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***In situ* control of synchronous germanide/silicide reactions with Ge/Si core/shell nanowires to monitor formation and strain evolution in abrupt 2.7 nm channel length**

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The metal-semiconductor interface in self-aligned contact formation can determine the overall performance of nanoscale devices. This interfacial morphology is predicted and well researched in homogenous semiconductor nanowires (NWs) but was not pursued in heterostructured core/shell nanowires. We found here that the solid-state reactions between Ni and Ge/Si core/shell nanowires resulted in a protruded and a leading NiSi_y segment into the channel. A single Ni₂Ge/NiSi_y to Ge/Si core/shell interface was achieved by the selective shell removal near the Ni source/drain contact areas. Using *in situ* transmission electron microscopy, we measured the growth rate and anisotropic strain evolution in ultra-short channels. We found elevated compressive strains near the interface between the compound contact and the NW and relatively lower strains near the center of the channel which increased exponentially below the 10 nm channel length to exceed 10% strain at ~3 nm lengths. These compressive strains are expected to result in a non-homogeneous energy band structure in Ge/Si core/shell NWs below 10 nm and potentially benefit their transistor performance.

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Semiconductor heterostructured transistors play a pivotal role in advanced high-speed analog telecommunications circuits and amplifiers.^{1,2} The performance of these analog devices and that of logic devices are enhanced at shorter channel lengths and can be tailored by strain engineering through compositional change parallel to or in the direction of current transport, such as in the SiGe source/drain regrowth for Si channels.^{3,4} The standard practice in obtaining short channel devices is enabled by the solid-state reaction of self-aligned contacts.^{5,6} The combination of heterostructure engineering and self-aligned contacts at atomic to nano-scales is expected to refine the benefits of the two approaches. However, despite macroscopic studies on forming short transistor channels in heterostructured nanowires,^{7–10} detailed metallurgical studies on solid-state reaction with heterostructured nanowires and the morphology of the contact/semiconductor interface have not been pursued thus far. Prior detailed efforts in this field have primarily focused on metallic contact formation in homogeneous semiconductor nanowires, such as in Si,^{11,12} Ge,^{13,14} and III-V^{15,16} NWs. We focus in this work on Ge/Si core/shell NWs because they have several advantages including alleviated surface scattering and accumulated holes in the core due to the type-II band alignment between Ge and Si¹⁷ and therefore larger hole-mobility in the Ge core.

Additionally, the lower strain in the Si shell—due to strain sharing in core/shell NW geometry—than its thin-film counterpart^{18,19} leads to the growth of more uniform Si layers on Ge. Therefore, we used the Ge/Si core/shell NW as a model system to investigate the compound contact formation between metal (Ni) and semiconductor heterostructures *in situ* inside a transmission electron microscope (TEM).

The Ge, Si, and Ge/Si core/shell NWs in this work were grown in a low pressure, cold wall chemical vapor deposition (CVD) system.^{20,21} The diameters of Ge cores ranged from 10 to 35 nm and the Si shells had an average thickness of 2 nm, both measured by high-resolution TEM (HRTEM). The fabrication steps and methods are detailed in the [supplementary material](#). Under the same fabrication and imaging conditions, we found distinct growth-rate behaviors and interface morphologies in the Ni reaction with Ge, Si, and Ge/Si core/shell NWs that have similar diameters (Fig. 1). The three samples underwent a simultaneous *ex situ* rapid thermal anneal (RTA) process at 300 °C for 30 s with a forming gas (N₂/H₂) flow at a chamber pressure of 1 Torr. We observed that the nickel germanide/germanium (Ni_xGe/Ge) interface remained flat during the reaction as shown in Fig. 1(a), similar to the nickel silicide/silicon (NiSi_y/Si) interface in Fig. 1(b). However, for the Ge/Si core/shell NW [Fig. 1(c)], the Ni_xGe/Ge and NiSi_y/Si interfaces each remained flat in the core and shell, respectively, while the reaction front of the NiSi_y shell leads the

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Ni_xGe core by ~ 20 nm. We then investigated the origin of this behavior in order to control the morphology of the compound contact to the Ge/Si core/shell NW.

