Simultaneous Positioning and Orientation of Single Nano-Wires Using Flow Control†

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Received Xth XXXXXXXXXX 20XX, Accepted Xth XXXXXXXXX 20XX
First published on the web Xth XXXXXXXXXX 20XX
DOI:

We present a material-property independent method for manipulating both the position and orientation of nanowires (NWs), by feedback control of flows. For example, the NWs need not be electromagnetically polarizable. Control of NWs in a microfluidic device is demonstrated across a 170 μm $\times$ 170 μm region with on-demand trapping, translation, and simultaneous rotation of dielectric, semiconducting, and metallic NWs. An average trapping precision of 0.6 μm in position and 5.4° in orientation is achieved for the NWs considered, making it attractive for sensing and directed assembly applications.

1 Introduction

Sensing and directed assembly applications that use single nanoscale objects require control over their position and orientation. The ability to position and orient individual NWs can be used to build structures that sense and guide electromagnetic waves,[1,2] steer a nanowire (NW) light source to image nanostructures,[3] manipulate NWs to precisely deliver chemical doses to specific parts of a cell,[4] and align NWs to detect chemicals[5] by monitoring structural changes along the alignment direction caused by chemical adsorption. Such an ability could also be used to map intensities and polarizations of microscale force fields over a large area using nanowire probes. Flow control has the advantage that the controlled fluid can exert forces and hence manipulate objects of any material type. Since viscous forces scale with object length rather than volume, electro-osmotic flow control (EOFC) has enabled the positional manipulation of nanoscopic objects that are difficult to actuate by optical and dielectrophoretic means, and has done so with nanoscale precision.[6]

Prior techniques for manipulating both the position and orientation of nanoscale objects include sculpting optical wavefronts to rotate optically polarizable objects[7–14] or using plane wavefronts to rotate birefringent ones,[15,16] using dielectrophoretic forces to position and orient objects that are polarizable at radio frequencies,[17–25] and magnetic actuation to manipulate magnetically susceptible objects.[26–29] A common feature of all these techniques is that they rely on intrinsic material properties, such as polarizability, of the manipulated object[7–29] (or of those in its vicinity[30]). A magnetic field for example, cannot directly control a non-magnetic object.

Here we show how precise flow control using EOF can be used to control not only the position, but also simultaneously the orientation, of NWs in a 2D plane. Instead of using forces and torques that rely on the properties of the wire, we create electro-osmotically actuated flow patterns that move and rotate the wire by viscous stresses. We achieve essentially the same degree of control over dielectric, semi-conducting and conducting NWs, thus showing that the technique is effective regardless of the material properties of the nano-object. The NWs are controlled in a simple polydimethyl siloxane (PDMS)-mold-based device, with a one-time requirement for mold microfabrication.

2 Results and discussion

An electric field applied across a microfluidic channel creates a plug flow (EOF) whose velocity is directly proportional to the field strength[31,32] (see SI for details on EOF physics). An immersed nano-object moves with the local flow velocity due to the applied viscous stress, which depends on the object's dimensions and orientation.

Here we use EOF within a vision-based feedback loop to control the NW motion as shown in Fig. 1. Since electric fields are curl-free one cannot create a circulating flow to rotate the object. Instead, shear flows are used to rotate objects with less symmetry than a sphere, e.g. a NW,[33] as shown in the top panel of Fig. 1. Voltages applied at peripheral elec-

† Supplementary Information (SI): Supplementary text and movies can be downloaded. Movie M1 (sped up to 4X real time) shows control of the SU8 rod along the "NIST" path. Movies M2, M3 and M4 (all real time) shows rotation and trapping of SU8, silicon and gold NWs respectively.DOI:

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trodes create EOF in the four microchannels and in the central control region where the four channels meet. Precise NW manipulation, using controlled translational and shear flows, is achieved by choosing the strength and orientation of the electric field guiding the EOF based on the NW's current position and orientation.

