## Micro and Nano Transducers for Autonomous Sensing Applications

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Abstract—With the advent of Wireless Sensor Networks and the Internet of Things, the need for energy efficient components has increased significantly. Sensors consume sometimes a significant part of the energy budget of an autonomous system, while it is not always clear how to make them more efficient. Here we attempt to identify strategies for lowering power consumption in transducers. The discussion is constructed around a formula that relates the power consumption to the other relevant parameters for sensing such as noise, sensitivity, and conversion rate.

Sensors and transducers; low power cosumption; eventproportional sensing; miniaturization and nanomaterials;

### I. INTRODUCTION

Autonomous, wireless sensor networks promise to transform society via a plethora of applications in physiological, asset and environmental monitoring, or other yet unforeseen applications. Energy efficiency of the sensor nodes is of central concern, because in many scenarios, the sensors have to rely on compact batteries or energy harvesters for operation. An intelligent system like a sensor node can be factored into a few functions, namely computation, communication, sensing, actuation, and energy generation. For a limited energy budget, it is natural to ask how much energy each of these essential functions requires, and furthermore, to which extent the energy dissipation per function can be reduced.

At the elementary level, it appears that an energy minimum of kT ln 2 seems to be required regardless of whether one tries to send [1], measure [2] or process [3] a bit of information. Shifting the abstraction level away from the bit-level operations, there are many other essential functions in intelligent systems, such as signal amplification and conditioning, data conversion and transmission, and others still. Power dissipation bounds have emerged over the years for some of these operations as well [4]. For example, a bound for the power dissipation in amplifiers has been derived by Vittoz [5] for class-B and Annema [6] for class-A amplifiers. Walden [7] derived a similar bound for the power dissipation in data converters. Empirical data gathered by Murmann [8] showed that the other non-idealities limit the dissipation well before the thermal noise limitation is reached in practice. Doherty et al. [4] provide further analysis into the energy dissipation of other operations.

Due to the wide variety of sensing principles, it seems hopeless to find a similar bound for sensors. Nonetheless, in this work we propose a power dissipation bound for electronic transducers. The view assumed here is that the sensor task is to measure an input quantity within a prescribed range with a resolution represented in an equivalent number of bits at a certain sampling rate. The focus will be the transducer and not the adjacent circuitry needed for measuring it. This bound will allow us to systemize strategies for lowering dissipation in sensors.

#### II. POWER DISSIPATION BOUND

Let P denote the power dissipated by the transducer, N the equivalent number of bits of required resolution, and  $\Delta f$  and  $\Delta x$  the required input bandwidth and amplitude range, respectively. Then,

$$P \ge c_{\alpha} \frac{4kT \cdot \Delta f}{\left(\Delta x / 2^{N}\right)^{2} \cdot \left(S_{Q} / Q_{0}\right)^{2}} \tag{1}$$

This expression consists basically of four factors: *the* confidence factor  $c_a$ , the noise factor  $4kT\cdot\Delta f$ , the resolution factor  $(\Delta x/2^N)^2$ , and the transducer figure-of-merit  $(S_Q/Q_0)^2$ . Intuitively, (1) states that the minimum power required by a transducer is determined by the required resolution, the noise power, and the sensitivity.

The expression is dimensionally correct, and it can be derived for linear, ratiometric transducers with additive Gaussian noise. In addition, it can be derived for the class of electronic sensors such as resistive or capacitive transducers regardless of the readout circuit topology. Because of these particular examples and because it exhibits the expected scaling with respect to the different factors, we postulate this power dissipation bound to be valid for general nonlinear sensors.

#### III. APPLICATION-SPECIFIC STRATEGIES

A few parameters in (1) are external, i.e. they come with the sensing application itself. These are the sensing rate  $\Delta f$ , the input range  $\Delta x$  and the required resolution in number of effective bits N. To no surprise, the better one knows the ranges and resolution required for the application to still run appropriately, the more energy efficient the sensor module can be made.

An extreme case is reached in the case of trigger sensors, for which the effective number of bits N is 1. Such a sensor will only be able to tell if the input crosses some predefined threshold, in which case we say that it has detected an event. In certain applications, the event rate is extremely irregular. An example are micro seismic events whose rate and intensities have been shown to be good predictors of cliff collapse [9]. The seismic event rate in the frequency range up to 10kHz varied over 3 orders of magnitude. Sampling above the minimal Nyquist rate, would result according to (1) in unjustified energy dissipation, since most of the time, there is no event in the signal. A much better approach is one in which the sensor is idle most of the time consuming little to no power and it wakes up only when an acoustic event arrives. We call this strategy event-proportional sensing. Embracing this strategy, we have proposed recently an energy-efficient acoustic emission sensor. This device consumes very little energy in the absence of an acoustic event. When such an event arrives, the mechanical transducer, amplifies the acoustic wave in a passive fashion via wave shoaling on a tapered, lumped massspring chain [10]. The amplified wave can either be transduced capacitively [11], or further converted into an electrical event via a pull-in instability.

#### IV. LOW NOISE SENSORS

The second category of parameters appearing in (1) is specific to the sensor technology itself. These are the noise power, the sensitivity  $S_Q$  and the offset or quiescent output value  $Q_0$ . Here the general strategy is to optimize the architecture, consider miniaturization or look for different sensing materials, notably nanomaterials.

