

A Statistical Approach to Characterizing and Testing Functionalized Nanowires

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Abstract

Unlike the top-down photolithographic CMOS VLSI process, cost-effective bulk fabrication of nanodevices calls for a bottom-up approach, generally called self-assembly. Self-assembly, however, inherently lends itself to innate disparities in the structure of nominally identical nanodevices and, consequently, wide inter-device variance in their functionality. As a result, nanodevice characterization and testing calls for a slow and tedious procedure involving a large number of measurements. In this work, we discuss a statistical approach which learns measurement correlations from a small set of fully characterized nanodevices and utilizes the extracted knowledge to simplify the process for the rest of the nanodevices. More specifically, we employ various machine-learning methods which rely on a small subset of measurements to i) predict the performances of a fabricated nanodevice, ii) decide whether a nanodevice passes or fails a given set of specifications, and iii) bin a nanodevice with regards to several sets of increasingly strict specifications. The proposed methods are demonstrated and their effectiveness is assessed, within the context of nanowire-based chemical sensing, using a set of fabricated and fully characterized nanowires.

1. Introduction

Nanotechnology promises a broad range of new capabilities and a revolution in the way that atom or molecule-scale science and engineering will impact various aspects of our lives. These promises have sparked tremendous research interest in this area, with the majority of efforts directed in the physical domain of developing and characterizing a single or a handful of nanodevices. Yet, proliferation of nanoscale systems capable of performing computationally interesting tasks will eventually require general, robust, and well-understood structural and behavioral models of these devices. A key challenge in this quest stems from the uncertainty and randomness associated with the batch fabrication of nanodevices. Indeed, with feature dimensions between a fraction of a nanometer and a few nanometers, nanodevice fabrication and placement is to date not controllable to the precision we are accustomed to in a CMOS VLSI process. Unlike the top-down photolithographic

approach, cost-effective bulk fabrication of nanodevices calls for a bottom-up approach, generally called self-assembly. The latter, however, inherently lends itself to innate disparities in the structure of nominally identical nanodevices and, consequently, wide inter-device variance in their functionality. Thus, each nanodevice has to be individually characterized through a laborious process that involves a large number of measurements and performance computations.

In this paper, we aim to expedite and simplify the process of nanodevice characterization through the use of statistical analysis. Specifically, we exploit the ability of machine learning methods to examine a representative set of characterized nanodevices and identify correlations between the various measurements that are typically used for such characterization. Assuming that the nanofabrication process yields nanodevices whose performances belong to a distribution, the extracted knowledge can then be leveraged to reduce the number of required measurements and, thus, simplify the characterization procedure for the rest of the nanodevices.

The effectiveness of the various statistical methods employed in this study is assessed on a set of nanowires that have been fabricated and fully characterized within the context of a nanowire-based chemical sensing application. The objective of this application is to use nanowires that are functionalized as chemical sensors, in order to distinguish among different trigger elements. Such chemical sensing results in a shifting of the I-V curve of the nanowire, the magnitude of which depends on the sensed element. Interestingly, both the baseline I-V curve and the shifted versions in the presence of a target chemical vary widely even for nominally identical functionalized nanowires. Therefore, each fabricated nanowire has to be characterized with respect to a given set of specifications that it needs to comply to, in order to achieve the aforementioned objective. To simplify this process, the statistical methods proposed herein focus on the following three tasks:

- **Performance Estimation:** We examine the effectiveness of using regression models to predict the performances of a nanowire from a small subset of the measurements that are typically obtained for characterization. As we demonstrate experimentally, the average prediction error is very small and remains within a small zone around the actual performance value, while the number of required measurements is drastically reduced.

- **Pass/fail Testing:** We examine the effectiveness of using the predicted performances to decide whether a nanowire passes or fails a given set of specifications. We also examine the effectiveness of various classifiers, including a Support Vector Machine (SVM) [1] and an Ontogenic Artificial Neural Network (ONN) [2], to directly predict the pass/fail label of a nanowire. As we demonstrate experimentally, classification accuracy of over 90% can be achieved with a very small subset of measurements. Moreover, a guardbanding method can further boost this accuracy at the expense of obtaining the complete set of measurements on a small fraction of the devices.
- **Binning:** We examine the effectiveness of using the predicted performances to bin a nanowire with regards to four sets of increasingly strict specifications. We also examine the effectiveness of a multi-class classifier to achieve the same. As we demonstrate experimentally, binning accuracy of over 80% and an average incorrect binning distance of 1.2 can be achieved with a very small subset of measurements.

