Improving the contact resistance at low force using gold coated carbon nanotube surfaces.

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Summary
Investigations to determine the electrical contact performance under repeated load cycles at low force conditions for carbon-nanotube (CNT) coated surfaces were performed. The surfaces under investigation consisted of multi-walled CNT synthesized on a silicon substrate and coated with a gold film. These planar surfaces were mounted on the tip of a PZT cantilever and contacted with a hemispherical Au plated probe. The dynamic applied force used was 1mN. The contact resistance (R_c) of these surfaces was investigated with the applied force and with repeated loading cycles performed for stability testing. The surfaces were compared with a reference Au-Au contact under the same experimental conditions. This initial study shows the potential for the application of gold coated CNT surfaces as an interface in low force electrical contact applications.

Key words:
contact force, contact resistance, carbon nanotubes, and Au/multi walled carbon nanotubes.

1. Introduction

This paper presents a study of electrical contact between surfaces under low dynamic force conditions, typically 1mN. Such conditions are relevant to a number of micro-contact applications, for example MEMS relay devices. There are a number of potential materials commonly used for this application including gold, palladium and platinum [1]. The weakness of such materials is that they are relatively soft and wear easily. Other materials which are of interest on MEMS relay's micro-contact include silicon carbide and diamond films. Both have high moduli but low electrical conductivity. This latter makes them unsuitable for electrical contact applications. There have been attempts to increase the conductivity. When doping SiC film with NH3 the resistivity drops to 1 x 10^{-4} Ωm [2] and doping DLC with ruthenium the resistivity drops to 1 x 10^{-5} Ωm [3], however, both materials still have a high resistivity compared to gold and even gold alloys (for example Au-6.3% Pt has a resistivity of 7.17 x 10^{-8} Ωm) [1].

A carbon nanotube (CNT) coated surface has potential as a material for MEMS relay applications specifically as a contact material because of its excellent mechanical and electrical properties. An experiment has been performed [4] to measure the contact resistance, R_c between CNT coated electrodes in ambient air and in a vacuum. The author concluded that the contact resistance, R_c was found to be much lower in ambient air (~160 Ω) than in vacuum (>4kΩ). In a more recent experiment [5], Au contacts with a substrate coated with tangled single walled carbon nanotubes were investigated. The authors concluded that a tangled Single Walled Carbon Nanotube (SWCNT) film against an Au coated surface works better than two contacting tangled films.

The following mechanical properties have been determined; CNTs tensile strength of up to 63 GPa has been measured [6]. Experiments using an atomic force microscope were performed to measure the elastic modulus and bending strength of individual, structurally isolated, multi-wall carbon nanotubes and indicated values of 1.26 TPa and 14.2 GPa [7] respectively. Experiments were also conducted on CNTs using nano-indentation apparatus and values were obtained for the bending modulus; 1.24 TPa, axial modulus; 1.23 TPa and wall modulus; 5.61 TPa [8]. Another report shows that CNT's have an elastic modulus greater than 1 TPa [9] compared to diamond, which has a modulus of 1.2 TPa.

In terms of its electrical properties, it is calculated that a 4-10 μm long SWCNT with a diameter of 1.2nm has a resistivity of 0.88 x 10^{-8} Ωm and is thought to exhibit ballistic electrical conduction. The calculation is performed using the theory of ballistic conductors and it is assumed that the CNT is defect-free. In addition, if a CNT were to be filled with metal, to form a composite its resistivity would fall to 0.35 x 10^{-8} Ωm [10]. The mechanical and electrical properties are therefore potentially comparable to diamond and gold respectively, however, as yet no experiments have been reported on CNT metal composites for micro-contact applications. In the present work a novel approach is used in which a CNT "forest" is over coated with gold, in order to provide a high conductivity surface layer with a compliant under layer.

This paper presents a continuation of previous experimental work. In this previous work a modified nano-indentation apparatus [11] was used to determine the contact resistance, R_c, as a function of contact force and load cycling up to ten load cycles. These initial results showed that the performance and contact resistance of Au-Au/MWCNT contact pairs is comparable to Au-Au contact pairs and during ten load cycles of Au-Au/MWNT contact pair shows stable and constant contact resistance.

