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1 Statistical Reproducibility of Selective Area Grown InAs Nanowire Devices

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11 **Abstract:** New approaches such as selective area growth (SAG), where crystal growth is litho-
 12 graphically controlled, allow the integration of bottom-up grown semiconductor nanomaterials in
 13 large-scale classical and quantum nanoelectronics. This calls for assessment and optimization of the
 14 reproducibility between individual components. We quantify the structural and electronic statistical
 15 reproducibility within large arrays of nominally identical selective area growth InAs nanowires. The
 16 distribution of structural parameters is acquired through comprehensive atomic force microscopy
 17 studies and transmission electron microscopy. These are compared to the statistical distributions of
 18 the cryogenic electrical properties of 256 individual SAG nanowire field effect transistors addressed
 19 using cryogenic multiplexer circuits. Correlating measurements between successive thermal cycles
 20 allows distinguishing between the contributions of surface impurity scattering and fixed structural
 21 properties to device reproducibility. The results confirm the potential of SAG nanomaterials, and
 22 the methodologies for quantifying statistical metrics are essential for further optimization of repro-
 23 ducibility.

24 **Keywords:** nanowires, selective area growth, semiconductors, multiplexers, reproducibility

25 The characteristics of nanoscale electrical devices can
 26 be significantly influenced by rearrangements of only a
 27 few atoms in the vicinity of the active transport channel,
 28 especially at low temperatures.^{1,2} Material purity and
 29 device reproducibility are thus key concerns in the devel-
 30 opment of increasingly complex and powerful electrical
 31 quantum circuits operating at ultra-low temperatures.³
 32 While bottom-up semiconductor nanowires have been an
 33 important platform for mesoscopic quantum electron-
 34 ics due to the high control of crystal properties,⁴ in-
 35 trinsic quantum confinement,⁵ and flexible epitaxial in-
 36 tegration of dissimilar materials,^{6,7} the traditional out-
 37 of-plane geometry is incompatible with standard semi-
 38 conductor processing and has prevented up-scaling of
 39 nanowire circuits. The method of SAG^{8–11} potentially re-
 40 moves this roadblock by allowing large-scale lithographic
 41 control of planar nanowire growth. Already, key device
 42 concepts based on SAG nanowires have been realized,
 43 such as field effect transistors (FETs),^{12,13} nanowire Hall
 44 bars,^{14,15} quantum interferometers,^{16,17} hybrid supercon-
 45 ducting devices,^{16,18,19} and quantum dots.²⁰ So far, how-
 46 ever, the focus has been on proof-of-principle experiments
 47 based on a few devices. We here take the next step
 48 towards up-scaling by quantifying the statistical repro-
 49 ducibility of SAG devices operated at the deep cryogenic
 50 conditions relevant for quantum circuitry.

51 Statistical assessment of reproducibility requires re-
 52 peated characterization of the electrical parameters for

53 large ensembles of devices. To bypass the device-count
 54 limitation usually imposed on cryogenic nanowire ex-
 55 periments, we employ cryogenic multiplexers (MUX) for
 56 semi-automated characterization of large numbers of de-
 57 vices in a single cool-down.^{21–23} The variability between
 58 nominally identical devices is influenced by both struc-
 59 tural variations related to fabrication tolerances and local
 60 fluctuations in growth conditions and from the random
 61 electrostatic potential from charged impurities or adsor-
 62 bates in the vicinity of the device. We study these relative
 63 contributions by statistically correlating measurements
 64 of successive temperature cycles between the cryogenic
 65 regime and room temperature, where impurity configu-
 66 rations are randomized.²⁴ These distributions are com-
 67 compared to the fixed structural variability that is quantified
 68 through systematic atomic force microscopy (AFM) anal-
 69 ysis of 180 nominally identical nanowires.

70 The aim of this work is thus to quantify the statistical
 71 variations in the specific SAG InAs nanowires grown here,
 72 which serves as a reference for ongoing efforts to opti-
 73 mize SAG as a scalable platform for quantum electronics.
 74 Secondly, our work demonstrates the methods for acquir-
 75 ing the required statistical metrics in SAG circuits. We
 76 compare the results to alternative material candidates
 77 for cryogenic electronics, such as AlGaAs/GaAs 2D het-
 78 erostructures and cryogenic CMOS.

