Gold coated Carbon Nanotube Surfaces as Low Force Electrical Contacts for MEMS devices: Part 1.

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Abstract— **An experimental investigation of a gold coated vertically aligned carbon nanotube surfaces is undertaken to determine the limits of the electrical contact performance over a large number of switching cycles under low force conditions and with current loading (1mA-50mA at 4V). The multi-walled CNT's (MWCNT's) are synthesized on a silicon planar and sputter coated with a gold film. The planar surfaces are mounted on the tip of a PZT actuator and mated with a coated Au hemispherical probe. The electrical load is selected to reflect typical MEMs relay loads with a 4V supply, 1 and 10mA current load with an applied force of 1mN. The surfaces tested maintain a stable contact resistance over 10^6 switching cycles. To determine the limits, the contact force is increased to 3mN under dry circuit conditions and the current increased at the 1mN load to 20mA-50mA. The surfaces are compared with a reference Au-Au contact under the same experimental conditions. For the surfaces investigated the current loading limit was determined to be 20mA where the contacts failed after $50x10^6$ cycles.**

Keywords-component; Carbon nanotube surfaces, MEMS switching surfaces, Carbon nanatube composites.

I. INTRODUCTION

Reliable electrical contact surfaces for MEMS switching devices, has been a limitation to the full development of the technology. Typically, metallic surfaces such as Au, degrade when switching even very low electrical loads, as a result of localized thermal phenomena such as the molten metal bridge phenomena, [1]. The switching of electrical loads is often referred to as "hot switching" and is the term used in this paper. The "dry circuit" condition, also used here, is associated with the mechanical switching of the surfaces, where the applied voltage and current have no physical influence on the surface, [2].

In this paper consideration is given to the development of a compliant substructure to the metallic surfaces, using carbon nanotubes. In this study the main focus is on the experimental results, while Part 2, [3], will build upon the results with observations on the material and electrical properties of the surfaces. To replicate MEMS switching conditions, the static contact force used is 1mN, and the "hot switching" conditions, 1mA-50mA at 4V. There are a number of prospective materials commonly used for this application including gold, palladium and platinum [4] but they are relatively soft and wear easily. The low contact resistance requirement, for MEMS relays requires a contact material with low resistivity, high wear resistance and high resistance to oxidation [5]. It is desirable to have a stable contact resistance of less than 1~2Ω during millions of "hot switched" cycles [4,6-9]. Other materials which are of interest for MEMS relay contacts include silicon carbide and diamond films. Both have high elastic moduli but also a high electrical resistivity. There have been attempts to lower the resistivity. When doping SiC film with NH_3 the resistivity drops to 1×10^{-4} Ωm [10] and doping DLC with ruthenium the resistivity drops to 1×10^{-5} Ωm [11], however, both materials have a high resistivity compared to gold and gold alloys (for example Au-6.3% Pt has a resistivity of 7.17×10^{-8} Ωm), [4].

A carbon nanotube (CNT) coated surface has potential as a material for MEMS relay applications specifically as a contact material since the mechanical and electrical properties are potentially comparable to diamond and gold respectively [12-16]. In [17] the contact resistance against applied force relationship was investigated between two multi-walled CNT's (MWCNT's) surfaces. The contact resistance was measured in ambient air and in a vacuum. It was concluded that the contact resistance was lower in ambient air (~160 Ω) than in vacuum (>4kΩ). In [18] a micro-tribometer was used to determine the contact resistance between electrodes where the upper and lower electrodes were coated with Au and CNT respectively. The CNT surface in this case was a tangled single walled carbon nanotubes structure, and the applied force ~150 mN. The authors concluded that a tangled single walled carbon nanotube (SWCNT) film in contact with a Au coated surface has a lower contact resistance than two contacting SWCNT films.

In this paper a MWCNT "forest" is coated with gold, in order to form an Au composite and provide a high conductivity surface layer with a compliant under layer. The results presented are a continuation of previous experimental studies, [19-21]. In [19], a modified nano-indentation apparatus was used to determine the contact resistance as a function of contact force (1mN) with up to 10 "dry circuit" switching operations. In [20] a PZT actuator was used to support planar coated surfaces for "dry circuit" switching up to 1000 cycles. The results showed that the mechanical performance and contact resistance of Au-Au/MWCNT contact pairs were comparable to Au-Au contact pairs. In [21] a PZT apparatus was used in an initial study of the "hot switching" condition (1mA-10mA at 4V). The results showed that the Au-Au/MWCNT contact pair maintained a stable contact resistance over $2x10^6$ switching cycles.

