 0.0311 |
| Cantilever #3  | 0.1074 | 0.0022 |
| Cantilever #2  | 0.0120 | 0.0063 |
| Cantilever #1  | 0.0157 | 0.0022 |

|        | \( Q \)
|--------|-----------------|
|        | Before | After |
| Cantilever #4 | 15311.2 | 1684.0 |
| Cantilever #3  | 11838.8 | 3982.8 |
| Cantilever #2  | 11660.8 | 2290.7 |
| Cantilever #1  | 18901.9 | 3715.8 |

In table 3 the cantilevers loss parameters and their minimum detectable force are compared before and after they went through the ion milling process. The minimum force values are calculated at \( T = 300K \) using (2) and using the values of table 2. We have used the relative percentage change in equation (9) to emphasize the difference between the before and after values. For the case of the minimum force it leads to the following expression:

\[
\text{Relative percentage change} \% = 100\times \frac{F_{\text{min}}^\text{Before} - F_{\text{min}}^\text{After}}{F_{\text{min}}^\text{Before}}
\]

The results in table 3 show that only 1 out of 4 cantilevers present an increase in force sensitivity while the other 3 result in decreased values. The cantilevers minimum detectable force and the loss parameter depend on the values from table 2. Therefore a careful study of the values of this table should result in understanding why the minimum detectable force/loss parameter increased. However the data in table 3 is valuable because it demonstrates the combined effect of the values in table 2.

### Table 3. Modified cantilevers minimum force \( (F_{\text{min}}) \) and loss parameter \( (\gamma) \) before and after FIB milling occurred. Columns 4 and 5 present the relative percentage change of these quantities due to the FIB process.

|        | \( F_{\text{min}} \) (femtoN)
|--------|-----------------|
|        | Before | After |
| Cantilever #4 | 3.099 | 4.635 |
| Cantilever #3  | 2.048 | 0.803 |
| Cantilever #2  | 0.667 | 1.691 |
| Cantilever #1  | 0.636 | 0.896 |

|        | \( F_{\text{min}} \) percentage change (\%)
|--------|-----------------|
|        | Before | After |
| Cantilever #4 | -49.6 | 3.64 |
| Cantilever #3  | 60.8 | 1.59 |
| Cantilever #2  | -153.4 | 0.17 |
| Cantilever #1  | -40.8 | 0.15 |

|        | \( \gamma \)
|--------|-----------------|
|        | (nN/m)(Hz)^{-1}|
|        | Before | After |
| Cantilever #4 | 8.12 | 0.25 |
| Cantilever #3  | 0.25 | 84.6 |
| Cantilever #2  | 1.08 | -542.2 |
| Cantilever #1  | 0.30 | -98.3 |

This data shows us the range of minimum force achieved by the modified cantilevers before milling. For all these cantilevers the minimum force already surpasses the sensitivity achieved in previous Casimir force experiments [6,7]. But if this milling technique on the cantilevers is successful the minimum detectible force can be substantially reduced with minimal nano processing. For instance:
Cantilever No. 3 increased its minimum force by 60% mainly due to the 2 order of magnitude decrease in the spring constant \(k\). We believe that during milling not only is the cantilever’s width reduced but also its thickness \(t\) was decreased. Consequently this cantilever’s minimum force decreased. If future research is able to keep the milled cantilevers energy dissipation closer to the non-milled values, and the thickness and width reduction is the same, sensitivities of the order of tens of attonewtons could be achieved.

One of our objectives is to understand if the sphere and the Ag epoxy affect any of the mechanical properties of the unmodified single crystal Si cantilever. Since the cantilever mass, regardless of milling is comparable to the mass of the hollow sphere plus Ag epoxy, it is possible that the quality factor might have decreased because of the internal stresses introduced when the sphere is attached. In addition, we are studying the possibility that the theoretical model of a prismatic bar fixed at one end is no longer suitable for our milled cantilevers and that a better model with boundary conditions which resemble our modified cantilever with sphere attached will be more appropriate. In fact if a different theoretical model is used, the cantilevers theoretical characteristics might be different from the ones resulting from equations (4) and (5), changing the expected cantilever’s minimum force. This is indeed the case for non-prismatic cantilevers [23].

To understand the origin of the internal stress in the cantilever, we annealed the milled cantilever. The result was promising; the cantilevers \(Q\) returned approximately to their previous non-milled value. However during the annealing process the cantilevers lost their sphere. The positive aspect of these results is that the cantilevers \(f_0\) and \(k\) were close to the milled cantilevers values obtained previous to annealing with the hollow sphere attached to its free end. We believe that the reason for this is that most of the cantilevers added mass come from the Ag epoxy, which remained on the milled cantilever after the annealing process, i.e. the hollow metal coated sphere does not add much mass to the system. In any case further studies are currently being done to obtain a more accurate solution that better describes our modified cantilevers.

5. Conclusions
Reducing the width of single-crystal silicon cantilevers with a Cr/Au-coated-hollow sphere attached at their free-end leads to higher force sensitivities appropriate for better Casimir force detection. The FIB milling technique used to reduce the width seems to damage the material of the Si cantilevers and lead to a decrease in the cantilever quality factor. Annealing of the modified cantilevers suggests that this effect could be reversed. Future studies could allow us to attain the minimum detection forces of the order of tens of attonewtons for cantilevers with attached metal coated spheres required for sensitive Casimir force measurements.

Acknowledgments
The experimental section of this work was supported by NSF Grant No.PHY0653657 and the instrumental supplies along with the analysis by DOE Grant No. DE-FG02-04ER46131. The nanofabrication was supported by DOD/DMEA H94003-07-2-0703. R C-G is grateful for the financial support from UCMexus and CONACYT. We also appreciate the technical assistance of Dr. Dong Yan with the FIB milling technique.

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