What is not immediately apparent in (1) is that in most cases, the noise factor should include excess noise besides the thermal noise 4kT. In this respect each device architecture, materials and processing conditions bring a different noise figure. When it comes to electronic excess noise, device miniaturization is in general unfavorable, simply because the surface to volume ratio has to increase, rendering the fewer charge carriers more susceptible to environmental fluctuations. In the case of carbon nanotubes, we have shown that fabrication process cleanliness can result in a noise factor reduction by up to an order of magnitude. Nonetheless, it is not known if noise can further be reduced in nanodevices.

Even though miniaturization seems to be detrimental to noise, in some cases it is beneficial to sensitivity. Interestingly, the sensitivity gain seems to overcompensate the loss in noise figure. This is the case of carbon nanotube resonators, for which monoatomic mass resolution has been demonstrated [14].

The offset factor  $Q_0$ , can be reduced by utilizing sensors which are normally off and turn on in the presence of the input stimulus. One example has already been given in the previous subsection, the acoustic event detector. Another example would be a carbon nanotube field effect transistor utilized as a gas sensor, biased such that it is off in the absence of the gas and turns on proportionally with the gas concentration. Finally, nanomaterials like carbon nanotubes offer another energy efficiency improvement strategy that is not immediately apparent in (1). In particular, solid state gas sensors, are notorious for their energy inefficiency as heating of the sensing material is required either for activation or for refreshing the sensing material. In [14] we have shown that it is possible to reach micro-power consumption levels by utilizing, individual suspended carbon nanotubes as gas sensing material and Joule selfheating for refreshing the sensor.

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- C. Shannon, "A mathematical theory of communication," Bell System Technical Journal, The, vol. 27, pp. 379–423, July 1948.
- [2] L. Brillouin, "Maxwell's demon cannot operate: Information and entropy. I," Journal of Applied Physics, vol. 22, no. 3, pp. 334– 337, 1951.
- [3] R. Landauer, "Irreversibility and heat generation in the computing process," IBM journal of research and development, vol. 5, no. 3, pp. 183–191, 1961.
- [4] L. Doherty, B. Warneke, B. Boser, and K. Pister, "Energy and performance considerations for smart dust," International Journal of Parallel and Distributed Systems and Networks, vol. 4, no. 3, pp. 121–133, 2001.
- [5] E. Vittoz, "Future of analog in the VLSI environment," in Circuits and Systems, 1990., IEEE International Symposium on, pp. 1372–1375 vol.2, May 1990.
- [6] A.-J. Annema, "Analog circuit performance and process scaling," Circuitsand Sys tems II: Analog and Digital Signal Processing, IEEE Transactions on, vol. 46, no. 6, pp. 711–725, 1999.
- [7] R. H. Walden, "Analog-to-digital converter survey and analysis," Selected Areas in Communications, IEEE Journal on, vol. 17, no. 4, pp. 539–550, 1999.
- [8] B. Murmann, "A/D converter trends: Power dissipation, scaling and digitally assisted architectures," in Custom Integrated Circuits Conference, 2008. CICC 2008. IEEE, pp. 105–112, Sept 2008.
- [9] D. Amitrano, J.R. Grasso, G. Senfaute, "Seismic precursory patterns before a cliff collapse and critical point phenomena", Geophysical Research Letters, vol. 32, p. L08314, 2005.
- [10] M. Müller, V. Maiwald, M. Käch, C. Hierold, C. Roman, "A passive micromechanical broadband amplifier for acoustic emission sensing", in TRANSDUCERS 2015, pp. 1129-1132, June 2015.
- [11] V. Maiwald, M. Müller, C. Ritz, C. Hierold, C. Roman, "Shoaling vibration amplifier with flattened transfer function and suppressed spurious modes", in IEEE MEMS 2017, January 2017.
- [12] K. Chikkadi, M. Muoth, W. Liu, V. Maiwald, C. Hierold, "Enhanced signal-to-noise ratio in pristine, suspended carbon nanotube gas sensors ", Sensors and Actuators B: Chemical, vol. 196, pp. 682-690, 2014.
- [13] B. Lassagne, D. Garcia-Sanchez, A. Aguasca, and A. Bachtold, "Ultrasensitive Mass Sensing with a Nanotube Electromechanical Resonator", Nano Letters vol. 8, pp. 3735-3738, 2008.
- [14] K. Chikkadi, M. Muoth, V. Maiwald, C. Roman, C. Hierold, "Ultra-low power operation of self-heated, suspended carbon nanotube gas sensors", Applied Physics Letters, vol. 103, p. 223109, 2013.

### Out-of-Equilibrium Body Potential Measurements in Ψ-MOSFET for Biosensing

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Abstract - A novel method for surface charge sensing using the  $\Psi$ -MOSFET is presented. Systematic measurements of out-of-equilibrium body potential were performed on SOI wafers. The interest of  $\Psi$ -MOSFET as a biochemical sensor is experimentally proved. The advantages of this method are high sensitivity, simplicity and low-bias reading compared to the conventional I<sub>D</sub>-V<sub>G</sub> method.

### *Keywords – SOI, Ψ-MOSFET, body potential, biochemical sensor, field-effect*

#### I. INTRODUCTION

The  $\Psi$ -MOSFET characterization of SOI wafers serves to determine electrical parameters such as the threshold voltage, the mobility or the interface trap density [1]–[4]. This technique uses the bulk silicon substrate as a backgate and the buried oxide (BOX) as a gate dielectric. The gate voltage (V<sub>G</sub>) induces a channel at the interface between the silicon film and the BOX. The conduction from source to drain is ensured by either holes or electrons depending on the V<sub>G</sub> polarity. The coupling effect between the top free surface charge and the channel leads to threshold voltage variations useful for sensing purposes [5].

