

February 21, 2007

Three dimensional controlled assembly of gold nanoparticles using a micromachined platform

Nishant Khanduja

Department of Electrical and Computer Engineering, Northeastern University

Selvapraha Selvarasah

Department of Electrical and Computer Engineering, Northeastern University

Chia-Ling Chen

Department of Electrical and Computer Engineering, Northeastern University

Mehmet R. Dokmeci

Department of Electrical and Computer Engineering, Northeastern University

Xugang Xiong

Department of Mechanical and Industrial Engineering, Northeastern University

See next page for additional authors

Recommended Citation

Khanduja, Nishant; Selvarasah, Selvapraha; Chen, Chia-Ling; Dokmeci, Mehmet R.; Xiong, Xugang; Makaram, Prashanth; and Busnaina, Ahmed A., "Three dimensional controlled assembly of gold nanoparticles using a micromachined platform" (2007). *Center for High-Rate Nanomanufacturing Publications*. Paper 11. <http://hdl.handle.net/2047/d20000922>

Author(s)

Nishant Khanduja, Selvapraba Selvarasah, Chia-Ling Chen, Mehmet R. Dokmeci, Xugang Xiong, Prashanth Makaram, and Ahmed A. Busnaina

Three dimensional controlled assembly of gold nanoparticles using a micromachined platform

Nishant Khanduja, Selvapraba Selvarasah, Chia-Ling Chen, and Mehmet R. Dokmeci^{a)}
*Department of Electrical and Computer Engineering, Northeastern University,
 Boston, Massachusetts 02115*

Xugang Xiong, Prashanth Makaram, and Ahmed Busnaina
*Department of Mechanical and Industrial Engineering, Northeastern University,
 Boston, Massachusetts 02115*

(Received 16 October 2006; accepted 18 January 2007; published online 21 February 2007)

By using optical lithographic procedures, the authors present a micromachined platform for large scale three dimensional (3D) assembly of gold nanoparticles with diameters of ~ 50 nm. The gold nanoparticles are formed into 3D low resistance bridges (two terminal resistance of ~ 40 Ω) interconnecting the two microelectrodes using ac dielectrophoresis. The thickness of the parylene interlevel dielectric can be adjusted to vary the height of the 3D platform for meeting different application requirements. This research represents a step towards realizing high density, three dimensional structures and devices for applications such as nanosensors, vertical integration of nanosystems, and characterization of nanomaterials. © 2007 American Institute of Physics.

[DOI: [10.1063/1.2679882](https://doi.org/10.1063/1.2679882)]

The organization of nanoscale materials (of size <100 nm)¹⁻³ into ordered structures serves as a building block towards realizing devices of the future having significant technological importance. Attractive properties of nanomaterials such as large surface to volume ratio,^{4,5} high packing density, and long-range order⁶ may serve to be potentially useful in emerging commercial applications. However, the lack of progress and innovation for integrating these nanomaterials into microdevices and/or providing a means to characterize the manufactured nanomaterials has been a bottleneck which prevents the translation of these discoveries into commercial products of nanotechnology. A possible solution could be to develop methods for the incorporation of nanoscale materials onto platforms as components of microsystems or for in-line characterization of nanostructures after manufacturing.

In this respect, there have been several approaches for selective positioning of nanometer sized materials as components of functional devices. However, most of these approaches have shortcomings to find wide spread use specifically for high rate nanomanufacturing. The use of atomic⁷⁻⁹ and scanning force microscopy^{10,11} has been demonstrated, yet they have a limited range of operation with a relatively low throughput. Optical tweezers,^{12,13} carbon nanotube nanotweezers,¹⁴ and nanorobotic manipulations¹⁵ are other alternatives for achieving nanoassembly; however, their initial equipment setup is quite complicated. Hence trapping and manipulating nanostructures using these approaches are severely limited by the size and type of nanomaterials used. Electron-beam lithography is another common tool for creating nanoassembly platforms, yet it suffers from low throughput and is not suitable for volume manufacturing. Another method for selective growth of nanostructures as demonstrated by Kreupke *et al.*¹⁶ for growing carbon nanotubes requires elevated temperatures (>500 °C) and hence is not compatible with integrated circuit fabrication techniques.

Most importantly, many of these methods are achieved on a planar two dimensional (2D) surface.

