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Polycrystalline silicon nanowires synthesis compatible with CMOS technology for integrated gas sensing applications

R. Rogel, E. Jacques, L. Pichon, and A.C. Salaun

Abstract—Polysilicon nanowires are synthesized following a classical top-down approach using conventional UV lithography technique fully compatible with the existing silicon CMOS technology. N- and P-type *in-situ* doping of these nanowires is controlled over a large range of doping levels and electrical properties of these nanowires are analyzed. Results show that resistivity dependence with the doping level is both related to the nanowires size dependent structural quality and doping specie. Charged gas species (ammonia) sensitivity of these nanowires has also been studied. In addition, feasibility of N- and P-channel polysilicon nanowires transistors is demonstrated.

Index Terms— polysilicon, nanowires, in situ doping, LPCVD, TFT.

I. INTRODUCTION

Semiconducting nanowires are currently attracting much attention as promising components for future nanoelectronic devices such as nanowire field effect transistors [1], photonic and optoelectronic devices [2], and more particularly as chemical or biological sensors [3-5]. The needs of a fast and precise detection of early disease symptoms, as well as the need of environment safety, become now the main leitmotiv of the societal development. The incorporation of semiconducting nanowires into chemical and biological sensors applications receives a great interest. As their surface can be sensitive to charged species combined with their high surface to volume ratio, semiconducting nanowires are the subject of intense research activities for high sensitivity chemical sensor fabrication. In particular, for silicon nanowires (SiNWs) based electronic devices, the highly sensitive detection based on SiNWs enables a change in

current when, after surface functionalization, the analytical (charged) molecules bind to specific recognition molecule at the SiNWs surface. In this way, many studies reported on biological sensors for DNA hybridation [4], chemical [6] and gas detection [7].

SiNWs can be prepared by one of two approaches, “top-down” and “bottom up”. In a bottom up strategy the individual base elements (atoms, molecules...) of the system are linked together to form larger subsystems. Synthesis methods most developed are layer-by-layer self assembly [8], Vapor Liquid Solid (VLS) and Solid Liquid Solid (SLS) growth techniques [9,10], and using matrix template [11]. The main drawbacks of these synthesis methods for a 3D integration are the difficulty in control of size and positioning of the nanowires. In this case, nanowires need to be selectively collected and manipulated to be assembled in a planar layout. The “top down” approach starts from bulk materials and scales down the patterned areas. In this way, several advanced nanopatterning techniques were developed such as e-beam [12], atomic force microscopy (AFM) [13,14], deep UV [15] and nanoimprint lithographies [16,17], to obtain SiNWs. The main drawbacks of these advanced lithographic tools with nanometer size resolution rest on the high cost generated, and more generally the low throughput capability unsuitable with mass production. Because SiNWs synthesis can be compatible with the established silicon technology, SiNWs based sensor integration will allow a lower manufacturing cost, in addition to the advantageous electronic features of embedded detection and signal processing in silicon technology. The intrinsic reliability of the well-known semiconductor CMOS (Complementary Metal Oxide Semiconductor) process also guarantees reproducible and reliable performances. Recently, previous works demonstrated the CMOS compatibility of SiNWs top down fabrication. Some of them [18] reported on SiNWs electronic building blocks, with nanowires fabricated from silicon bulk substrate and then collected to another (plastic) substrate. Others authors [19,20] used e-beam lithography techniques for SiNWs fabrication using costly SOI (Silicon On Insulator) substrates.

In addition, a high reliable doping control at nanoscale still remains a challenge for development of nanoscale device

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integrated in electronic, sensing, photonic,... systems. The ability to predict and control electronic doping level of SiNWs, using knowledge obtained from the planar silicon technology, is a key feature for nanoscale device applications. In other words, high and uniform doping control is required for a precise control of electrical properties of SiNWs based devices. In this study, we present a direct patterning technique of *in-situ* doped SiNWs planar arrays by conventional optical lithography, compatible with the existing planar CMOS silicon technology. Such SiNWS are promising active building blocks for gas (ammonia) sensors and electronics.