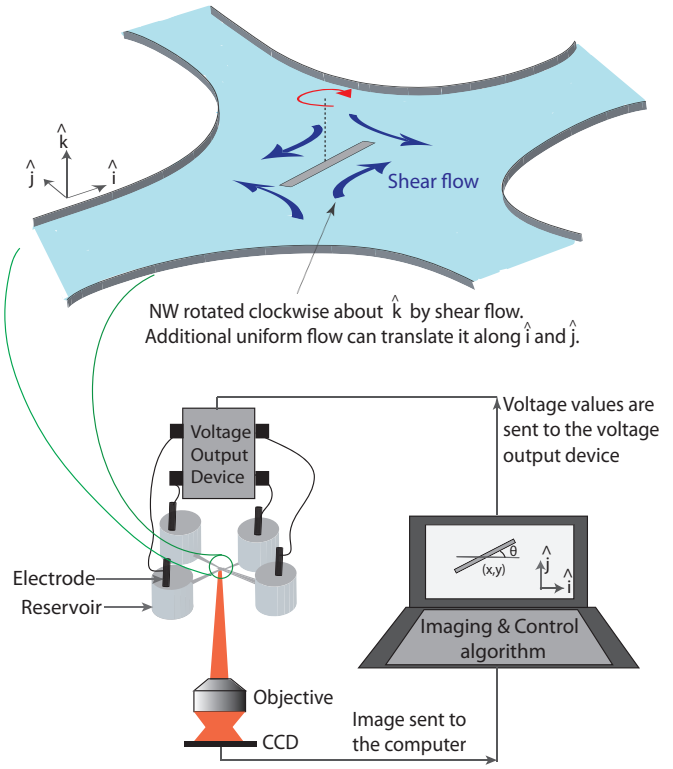

Fig. 1 Schematic of the microfluidic device (top, magnified) and the feedback hardware loop (bottom). The position and orientation of the NW (grey rod) is observed through the microscope objective at each time step. Based on this, a computer algorithm determines the quartet of voltages that will create a fluid flow (blue arrows) to translate and rotate the NW from where it is towards where it should be (only rotation is shown in the figure). The same process repeats at the next time step to continuously move and rotate the NW along its desired trajectory.

Each cycle of the feedback loop starts with the charge-coupled device (CCD) camera sensing the NW image. An image processing algorithm estimates the NW's position and orientation using centroiding and least-squared best-fits respectively (see SI for details of position and orientation estimation). These estimates are compared to the next targeted position and orientation along a user-specified trajectory. The difference is multiplied with constant gain parameters - one for position and one for orientation - giving the needed translational and rotational velocity of the NW that will take it to the next desired target position and orientation. The quartet of voltages that can impart these needed velocities is calculated using a pre-computed map that linearly relates the voltages to NW-velocities. See Equation 10 of our previous work[33] for a detailed theoretical derivation of the control law used in the present experiments. An output device applies this quartet of voltages at the electrodes, entraining the NW in fluid flow towards the target. Repeated executions of this feedback loop continually shape the fluid flow near the NW in a way that counteracts Brownian perturbations and enables the object to accurately track the desired trajectory. Specifically, translational flows move the wire and applied shear flows can rotate it. Although the shear flow shown in Fig. 1 is curl-free (irrotational), it will rotate the NW clockwise because the clockwise components of the flow will apply more torque on the long axis of the wire than the equal-and-opposite counter-clockwise components will apply on the short axis. Thus, the asymmetry of the wire allows it to be controllably rotated by an irrotational electro-osmotic flow.

We demonstrate the controlled translation and rotation of three electrically different objects - dielectric rods (made of SU8[34], a photoresist), metallic (gold) NWs, and semiconducting (silicon) NWs - in water. Perturbations due to Brownian motion, residual pressure flows due to unequal heights of fluid in the device reservoirs, and possible non-uniformities in the zeta potential (potential differences across the Debye layer) at the device-fluid interface[35] are all compensated for by the feedback control loop.