By selective assembly of nanostructures in a three dimensional architecture, one can drastically increase the density of nanosystems and simultaneously provide a means for their integration into microdevices. Accordingly, by utilizing a simple, two mask, self-aligned process, we introduce a micromachined platform for the integration of nanostructures into microdevices in a three dimensional architecture using dielectrophoresis. Gold nanoparticles have small size, good stability, and established bioconjugation properties which make them ideal as assays for heterogenous DNA detection.^{17,18} Velev and Kaler¹⁹ have functionalized gold nanoparticles with DNA molecules and utilized this device as a biosensor. Thiolate-coated gold nanoparticles have also been used for gas/vapor sensors.²⁰⁻²² The possibility of tuning the properties of gold nanoparticles makes them promising candidates for a group of electrical and optical applications. Our technology provides a high density platform for these emerging nanotechnology applications.

Dielectrophoresis (DEP) is a popular method for manipulation, trapping, and separating micro- and nanoparticles, as it is inexpensive and achieved at low temperatures with a high yield. The biasing scheme for DEP together with the 3D platform is illustrated in Fig. 1(a). When a polarizable object is placed in a nonuniform electric field, due to its interaction with the field, it exhibits a translation motion which forms the basis of dielectrophoretic assembly.²³ By applying an ac voltage between the two electrodes, an electric field is created which results in a dielectrophoretic force that attracts the nanocomponents into gaps where the gradient of the electric field is maximum or minimum. Dielectrophoresis occurs in both ac and dc electric fields; however, ac fields are preferred since they allow manipulation and assembly of the nanocomponents while minimizing and/or suppressing the electrochemical and particle migration effects present using dc fields.^{24,25} Chan *et al.*²⁶ have demonstrated the use of DEP for manipulation of carbon nanotubes for

^{a)}Electronic mail: mehmetd@ece.neu.edu

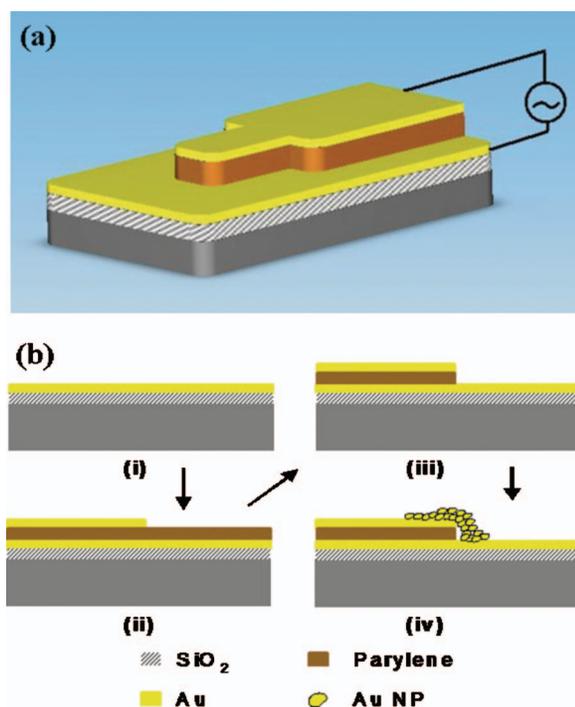


FIG. 1. (Color online) (a) Schematic of the 3D micromachined platform for dielectrophoretic assembly of nanostructures. (b) Process flow for the fabrication of the 3D platform.

nanosensing applications while Washizu and Kurosawa²⁷ and Zheng *et al.*²⁸ have shown that DNA and proteins can also be assembled using ac electric fields. Amlani *et al.*²⁹ and Kretschmer Fritzsche³⁰ have demonstrated DEP assembly of gold nanoparticles over a 2D structure, and we extend this method for assembling gold nanoparticles on our 3D platform.