79 SAG nanowires were grown by molecular beam epitaxy
 80 (MBE) on undoped (311)A GaAs substrates, which are
 81 electrically insulating at low temperatures. A growth
 82 mask (10 nm of SiO₂) was patterned using electron beam
 83 lithography and dry etching to expose the GaAs sub-
 84 strate in large arrays of nominally identical 0.15 × 10 μm

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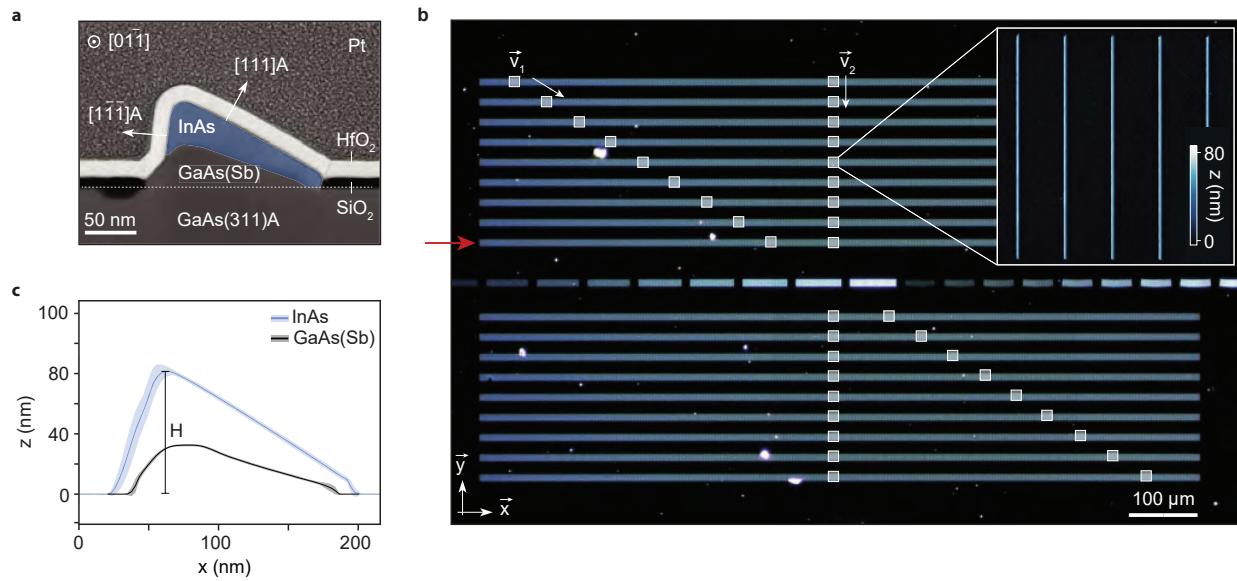


FIG. 1. **a** Cross-section transmission electron micrograph of a SAG InAs nanowire (blue). The asymmetric shape is a consequence of the (311) substrate symmetry. **b** Optical dark-field microscope image of an array consisting of two blocks of 9 rows, each holding 512 individual, nominally identical SAG nanowires with a length of $10\text{ }\mu\text{m}$ and cross-sections as in panel a. 180 nanowires were structurally characterized by AFM acquired at the positions of the white squares. An example of an AFM image is shown in the inset. The red arrow indicates the row of nanowires used in the transport measurements. **c** Example of cross-section AFM profile averaged over $1\text{ }\mu\text{m}$ of an InAs nanowire and the GaAs(Sb) buffer. The shaded regions correspond to the standard deviation. The maximum height, H , is used to quantify the morphological variations in the nanowire array.

rectangular openings aligned along the $[0\bar{1}\bar{1}]$ direction. The arrays contain two blocks. Each block consists of 9 rows spaced $35\text{ }\mu\text{m}$ apart, and holds 512 nanowires with a pitch of $2\text{ }\mu\text{m}$. The prepared substrates were introduced into the MBE chamber, and a GaAs(Sb) buffer layer was grown to improve the GaAs substrate surface for the subsequent growth of InAs which serves as the active conducting channel in the transport experiments.^{12,23} Further details of substrate preparation and growth parameters are included in Supplementary Section I. Figure 1a shows a high-angle annular dark field scanning transmission electron microscope (HAADF STEM) micrograph of a SAG nanowire cross-section. The nanowire has an asymmetric profile imposed by the (311) symmetry of the substrate. Figure 1b shows a dark-field optical microscope image of an array consisting of 8192 nanowires. Three such arrays from the same growth were used in this study for the TEM, AFM, and transport characterization.