II. MATERIAL PREPARATION

In this study two contact pairs are investigated; Au to Au and Au to a Au/multi walled carbon nanotube (MWCNTs) composite. The geometry selected is a 2mm diameter hemisphere probe contacting a flat surface, [19-21]. In all cases the hemisphere probe consists of a stainless steel base, sputter coated with Au, ~500 nm thick, with surface roughness $R_a \approx 400$nm. In the Au to Au case, the flat surface is a silicon (Si) substrate, sputter coated with Au ~500 nm, with a surface roughness $R_a \approx 30$nm.

For the Au to Au/MWCNT case, a "forest" of MWCNTs is grown on a Si wafer using thermal CVD. The catalyst used is sputter deposited Fe and the gaseous carbon source is ethylene. The growth temperature and time is 875^oC and 3 minutes respectively to produce a dense forest of vertically aligned MWCNT of an average length of ~50µm as shown in Fig 1. The packing density of the structure has yet to be determined however Fig.1 shows the uniformity of the structure and the apparent roughness of the top surface of the structure. The surface is sputtered on the upper surface of the MWCNT forest to produce Au/MWCNT composite coatings as shown in Fig 2, with surface roughness $R_a \approx 1.5$µm. Fig.2 shows that the Au film has a number of sub-micron voids. The thickness of the Au has yet to be determined however pervious SEMs studies suggest that the Au diffuses into the CNT structure.

III. EXPERIMENTAL METHOD

The apparatus used is shown in Fig 3, and described in detail in, [20]. The PZT actuator is used to support the planar coated surfaces, and to make contact with the lower Au-coated hemispherical ball. The system has been designed to model a MEMS relay electrical contact. The contact force is controlled by controlling the contact gap and is set at 1mN and 3mN for the results presented. The apparatus is mounted in a sealed enclosure at room temperature. The performance of the Au-Au/MWCNT surfaces is compared to a reference Au-Au contact pair under the same experimental conditions in order to assess the mechanical and electrical stability and reliability. SEM and 3D surface profiling (TaiCaan Technologies XYRIS 4000CL) are used to measure changes to the surfaces for three experimental conditions.

A. Experiment 1: Determining the Mechanical Switching Limits of the Au to Au/MWCNT surface.

In the previous mechanical studies the contact force was limited to 1mN, [19]; in this study the static force is increased to 3mN, using the PZT system. It is expected that the impact forces will be much higher than the static force. The aim is to determine the limits of the mechanical or "dry switching" characteristics.

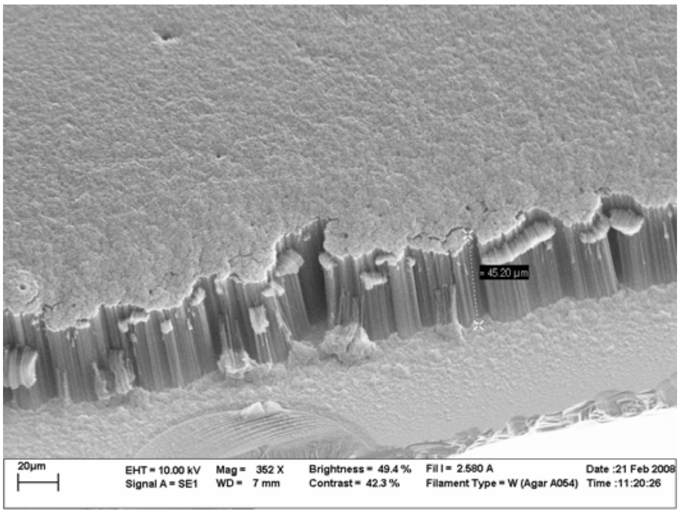

Figure 1. MWCNT with average length ~50µm.