II. EXPERIMENTS

The key fabrication steps of the polycrystalline silicon NWS are illustrated in figure 1 (a). At first, a dielectric film A is deposited and patterned into islands by conventional UV lithography. Then, a polycrystalline silicon layer is deposited by LPCVD technique. Accurate control of the polycrystalline silicon layer reactive ion etching (RIE) rate leads to the formation of nanometric size sidewall spacers that can be used as nanowires. The feasibility of these polycrystalline silicon NWs with curvature radius as low as 50 nm was previously demonstrated [21]. This method allows the fabrication of parallel SiNWs network.

Phosphorus (or Boron) *in-situ* doped polycrystalline silicon layers used for such nanowires were deposited by thermal decomposition of a mixture of pure silane, SiH_4 , and phosphine, PH_3 , (or diborane, B_2H_6). The *in-situ* doping level is controlled by adjusting the PH_3/SiH_4 (or $\text{B}_2\text{H}_6/\text{SiH}_4$) mole ratio varying from 0 for undoped films to 4×10^{-4} (or 5×10^{-4}) for heavily doped films. Silicon films were deposited in an amorphous state at 550°C and 90 Pa, and then crystallized by a thermal annealing in vacuum at 600°C during 12 hours. The corresponding incorporated phosphorus (boron) atoms concentrations, C_P (C_B), previously determined from SIMS (Secondary Ions Mass Spectroscopy) analysis, varies from 2×10^{16} to $2 \times 10^{20} \text{ cm}^{-3}$ (or 2×10^{16} to $5 \times 10^{19} \text{ cm}^{-3}$) [22]. Such results stand as reference for doping concentration in our processed SiNWs, because SIMS analysis on single nanowires is unfortunately not easy to implement.

The *in-situ* doped polycrystalline silicon NWs were integrated into resistors devices in coplanar structure (fig. 1 (b)) for electrical characterization. In this way, the nanowires were capped by a (100nm thick) SiO_2 layer deposited by Atmospheric Pressure Chemical Vapor Deposition (APCVD) technique at 420°C and contact openings wet etched. Contacts electrodes were made of thermally evaporated aluminium and defined by wet etching. Finally a thermal annealing in forming gas ($\text{N}_2/\text{H}_2:0.9$) was carried out at 390°C to ensure good electrical contacts. Resistors were fabricated with 10 μm length parallel SiNWs.

III. RESULTS AND DISCUSSION

Electrical resistivities, ρ_n and ρ_p , of N- and P-type doped polycrystalline silicon NWS respectively were deduced from the I-V characteristics (slope) of polycrystalline silicon NWs based resistors, collected at room temperature using a HP 4155 B semiconductor parameter analyzer. The *in-situ* doping effect is studied through the dependence of the resistivity with the doping concentration for two different radius curvature (50 and 100nm). As shown in figures 2 (a) and (b), doping effect is different with phosphorus or boron doping species.

A first partial study, previously reported by our group [23] on the N-type *in-situ* doping control on high curvature radius (100nm) —polycrystalline silicon NWs, showed that the resistivity increasing the doping level from about $1 \times 10^{18} \text{ cm}^{-3}$ results in an abrupt resistivity drop of about 6 orders of magnitude for only a factor of 10 further increase in doping concentration. Beyond that range resistivity decreases to reach the same value as for crystalline silicon. For N-type polycrystalline silicon NWs with 50nm curvature radius similar behaviour is observed, with a lower magnitude for the abrupt resistivity drop at higher doping concentrations, and higher resistivity values at high doping levels. However, the shape of these two corresponding curves, quite similar to previously observed results for *in-situ* doped polycrystalline silicon layers [22, 24], indicates that polycrystalline silicon NWs doping effect is in accordance with the Seto's theory [25], in particular for high curvature radius. These results can be related to the spatial defect distribution related to the curvature radius of the polycrystalline silicon NWs. Indeed, two types of defects can be involved either located i) at the surface of the wire and/or ii) in the core of the polysilicon layer. In this last case, because the amorphous silicon crystallization process begins at the dielectric film A/amorphous silicon interface, the defects density (including grain boundaries) is higher in the inferior part of the polycrystalline silicon layer (few nanometers thick) constituting the nanowires (see figure 3). This grain morphology and defect density were previously revealed by electron microscopy study [26].