The feedback loop in all experiments is updated in 33 ms time-steps (corresponding to the frame rate of the camera) with the magnitude of the maximum voltage at each of the electrodes limited to $V_{max} = 10$ V to prevent hydrolysis.[36]

Control of a 10 μm $\times$ 1 μm fluorescently labeled SU8 rod (lithographically fabricated in-house) in water is shown in movie M1 (see SI, movie is sped up to 4X real time), four snapshots of which are shown in Fig. 2. In each update of the feedback loop, the center of mass of the rod is directed towards a different target on the pre-planned path - the word "NIST" in Fig. 2 - at an average speed of 4 μm/s. The rod is simultaneously rotated until its long axis is tangential to each segment in this path by the time its center of mass completes tracking that segment.[37]

The center of mass positional error along the x-axis at time-step i is defined as $e_{x,exp}(i) = x_{des}(i-1) - x_{obs}(i)$ where $x_{des}(i)$ and $x_{obs}(i)$ are the desired and observed x-coordinates of the rod center of mass at time step i. This definition respects the fact that the observed center of mass position at time-step i should be compared to the one demanded at time-step $i-1$. Mean $\langle e_{x,exp}(i)\rangle$ and root mean square (RMS) $\langle e^2_{x,exp}(i)\rangle^{1/2}$ values are measures of the x-positioning accuracy and precision respectively, averaged over the entire trajectory. The y-position error $e_{y,exp}(i) = y_{des}(i-1) - y_{obs}(i)$ and the orientational error $e_{\theta,exp}(i) = \theta_{des}(i-1) - \theta_{obs}(i)$ are defined similarly. Mean errors for this experiment were mea-

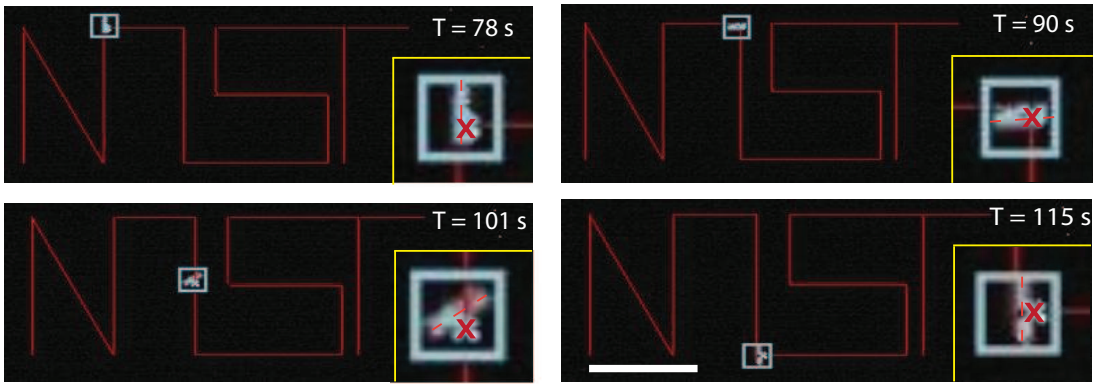

Fig. 2 Snapshots of a 10 μm $\times$ 1 μm fluorescently labeled SU8 rod being controlled along the "NIST" path graphically underlaid in red (movie M1 in SI). The rod is made to simultaneously rotate and align itself tangential to each of the 12 line segments by the time its center of mass reaches the end of a segment. Each image shows the rod at different locations along the trajectory at the noted times. The orientation of the rod is clarified in each inset with a dotted red line. The desired position of the rod's center of mass at the next time step is clarified in each inset with a red cross. Scale bar = 50 μm.

sured to be $\langle e_{x,exp}(i)\rangle = 0.22$ μm, $\langle e_{y,exp}(i)\rangle = 0.28$ μm and $\langle e_{\theta,exp}(i)\rangle = 1.3^\circ$. The RMS errors were $\langle e^2_{x,exp}(i)\rangle^{1/2} = 1.1$ μm, $\langle e^2_{y,exp}(i)\rangle^{1/2} = 1.2$ μm and $\langle e^2_{\theta,exp}(i)\rangle^{1/2} = 15.4^\circ$.

Next, the device performance was characterized by trapping individual SU8 rods (10 μm long, 1 μm wide, lithographically fabricated, in-house), silicon NWs (10 μm long, 100 nm wide, grown in-house) and gold NWs (10 μm long, 100 nm wide, methyl-terminated, Nanopartz Inc.[34]) at a fixed position and orientation. In each case, the NW was trapped positionally, rotated to an orientation of 0° and then maintained at that position and orientation for at least 45 s. Movies M2, M3 and M4 (see SI, all three movies in real time) show trapping of SU8, Si and Au NWs respectively. Fig. 3 shows snapshots and trajectories of the NWs while Table 1 lists errors.

