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Electrostatic actuated strain engineering in monolithically integrated VLS grown silicon nanowires

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Abstract
In this paper we demonstrate the fabrication and application of an electrostatic actuated tensile straining test (EATEST) device enabling strain engineering in individual suspended nanowires (NWs). Contrary to previously reported approaches, this special setup guarantees the application of pure uniaxial tensile strain with no shear component of the stress while e.g. simultaneously measuring the resistance change of the NW. To demonstrate the potential of this approach we investigated the piezoresistivity of about 3 μm long and 100 nm thick SiNWs but in the same way one can think about the application of such a device on other geometries, other materials beyond Si as well as the use of other characterization techniques beyond electrical measurements. Therefore single-crystal SiNWs were monolithically integrated in a comb drive actuated MEMS device based on a silicon-on-insulator (SOI) wafer using the vapor–liquid–solid (VLS) growth technique. Strain values were verified by a precise measurement of the NW elongation with scanning electron microscopy (SEM). Further we employed confocal μ-Raman microscopy for in situ, high spatial resolution measurements of the strain in individual SiNWs during electrical characterization. A giant piezoresistive effect was observed, resulting in a fivefold increase in conductivity for 3% uniaxially strained SiNWs. As the EATEST approach can be easily integrated into an existing Si technology platform this architecture may pave the way toward a new generation of nonconventional devices by leveraging the strain degree of freedom.

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Keywords: strain engineering, silicon, nanowire, MEMS

(Some figures may appear in colour only in the online journal)

1. Introduction
In recent decades strain tuning of the electrical and optical properties, in combination with quantum confinement effects in nanostructures, has become an increasingly promising subdomain of material research. In particular, semiconductors common in the microelectronic industry, such as Si semiconductors, feature several technically attractive strain dependent effects which improve performance or even enable new fields of application. In particular the piezoresistance effect—changing electrical resistivity of a material due to applied mechanical stress—was quickly adapted to realize micro-electro-mechanical sensor systems [1]. This strain effect on the transduction physics of silicon based piezoresistive sensors is closely related to the carrier mobility...
enhancement in strained Si-CMOS [2–4]. These strained channel field effect transistors, for example, have succeeded in moving beyond the research lab into mass production in the semiconductor industry [5, 6]. However, besides the carrier mobility [7] and the effective mass of carriers [7, 8], the band alignment [7–10], electrical conductivity [11–14] and thermal conductivity [15, 16] can also be modulated via strain and thus may lead to a seminal way to develop future devices that leverage the strain degree of freedom.

The amount of elastic strain which can be induced in a material is limited by the maximum fracture strength. For bulk Si yield stress lower than 3 GPa [17] can be applied without damaging the crystal structure, thus strain tuning is subjected to physical limits due to intrinsic materials properties. However, by reducing the dimensions the fracture stress limits are much higher at nearly the same Young’s modulus [18]. Thus VLS grown SiNWs with diameters below 200 nm have shown fracture stress values of 12 ± 3 GPa on average [19]. In GeNWs, uniaxial tensile strain values of more than 10% have already been demonstrated without fatigue failure [20]. Hence, the ability to fabricate single-crystal NWs that are widely free of structural defects will make it possible to apply high strain without creating fractures. Thus, in any application where crystallinity and strain are important, the implementation of NWs should therefore be advantageous.

However, strain engineering applications of NWs require accurate mechanical characterization, which in turn requires development of novel experimental techniques. Atomic force microscopy (AFM) setups or nano-manipulation stages [21–23] were used to apply mechanical stress on NWs. The drawback of these techniques are the problems of handling and positioning nanometer-sized objects and the limited portability and thus incompatibility with other in situ characterization techniques e.g. cryo measurements. Other concepts to induce strain into NWs, such as micromechanical three-or four-point bending methods, enable more flexibility to accomplish various measurements [11, 24], but shear forces on the NW have to be considered since strain will not be induced directly and uniaxially on the NW.