According to Seto's theory [25], carriers transport is controlled by energy barriers induced by the trapping effect of carriers at defects located at grain boundaries. However, for lowest curvature radius (50nm) polycrystalline silicon NWs, this conduction mechanism is not dominant. Indeed, as illustrated in figure 3, grain boundaries as well as intra-grain defects densities are higher, and it is more realistic to consider structural quality quite similar to (amorphous-type or) highly disordered silicon material. Therefore, one can consider that defect density is uniformly distributed in the core of the polycrystalline silicon NWs. In this case, a more appropriate carriers transport is the variable range hopping of carriers [27]. In this model, firstly described by Mott [28], hopping refers to carriers tunneling transitions from occupied to unoccupied localized states, the state energy difference being bridged by

emission of absorption of one or several phonons.

For P-doped polycrystalline silicon NWs, the size of the nanowires does not influence the dependence of the electrical resistivity with doping concentration (fig. 2(b)): for both the electrical resistivity decreases with the increase of the doping concentration. In addition, the similar doping level dependence as for N-doped polycrystalline silicon NWs is not so evident, in particular with a slight resistivity drop region. This behaviour agrees with results previously observed for boron *in-situ* doped polycrystalline silicon layers [22]. In fact, the *in-situ* doping effect of boron is different from the phosphorus, and depends on the pressure deposition. Indeed, similar ρ_p dependence with C_p , as for *in-situ* N-doped polycrystalline silicon layer, was observed for P-doped polysilicon layer deposited at lower deposition pressure (30Pa) [22]. This phenomenon is not well clarified for thin film polycrystalline silicon layers, and thus for polycrystalline silicon NWs. However, another study reported that for the 50nm curvature radius *in-situ* boron doped polycrystalline silicon NWs carriers transport follows variable range hopping mechanism [27].

Our results highlight the good control of the doping level on low curvature radius ($\leq 100\text{nm}$) polycrystalline silicon NWs over a wide range. The polycrystalline SiNWs gas (ammonia) sensitivity was studied using resistors based on uncapped undoped polycrystalline silicon NWs used as gas sensors. Electrical resistance ($R=V/I$) measurements, deduced from the slope of the linear I-V curves, are reported as function of time for devices under exposure to ammonia. Prior to measurements, devices were submitted to high diluted hydrofluoric acid (2 %) aqueous solution to remove the native oxide on the SiNWs surface in order to promote chemical species adsorption. The electrical measurements were carried out at room temperature in nitrogen with a protocol described as follow. At first, SiNWs based devices were put into a vacuum chamber for 2 hours ($P=10^{-4}\text{mbar}$). Prior ammonia injection nitrogen gas injected during few minutes to guarantee baseline leveled off. Then, the sensor was exposed to ammonia until realizing the onset of sensor response saturation. Flow and gas pressure dependences were monitored, and arbitrary ammonia pressure in the chamber during SiNWs based resistors exposure is about 500mbar. The potential use of polycrystalline SiNWs as sensitive units to ammonia was checked by measuring the detector response, S_g , defined as:

$$S_g = \frac{I - I_g}{I} \quad (1)$$

where (I) and (I_g) are the current values for devices in nitrogen and reactive ambient respectively.