The present paper aims to propose an alternative approach, by measuring the potential rather than the current. This unconventional technique presents noticeable advantages in biochemical sensing. It derives from body potential measurements in the out-ofequilibrium regime already performed in FD-SOI MOSFETs [6].

#### II. MATERIALS AND METHODS

The SOI substrates analysed have a BOX thickness of 145nm and a film thickness of 88nm. Islands of  $5x5mm^2$  were patterned using lithography and reactive ion etching. The electrical measurements were carried out with a HP4155 Analyser. In our specific setup, three metallic probes with controlled position and pressure were placed on the wafer surface (Fig. 1). Two of them (no. 1 and 3) play the role of source and drain whereas the third one (no. 2) measures the body potential. To avoid leakage current, the Source/Measurement Unit (SMU) fitted with high input impedance was used to probe the body potential by imposing a zero current condition. The drain current (I<sub>D</sub>) and gate current (I<sub>G</sub>) vs. gate voltage (V<sub>G</sub>) are monitored

simultaneously with the body potential  $(V_B)$  for validation purposes.

#### III. RESULTS

#### A. Body potential measurements

When  $V_G$  is scanned from accumulation to inversion (Fig. 2), the body potential starts to increase with a quasi linear behaviour and then decreases. The body potential variation occurs only in the region of full depletion where drain current is negligible. Significant variations of  $V_B$  are associated with significant variations of  $I_G$  (for  $V_G$  between -1.3V and -0.1V). This particular  $I_G$  behaviour demonstrates that the  $V_B$  variations are related to transient mechanisms when switching  $V_G$  between the accumulation and inversion regimes.

In Fig. 3 and Fig. 4, the I<sub>G</sub>-V<sub>G</sub> and V<sub>B</sub>-V<sub>G</sub> characteristics are assessed for different delay times between measurement points. Faster sweeps are beneficial for sensitivity as they induce higher peaks of both I<sub>G</sub> and V<sub>B</sub>. This reflects the time-dependent characteristic of this type of measurement, for which a complete understanding will be provided through simulations.

#### B. Application to APTES and gold nanoparticles

The suitability of body potential measurements for sensing applications was tested by preparing samples with different top silicon film functionalization. The measurements were performed after APTES treatment and gold nanoparticles attachment to the surface (process described in [7]). In both cases, the chemical treatment modifies the top surface charge (positive after APTES and negative after gold nanoparticles deposition). Both I<sub>D</sub>-V<sub>G</sub> (Fig. 5) and  $V_B$ - $V_G$  (Fig. 6) curves are shifted to the left after APTES, and to the right after gold nanoparticles deposition as expected, given the sign of the charge added to the surface [7], [8]. While  $I_D$ -V<sub>G</sub> monitoring necessitates a gate voltage high enough to measure a voltage shift or a very weak current difference, for the VB-V<sub>G</sub> curve, the maximum response is obtained for rather low gate voltages. The lower gate bias for body potential reading and the simplicity of the set-up are outstanding advantages for practical applications with respect to current measurements.

#### IV. CONCLUSION

Body-potential measurement in the Ψ-MOSFET configuration is a promising sensing method due to its simplicity, sensitivity and lower bias needed for detection. Research in progress aims at characterizing the surface charge resulting from the deposition of various molecules.

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- S. Williams et al., Mater. Sci. Eng. B, 12, pp. 191-194, 1992. [1]
- D. Munteanu et al., Solid-State Electron, 43, pp. 547-554, 1999. [2] [3] S. Cristoloveanu et al., IEEE Trans. Electron Devices, 47, pp.
- 1018-1027, 2000. G. Hamaide et al. J. Appl. Phys., 101, p. 114513, Jun. 2007.
- [4]
- C. Fernandez et al., EUROSOI-ULIS, pp. 289-292, 2015. [5]
- M. Bawedin et al., Solid-State Electron., 54, pp. 104-114, 2010. [6]
- [7] I. Ionica et al., IEEE-NANO, pp. 38-43, 2011.
- D. Landheer et al., J. Appl. Phys., 98, p. 44701, 2005. [8]



Figure 1. Experimental setup for body-potential measurements in  $\Psi$ -MOSFET configuration.



Figure 2. Drain current, gate current and body potential vs. gate voltage sweep from accumulation to inversion. All curves were measured simultaneously.



Figure 3. Gate current vs. gate voltage for different delay times.



Figure 4. Body potential vs. gate voltage for different delay times.



Figure 5. Drain current vs. gate voltage for different surface treatments.



Figure 6. Body potential vs. gate voltage for different surface treatments.

# Electrical Characterization of Percolating Si-Nanonet FET for sensing applications

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Abstract—Static electrical characterizations on Percolating Si-Nanonet FET based on Si-nanowires were presented. For the electrical parameter extraction, Lambert-function model was used. Dependencies of electrical parameters with length of devices and density of nanowires were studied.