2. Material Preparation

In the present study two contact pairs have been investigated; Au to Au and Au to Au/multi walled carbon nano-tubes (MWCNTs) composite. The geometry selected is a 2mm diameter hemisphere contacting a flat surface. In all cases the hemisphere consists of a stainless steel base, sputter coated with Au, ~500 nm thick, with surface roughness Ra≈400nm. In the Au to Au case (Sample 1), the flat surface is a silicon (Si) substrate (~2mm by 7mm), sputter coated with Au ~500 nm, with a surface roughness Ra≈30nm.

For the Au to Au/MWCNT case (Sample 2), a "forest" of MWCNTs is grown on the 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°C and 3 minutes respectively to produce a dense forest of vertically aligned MWCNT of an average length of ~50µm as shown in Fig 1. Au is then sputtered on the upper surface of the MWCNT forest to produce Au/MWCNT composite coatings as shown in Fig 2. It is shown in Fig 3, that the Au penetrates the MWCNT surface to a depth of ~4 µm.

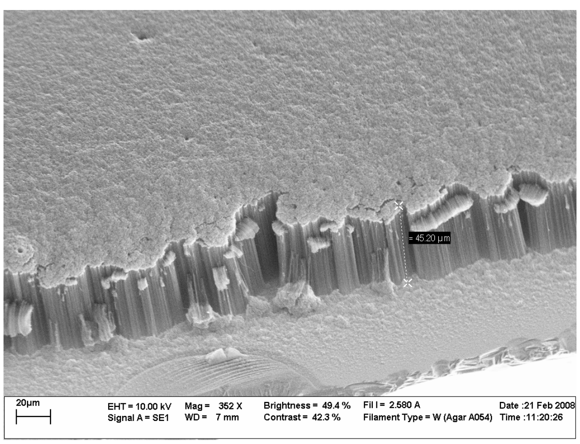

Figure 1: Dense forest of MWCNT with average length ~50µm.

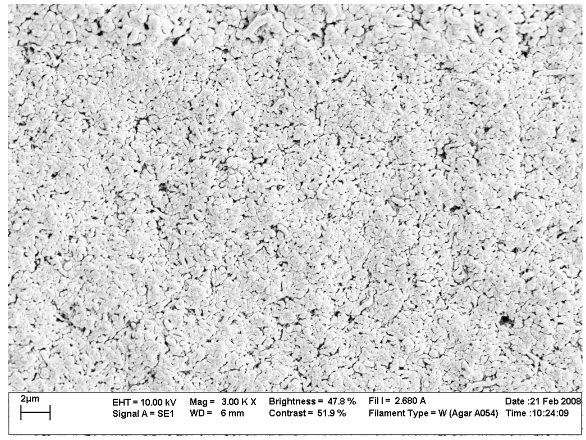

Figure 2: Sample 2, Au/MWCNT composite contact surface.

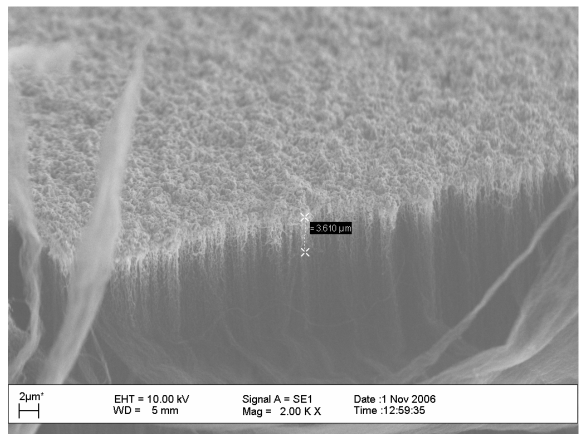

Figure 3: Au penetration on MWCNT by sputtering.

3. Experimental Method

In order to determine the performance of the surfaces under repeated switching actions an apparatus has been designed, in which a PZT bender/cantilever is used to support the planar coated surfaces as shown in Fig 4. This surface makes electrical contact with the hemispherical Au-coated probe to mimic the actuation of a MEMS relay micro-contact. The apparatus has been designed to allow control of the gap and to allow the performance of the contact materials to be investigated ultimately over large numbers of switching cycles ($>10^6$).