Small and non-uniform optical scattering cross-sections of the Si and Au NWs occasionally results in NW image intensity falling below the camera noise threshold. Resultant errors in orientation estimates at such instants are recorded as spikes in the orientation time trace in Fig. 3. However, the control scheme is robust and control is resumed after the NW rotates past the spikes, as shown in the figure. Such errors could be reduced for critical applications by using a more sensitive camera and a darkfield objective.

Trapping errors scale with the relative strengths of the actuation and the diffusional perturbations. For a NW of length l, the rotational diffusion coefficient D_θ scales as l^{-3} whereas the translational diffusion coefficient scales as l^{-1}.[38] Simulations showed[33] that most of the voltage actuation is spent in compensating rotational, rather than translational, diffusion. Voltages required to completely compensate for the rotational diffusion perturbation frequently exceed the -10 V to 10 V range that was set to prevent hydrolysis. If the entirety of this range is available to combat rotational diffusion, then a Fokker-Planck analysis[39] yields (neglecting orientation measurement errors) the following theoretical RMS orientation error estimate for trapping

$$\langle e^2_{\theta,theo}\rangle^{1/2} = \sqrt{2}\frac{D_\theta}{\omega} \quad (1)$$

where ω is the angular velocity afforded by the voltage range that is available to combat rotational diffusion.

Table 1 Trapping errors for dielectric (SU8), semi-conducting (silicon) and conducting (gold) NWs: Each NW was trapped at a fixed position and orientation for at least 45 s. In the traps' absence, the expected RMS orientational deviation $\sqrt{\langle e^2_{\theta,exp}(i)\rangle}$ would exceed 40° within 45 s due to rotational diffusion. In the traps' presence, $\sqrt{\langle e^2_{\theta,exp}(i)\rangle}$ is lower than the theoretical estimate (last row) indicating that the available angular velocity is higher than the median used in the estimate.

Error Metric	SU8	SiNW	AuNW
$\langle e_{x,exp}(i)\rangle \pm \sqrt{\langle e^2_{x,exp}(i)\rangle}$	0.60 μm $\pm$0.28 μm	0.44 μm $\pm$0.20 μm	$-$0.81 μm $\pm$0.21 μm
$\langle e_{y,exp}(i)\rangle \pm \sqrt{\langle e^2_{y,exp}(i)\rangle}$	0.73 μm $\pm$0.37 μm	$-$1.31 μm $\pm$0.64 μm	$-$1.48 μm $\pm$0.60 μm
$\langle e_{\theta,exp}(i)\rangle \pm \sqrt{\langle e^2_{\theta,exp}(i)\rangle}$	1.52° $\pm$4.71°	$-$1.14° $\pm$5.33°	$-$4.28° $\pm$6.16°
$\sqrt{\langle e^2_{\theta,theo}\rangle} = \frac{180}{\pi}.\frac{\sqrt{2}\langle D_\theta\rangle}{\omega}$	6.94°	6.48°	8.10°

We now compare Eqn. 1 to experiments by independently measuring D_θ and ω for the three NWs that were trapped.

An additive diffusion noise model was used to estimate D_θ and the measurement noise $\sqrt{\langle\theta^2\rangle_{meas}}$, which arises due to