Nanonet; silicon nanowire; Static electrical characterization; Electrical parameter extraction

#### I. INTRODUCTION

Nanonets (NNs) consist in random networks of nanowires (NWs). They can be obtained using a low cost bottom-up approach [1] with no requirement of high resolution lithography techniques. They combine the advantages of NWs in terms of sensitivity to surface charges with the ability to be transferred on any kind of substrates. This makes them perfectly suited to the above-IC integration of chemical, bio or gas sensors, with direct vertical wiring to the CMOS conditioning and readout electronics. Such a compact 3D integration scheme has the potential of providing low-cost, labelfree, multiplex sensors based on the field-effect. The development of this type of devices has been recently pushed by the demonstration that conduction in Si NNs could be promoted by the recrystallization of inter-NW junctions after sintering at a low temperature, compatible with CMOS integration (< 400 °C) [2]. In this paper, we analyze the electrical properties and provide a compact model of such nanonet-based field-effect devices.

#### II. FABRICATION

The NNs used in this study were self-assembled from Si-NWs grown by VLS-CVD. NW length and diameter were 6.9  $\mu$ m  $\pm$  2.8  $\mu$ m and 39 nm. NWs were dispersed with a reference density in a solution and filtered through a porous membrane to form the NN. The density of NWs in the NN was demonstrated to be proportional to the volume filtered. Two different values, d<sub>0</sub> and 1.4×d<sub>0</sub> were used here. NNs were then transferred on a Si/SiO<sub>2</sub> wafer and contacted by Ni/Au metal pads (Fig.1). Field-effect control was obtained by using the Si/(200nm)SiO<sub>2</sub> substrate as a backgate. Several device geometries were available with channel lengths L ranging from 5 $\mu$ m to 100 $\mu$ m.



Figure 1. SEM image of the fabricated device.

#### III. EXPERIMENTAL PROTOCOL AND RESULTS

Drain current (I<sub>d</sub>) was measured as a function of the voltage applied on the backgate  $(V_g)$  with the source contact linked to ground and a constant voltage (V<sub>d</sub>=-4 V) applied to drain (Fig.2). We used a Karl Süss probe station and a HP 4155A semiconductor parameter analyzer. The drain voltage was chosen to put NN FET in the linear regime of operation. These curves were used to extract the main electrical parameters such as low field mobility  $\mu_0$ , subthreshold slope ideality factor n. threshold voltage V<sub>t</sub> and first order mobility attenuation factor  $\theta_1$ . Extraction was performed by fitting the experimental curves with a Lambert W function [3] (Fig.3). Model fitted well with experimental characteristics (Fig.4) for most devices. Measurements were carried out on up to 30 identical devices for each combination of density and geometry in order to get an order of magnitude of statistical variability.



Figure 2. Id=f(Vg) for differents lengths with source linked to the ground and Vd = -4 V for the reference density of nanowires  $d_0$ 

$$Id = \frac{W}{L} \frac{\mu 0}{1+\theta 1 \frac{QL}{Cox}} Qi(Vg) Vd$$
(1)  
$$Qi(Vg) = Cox n \frac{kT}{q} LW\left(e^{q \frac{Vg-Vt}{nkT}}\right)$$
(2)

Figure 3. Lambert W-function (LW) model, with drain current Id, channel width W, channel length L, charge inversion Qi, drain voltage Vd, gate voltage Vg, gate capacitance Cox, Boltzmann constant k, temperature T, electron charge q and  $\mu$ 0, n, Vt and  $\theta$ 1 the electrical parameters to be extracted.





#### IV. DISCUSSION

The values of  $\mu_0$ , n, and V<sub>t</sub> were obtained using contact pad width and source(S)- drain(D) distance as channel width and length, respectively. This provided effective values suitable for compact modeling. Fig.5, Fig.6 and Fig.7 are plotting  $\mu$ 0, n and Vt versus channel length, respectively. A strong variation was observed at short channel length, while plateau values were obtained for large channel lengths. We believe that this change in the variation with channel length can be related to the transition from parallel conduction through several NWs, each linking directly the two contacts, to the percolation regime, where SD conduction paths involve a series of interconnected NWs. The apparent dependence of µ0 with NW density would then be related to the variation of the physical channel width, which is then expected to be proportional to the number of NWs in parallel. The quantitative analysis at long channel length, with a visibly huge variation of  $\mu_0$  with the density, would require much more involved modelling accounting for percolation effects [4].



Figure 5.  $\mu_0 = f(L)$  for differents densities.



Figure 6. n = f(L) for differents densities.



Figure 7. Vt = f(L) for differents densities.

#### V. CONCLUSIONS

Considering Fig.5, a NN FET with low L is better for transistor application, because of high value of  $\mu_0$ . For sensor applications, it can be better to have a large surface, and even for a large L, current being still measurable. Fit of experiment with Lambert-function model is a proof that this model is a promising base for compact model of NN-FET.

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- P. Serre, M. Mongillo, P. Periwal, T. Baron, and C. Ternon, "Percolating silicon nanowire networks with highly reproducible electrical properties," Nanotechnology, vol. 26, no. 1, pp. 1–10, Jan. 2015.
- [2] C. Ternon, P. Serre, J. M. Lebrun, V. Brouzet, M. Legallais, S. David, T. Luciani, C. Pascal, T. Baron, and J. M. Missiaen, "Low Temperature Processing to Form Oxidation Insensitive Electrical Contact at Silicon Nanowire/Nanowire Junctions," *Adv. Electron. Mater.*, vol. 1, no. 10, pp. 1–8, Oct. 2015.
- [3] T.A. Karatsori, C.G. Theodorou, E.G. Ioannidis, S.Haendler, E.Josse, C.A. Dimitriadis and G.Ghibaudo, "Full gate voltage range Lambert-function based methodology for FDSOI MOSFET parameter extraction", Solide-State Electronics, vol.11, pp.123-128, Sept.2015.
- [4] M.-K. Joo, M. Mouis, D.-Y. Jeon, G.-T. Kim, U. J. Kim and G. Ghibaudo, "Static and low frequency noise characterization of N-type random network of carbon nanotubes thin film transistors", Journal of Applied Physics, Vol. 114, no. 154503, Oct. 2013.