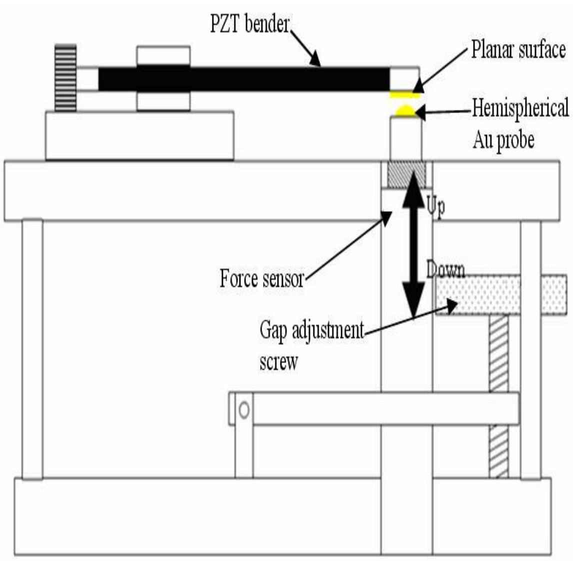

Figure 4: Schematic side view of the test rig.

A signal generator with voltage amplification is used to actuate the PZT bender as shown in Fig 5. The PZT bender's layers consist of Nickel (1st layer), PZT material (Lead Zirconate Titanate) (2nd layer), Nickel (3rd layer) and Kovar (Nickel-Cobalt ferrous alloy, final layer). The resonance frequency of the PZT bender is ~900Hz (1st harmonic). In this experiment the PZT bender is actuated at low frequency 0.2Hz to allow a quasi-static study of the contact surfaces. The dynamic force is measured using a piezoelectric force sensor [12-15] situated as shown in Figs 4 and 5. The force sensor is amplified using a charge amplifier and the dynamic force monitored.

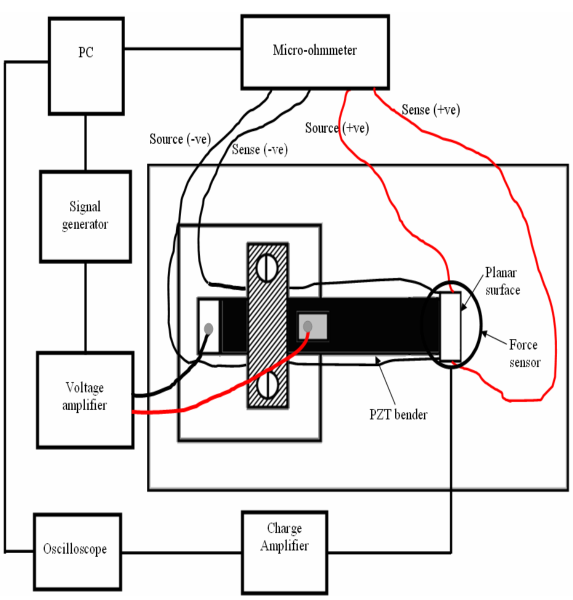

Figure 5: Schematic top view of the test system

There are several methods to control the applied force as follows; 1) By controlling the gap [16]. This can be achieved by turning the adjustment screw as shown in Fig 4 and the contact gap is monitored using a digital DTI with a resolution of 1µm, 2) By controlling the length of the bender [14], and 3) By controlling the amplitude of the supply voltage to the bender. In the experiment presented here all other parameters are held constant, with the contact gap used to set the contact force at 1mN.

The contact resistance, R_c is measured using the 4 wire-measurement methods as shown in Fig 5 and 6. The DC current source across the planar coated surfaces and micro-contact is set at 1mA using a Keithley 580 micro-ohmmeter. The number of cycles and contact resistance can be controlled and extracted by using the control and data acquisition program (Lab View).

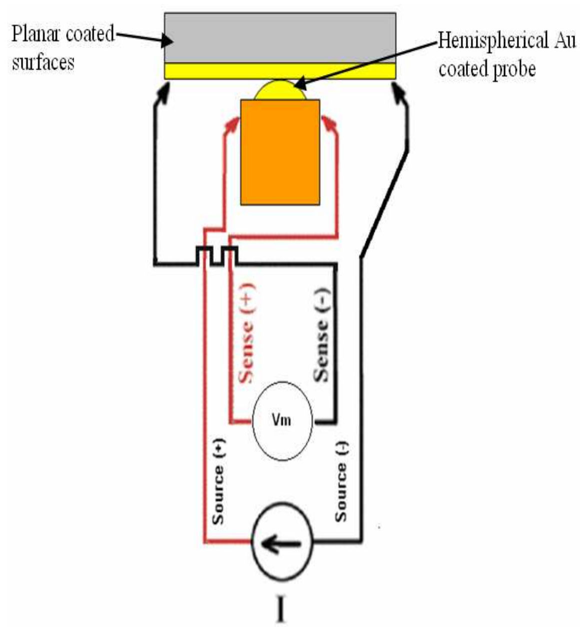

Figure 6: Schematic of contact zone with its electrode and R_c measurement.

