Cooling of Levitated Graphene Nanoplatelets in High Vacuum

Pavel Nagornykh and Joyce E. Coppock
Department of Physics, University of Maryland, College Park, MD

B. E. Kane
Joint Quantum Institute, University of Maryland, College Park, MD and Laboratory for Physical Sciences, University of Maryland, College Park, MD

We demonstrate cooling of the center of mass motion of charged graphene nanoplatelets levitated in a quadrupole ion trap in high vacuum down to temperatures of 20 K. Parametric feedback based on optical measurements of particle motion was used to achieve the particle cooling at pressure \( p < 10^{-6} \) Torr, and cooling along all three axes of motion was observed. Dependence of cooling on the electric fields was measured by varying DC voltages on a set of auxiliary electrodes used to spatially shift the trap minimum. Methods to calibrate mass and charge of the nanoplatelet by measuring its motion frequency dependence on discharge were also explored.

While trapping and cooling technologies have been primarily applied to atomic systems \([1,3]\), they have also found applications in the study of nanoscale systems of condensed matter \([4,10]\). One of many 2D materials that could benefit from these techniques is graphene, a material that garnered attention after its discovery in 2004 \([11]\). A plethora of new phenomena related to unique behavior of electrons in graphene were predicted \([12,13]\). While big progress has been made in getting closer to graphene’s intrinsic behavior \([14-16]\), there are certain properties that are hard to measure in conventional experiments, and consequently a complementary approach of decoupling of graphene from the environment by levitating small graphene samples was proposed \([17]\). This experimental setup has the advantage of allowing the study of free graphene membrane motion at ultrahigh rotation speeds, which can be used to apply strain to the membrane \([17,18]\). Thermal properties of graphene near its melting point, predicted to be close to temperature \( T = 5000 \) K \([19]\), are also out of reach for standard measurements while easily achievable by laser heating \([19]\). Finally, the levitation approach can be used to conduct research on ultra high vacuum (UHV) crystal growth, graphene manipulation and deposition onto a substrate as well as flakes’ functionalization \([20,21]\).

In previous experiments \([17]\), a quadrupole ion trap was used to levitate graphene nanoplatelets separated by a distance of about 2 mm from the trap electrodes. During the experiment, the time dependence of the center of mass motion of the particle and its visible intensity were measured at different pressure \( p \) levels, and circularly polarized light was used to provide spin to the particle. While these measurements produced estimates of the mass, thickness, and charge, it was also found that the time particles stay trapped before they escape was limited to a few hours at \( p < 10^{-7} \) Torr \([17]\). However, lower \( p \) values are required to obtain a low contamination environment necessary for material deposition, thermal experiments, and high spinning frequency measurements on graphene flakes. Consequently, a solution to the problem of low trap life times is required.

A similar problem has been known to exist for optically trapped microscopic particles, and parametric feedback cooling was developed as a possible solution \([22]\). Experiments conducted in Ref. \([22]\) involved silica nanospheres trapped in an optical trap and cooling to 50 mK was observed. We implement the parametric feedback approach for graphene flakes in the quadrupole ion trap in order to improve the trapping times of the flakes, to stabilize and cool down their motion in high vacuum conditions, and to show that it is a viable method for investigation of levitated graphene in UHV.

RESULTS

Experimental setup

The experimental setup used for our measurements is similar to the design used in Ref. \([17]\) with modifications related to transfer of the particles after trapping and the way their motion is detected. The system consists of two chambers separated by a gate valve, with ionized particles introduced into the loading chamber by use of the electrospray technique \([23]\). After trapping in the loading chamber, particles are transferred to the high vacuum chamber (see Fig. 1b), where the cooldown experiment is conducted (see paper \([24]\) for discussion of trapping and transfer at the current experimental setup in more detail). The use of two chambers minimizes contamination of the high vacuum chamber by volatile substances that are created during electrospray operation.

