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# Nanometer Scale Electrode Separation (Nanogap) Using Electromigration at Room Temperature

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**Abstract**—Pairs of electrodes with nanometer separation (nanogap) are achieved through an electromigration-induced break-junction (EIBJ) technique at room temperature. Lithographically defined gold (Au) wires are formed by e-beam evaporation over oxide-coated silicon substrates silanized with (3-Mercaptopropyl)trimethoxysilane (MPTMS) and then subjected to electromigration at room temperature to create a nanometer scale gap between the two newly formed Au electrodes. The MPTMS is an efficient adhesive monolayer between SiO<sub>2</sub> and Au. Although the Au wires are initially 2 μm wide, gaps with length ~1 nm and width ~5 nm are observed after breaking and imaging through a field effect scanning electron microscope. This technique eliminates the presence of any residual metal interlink in the adhesion layer (chromium or titanium for Au deposition over SiO<sub>2</sub>) after breaking the gold wire, and it is much easier to implement than the commonly used low-temperature EIBJ technique which needs to be executed at 4.2 K. Metal–molecule–metal structures with symmetrical metal–molecule contacts at both ends of the molecule are fabricated by forming a self-assembled monolayer of dithiol molecules between the EIBJ-created Au electrodes with nanometer separation. Electrical conduction through single molecules of 1,4-Benzenedimethanethiol (XYL) is tested using the Au/XYL/Au structure with chemisorbed gold–sulfur coupling at both contacts.

**Index Terms**—Electromigration, molecular adhesion, molecular conduction, nanogap, symmetric contacts.

## I. INTRODUCTION

**E**FFICIENT device fabrication requires developing a suitable contact structure for characterization of few-molecule systems. Transport properties of single molecules are difficult to study by probing them between two symmetrical metal contacts due to the lack of a means to define the electrode structures with separations in molecular dimension. Efforts have been made to study the electrical conductivity through molecules by fabricating metal–molecule–metal (M–M–M) structures. Two general approaches involving preformed contacts have been employed. The first involves vertical device structures (VDS), where a self-assembled monolayer (SAM) of molecules is prepared on a metallic surface and a second contact is made using scanning tunneling microscopy [1], [2] (STM), gold

cluster-assisted STM [3], conductive probe atomic force microscopy (C-AFM) [4], flip chip [5], and crosswire techniques [6]. The second approach involves lateral device structures (LDS), where a pair of electrodes with nanometer separation is formed, and molecules are localized within the gap, typically through the use of end groups. Such electrodes are formed using mechanically controlled break junction [7], [8] (MCEB), electromigration-induced break junctions (EIBJ) [9]–[13] at low temperature (4.2 K), and electrodeposition [14]–[16]. In VDSs the top contact to the molecule is made by a physical process; whereas, the bottom contact is chemically coupled to the molecule. Here, the presence of any asymmetry, due to different contact structures at both ends of the molecule, cannot be avoided in electrical transport measurements. In LDSs that have been demonstrated to create a nanogap between two electrodes, the Au wires are either free-standing microbridges [8], [17] or formed over SiO<sub>2</sub> substrates with metal (titanium [12] or a chromium [13]) adhesion layer [11]. For molecular conduction studies with electromigration facilitated nanogaps, molecules were typically deposited on the metal surface before breaking and induced to migrate into the gap by subsequent heating to approximate liquid nitrogen temperatures. In these cases, after making the break junction, the residual interlink of metallic adhesion layer, in devices employing a metallic adhesion layer over SiO<sub>2</sub>, and the cantilever effect [18] of the suspended Au microelectrodes, in the case of free-standing structures, cannot be ruled out.

In this paper, we present a simple method of fabricating a pair of electrodes with nanometer sized separation (nanogap) between them, through an EIBJ technique performed at room temperature. The reported structure uses a molecular adhesion layer beneath the Au wire. This allows the break junction to be first characterized at room temperature, prior to deposition of molecular-scale conductors. Gaps in the range of ~1–2 nm have been realized, as inferred from imaging and electrical conductivity. The formation and characterization of the nanogaps at room temperature allows the deposition of desired organic molecules after initial characterization of the nanogaps, as evidenced by a study of conduction through single (or few) molecules of (1,4-Benzenedimethanethiol (XYL)) in an M–M–M structure (Au/XYL/Au) with nominally symmetric metal–molecule (Au–S) contacts.

