Video Article

Trapping of Micro Particles in Nanoplasmonic Optical Lattice

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Abstract

The plasmonic optical tweezer has been developed to overcome the diffraction limits of the conventional far field optical tweezer. Plasmonic optical lattice consists of an array of nanostructures, which exhibit a variety of trapping and transport behaviors. We report the experimental procedures to trap micro-particles in a simple square nanoplasmonic optical lattice. We also describe the optical setup and the nanofabrication of a nanoplasmonic array. The optical potential is created by illuminating an array of gold nanodiscs with a Gaussian beam of 980 nm wavelength, and exciting plasmon resonance. The motion of particles is monitored by fluorescence imaging. A scheme to suppress photothermal convection is also described to increase usable optical power for optimal trapping. Suppression of convection is achieved by cooling the sample to a low temperature, and utilizing the near-zero thermal expansion coefficient of a water medium. Both single particle transport and multiple particle trapping are reported here.

Video Link

The video component of this article can be found at https://www.jove.com/video/56151/

Introduction

The optical trapping of micro-scale particles was originally developed by Arthur Askin in the early 1970s. Ever since its invention, the technique has been developed as a versatile tool for micro- and nanomanipulation[1,2]. Conventional optical trapping based on the far-field focusing principle is inherently limited by the diffraction in its spatial confinement, wherein the trapping force decreases dramatically (following an $\sim a^3$ law for a particle of radius a)[3]. To overcome such diffraction limits, researchers have developed near-field optical trapping techniques based on the evanescent optical field using plasmonic metallic nanostructures and, furthermore, the trapping of nanoscale objects down to single protein molecules has been demonstrated[4,5,6,7,8,9,10,11]. Moreover, the plasmonic optical lattice is created from arrays of periodic plasmonic nanostructures to confer long range transport of micro- and nanoparticles and multiple particle stacking[11,12]. One major obstacle to disrupt trapping in an optical lattice is photothermal convection and efforts have been made to elucidate its effects by several groups[14,15,16,17]. Using Green's function, Baffou *et al.* have calculated a temperature profile by modeling each plasmonic nanostructure as a point heater and then experimentally validated their model[14]. Toussant's group has also measured the plasmon-induced convection with particle velocimetry[15]. The author's group has also characterized both near-field and convectional transport and demonstrated an engineering strategy to suppress photothermal convection[16,17].

Here we present the design of an optical setup and a detailed procedure specifically for trapping experiments with plasmonic optical lattice. The optical potential was created by illuminating an array of gold nanodiscs with a loosely focused Gaussian beam. A scheme to suppress the photothermal convection by cooling down the sample to a low temperature (~4 °C) for optimal trapping is also describe here[17]. Under Boussinesq approximation, an order of magnitude estimate for the natural convection velocity u is given by $u \sim L^2 g\beta\Delta T / \nu$, where L is the length scale of the heat source and ΔT is the temperature increase relative to the reference due to the heating. g and β are the gravitational acceleration and thermal expansion coefficient, respectively. At temperatures near 4 °C, the density of the water medium exhibits anomalous temperature dependence and this translates into a near-zero thermal expansion coefficient and, therefore, a vanishingly small photothermal convection.

Protocol

1. Optical Setup

Note: The principle of the optical setup is illustrated in **Figure 1**.

1. Set up the optical tweezer kit (see the **Table of Materials**) and the fluorescence module (see **Table of Materials**) as per their manuals. Connect a 470 nm blue light emitting diode (LED) light source to the fluorescent module.

2. Replace the high numerical aperture (NA) (NA= 1.25, magnification 100x) oil immersion objective by a long working distance (WD) microscope objective (focal length 3.6 mm, WD=10.6 mm, NA=0.5).
3. Remove the lens in the beam expansion section of the assembled kit to achieve loose focusing of the laser beam.
4. Turn on the power supply and the current for the laser diode of wavelength 980 nm and use the charged coupled device (CCD) camera to make sure the laser beam is aligned properly.
   NOTE: If the laser beam is well-aligned, the CCD camera will read a Gaussian spot.