When trapped in the high vacuum chamber, charged particles are levitated in an electric quadrupole trap \([25]\) that consists of two coaxial conical electrodes (Fig. 1a). The inner electrode of the trap is kept at zero voltage while an AC voltage \( V_{AC} = 300 \) V at frequency \( \Omega/2\pi \) (chosen to allow stable trapping of particles) is applied to the outer electrode. The AC voltage is created by a

* pnagorny@lps.umd.edu
Motion has three main frequencies of oscillation [25]: the outer electrode, which allows us to distinguish motion in the optical signal coming from a single detector. The ability to resolve all three degrees of motion present as asymmetry is crucial for our experiments, since we rely on the trap asymmetry introduced to the system. This considerations (See Fig. 1.a) that motion in the X and Y directions is non-degenerate only if there is some intentional trap asymmetry introduced to the system. This asymmetry is crucial for our experiments, since we rely on the ability to resolve all three degrees of motion present in the optical signal coming from a single detector. The splitting of the degeneracy is done by making a slot in the direction of the light scattered from the particle. X’, Y’ and Z’ show the projections of the main axis of motion of the particle onto the lens’ focal plane for three different orientations of the prism beam splitter. Photodetectors are shown schematically as A and B.

In the pseudopotential approximation, the particle motion has three frequencies of oscillation [25]:

$$\omega_{x,y,z} = \frac{q x, y, z}{m} \frac{V_{AC}}{\sqrt{2 z_0^2 + 2 \omega^2}}$$, (2)

where q and m are the charge and mass of the particle. The pseudopotential approach is applicable when $\omega_x, \omega_y, \omega_z < \Omega$, conditions which are true for our experimental parameters of $\omega_{x,y,z}/2\pi$ and $\Omega/2\pi$ which lie in the range of 150-1000 Hz and 15-30 kHz respectively.

Charge to mass ratio values of about 10-100 $C \cdot kg^{-1}$ are typical for particles trapped using the electrospray technique, and the data presented in the paper are taken on one of these particles. To minimize charge loss of the particle, laser power for all experiments was limited to 1 mW or lower. Discharge observed at higher laser powers may be related to evaporation of the solution residue left on particles after their preparation [26]. A 671 nm circularly polarized laser propagating along the Z axis of the trap was used to stabilize the rotation of the particle, and minimize brightness fluctuations attributable to nanoflake reorientation [17].

**Particle motion detection**

Motion of the trapped particle was tracked using a 532 nm linearly polarized laser beam with a width of 0.16 mm (See Fig. 1[b]). The light scattered from the particle is focused by a lens with a 40 mm diameter located at a distance of 12.5 cm from the center of the chamber and is afterwards divided by a 50:50 beam splitter. One of the split beams is used for rough particle positioning with a CCD (charge-coupled device) camera and provides information about average position of the particle and amount of scattered light at a frame rate of 1 sec. The second beam falls on a knifeedge of a 90° prism mirror beam splitter at the lens’ focal point. This directs light to one of two avalanche photodiodes (A and B in Fig. 1[c]), depending on the position of the particle. The difference between the photodetector signals, $S = A - B$, can be used to study the center of mass motion of the trapped particle. Here we intentionally choose to use the normalized difference of signals in order to minimize the signal dependence on the laser power and on variations of intensity of scattered light.

Only components of motion that have non-zero projection onto the image plane in a direction perpendicular to the prism knifeedge will contribute to the signal S. Indeed, orienting the prism edge orthogonally to the Z axis of the trap (see Fig. 1[c], where prism is shown as viewed along the deflected light direction from the viewpoint at the center of the trap) will maximize the amount of motion detected from the particle oscillations in Z direction. Similarly, rotating it by 90 degrees to direction parallel to the Z axis will zero the signal from the oscillations along Z and maximize the X and Y projections, albeit at amplitude $1/\sqrt{2}$ lower than amplitude along Z in the previous case. A rotation angle of 54.7 degrees is chosen in order to make scaling factors for contributions from each of the frequencies to be the same. In this configuration, the fast Fourier transform (FFT) of the signal $S$ contains all three peaks, one for each of the eigenmodes of motion (Fig. 2).
Figure 2. Power spectrum of signal $S$ at (a) $p = 4$ mTorr and (b) $p = 4 \times 10^{-7}$ Torr.

**FPGA setup for particle control and cooling**

The signal $S$ is sent to an FPGA (Field-Programmable Gate Array) for digital filtering and processing as well as for generation of slow feedback (cut-off frequency of 4.4 Hz) that is used to keep $A = B$ by adjustment of lens’ position on a piezostage. The speed of this feedback is set to be much slower than the typical eigenfrequencies of the particle motion to avoid any cross-talk between the feedback and the motion.