### Influence of Free Radical Surface Activation on Si/SiC Heterogeneous Integration by Direct Wafer Bonding

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Abstract— In this study, a surface activated bonding method using remote plasma is applied to realize the direct wafer bonding of Si and SiC. A comparison of different surface treatments is reported. Hydrophilic and hydrophobic wafers have been exposed to *in-situ* argon and nitrogen radicals generated by remote plasma for surface activation before bonding. A comparison of the bonding yield and surface condition has been conducted and analyzed as a function of the surface treatments. It has been shown that N<sub>2</sub> plasma leads to the highest yield of > 97 %, strongest bond of > 360 N and interfacial layer (IL) thickness of ~1.5 nm.

### Keywords-direct wafer bonding; free radical surface activation; heterogenious integration; Si; SiC

#### I. INTRODUCTION

Silicon Carbide (SiC) is a wide bandgap semiconductor material with a thermal conductivity three times that of Silicon (Si). This minimizes the self-heating effects as power is dissipated in this material in Si-on-SiC devices [1]. Furthermore, SiC is a material traditionally used for its mechanical properties; i.e., it can be thinned down to a thickness as low as 50  $\mu$ m and remain freestanding. This is another major boost to the thermal performance of Si-on-SiC devices providing a short path to the heat sink.

The large lattice mismatch between Si and SiC results in a polycrystalline Si film containing a large density of defects and dislocations originated from the material interface [2]. An alternative promising approach to circumvent the problems of epitaxy for hybrid integration is direct wafer bonding (DWB) resulting in defect-free layers at both sides of the bonded pair [3]. DWB is accomplished by activating the surfaces of separated wafers by wet chemistry, direct or remote plasma exposure, or ultraviolet exposure, before physically pressing them together. In this study, we have investigated the use of argon and nitrogen free radicals formed by remote plasma for Si/SiC wafer bonding with attenuated electron and ion concentrations.

#### II. EXPERIMENT

Commercial 4-inch semi-insulating SiC wafers were bonded to 4-inch [100]-oriented Si wafers. Both wafers were cleaned using Standard Cleaning 1 and Standard Cleaning 2 solutions. Wafers were then further cleaned using an EVG wafer cleaning tool equipped with DI megasonic nozzle. For the hydrophobic bonding, the wafers were dipped in diluted HF solution for 60 s prior to the bonding. The wafers were then loaded in a bonder fitted with an *in-situ* remote plasma source. The bonded pairs were then diced into  $1 \times 1$  cm<sup>2</sup> dies and the Si substrate was ground to ~10 µm for TEM analysis.



N<sub>2</sub> plasma

Ar plasma



#### III. RESULTS AND DISCUSSIONS

SiC wafers are transparent in visible range and some voids with interference fringes can be observed at the interface, as shown in Fig. 1. The voided area percentage (Fig.1), number of voids and their distribution should be considered to evaluate the quality of the bonding.

TEM images of the bonded interfaces are shown in Figs. 2(a) and 2(b) for the  $N_2$ /hydrophobic and  $N_2$ /hydrophilic, respectively. All interfaces have an amorphous interfacial layer (IL) between 0.65 nm - 1.6 nm thick.

The average tensile strength of the Si/SiC bonded pairs is obtained by pulling tests, results of which are summarized in Table I. As can be seen, the bond strength varies depending on surface treatment and for the  $N_2$ plasma it is at least equal to bulk Si strength, as the fractures always occurred in the bulk of the Si wafer.



Figure 2. Cross-sectional TEM image of (a)  $N_2$ /hydrophobic, and (b)  $N_2$ /hydrophilic bonded interface.

TABLE I. BOND STRENGTH OF THE SI/SIC BONDED PAIRS.

Plasma/surface	Bond strength (N)		
N <sub>2</sub> /hydrophilic	> 360 (no delamination)		
N <sub>2</sub> /hydrophobic	> 360 (no delamination)		
Ar/hydrophilic	285		
Ar/hydrophobic	118		

All samples were de-bonded in air, near the XPS UHV chamber, resulting in an exposure to air for less than 10 s prior to inserting into the XPS spectrometer. The Si samples displayed broadly similar surface chemistries as shown in the Si 2p overlay in Fig. 3(a). The Si 2p corelevel data shows a primary peak at approximately 99 eV, which is indicative of elemental, non-oxidized Si. A secondary peak present at approximately 103 eV may be attributed to the presence of SiO<sub>2</sub> formed by oxidation of the pristine Si surface. Interestingly, Ar/hydrophobic sample contained the largest amount of SiO<sub>2</sub> with a concentration of approximately 18 at.% while N<sub>2</sub>/ hydrophobic sample contained trace amounts of SiO<sub>2</sub> at ~2 at.%. High-resolution core-level data for the SiC samples is shown in Fig. 3(b). The Si 2p spectrum for Ar/hydrophobic and N<sub>2</sub>/hydrophobic could be fitted by two components: SiC at ~101 eV and SiO<sub>x</sub>C<sub>v</sub>/SiO<sub>2</sub> at ~102 eV. This would indicate the presence of surface contamination and partial oxidation of the SiC surface.