We first consider the contributions to structural variations between neighboring nanowires and across the array. Although all nanowires are defined to be nominally identical, different effects contribute to variations at different length scales. While variations that occur at length scales significantly below the typical device length ($L_D \sim 1\text{ }\mu\text{m}$, see below) alter the average electrical parameters, the variations at longer scales re-

duce reproducibility between neighboring devices or lead to long-range modulations across the array. For example, on the few-nanometre scale, dislocations and stacking faults occur in the crystal due to the lattice mismatch at the GaAs/InAs interface. This is confirmed by cross-sectional TEM performed on four representative nanowires (see Supplementary Section III). Elastic relaxation is observed around the rounded top corner and the shorter $(1\bar{1}\bar{1})\text{A}$ nanowire facet, while misfit dislocations occur with an average distance of $\sim 9\text{ nm}$ along the longer $(1\bar{1}\bar{1})\text{A}$ InAs/GaAs(Sb) interface. In addition, based on a limited number of HR-TEM micrographs, a typical number of stacking faults between 1 and 4 are observed in each cross-section. As discussed previously,¹² the strain in the system and the elevated temperature during growth promote inhomogeneous diffusion of Ga from the GaAs(Sb) buffer layer during InAs growth as confirmed in cross-sectional HAADF STEM and electron energy loss spectroscopy (EELS) (Supplementary Sections III and IV). Such material intermixing will affect the electrical properties locally due to associated distortions of the band structure or defect states. As dislocations and stacking faults occur on a scale much shorter than L_D we expect that they mainly affect average electrical parameters such as the mobility, and contribute less to device-to-device variation. The impact of stacking faults on the mobility has been shown previously for out-

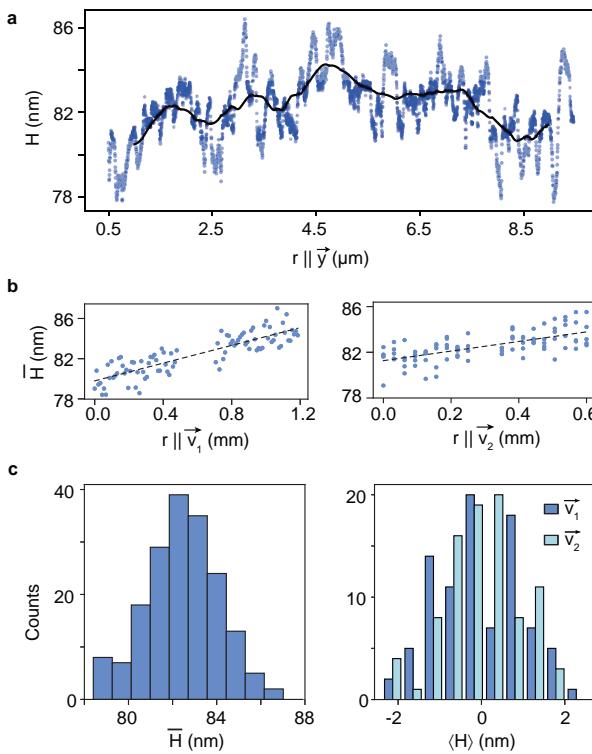


FIG. 2. **a** The height, H , defined in Fig. 1c along a typical SAG nanowire in the array of Fig. 1. The black curve shows the running average over 1 μm . **b** H averaged over a middle 1 μm segment for nanowires located along \vec{v}_1 (\vec{v}_2) as defined in Fig. 1b. The dashed lines are linear fits. **c** Histograms of the average \bar{H} and the corresponding residuals $\langle H \rangle$ after subtracting linear fits to data in panels b, quantifying short-range variation between wires of the array.

of-plane InAs nanowires²⁵, and a significantly improved mobility was observed in Ref.¹² by suppressing the formation of crystal faults.

