Resistivity changes due to environmental contamination in films used for charge reduction in electrostatic electron optics

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In electrostatic electron optics charging of the surfaces of insulators separating the electrodes can cause undesired beam fluctuation. In prior work we showed that coating the insulators with a film deposited by atomic layer deposition (ALD) could lead to acceptably low charging effects in the REBL electron beam system [1]. However, the stability of the resistivity is affected by contaminants present in the vacuum environment of the electron beam tool. The mechanism of formation for carbon layers typically involves the cracking of hydrocarbon contaminants adsorbed on the film surface by photon, electrons, or heat.

Here we describe changes in resistivity of ALD films 40 nm thick under different operating conditions. The films, comprising layers of zinc and zirconium oxides, were deposited using a Cambridge Nanotech Savannah system on sapphire plates. The metal oxide layers were deposited by sequential pulses of a metal organic precursor and an oxidizing precursor. Between each pulse, the reactor was purged to remove all physisorbed and gas phase precursor. The metal organic precursors used were tertrakis (dimethylamido) zirconium (TDMAZr) and diethylzinc (DEZ). Water was used the oxidizer. Mixed ZnZrO films were obtained by substituting a DEZ pulse with a TDMAZr pulse; the frequency of the substitution determined the Zr:Zn ratio. Substrate temperature was 200degC. The samples were 1x1 cm^2 and a single set or an array of Au/Cr electrodes were deposited as described below.

To demonstrate the effect of vacuum conditions on resistivity, two vacuum systems were utilized. A moderate vacuum system (1E-6 Torr) that consists of a conventional bell jar, an oil roughing pump and a turbo pump was used for both the evaporative deposition of Cr/Au electrodes and for electron bombardment with 309 Volts electrons. A higher vacuum system was ion pumped to about 5E-8 torr and included an electron flood gun (Kimball Physics model EGA-1106) capable of lower electron landing energy. The electrodes for the samples in this system were sputter-deposited. XPS measurements were performed to determine the composition and the surface conditions of the samples at different stages. The results indicated the presence of a thin carbon layer (3-4nm/ 56% atomic) in the samples patterned and measured in the bell jar system (probably due to cracking of the roughing pump oil). About 22% atomic carbon was observed on untreated samples. The first experiments in the bell jar system consisted of applying a low electric field ~3090 V/cm along the sample surface and irradiating it with both heat and electrons. Heating was performed by irradiation from a hot W filament and negatively biasing the film. Simultaneous electron irradiation was performed by reversing the bias of the film. Figure 1 indicates a decrease in resistivity with both heating and electron irradiation. On the other hand, in the ion-pumped system (Fig. 2) the resistivity increases with low voltage electron irradiation. In the ion pump system heating effects are reduced due to the e-gun housing. The bell jar irradiation behavior is consistent with the formation of a carbon surface layer that acts as a parallel current path. The opposite effect observed in the ion pump system indicates a possible change in the microstructure of the film or a surprisingly low energy electron carbon removal mechanism which needs to be studied further.

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Reference 1 – Brodie, A.D., et al, ECS Transactions, 33(2) 101-110 (2010)