Numerous studies on the Ni reaction with homogeneous semiconductor NWs (i.e., Si, Ge) have demonstrated Ni as the dominant diffusion species into NWs and a Ni-diffusion limited (mass-transport limited) growth.^{22–24} Ni has a much higher estimated diffusivity in Ge ($\sim 8 \times 10^{-9}$ cm²/s, at 300 °C)²⁵ than that in Si ($\sim 1 \times 10^{-10}$ cm²/s, at 300 °C)²⁶ due to the larger atomic spacing in the Ge lattice.²⁷ Therefore, the rate of arrival for Ni atoms to the growth interface is less inhibited in Ge than in Si and the reaction rate is consequently faster in Ge, leading to a longer Ni_xGe segment [Fig. 1(a)] in Ge NW channels than the NiSi_y segment [Fig. 1(b)] in Si NW channels under the same reaction conditions. In Ge/Si NWs, the Ge core and Si shell share the strain due to the nearly 4.2% lattice mismatch between the tensile strained Si core and the compressive strained Ge shell. Since the Ge core dominates the volume of the entire NW body, it experiences less strain than the Si shell. Therefore, the Ni diffusivity in Ge/Si core/shell NWs is expected to be little influenced by strain and the reacted Ni_xGe length in Ge/Si core/shell NWs [Fig. 1(c)] was very close to that in Ge NWs. Here, the protruded NiSi_y phase cannot be well explained with the change of Ni diffusivity in the strained Si shell, and other factors may play roles in this phenomenon as we will discuss next.

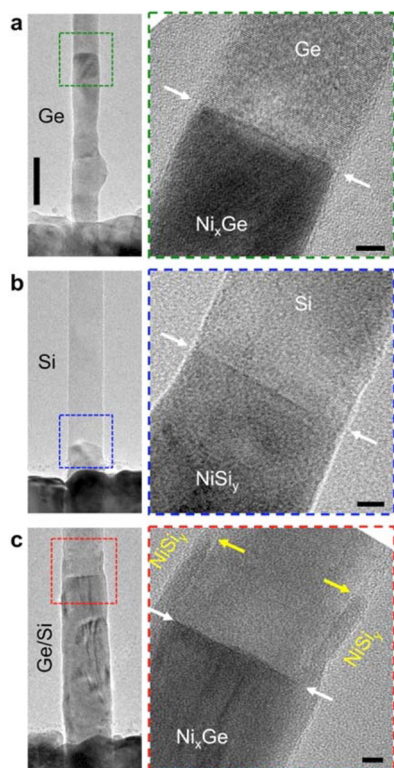


FIG. 1. (a) TEM image of nickel germanide formation in a Ge NW, with the high magnification TEM (HRTEM) image showing the $\text{Ni}_x\text{Ge}/\text{Ge}$ interface. (b) TEM image of nickel silicide formation in a Si NW, with the HRTEM image showing the NiSi_y/Si interface. (c) TEM image of nickel germanide/silicide formation in a Ge/Si core/shell NW, with the HRTEM image showing the protruded silicide shell than the germanide core. All three samples were annealed in RTA with forming gas (N_2/H_2) at 300 °C for 30 s. Low magnification TEM images share the same scale bar of 50 nm, and the scale bars for all HRTEM images are 5 nm.

From separate observations on Ni reaction with other NW materials, we observed distinct ledge nucleation and movement behaviors under the metal pad than in the NW cross-section.²⁸ In InGaAs, we found that a nickelide shell is quickly formed on the NW surface as the reaction started and then grew evenly but slowly into the NW core with multiple nucleation sites and reaction interfaces under the metal pad. This is different from the single interface in elementary NW channels when the reaction extends outside the metal pad.^{29,30} In the case of Ge/Si nanowires, as the reaction starts underneath the source/drain Ni pads, the Si shell will first be reacted to form a NiSi_y shell before the Ge core is completely reacted to form a Ni_xGe core. As a result, the NiSi_y shell protrudes into the channel earlier than the Ni_xGe core extends outside the Ni pad, resulting in a leading NiSi_y reaction front [Fig. 1(c)]. We therefore concluded that the Ni_xGe core needs to protrude outside the metal pad and into the channel simultaneously with the NiSi_y shell in order to balance and simultaneously grow the Ni_xGe and NiSi_y fronts. To validate this hypothesis, we carried out *in situ* heating experiments and monitored the dynamic reactions between Ni and Ge/Si NWs inside TEM as is schematically shown in Figs. 2(a) and 2(b). Figure 2(c) shows the sequence of reactions near the Ni contact area. The Si shell had been removed prior to Ni deposition by five cycles of oxidation (ambient environment, 1 h) and oxide stripping (BOE 1:20 in deionized water, dip for 30 s). We found that the left portion of the NW had a smaller diameter than its right portion due to the Si shell removal on the left. The reaction happened first in the Ge core and then gradually extended into the Ge/Si core/shell region. During this process, $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ initially did not have a fixed interface on certain atomic planes of the Ge/Si NW and the upper portion of the reacted NW extended further into the channel. Later, the $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ compounds gradually started to react uniformly across the entire NW body, and the reaction fronts became flat for both Ni_xGe and NiSi_y at time $t_0 + 14$ min. To validate that the reacted $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ phases still had a flat front at very short channel lengths, we monitored the reaction in another Ge/Si NW at a channel length below 30 nm as shown in Fig. 2(d). The $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ phases nucleated heterogeneously from one side of the NW near the surface and the nucleuses had a step height of multiple atomic layers on the Ge (111) planes as shown in the colored arrows overlaid on Fig. 2(d) panels. The $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ reaction front remained flat during further reactions.