On an intermediate scale ($\sim 20 - 100$ nm), etch roughness of the lithographically defined growth apertures and effects of random growth nucleation on the SiO_2 mask can lead to local structural variations along nanowires. Even larger scale modulations across the array ($\mu\text{m} - \text{mm}$) could arise from spatial variations in substrate temperature and adatom flux density across the substrate. To quantify these effects, detailed topographic AFM maps were acquired of 180 individual nanowires located at the positions of the white squares in Fig. 1b. An example of the average profiles of a 1 μm long segment from a complete nanowire structure, and from a GaAs(Sb) reference growth are shown in Fig. 1c, where the colored bands represent the standard deviation. As a characteristic parameter quantifying the morphological variations along nanowires and between nanowires of the array, an automatic procedure was developed to extract the maximum height, H , at each measured point along

each nanowire (See Supplementary Section V). As the cross-sectional shape is defined by the $[111]$ and $[1\bar{1}\bar{1}]$ crystal planes, other dimensions scales with H .

As an example, Fig. 2a shows H along the length of a single nanowire having an average height of 82.3 nm and a standard deviation of ± 0.9 nm. The black curve shows the running average, \bar{H} , using a 1 μm averaging interval, relevant for comparison with $L_D = 1 \mu\text{m}$ FET devices as discussed below. The relative standard deviation of \bar{H} along the length of the nanowire is 1.5%. To further quantify this and to investigate systematic variations also across the array, Fig. 2b shows \bar{H} from the center of each of 180 individual nanowires measured at positions along two directions across the array (\vec{v}_1 and \vec{v}_2 in Fig. 1). \bar{H} increases along both directions and linear fits yield slopes of 4.4 nm/mm and 4.2 nm/mm along \vec{v}_1 and \vec{v}_2 , respectively. Assuming that the linearity extends across the entire array, a plane-fit of $\bar{H}(x, y)$ using these slopes yields an estimate of a maximum of 6% variation of \bar{H} across a distance of 1 mm on the substrate. We attribute this variation to large-scale spatial modulation of the substrate temperature during growth²⁶. Such considerable variations could be important for integrated nanowire circuits covering a significant area. In addition to the overall trend observed in \bar{H} , variations between neighboring nanowires are clearly significant. To quantify this, Fig. 2c shows histograms of \bar{H} centered at 82.4 ± 1.6 nm and the residuals of \bar{H} after subtracting the linear trend along \vec{v}_1 and \vec{v}_2 . The standard deviation $\sigma_H = 0.97$ nm (1.1% of \bar{H}) corresponds to the average small-range height variations between the nominally identical nanowires.

Having discussed the structural variability across the array, we now consider the corresponding distribution and reproducibility of low-temperature electrical characteristics for nominally identical devices. A cryogenic, on-chip multiplexer/de-multiplexer circuit was used to enable the characterization of 256 individual, lithographically identical SAG nanowire FETs in a single cool-down (See Ref.²³ for details). Individual nanowires were electrically contacted by Ti/Au electrodes in an FET geometry keeping a constant channel length of $L_D = 1 \mu\text{m}$ and top-gates separated from the InAs channel by 15 nm of HfO_2 . Figure 3a shows an SEM micrograph of a part of the studied device array which is situated in the row indicated by the red arrow in Fig. 1b. The MUX circuit, which is shown schematically (an optical microscope image of the entire circuit is shown in Supplementary Section VIII.), addresses devices in pairs, and two gates (V_{G1}, V_{G2}) are used for the final selection; if one gate is active for a particular device, the other gate is inactive due to screening by the contact metal (insets to Fig. 3a). In the following, we denote the potential of the active gate of any device by V_G .