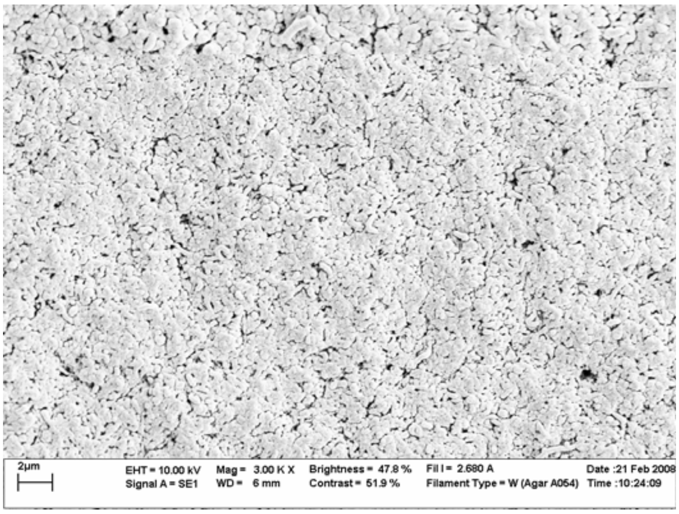

Figure 2. Au/MWCNT composite contact surface.

B. Experiment 2: Hot-switched condition with 1mA and 10mA at 4V

In this experiment the PZT actuator is initially controlled at low frequency (0.2Hz) to allow a quasi-static study of the contact surfaces up to 1000 cycles. At 0.2Hz the contact force is applied for ~3 seconds so that a representative average contact resistance value can be determined. To replicate the conditions of a MEMS relay the "hot-switching" loads used are 1mA and 10 mA at 4V, [22-24]. Fig 4 shows the circuit arrangement for the hot-switching experiment. After every 10th cycles the current and supply load is switched off and the contact resistance, (R_c) measured using the 4 wire-measurement method. To study the contact surfaces over a million cycles, the PZT actuator is actuated at a higher frequency (10 Hz). To determine the contact resistance the cycling frequency is reduced to the quasi-static frequency (0.2Hz), at 3000, 6000, 9000, 10000, 300000, 865000, 1 and 2 million cycles.

C. *Experiment 3: Hot-switched condition, 20mA-50mA, 4V.*

The current load is increased to 20mA-50mA to observe and benchmark the performance of these surfaces. For currents of 30mA-50mA the PZT actuator was actuated at 0.2Hz. After every 10 cycles the current and supply voltage was switched off and the contact resistance measured.

For the current loading of 20mA the sample was actuated at 0.2Hz, 10Hz and 20Hz. At 0.2Hz every 10 cycles the R_c was measured. After 1000 cycles the frequency was changed to 10Hz and the contact resistance measured after 10000, 100000 and 1 million cycles. After 1 million cycles the frequency was changed to 20Hz and the resistance monitored every 2 million cycles.