The chemical responses of these SiNWs based resistors to ammonia exposure are presented in function of exposure time in figure 4. Measurements are carried out with 50 parallel (100nm radius curvature) SiNWs based devices. Upon exposure to ammonia tests, time dependence change of the electrical resistance is found to indicate a quasi reversible trend because of the recovery baseline leveled off, suggesting

that polycrystalline SiNWs could be reusable after exposure. Electrical current increases because ammonia species act as donor of electrons (reducing agents) at the SiNWs surface [29]. This behavior was previously observed [30] for such polycrystalline SiNWs based resistors under ammonia exposure. In this case, after evacuating the vacuum chamber, diluted ammonia into nitrogen is injected under controlled concentrations (700, 350, and 175 ppm) and shows that the detector response S_g increase is proportional to the increase of the ammonia concentration. These first quantitative results, acting as proof of concept, show the potential use of polycrystalline SiNWs as gas (ammonia) sensitive units. However, further studies concerning sensitivity, low detection limits, and reproducibility are needed for gas sensors applications.

Such results are explained because the gas molecules adsorption occurs at the SiNWs surface, electrons are transferred to the polycrystalline silicon SiNWs core. Such interactions induce significant changes in the carriers transport along the nanowires and SiNWs electrical resistance (or current) in different possible cumulative ways. First, as the SiNWs conductance can be modulated by an applied voltage [29,31], positively charged gas molecules (electron donors) binded on SiNWs surface can modulate their conductance by changing the volume of the conductive layer. In this case, ammonia may act as chemical gates. In other words, it means that the Fermi level of the silicon nanowires is shifted to the conduction band edge reducing the sample electrical resistance. Moreover, carrier transport strongly depends on structural nanowires defects. So, we have to consider effects of grain boundaries. Through charge exchange, gas molecules adsorbed may play a significant role in decreasing the potential barrier height at the grain boundaries between two grains. Previous work reported such effect [32]. In this case, adsorbed gas molecules passivate defects like dangling bonds leading to an increase of current [33]. The electrical current increase under ammonia exposure suggests that electrons transfer and potential barriers lowering effects may dominate. Such results act as proof of concept and show the potential usefulness of the polycrystalline silicon nanowires for high sensitive gas sensors applications operating at room temperature.

N- and P-channel Thin Film Transistors (TFTs) are fabricated using undoped polycrystalline silicon NWs following the synthesis method described previously. In addition, two types of TFTs are studied: back gate and top gate devices (fig. 5). For back gate transistors the substrate is heavily doped crystalline silicon wafer acting as gate electrode (see fig. 5 (a)). The film A acting as gate insulator is a 100 nm thick Si_3N_4 layer deposited by LPCVD technique at 600°C . Source and drain regions are made of heavily *in-situ* N-type (P-type) doped polycrystalline silicon layer deposited by LPCVD technique for N- and P-channel transistors respectively. This layer is patterned by reactive ion etching with SF_6 plasma before thermal evaporation of aluminum used for source and drain electrodes. For the top gate TFT (fig. 5 (b)), the (100nm

thick) SiO₂ capping layer acting as gate insulator is deposited by CVD technique at atmospheric pressure at 420°C after heavily *in-situ* doped polycrystalline silicon Source/Drain regions plasma patterning, and wet etched for opening contacts. Gate electrode is patterned with source and drain electrodes.

