Low resistivity Pt interconnects developed by electron beam assisted deposition using novel gas injector system

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Low resistivity Pt interconnects developed by electron beam assisted deposition using novel gas injector system.

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Abstract. Electron beam-induced deposition (EBID) is a direct write process where an electron beam locally decomposes a precursor gas leaving behind non-volatile deposits. It is a fast and relatively in-expensive method designed to develop conductive (metal) or isolating (oxide) nanostructures. Unfortunately the EBID process results in deposition of metal nanostructures with relatively high resistivity because the gas precursors employed are hydrocarbon based. We have developed deposition protocols using novel gas-injector system (GIS) with a carbon free Pt precursor. Interconnect type structures were deposited on preformed metal architectures. The obtained structures were analysed by cross-sectional TEM and their electrical properties were analysed ex-situ using four point probe electrical tests. The results suggest that both the structural and electrical characteristics differ significantly from those of Pt interconnects deposited by conventional hydrocarbon based precursors, and show great promise for the development of low resistivity electrical contacts.

1. Introduction

The fabrication of nano-scale structures at desired locations, with ultimate precision and resolution is normally achieved by "top-down" nano-lithography processes such as electron beam lithography, nano-imprint lithography (NIL), scanning probe lithography and electron or ion beam induced depositions (IBID or EBID) [1]. Among them EBID is a mask-less lithography process in which an electron beam is focused on a substrate surface and it is used to decompose gas precursor molecules leaving behind non-volatile deposits [2]. The decomposition process is a result of the interaction of surface emitted secondary electrons (SEs) in the irradiated area with locally absorbed gas molecules delivered by a gas injector system (GIS).

Major advantage of the EBID process over EBL and NIL techniques is that it is a direct write process that offers a fast and inexpensive route to direct prototyping and contacting of device architectures as well as the ability to fabricate 3D nano-structures [3]. Therefore FIB/SEM systems

equipped with GIS have now numerous important applications such as carrying out device edits on prototype devices to fix design errors or to probe circuits for failure analysis.

Unfortunately, a major disadvantage of the EBID process with regards to metal deposition is that the deposits are highly resistive [2]. For example Pt structures developed by EBID show resistivities up to 5 orders of magnitude higher than that of bulk Pt which is at 10.6 μΩ cm. This is normally accompanied by very high variation in the reported values, which can only be explained by large variation in the deposition conditions including contamination within the vacuum chamber. Nevertheless, it is well accepted that the main reason for the high resistivity of the Pt interconnects developed by EBID originates from the type of precursor used [4]. The standard precursor used in the GIS is an organometallic Pt compound, methylcyclopentadienyl platinum trimethyl (MeCpPtMe3), consisting of a large hydrocarbon backbone. As a result, the Pt deposits normally suffer from a very large (up to 80%) amorphous carbon content, and show inferior structural integrity. In an effort to reduce the resistivity of such deposits, post-deposition treatments such as high temperature annealing or additional irradiation under high electron beam current have been explored [2]. Unfortunately such steps introduce additional complexity to the process and can be counteractive to fast and inexpensive prototyping *via* EBID. It is postulated that, reducing the hydrocarbon amount within the precursor molecule or using fully carbon-free Pt precursors can lead to depositions with reduced carbon content and improved structural integrity, and therefore improved resistivity, approaching that of bulk Pt. It has been suggested that one such precursor is $Pt(PF_3)_4$ [5].

In this study we examine the applicability of a newly developed GIS for EBID, fitted onto a Helios NanoLab Dual Beam system that operates with $Pt(PF_3)_4$ precursor molecules. A detailed study of the deposition parameters and GIS operation guide lines are presented. Structural and electrical characterization of the obtained interconnects are compared for varying deposition conditions.

2. Experimental

2.1. Pt EBID using $Pt(PF_3)_4$ precursor

Pt interconnects were developed by EBID on FEI's Nova 600i and Helios Dual Beam systems using a GIS specifically designed to incorporate $Pt(PF_3)_4$ precursor. The $Pt(PF_3)_4$ precursor was purchased from Strem Chemicals and introduced into the GIS using a glove box. The depositions were performed after overnight evacuation of the microscope chamber (normally reaching pressures of 10^{-6} mbar). Before deposition the corresponding beam currents were measured using a Faraday cup at various apertures. Changes in the chamber pressure upon dosing $Pt(PF_3)_4$ were strictly monitored and normally resulted in pressure increase of one order of magnitude after opening the GIS valve. The pressure equilibrium is normally reached within less than 5 s after opening the GIS valve. Successive depositions were performed after evacuating the chamber back to its base pressure of about 10^{-6} mbar. Before developing Pt interconnect lines, the performance of the GIS and corresponding deposition rates were monitored by developing 1 μm^2 square patterns at 5 kV and varying beam currents at constant deposition times. Notably, the performance of the GIS system started to deteriorate (e.g. the dosing of the precursor was considerably hindered) after 3 months of usage reflecting the corrosive nature of the precursor that resulted in blockage of the GIS aperture. A new improved designed of the GIS aperture was developed and resulted in reliable performance of the GIS. The improvements required choice of a new material for the manufacture of the isolating O-rings and apertures.