The apparatus is enclosed and held at ambient air and room temperature.

In this experiment, the PZT bender is actuated up to 1000 cycles at 0.2 Hz under dry circuit conditions with the contact resistance R_c measured simultaneously. The aim of this initial study is to determine the stability of the contact surfaces, prior to longer duration testing at higher frequencies and under variable load conditions.

In order to replicate the conditions of a MEMS relay, a dynamic applied force of 1mN is used. The coated planar surface and Au ball are brought into contact at 0.2Hz using an applied square wave form, and the gap adjusted so that a maximum load of 1 mN is reached. The targeted load is applied for ~3 seconds so that a representative average contact resistance value can be determined. Fig 7 shows an example of the load history over a period of time. The measured load versus time for Au-Au contact pair is shown to have a noisy signal. This is partially due to the use of a low-pass filter of 10Hz on the charge amplifier. Since the frequency used to actuate the PZT bender is 0.2 Hz, thus any external signal or disturbance above this frequency is then detected. Fig 8 shows of the variation in R_c over a period of time at a frequency of 0.2Hz. This procedure is repeated in order to detect any cyclic changes in the electrical contact resistance.

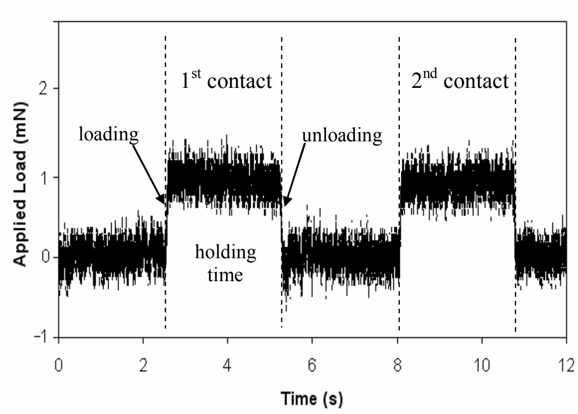

Figure 7: Example of load cycles (0.2 Hz) for an Au-Au contact pair at 1mN.

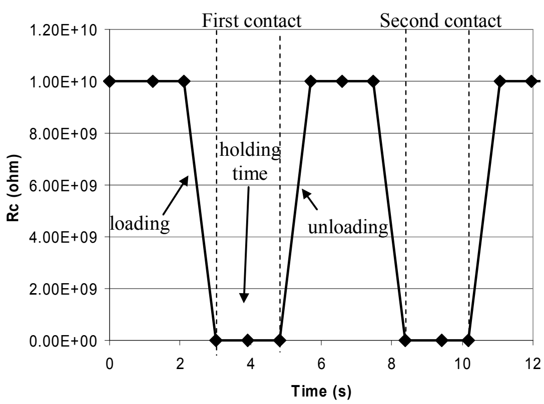

Figure 8: Example of contact resistance during load cycling for Au-Au contact pair.

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 their mechanical and electrical stability. A TaiCaan Technologies XYRIS 4000L laser scanner is used to confirm any changes on the contact surfaces samples such as degradation and wear.

4. Results and Discussion

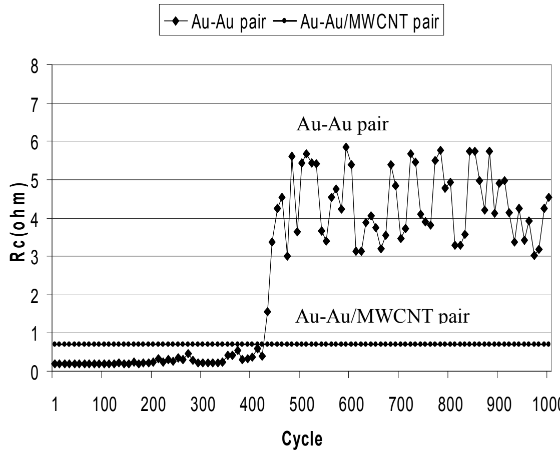

Figure 9: Cyclic contact resistance of Au-Au and Au-Au/MWNT contact pairs.