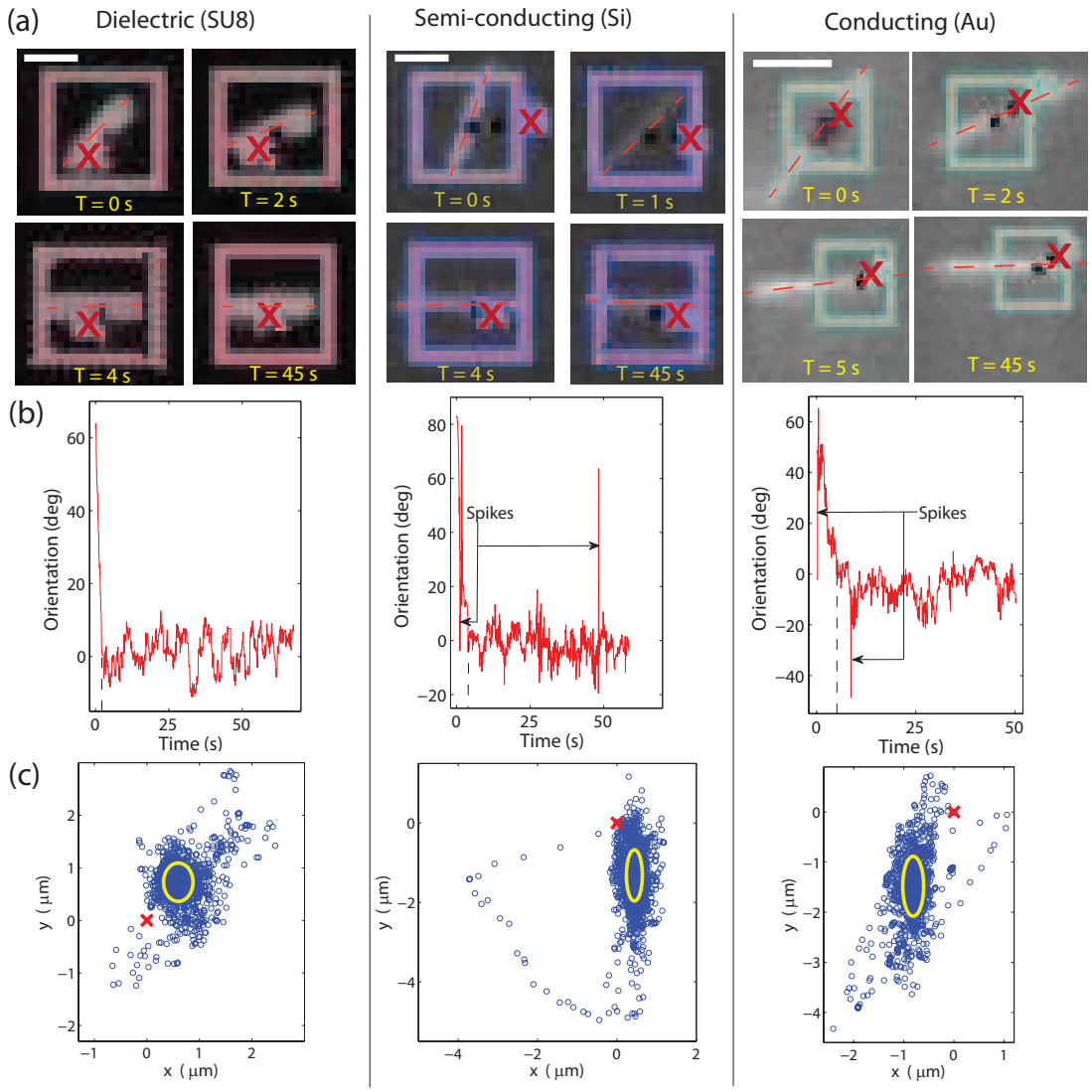

Fig. 3 Each of the three columns show rotation of a NW to $\theta = 0^\circ$ about a trapping location followed by holding at $\theta = 0^\circ$. Row (a) shows snapshots of NWs at indicated times with their orientations clarified by dotted red lines. Red crosses show the intended trapping position at $(0,0)$. Scale bars $= 5\ \mu$m. NW orientation data θ versus time t is shown in row (b). Spikes in $\theta(t)$ for Si and Au NWs indicate momentary errors in the orientation estimates due to NW image intensity falling below the camera's noise threshold but control is resumed after the NW rotates past the spikes. Black dotted lines indicate the time t_{FP} when the orientation of each NW first passes $\theta = 0^\circ$. Blue circles in row (c) plots show the NW center of mass estimates for times $t > 0$. Axes lengths of the yellow ellipse indicate RMS errors $\langle e^2_{x,exp}(i)\rangle^{1/2}$ and $\langle e^2_{y,exp}(i)\rangle^{1/2}$ for times $t(i) > t_{FP}$.

camera noise and thresholding/pixelation errors in image processing (see SI and references[40,41] for details of this estimation). The diffusion coefficients for the SU8, Si and Au NWs were measured to be $(6 \pm 2) \times 10^{-3}$ rad^2/s, $(4 \pm 1) \times 10^{-3}$ rad^2/s and $(8 \pm 2) \times 10^{-3}$ rad^2/s respectively while $\sqrt{\langle \theta^2 \rangle_{meas}}$ measured 1.15°, 0.57° and 3.44° respectively.