Transfer and output characteristics are displayed on the figures 6 and 7 for the two types 100nm radius curvature polysilicon NWs TFTs. Electrical parameters, threshold voltage, V_T , and optimum field effect mobility, μ , are determined according to the classical conduction electrical model used for the MOSFET (Metal Oxide Semiconductor Field Effect Transistor) [34]. μ is deduced from the maximum slope of the $I_{DS}(V_{GS})$ curve (transconductance) measured in the saturation mode, and V_T is determined by the intercept of the $I_{DS}^{1/2}(V_{GS})$ curve with the gate axis voltage. Switching ratio is defined as maximum (On state) to minimum (Off state) currents ratio. Average values of electrical parameters summarized in the table 1 show better electrical properties for the top gate polycrystalline silicon NWs TFTs, with a higher switching ratio and field effect mobility, and lower threshold voltage. These results are explained to the better gate insulator/polycrystalline silicon NW interface quality. Indeed, as illustrated in the figure 3, for the back gate TFTs channel region takes place in the seed layer with a higher defects density than the upper polycrystalline silicon NW surface [26]. In addition, such defects result to a poor the electrode/channel interface quality responsible of parasitic contact resistances because of the possible non linear behavior of drain current at low source-drain voltages. A lowering of the electrical properties was also observed for polycrystalline silicon NWs TFTs made with lower curvature radius polysilicon NWs, due to a higher defects density [35].

Polycrystalline SiNWs based TFTs show lower electrical properties than for those based on polycrystalline silicon layer. However, our study shows that back gate TFT could be used as sensitive blocks because in this configuration polycrystalline silicon NW can be submitted to charged species ambient, and thus the field effect could be promising to amplify chemical species detection. Thanks to their promising electrical properties, top gate transistors are potential candidates for electronics conditioning sensing signal.

IV. CONCLUSION

Our study shows the great flexibility in design of planar SiNWs array by direct patterning technique using conventional lithographic tools. The N- and P-type polycrystalline silicon NWs doping control make them good candidates for the fabrication of electrically controlled thin film devices (resistors and transistors), in particularly for specific gas (reducing) sensing or electronics applications.

Results show the full compatibility of the nanospacer polysilicon nanowires technology with the existing silicon CMOS technology, using nanowires as potential sensitive units

for integrated gas sensors applications. Indeed, field effect behaviour observed in polycrystalline silicon NWs based transistors is promising to amplify chemical species detection as well as for electronics conditioning sensing signal for back gate and top gate configurations respectively.

In addition, thanks to silicon surface functionalization possibilities, such results offer a great potential for further developments of integrated SiNWs based (bio)chemical sensors and their implementation in electronic systems.

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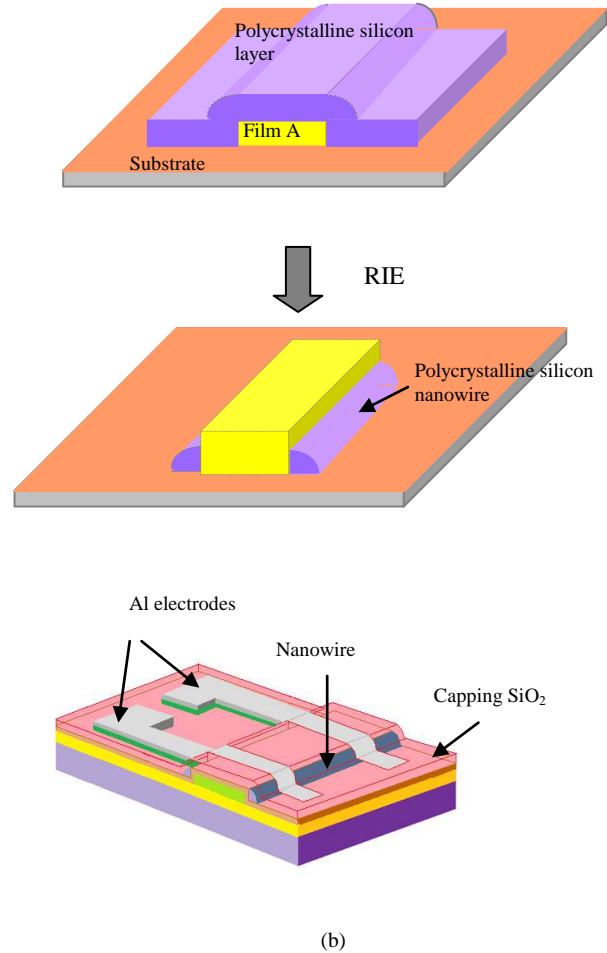


