Direct-write single electron transistors by focused electron beam induced deposition

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Abstract

Single-electron transistor (SET) device fabrication for operation in the tens of Kelvin range is still challenging due to the need of controlled definition of the metallic island with a diameter far below 100 nm and proper tuning of the island’s tunnel couplings to the drain and source leads. Here we present results on SET device fabrication using focused electron beam induced deposition (FEBID) for island definition between pre-fabricated SET electrode structures. The island’s nano-granular microstructure allows us, in conjunction with in situ tuning of the inter-grain tunnel coupling by post-growth electron irradiation, to study the effect of the island’s electronic granularity on SET device performance. In addition we show that for reliable SET operation FEBID-associated co-deposit in proximity of the island has to be removed which can be accomplished by a novel in situ Ar ion etching process. For the low-temperature properties of functioning SET devices we obtain good agreement of capacitance values deduced from the current–voltage characteristics and capacitance calculations based on the geometry of the device electrodes and the microstructure of the island. Complementary simulations of the SET current–voltage characteristics based on the master equation approach are in good agreement with the experimental data. The observation of well-defined Coulomb oscillations indicates that FEBID-based SET structures can be useful as on-demand charge monitor devices with high lateral positioning flexibility.

Introduction

Over the last 40 years, increasing interest in ultra-low-power and ultra-high-density integrated circuits has pushed the development to smaller and smaller devices. With achieving the ability of making miniaturized elements on the scale of less than few hundreds of nanometers, devices based on single-electron effects have been realized and even proposed as replacement for silicon transistors [1–6], albeit not for very large scale integration.

The single-electron transistor (SET) is based on a nano-island which is connected by tunnel junctions to source and drain electrode leads and is capacitively coupled to one or more gate electrodes. In the island, electrons are confined and their number is quantized. The current through the island can be tuned by the gate voltage which controls the number of excess charges on the SET island. Coulomb blockade in SETs is the basis for the realization of ultra sensitive electrometers [7–10], memory devices [11–15], logic switches and gates [16–19], molecule-based transistors [20, 21], displacement [22, 23] and chemical sensors [24, 25].

The most used SET implementation methods are shadow evaporation [26–29], scanning probe microscope tip modification [30, 31], electromigration [32–34], nanoscale oxidation [35, 36] and mechanically controlled break junction formation [37, 38]. By the advance of nanofabrication processes and technology the use of nanoscale objects like nanoparticles [39–44], nanotubes [45–47], fullerenes [48, 49] and single-molecules [20, 50] as nano-islands for SET devices has been demonstrated. However, controlling the size and position of the nano-island as part of an SET device can be very challenging if self-assembly process steps are involved. In this framework, the use of focused electron beam induced deposition (FEBID) has been reported as suitable
technique for SET fabrication [51–55], FEBID is a direct-write technique based on the electron beam assisted dissociation of precursor molecules adsorbed on a substrate surface inside a scanning electron microscope (SEM). The precursor is injected through a gas injection system into the SEM chamber in close proximity to the substrate surface and in the focus of the electron beam it dissociates into volatile fragments and the non-volatile deposit [56]. FEBID serves numerous application fields since it can be used on any kind of substrate, is capable of sub-10 nm resolution, and a wide range of materials can be deposited with high flexibility in pattern design, even in 3D [57, 58]. In particular, FEBID is used in mask repair [59, 60], plasmonics [61, 62], nano-magnetism [58, 63], superconductivity [64, 65] and strain sensing [66–68]. The main advantage of FEBID as compared to conventional lithographic techniques is that, being a direct-write technique, intermediate steps such as resist-coating, developing, metal deposition and lift-off are not necessary. In this way, possible modifications of the surface chemistry and shape of the nanostructures are bypassed. The majority of FEBID precursors are metalorganic which generally results in carbon-rich deposits with low metal content. This is the case for the platinum precursor MeCpPtMe3 (Me: methyl, Cp: cyclopentadienyl) which leads to a Pt content of 16–26at% [69]. The deposit consists of nm-sized grains of fcc Pt embedded in a carbon matrix and the distance between the grains is on the 1 nm scale [70]. Therefore, as-grown Pt FEBID deposits are nano-granular metals whose electrical transport is the result of the interplay of diffusive charge transport inside the Pt grains and thermally assisted tunneling between those grains [57]. The delocalization effects due to tunneling and the tendency for localization can be finely tuned from insulating to metallic behavior through a post-growth electron irradiation treatment [71–74]. Lately, several other methods have been reported adopting high temperature treatments (300°C–550°C) and/or exposure to O2, H2O or H-radicals [69, 75–81] or pulsed laser light exposure [82]. Although FEBID provides very defined structures, it has the disadvantage of a co-deposition halo in close proximity to the defined structure caused by precursor dissociation beyond the incident beam location mainly due to secondaries caused by backscattered electrons [83–86]. For SET fabrication with FEBID the halo can undermine device operation, e.g. by undesired charge trapping in localized states near the nano-island. In the work reported here we have used post-growth irradiation of a nano-granular Pt island positioned between pre-fabricated drain, source and gate electrodes in conjunction with in situ localized Ar+ ion-etching [87] in order to remove the halo in proximity of the island. By this we were able to tune the tunnel coupling strength inside the island and between the leads so that stable SET operation was achieved. The obtained results have been confirmed by capacitance simulations performed with the software Fastcap2 [88] and complementary simulations of the SET current–voltage characteristics based on the master equation approach, see e.g. [89, 90]. The observation of well-defined Coulomb oscillations indicates that FEBID-based SET structures can be useful as on-demand charge monitor devices with high lateral positioning flexibility and may serve as building blocks of more complex structures.