The second and main purpose of the FPGA is to generate a feedback signal that is fed to the signal generator as an amplitude modulation (AM) input. The feedback signal $S_{feed} = gSS\dot{S}$ is created by multiplying the filtered signal $S$ by its derivative \cite{22}, where $g$ is the gain setting of the feedback. For sinusoidal signals, this operation creates an output at twice the particle oscillation frequency. The feedback modulation is applied to the trap potential $V_{AC} = V_{0AC}(1 + G_{AM}S_{feed})$, where $G_{AM}$ is the AM value set at the signal generator.

**Particle cooling and stabilization in high vacuum**

Once the $p$ is lowered below 1 mTorr, the cooldown of the particle motion is observed if proper feedback conditions are chosen. We have used AM setting of 30% at feedback gain of $g = 2.4 \times 10^{-4}$ s to show the cooling dependence on $p$. At these parameters, all three degrees of particle motion (Fig.3a) reach $T \sim 20$ K at $p = 4 \times 10^{-7}$ Torr. In the region between $10^{-3}$ and $10^{-7}$ Torr, $T$ is strongly affected by the pressure, and even changing $p$ from $10^{-6}$ Torr down to $4 \times 10^{-7}$ Torr improves cooling from $T \sim 40$ K down to $T = 20$ K.

To prove that the AM setting of 30% chosen for the experiment is optimal and to show that the cooling is the result of parametric feedback, we measure the $T$ dependence on the AM setting. While higher AM settings result in better cooling (Fig.3b), settings close to zero lead to higher average $T$ and its fluctuations. At the same time, one can notice that increasing the AM setting to values higher than 30% does not improve the cooling, which makes $G_{AM} = 30\%$ the best value for experiments. Absence of further cooling for AM values greater than 30\% indicates that noise present in the feedback is likely limiting cooling.

**Cooling dependence on auxiliary electrodes’ settings**

The sensitivity of cooling to the feedback noise is enhanced by any non-zero DC electric fields (see Methods)
While only with a distinct discharge quantization is shown on Fig. 4.

The ability to extract the mass of the particle through its discharge allows for a separate estimate of particle’s $T$ if a proper calibration of signal $S$ in terms of spatial displacement is done. However, it should be pointed out that there is an intrinsic nonlinearity in our signal detection that makes the $T$ extraction potentially inaccurate and proper calibration is dependent on exact value of the reflectivity of the particle as well as high signal to noise ratio.

In conclusion, we have presented experimental results on cooling and stabilization of the graphene nanoflakes.
in high vacuum conditions. It was shown that at $p$ of $4 \times 10^{-7}$ Torr the particle can stay trapped on a time scale longer than a few days and cooled down below 20 K in all three degrees of motion, reaching particle localization below 1 $\mu$m in the cooled state. Achieving stable motion of the particle allows us to observe the discharge of the particle and to extract its mass and charge. Finally, it was shown that the elimination of stray fields was necessary to achieve optimal cooling.

METHODS

Signal calibration

In order to calibrate the $T$ of the particle, we use the particle motion at $p$'s of 5-10 mTorr, where the Brownian motion of the particle is well thermalized and the laser power does not affect the $T$ of the particle motion. For thermalized motion, the fluctuation-dissipation theorem [34] can be applied for calibration of the power spectrum of signal $S$ [32]:

$$\int P_S(\omega) \, d\omega = \xi T,$$

where $P_S(\omega)$ is a power spectrum of signal $S$ and $\xi$ is the scaling factor extracted by normalizing the signal to give ambient $T$ value ($T = 293$ K) at $p$ of 6 mTorr. The value of $\xi$ for motion along each of three axes of motion is found by integrating the area under the corresponding peak in the power spectrum of $S$ (see Fig 2). After the $T$s are calibrated, we can keep track of their individual values during the measurements. The $T$s start to deviate from one another when $p$ is below the level required for proper thermalization ($p \sim 1$ mTorr).

Explanation of cooling effect

When the feedback modulation is applied to the AC potential, the particle motion in all three directions becomes nonlinear:

$$\ddot{x}_{x,y,z} + \Gamma_0^{x,y,z} \dot{x}_{x,y,z} + \left[\omega_{x,y,z}(S, g)\right]^2 S_{x,y,z} = 0,$$

where $\omega_{x,y,z}(S, g) \approx V_{AC}^0 (1 + G_{AM} S_{feed})$ and $\Gamma_0^{x,y,z} = \Gamma_0$ is a $p$ dependent damping ratio [34]. For the case of $G_{AM} S_{feed} \ll 1$, which is true for our experimental settings, the equation of motion can be simplified down to an equation of a parametric harmonic oscillator [35] that can be thought of as a harmonic oscillator with a position dependent damping $\Gamma$ [34].

$$\Gamma_{x,y,z} = \Gamma_0^{x,y,z} + 2 g G_{AM} \omega_{x,y,z}^2 S^2 > \Gamma_0^{x,y,z}.$$  

(5)

Since effective damping is enhanced by the feedback, the particle's center of mass motion will be cooled down when $g$ or $G_{AM}$ are increased.