## II. FABRICATION AND CHARACTERIZATION OF NANOMETER-SIZED ELECTRODE SEPARATION

The 5000-Å thermal oxide coated silicon(Si) substrates from Silicon Quest International, Inc., were silanized with a monolayer of (3-Mercaptopropyl)trimethoxysilane (MPTMS)

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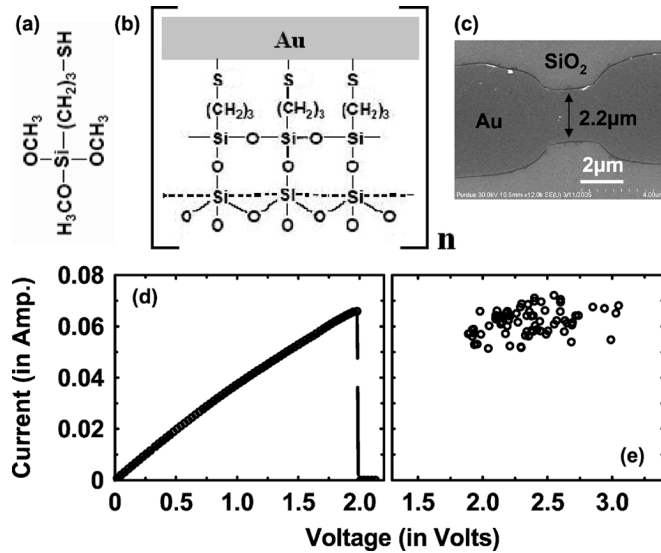


Fig. 1. (a) Molecular formula of MPTMS molecule. (b) Schematic diagram of MPTMS monolayer adhesion that forms Si–O–Si covalent bonds with SiO<sub>2</sub> surface silanols and Au–S bond with evaporated Au thin layers. (c) FESEM image (with 2- $\mu\text{m}$  scale bar) of lithographically defined Au microwire before electromigration with 2- $\mu\text{m}$  scale bar. (d) Current–voltage characteristics in Au wire during electromigration. (e) Threshold current and voltage values at break point for some EIBJ devices.

procured from Aldrich and Co., USA. In silanization, the SiO<sub>2</sub> substrate was processed as follows: 1) piranha clean; 2) oxygen plasma; 3) hydroxylation; and 4) four hours of exposure to the MPTMS gaseous molecules [molecular formula is shown in Fig. 1(a)] inside a vacuum desiccator containing an open bottle of MPTMS. The silanized SiO<sub>2</sub> samples were transferred immediately into the evaporation chamber for deposition of a 200-Å Au layer by e-beam evaporation at room temperature. The detailed experimental procedure and the chemical processes involved are explained elsewhere [21]. A drawing showing the Au film over an MPTMS capped SiO<sub>2</sub> substrate is shown in Fig. 1(b). The silane functional group of the MPTMS forms Si–O–Si covalent bonds with the SiO<sub>2</sub> surface silanols, leaving the -thiol group (-SH) of the molecule on the top, which on Au deposition forms an Au–S bond strong enough to hold the Au thin film tightly. A field effect scanning electron microscope (FESEM) image of the lithographically defined Au wire is shown in Fig. 1(c). For electrical connections, each end of the Au microwire is connected to a thick (4000 Å) Au pad layer deposited with e-beam evaporation. In all depositions, the pressure was maintained at  $2\text{--}3 \times 10^{-7}$  torr, and the rate of deposition was controlled at 1 Å/s, which was monitored using a quartz crystal thickness monitor. The positions of the nanogaps are controlled by placing a notch structure at the middle of the Au wire [shown in Fig. 1(c)].