2. Nanofabrication

1. Marker Fabrication.
   NOTE: Markers will help to position the nanoplasmonic array during the fabrication process and subsequent trapping experiment. The detailed process is illustrated in **Supplementary Figure 1**.
   1. Deposit 40 nm indium tin oxide (ITO) film on a coverslip of thickness 0.17 mm with sputtering.
      NOTE: The ITO film will help discharge electrons during the subsequent e-beam lithography process.
   2. Spin coat an 8 µm layer of positive photoresist with spin speed 4000 rpm and time 30 s with a spin coater.
   3. Soft bake the sample at 90 °C for 5 min and align the sample with the photomask for marker and expose the sample to UV light for 80 s in the mask aligner.
   4. Soak the sample in the photoresist developer for 130 s.
   5. Deposit a 2 nm layer of chromium and a 40 nm layer of gold onto the sample using thermal evaporation.[18]
   6. Soak the sample in acetone and place it in an ultrasonic cleaner operating at 43 kHz and 150 W for 5 min for lift off.

2. Fabrication of Nanoplasmonic Array
   1. Spin coat a layer of e-beam resist PMMA 120K with spin speed 5000 rpm for 30 s on a spin coater. Bake the sample at 160 °C for 3 min on a hot plate.
   2. Spin coat another layer of e-beam resist PMMA 960K with spin speed 5000 rpm for 30 s on a spin coater. Bake the sample at 160 °C for 3 min on a hot plate.
   3. Use e-beam writer to expose the e-beam resist with acceleration voltage 30 kV and dosage 400 C/cm^2.
   4. Deposit a 40 nm layer of gold in a thermal evaporator.
   5. Soak the sample in acetone and place it in an ultrasonic cleaner for 5 min for lift off.

3. Sample cooling system and its temperature calibration

NOTE: The sample cooling stage design is shown in **Supplementary Figure 2**.

1. Making the driver circuit for sample cooling
   1. Place the resistors, bipolar junction transistors, and power metal oxide field effect transistors on the custom circuit board by following the circuit diagram in **Supplementary Figure 3**. Solder all these components with soldering iron.
   2. Connect wires between the control port of the circuit board and the electronic control board. Connect the wires between the output port of the circuit board and thermoelectrical cooling (TEC) element. Place the TEC element on the sample stage with heat sinking.
      NOTE: The TEC element has a hole in the center to allow the laser beam to go through.
   3. Connect wires from the circuit board to 5 V power supply. Use the forward-looking infrared camera to monitor the temperature to check if the thermoelectrical cooling is properly cooling down.

2. Calibration of measured temperature in the forward-looking infrared camera and resistance temperature detector (RTD) thermometer.
   1. Place the RTD thermometer on a blank coverslip and apply a small amount of thermal paste onto it to ensure proper thermal contact between the RTD thermometer and coverslip.
   2. Change the output power setting of the electronic control circuit to TEC element by changing the duty cycle of pulse width modulation setting and wait for 3 min to make sure the steady state temperature is reached. Read the temperature using the RTD thermometer.
   3. Turn on the forward looking infrared camera and monitor the temperature. Repeat this at various output power settings to obtain the temperature calibration curve. A representative temperature calibration curve is shown in **Supplementary Figure 4**.
      NOTE: It is crucial to do calibration between RTD thermometer and the forward looking infrared camera because the temperature reading of the forward-looking infrared camera must be accurate to ensure the correct temperature is reached.

4. Trapping of Microparticles

1. Dilute micro polystyrene particles of diameter 2 µm in deionized water in a microcentrifugetube with proper volume ratio.
   NOTE: The concentration of micro particles can be adjusted according to the objective of the experiment. While lower concentration allows a sample time interval between single particle trapping events, higher concentration will shorten the time for multiple particle trapping. For single particle trapping, a typical concentration is ~0.05% (w/v).
2. Put the sample with nanoplasmonic array on the stage and turn on a 470 nm LED as the fluorescence light source and manually set the power to 5 mW for bright field imaging.
3. Use the marker to locate the nanoplasmonic array, align the sample, and use the CCD camera to make sure the array is in the center of the region of interest on the computer screen.
4. Dispense 10 µL of the diluted micro particles of diameter 2 µm on the sample with a micro pipette.
5. Turn on the current supply to the laser diode of wavelength 980 nm to excite the plasmonic resonance of the array with a power in the range ~1 mW to 10 mW.
6. Manually turn on the power supply to the electronic control board to cool down the sample to a steady state temperature ~4 °C.