After de-bonding the samples, both SiC and Si dies were analyzed by AFM. Sub-nanometer values of the rms roughness over a scanned area of  $2 \times 2 \mu m^2$  indicate that no significant degradation of the surface is detectable



Figure 3. Si 2p core-level data acquired on (a) Si, and (b) SiC.



after radical exposure. All samples show parallel stripes whose widths match in the SiC and Si scans when debonded from the same die, but vary for different plasma exposures. Even though the difference in height from tops and valleys of the stripes is very small and close to the resolution limit of the equipment, their movement with physical rotations of the scanned dies confirms that they are real and not due to any form of artifact.

#### IV. CONCLUSIONS

Remote plasma surface pre-bonding treatment has been presented as a promising solution to increase the surface chemical reaction for high yield and high bond strength integration of Si/SiC by direct wafer bonding. An amorphous interfacial layer (thickness between 0.65 nm and 1.6 nm) is formed between the two wafers. XPS analysis confirms the presence of oxide on both Si and SiC surfaces.

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- P. M. Gammon *et al.*, "The effect of interfacial charge in the development of silicon-on-silicon-carbide power devices," Proceedings of ECSCRM 2016, Materials Science Forum, In Press (2017).
- [2] L. B. Li, Z. M. Chen, and Y. Zang, "Interface-structure of the Si/SiC heterojunction grown on 6H-SiC," Journal of Applied Physics, vol. 17, 013104, 2015.
- [3] F. Gity et al., "Characterization of germanium/silicon p-n junction fabricated by low temperature direct wafer bonding and layer exfoliation," Appl. Phys. Lett., vol. 100, 092102, 2012.

# Hall-effect Mobility for a Selection of Natural and Synthetic 2D Semiconductor Crystals

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Abstract—We present a DC-AC Hall-effect analysis on transition-metal-dichalcogenides comprising natural crystals of MoS<sub>2</sub> and WSe<sub>2</sub>, and synthetic crystals of HfSe<sub>2</sub>, MoTe<sub>2</sub>, MoSe<sub>2</sub> and Nb-doped MoS<sub>2</sub>. We observe mobility (carrier concentration) values from ~7.6 cm<sup>2</sup>/V.s ( $4.3 \times 10^{19}$  cm<sup>-3</sup>, *p*-type) for a synthetic Nb-doped MoS<sub>2</sub> crystal to 171.3 cm<sup>2</sup>/V.s ( $1.1 \times 10^{17}$  cm<sup>-3</sup>, *p*-type) for a non-intentionally doped natural MoS<sub>2</sub> crystal. All crystals exhibit a net *n*-type or *p*-type doping. Secondary ion mass spectrometry shows higher counts of Nb in the intentionally-doped *p*-type synthetic MoS<sub>2</sub> crystal, in addition to various other high contamination counts in both the natural and synthetic MoS<sub>2</sub>, correlating well with the significant range of resistivity observed.

Keywords-DC Hall; AC Hall; 2D materials; transition-metaldichalcogenides; TMDs; crystals; transition metals; chalcogens; resistivity; mobility; semiconductor; secondary ion mass spectrometry (SIMS)

#### I. INTRODUCTION

One class of material which demonstrates considerable promise as the active semiconductor layer for improved energy efficiency in nanoelectronics, optoelectronics and sensors are transition-metal-dichalcogenides (TMDs), or otherwise known as 2D materials, which have a range of energy gaps that span from semi-metals through to wide band gap semiconductors and as a consequence open up a range of potential applications including those in tunnel FETs, flexible electronics, photovoltaics, and the production of solar fuels using photoelectrochemical cells. While some progress has already been made in fabricating devices, the control and understanding of what influences carrier properties in the 2D material layers remains at an early stage of research and development [1].

#### Experiment

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Eight natural and synthetic 2D material crystals, as shown in Figure 1 (details in [2]), were received from a commercial supplier and mechanically cleaved to obtain different approximate thicknesses for forming Van der Pauw structures with various contact metals (In, Ag, Ni/Au, and Au). Approximate squares are formed with side length (L), where 0.6 cm $\le$ L $\le$ 1 cm, with the deliberate exception of S3 which is measured firstly with an irregular shape (S3a) and again after forming an approximate square shape (S3b) in order to quantify the changing effect on the solution to the Van der Pauw equation (and on the resulting 4-point resistivity), as well as on the Hall carrier properties. Room temperature Hall-effect measurements are performed with a LakeShore Model 8404 Hall-Effect Measurement System with DC and AC magnetic fields of  $\pm 1.7T$  (DC) and  $\sim 1.2T$ RMS (AC, 100mHz), respectively. The Hall system uses all combined optimum methods available to calibrate and remove error components [3]. Hall mobility is extracted from the measured Hall voltage and the measured 4-point resistivity for the particular excitation current and magnetic field applied (Hall factor set to unity). Sheet carrier concentration is extracted likewise from the Hall voltage.