Figure 3b shows three representative examples of the conductance as a function of gate voltage, $G(V_G)$, measured at $T = 20$ mK. Typical n -type depletion mode FET behavior is observed as expected for InAs

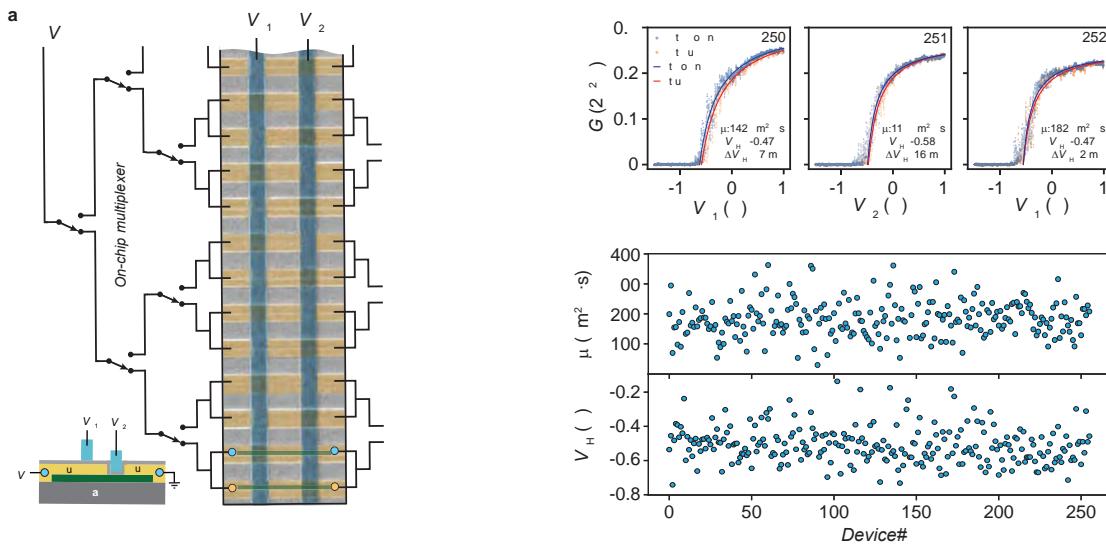


FIG. 3. **a** Schematic illustration of the multiplexer circuit used to address 256 individual SAG nanowire FET devices. The middle image shows a scanning electron micrograph of a part of the device array. Insets show schematic cross-sections of two neighboring devices. Note that the position of the exposed semiconductor segment alternates between devices, thus allowing the final selection to be carried out with the two top gates (V_{G1} and V_{G2}). **b** Example of the conductance as a function of gate voltage for three consecutive devices in the array. Data for the gate sweep towards negative voltage (down) is shown in blue, and towards positive voltage (up) - in orange. Blue and red lines are fits to the expression $G^{-1} = R_s + L^2/(\mu FE(V - V_{TH}))$. Fit parameters for the threshold voltage, V_{TH} and mobility, μ , for the down traces are stated in each case, and panel **c** shows the extracted parameters for all 256 devices in the array.

nanowires. By fitting $G(V_G)$ to the standard expression²⁷ $G^{-1} = R_s + L_D^2/(\mu FE(V - V_{TH}))$ – shown as orange lines in Fig. 3b – we extract the field effect mobility, μ_{FE} , threshold voltage, V_{TH} , and series resistance, R_s . The series resistance R_s accounts for contact resistance and the resistance of the measurement circuit. Numerical simulation was used to estimate the gate capacitance $C = 5.3\text{fF}$ ²³, which was used for all fits. Also stated in the figure are values for the difference in threshold voltage between positive (up) and negative (down) V_G sweep directions $\Delta V_{TH} = V_{TH}^{\text{down}} - V_{TH}^{\text{up}}$, used here as a measure of hysteresis.

Since effects of quantum confinement and surface scattering decrease with increasing nanowire dimensions, lower V_{TH} and larger μ are expected^{5,28–32} for larger \bar{H} . In Ref.³³ we investigated this relationship for InAs SAG nanowire and found approximately linear relationships with $\partial V_{TH}/\partial H = -14.1 \pm 1.0\text{mV/nm}$, and $\partial \mu/\partial H = 12.0 \pm 2.3\text{cm}^2/\text{Vs} \cdot \text{nm}$. This connects the structural properties and geometric variability discussed above with the variability of the electrical characteristics. If the geometric variability in Fig. 2 is dominating the statistics of the electrical properties, the standard deviations of the \bar{H} distribution, $\sigma_H = 0.97\text{ nm}$ would lead to a spread in V_{TH} of 13.7 mV and in μ of 11.6 cm^2/Vs .