Fig 9 shows the contact resistance of Au-Au and Au-Au/MWCNT pairs over 1000 load cycles at a maximum (quasi static) applied load of 1mN. The contact resistance of the Au-Au pair is initially ~0.2Ω and increases rapidly to 4-6Ω at 450 cycles. Under dry circuit conditions with 1mA current and a maximum voltage of 20mV the contacts are unlikely to degrade by "hot-switching", therefore the increase in contact resistance is solely due to the mechanical deterioration of the Au-Au contact pair surfaces, reflecting the recognized problems of using soft metals for electrical contacts.

The reason for the sharp increase in R_c of Au-Au pair at ~430 cycles is believed to be due to the initial smoothing of the Au surfaces which leads to increased adhesion [17].. The smoothing is the result of the repeated impacts and time-dependent deformation of the Au. This can be seen in Fig 10 which shows the damaged Au surface planar for the Au-Au contact pair.

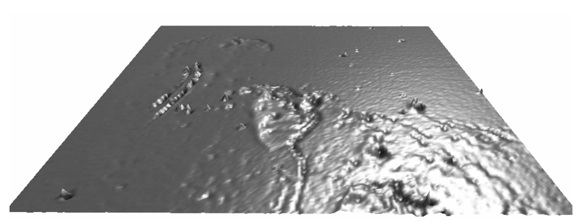

Figure 10: Scanned image of Au planar surface 201x201(0.2mmx0.2mm) using TaiCaan (Xyris 4000CL)

The adhesive force increases with the number of cycles and is consistent with creep being one of the underlying physical mechanisms for the increase in adhesion [18]. The creep of the gold was shown in [11] during a single load cycle, as shown in Fig 11. Fig 11 shows the graph of load against displacement for an Au-Au contact pair. The experiment was carried out using a nanoindentation apparatus. The curve shows there is creep, a deformation that occurs over a period of time when a material is subjected to constant stress, even at room temperature. The force cycle exhibited in Fig 11 occurs over a longer time scale than that used in the current test procedure, with an impact period of approximately 3 seconds.

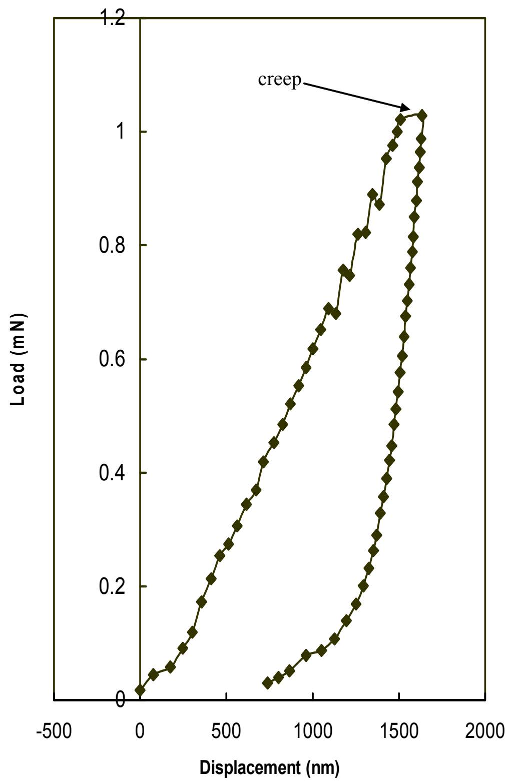

Figure 11: Graph of load against displacement for Au-Au contact pair.

Fig 9 also shows contact resistance of Au-Au/MWCNT contact pair. The initial contact resistance (~0.7Ω) is higher for the Au-Au/MWNT pair. The likely cause is the difference of surface roughness between Au and Au/MWCNT coated planar surface. It is anticipated that this can be improved in subsequent experiments by better process control. The contact resistance is much more stable than for the Au-Au pair over the 1000 loading cycles. This is believed to be due to the deformation required to form the contact being provided by elastic deflection of the MWNTs rather than plastic deformation of the Au surface. As the applied load is increased, more deflection occurs of the MWNTs closing the air gaps between the vertically aligned MWNTs thus improving the transfer of electrons [11].

The hypothesis that the MWCNT's provide elastic deformation is supported by the results in [8] where the use of nanoindentation apparatus with a diamond tip performed indentation on a planar surface coated with vertically aligned CNT was investigated. It was shown that the CNTs deflected elastically as the diamond tip indented the surface layer. The resistance of the CNT forests to penetration is a result of superposition of the bending responses of the nanotubes as the indentor successively encounters nanotubes. In addition, Qi. *et. al.* pointed out that the shorter the CNT the higher the indentation resistance. A graph of load against displacement shown in Fig. 12 shows that MWCNTs are elastic [11]. The curve in Fig 12 shows that there is much less permanent displacement once the indentation load is removed. This is consistent with the MWNT deforming elastically whereas the Au undergoes plastic deformation.