The rest of the paper is organized as follows. In section 2, we provide an overview of the process through which the nanowires employed in this study are fabricated and functionalized. In section 3, we describe the target chemical sensing application and we review the specifications that the performances of the fabricated nanowires should satisfy, as well as the process through which these performances are currently computed. Then, in section 4, we assess the effectiveness of the proposed statistical methods in predicting these performances from a small number of measurements, as well as the effectiveness of using the predicted performances for testing and binning the nanowires. We should note that the proposed statistical methods are motivated by and resemble the specification test compaction and alternate test methods that have become popular in analog/RF circuits [3], [4], [5].

2. Nanowires as FETs and Sensors

We start by reviewing the general procedure through which the nanowires employed in this study are fabricated, integrated, and characterized to date. We also briefly discuss the ability of these nanowires to be functionalized and act as chemical receptors in order to support the targeted sensing application.

2.1. Fabrication

The electronic and optoelectronic properties of semiconducting inorganic nanowires is the subject of intense contemporary interest. Semiconducting crystals grown by highly anisotropic, unidirectional methods have been known since the pioneering works of Wagner et. al [6] and Hiruma et. al [7], and are a subject of concentrated research [8], [9], [10], [11], [12] because they represent the limit of semiconductor crystalline solids. These structures, termed nanowires (NWs), are typically single-crystal, solid cylindrical structures nanometers in diameter and

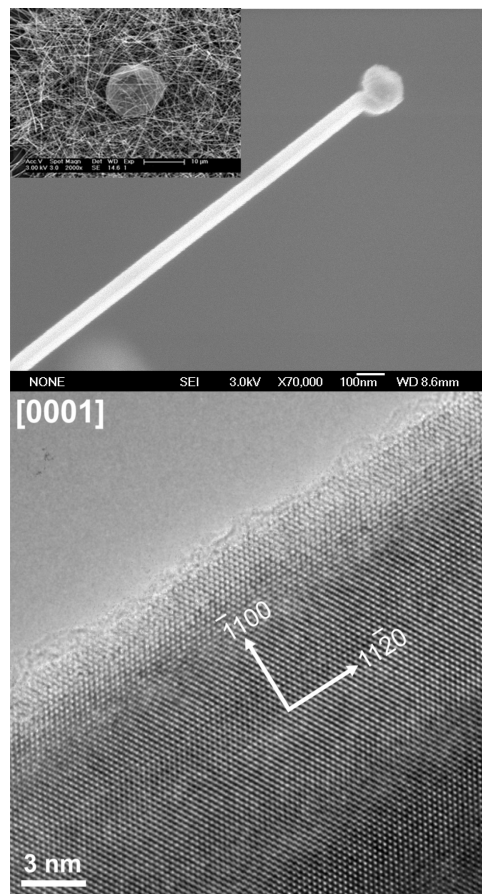


Fig. 1. TEMs of GaN nanowires grown in Reed's laboratory: (a) The top illustrates the nucleating catalyst at the end of the nanowire. The inset shows a cluster of NWs on the growth substrate at lower magnification (the ball in the center is a Ga pellet). (b) The bottom illustrates that the nanowires are grown in the $11\bar{2}0$ direction and are single-crystal hexagonal wurtzite.

microns in length and can be synthesized from a vast array of traditional semiconducting materials (such as Si, GaN, ZnO, and others). Over the last few years a wide variety of devices have been demonstrated, such as diodes, FETs (and circuits), LEDs, chemical and biological detectors, and even quantum effect devices such as resonant tunneling diodes. Along with carbon nanotubes, these structures are considered potential candidates for post-CMOS electronic devices.

However, a number of limitations must be overcome before these nanowires can be implemented in real applications. Typically, these structures (both nanowires and carbon nanotubes) are synthesized by a bottom-up approach utilizing a catalyst in a growth system such as an oven. An example of this process is shown in Fig. 1. They are then transferred onto planar systems, where they are ultimately integrated with electronic fanouts. Thus, the integration challenge is severe, as the nanowires must be laboriously manipulated and serially fabricated. Labor