Using this fabrication process [Fig. 2(b)] and the *in situ* heating and monitoring method, we were able to control the reacted $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ segment lengths and achieved a channel length of 2.7 nm that corresponds to 7 layers of Ge (111) atomic planes as shown in Fig. 3(a). Ultra-small channel lengths have been demonstrated in elementary NWs by similar compound contact reactions monitored *in situ* by TEM, such as 8 nm-channel in PtSi/Si/PtSi NW,³¹ 15 nm-channel in $\text{Cu}_3\text{Ge}/\text{Ge}/\text{Cu}_3\text{Ge}$ NW,³² and 2 nm-channel in NiSi/Si/NiSi NW³³ but not in heterostructured NWs. Energy-dispersive X-ray (EDX) line-scan across the NW diameter in the reacted segment is summarized in Fig. 3(b). The high-angle annular dark-field (HAADF) image is shown as an inset and its contrast contour is also plotted in Fig. 3(b) with the peak intensity normalized with the height of Ni signal

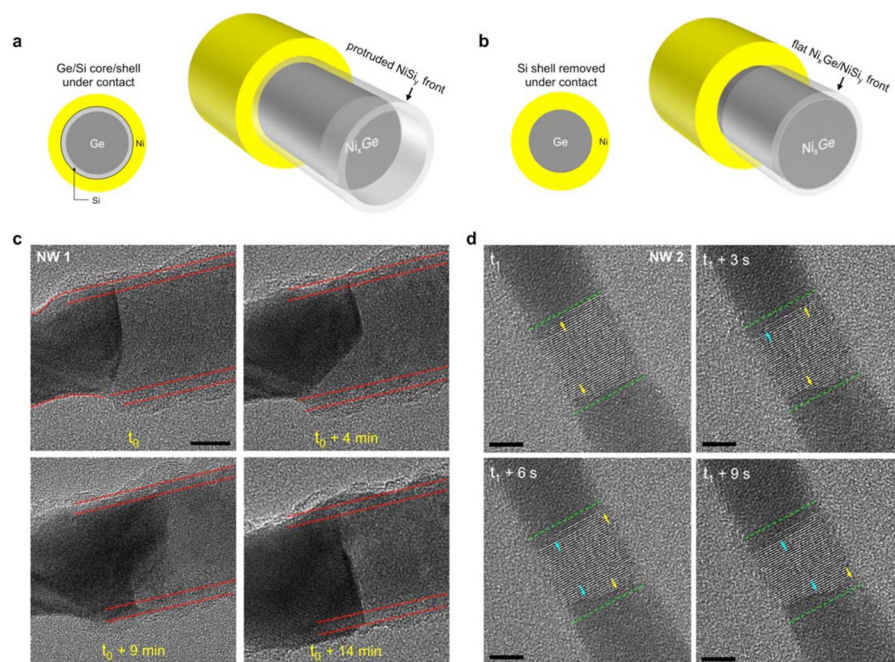


FIG. 2. (a) and (b) Schematics of $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ contact formation in the Ge/Si NW before and after removing the Si shell at the metal contact area prior to Ni deposition, respectively. (c) HRTEM sequences from a Ge/Si NW showing the $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ formation near the contact where the Si shell has been removed. (d) HRTEM sequences from another Ge/Si NW showing the flat $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ front when approaching very short channel lengths. Scale bars are 5 nm.