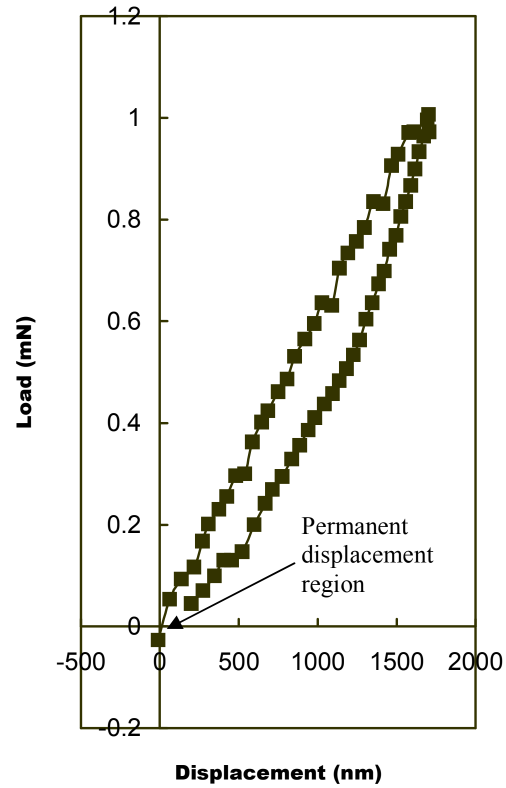

Figure 12: Graph of load against displacement for an Au-MWCNT contact pair.

In addition, the MWNTs conform to the shape of the Au ball probe, increasing the contact area. There are significant indentation marks on the Au ball covering the surface of the Au ball as shown in Fig 13. No visible damage can be detected on the Au/MWCNT planar composite, further suggesting that the CNT under layer has improved the mechanical integrity of the gold surface. The other probable reason for the stable contact resistance of the Au-Au/MWCNT pair is that the CNT deformation reduces the dynamic forces due to the impact of the Au ball, thus decreasing the tendency of smoothing and damaging the surfaces.

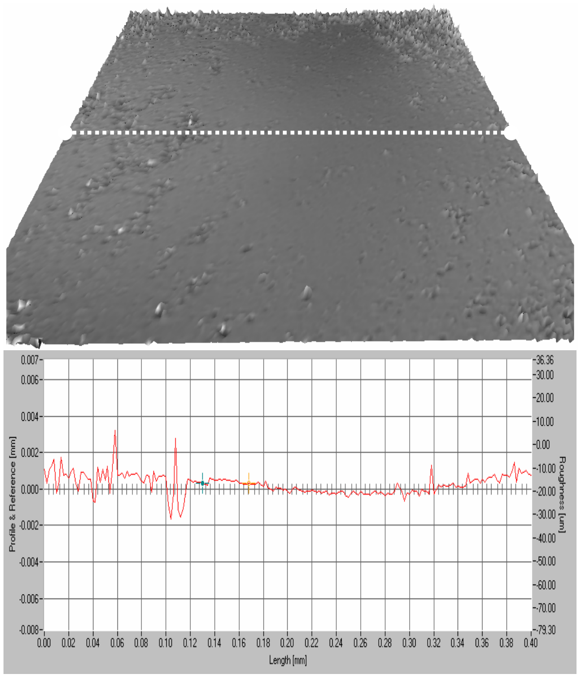

Figure 13: Scanned image of Au ball for Au-Au/MWCNT with the sphere removed, contact pair 201x201 (0.4mmx0.4mm) using TaiCaan (Xyris 4000CL).

5. Conclusion

The applied cyclic load and contact resistance between Au-Au/MWCNT composite contact pairs was investigated using a PZT bender apparatus and R_c measurement methods. This contact pair combination was compared with an Au-Au contact pair. Over 1000 load cycles the Au-Au/MWCNT contact pair demonstrated a much more stable contact resistance than Au-Au contact pair. This improvement is believed to be due to the elastic deformation of the underlying MWCNTs which reduces the plastic deformation and subsequent adhesion between the Au contact surfaces. The test approach appears to be a very promising route towards obtaining in situ measurements of contact resistance on representative surfaces. Future work will conduct testing out to realistic numbers of cycles for MEMS switch applications ($>10^6$).

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