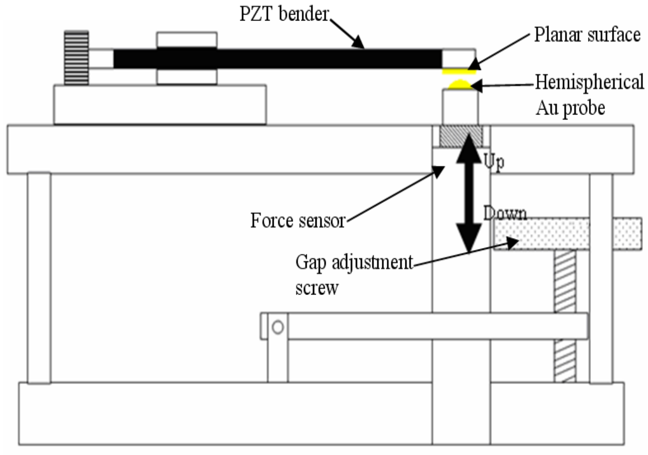

Figure 3. Schematic side view of the PZT test rig

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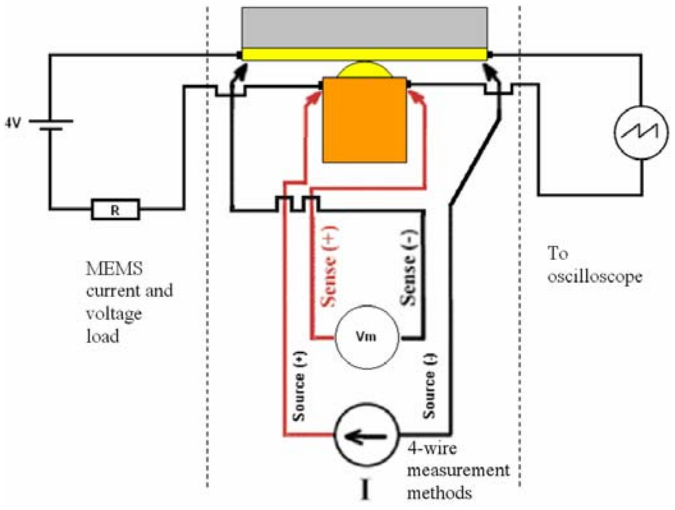


Figure 4. Schematic of contact zone with its electrode, current and supply load, 4 wire measurement and the monitoring.

IV. RESULTS

A. *Experiment 1: Determining the Mechanical Switching Limits of the Au to Au/MWCNT surface*

Fig.5 shows the switching surfaces after 20x10^6 mechanical "dry circuit" switching cycles. There is limited damage to the ball surfaces in (a), while in (b) the Au/MWCNT surface shows evidence of cracking surrounding the impact area, further emphasized in the 3D data shown in (c). The depression in the surface is 60µm, which is a significant departure from results with 1mN where the deformation was typically 1-2 µm, [19]. It is expected that the increase in the applied force has significantly increased the transient impact forces. There was no increase in the contact resistance during this study.

The depth of the crater (60µm) emphasizes the difficulty in determining the height of the CNT structure prior to testing. It was suggested in Fig.1 that the height of the CNT structure is approximately 50µm. It is expected that for the sample tested the 3mN force has deformed the surface to the base Si surface level. Fig.5 also shows that the Au plating on the MWCNT structure is cracked. This is apparent in both SEM and 3D data images. It is expected that the cracking is associated with the fatigue performance of the Au plated structure.

B. *Experiment 2. Hot-switched condition with 1mA and 10mA at 4V*

Fig 6 shows the contact resistance of the Au-Au reference surface over 1000 cycles at a maximum (quasi static) applied load of 1mN, with a 1mA current. The contact resistance is initially ~0.58Ω and increases rapidly to 4-10Ω at ~220 cycles. The increase in the contact resistance corresponds to the adhesion of the Au-Au pair and the resultant delamination of the Au coating on the ball, as shown in Fig 7. Fig.7 (a) shows the damaged Au hemispherical probe surface and the corresponding adhesion of the delaminated film to the Au planar surface. Fig 7 (c) shows 3D profile of the damaged Au hemispherical probe, to determine the thickness of the Au film. A is the top of the Au surface while B is the top of the underlying ball surface. This result confirms the thickness of the Au film on the ball to be ~500nm. The peaks on the surface are cause by the tearing of the surface during the delamination process. The adhesion between the Au surfaces is a well know phenomena, and is in this case a combination of the softening and melting the Au layer, associated with the bridging phenomena resulting from the localized current density at the asperity contact peaks [1] and Au-Au adhesion, [25]. Fig 6 also shows the contact resistance of the Au-Au/MWCNT contact pair at two different current loads (1mA and 10mA, 4V). The contact resistance is higher than for the Au-Au pair at the start of the cycling, (~0.68Ω) for both current load conditions. This observation is consistent with previous force, contact resistance studies, [19].

Fig. 8 shows the contact resistance during extended cycling over 2 million cycles. The contact resistance is similar and stable for both current load conditions, 1mA and 10mA. Fig.9 shows the surfaces after the testing with a small amount of Au adhesion on the Au hemispherical probe in Fig. 9(a), at 1mA and 10mA in (b). This Au adhesion appears to be in one direction, from the Au/MWCNT planar surfaces to the Au ball. No observable damage on Au/MWCNT planar composite surfaces has been detected.