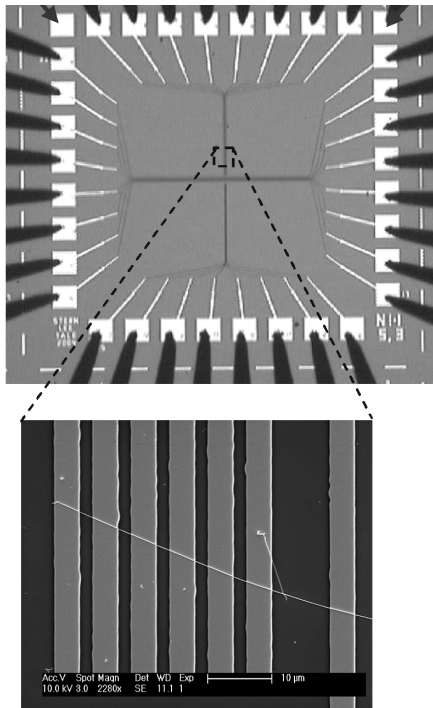


Fig. 2. Optical micrograph of dies used for nanowire device fabrication (top). Probe tips are visible touching the contact pads. A representative magnified device is shown in the SEM (bottom), in this case illustrating a multipoint device to characterize contact resistance.

intensive custom lithography is often used since there exist no suitable high-throughput NW-to-individually-addressable-interconnect alignment techniques. Consequently, this serial method is impractical for obtaining useful integration.

A more practical method is the use of a parallel technique such as optical lithography to define these devices. To this end, Reed's Group at Yale University has recently developed a high throughput capability to make and study a large number of devices, which is crucial to understanding nanowire material and device properties enough to successfully integrate into circuits. This method, the details of which can be found in [13], was the first to report a statistically significant sample size of nanowires, sufficient for extracting correlations with material synthesis and device fabrication parameters, or for quantifying interdevice behavioral fluctuations. The developed process combines controlled dispersion of nanowires with appropriate metallization to yield dies with individually addressable nanowire FETs. Specifically, the dispersed nanowires form contacts between adjacent pairs of metal lines, which can then be successively probed to identify whether a nanowire has, indeed, established contact. In this case, the metal access points can be used to examine the transfer characteristics of the nanowire and decide whether it is a functional device. With 150 dies per 4" wafer, a large number of nanowire devices per wafer can be created. As an example, Fig. 2 shows a nanowire establishing contact among several adjacent metal lines.

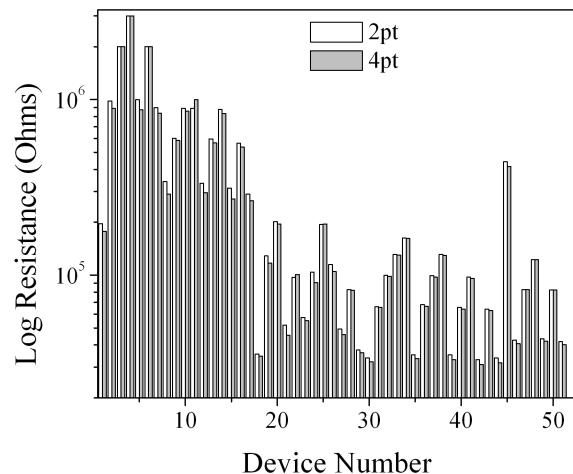


Fig. 3. Channel resistance (and a comparison of 2-point versus 4-point) for various devices fabricated from nominally identical nanowires.

With the integration issue for accessing and characterizing individual nanowires minimized, another limiting problem of nanowire devices now becomes obvious. Characterization of a full wafer containing about 50 devices that are 4-pt or greater (i.e. they establish contact between at least 4 consecutive metal lines) yields Fig. 3. This bar-chart illustrates a major problem with nanodevices, namely *pronounced inter-device fluctuations*. The multi-point measurements reveal an important insight - the contact and device variables fluctuate little, leaving intrinsic material properties as the main source of fluctuation. The resistivity measurements shown are equally reflected in the mobility and carrier density measurements, as well as the resulting transconductances and circuit performance.

These measurements illustrate the inter-device fluctuations inherent in nanoscale material synthesis, which will certainly inhibit system-level integration. And as device dimensions scale, both material properties and device fabrication variations will give rise to even more pronounced innate device-level fluctuations. It is, therefore, foreseeable that extensive device characterization and individual tuning of nanoscale systems will be required. In this sense, methods for reducing the burden of nanodevice characterization and testing are expected to play a key role in widespread deployment of nanoscale systems.