A linearly increasing voltage  $V$  is ramped across the Au wires in steps of 20 mV at a rate of 0.25 s/step. At a threshold voltage  $V_{\text{th}}$  (corresponding current density  $J_{\text{th}}$ ), a  $\sim 10^7$  order decrease in current due to the formation of a gap is observed, where Ohmic current through the Au microwires [a FESEM image is shown in Fig. 1(c)] change to tunneling current through the nanogap created between two newly formed Au electrodes (FESEM image is shown in Fig. 2).  $I$ – $V$  characteristics through

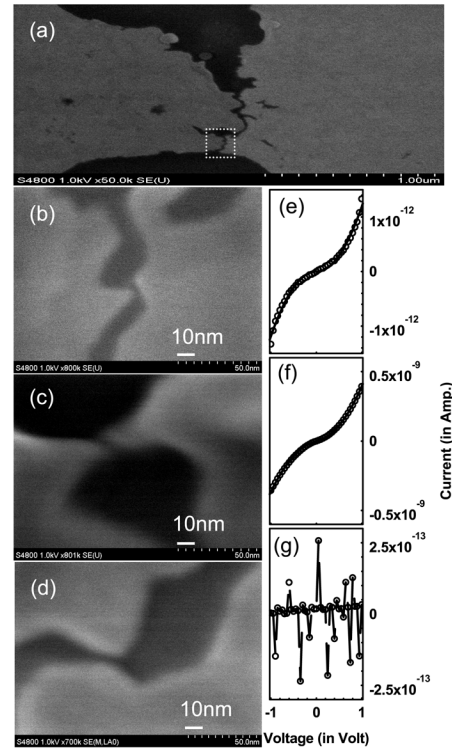


Fig. 2. (a)–(d) FESEM images of EIBJ devices that show  $\sim 1$ -nm gap created between two Au electrodes. (a) Although Au wires are initially 2- $\mu\text{m}$  wide, gap of length  $\sim 1$  nm and width  $\sim 5$  nm is observed (b) after break junction. Image (b) is zooming picture of selected portion in image (a). (e)–(g) Corresponds to empty gap  $I$ – $V$  characteristics of EIBJ devices, respectively. Here, solid lines are Fowler–Nordheim fit to experimental data (open circles). Significant conductivity is measured (e), (f) for Au/empty-gap/Au devices with gaps  $\sim 2$  nm, as confirmed through FESEM image (b) and (c). For devices with larger gap lengths ( $\sim 5$  nm), conductivity falls below noise level of instrument (g).

an Au wire during electromigration are shown in Fig. 1(d). The resistances of the Au wires, which were 35–40  $\Omega$  before electromigration, change to  $\sim M\Omega$ – $G\Omega$  after the break. The break occurs when the driving voltage exceeds 1.7 V and a current over 50 mA is flowing through the wire. At this point, a gap is formed due to the physical motion of atoms (Au) out of the high-current density areas, due to electromigration, a common phenomena of circuit breaking observed in large-scale integrated electronics [19], [20]. The movement is the net result of: 1) the force due to the direct action of the external field on the charge of the migrating ion and 2) the wind force due to the scattering of the conduction electrons by the metal atom under consideration. The threshold values of the current  $J_{\text{th}}$  and voltage  $V_{\text{th}}$  at the break point varies from wire to wire [plotted in Fig. 1(e)].

It has been observed that wires with longer notch width ( $\sim 4 \mu\text{m}$ ) give a wider gap compared to that of shorter ones ( $\sim 2 \mu\text{m}$ ). This can be explained as the threshold current density ( $J_{\text{th}} = I_{\text{th}}/A$ , where  $I_{\text{th}}$  is the current through the wire and  $A$  is the effective area of cross section); hence, the required Joule heating reaches a lower bias range for shorter width wires compared to the longer width wires. At the same time, a larger width wires experiences a large electric field due to a high bias requirement to reach the required Joule heating (or  $J_{\text{th}}$ ), which makes the Au atoms move further apart with high field. Also,  $I_{\text{th}}$  for thin wires is found to be less compared to thicker wires.

This indicates that electromigration is a combined effect of the local Joule heating experienced at the notch and the net force between migrating ions and following electrons.