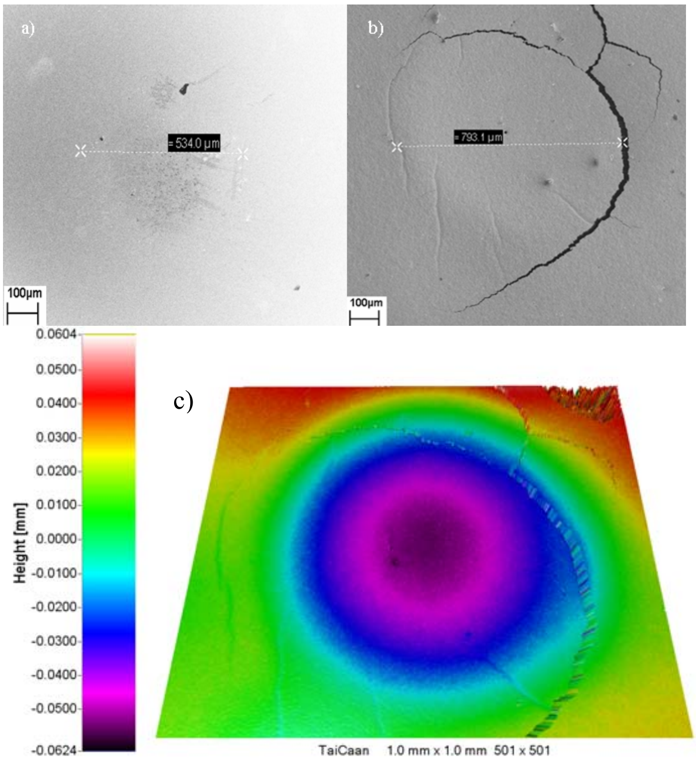


Figure 5. SEM images of a) Au hemispherical probe and b) Au/MWCNT surface after 20 million cycles at 3mN, c) 501x501(1mm x 1mm), 3D scan of Au/MWCNT surface.

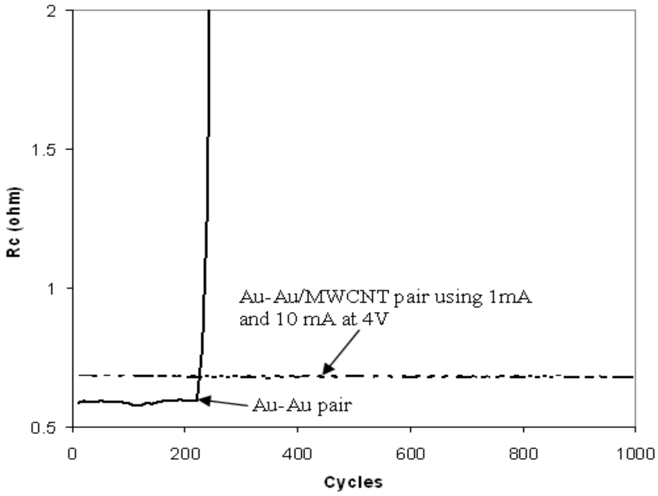


Figure 6: Contact resistance for Au-Au and Au-Au/MWCNT contact pair.

