Precision mass and density measurement of individual optically-levitated microspheres

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We report on an in situ mass measurement of ~4.7-μm-diameter, optically levitated microspheres with an electrostatic co-levitation technique. The mass of a trapped, charged microsphere is measured by holding its axial (vertical) position fixed with an optical feedback force, under the influence of a known electrostatic force. A mass measurement with 1.8% systematic uncertainty is obtained by extrapolating to the electrostatic force required to support the microsphere against gravity in the absence of optical power. In three cases, the microspheres were recovered from the trap on a polymer-coated silicon beam and imaged with an electron microscope to measure their radii. The simultaneous precision characterization of the mass and radius of individual microspheres implies a density of 1.55 ± 0.08 g/cm³. The ability to recover individual microspheres from an optical trap opens the door to further diagnostics.

Optical trapping and manipulation of micron-sized dielectric particles in vacuum has been applied to optomechanics [1–6] and cavity cooling [7–9], fundamental forces and interactions [10–16], quantum mechanics [17, 18] and quantum information [19], and surface science [20]. In many of these applications, knowing the size, mass and other characteristics of the trapped particles is critical to drawing conclusions about moments of inertia, optical spring constants, and force sensitivity.

We present a technique to measure the mass of individually trapped microspheres (MSs), by balancing a known electrostatic force, the optical levitation force, and earth’s gravity. The electrostatic force is extrapolated to the condition of no optical power to determine the gravitational force on the MS, and thus infer its mass. This measurement requires fewer assumptions than other techniques [21, 22] and is found to be independent of environmental conditions.

It may be possible to derive a direct relation between the optical power required to levitate a MS at the center of the trap and the mass of the MS using numerical methods to solve Mie scattering theory [23]. However, this requires a detailed understanding of the MS radius, non-sphericity, and index refraction, as well as a full description of the optical potential in three dimensions. The technique described here bypasses these complications and their associated systematics, resulting in improved accuracy.

A subset of MSs are also individually collected from the optical trap using a mechanical probe, and imaged via scanning electron microscopy (SEM) to determine their radii. Knowing both the mass and radius of individual MSs, their density can be calculated. The radii determined from SEM images of those specific MSs are compared to the radii determined from SEM images of large populations of ~10³ MSs that have never been in the optical trap.

The optical trap used here is described in Refs. [20, 24]. Silica MSs obtained from the Stöber process [25, 26] with ~4.7-μm-diameter are loaded into the trap by ejection from a vibrating glass slide placed above it. To efficiently load MSs, 1 mbar of residual gas is used to provide viscous damping. The chamber can then be evacuated to a final pressure of 10⁻⁶ mbar, in order to reduce thermal noise. Below 0.1 mbar, the trap requires active feedback for stabilization. The feedback system [24] serves to provide viscous damping in all three degrees of freedom (DOF). Importantly, the axial degree of freedom of the MS, stabilized by modulating the power of the trapping beam with an acousto-optic modulator, is held at a fixed position by proportional and integral feedback terms.

The above loading procedure triboelectrically charges the MSs. A xenon flash-lamp, emitting ultraviolet light, is used to alter the MS charge state, q_{MS}, over a wide range 500e < q_{MS} < 500e, where e is the elementary charge [11, 13, 20, 24]. The arbitrarily set MS charge state, stable over timescales of order one month, is known with sub-e precision, as individual quanta are added to or removed from a state of overall neutrality.

Charged MSs are shielded from external electric fields by a Faraday cage made of six electrodes, each with an independent bias voltage. The two electrodes directly above and below the trapped MS are used to generate a uniform, slowly-varying electric field at the trap location, exerting an axial force on a charged MS. The relation between the applied voltage and the electric field within the trapping region is modeled with finite-element analysis, with an uncertainty that is much smaller than any other systematics.

After measuring their mass, three MSs are collected onto the end of a polymer-coated silicon beam, described in Refs. [20, 27], where they remain attached via Van der Waals forces. Individual MSs are addressed to particular locations, recognizable from features on the silicon beam.
FIG. 1. Schematic depiction of the measurement technique. A charged MS is trapped by a Gaussian laser beam and held at fixed axial position with active feedback. A slowly varying electric field is applied, depicted with a black arrow. The active feedback reduces the optical power, indicated by the intensity of the trapping beam, such that the sum of the optical and electrostatic levitation forces opposing gravity is constant. The relation between optical power and applied field is then extrapolated to zero optical power, allowing a determination of mass from the implied electrostatic levitation field and the known charge.