It is informative to compare and contrast the current structure with prior EIBJ devices. In our experiments, with Au wire dimensions of  $\sim 2.0 \mu\text{m}$  width and  $\sim 20 \text{ nm}$  thickness, a threshold current densities of  $1.25 \times 10^{-8} \text{ A/cm}^2$  and  $1.1 \times 10^{-8} \text{ A/cm}^2$  are observed for Au/MPTMS and Au/Ti structures, respectively. Comparable  $J_{\text{th}}$  of  $2.5 \times 10^{-8} \text{ A/cm}^2$  at the break point has been reported for experiments on electromigration facilitated nanogaps using Au microwires of different dimensions ( $\sim 0.2 \mu\text{m}$  width and  $\sim 10\text{--}15 \text{ nm}$  thickness) over Cr [13] with a threshold current density. This implies that the breaking mechanism for Au microwires is similar to that in previous reports. It has been observed that at room temperature the Au microwires over MPTMS show breaks of sub-2-nm gap length, while the breaks in Au microwires having the same dimension over Ti generally yield wider gaps ( $\sim 20 \text{ nm}$ ), even if the same procedure for breaking is followed (FESEM image is not shown). In another study [21], we have observed that the Au film evaporated over MPTMS is much flatter than films evaporated over Ti ( $\sim 2 \text{ nm}$  rms roughness for Au/MPTMS vs.  $\sim 4.0 \text{ nm}$  for Au-Ti), both measured by atomic force microscopy over an area of  $1 \mu\text{m}^2$ . This indicates a significant difference in grain size/structure between the two films, which could explain the difference in minimum gap sizes observed under room temperature electromigration. The EIBJ devices formed using the same structure and procedures with Ti as the adhesion layer give conductances of  $\sim 10^{-7} \text{ S}$  (measured between the newly formed two electrodes after break), even when the gap length of  $\sim 15 \text{ nm}$  is confirmed through FESEM (not shown) with a decrease in conductance level with time. This anomalous  $I$ - $V$  curve with devices having metal adhesion layers is most likely due to the presence of residual metal interlinks through the adhesion layer after breaking the Au portions of the wire. This infers that even if we are following the same mechanism (electromigration) for breaking the Au wires, the present method of introducing a molecular adhesion monolayer for Au deposition over  $\text{SiO}_2$  produces isolated pairs of nanoscale separated Au electrodes with comparable high yield.

Following electromigration, the nanogaps are characterized through FESEM and current-voltage ( $I$ - $V$ ) characteristics. Although the Au wires are initially  $\sim 2 \mu\text{m}$  wide [Fig. 1(c)], imaging using FESEM indicates final gaps (the regions with shortest separation distance between newly created Au electrodes) of length  $\sim 1 \text{ nm}$  over  $\sim 5 \text{ nm}$  width. Hundreds of EIBJ devices were examined under FESEM, and it is generally found that 80% of the total width ( $2.5 \mu\text{m}$ ) shows a gap length more than  $50 \text{ nm}$ . The nanogaps with molecular dimension were composed of  $5\text{--}10\text{-nm}$  areas of the junctions separated by  $1\text{--}5 \text{ nm}$ , with the remainder of the junctions showing gaps with  $5\text{--}15\text{-nm}$  separation. Fig. 2(b) and (c) shows the FESEM image for two pairs of Au electrodes with  $\sim$  sub 2-nm separation observed through the EIBJ technique. Out of the field of view of these figures, there are wide gaps of more than  $20 \text{ nm}$ . The device yield is more than 20%, which is an improvement to the previously reported 10%–15% device yield [9], [10], [13] using break junctions formed by electromigration.