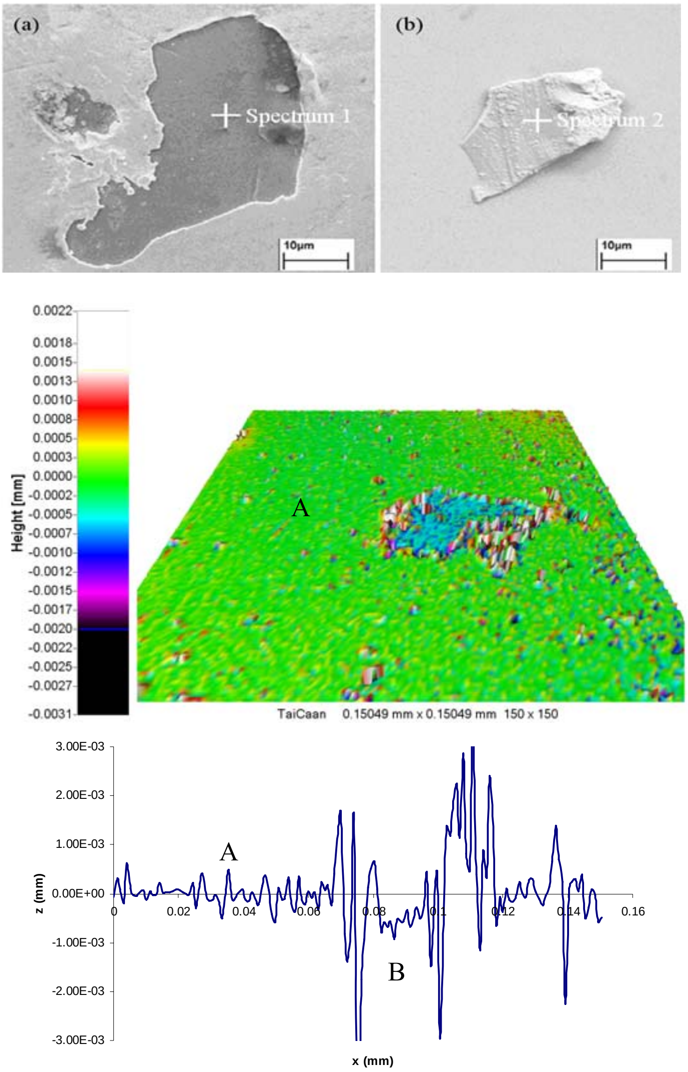


Figure 7: (a) SEM image of Au hemispherical probe degradation (b) SEM image of Au planar with Au debris (c) Scanned image of damaged Au ball for Au-Au pair (1mA,4V) with the sphere removed, contact pair 150x150 (0.15mmx0.15mm) using TaiCaan (Xyris 4000CL).

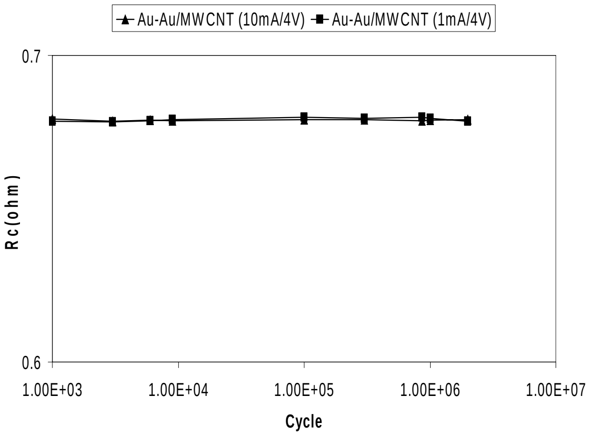


Figure 8: Contact resistance for 1mA and 10mA over 2 million cycles for the Au-Au/MWCNT contact.

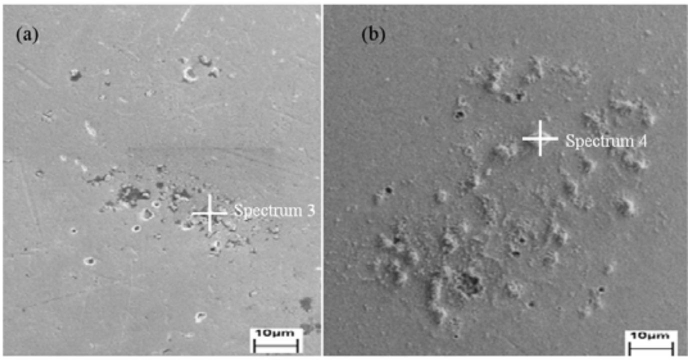
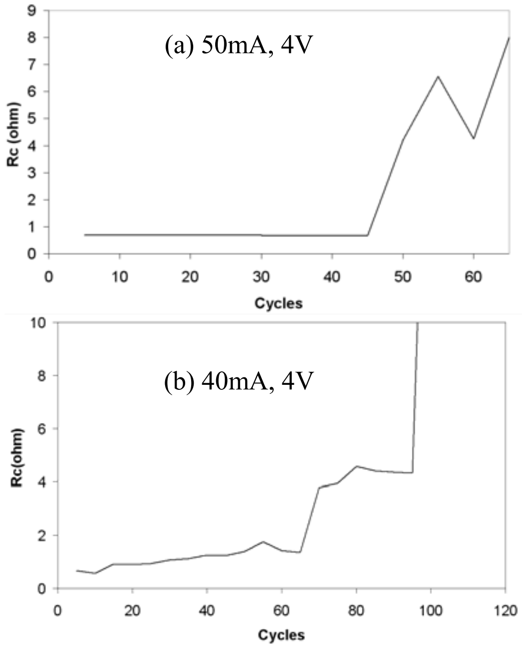


Figure 9: SEM image of (a) Au hemispherical probe for Au-Au/MWCNT contact pair after 2 million cycles at current load 1mA, 4V and (b) Au hemispherical probe at current load 10mA, 4V.