7. In the viewer software, click the "record video" sequence to open the recording dialog. Click the "record" button to start the video recording 1.5 of the motion of the micro particles at a frame rate of 10 frames/s over the sample under the influence of laser beam using the CCD camera. Click the "stop" button to stop the recording. See **Video 1**.

Representative Results

Single particle trajectories were recorded by a CCD camera in our experiment and the images were then processed with a custom program to extract each particle's trajectory[16]. Representative results are displayed in **Figure 3 and Video 1** for micro-spheres with diameters of 2 µm. Multiple particle trappings inside the optical lattice were observed. Successive images extracted from a representative motion video of the particle are displayed in **Figure 4**. For microparticles of diameter 2 µm, one can see the clustering of microparticles formed a hexagonal close packed (hcp) structure. The sample can also be warmed up by turning off the TEC element; the observed trapped cluster would disperse due to photothermal convection.

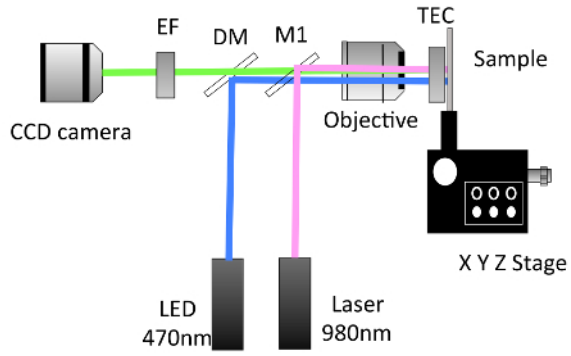


Figure 1. Schematic of the Optical Setup.
A Gaussian beam with wavelength of 980 nm is used to excite the plasmonic optical lattice sample to create trapping potential. A fiber-coupled laser diode of wavelength 980 nm goes through mirror (M1), gets loosely focused by a long working distance microscope objective, and excites the plasmonic sample. The fluorescent image is taken with same objective in conjunction with dichroic mirror (DM) and emission filter (EF) under the fluorescent excitation at 470 nm from light emitting diode light source. The excitation light at 980 nm for the plasmonic resonance is color coded 'pink' and the excitation and emission light for fluorescent imaging are color coded 'blue', and 'green', respectively. The motion is recorded with the CCD camera. Thermoelectrical cooling (TEC) is used to cool down the sample. Please click here to view a larger version of this figure.

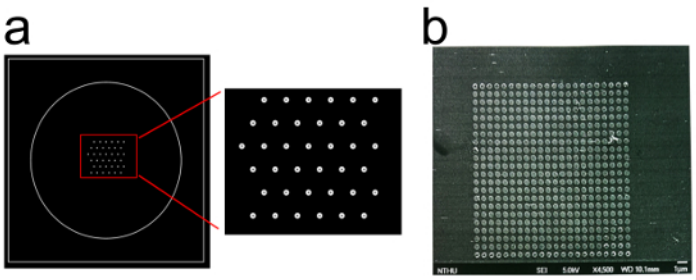


Figure 2. Nano Plasmonic Array Fabricated by E-Beam Lithography. (**a**) Marker design used to locate and align the sample in the e-beam writer. The dimensions of the outer white square are 22 mm x 22 mm, and the annular marker in the inset has an outer diameter of 150 µm and inner diameter of 50 µm. (**b**) A scanning electron microscope (SEM) image of a nanoplasmonic array. A simple square array of 22 x 22 nanodiscs is used and each unit cell contains one nanodisc of thickness 40 nm and diameter 550 nm with inter-disc distance 750 nm. Please click here to view a larger version of this figure.