Experimental details

We used p-doped (100) silicon as substrate material insulated with a 200 nm thick thermally grown SiO2 layer on which contact electrodes of 35 nm Au/Cr were prepared by UV-lithography in a cross configuration. The chip was cleaned in a solution of H2O2 and H2SO4 in a 1:3 ratio (piranha solution) to remove any surface contaminants. The device definition entailing Ga+ ion-milling of the contacts, island deposition, post-growth irradiation and localized Ar+ ion-etching, was carried out in a dualbeam SEM/FIB microscope (FEI, Nova Nanolab 600) equipped with a Schottky electron emitter.

In the first step, a Ga+ ion beam with 30 kV acceleration voltage and 10 pA current was used to etch the cross region of the contacts in order to obtain a base SET design with two gates (figure 1(a)). The distance between source and drain electrodes was around 50 nm and the distance between the cross center and the two gate electrodes was 150 nm.

For FEBID of the nano-granular Pt island we used the precursor MeCpPtMe3 which was heated to 44 °C for 60 min prior to deposition. The precursor was injected in the SEM by a capillary with 0.5 mm inner diameter in close proximity to the focus of the electron beam on the substrate surface. The island was written with an electron beam energy of 15 keV, a current of 140 pA, 25 μs dwell time and 200 passes. The chamber pressure during deposition was about 7.5 × 10⁻⁶ mbar. In order to increase the inter-grain tunnel coupling inside the islands, the islands were subjected to a post-growth electron irradiation treatment. This was carried out 3 h after deposition in order to ensure complete removal of residual precursor before the irradiation. Samples A and B, presented here, were treated with irradiation doses of 3 μC μm⁻² and 1 μC μm⁻², respectively. Samples subjected to lower irradiation doses were also fabricated but are not reported here since they did not show SET behavior.