From FESEM imaging, it is difficult to estimate the exact gap length below  $2 \text{ nm}$ , but this can be inferred from the conductivity level of the newly formed nanogaps after the break. Fig. 3 represents the histogram of the spectrum of conductances from  $\sim \mu\text{S}$  to  $\sim \text{pS}$ , observed in some nanogaps formed through EIBJ. The smallest separation distance is observed to be located at one place (a protruding edge of  $5\text{--}10\text{-nm}$  length, as viewed through the FESEM imaging in Fig. 2) along the whole width ( $2.2 \mu\text{m}$ ) of the Au microwire. This observed conductivity is attributed to the smallest gap region and is not effected by the other portions of the gap. In general, the conductivity is not believed to be due to multiple gaps. We have observed a recordable  $I$ - $V$  characteristics in the nanoampere to picoampere range [Fig. 2(e) and (f)], only for devices with gap lengths  $\sim 2 \text{ nm}$  or less [Fig. 2(b) and (c)], but not through gaps showing more than  $2 \text{ nm}$  [Fig. 2(d)] (as viewed through FESEM), where the conductance level falls much below the measurable range and the  $I$ - $V$  data shows the noise level of the instrument [Fig. 2(g)]. The current after break is due to the Au-nanogap-Au structure, formed at the cracking portion of the Au microwire. The empty gap currents are in excellent agreement with the Fowler-Nordheim tunneling expression [22] and are shown in Fig. 2(e) and (f). The empty gap  $I$ - $V$  characteristic just after the break is reproducible in further repeated scans, which confirms the stability of the newly formed gap with time and to further external fields. The tunneling conductance of  $\sim \text{nS}$  is known to be due to approximately  $\sim 1 \text{ nm}$  electrode separation [8], [14], [15], [17] and decreases exponentially [23] as separation distance increases. The length of the gap is related to the tunneling conductance by the expression [15]  $G_{\text{tun}} = \alpha \exp(-kd)$ , where  $G_{\text{tun}}$  is the tunneling conductance,  $d$  is the gap length, and  $\alpha$  and  $k$  are constants which depend on the local structure. The tunneling conductance versus separation distance is reported [8] for an Au-vacuum-Au structure comparable to our current structure. This relationship can be described by values of  $\alpha = 6.67 \times 10^{-8}$ , and  $k = 1.25 \times 10^{10} \text{ m}^{-1}$ . From the tunneling conductance of the empty gaps, the separation distance can be easily estimated. Since there is one order decrease in tunneling current in every  $2 \text{ \AA}$ , the observed conductivity is dominated by the smallest possible separation localized at one point over the whole width.

In order to verify the suitability of the nano gaps for small molecule conductivity studies, the conductivity of XYL has been studied. The break junction chip is plasma cleaned to take off any residual MPTMS or any other organic contaminations from the gap region and rinsed with Ethyl alcohol to take off the oxides from Au layer, then immersed in a 1-mM solution of XYL in ethanol. MPTMS is not soluble in ethanol and does not distort the Au thin film [21]; therefore, it is not expected to migrate into the gap during deposition of XYL. In this case, the metal-molecule contacts are formed through chemisorbed Au-S coupling, which forms nominally symmetric contacts at both ends of the molecule. Au-S is known to be a strong bond and is frequently used in molecular electronics [7], [10]. Fig. 4 represents the symmetric  $I$ - $V$  characteristics of Au/XYL/Au device at room temperature. An increase in current of  $\sim 10^5$  is observed in these devices, compared to the currents through the empty gap devices. This increase in conductivity is observed only in devices with gaps of  $\sim 1\text{--}2 \text{ nm}$ , but not in devices

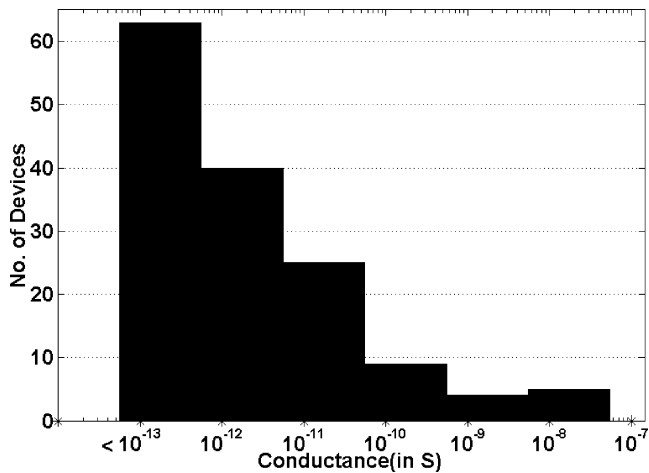


Fig. 3. Histogram of conductances observed in 150 nanogap EIBJ (Au/empty-gap/Au) devices.