The 3D nanoassembly platform is fabricated using a two mask self-aligned process. A schematic of the fabrication process is illustrated in Fig. 1(b). A 1 μm thick isolation oxide is first thermally grown on a 3 in. silicon wafer. A Cr/Au (400 \AA /1500 \AA) layer is deposited and patterned using lift-off to serve as the first metal layer [Fig. 1(b)(i)]. Next, a thin (0.7 μm), pinhole-free parylene-C dielectric layer is deposited on the wafer at room temperature. The second metal layer [Cr/Au (400 \AA /1500 \AA)] is then deposited and patterned using lift-off [Fig. 1(b)(ii)]. The two metal layers serve as the electrodes to assemble the nanostructures. By using the second metal layer as a hard mask (in a self-aligned manner), we etch the parylene-C layer in an inductively coupled plasma (Plasmaterm 790) using O₂ [Fig. 1(b)(iii)]. In these experiments, commercially available Au nanoparticles (Ted Pella, Inc., Redding, CA) dissolved in de-ionized water are utilized (4.5×10^{10} particles/ml). The average diameter of the Au nanoparticles is between 40 and 50 nm. After applying an ac voltage of 8 V_{p-p} (peak to peak) at 1 MHz²⁹ to the microelectrodes using a function generator (Agilent 33220A), a droplet (2–3 μl) of the solution containing the nanoparticles is dispensed onto the chip with a pipette. After 5 min of assembly at room temperature, the sample is blow dried with nitrogen and the power is turned off resulting in the 3D assembled nanoparticle bridges [Fig. 1(b)(iv)].

For these assembly experiments, we have designed microelectrodes with multiple fingers as illustrated in the opti-

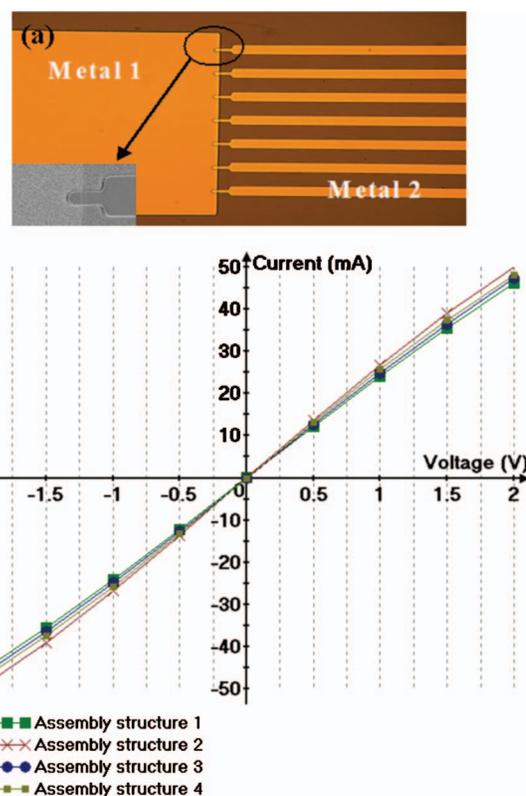


FIG. 2. (Color online) (a) Optical image of an assembly finger structure designed for high rate nanoassembly. The inset shows a SEM micrograph of one of the fingers. (b) I - V characteristics of the assembled gold nanoparticles. The curve scales linearly over the entire voltage range with a low resistance.

cal image of Fig. 2(a) with a scanning electron microscopy (SEM) (Zeiss Supra 25) micrograph shown in the inset of the same figure. The assembly width of each finger is designed as 2 μm with a 3 μm overlap with the first metal layer. Kretschmer and Fritzsche³⁰ have investigated the relationship between the nanoparticle size and the microelectrode gaps. They claim that one cannot reliably achieve controlled assembly of 50 nm Au nanoparticles with electrode gaps exceeding 1 μm . The assembly gap for our approach was 0.7 μm (thickness of the parylene-C layer), yet it can be easily changed for assembling other nanostructures.

After performing the nanoassembly, the current-voltage (I - V) characteristics are first measured to verify the continuity of the assembly. Figure 2(b) shows the output curves measured from the nanoparticle bridges [Fig. 3(a)] connecting the two microelectrodes using an HP 4155A parameter analyzer. From the graph, we see a near Ohmic behavior, as expected since DEP is effective on conductive elements. The measured resistance values ranged between 37 and 50 Ω , with variations due to different assembly times. An average two terminal resistance of $\sim 40 \Omega$ was obtained reproducibly. The resistances reported by Zheng *et al.*²⁸ Amlani *et al.*,²⁹ and Kretschmer and Fritzsche³⁰ using Au nanoparticles on planar surfaces are relatively greater than what we have obtained which may be justified by the differences in size and amount of nanoparticles and also the difference in the assembly platform.