As a last step in SET fabrication, the island region was subjected to localized Ar+ ion-etching inside the SEM using a beam induced polishing and sputtering system installed in our SEM by Thermo Fisher Scientific [87]. A
localized Ar flow over the sample was provided by a capillary having an inner radius of 17.5 μm which was positioned in close proximity to the sample. The substrate was tilted by 45° from the e-beam axis, maintaining 100 μm distance in vertical direction from the Ar gas nozzle. A voltage of 100 V was applied between the gas nozzle and the substrate surface. Ionization of the Ar flux was established by scanning the e-beam over a slit previously cut with the Ga⁺+ FIB close to the nozzle aperture (see figure 1(b)). Thus generated thermal Ar⁺ ions were accelerated in the electrostatic field between the nozzle and the substrate causing sputtering of the sample surface over a disc-like field of approximately 20 μm diameter. The island region was exposed for 60 s overall etching time divided in 15 s intervals. This ion etching timing was chosen such that the in situ measured drain-source resistance at room-temperature ended up in the range of 500 kΩ–5 MΩ.

After fabrication the samples were inserted in a 3He cryostat equipped with a vacuum-sealed insert. The time period between venting the SEM and mounting the samples in the evacuated cryostat insert was kept as short as possible in order to minimize deposit aging effects [77, 91]. Current–voltage curves were taken at the lowest accessible temperature of 270 mK and up to 160 K. For the measurements a Keithley two-channel sourcemeter (model 2636A) was used. The source contact was forced to ground potential defined by the insert. Data were taken in two-probe geometry. Contact and wiring resistances were about 300 Ω as was verified by complimentary four-probe measurements.

Results

In figure 2(a) we show a SEM image of sample A and in figure 2(b) the corresponding circuit diagram. The tunnel junctions drain-island (Jdi) and island-source (Jis) can be represented as capacitances (Cis, Cdi) and resistors in parallel (Ris, Rdi), respectively. The island is capacitively coupled to two gates having the capacitances Cgs1 and Cgs2. The two gate capacitances act in parallel and are connected to the gate voltage source Vgs. The total gate
Capacitance is $C_{cc} = C_{gs} + C_{gd} + C_0$, where $C_0$ is the background capacitance formed by the gate electrodes and the p-doped Si substrate.

Before the exposure to the $Ar^+$ ion-etching, Sample A and Sample B showed a total resistance $(R_{tt} = R_{ds} + R_{ss})$ at room-temperature (290 K) of 2.7 kΩ and 6.3 kΩ, respectively. Their resistance strongly decreased after exposing them to 60 s (in 15 s intervals) of $Ar^+$ ion-etching. In particular, after the etching process and still inside the SEM chamber Sample A had a total resistance of 1.6 MΩ and Sample B of 2.8 MΩ. These resistance values did not change by more than 5%–10% towards low temperature.

In Figure 3(a) we present the current–voltage characteristics of sample A at 270 mK for various gate voltages in 0.05 V steps up to 5 V. The diamond-shaped features around zero drain-source voltage $V_{ds}$ as well as the step-like increase of the drain-source current $I_{ds}$ versus $V_{ds}$ at a gate-voltage dependent threshold voltage (Coulomb blockade) are typical for a functional SET [90, 92–94]. In particular, the first step-like increase is due to the probability $p(n = ±1)$ of having one excess charge on the island beginning to increase from 0. In parallel, the probability $p(n = 0)$ of having no excess charge begins to fall from 1. At the second step in addition the probability of having two excess charges $p(n = ±2)$ starts to rise from zero at the expense of the probability $p(n = 0)$.

A particularly clear representation of the Coulomb blockade regions is provided by the stability diagram shown in Figure 4(a), where the differential conductance $dI/dV$ as obtained by numerical differentiation of the $I$–$V$ curves from figure 3(a) is drawn as filled contours versus $V_{gd}$ and $V_{gs}$. Apparently, the Coulomb blockade diamonds appear periodically and with constant shape. We observe the largest Coulomb blockade for $V_{gs} = 0.15$ V which repeats itself every 0.75 V interval. This interval corresponds to $\frac{1}{e}$, where $e$ is the electron
charge. From this we obtain \( C_{gg} = 0.21 \, \text{aF} \). The initial shift of 0.15 V on the \( V_{gg} \)-axis is due to a background charge of \( Q_0 = C_{gg} \times (0.75 - 0.15) \, \text{V} \) leading to \( Q_0 = 0.76|e| \).