2.2. Functionalization

It has been known for some time that one can potentially achieve unprecedented sensitivity in ChemFET-like sensors by utilizing quasi-1D channels, since the "bulk" channel conductivity is essentially modulated by the surface chemistry of the device. Semiconducting nanowires have been shown to be chemical sensors of excellent sensitivity. The top of Fig. 4 shows a schematic of this type of sensors in an FET configuration. Note that the chemical potential and carrier density of the wire is set by the gate that lies under the nanowire, to maximize the available surface (although Reed's

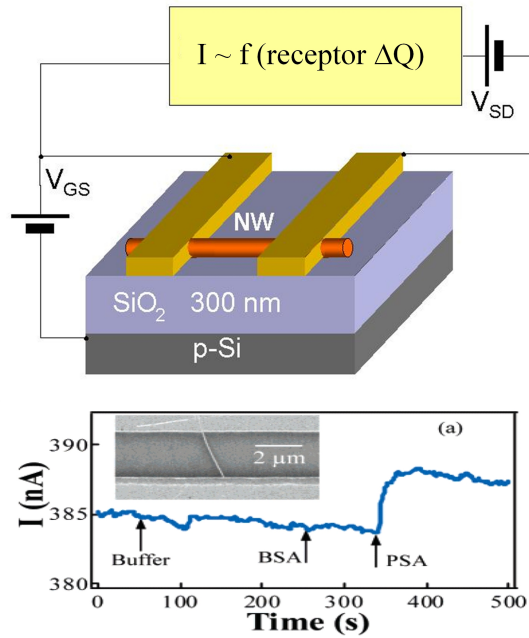


Fig. 4. Schematic of a nanowire chemical/biological sensor in an FET configuration (drawing courtesy of M. Moskovits) (top). Response of n-type In_2O_3 semi-conducting nanowire (from [14]) (bottom).

lab has also demonstrated identical FET performance by a top gate). The bottom curve shows the response of suitably functionalized nanowires (n-type semiconductor (In_2O_3)) to the effector PSA (Prostate Specific Antigen), demonstrating selective response of the nanowires' conductivity (note the control BSA (Bovine Serum Albumin) is similar to PSA, but shows no response).

3. Target Application: Chemical Sensing

The end application for the nanowires employed in this study is the ability to sense and distinguish between different chemicals. In other words, the objective is to be able to functionalize a nanowire such that it reacts distinctly with different trigger elements. As shown in Fig. 5, the nanowire is essentially configured as a three terminal FET device. For a given bias voltage between its gate and source, V_G , when the nanowire senses a trigger chemical, the I_{SD} - V_{SD} curve between the source and the drain of the FET is shifted, as implied by the bottom curve of Fig. 4. Assuming that this shift is different for each targeted chemical, one may use the I_{SD} - V_{SD} curve to identify the absorbate. While selective nanowire functionalization for different trigger elements has yet to be done, it has been shown that different chemicals indeed result in different shifts in this curve. Furthermore, it has been shown that the impact of such chemical sensing is equivalent to varying V_G . In other words, the I_{SD} - V_{SD} curve shift instilled

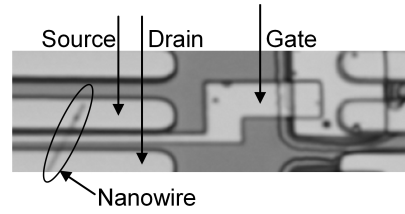


Fig. 5. Nanowire configured as three-terminal FET.

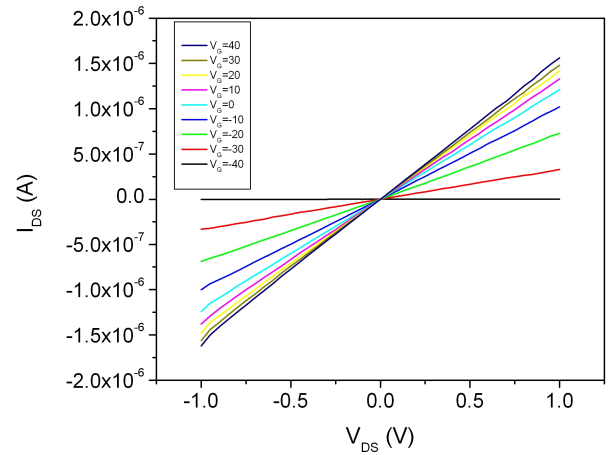


Fig. 6. I_{SD} - V_{SD} plot for varied V_G . Each curve emulates the nanowire response in the presence of a different chemical.

by a chemical trigger can be mimicked by changing the bias voltage between the gate and source of the nanowire FET. Therefore, by stepping V_G through 17 distinct voltage values and plotting the corresponding 17 I_{SD} - V_{SD} curves, we can emulate the response of the nanowire in the absence of a trigger and in the presence of each of 16 targeted chemicals. An example of such measurements is shown in Fig. 6, where V_G is varied from -40V to 40V at a step of 5V. For the purpose of not cluttering the figure, only 9 of the 17 plots (at a step of 10V) are shown.