C. *Experiment 3: Testing the Limits; 20mA-50mA at 4V*

Fig 10 (a), (b) and (c) shows the contact resistance against number of cycles for the Au-Au/MWCNT contact pair using a current load between 30mA-50mA, 4V. In all cases it was observed that degradation has occurred between 45 and 150 cycles. As with the 1mA test on Au-Au surfaces in Fig.7 the degradation has occurred on the surface of the Au coated ball shown in Fig 11, for the 50 mA test. The delamination corresponds to an increase in the contact resistance.

Fig 12 shows the contact resistance against number of cycles for Au-Au/MWCNT composite with a current load of 20mA. The contact resistance remains stable until ~50 million cycles. For this condition the degradation is no longer by the delamination process, but by the material transfer from the anode surface to the cathode, as shown in Fig 13 (a) and (b). Au from the top surface of the Au/MWCNT composite surface (anode) has transferred to the Au hemispherical probe (cathode).



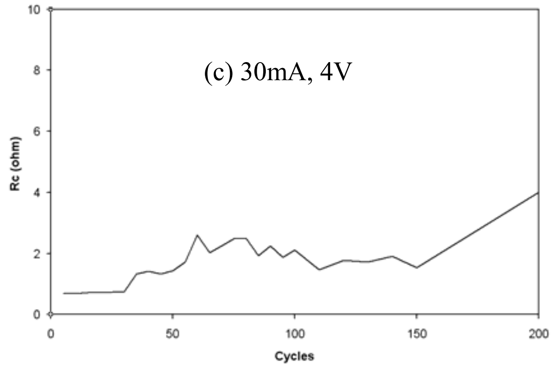


Figure 10: Graph of contact resistance against number of cycles for Au-Au/MWCNT contact pair with current load of (a) 50mA, and (b) 40mA and (c) 30mA 4V (all 1mN).

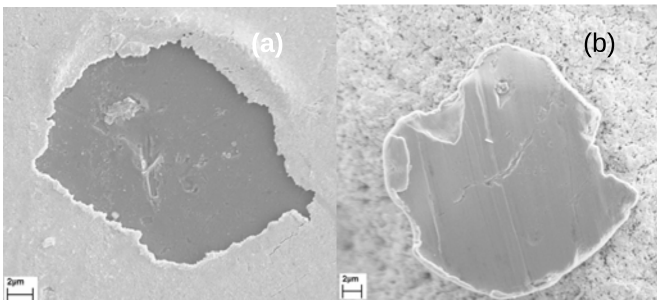


Figure 11: Damaged surface of Au hemispherical probe after using current load (a), and the Au adhered on the Au/MWCNT composite surface (b) for 50mA, 4V

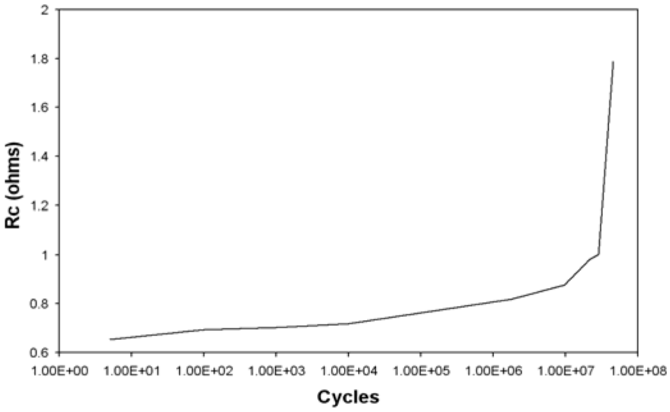


Figure 12: Graph of contact resistance against number of cycles for Au-Au/MWCNT contact pair with current load of 20mA, 4V (1mN).

V. DISUSSION

The results show there to be two degradation mechanism in the "hot switching" of the Au-Au/MWCNT structure. In the first, at higher current levels >30mA the process is consistent with the adhesion of the Au coating on the ball to the top of the Au/MWCNT structure, as shown in Fig.11. This is a similar to that obtained on the reference Au-Au sample at a much lower current level of 1mA. Au-Au systems are known to exhibit cold welding even at very low contact forces, however in this case the phenomena observed is linked to the current density through the asperities of the surface and reflected in the current loading. The results presented here and in [19-21] show that the Au-MWCNT structure deforms to the applied force, increasing the apparent area between the contact surfaces, (see Fig.5).