Figure 1. Fabrication of polycrystalline silicon NWs by the sidewall spacer formation technique (a). Polycrystalline silicon nanowires based resistor for electrical characterization (b)

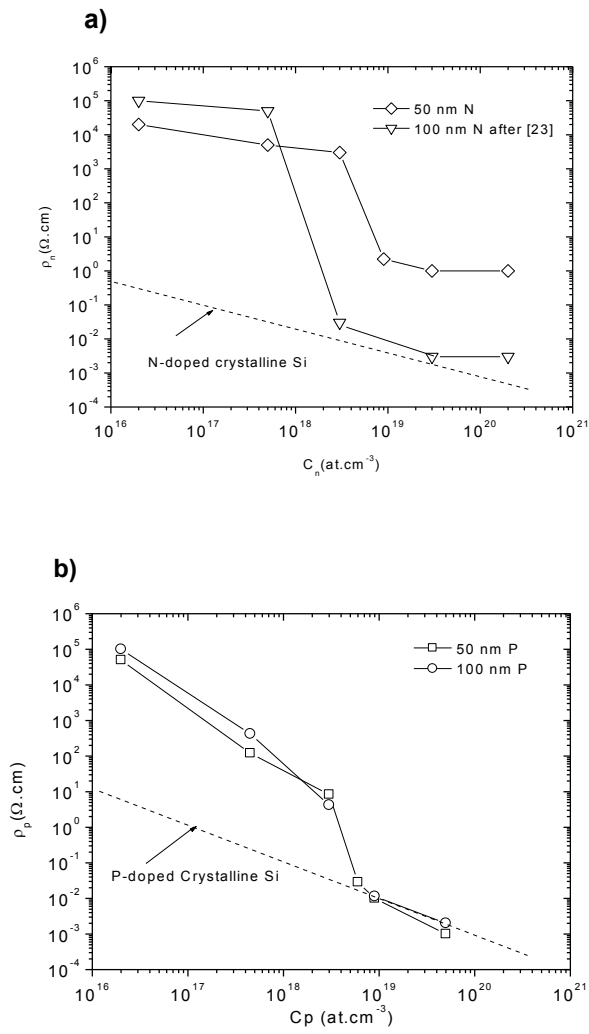


Figure 2. Electrical resistivities of (50nm and 100nm curvature radius) polycrystalline SiNWs variations versus phosphorus (a), boron (b) concentration.

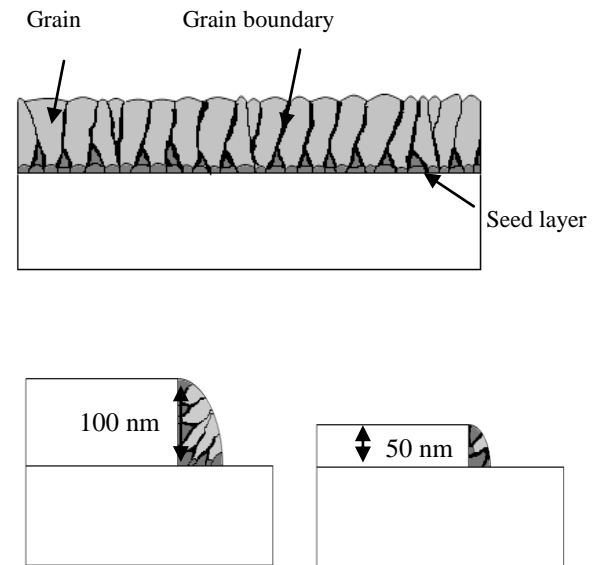


Figure 3: Schematic cross section view illustrating the columnar type structure of a polycrystalline silicon layer: size lowering leads to a higher defects density within the polycrystalline SiNWs.