3.1. Nanowire Characterization

Essentially, each of these I_{SD} - V_{SD} curves corresponds to the response of a nanowire when it senses a target chemical. However, for a nanowire to be effective as a chemical sensor, these responses need to exhibit performances that abide by certain specifications. In order to characterize each nanowire, a tedious process involving a large number of measurements is currently employed. Specifically, for each of the 17 V_G values, the voltage V_{SD} between the source and the drain of the three-terminal nanowire FET is swept between -1V and 1V and 41 measurements of the current I_{SD} are taken to plot the corresponding I_{SD} - V_{SD} curve. In total, 697 current measurements are performed on each device. Then a least-

squares slope value fit to each curve and the correlation of the actual measurements to this slope are computed. From these 34 slopes and correlations (hereafter referred to as the ‘measurements’), the following 5 performances are computed:

- 1) **Min-slope**: The smallest fitted slope.
- 2) **Max-slope**: The largest fitted slope.
- 3) **Span**: The difference between the Min-slope and Max-slope.
- 4) **Linearity**: The smallest correlation value of the fitted slopes.
- 5) **Distribution**: A measure of how evenly distributed the slope values are throughout the span. The distribution value is computed by first normalizing each of the 17 slopes to the span, i.e. expressing them as percentages of the span such that the Min-slope value is at 0% and the Max-slope value is at 100%. Then, a least-squares slope value fit is applied to these normalized slopes and its correlation value is calculated and reported as the distribution.

These performances are subsequently compared to 6 specifications to decide whether the nanowire can be used effectively in the target application (note that *Span* has a double-sided specification while the rest of the performances have single-sided specifications): *Span-low*, *Span-high*, *Min-slope-low*, *Max-slope-high*, *Linearity-low*, and *Distribution-low*.

4. Statistical Characterization & Testing

While the aforementioned process yields the nanowire performances and can be used for characterization, pass/fail testing, and binning of a nanowire, it is slow and tedious due to the large number of measurements involved. To alleviate this problem, we employ statistical methods that rely on a small subset of the 34 measurements (and, by extension, the 697 current measurements) to predict the performances, to test, and to bin each nanowire. We point out that, as the functionality of nanowires becomes more elaborate and as their complexity increases, the number of measurements needed for characterization will also increase. Thus, statistical approaches, such as the ones described herein, can prove particularly handy.

4.1. Experimental Setup

The described methods are assessed on a set of 842 nanowires that were fabricated and fully characterized in Reed’s laboratory through the process described in section 3.1. The nanowires are split uniformly at random into three sets. The first set is called the *training set* and consists of half of the devices. This set is used for building the statistical models for regression and classification. The second set is called the *hold-out set* and consists of one quarter of the devices. This set is used as an independent set during the selection of measurements subsets (feature selection). The third set is called the *validation set* and consists of the remaining one quarter

of the devices. This set is used as a final independent set on which the statistical model built from the selected subset of measurements is assessed.

4.2. Performance Prediction

We first focus on the task of predicting the 5 performances of a nanowire using only a subset of the 34 measurements. For this purpose, we employ regression using MARS [15]. More specifically, we use these 34 measurements from the nanowires in the training set to build regression models for each of the 5 performances. These models are then applied to predict the performances of the devices in the validation set and to compare them to the actual performances in order to compute the prediction error. The latter varies greatly depending on the cardinality of the subset of measurements that is used to build the regression models, as well as the actual measurements that this subset includes. Naturally, our objective is to minimize the error, but also to keep the cardinality of the required measurement subset as low as possible to expedite the nanowire characterization process.

For this purpose, we employ a simple greedy algorithm to search in the powerset of measurements for appropriate subsets. Starting with all 34 measurements, the algorithm builds regression models and assesses them on the hold out set to compute the prediction error. Then, each of the 34 measurements is excluded, in turn, and 34 regression models, each with 33 measurements, are built and assessed on the hold out set. Then, the measurement whose exclusion results in the least increase in prediction error is permanently eliminated and the process is repeated until only one measurement is left. In the end, the algorithm reports the prediction error achieved on the hold-out set by each of the subsets of cardinality 1 through 34. Thus, given a targeted minimum acceptable prediction error, the subset of minimal cardinality that achieves this target on the hold-out set is selected. Finally, the regression models that are built in the training set using the selected measurement subset are applied to predict the performances of the nanowires in the validation set and the average prediction error is reported as the figure of merit.