Figure 3c shows μ and V_{TH} extracted for all 256 devices as a function of the position in the array (see Supplemen-

tary Sections VI and VII for the underlying data and fitting procedure). Device #1 and #256 are separated on the chip by $\sim 0.5\text{ mm}$ and the absence of any clear trend shows that either large scale, systematic structural variations (cf. Fig. 2b) are not significant in this array or that other effects dominate the electrical properties. Figure 4a shows the distribution of V_{TH} , which provides important information about the gate ranges required for optimal operation large-scale SAG nanowire circuits. In the present case, all devices in the array are in pinch-off (accumulation) for $V_G < -0.8\text{ V}$, ($V_G > -0.2\text{ V}$), respectively. The FETs of the MUX circuit (cf. Fig. 3 and Ref.²³) were operated at $V_G \pm 1\text{ V}$ to ensure robust closed (open) states²³.

The threshold voltage has a mean value of $\bar{V}_{TH} \approx -530\text{ mV}$ and a standard deviation $\sigma_{V_{TH}} = 90\text{ mV}$ ($\sim 17\%$ of \bar{V}_{TH}). The larger relative standard deviation of V_{TH} compared to those of \bar{H} suggests significant contributions to the variability from sources not directly detectable through H . One example could be microscopic crystal defects, as discussed above, or the effects of scattering and screening from random, charged impurities in the vicinity of the device. The latter depends on material quality, the quality of oxides, surface adsorbents, the parameters of processing, and experimental conditions. The charged impurity configuration remains stable if the impurity energy depth exceeds the thermal energy