The angular velocity ω that is available in experiments is lower than that suggested by the -10 V to 10 V voltage range. This is because part of the actuation is consumed in compensating for the residual pressure flow perturbation to NW motion that is observed in experiments (this perturbation is translatory in nature, see SI). The voltage range, and thus ω, that is effectively available for combating rotational diffusion is reduced as a result. The available ω is conservatively estimated to be the median of the angular velocities imparted by the actuation in each update of the feedback loop. The angular velocity imparted by EOFC in each update can be estimated by substituting the voltages, NW position and orientation values from each update into the EOF-physics based map relating the NW angular velocity to the applied voltages (see SI). The

median angular velocity while trapping SU8, Si and Au NWs were measured to be 0.07 rad/s, 0.05 rad/s and 0.08 rad/s respectively. These values are substituted for ω in Eqn. 1, yielding theoretical estimates of RMS orientational precision that is comparable to the $\approx 5.4^\circ$ precision observed experimentally (see Table 1). The lower values of the experimental precision compared to theoretical estimates in Table 1 suggests that the available angular velocity is in fact larger than the median used in the estimates.

Compared to other techniques for manipulating both the position and orientation of nano-objects, our main advantage is that we can manipulate NWs of any type (dielectric, semiconducting or metallic). Prior techniques for manipulating NWs have created potential wells, that counteract Brownian perturbations, by exploiting specific material properties of the NW, like its polarizability. For example, a 2 μm long SiNW was optically trapped in a recent experiment[11] to an oriental precision of 3°. Micron sized silica and quartz particles have been rotated with angular velocities between 10 and 200 rad/s - a higher range than our technique - by exploiting properties like optical polarizability[10] and birefringence[16] (trapping precisions were not reported). For both optical and dielectrophoretic (DEP) actuations, trapping only works when the optical beam and AC electric fields respectively, are tuned to the dielectric properties of the NW.[13,25] Additionally, unlike our technique, high electric fields required for DEP necessitate electrode patterning near the control region (with such patterns however, parallel assembly of 15 μm long NWs, each spanning 10 μm $\times$ 1 μm sized inter-electrode gaps, has been shown[18]). In our technique, as long as the NW is bright enough to be imaged and thus spatially and orientationally localized, the feedback control technique does not depend on, nor is it significantly perturbed by, any other material properties of the NW.

3 Conclusion

We have experimentally demonstrated simultaneous position and orientation control of 10 μm long nanowires using electro-osmotic flow control (EOFC). Since fluid flow applies viscous forces to all material types, our method can be used to manipulate nanowires irrespective of which material they are made from - we demonstrate this by manipulating dielectric, semiconducting and metallic NWs. The wires are manipulated in a microfluidic device over a 170 μm $\times$ 170 μm control region with an average trapping precision of 0.6 μm in position and 5.4° in orientation. The flow control capability that we have shown here relies on fluid shear turning asymmetric objects, and will equally apply to manipulation of objects of other shapes. This technique can, in principle, be employed to map intensities and polarizations of microscale fields by scanning a probe object over the control region - material-independence would allow the selection of probes based on optimum sensitivity to the target field, without any constraint from the actuation mechanism.

Acknowledgments. We thank A. Berglund (CNST-NIST), R. Probst and Z. Cummins (both UMD) for helpful discussions. We also thank the following people at CNST-NIST: A. Band, D. Rutter and G. Holland for instrument setup; A. Talin and D. Ruzmetov for providing a sample of SiNWs. PPM acknowledges support under the cooperative research agreement between UMD and NIST-CNST (award 70NANB10H193).

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