Noise coupling in absence of DC fields

In our system, the noise in the feedback originates from the shot noise from photodetectors $A$ and $B$. The resulting correction to the feedback leads to coupling of the noise to the motion of the particle and the minimum cooling $T$ becomes modified [35]. Since the highest energy transfer comes from the component of the feedback at a frequency of $\omega_{fd} = 2 \times \omega_{x,y,z}$ and our feedback is the product of the measured signal $S_m = S + \eta$ and its derivative, the efficiency of the feedback will be mostly limited by the cross-terms between the signal $S$ and the noise $\eta$ that have frequencies adding up to $\omega_{fd}$. Considering that main components of the signal have frequencies of $\omega_{x,y,z}$, the noise components at $-\omega_{x,y,z} \pm \omega_{fd}$ are going to dominate in the feedback noise.

Noise coupling in the presence of non-zero DC fields

In order to study how the noise coupling is affected by DC fields, let us start with a derivation of the particle motion under the influence of small $\delta V_{AC}$ modulation of the trap potential. For simplicity, we analyze only the motion along the $X$ axis in the presence of a non-zero DC field $E_x$, but similar calculations can be done for two other axes of motion.

The DC electrical force displaces the particle from the trap center by $x_{dc}$, where $x_{dc}$ is defined from an equilibrium condition between the trapping force and the DC field:

$$F_x = qE_x - m \omega_{x}^2 x_{dc} = 0.$$

When a small modulation of AC potential $\delta V_{AC} \epsilon_{i\omega t}$ is introduced by the lock-in or by noise from the feedback, the resulting variance in frequency $\delta \omega_{x} \epsilon_{i\omega t}$ creates a varying force component:

$$\delta F_x(t) = -2 m \omega_{x} x_{dc} \delta \omega_{x} \epsilon_{i\omega t},$$

which in turn leads to modulation of particle motion $\delta x(t)$. The modulation $\delta x(t)$ can be expressed via the response function of the simple harmonic oscillator to the external force $\delta F_x(t)$ at a frequency $\omega$:

$$\delta x(t) = \frac{1}{\omega_x^2 - \omega^2 - i \Gamma \omega_x} \frac{\delta F_x(t)}{m}.$$  

(6)

By using equation (2) for $\omega$, and equation (6) for $\delta F_x(t)$, we can rewrite $\delta x(t)$ in terms of $\delta V_{AC}$:

$$\delta x(t) = -x_{dc} \frac{2 \omega_x^2}{\omega_x^2 - \omega^2 - i \Gamma \omega_x} \frac{\delta V_{AC}}{V_{AC}^0} \epsilon_{i\omega t}.$$  

We can see that in the case of non-zero displacement $x_{dc}$, presence of feedback noise generated variance in the trap potential $\delta V_{AC}$ will create an additional noise coupling channel via $\delta x(t)$ at a frequency $\omega_{x,y,z}$. This
noise term is different from the previously discussed noise term at $\omega_{fd}$ and for high enough DC fields starts to limit the cooling temperature, meaning that some way to null DC fields is required.

In our system, the nulling procedure involves measuring the lock-in response to excitation frequencies set way to null DC fields is required. Varying DC voltages at the electrodes modifies the total noise term at $\omega_{fd}$ and for high enough DC fields starts to limit the cooling temperature, meaning that some way to null DC fields is required.

Varying DC voltages at the electrodes modifies the total electric field $E_{dc}$ acting on the particle and by finding a configuration that shows the zero lock-in response (corresponding to $E_{dc} = 0$ and $x_{dc}y_{dc}z_{dc} = 0$ [36]), we can minimize the sensitivity of cooling to the feedback noise effectively. These settings are called the nulling settings and are considered optimal for any further experiments on the nanoplatelets in UHV.

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Author contributions

P.N. performed the experiments, data analysis and wrote the manuscript; J.E.C. helped with graphene nanoplatelets trapping and transfer; B.E.K. designed the experimental setup, initiated and supervised the project.

Competing financial interests

The authors declare no competing financial interests.