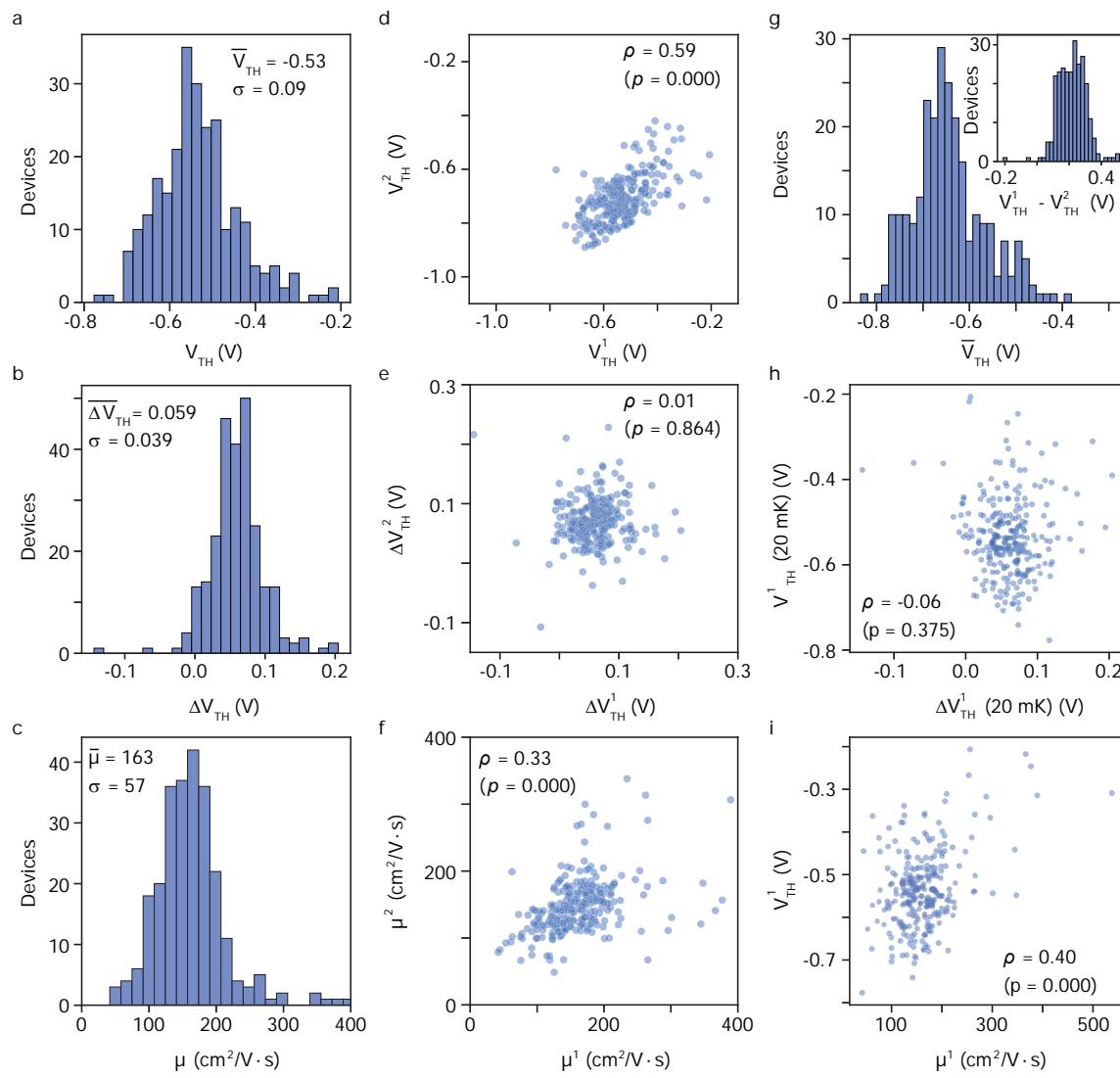


FIG. 4. **a,b,c** Histograms of threshold voltage V_{TH} , hysteresis ΔV_{TH} , and mobility μ , respectively, for 256 devices measured at 20 mK. **d,e,f** Correlations between two cool-downs to 20 mK for V_{TH} , ΔV_{TH} , and μ_{FE} respectively. Pearson's correlation coefficient ρ and the corresponding p -value shown in each panel. **g** Distribution of $\bar{V}_{TH} = (V_{TH}^1 + V_{TH}^2)/2$ and the differences $(V_{TH}^1 - V_{TH}^2)$ of threshold values measured for the two cool-downs. **h** Correlation between V_{TH} and ΔV_{TH} **i** Correlation between V_{TH} and μ .

and the energy scale of external electrical fields, and we, therefore, expect a static background at the millikelvin temperatures and V_G ranges used here. However, thermally cycling the device 20 mK \rightarrow 300 K \rightarrow 20 mK causes a random re-configuration of the charged impurities,^{24,34} allowing us to study their contribution to device reproducibility.

Figure 4d shows the correlation of the two V_{TH} values measured for each device at base temperature before and after thermal cycling having a correlation coefficient of $\rho = 0.59$. This shows that - although not detectable

through H - fixed structural/intrinsic properties partly govern the transport parameters. At the same time, however, a significant scatter in Fig. 4d shows that charged impurities - randomized by thermal cycling - also contribute to the overall spread in the V_{TH} . The corresponding distribution in Fig. 4g of the mean for each device estimates the structural distribution with a standard deviation $\sigma_{\bar{V}_{TH}} = 103$ mV which again exceeds the expectation from the variations of \bar{H} . We speculate that intrinsic defects such as atomic-scale crystal defects, stacking faults, and material intermixing could be responsible for these

1 thermal cycling independent variations between devices,
 2 which do not show up in the distribution of the geometric
 3 parameter \bar{H} .

4 The mobility is influenced by the same structural properties, and the distribution (Fig. 4c) and correlations
 5 (Fig. 4f) show qualitatively similar behavior, albeit with a larger dispersion caused by impurity charges. The
 6 hysteresis, ΔV_{TH} , on the other hand, is linked to gate-activated charge traps near the nanowire channel and is
 7 thus expected to be less dependent on the structure. This is consistent with the absence of correlations between the
 8 values obtained before and after thermal cycling, as seen in Fig. 4e.