In our study, we set the threshold for the acceptable error between an actual performance and its prediction to 3σ , where σ is the standard deviation of this performance over all the characterized nanowires. In other words, we pick the minimal cardinality subset of measurements that yields predictions of error smaller than $\pm 3\sigma$ in the hold-out set. The cardinality of this set is 4 and the results of applying the corresponding regression models to the validation set are shown in Fig. 7, where we plot the actual vs. the predicted value for each of the 5 performances for all the nanowires in the validation set. In each of the 5 plots, the middle line represents the ideal case of zero prediction error, while the upper and lower lines represent the $\pm 3\sigma$ zone. As can be seen, for the vast majority of the devices in the validation set, the prediction error remains well within the $\pm 3\sigma$ zone for all 5 performances, despite the fact that only 4 out of the 34 measurements are used.

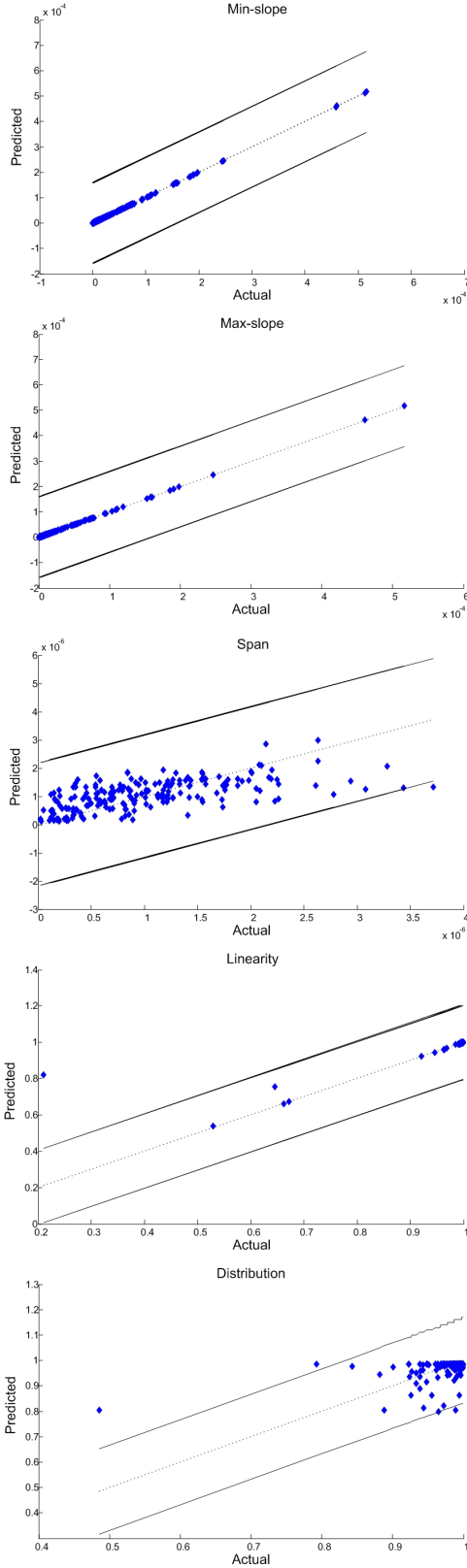


Fig. 7. Actual vs. predicted performances. Regression error remains for most devices within a $\pm 3\sigma$ band.

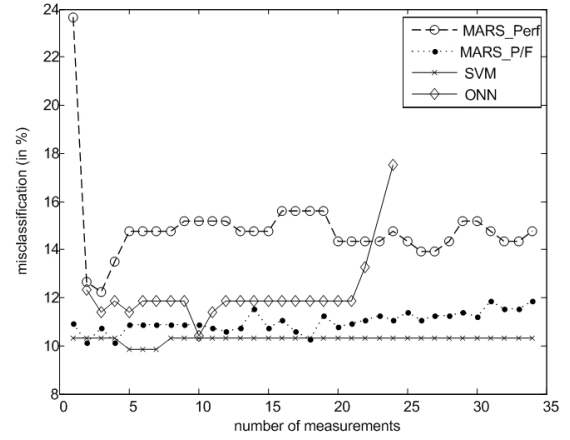


Fig. 8. Comparison of pass/fail prediction methods.

4.3. Pass/Fail Testing

The next task that we focus on is predicting whether the performances of a nanowire pass or fail a given set of specifications, as described in section 3.1. The simplest way of achieving this is to compare the predicted performances that are calculated from the MARS models to the given specifications. We refer to this two-step approach, which first learns the mapping of measurements to performances and then uses this mapping to decide on a pass/fail label, as “classification through regression”. Once again, a greedy algorithm is employed to report the subset of measurements of cardinality 1 through 34 that yields the minimal misclassification error for the nanowires of the hold-out set. These measurement subsets are subsequently applied to the nanowires of the validation set and the MARS_Perf. plot of Fig. 8 is derived. As may be observed, this method performs respectably, as it achieves over 85% classification accuracy with only 3 measurements.