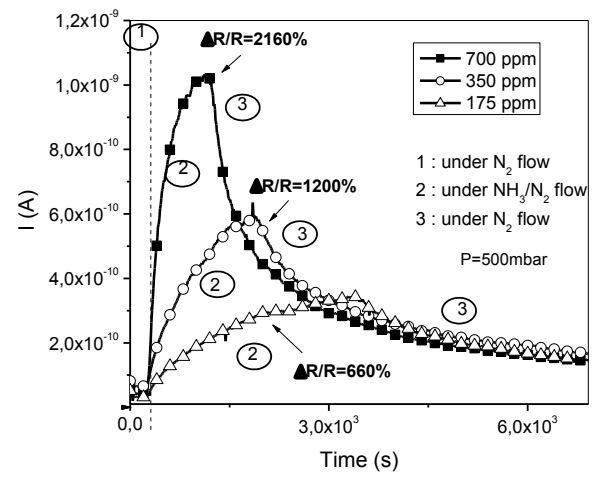


Figure 4. Variations of the current versus time under controlled ammonia exposure. The pressure adjusted in the chamber is 500 mbar, and measurements are carried out at room temperature. SiNWs radius curvature: 100nm

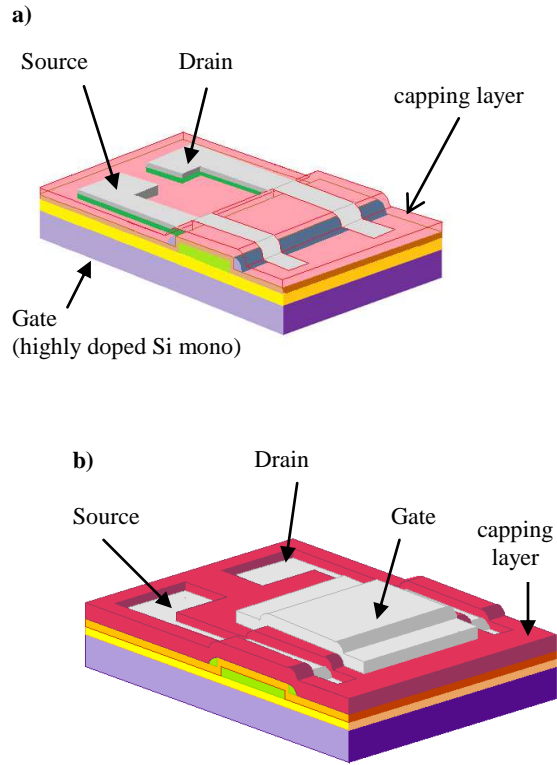


Figure 5: Back gate (a) and Top gate (b) polycrystalline SiNWs based TFTs.