Analyzing further, from the maximum threshold voltage \( V_{ds}^{\text{max}} \) the capacitance \( C_S \) can be derived using \( V_{ds}^{\text{max}} = \frac{|e|}{C_S} \) with \( C_S = C_{di} + C_{is} + C_{gg} \). The symmetric shape of the diamonds furthermore indicates that the two junction capacitances are very similar \( (C_{si} \approx C_{di}) \) and that the gate capacitance is small in relation \( (C_{gg} \ll C_{si}) \). The remaining capacitances and the charging energy \( E_C \) can now be extracted from the stability diagram. We obtain \( C_{gg} \approx C_{di} = 2.06 \, \text{aF}, C_S = C_{gg} + 2C_{Is} \approx 4.33 \, \text{aF} \) and \( E_C = \frac{|e|^2}{C_S} \approx 0.018 \, \text{eV} \).

In figure 4(b) we show for sample A the gate-voltage dependent current through the island for different drain-source voltages as indicated. Peaks in the current—the Coulomb oscillations—occur whenever the Coulomb blockade is suppressed due to the gate voltage. The excess charge confined to the island in the blockade region is quantized and each peak is associated with the change of the total excess charge by one electron.

We now turn to the temperature dependence of the device characteristic. Thermally assisted tunneling through the barriers leads to a shrinkage of the Coulomb blockade region. In figure 5 we present the evolution of the \( I_d(V_{ds}) \)-characteristics for \( V_{gs} = 0 \) for a selection of temperatures. With increasing temperature the Coulomb blockade region shrinks until at about 160 K the Coulomb blockade is completely gone and the \( I-V \) characteristic becomes linear. This corresponds favorably with the calculated charging energy \( E_C = 0.018 \, \text{eV} \) which implies about 210 K thermal energy. From this, reasonable SET operation for sample A is expected up to about \( T_{\text{max}} \approx 30 \, \text{K} \), corresponding to the criterion \( E_C > 7k_B T_{\text{max}} \).

We now show selected results obtained for sample B, which has been subjected to a smaller post-growth irradiation dose. A SEM image is presented in figure 6.

Similar to sample A, sample B shows Coulomb blockade regions in the stability diagram (see figure 7(a)). However, the diamonds are not simply periodic with increasing \( V_{gs} \). This is also apparent from figure 7(b) which reveals a much more complex structure of the Coulomb oscillations. In addition, the threshold voltages are diode-like asymmetric reaching maximum absolute values of 0.023 and 0.015 V for positive and negative bias, respectively (see figure 8). This corresponds to less than half the value observed for sample A. We conclude this section by noting that for sample B, in contradistinction to sample A, the Coulomb blockade disappears already for \( T \) larger than about 77 K, as is expected due to the smaller charging energy.

**Discussion**

We begin our discussion by analyzing whether the device parameters \( (C_{si} \approx C_{di}, C_{gg}) \) deduced from the stability diagram are plausible with regard to the device geometry and material properties. In a first step a 3D-model of the devices was generated considering the geometry of sample A. This is straightforward for the pre-defined drain, source and gate electrode structures but less so with regard to the nano-granular Pt island. For this we assumed an overall composition of Pt\(_{17}\)C\(_{83}\) consistent with the beam parameters during deposition. For the island geometry after the irradiation treatment we assumed a core–shell configuration with a metallic Pt core of semi-spherical shape with 37 nm diameter and a hollow carbon semi-spherical shell of 3.5 nm thickness. The
SET model structure was located on top of a homogenous SiO₂ dielectric with a relative dielectric constant of 3.7 at 0.3 K \[95\]. Ga⁺-ions implantation \[96\] in the SiO₂ did not lead to appreciable leakage between source and drain contacts as evidenced by a current level of about 0.1 pA at 2 V after Ga⁺-milling. The p-doped Si was assumed to be either metallic, thus representing an additional electrode, or insulating, as is expected to be the case at 0.3 K. The results of the capacitance calculation reported here refer to the insulating case. Next, all surfaces of the model were subdivided into small quadrangular- or triangular-shaped elements (see figure 9) which were used in capacitance calculations employing Fastcap2 \[88\], a fast field solver that uses an adaptive multipole algorithm based on a generalized conjugate residual iterative technique that allows to solve for the elements of the capacitance matrix. The capacitances resulting from the simulation were \(C_{is} = C_{di} = 2.03 \text{ aF}\) and \(C_{gs} = 0.14 \text{ aF}\) in very reasonable agreement with the deduced experimental values.