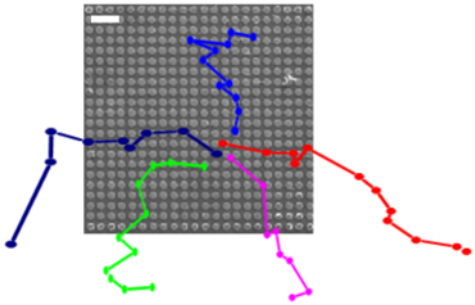


Figure 3. Single Particle Trajectories. The trajectories of the microparticles extracted using image processing are compiled using the centroid algorithm[16] and displayed here. The optical power used for plasmonic resonance excitation at 980 nm is 5 mW. A scale bar of 2 μm is displayed. Please click here to view a larger version of this figure.

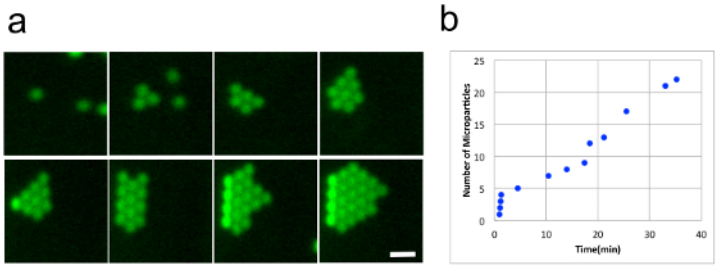
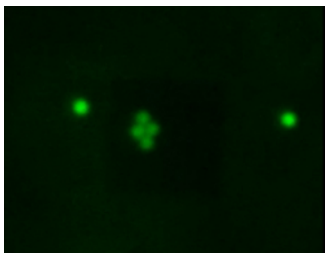
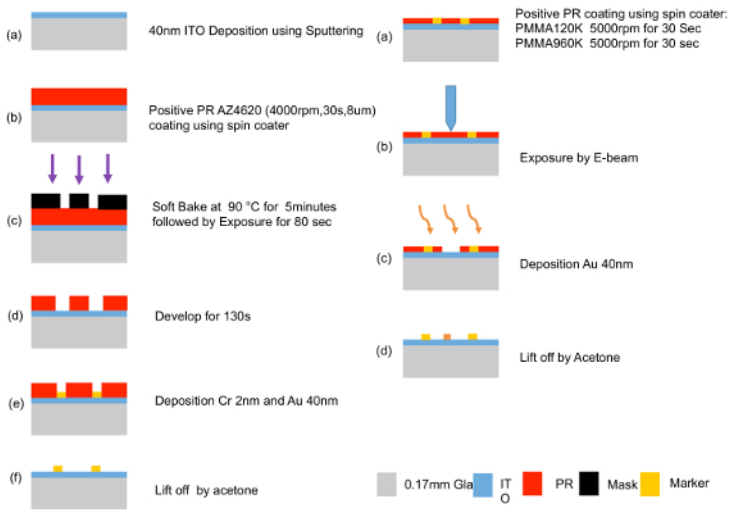


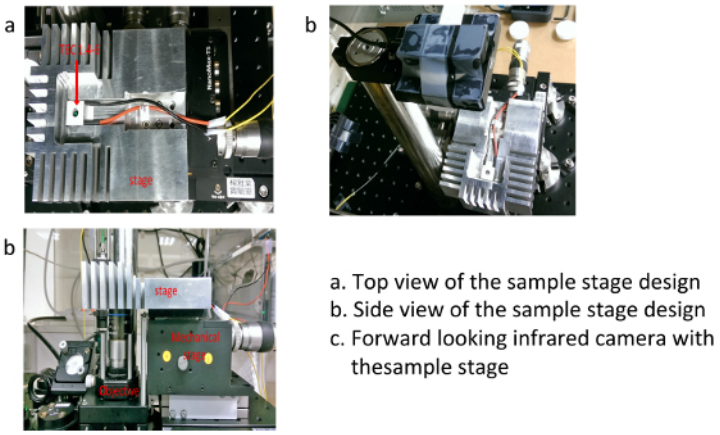
Figure 4. Image of a Cluster of Microparticles Trapped in Plasmonic Optical Lattice and Accumulation of Microparticles Over Time at an Optical Power of 5 mW. (**a**) Successive fluorescence images showing the accumulation of trapped microparticles forming clusters. A white scale bar of 4 μm is displayed. (**b**) Number of trapped microparticles versus time, extracted from (**a**). Please click here to view a larger version of this figure.