Figure 1: The bulk crystals that sourced **S1-S8** of this work (details in [2]): **S1** MoS<sub>2</sub>, **S2** MoS<sub>2</sub>, **S3** Nb-MoS<sub>2</sub>, **S4** MoS<sub>2</sub>, **S5** MoSe<sub>2</sub>, **S6** MoTe<sub>2</sub>, **S7** WSe<sub>2</sub>, and **S8** HfSe<sub>2</sub>. All sample areas are macroscale for Hall testing.

Secondary Ion Mass Spectrometry (SIMS) chemical analysis is performed on the natural MoS<sub>2</sub> crystal **S1** and on the synthetic crystal **S3** (Nb-doped MoS<sub>2</sub>). Two different primary beams,  $O_2^+$  or Cs<sup>+</sup>, where used with a Cameca IMS-4f instrument while collecting positive or negative secondary ions, respectively, in the whole mass range of 1u-210u. For quantitative analysis, SIMS requires suitable standards that were not available for MoS<sub>2</sub>. However, the technique provides useful information on a relative basis by comparing yields collected in different samples.

#### III. RESULTS AND DISCUSSION

A results summary of the DC-AC Hall-effect analysis on samples **S1-S8** is provided in Table I. The first observation is that there is good agreement in the extracted properties between the DC and AC Hall-effect methods, and since the AC method is highly efficient at removing error components with a lock-in amplifier, these AC Hall properties are more accurate than the DC extractions [3]. It is observed that all Se-based and Te-based samples (electron shells 4 and 5, respectively, that include *d*orbitals) appear *n*-type (**S5-S8**), whereas most S-based samples (electron shell 3 with no *d*-orbitals, similarly configured to oxygen of electron shell 2) appear *p*-type (**S1-S3**), with the exception of the natural *n*-type **S4** crystal with a much lower carrier concentration and a high mobility.

While in general a wide range of Hall mobility, resistivity and carrier concentration values are observed, it is noticeable that the Nb-doped  $MoS_2$  crystal **S3** has a much lower resistivity and a much higher carrier concentration compared to all the other 2D crystals. It is instructive to plot the Hall mobility versus the sheet carrier concentration from Table I, and this is shown in Figure 2(a). We also plot the Hall mobility versus the carrier concentration in Figure 2(b). Apart from the unusually high resistive sample **S1** discussed later, we observe very similar best fit trends to the **S2-S8** data in both plots, confirming that the dominant carriers are distributed homogeneously in the crystals, each with different thicknesses in the ~130µm-765µm range.

TABLE I. HALL-EFFECT PROPERTIES FOR THE S1-S8 CRYSTAL	LS
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Material (ID used)	B Field Type	Carrier Type	Hall Mobility (cm <sup>2</sup> /V.s)	Resistivity (Ω.cm)	Carrier Conc. (cm <sup>-3</sup> )
MoS2 (S1)	DC	Р	4.571	18.66	7.318x10 <sup>16</sup>
MoS2 (S1)	AC	Р	4.442	18.37	7.647x10 <sup>16</sup>
MoS2 (S2)	DC	Р	171.3	0.30	1.056x10 <sup>17</sup>
MoS2 (S2)	AC	Р	170.9	0.30	1.059x10 <sup>17</sup>
Nb-MoS2 (S3a)	DC	Р	6.204	0.014	7.427x10 <sup>19</sup>
Nb-MoS2 (S3a)	AC	Р	6.189	0.014	7.445x10 <sup>19</sup>
Nb-MoS2 (S3b)	DC	Р	7.962	0.019	4.068x10 <sup>19</sup>
Nb-MoS2 (S3b)	AC	Р	7.551	0.019	4.296x10 <sup>19</sup>
MoS2 (S4)	DC	N	124.8	10.71	4.666x10 <sup>15</sup>
MoS2 (S4)	AC	N	113.9	10.69	5.127x10 <sup>15</sup>
MoSe <sub>2</sub> (S5)	DC	N	103.1	11.67	5.187x10 <sup>15</sup>
MoSe2 (S5)	AC	N	105.6	11.72	5.045x10 <sup>15</sup>
MoTe <sup>2</sup> (S6)	DC	N	61.10	0.359	2.844x10 <sup>17</sup>
MoTe <sup>2</sup> (S6)	AC	N	62.09	0.351	2.866x10 <sup>17</sup>
WSe <sub>2</sub> (S7)	DC	N	142.8	9.576	4.565x10 <sup>15</sup>
WSe <sub>2</sub> (S7)	AC	N	130.5	9.672	4.944x10 <sup>15</sup>
HfSe <sub>2</sub> (S8)	DC	N	7.767	0.18	4.471x10 <sup>18</sup>
HfSe <sub>2</sub> (S8)	AC	N	7.422	0.18	4.680x10 <sup>18</sup>

These trends observed in Figure 2 are not overly dissimilar to the drift mobility versus the *n*-type and *p*-type impurity concentrations for Si and GaAs [4]. Importantly,  $MoS_2$  is observed to potentially exhibit a net *p*-type behavior with a hole mobility as high as ~171 cm<sup>2</sup>/V-s.



Figure 2: DC-AC Hall mobility versus (a) sheet carrier concentration and (b) carrier concentration. Best non-linear curve fit lines to crystals **S2-S8** (**S1** is excluded from the best fit exercise as it is abnormally too resistive). Note that the y-axis of plots (a) and (b) have the same Hall mobility scale.

Figure 3 reports the SIMS yields of the detected impurities in samples **S1** and **S3**. A large range of (unintentional) impurities are present in both the natural and synthetic  $MoS_2$ , consistent with reports via inductively coupled plasma mass spectrometry analysis [5], and likely limiting the achievable mobility as expected by theory [6].