We proceed now to simulating the SET \(I-V\) characteristics. To this end we employ the master equation approach in the stationary case, as, e.g. detailed in \[99\]. Very briefly, we calculate the tunneling rates between the island and the drain and source electrodes using Fermi’s golden rule under the assumption of energy-independent tunnel matrix elements. These rates depend on the change of free energy of the SET as the island charge is changed by \(\pm |e|\). In addition, we calculate the probabilities for excess charge occupation \(p(n)\) of the island from the probability continuity equations assuming stationary conditions. This then allows us to calculate the drain-source current for any given drain-source and gate voltage. We used the capacitance values deduced from the stability diagram of sample A. The junction resistances at low temperatures are not known to us, since we could not measure to such high \(V_{ds}\) values to be in the linear regime without destroying the SET structure.

Figure 6. SEM image of sample B.

Figure 7. (a) Stability diagram of sample B. The diamonds are not simply periodic and do also vary in size in contradistinction to the stability diagram of sample A (figure 4 (a)). The black, red and blue lines correspond to the \(V_{gs}\) values for which the Coulomb oscillations in (b) are reported. (b) Complex Coulomb oscillation characteristic for sample B for different \(V_{gs}\) as indicated.
However, by comparing our simulation results for $I_{ds}(V_{ds})$ for different values of $R_{is} = R_{di}$ with the measured $I$–$V$ characteristics, we found good correspondence for $R_{is} = R_{di} = 750 \, \Omega$ or $R_{is} + R_{di} = 1.5 \, \text{M} \Omega$ which is almost identical with the measured room-temperature drain-source resistance of sample A. In figure 8 we show the results of our simulations of the stability diagram and the Coulomb oscillations for direct comparison with the experimental data presented in figure 4. The agreement is quite satisfactory, as is particularly evident by directly comparing the experimental results (device parameters, current peak positions of Coulomb oscillations) with the simulation, see figure 11. The values differ by less than 5%.

We now turn to the specific advantages we see in using the SET fabrication approach presented here. Starting from the UV-lithography contacts the complete SET fabrication as well as electrical testing can be done in situ. This is a clear advantage when using a direct-write technique such as FEBID in the prototyping stage or for few device fabrication. An additional advantage lies in the type of island material deposited. The island consists of a nano-granular metal with only 2–3 nm Pt grain size [70]. Nano-granular metals are tunable systems in which the electronic properties are the result of the interplay of the diffusive charge transport inside the grains and the (thermally assisted) tunnel processes between the grains combined with the essential features of general disordered system [57, 97]. By means of the post-growth irradiation treatment the inter-granular tunnel coupling strength can be finely tuned in the vicinity of the critical tunnel coupling at which an insulator-metal
transition occurs in the three-dimensional case [72]. This increase in tunnel coupling strength derives mainly from an enhanced graphitization of the carbon matrix [71].