counts. The profile width of the Ni signal-counts was close to that of the HAADF contour, indicating the full reaction of the NW body with Ni. The profile width of the Ge signal was narrower than that of Ni, suggesting that the Ge element was still limited to the core without any intermixing with the Si shell during the reaction with Ni and that the shell had a thickness of ~ 2 nm on each side. The EDX signal of the thin Si shell was barely detected and the Si_3N_4 TEM window underneath provided large background Si counts to which we performed the subtraction of the baseline, thus resulting in nearly zero Si counts.

At a few nanometer channel lengths, unprecedented levels of strains (along the channel direction) are expected from the 57% volume expansion due to the phase

transformation from Ge to Ni_2Ge , a phase that we will discuss its validation later in this work. To systematically investigate the channel strains at different lengths and their influence on the $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ growth, we captured *in situ* videos of the reactions at very short channel lengths with HRTEM as shown in Fig. 4(a). We noticed that the reaction fronts of $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ remained aligned on Ge/Si (111) atomic planes and were perpendicular to the channel direction. Labeled on the left-most figure are the directions of the in-plane ($\epsilon_{\parallel}$) and the out-of-plane ($\epsilon_{\perp}$) uniaxial strains along the [111] channel direction.^{34,35} The non-reacted channel length as a function of time exhibited a linear dependence [Fig. 4(b)], indicating that the kinetic reaction rate remains constant even with strain accumulation at the reaction front for such nanoscale channel lengths. The $\epsilon_{\perp}$ strains were analyzed by measuring the inter-plane distances, both near the left and right interfaces of Ge/Si with $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ compound contacts and at the center of Ge/Si channel, which are plotted in Fig. 4(c). Each data point was the average of three measurements (top, center, and bottom) at the same distance (within 2 nm) from the $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ interfaces or at the channel center. The interfacial $\epsilon_{\perp}$ showed an average value of 5% of compression at a channel length of 40 nm and gradually increased to $\sim 10\%$ of compression at a channel length of 3 nm. However, the $\epsilon_{\perp}$ at Ge/Si channel center showed a different behavior than the interfacial strains and remained below 1% of compression when the channel length was larger than 10 nm. As the channel length reached 10 nm and smaller, the center of the overall channel saw a dramatic increase of compressive $\epsilon_{\perp}$ and reached $\sim 10.5\%$ at the 3 nm channel length. Such a high channel strain is possible in nanowire channels due to the greatly reduced Young's modulus of the material at nanoscale.^{36,37} A very high channel strain up to 12% of compression was also reported in 2 nm channels of $\text{NiSi}/\text{Si}/\text{NiSi}$ NW.³³ Uniaxial strain is known to lift the valence-band degeneracy and lead to the crossover of heavy-hole (HH) and light-hole (LH)

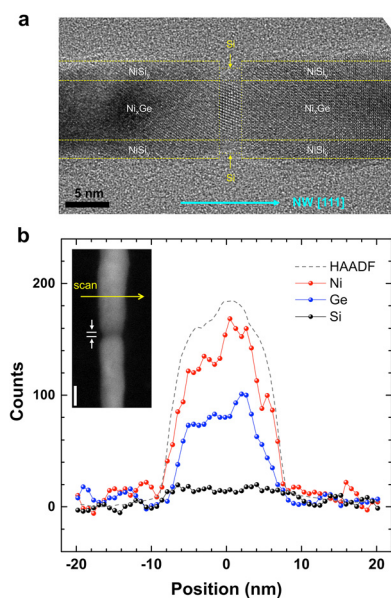


FIG. 3. (a) HRTEM image of an ultra-short Ge/Si channel of 2.7 nm with $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ contacts at both ends. (b) The EDX spectrum with line-scan across the $\text{Ni}_x\text{Ge}/\text{NiSi}_y$ contact, and insertion is the HAADF image.