With the application of the proposed MEMS devices, highly resolved and reproducible levels of pure tensile strain without any shear components can be applied to individual as well as arrays of NWs. Furthermore the compactness and portability of such devices allows the integration into various measurement setups which makes the EATEST devices a versatile material characterisation tool. Basically two actuation modes have been established for MEMS based straining of NWs: thermal [25–27] and electrostatic actuation [14, 28–32]. MEMS devices bring a huge design space and the main challenge is to find a good trade-off between robustness, stability, functionality and the ability to apply a high level of strain on the NW with a maintainable actuator voltage. Previous work has been shown that such MEMS devices are perfectly suitable for piezoresistivity characterization of SiNWs [14, 33, 34]. Thereby NWs are integrated by pick and place techniques using nano-manipulators and subsequently fixed for contact formation. As a more practical way of overcoming the aforementioned obstacles for the integration of NWs, in-place epitaxial NW growth has been proposed with robust linkages to pre-patterned electrodes [35, 36]. Particularly the selective deposition of the nanowire growth-promoting catalyst as well as the growth parameters must be optimized. With respect to the growth uncatalytic deposition of poly-Si has to be avoided as this induces leakage currents which can disturb the electrical measurements. The main advantages of the monolithic integration are the self-aligned contact formation, the lack of metal-semiconductor Schottky contacts and no additional strain dependent contact factor due to metallic NW pinning [34]. Additionally, when ion beam induced metal deposition is used for NW fixation, no amorphization of the NW due to the ion beam can occur [37]. Such monolithic integration of VLS grown NWs in the EATEST device appears as an ideal platform for the exploration of strain-related effects even on an industrial scale using standard Si processing technology.

2. Experimental section

Basically the EATEST device comprises three functional parts as schematically illustrated in figure 1(a).

The comb drive actuator consists of interdigitated structures of the fixed stator and the movable suspended cantilever of the stage (see inset of figure 1(a) and the SEM in figure 1(b)). The test object, in our case a SiNW about 3 μm long and 120 nm in diameter, is integrated monolithically via VLS growth between this suspended cantilever of the stage part and the specimen part. Figure 1(b) shows the respective SEM image of the EATEST device with the detailed view of the integrated SiNW in the inset. By applying a voltage on the stator part, electrostatic forces pull the freestanding cantilever of the stage towards the stator part thus straining the suspended SiNW. The EATEST device was fabricated using a SOI wafer with a 2 μm thick, (110) oriented device layer on top of a 2 μm thick buried oxide (BOX). After cleaning the substrate by rinsing it with pure acetone and isopropanol, conventional photolithography was used for EATEST pattern definition. For the pattern transfer a chromium hard mask was deposited and after the lift off, plasma enhanced reactive ion etching was used for device layer patterning. To ensure the application of pure tensile strain later on, the NMU must be perfectly aligned along the actuation path of the EATEST, thus perpendicular to the facing surface of the specimen and the stator part. Controlling the alignment of the EATEST device with respect to the (110) oriented device layer of the SOI wafer can on can end up with (111) oriented end faces of the stator and the specimen part. Thus by epitaxial growth of <111> oriented SiNWs perpendicular to the end face of the specimen part, perfect alignment with respect to the actuation path of the MEMS device can be achieved.

After hard mask removal, the stage was partly underetched with buffered HF solution, removing the underlying BOX layer resulting in the freestanding and thus movable suspended cantilever close to the specimen part. The freestanding cantilever is fixed during BOX removal, as well as
the successive processing, by stabilizing Si-beams marked with white arrows in figure 1(b). These stabilizing bridges avoid sticking of the moveable part to the handle layer of the substrate due to wet-chemical etching, spin coating of photoresist and during NW growth, and are cut through by focused ion beam (FIB) milling at the very end of the device processing.

SiNWs are integrated monolithically into the EATEST device via epitaxial VLS growth [38] in a low pressure chemical vapor deposition (CVD) system. As VLS growth-promoting catalysts, 80 nm gold colloids are deposited on the (111) facet of the specimen part opposite to the moveable stage part by dielectrophoresis. After loading the sample into the CVD reactor, NWs were grown with 2% silane diluted in He as precursor at T = 500 °C and 3 mbar total pressure. Under these growth conditions epitaxial SiNWs grow preferentially in the <111> direction and thus perpendicular from the facet of the specimen part to the stage part bridging the 3 μm wide gap. Finally, we ended up with a suspended SiNW anchored with one end to the specimen part and the other to the moveable part of the stage.

The interface region between such epitaxially grown SiNWs and the (111) surface of Si is shown in the high resolution transmission electron microscopy (HRTEM) image of figure 2(a). The dashed line represents the substrate/SiNW interface. For the growth conditions mentioned above, no grain boundaries, abrupt interfaces, or misfit dislocations were observed in this region and the interface appears to be coherent, unstrained, and epitaxial Si to Si. It was demonstrated previously that such grown NWs exhibit extraordinarily robust electrical and mechanical contacts on both sides [39, 40]. The two terminal I/V characteristic shown in figure 2(b) demonstrates ohmic contact behavior of the crystalline SiNW monolithically integrated into the EATEST device. The intimate contact between the epitaxial SiNWs and the stage and specimen part enables later on to apply exceptional high strain levels and to measure the resistance change in the NW simultaneously. As mentioned above, the ultimate processing step is the removal of the anchor points by FIB cutting.