As indicated in figure 12, different transmission channels exist which can be described through different types of conductances normalized to the quantum conductance $G_0$: $g_0$, the intra-grain conductance, $g$, the conductance between an electrode lead and the most favorable metal grain in the island, and $g'$, the conductance between the grains in the island. $g'$ is an effective quantity and may itself be written following Landauer [98] as the sum of all conductance channels the electron has when propagating through the nano-granular island. For a nano-granular metal the identification of the most important conductance channels is a hard problem due to the large charging energies associated with the small grain size [97]. If $g' \ll 1$, the island is insulating at low temperatures. On the other hand, if $g' \gg 1$ the nano-granular island is metallic. Considering that $g_0 \gg g, g'$ we are left with different regimes depending on the ratio $g'/g$. For $g'/g \gg 1$ the island is behaving as a metallic system and the device resistance is dominated by the small parameter $g$. In the weak lead-coupling regime $g \ll G_0$ the charge is quantized and confined to the island. This represents the standard working regime of a SET. For $g'/g \approx 1$ and if $g' \geq 1$ at least one of the junctions is in the strong lead-coupling regime. In this case the probability of co-tunneling is strongly enhanced resulting in undesired leakage currents through the SET [99].

Here the nano-granular nature of the island can improve the situation, since for $g' \approx 0.1$ co-tunneling events spanning from drain to source can be effectively blocked if the number of grains is not too small. Yet, the conductance inside the island is still large enough to not compromise its quasi-metallic behavior. For this reason, the use of FEBID for nano-granular island definition in conjunction with post-growth irradiation allows for fine tuning the device towards effective suppression of leakage currents in the Coulomb blockade regime. Finally, if $g'/g \ll 1$ the device is too highly resistive. In our case, sample A shows very defined and reproducible Coulomb diamonds and Coulomb oscillations typical of a device with metallic island. The irradiation dose of
3 μC μm⁻² used for this sample relates to bulk nano-granular Pt FEBID deposits with quasi-metallic behavior of the temperature-dependent conductance [72]. For a single island, this dose appears to be enough to promote metallic behavior. In contradistinction, for sample B (irradiation dose 1 μC μm⁻²) the Coulomb diamonds and the Coulomb oscillations are not as well defined, albeit still clearly visible. We speculate that for this dose g′ is slightly too small, so that different excess charge trapping distributions over the nano-grains in the island occur with comparable probability. We consider this multiplicity of charge trapping states within the island to be the most important factor for the weaker performance of sample B. An additional factor to be considered is that for lower irradiation doses one expects an increased island to drain/source resistance for otherwise identical geometrical dimensions. Furthermore, the effective dielectric constant of the carbon matrix of the island does also depend on the irradiation dose.

We conclude our discussion with some remarks concerning the halo. As a consequence of the post-growth irradiation of the island region an associated conductance increase of the halo may be promoted. This can and does cause several problems, such as leakage currents, parasitic capacitances and charge trapping, all of which compromise SET operation. In preceding experiments comparable structures not treated with Ar⁺ ion-etching did not show any evident conductance dependence on Vgs. From this we conclude that the Ar⁺ ion-milling step for halo removal is essential. The parallel milling of the island and part of the leads is uncritical as the halo thickness is only in the 1 nm range for nano-islands of the size fabricated in this work. Nevertheless, the brief milling step is apparently enough to remove electrostatic inhomogeneities around the island. Thanks to the in situ electrical monitoring of the source-drain current in between the Ar⁺ ion-milling intervals, the associated resistance increase could be nicely followed and ensured that the conditions for which Coulomb blockade effects occur can be met, such as R_{ds/di} 26 kΩ.

Conclusions

In this work we present the focused electron beam induced deposition direct-write fabrication of lateral-gate single electron transistors where the nano-island consists of a nano-granular Pt dot of roughly semi-spherical shape of about 40 nm diameter. By means of a post-growth electron irradiation treatment of the island, we were able to tune the tunnel-coupling strength between the Pt grains in the island, as well as the junction couplings to source and drain. By this, different transport regimes of the SET are established. As a result of localized Ar⁺ ion-etching undesired co-deposit in the island region is removed, a process that can be conveniently monitored by in situ drain-source conductance measurement. By drain-source I–V characteristics measured for different gate voltages and at different temperatures we demonstrate stable transistor operation for SET structures subject to optimally tuned post-growth irradiation doses. Capacitance simulations of the working device and simulations of the SET I–V curves are found to be in very good agreement with the experimental data. We consider our approach to be particularly useful for prototyping and single to few SET device fabrication with high flexibility.

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