Size and Orientation Effects on the Kinetics and Structure of Nickelide Contacts to InGaAs Fin Structures

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Supporting Information

ABSTRACT: The rapid development of ultrascaled III–V compound semiconductor devices urges the detailed investigation of metal–semiconductor contacts at nanoscale where crystal orientation, size, and structural phase play dominant roles in device performance. Here, we report the first study on the solid-state reaction between metal (Ni) and ternary III–V semiconductor ($In_{0.53}Ga_{0.47}As$) nanochannels to reveal the reaction kinetics, formed crystal structure, and interfacial properties. We observe a size-dependent Ni surface diffusion dominant kinetic process that gradually departs to a volume diffusion process as the Fin width increases, as properly



depicted with our Fin-specific growth model. The interfacial relationship was found to be Ni₄InGaAs₂ (0001) || In_{0.53}Ga_{0.47}As (111) with a single Ni₄InGaAs₂ phase whose [0001] axis exhibit a peculiar rotation away from the nickelide/InGaAs interface due to surface energy minimization. This crystalline interfacial relationship is responsible for introducing a uniaxial height expansion of 33% \pm 5% in the formed nickelide segments. Further, the nickelide formation resulted in both in-plane and out-of-plane compressive strains in the Fin channels, significantly altering the In_{0.53}Ga_{0.47}As energy band-edge structure near the interface with a peak bandgap energy of ~1.26 eV. These timely observations advance our understanding and development for self-aligned contacts to III–V nanochannels and for engineering new processes that can maximize their device performance.

KEYWORDS: Indium gallium arsenide, Fin, size effect, orientation dependence, compound contact, strain

dvanced semiconductor devices that employ geometric **A**scaling for performance enhancement encounter size effects, which emerge at nanometer scales, and crystal orientation effects that can dominate their physical properties and govern their performance.^{1,2} An important aspect of ultrascaled devices is the phase transformation that accompanies the formation of metal-semiconductor compound contacts to semiconductor channels that has critical fabrication and low-resistance ohmic contact requirements.³ These compound (stoichiometric) and alloyed (nonstoichiometric) contacts are particularly important for new channel materials or materials with renewed interest in nanoscale architectures, such as indium gallium arsenide (InGaAs)⁴ in a Fin or nanowire geometry, which has been touted as a replacement channel material for sub-10 nm technology nodes.^{5,6} The development of ultrascaled InGaAs devices calls for a detailed investigation that can reveal the influence of crystal orientation and size effects on the kinetics of formation, phase, and structural characteristics of compound contacts to InGaAs Fin/nanowire channels.

Throughout the evolution of the Si complementary metaloxide-semiconductor (CMOS) technology, nickel silicide has been and continues to be the standard compound contact to Si and SiGe channels owing to its low processing temperature, low resistivity, superior scaling to sub-100 nm line widths, and good compatibility for self-aligned source/drain (S/D) contact

formation.^{7,8} On the other hand, compound and alloyed contacts to III–V channels have been well studied for planar geometries.^{9–13} Recent efforts fueled with the desire to develop a suitable self-aligned contact to III-V channels, analogous to saliside (self-aligned silicide) contacts to Si channels, have introduced the Ni-InGaAs compound contact, conventionally named as Nickelide, as a serious candidate.^{14,15} The nickelide contact has a number of attributes that satisfies the stringent requirements for S/D contact technology. First, the nickelide contact is reproducible and stable where the Ni and InGaAs reaction starts at \sim 230 °C, meeting a good thermal budget, and the formed phase is thermally stable between 350 and 450 $^\circ ext{C.}^{16}$ During the formation of crystalline nickelide, Ni is found to be the diffusing species, which provides the feasibility to form metal contact to ultrathin body devices with controlled Ni supply.¹⁷ Second, the nickelide contact possesses a low specific contact resistance $(4 \times 10^{-8} \ \Omega \text{-cm}^2)^{18}$ accompanied by a low sheet resistance $(20-25 \ \Omega/\text{square})$,^{19,20} and a low Schottky barrier height $(SBH)^{21-23}$ to the InGaAs channel. Third, the nickelide contact can be self-aligned to the S/D regions through the selective etching of excess Ni by concentrated HCl,²⁴ allowing

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Figure 1. Nickelide formation in $In_{0.53}Ga_{0.47}As$ Fin channels. (a) Schematic illustration of the fabrication procedure of $In_{0.53}Ga_{0.47}As$ Fin channels on insulator on Si. (b,c) SEM images illustrating the size-dependent and orientation dependent morphologies for nickelide contacts with $In_{0.53}Ga_{0.47}As$ Fin channels predefined in $\langle 110 \rangle$ and $\langle 100 \rangle$ orientations, respectively. Scale bars are 5 μ m. (d,e) SEM images illustrating the morphologies of formed nickelide contacts with $In_{0.53}Ga_{0.47}As$ planar films for comparison. Scale bars are 2 μ m. (f,g) AFM topography plots of nickelide–InGaAs Fin channels. Scale bars are 2 μ m. The brighter portions in the topography images are volume-expanded nickelide regions. (h,i) The height profiles for nickelide segments (red) and nonreacted $In_{0.53}Ga_{0.47}As$ segments (turquoise), corresponding to the color-marked line cuts in (f,g). The nickelide segments were found to have an increased height of $33\% \pm 5\%$. The height profiles do not represent real aspect ratios of each Fin structure, as the units of two axes are different. (j,k) The 3D topography plots further reveal that the volume expansion in nickelide segments is predominant in the direction normal to the substrate surface.