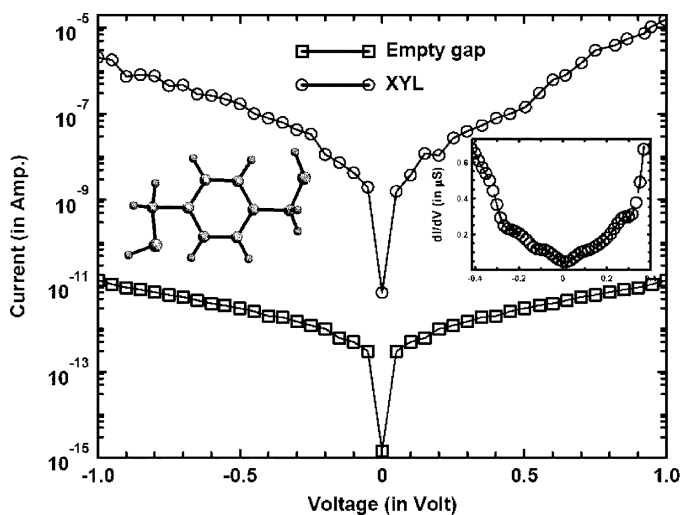


Fig. 4. Room temperature  $I$ - $V$  characteristics of Au/XYL/Au (squares) and Au/empty-gap/Au (circles) structures. Insets show molecular formula of XYL and low bias  $dI/dV$  of XYL molecule.

with gap larger than 2 nm, as determined from empty gap conductivity levels and subsequent FESEM imaging. Since XYL is approximately 1 nm long, binding the two ends of the molecule with the two nanoelectrodes is possible only for the gaps of sizes comparable to the molecular dimension ( $\sim 1$ –2 nm) but not in gaps of several nanometers in length. The low conductivity before XYL deposition indicates that the MPTMS conductivity is negligible compared to observed XYL conductivity. Also, a featureless  $I$ - $V$  characteristics is observed for EIBJ devices measured after immersing in pure solvent (Ethanol). Molecular fine structures are reflected in the  $dI/dV$  versus voltage (inset in Fig. 4) plot. This molecular signature [25] confirms the presence of molecules between the nanogap, hence the successful bridging of the dithiol molecules of length comparable to the nanogap between the pairs of Au nanoelectrodes. In another experiment [24], we have immobilized dithiol derivatized 15 base-pair double stranded DNAs between the nanogaps and a significant increase in conductivity is observed. This nanogap takes advantage of not having any

interference in  $I$ - $V$  results due to presence of residual metal (Cr or Ti) interlink at the breaking site or due to the cantilever effect in case of free-hanging structures.

### III. CONCLUSION

In conclusion, we present a simple method to achieve a pair of electrodes with nanometer separation through EIBJ performed at room temperature. The structure involves lithographically defined Au wires formed over  $\text{SiO}_2$  substrates silanised with MPTMS. The MPTMS acts as an efficient molecular adhesion monolayer between Au and  $\text{SiO}_2$  and results in a film with subnanometer root mean square roughness. This technique of fabricating M-M-M structures with symmetric metal-molecule contacts eliminates the presence of any residual metal interlink in the adhesion layer (chromium or titanium) after breaking the gold wire, and it is much easier to implement than the commonly used low-temperature breaking technique, which needs to be executed at 4.2 K. Electrical conductivity through single molecules of XYL is measured for a Au/XYL/Au structure with chemisorbed Au-S coupling at both contacts. Fabricating nanometer separated metal electrodes will be extremely useful for nanotechnology to build efficient nano/bio inspired devices by successfully probing the nanoscale materials (molecules, DNA, and nanometer sized clusters) for electrical studies, which is still a challenging job for today's science and technology.

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