The silicon beam is removed from the chamber and the three MSs are imaged with SEM, in order to determine their individual radii. A population of MSs of the same variety and lot as those used in the trap are also measured with SEM. For this purpose, a monolayer of MSs is spread onto a silicon wafer, and subsequently imaged with SEM. Various diffraction gratings [28] are used to calibrate the instrument at each of the magnifications.

Once a constant, known charge is obtained for a trapped MS, its axial position is fixed near the focus of the optical trap using the feedback. The slowly-varying (0.5 Hz) electric field is applied in the vertical direction, while the power of the trapping beam injected into the chamber, controlled by the feedback, is monitored with a beam pickoff and a photodiode. As the applied electrostatic force increases, the axial feedback reduces the optical power required to maintain a net force of zero, counteracting gravity. The electric field can then be extrapolated to zero optical power, allowing a determination of mass from the implied electrostatic levitation field and the known charge.

The equilibrium of axial forces $F_z$ is expressed as,

$$\sum F_z = qE(t) - mg + F_{\text{opt},z}(t) = 0,$$

where $q$ and $m$ are the charge and mass of the MS, respectively, $g = 9.806 \text{ m/s}^2$ is the local gravitational field strength [29], $F_{\text{opt},z}(t)$ is the optical levitation force, assumed to be proportional to the trapping beam power, and $E(t)$ is the applied electric field strength. For each MS and charge state combination, the slowly-varying electric field and power are measured at least 50 times, each with a 50 s integration. An exemplary dataset is shown in Fig. 2, with the calculated mass from the extrapolation to zero optical power.

The mass measurement is performed on 13 MSs, in various charge states around $|q| = 20e$, with both signs of charge, as well as in two vacuum pressure regimes: trapping pressure, $\sim 1 \text{ mbar}$, and chamber base pressure, $\leq 10^{-6} \text{ mbar}$. The use of different pressures tests whether MS mass is lost due to heating, as observed for larger MSs in Ref [30]. Cooling via residual gas decreases significantly with decreasing pressure, while absorption and scattering of laser light, the dominant heating mechanisms, remain constant.

FIG. 2. Normalized optical power vs applied electric field for 100×50 s integrations with a single MS. The extrapolation is performed separately for each integration. The mean of all extrapolations is shown with a dashed black line. (Inset) distribution of the 100 extrapolated masses.
Results of mass measurements for all experimental conditions are shown in Fig. 3, while the results from the final three MSs later transferred to SEM are provided in the first column of Table I. Fluctuations in the mass measurement over the ≥ 50 distinct 50 s integrations for a set of experimental conditions are normally distributed with a standard deviation on the order of 0.5 pg as seen in the inset of Fig. 2. However, the total uncertainty of the measurement is dominated by common systematics which are enumerated in Table II.

Each effect listed has been interpreted as an uncertainty on either the applied electric field, the measured optical power, or the assumed value of \( g \). From Eq. 1, these relative uncertainties directly propagate onto the extrapolated mass, whose uncertainty is computed as a quadrature sum of all contributions. The accuracy of the high-voltage amplifier’s output monitor and the tolerance on the trapping lens focal length dominate the overall uncertainty. The second effect may offset the trap axially, thus sampling a different electric field strength. Each of the effects in Table II should result in a systematic shift common to all mass measurements. The total uncertainty obtained is 1.8%, which is included as a systematic on the mean mass for each MS.

We also observe scatter in the measured mass of a single MS between different experimental conditions, as seen in Fig. 3. These variations could be due to a number of effects including optical path length fluctuations in the axial feedback, electronic fluctuations in the axial feedback, as well as real changes in the mass of a MS. We do not observe any correlations between measured mass and experimental parameters such as the MS charge state or the vacuum pressure. The fluctuations are quantified by the standard deviation of measurements with different experimental conditions, which is included as part of the statistical uncertainty on the measured mass.

To collect the final three MSs, the polymer-coated silicon beam is rapidly inserted between the trapping laser and the MS, allowing the MS to fall under the influence of gravity. Each distinct MS can be associated to the MS, allowing the MS to fall under the influence of gravity. Each distinct MS can be associated to the MS, allowing the MS to fall under the influence of gravity. Each distinct MS can be associated to the MS, allowing the MS to fall under the influence of gravity.

The fluorocarbon polymer coating is estimated from EGM2008 [29].