ease of processing to achieve ultrashort nm scale channel lengths.²⁵ At nanoscale dimensions with Fin and nanowire architectures, the nickelide phase, the epitaxial relationship with InGaAs layer, and the kinetic and thermodynamic effects that are size and orientation dependent are yet to be determined.

This work focuses on nickelide phase formation in In_{0.53}Ga_{0.47}As Fin structures under the influence of a rapid thermal annealing (RTA) process. We observed a strong dependence of the morphology of the formed nickelide segments on the Fin orientation as well as the Fin width. Through systematic studies of different annealing times, temperatures, and Fin widths, we found that the nickelide contact growth behavior is diffusion-limited and developed a Fin-specific kinetic competition model that strongly agrees with our experimental findings. As the Fin width increased, we observed a transition from a surface-diffusion limited growth to a volume-diffusion

limited growth. We carried out detailed transmission electron microscopy (TEM) characterizations of the nickelide phase and interfacial crystalline relationship and developed a crystalline model that explains the uniaxial anisotropic volume expansion of the nickelide contacts observed in experiment. Further, we utilized the cross-sectional TEM images to determine the strain distribution in the channel and relate it to the energy band-edge profiles in the transport direction. These extensive kinetic and structural investigations provide a comprehensive framework to understand and control the formation of nickelide/InGaAs Fin contacts and may inspire new contact engineering opportunities to enhance transport in nickelide-contacted InGaAs channels.

Our studies started with a 50 nm $In_{0.53}Ga_{0.47}As$ film on insulator on Si substrate that is accomplished with a novel solidstate wafer bonding approach that we developed in our laboratory as schematically illustrated in Figure 1a (detailed

process in Supporting Information).²⁶ This integration approach can eliminate the substrate influence (reaction) during thermally driven compound contact formation to III-V channels, and additionally provides a platform to complement and combine the high-performance III-V devices with low-cost Si circuitry underneath. The Fin structures are patterned on top of the In_{0.53}Ga_{0.47}As layer utilizing a 100 kV e-beam writer (JEOL JBX-6300FS) with beam size ~ 10 nm. Negative e-beam resist, hydrogen silsesquioxane (HSQ XR-1541-004), was used as the etch mask for a Cl_2/N_2 composite reactive ion etch (RIE)/ inductive coupled plasma (ICP) etch of the In_{0.53}Ga_{0.47}As Fin channels. After the RIE/ICP etching step, HSQ atop the Fins is removed with three consecutive cycles of O₂ plasma treatment followed by a short diluted HF dip, which also reduces the plasma-induced surface damage and smoothens the InGaAs surface. During this surface conditioning process, the height of In_{0.53}Ga_{0.47}As Fin was found to reduce from 50 to 40 nm. A 200 nm Ni film was then evaporated at the two ends of the Fin structures. The nickelide formation was controlled by RTA in the temperature range from 250 to 300 °C. The surface topographical measurements were performed with an atomic force microscope (AFM, DI Veeco), and the crystal structure of nickelide phase was characterized by an FEI Tecnai F30 transmission electron microscope (TEM). Cross-sectional lamellas across the Fin length and Fin cross-section were prepared with focused ion beam (FIB, FEI Nova 600) milling and Ar ion post cleaning (Fischione Model 1010 ion mill).