9 Fig. 4h shows V_{TH} vs. ΔV_{TH} for all devices in the same
 10 cool-down. No correlations are observed, which is consistent with the hypothesis that the hysteresis is caused
 11 by rearranging surface charges - a process that is independent of the threshold voltage of the device. Similarly,
 12 Fig. 4i shows V_{TH} vs. μ . Here, both properties are dependent on the structure, and correlation is observed, which
 13 indicates that the intrinsic features governing the variation of threshold voltage are related to those determining
 14 the mobility.

15 Combined, the results show that the reproducibility in the values of μ and V_{TH} is influenced approximately
 16 equally by fixed structural/intrinsic properties and by the distribution of charged impurities, which vary between
 17 cool-downs. Thus, to improve the reproducibility of SAG
 18 nanowire transport properties at low temperatures, these
 19 results suggest that efforts should target both contributions.
 20 The intrinsic contribution cannot be accounted
 21 for by variation in the physical dimensions of the SAG
 22 nanowires, and thus, variations in composition or crystal
 23 defects may be the main source.

24 In summary, we have presented a comprehensive study
 25 of the reproducibility of structural and electrical parameters
 26 of nominally identical InAs nanowires realized by
 27 selective area growth. Large arrays of nanowires were
 28 grown, and statistics on the structural properties were ac-
 29 quired by high-resolution AFM analysis of 180 nanowires
 30 systematically spaced across the array. We quantify the
 31 nanowire-to-nanowire variability caused by local fluctu-
 32 ations in growth dynamics, lithographic accuracy, and
 33 large-scale systematic trends, presumably due to spatial
 34 temperature fluctuations across the growth wafer. To ac-
 35 quire statistically significant distributions of electron mo-
 36 bilities, threshold voltages, and hysteresis, we employed
 37 an on-chip cryogenic multiplexer design, allowing inde-
 38 pendent characterization of 256 individual devices. Com-
 39 paring the spread of values to that expected from the
 40 distribution of structural parameters and by correlating
 41 values between successive cool-downs to millikelvin tem-
 42 peratures, we quantify the contributions to the variability
 43 from intrinsic fixed properties and from charged impuri-
 44 ties in the vicinity of the devices.

45 The $\sigma_{V_{TH}}$ in our system is comparable to values found
 46 in AlGaAs/GaAs 2DEGs^{35,36} and foundry cryo-CMOS
 47 samples^{37,38}. However, it must be noted that the width
 48

49 of the gates in our system is larger than those of Ref.^{37,38},
 50 and variability is expected to decrease with increasing
 51 gate size³⁷ as the influence of impurities on the electro-
 52 static environment averages over a larger scale³⁹. Never-
 53 theless, given that this is the first study of reproducibility
 54 of InAs SAG and no specific attempts were made to opti-
 55 mize reproducibility in the studied structures, the results
 56 clearly show the potential of the platform. In further ex-
 57 periments intrinsic defects can be reduced by including
 58 an intermediary buffer layer¹² and the amount of charged
 59 impurities can be reduced by employing in-situ or resist-
 60 free processing⁴⁰.

61 The statistical approach developed here will be an im-
 62 portant tool for optimizing SAG nanowires and other
 63 bottom-up nanomaterials towards large-scale circuits and
 64 may motivate efforts towards developing high through-
 65 put TEM methodologies enabling statistical correlations
 66 between electrical quantum properties and the atomic
 67 scale structures. Finally, we emphasize that concerning
 68 the SAG nanowires, even with the current, low-
 69 mobility structures, reproducibility is sufficient for real-
 70 izing large-scale functional circuits such as multiplexer/de-
 71 multiplexer²³ and other logic circuitry. For circuits re-
 72 lying on high μ_{FE} or bandwidth, further optimization
 73 of μ_{FE} and the reproducibility of μ_{FE} is needed. The
 74 insights developed here will inform future optimization
 75 toward quantum devices with even stricter tolerances.

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Competing interests

98 The authors declare no competing interests.

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Supplementary Information

Supplementary information contains extended information on structural characterisation and extended transport datasets.

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Data Availability

The data supporting the findings is available at
<https://doi.org/10.11583/DTU.24967755>