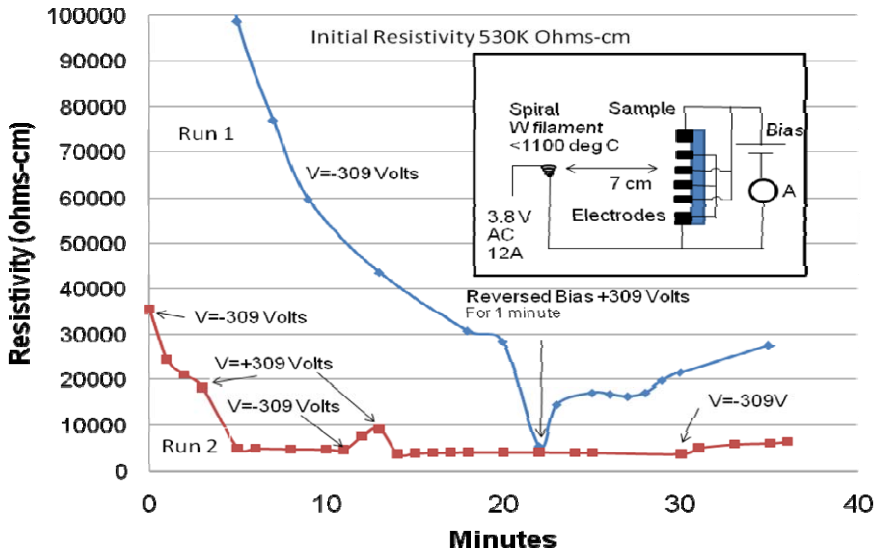

Figure 1. In-plane resistivity measurements in the bell jar system. The sample (51%/49%Zr) patterned with a Au/Cr finger array with 0.1 cm gaps was radiation heated and electron irradiated simultaneously utilizing an applied field of +/-3090 V/cm. The initial in-plane resistivity (Run 1 taken during heating) decreased rapidly after the filament was activated with the AC voltage and out-gassed (top trace). No electrons reached the sample with the negative bias (-309V). After electron irradiation and cooling there was a further decrease in resistivity with more heating and electron irradiation (Run 2 lower trace also taken during heating). With the positive 309 Volts applied, the electron dose was not uniform due to the voltage drop on the film surface (this was somewhat reduced by the interdigital electrode array which established a quasi periodic potential distribution on the whole sample surface allowing several areas between fingers to be exposed simultaneously). However, the point of the experiment was to show the effect of contamination during electron irradiation and quantitative results were only expected from the clean system described below.

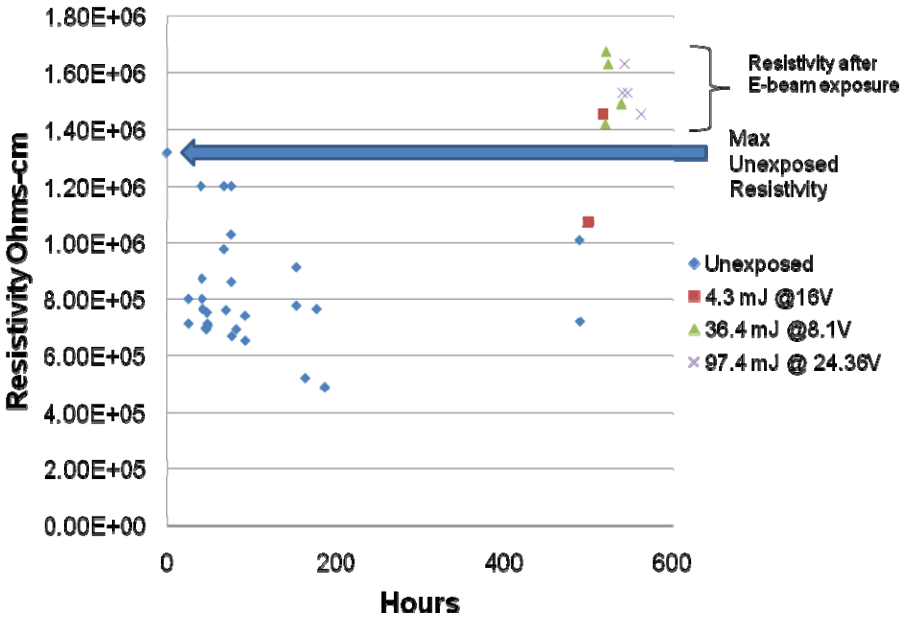

Figure 2. The in-plane resistivity in the ion-pumped system of one 1x1 cm^2 sample (80.2%Zn/19.8%Zr) with a single set of Au/Cr electrodes was measured with an applied field of -309 V/cm prior to e-beam exposure. No high energy electrons reached the sample during all resistivity measurements using the control grid of the Kimball Physics gun. The resistivity varies randomly between 0.5 and 1.3 MΩ–cm with time as shown. The set of sample electrodes were short circuited and grounded during e-beam irradiation to reduce the field non uniformity, and the gun cathode was kept at a negative potential. After an initial dose of ~4.3 mJ/cm^2 at 16 Volts, the resistivity increased substantially measured with the same field of -309 V/cm. Subsequent doses to 8.1 and 24.86 volt electrons are shown. The dose values are shown for the total exposure at a given voltage. The resistivity stabilizes with an applied field of -309V/cm to about 1.5 MΩ-cm (somewhat higher than the maximum measured before e-beam exposure as shown in the figure).