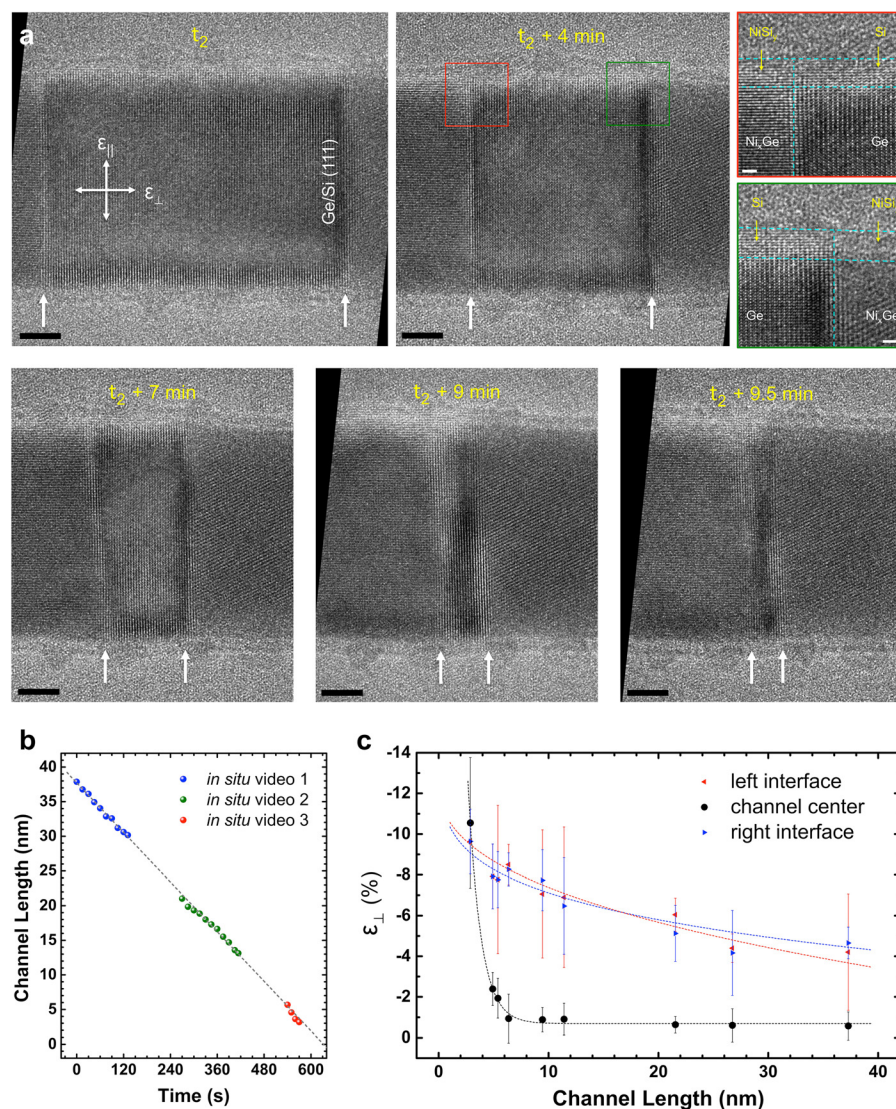


FIG. 4. (a) HRTEM sequences that show the consecutive formation of Ni_xGe/NiSi_y contacts from both ends of the channel for reaching a very short-channel length. Scale bars are 5 nm. The highlighted HRTEM images show the flat reaction front of Ni_xGe and NiSi_y. Scale bars are 1 nm. (b) Plot of the channel length as a function of time from the recorded *in situ* videos. (c) Plot of channel strain ($\epsilon_{\perp}$) as a function of channel length with measurements from left interface, channel center, and right interface, respectively.

bands under compressive stresses.^{38,39} As more holes populate the LH bands, the effective mass of holes is reduced, leading to an enhanced hole mobility that boosts transistor performance. Mapping and evaluating the stress distributions within short NW transistor channel length becomes inevitable to design nanowire transistor channels below 10 nm. Future efforts will focus on the electrical characterization to quantify the strain effects and to improve the gate-modulation over such small channel lengths.