Alternatively, one may also attempt to learn directly the mapping between the measurements and the pass/fail label. In our study, we employ three such direct classification methods; a binary regression, a support vector machine (SVM) [1], and an ontogenic artificial neural network (ONN)[2]. The first method still relies on MARS models, only this time the range of these functions is $\{0,1\}$, signifying failing or passing nanowires, respectively. Predicted values under 0.5 are rounded down to 0, while predicted values over 0.5 are rounded up to 1, which causes MARS to act like a classifier. Combining the greedy selection algorithm with this classifier yields the results shown in the MARS_P/F plot of Fig. 8. In the second and third methods, the SVM and the ONN attempt to learn the boundary that separates the passing from the failing nanowire populations in the hyper-dimensional space of the available measurements. Hence, they directly classify a nanowire as passing or failing by simply comparing the footprint of its measurement pattern on the hyper-dimensional space to the location of the separation boundary. The SVM employs an internal feature selection algorithm which relies on Principal

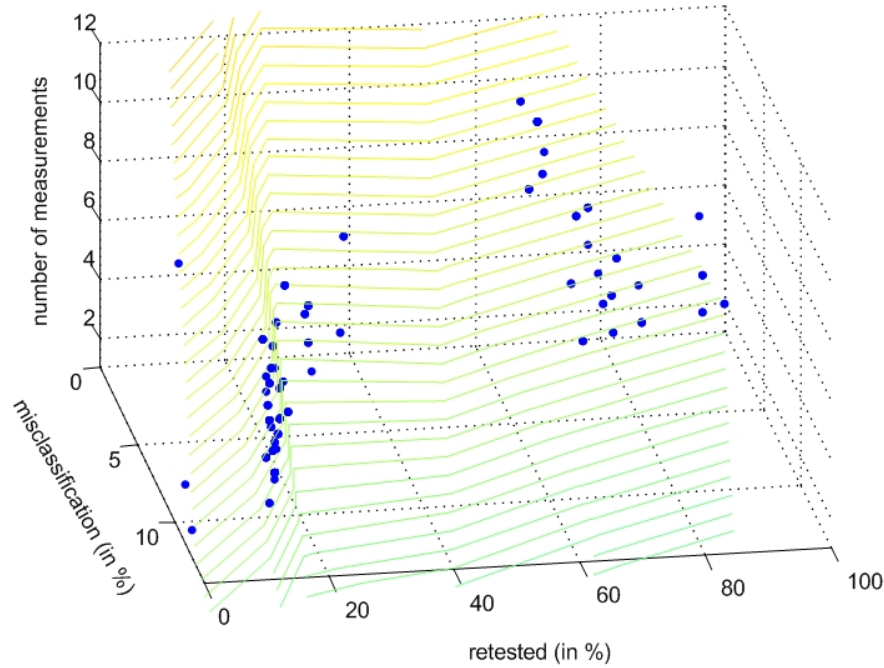


Fig. 9. Trade-off between misprediction error, number of retested nanowires, and number of measurements.

Component Analysis (PCA) to identify the most relevant measurements. Hence, combining it with greedy measurement selection incurs a minimal impact on its effectiveness, as shown in the SVM plot of Fig. 8. In contrast, the ONN is paired with a multi-objective Genetic Algorithm (GA) [16], which searches in the powerset of measurements. In the ONN plot of Fig. 8¹, we report the minimum misclassification error observed for a given subset cardinality.

The results show that all three methods perform similarly, with the MARS-P/F and the SVM achieving just under 90% classification accuracy with 2-3 measurements. With 5 measurements, the SVM performs at accuracy levels of over 90%. For this experiment, the ONN performs slightly worse, with 3 measurements garnering just below 89% accuracy, while with 10 measurements, it reaches an accuracy level of almost 90%. One more noticeable point is that all three direct classification methods outperform the classification through regression approach. This is expected, since the latter tunes the learned mappings to minimize the average performance prediction error, which does not necessarily lead to the optimal mappings for minimizing the pass/fail classification error.