Figure 3: SIMS raw yields of impurities in natural  $MoS_2$  (S1) and in synthetically-grown Nb-doped  $MoS_2$  (S3). H, C, O, F and Sb were collected as negative ions while sputtering with a  $Cs^+$  beam. Na, Cr and Nb were collected as positive ions while sputtering with a  $O_2^+$  beam. No direct same-sample yield comparisons are given, only between different samples.

Niobium can clearly be seen in the intentionally-doped synthetically-grown S3 sample with a yield ~450 higher than in the natural S1 sample, which is in good agreement with the measured *p*-type carrier concentration levels for S3, even if we cannot exclude that the detected Sb is not contributing to the observed *p*-type doping in the natural S1 sample. Na and Cr are present in the synthetically-grown

sample but, as those elements are expected with high sensitivity, their concentrations are presumed moderate. H, C, and O are observed in the natural S1 sample with significantly higher yields than in the synthetically-grown S3 sample, which we believe are remarkably high and leads to significant increases in the yields of other secondary molecular ions (not shown) formed with such species (e.g. MoC<sup>-</sup>, MgO<sup>-</sup>, etc.). We speculate that the presence of such impurities at anomalously high concentrations may contribute to the much larger resistance observed in S1. We also observe a possible correlation between the *p*-type behavior of S1-S3 from Table I, the very high oxygen content of S1 from Figure 3, and the known preference for higher oxidized Molybdenum (in a tri-oxide form) to give *p*-type behavior, as observed in solar cell research and in sub-stoichiometric form for MoO<sub>x</sub>-MoS<sub>2</sub> MOSFETs [7]. This may suggest a possible link between oxygen substitution of sulfur (especially given the similar electron configurations of these two elements noted earlier) with or without structural change and a more dominant *p*-type behavior, in contrast to the dominant n-type behavior we see for the Selenium and Tellurium based 2D crystals (S5-**S8**) that have the additional *d*-orbitals in these elements.

#### IV. CONCLUSIONS

We present a DC-AC Hall-effect analysis on transitionmetal-dichalcogenide 2D materials comprising natural crystals of MoS<sub>2</sub> and WSe<sub>2</sub>, and synthetic crystals of HfSe<sub>2</sub>, MoTe<sub>2</sub>, MoSe<sub>2</sub> and Nb-doped MoS<sub>2</sub>, which have potential applications in tunnel FETs, nanoelectronics, optical and sensing devices. We observe Hall mobility (carrier concentration) values from ~7.6 cm<sup>2</sup>/V.s ( $4.3 \times 10^{19}$  cm<sup>-3</sup>. p-type) for a synthetic Nb-doped MoS<sub>2</sub> crystal to 171.3 cm<sup>2</sup>/V.s (1.1 × 10<sup>17</sup> cm<sup>-3</sup>, *p*-type) for a non-intentionally doped natural MoS<sub>2</sub> crystal. The relatively high hole mobility observed in MoS<sub>2</sub> (~171 cm<sup>2</sup>/V-s) is of particular note, as it points to the possibility of MoS<sub>2</sub> as a single 2D material solution for CMOS based circuits. Secondary ion mass spectrometry shows higher counts of Nb for an intentionally-doped *p*-type synthetic MoS<sub>2</sub> crystal, in addition to various other high contamination counts in both the natural and synthetic MoS<sub>2</sub>, correlating well with the significant range of resistivity and suggests that higher mobility values may be achievable in 2D semiconductors with higher material purity levels. Finally, these results also suggest that intentionally doping 2D materials is feasible and can have a significant effect on the electrical properties.

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References

- Z. Lin *et al.*, 2D Mater. 3, 042001 (2016)
   S1 (t~615um): Natural MoS<sub>2</sub> (CAS: 1317-33-5, Source A) supplied by HQ Graphene, Groningen, the Netherlands (HQG); S2 (t~130um): Natural MoS<sub>2</sub> (CAS: 1317-33-5; Source B, HQG); S3 (t~190um): Synthetically-grown Niobium (Nb) doped MoS<sub>2</sub> (CAS: 1317-33-5, HQG); S4 (t~450um): Natural MoS<sub>2</sub> (CAS: 1317-33-5, HQG); S5 (t~210um): Synthetic MoSe<sub>2</sub> (CAS: 12058-18-3, HQG); S6 (t~425um): Synthetic MoTe<sub>2</sub> (CAS: 12058-20-7, HQG); S7 (t~765um): Natural WSe<sub>2</sub> (CAS: 12067-46-8, HQG); and S8 (t~405um): Synthetic HfSe<sub>2</sub> (CAS: 12162-21-9, HQG)
- [3] J. Lindemuth and S. -I. Mizuta, Thin Film Solar Technology III, Proc. of SPIE 8110, 81100I (2011)
- [4] S. M. Sze, K. K. Ng, Physics of Semiconductor Devices, 3<sup>rd</sup> Ed. (2007), John Wiley & Sons Inc., New Jersey, USA (p.29)
- [5] R. Addou *et al.*, *ACS Nano* **9** (9), 9124 (2015)
- [6] N. Ma, D. Jena, Phys. Rev. X 4, 011043 (2014)
- [7] S. Chuang et al., ACS Nano Lett. 14 (3), 1337 (2014)