Since the size of the nanoparticles (~ 50 nm) is much smaller than the electrode gap (0.7 μm), a large number of them cluster together to form a bridge as shown in the SEM

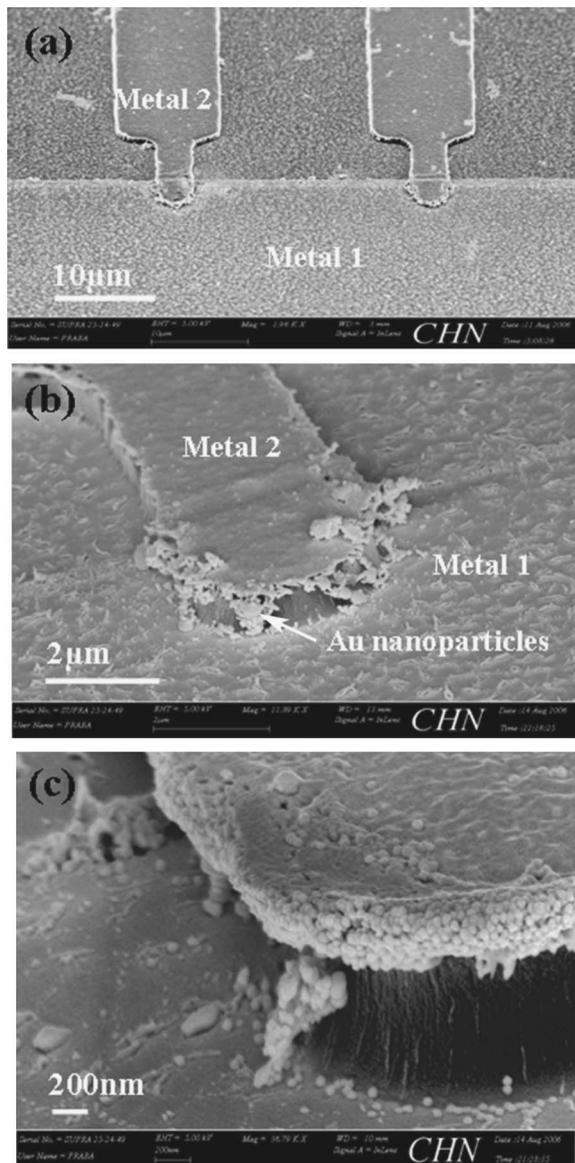


FIG. 3. (a) Distant SEM micrograph of the fabricated finger structure after assembly and [(b) and (c)] magnified view showing the 3D nanoparticles.

micrographs [Figs. 3(b) and 3(c)]. The large globular structures observed in Fig. 3(c) are possibly due to the melting of the particles due to current heating after a path is formed between the two electrodes.²⁷ One possible solution to prevent this would be to reduce the applied voltage; however, this may proportionally increase the assembly time. Another alternative is to use a series resistance in the current path or the use of a nanofuse²⁷ which could reduce the fusing of nanoparticles caused after a connection is formed between the two electrodes.

In summary, we have designed and fabricated a two mask micromachined platform for three dimensional assembly of nanomaterials. Using dielectrophoresis, we have integrated Au nanoparticles of diameter of ~ 50 nm into 3D bridges with a two terminal resistance of $\sim 40 \Omega$. The I - V curves of the room temperature assembled structures demonstrate a linear behavior. This approach is versatile and can be

extended to the integration of other conductive nanomaterials including nanowires, nanobelts, and nanorods into miniaturized devices and systems for the amalgamation of micro- and nanotechnologies. Furthermore, due to its vertical nature, it can potentially be used in the making of high density stacked systems, high rate nanoassembly of 3D nanodevices, and in-line characterization of nanostructures.

The authors acknowledge financial support from the Air Force Research Laboratory, Hanscom, MA, Contract No. FA8718-06-C-0045, the Nanoscale Science and Engineering Research Centers program of the National Science Foundation (Award No. NSF-0425826), and the National Science Foundation (Award No. NER-0608892).