Pt interconnects were deposited at 5 kV on pre-fabricated Au contacts developed by optical lithography on 200 nm thermally grown SiO_2 on Si substrates. Various deposition conditions were explored and the obtained Pt interconnect structures and their electrical responses are discussed below. For comparison, similar depositions were obtained using standard Pt precursor, $MeCpPtMe_3$.

2.2. Structural and electrical characterisation

The morphology of the obtained Pt structures was investigated directly after disposition using the SEM on the Dual Beam systems. Additionally, cross-sections for TEM of the obtained interconnects were prepared and examined by JEOL 2100 TEM. The electrical response of the as-deposited

interconnects was examined by four point electrical testing with a parameter analyzer on a Cascade Probe-station 1200 series.

3. Results and Discussion

3.1. Testing the GIS performance using $Pt(PF_3)_4$ precursor.

The performance of the GIS fitted to FEI's Nova NanoLab systems was monitored for 3 months by writing standard 1 μm^2 square patterns. One such example is shown in Figure 1 (a), where depositions were performed at increasing beam currents. Such depositions were used to calculate the deposition rate after accurately determining the thickness of the deposits. Tilt-view SEM imaging was used as a fast and simple method to determine the thickness of the as-deposited structures. Unfortunately, after comparing the thickness of the structures determined by tilt-view SEM to the thickness determined by cross-sectional SEM, a discrepancy of about 10 % was found. This is due to difficulties in accurately identifying the edges of the deposited structures under tilt-view SEM.