The second degradation mechanism is consistent with bridge transfer between surfaces. This particular mechanism was widely studied in the 1950 and 1960, for communication relays, which were super-seeded by electronic switching devices. The surface shown in Fig 13(a) on the Au ball, corresponds to the cathode surface, and shows a build up of material from the anode in Fig 13(b), for 20mA. The increase in the resistance is consistent with the removal of the Au conduction path on the top of the MWCNT surface. Fig. 9 shows a much lower level of transfer over the low number of cycles, and lower current levels, 1mA and 10mA. It is interesting to note that although the conduction path has become depleted in Fig. 13, the contact resistance level is still relatively low (<2Ω), showing the ability of the surface to also conduct through the CNT structure as observed in [19].

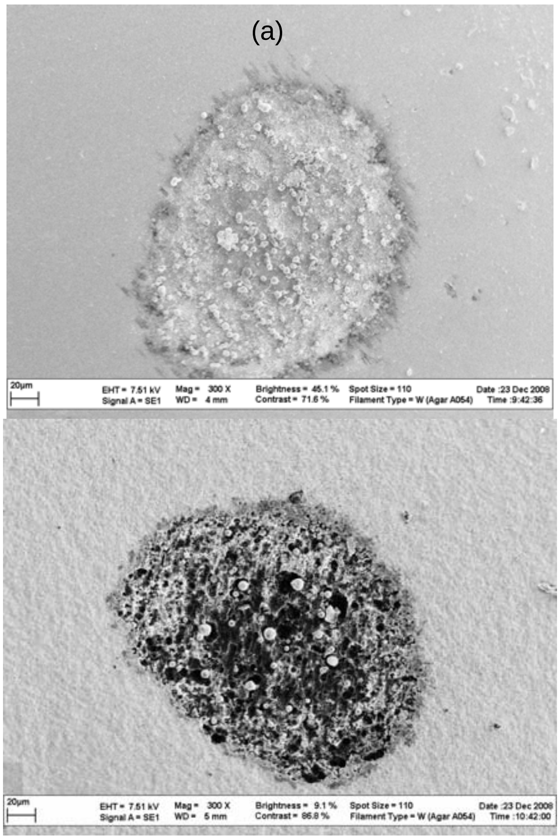


Figure 13: (a) Au adhering on the Au hemispherical probe (anode) and (b) damaged image on the Au/MWCNT composite surface (cathode).

VI. CONCLUSIONS

It has been shown that a Au surface coupled with a Au/MWCNT (gold coated multi-walled carbon nanotube structure), has the ability to sustain millions of switching cycles with a electrical load typical of current MEMS switch devices, typically 4V, 1 to10mA, with a low contact force of 1mN. This is an important observation, and lays the foundation for the application of carbon nanotube structures for electrical contact applications. The surfaces developed here use an approximate 500nm covering of Au on a Stainless steel ball surface and a MWCNT surface. The contact resistance across the interface is used as the parameter for determining the failure modes of these surfaces.

For the mechanical cycling of the surfaces, two contact force levels have been used, 1mN, and 3mN. In both cases no failure mechanisms have yet been reported, however the contact force has been shown to have a significant influence of the cyclic deformation of the Au/MWCNT surface. Results show that the surface deforms to the base level of the CNT structure.

For the "hot switching" or electrically loaded surfaces, the contact force was maintained at 1mN with a 4V supply, with a range of applied currents. An Au-Au reference surface was shown to fail after 250 switching cycles, due to adhesion of the Au surfaces. The Au-Au/MWCNT surfaces were subjected to current loads from 1mA to 50mA. Failure was shown to occur in the 30-50mArange after a low number of cycles with a mechanism similar to that exhibited in the Au-Au surface, with adhesion and delamination of the Au plating on the balled surface. For the Au-Au/MWCNT surfaces with current loads from 1mA to 20mA, an apparent failure was observed in the 20mA case after 50 Million cycles, as a result of a material transfer process associated with metallic bridge transfer. No failures were observed at the lower current levels; however these samples were only tested to 2 Million cycles.

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