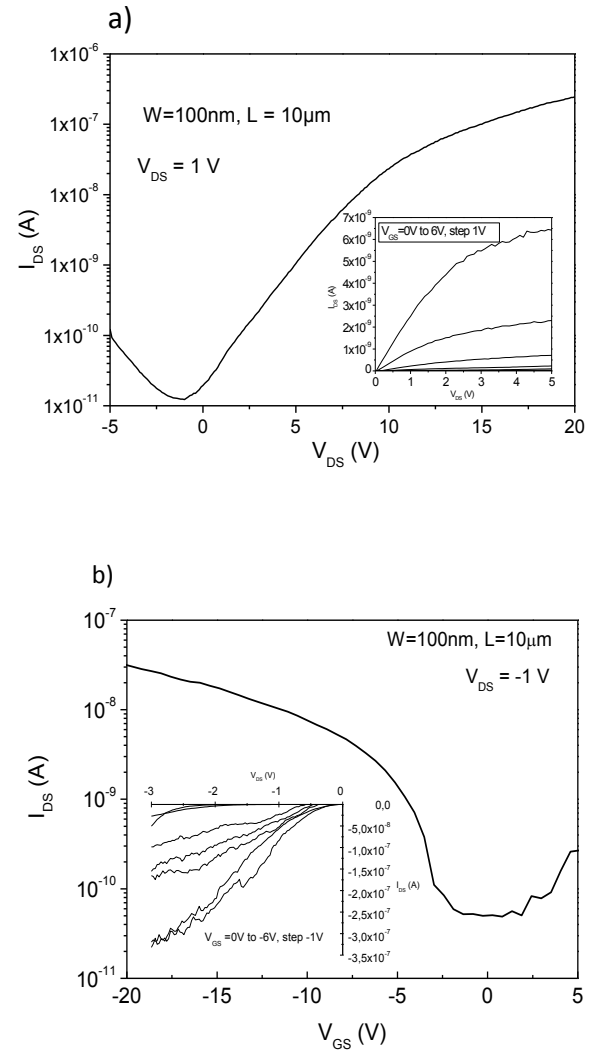


Figure 6: Transfer electrical characteristics of N-channel (a), P-channel (b) back gate polycrystalline SiNWS based TFTs. Inserts output electrical characteristics.

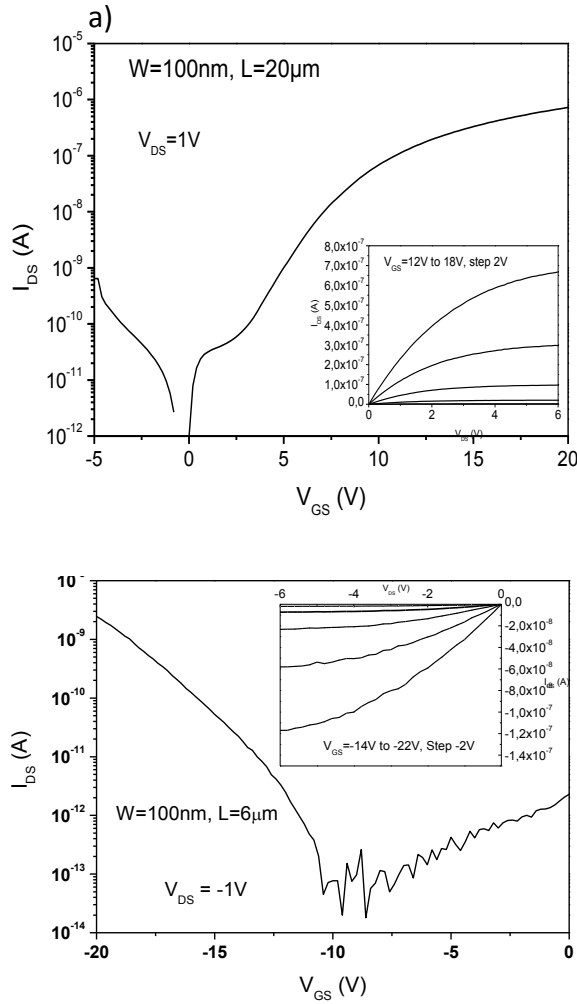


Figure 7. Transfer electrical characteristics of N-channel (a), P-channel (b) top gate polycrystalline SiNWS based TFTs. Inserts output electrical characteristics.

TABLE 1. SUMMARY OF THE ELECTRICAL PARAMETERS (AVERAGE VALUES) OF THE BACK AND TOP GATE TFTS MADE OF 100NM CURVATURE RADIUS POLYCRISTALLINE SILICON NWS.

| | Top Gate | | Back Gate | |
|-----------------------------|-----------|-------------|-------------|-----------|
| | N-channel | P-channel | N-channel* | P-channel |
| μ (cm ² /Vs) | 30 | 12 | ≤ 3 | < 3 |
| V_T (V) | 7 | - 16 | 7 | - 8 |
| Switching ratio | 10^6 | $\sim 10^7$ | $\sim 10^4$ | $>10^3$ |

* Results for back gate N-Channel polycrystalline SiNWS based transistors were previously reported in the ref [23]

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