### Table I. MS masses, \( m \), averaged over all experimental conditions; radii, \( r \), averaged from two distinct high magnifications; and the derived density, \( \rho \), for the three MSs caught on the silicon beam. All measurements include statistical and systematic uncertainties, and the relative contributions have been shown explicitly for the measured masses.

| MS | \( m \) [pg] | \( r \) [\( \mu \)m] | \( \rho \) [g/cm\(^3\)] |
|----|---------------|----------------|-------------------------|
| #1 | 84.0 ± 0.8 (stat) ± 1.5 (sys) | 2.348 ± 0.038 | 1.550 ± 0.080 |
| #2 | 83.9 ± 1.1 (stat) ± 1.5 (sys) | 2.345 ± 0.037 | 1.554 ± 0.079 |
| #3 | 85.5 ± 0.2 (stat) ± 1.5 (sys) | 2.355 ± 0.038 | 1.562 ± 0.081 |

### Table II. Systematic effects on the mass measurement.

| Effect | Uncertainty [×10\(^{-3}\)] |
|--------|----------------------------|
| Amplifier monitor accuracy\(^\dagger\) | \( \sigma_E/E \sim 15 \) |
| Lens focal length\(^\dagger\) | \( \sigma_E/E \sim 10 \) |
| Amplifier gain error\(^\dagger\) | \( \sigma_E/E \sim 2 \) |
| Tilt of field-axis | \( \sigma_E/E \sim 2 \) |
| Tilt of trap (optical) | \( \sigma_E/E \sim 1 \) |
| ADC offsets\(^\dagger\) | \( \sigma_P/P \sim 1 \) |
| Electrode voltage offset | \( \sigma_E/E \sim 0.5 \) |
| DC power offsets | \( \sigma_P/P \sim 0.3 \) |
| Local \( g \) \(^\ddagger\) | \( \sigma_g/g \sim 0.1 \) |
| Electrical pickup | \( \sigma_P/P \sim 0.02 \) |

\(^\dagger\) From manufacturer datasheets,
\(^\ddagger\) Estimated from EGM2008 [29].
made with a plasma deposition technique inherent to the Bosch process [31], using C₄F₈ and SF₆ gases in a 1.5 kW inductively-coupled plasma.

For the SEM measurements, the silicon beam with three MSs is first sputter-coated with 100 ± 50 nm of a Au/Pd alloy, in order to prevent charging and the resulting MS ejection from the silicon beam. Charging effects from the SEM are significantly exacerbated by the non-conductive polymer, necessitating the relatively thick coating. A diffraction grating with 1.000±0.005 μm pitch [28] is used to calibrate SEM images of individual MSs at high magnification, as seen in Fig. 4.

The MS diameter is first determined in terms of raw pixels. This is done via edge detection and contour tracing to outline the MSs. The contour is then fit with an ellipse to account for real ellipticity in the MSs, as well as astigmatism in the electron microscope. The radius is taken as the average of the semi-major and semi-minor axes, which differ by less than a percent. A systematic uncertainty of ±1 pixel in the determination of the semi-major and semi-minor axes is included.

At the same level of magnification, images of the calibration grating are used to convert from pixels to physical distances. This is done by locating the centroids of the grating’s repeated structure in the image, and averaging the pixel distance between neighboring centroids across the entire image. The ratio of grating pitch in microns to observed grating pitch in pixels serves to calibrate the pixel distance between neighboring centroids across a range of magnifications, together with images of the calibration grating. The same ellipse identification and calibration are used to characterize MS populations.

The radii of the final three MSs measured are compared to the distribution of radii from the MS population measurements, shown in Fig. 5. The conductive coating
reduces the apparent size of the MSs by \( \sim 20 \text{ nm} \), after accounting for the correction due to coating thickness. This may be the result of charging of the uncoated MSs.

After correcting for the thickness of the coating, the individually measured radii of conductively coated MSs are found to be consistent with the distribution of radii measured from the large population of conductively coated MSs. The apparent independence of the measured mass on the vacuum pressure, as well as the consistency between the measured radii of individual MSs that have been optically trapped and large populations of MSs that were never trapped, both indicate a negligible loss of MS material by heating, under the environmental conditions tested for \( r \approx 2.35 \mu m \) silica MSs. The simplicity and accuracy of the mass measurement, along with the reliable transfer of specific microspheres from the optical trap to air and subsequently to a different vacuum environment, opens the possibility for other correlated, precision measurements on microscopic objects.

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