Finally, we examined the evolution of the compound phase formation and its interfacial correlation with the NW by analyzing the Ni_xGe/NiSi_y phases at different locations of a Ge/Si NW as shown in Fig. 5 and detailed in the [supplementary material](#). The extracted lattice constants for the germanide from fast-Fourier transform (FFT) patterns and from the EDX analysis preserved the Ni₂Ge phase. Abrupt changes of diameter were observed in the nickelide region due to crystal rotation as is further elaborated in the [supplementary material](#). The interfacial correlations between Ni₂Ge and Ge were also found to be different at the two sides of the Ge/Si channel. On the left side, the Ni₂Ge/Ge followed a crystallographic relationship of Ni₂Ge $[\bar{1}21] \parallel \text{Ge } [220]$

and Ni₂Ge $[\bar{1}\bar{1}1] \parallel \text{Ge } [11\bar{2}]$, while on the right side, the interface followed a different crystallographic relationship of Ni₂Ge $[\bar{1}21] \parallel \text{Ge } [\bar{2}20]$ and Ni₂Ge $(11\bar{1}) \parallel \text{Ge } (111)$. In both cases, the $[\bar{1}21]$ direction of Ni₂Ge was in parallel with the Ge $[\bar{2}20]$ direction, due to the fact that the $(\bar{1}21)$ Ni₂Ge inter-plane distance (2.04 Å) is very close to the $(\bar{2}20)$ Ge inter-plane distance (2.00 Å) with a lattice mismatch of 2%.

In summary, we reported the systematic analysis of solid-state reactions between metal (Ni) and heterostructured (Ge/Si core/shell) NWs that exhibited a uniform and abrupt Ni₂Ge/NiSi_y to the Ge/Si core/shell interface with an unreacted channel length of 2.7 nm. The *in situ* monitoring of this thermally driven reaction under HRTEM allowed us to evaluate and monitor the strain evolution in ultra-short channel Ge/Si core/shell NWs. The interfacial strains between Ge/Si and Ni_xGe/NiSi_y compounds showed different ascending behaviors than the strains at the channel center, both of which exceeded 10% of compression below 3 nm unreacted Ge/Si core/shell NW. The formed Ni_xGe/NiSi_y compounds exhibited no intermixing between Ge and Si elements, and the phase of the reacted core is identified as Ni₂Ge with polycrystalline structures. The high compressive strain may benefit the hole-mobility for Ge/Si core/shell

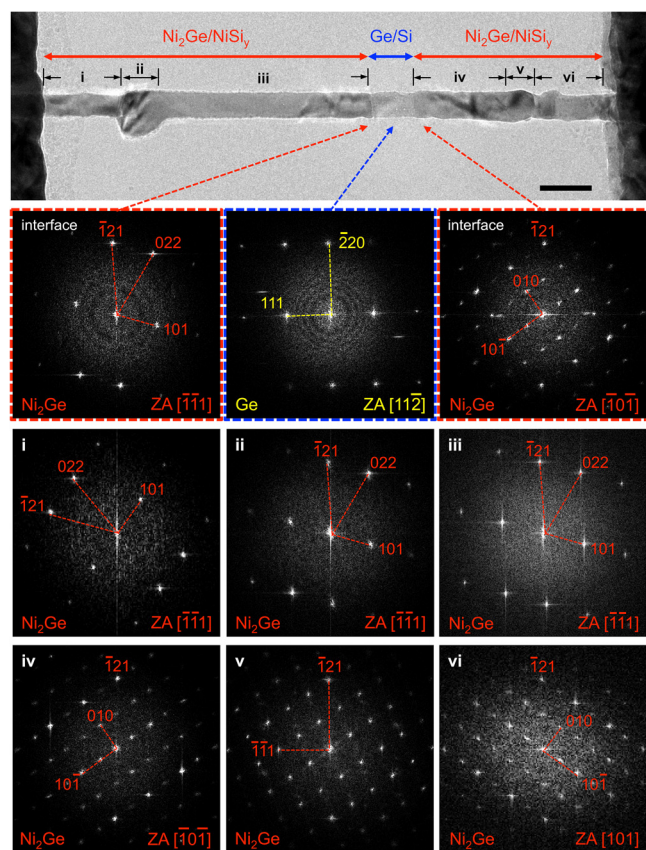


FIG. 5. TEM image of $\text{Ni}_2\text{Ge}/\text{NiSi}_3$ contact formation in a Ge/Si core/shell NW, and the fast-Fourier transform (FFT) images showing the diffraction patterns at different locations of the nanowire that have been labeled on the TEM image. The scale bar is 50 nm.

NW-FETs and the measured strain anisotropy could inform the design of sub 10 nm transistor channels.

See [supplementary material](#) for the TEM sample preparation methods and analysis of the germanide phase.

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