- ¹K. Keren, R. S. Berman, E. Buchstab, U. Sivan, and E. Braun, *Science* **302**, 1380 (2003).
- ²T. Ono, H. Miyashita, K. Iwami, S. J. Kim, Y. C. Lin, and M. Esashi, *Proceeding of the International Symposium on Micro-Nanomechanics and Human Science*, 31 October-3 November 2004, pp. 39–46.
- ³L. Sun, F. Banhart, A. V. Krashennikov, J. A. Rodriguez-Manzo, M. Terrones, and P. M. Ajayan, *Science* **312**, 1199 (2006).
- ⁴S. J. Limmer, T. P. Chou, and G. Cao, *J. Phys. Chem. B* **107**, 13313 (2003).
- ⁵S. J. Limmer and G. Cao, *Adv. Mater. (Weinheim, Ger.)* **15**, 427 (2003).
- ⁶A. Docoslis and P. Alexandridis, *Electrophoresis* **23**, 2174 (2002).
- ⁷T. Junno, K. Deppert, L. Montelius, and L. Samuelson, *Appl. Phys. Lett.* **66**, 3627 (1995).
- ⁸M. R. Falvo, G. Clary, A. Helsen, S. Paulson, R. M. Taylor, V. Chi, F. P. Brooks, S. Washburn, and R. Superfine, *Microsc. Microanal.* **4**, 504 (1998).
- ⁹M. Sitti and H. Hashimoto, *IEEE/ASME Trans. Mechatron.* **5**, 199 (2000).
- ¹⁰R. Resch, D. Lewis, S. Meltzer, N. Montoya, B. E. Koel, A. Madhukar, A. A. G. Requicha, and P. Will, *Ultramicroscopy* **82**, 135 (2000).
- ¹¹R. Resch, C. Baur, A. Bugacov, B. E. Koel, A. Madhukar, A. A. G. Requicha, and P. Will, *Langmuir* **14**, 6613 (1998).
- ¹²K. Castelino, S. Satyanarayana, and M. Sitti, *Proceedings of the 2003 IEEE-NANO*, 12–14 August 2003, Vol. 2, pp. 56–59.
- ¹³N. G. Dagalakis, T. LeBrun, and J. Lippiatt, *Proceedings of the 2002 IEEE-NANO*, 26–28 August 2002, p. 177–180.
- ¹⁴P. Kim and C. M. Lieber, *Science* **286**, 2148 (1999).
- ¹⁵A. A. G. Requicha, C. Baur, A. Bugacov, B. C. Gazen, B. Koel, A. Madhukar, T. R. Ramachandran, R. Resch, and P. Will, *Proceedings of the IEEE Conference on Robotics and Automation*, 16–20 May 1998, Vol. 4, pp. 3368–3374.
- ¹⁶R. Krupke, F. Henrich, H. V. Lohneysen, and M. M. Kappes, *Science* **301**, 344 (2003).
- ¹⁷W. Fritzsche and T. A. Taton, *Nanotechnology* **14**, R63 (2003).
- ¹⁸Y. W. C. Cao, R. Jin, and C. A. Mirkin, *Science* **297**, 1536 (2002).
- ¹⁹O. D. Velev and E. W. Kaler, *Langmuir* **15**, 3693 (1999).
- ²⁰H. L. Zhang, S. D. Evans, J. R. Henderson, R. E. Miles, and T. H. Shen, *Nanotechnology* **13**, 439 (2002).
- ²¹H. Wohltjen and A. W. Snow, *Anal. Chem.* **70**, 2856 (1998).
- ²²L. Han, D. R. Daniel, M. M. Maye, and C. J. Zhong, *Anal. Chem.* **73**, 4441 (2001).
- ²³H. A. Pohl, *Dielectrophoresis: The Behavior of Neutral Matter in Nonuniform Electric Fields* (Cambridge University Press, New York, 1978).
- ²⁴M. Riegelman, H. Liu, and H. H. Bau, *J. Fluids Eng.* **128**, 6 (2006).
- ²⁵K. D. Hermanson, S. O. Lumsdon, J. P. Williams, E. W. Kaler, and O. D. Velev, *Science* **294**, 1082 (2001).
- ²⁶R. H. M. Chan, C. K. M. Fung, and W. J. Li, *Nanotechnology* **15**, S672 (2004).
- ²⁷M. Washizu and O. Kurosawa, *IEEE Trans. Ind. Appl.* **26**, 1165 (1990).
- ²⁸L. Zheng, J. P. Brody, and P. J. Burke, *Biosens. Bioelectron.* **20**, 606 (2004).
- ²⁹I. Amlani, A. M. Rawlett, L. A. Nagahara, and R. K. Tsui, *Appl. Phys. Lett.* **80**, 2761 (2002).
- ³⁰R. Kretschmer and W. Fritzsche, *AIP Conf. Proc.* **725**, 85 (2004).