3. Results and discussion

The feasibility of the EATEST device for straining monolithically integrated NWs was tested by introducing the device into a scanning electron microscope (SEM) with electrical feedthroughs. Straining was achieved by applying an actuator voltage on the stator while forcing the stage as well as the handle layer of the sample to ground potential as shown in figure 1(a). The length and elongation of the SiNWs was thus determined in situ by SEM imaging, measuring the distance between the contact points of the NW with the specimen and the stage part facets. An animated SEM-image sequence of the SiNW elongation at different actuator voltages is given in the supporting information. The epitaxial growth of the SiNWs perpendicular to the Si pads (i.e. to the direction of the electron beam) enables a precise measurement of the NW elongation with approximately 10 nm precision without any ‘hidden’ displacement along the electron beam direction. Thus for the 2.85 μm long SiNW we determined an elongation of 48 nm corresponding to 1.7% tensile strain at an actuator voltage of 110 V.

To determine the strain in situ during the electrical characterization we used confocal μ-Raman spectroscopy. For excitation a frequency doubled Nd:YAG laser with a wavelength of 532 nm was used. With a 100x microscope objective (NA=0.9) the spatial resolution for Raman spectroscopy measurements is about 360 nm. Details on the Raman measurements are given in the supporting information. The upper inset in figure 3 shows the shift of the first-order Brillouin zone center Raman peak of the SiNW which appeared to be linear proportional to tensile strain [41]. For reliable measurement of strain related Raman peak shifts in nanostructures, phonon confinement [42] and NW heating [43] have to be considered since they also cause blueshifts of the Si Raman peak. Phonon confinement becomes
effective at SiNW diameters below 20 nm and can therefore be neglected for the NWs integrated in the EATEST device with diameter considerably above 100 nm. To avoid effective NW heating, excitation laser power was limited to 10 μW, hence the obtained Raman peak shifts observed for different actuator voltages, shown in the upper inset of figure 3, are solely related to strain in the suspended SiNW.

Zhu et al [18] have shown a constant Young’s modulus for tensile strained SiNWs in our diameter range, confirming the proportionality of force driven stress and strain. As the electrostatic force acting on a comb drive is proportional to the square of the actuator voltage [44], the strain in the SiNW applied by the EATEST device is also expected to be proportional to the square of the actuator voltage. The main plot in figure 3 shows the perfect agreement of strain values determined by measuring the NW elongation as well as by μ-Raman spectroscopy, being proportional to the square of the actuator voltage.

To calibrate the Raman measurements with respect to NW elongation we compared the peak shifts with the absolute physical length change of the NWs for different actuation voltages (see lower inset in figure 3). Thereby, the relation of the Raman peak shift and uniaxial strain is given by

$$\Delta \Omega \approx -k \cdot \varepsilon ||$$  \hspace{1cm} (1)

with $\Delta \Omega$ representing the shift in peak position, $k$ a proportionality factor, and $\varepsilon ||$ the strain along the $\langle 111 \rangle$ growth direction of the VLS grown SiNWs. Our calibration measurements reveal $k = 326 \text{ cm}^{-1}$ in good agreement with the bulk Si values from Ureña et al of $k = 343 \text{ cm}^{-1}$ [41]. The above equation is further used to quantify the strain level of the SiNWs in-situ immediately before the measurement of the piezoresistive response. Thus, an actuator voltage of 110 V leads to a peak shift of about 5.5 cm$^{-1}$ corresponding to a tensile strain of about 1.7%.

To characterize the piezoresistive response of the SiNW integrated in the EATEST device we applied a voltage sweep on the specimen part from $V_{NW} = -5$ V to +5 V and monitored the current flow in the NW at different actuator voltages thus varying the strain level. Thereby the stage and the handle layer of the substrate were grounded to avoid electrostatic attraction between the movable cantilever of the stage part and the substrate. To avoid a possible fracture of the NW due to an instant high strain level, the actuator voltage was ramped up in steps of 5 V. Instant release from high actuator voltages to zero showed no damage on the NW and Raman spectroscopy measurements showed a complete return to the original unstrained state as shown in the supporting material.

Figure 2. (a) Cross sectional HRTEM image of the substrate-nanowire interface region of a Si $\langle 111 \rangle$ NW grown epitaxial on a Si $\{111\}$ surface. (b) Two-terminal I/V characteristic of a SiNW monolithic integrated in an EATEST device (see inset). INW was measured while sweeping VNW from $-5$ V to $+5$ V. The measurements showed a nearly ohmic contact behavior of the SiNW.