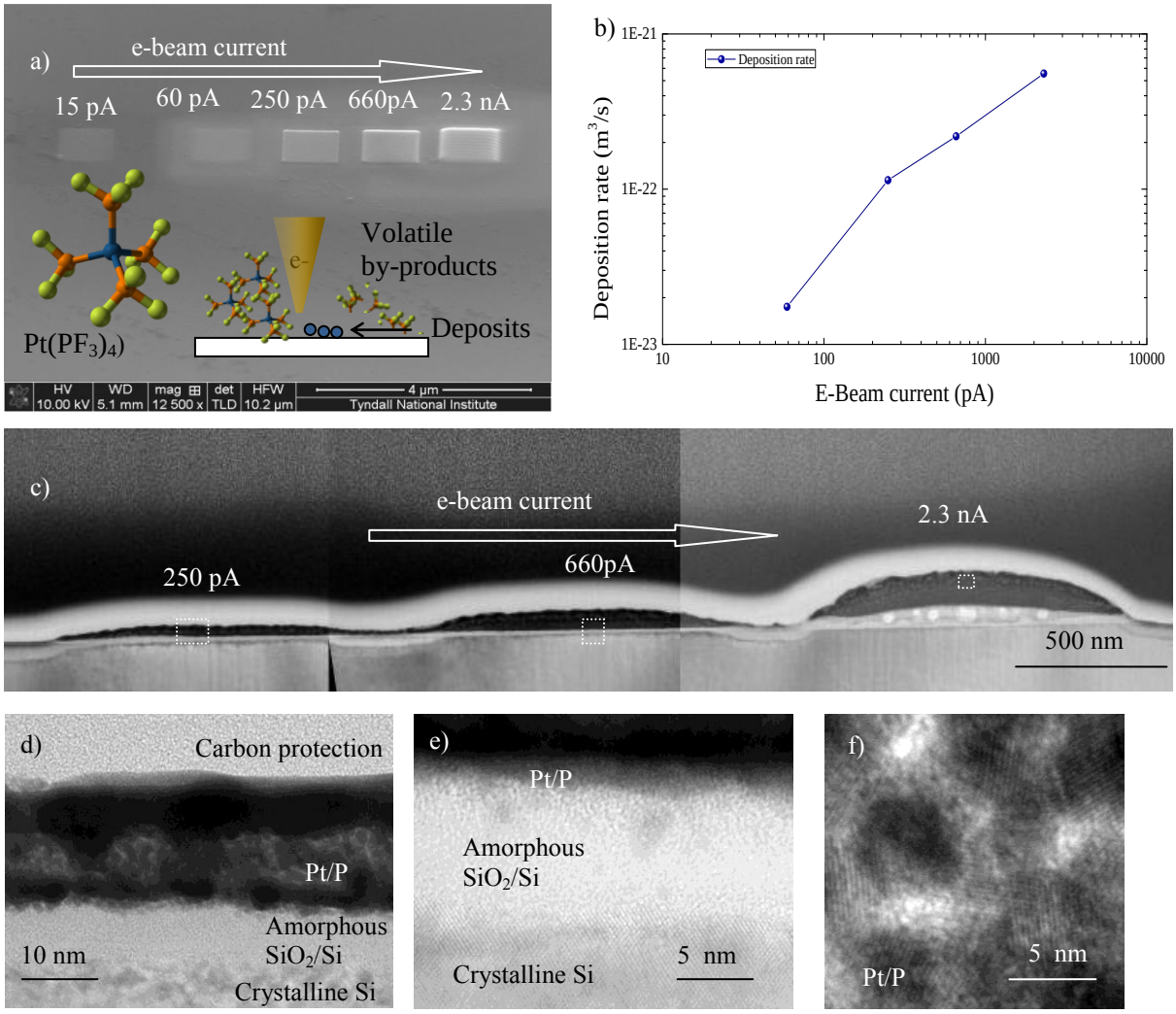

Figure 1. a) Tilt-view SEM image of a set of depositions performed at increasing beam current (inset: schematics of the deposition process using $Pt(PF_3)_4$ molecules), b) calculated deposition rate at increasing beam current, c) TEM cross-sections of deposits at increasing beam current, d), e) and f) corresponding HRTEM images taken at the mark areas. The elemental content was determined by EDX.

More accurate determination of the thickness of the deposited structures and therefore the depositions rates was achieved be performing cross-sectional TEM analysis (Figure 1(c)). Furthermore, cross-sectional imaging supplied important information regarding the shape of the Pt deposits. The images clearly show that the structures have no sharp edges and that the overall cross-section is not rectangular. Using the thickness values, measured at the center of the cross-sections the deposition rate was calculated (Figure 1(b)). The deposition rates show linear increase with the beam current and reach 5.6 10^{-4} $\mu m^3/s$ at 2.6 nA. It should be mentioned that this rate is overestimated since the accurate dimensions of the deposited structures deviate from perfectly rectangular boxes. The deposits are composed of Pt nanoparticles within amorphous phosphorous-containing matrix.

3.2. Electrical properties of Pt interconnects developed by EBID.

Figure 2 shows electrical characterisation of the as-deposited interconnects over pre-fabricated Au contact fingers. The depositions were performed at varying thickness and cross-sections of the interconnects at a set beam current of 880 pA. The length of the interconnects was determined by the spacing between the Au contact fingers (one such device is shown in Figure 2 (b)). The obtained mean resistivity value is at 1160 ±550 (μohms cm) which is about two orders of magnitude higher than that of bulk Pt but at the lower range of resistivity values reported in the literature for as-deposited Pt interconnects by EBID. The *IV*-curves show ohmic dependence (Figure 2 (c)), for voltages between -10 mV and +10 mV. This is different from the obtained non-ohmic behaviour of Pt interconnects developed by using standard Pt EBID precursors.

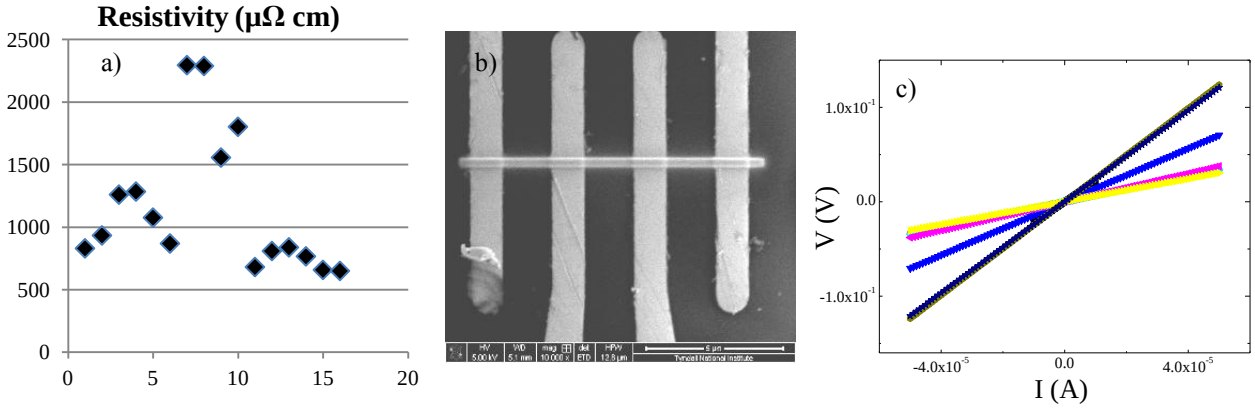

Figure 2. a) Calculated resistivity values for 16 different devices, b) example of one such device, c) experimental *IV*-curves for the corresponding devices.

4. Conclusions

Relatively low resistivity, ohmic Pt interconnects were developed by EBID using novel GIS and characterized structurally and electrically. Post-deposition treatments including gas annealing or in-situ carrier gas deposition might further improve the characteristics of the obtained structures.

Acknowledgements

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References

[1] McCord M A M, Rooks J *Handbook of Microlithography, Micromachining and Microfabrication,* 2000.
[2] Botman A, Mulders J J L, Hagen CW, 2009 *Nanotechnology* **20** 372001.
[3] Furuya K, 2008 *Sci. Technol. Adv. Mater.* **9** 014110.
[4] Botman A, Mulders J J L, Weemaes R, Mentink S, 2006 *Nanotechnology* **17** 3779–3785.
[5] Takeguchi M,·Shimojo M,·Furuya K, 2008 *Appl Phys A* **93** 439–442