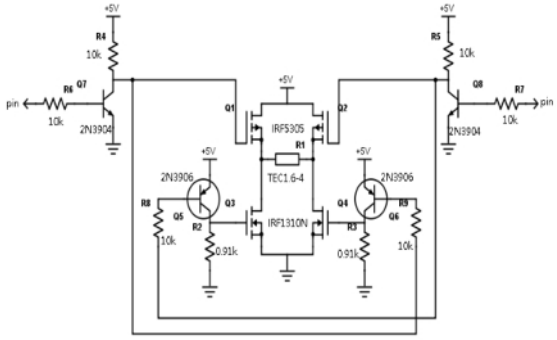
Video 1. Optical Trapping and Particle Accumulation of 2 μm Particles. The optical power used for plasmonic resonance excitation at 980 nm is 5 mW. Please click here to view this video. (Right-click to download.)



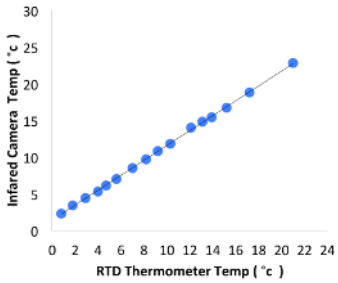
Supplementary Figure 1. Process Flow of Nanofabrication of Nanoplasmonic Array. Please click here to view a larger version of this figure.



Supplementary Figure 2. Sample Cooling Stage Design. Please click here to view a larger version of this figure.



Supplementary Figure 3. Driver Circuit for Sample Cooling. Please click here to view a larger version of this figure.



Supplementary Figure 4. Temperature Calibration between an rtd Thermometer and Forward-Looking Infrared Camera. Please click here to view a larger version of this figure.

Discussion

The procedure described here enables the reader to reliably reproduce trapping on a daily basis. A general empirical guideline to design a usable optical lattice is to use a comparable size for plasmonic nanoarray, interdisc distance, and trapped particle size. Compared to a single, isolated plasmonic nanostructure, the optical lattice design in conjunction with the high optical power afforded by cooling the sample to ~4 °C used here greatly enhances the trapping probability. If well separated, plasmonic nanostructures are used as trapping sites, one needs to wait for a long time for the migration of microparticles into the effective trapping volume near the plasmonic nanostructures. Also, increasing the inter-disc distance critically diminishes the probability of particle accumulation. Note that one can also perform the trapping experiment with the plasmonic optical lattice at room temperature but the usable optical power will be very limited. Also, at low optical power, one needs to wait for a long time (~ 1 h) for the microparticle trapping event. Typically, we turn on the CCD camera to record the particle motion and pick up the trapping event within a few minutes. The applicable laser power is as high as 10 mW. At a high optical power, a large aggregation of microparticles can be observed.

The critical step for success in this work is to cool down the plasmonic on a fragile cover slip and simultaneously monitor the sample temperature. We have chosen the forward looking infrared camera to measure the temperature because such a non-contact measurement greatly reduces the chance of sample breakage. Alternatively, one can choose to glue on a small size thermometer and use it to measure the temperature in real time. The cooling system together with non-contact temperature measurement is generally applicable for optical microscopy at low temperatures.

Although the demonstration here is done with micron-sized particles, one can trap nanoparticles by scaling down both the size and spacing of the gold nanodiscs. So far, trapping of nanoparticles with diameters as small as 100 nm has been demonstrated[19]. This is, however, not the ultimate limit of the plasmonic optical lattice. Also, we have used a long working distance microscope objective to ease the mechanical design of the sample stage. Better resolution in imaging can be achieved by replacing it with an oil immersion microscope objective. By scaling down the dimensions of the plasmonic nanostructures, the trapping of even smaller nanoparticles should also be feasible.[20,21]

Disclosures

The authors have nothing to disclose.

Acknowledgements

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