Figure 3. Uniaxial strain values on SiNW determined by SEM investigation and peak shift of the first order zone center Si Raman peak as function of the actuator voltage. Measured strains (black circles) as well as Raman peak shift values (red squares) are proportional to the square of the actuator voltage (dashed lines representing quadratic fitting curves). The upper inset shows the Raman spectra of the SiNW at different actuator voltages. With increasing voltage, the first order zone center Si Raman peak shifts to lower wavenumbers. The lower inset demonstrates the linear proportionality of strain determined by SEM imaging and Raman peak shift.
Relative resistivity change of a 3.3 μm long, VLS grown SiNW, 100 nm in diameter as function of uniaxial tensile strain. I/V characterization of the suspended SiNW was done by sweeping the voltage \( V_{\text{NW}} \) (see figure 1) on the specimen part while stepwise increasing the actuator voltage \( V_{\text{Act}} \). The I/V characteristics of the SiNW at different actuator voltages are shown in the inset. Curves are carefully calibrated to eliminate measurement offsets, so that no current or voltage shift is observed. Resistivity values are calculated by extracting the gradient of \( I_{\text{NW}} \) around 0 V bias voltage.

The equation for the relative change in resistivity \( \Delta \rho/\rho \) for a cylindrically shaped piezoresistive element as the NWs is given by

\[
\frac{\Delta \rho}{\rho} = \frac{\Delta R}{R} - (1 + 2\nu)e_{||} \cong \frac{\Delta R}{R},
\]

with \( \nu \) the Poisson’s ratio and \( e_{||} \) the strain along the cylinder axis. The influence of strain induced NW elongation and thinning on the resistance is small against the resistivity change due to the piezoresistive effect and will be neglected for resistivity calculations.

The inset in figure 4 shows typical I/V characteristics of a suspended 3.3 μm long SiNW for increasing actuator voltages and thus increasing strain levels. Resistivity values of the SiNW were calculated by extracting the gradients of \( I_{\text{NW}} \) around 0 V bias voltage. As the observed changes in resistance are significantly larger than the dimension changes, we converted relative changes in resistance to the relative change in resistivity, subtracting the dimensional parameters but neglecting the dimensional changes of the NWs. The calculated relative change in resistivity of a SiNW as a function of uniaxial tensile strain is plotted in figure 4. As expected for moderately strained (111) oriented SiNW \( \Delta \rho/\rho \) increases up to about 0.2% [11, 47]. He and Yang [11] found in 2006 a positive giant piezoresistance effect (PZR) in SiNWs however, only for small strain levels of up to 0.04%. They reported on an almost two orders of magnitude increase in the piezoresistance coefficient of NWs compared to bulk material. Even though they triggered a lot of experimental and theoretical effort, there still seems to be no consensus in the community on the origins of the effect. Modified carrier mobility [11], band switching between two surface states [48] or surface depletion [49] have been proposed to be the mechanisms involved. However for strain levels of about 0.25% and beyond, the anomalous piezoresistance effect becomes effective and \( \Delta \rho/\rho \) decreases exponentially and reached ~80% at 3% strain. This equals a fivefold increase of SiNW conductivity and compares well with previous strain experiments on VLS-grown intrinsic SiNWs [12].

Finally the actuator voltage was increased until the suspended NWs fail via sudden brittle fracture. For the particular NW mentioned above (3.3 μm long, 100 nm diameter) fracture stress of the SiNW was reached at 130 V actuator voltage which equals 3.3% tensile strain. Assuming a typical Young’s modulus of \( E=187 \) GPa [18], for (111) oriented SiNWs the calculated fracture stress of 6.17 GPa appeared to be two times higher compared to bulk Si [9].

4. Conclusions

We demonstrated an electrostatic actuated device as an ideal platform for uniaxial strain engineering which enables electrical as well as optical characterization of the NW under accurate and reproducible tunable tensile strain. \textit{In situ} strain measurements of SiNWs with SEM and \( \mu \)-Raman spectroscopy as well as electrical characterization using the EATEST device showed strain levels of up to 3.3% before fracture yielding fracture stress of 6.17 GPa, which is two times higher as compared to bulk Si. For the maximum tensile strain of 3%, the resistance of the SiNW decreased by a factor of 5. SiNWs were integrated monolithically into the EATEST device utilizing the VLS mechanism, but in principle this device is suitable for a multitude of colloid catalyzed NWs beyond Si.

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