While the pass/fail prediction accuracy using a very small number of measurements exceeds 90%, it may still be inadequate for the target application. In this case, a guardbanding methodology may be employed to identify the nanowires for which the pass/fail decision is prone to error. These nanowires can then be examined through a second test stage, where all the measurements used for nanowire characterization are

obtained, so that the nanowire can be definitively tested. By varying the width of the guardbands, we can add a third dimension in the trade-off exploration, namely the number of retested nanowires, along with the prediction error and the number of measurements. In this study, we use a guardbanding methodology that has been previously developed in conjunction with the ONN [4]. The results are demonstrated in the 3-D plot of Fig. 9. One can see, for example, that the genetic algorithm identifies a set of 5 measurements which, when used to train the ONN with the guardbands, result in an misprediction error of 2.84% by retesting 26.07% of the nanowires.

4.4. Binning

In addition to performance prediction and pass/fail testing, one may also wish to bin the nanowires in relation to increasing levels of strictness in the specifications. To that end, we first assume four different sets of specifications, each one stricter than the last. Then, we define the bins from one to five where nanowires in the first bin can pass the strictest specification set, nanowires in the second bin can pass all but the strictest specification set, and so on until the fifth bin contains nanowires that categorically fail. At this point, statistical methods can be used to predict into which bin a device will fall. Herein, we perform statistical binning in two ways. As a first option, we utilize the predicted performances from our previous regression experiment along with the four sets of specifications in order to assign each nanowire to a bin. As a second option, we employ the aforementioned ontogenic neural network, configured as a multi-class classifier, to attempt prediction of the bins directly.

A comparison between the results obtained from the neural network and those obtained by calculating from the predicted performances is provided in Fig. 10. As can be seen, the bins

¹We note that the GA employed for feature selection quickly figures out that the classification accuracy does not improve by using large measurement sets. Hence, it steers away from large sets and, therefore, data is only available for cardinality 2 to 24, skipping 23.

calculated from the predicted performances yield only about 75% accuracy even when using as many as 19 out of the 34 measurements. Additionally, in cases where the predicted bin is incorrect, it is on average over two bins away from the correct case (2.67 bins on average for the 19-measurement case). In contrast, the multi-class ontogenic neural network is able to perform at over 80% accuracy with only three measurements. Furthermore, of the misclassified devices, most are classified only one bin away from the correct case (1.261 bins on average in the 3-measurement case). These results corroborate the observation made in the previous section regarding the impact of the additional step required in classifying through regression.

5. Conclusion

As we reach the point where nanodevices can be fabricated and integrated in bulk, a pressing need to expand the nanotechnology research agenda is surfacing. Indeed, the leap from understanding nanowires at the device level to creating nanoscale architectures and functional nanoscale systems hinges upon the ability to effectively model, characterize and test nanodevices. Given the observed innate fluctuation in the structure and functionality of nanodevices, these tasks require a tedious and laborious effort. Towards alleviating this problem, we have demonstrated that statistical methods can effectively simplify this process. Specifically, we have shown the ability of machine learning methods to accurately predict nanodevice performances, classify nanodevices as good or bad, and bin nanodevices with respect to distinct sets of specifications. Furthermore, we have concluded that all of the above may be achieved with a drastically smaller set of measurements than what is currently employed. Our conjectures have been corroborated experimentally using data from fabricated nanowires, boosting our confidence that these results will translate to an equivalent decrease in characterization and test time and will help push nanotechnology research to the next level.

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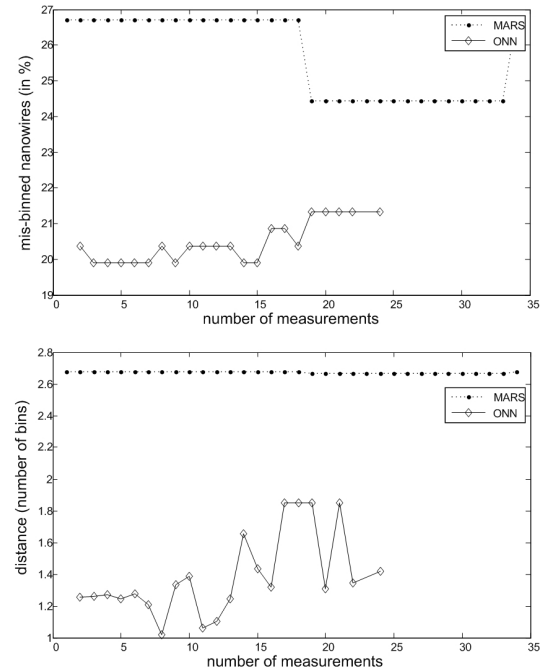


Fig. 10. Comparison of binning methods.