Proper investigation of the Nickelide formation mandated the fabrication of the InGaAs Fins in fixed crystallographic orientations. During the wafer-bonding step, the (001) In_{0.53}Ga_{0.47}As/InP was aligned with (001) Si substrate along the cleavage edges that are $\langle 110 \rangle$ oriented. The Fin structures were then defined by EBL with $\langle 110 \rangle$ orientation parallel to the cleavage edge and $\langle 100 \rangle$ orientation rotated 45° with respect to the cleavage edge (Supporting Information Figure S2). These orientations were further validated by detailed TEM characterization, where the misalignment to these crystallographic orientations were within an upper limit of $\pm 1^{\circ}$ for all investigated samples. For both orientations, the Fin heights were 40 nm and Fin widths were varied from 30 to 500 nm with 10 nm increment for sub-100 nm Fins and 50 nm for Fins with 100-500 nm widths. Figure 1b,c shows scanning electron microscope (SEM) images of the Ni on In_{0.53}Ga_{0.47}As Fins subjected to an RTA treatment at 250 °C for 20 min for $\langle 110 \rangle$ and $\langle 100 \rangle$ orientations, respectively. Figure 1d, e shows SEM images of reference samples fabricated also in $\langle 110 \rangle$ and $\langle 100 \rangle$ orientations, respectively. It can be readily observed from Figure 1b,c that the nickelide extension into the In_{0.53}Ga_{0.47}As channels is strongly dependent on the Fin width and that the nickelide and In_{0.53}Ga_{0.47}As segment widths are similar (no lateral nickelide volume expansion). In addition, the nickelide interface is flat for a $\langle 110 \rangle$ orientation and is rough for $\langle 100 \rangle$ orientation for both In_{0.53}Ga_{0.47}As Fins and planar layers (Figure 1d,e). AFM topographical measurements in Figure 1f,i show that the volume expansion is predominant in the vertical (001) direction normal to the substrate surface with a height increase from 40 nm in unreacted regions to around ~50-56 nm in reacted nickelide regions. The rough nickelide-InGaAs Fin interface is clearly visible in the AFM topography in Figure 1g. The 3D AFM topography image in Figure 1j,k further illustrates the features discussed above. The origin of these dependencies will be further elucidated with the kinetic and structural characterization of nickelide reacted Fins.

To delve into the details of the Ni and InGaAs reaction, we systematically studied the kinetics of nickelide formation as a function of time (2–95 min) and Fin width (30–500 nm) for $\langle 110 \rangle$ and $\langle 100 \rangle$ orientations. The measurements for nickelide lengths and widths were performed post RTA by SEM on 10 Fin sets per single data point to attain average and standard deviation lengths. This procedure was performed multiple times on the same sample for different reaction times in order to avoid sample-to-sample variations. The time-dependent nickelide segment lengths are shown in Figure 2a,b with three selected Fin widths



Figure 2. Time-dependence of nickelide phase formation. (a,b) The length of nickelide segments versus annealing time at 250 °C for $\langle 110 \rangle$ and $\langle 100 \rangle$ Fin orientations, respectively. In both orientations, the data were well fitted with $t^{1/2}$ dependencies. The insets display $L_{\text{Nickelide}}(t)$ for short reaction times where the nonzero intercept indicates an incubation time that is also size dependent. (c,d) $L_{\text{Nickelide}}(t^{1/2})$ at three different temperatures for 30 and 500 nm Fin widths, respectively. In all cases, the nickelide segment is longer in the $\langle 110 \rangle$ orientation.

and the reference planar film. In both $\langle 110 \rangle$ and $\langle 100 \rangle$ oriented Fins, the nickelide phase grew faster in narrower Fins and slowest in the planar film. Despite the slightly larger deviation in measurements for 30 nm Fin and at a fixed temperature of 250 $^{\circ}$ C, the nickelide length as a function of time, *t*, is well described by a hyperbolic $(t^{1/2})$ dependence as shown in Figure 2a,b. This is further validated at three different temperatures (250, 270, and 300 °C) in Figure 2c,d where the nickelide lengths versus $t^{1/2}$ for the narrowest (30 nm, Figure 2c) and for the widest (500 nm, Figure 2d) displayed linear characteristics for both orientations with a slightly longer nickelide segments for $\langle 110 \rangle$ oriented Fins. This $t^{1/2}$ dependence of nickelide growth is characteristic of Nidiffusion limited growth and was previously observed in nickelide formation in InAs nanowires,²⁷ in GaAs thin films,²⁸ and in nickel silicidation in Si nanowires.^{29,30} The insets of Figure 2a,b demonstrate an incubation time that is also size dependent for In_{0.53}Ga_{0.47}As Fins and the absence of an incubation time for planar reference films indicating different diffusion paths for the two cases. Next, we will validate that the nickelide growth is surface-diffusion limited in In_{0.53}Ga_{0.47}As Fins that should explain

the faster incubation times for smaller Fins (surface collection area/growth cross-section increases as Fin width decreases) and that volume diffusion of Ni into the growth front in planar films should not encounter any significant incubation time.

The top-down processed Fin structures in our studies present an ideal platform for studying compound metal contact formation with nanoscale channels at precisely defined widths and crystallographic orientations, which is in contrast to lesser control over such parameters in devices made on vapor-liquidsolid grown nanowires that have been subject to similar studies. For instance, depending on whether or not the Ni pads are in intimate contact with the Si nanowire surface, the kinetics of nickel silicide growth could be dominated by Ni source supply or by Ni diffusion along the silicide segments.³¹ The size of nanowires was also found to influence the reaction kinetics, as the diffusion limited process takes over interfacial-reaction limited one in smaller nanowires at elevated temperatures.³² Even in the Ni diffusion dominant kinetic process, debate still exists in whether Ni diffusion is primarily along the nanowire surface or through its body.^{33,34}

To shed light on the nickelide formation mechanism, we extended previous kinetic models for the reaction of Ni with Si nanowires^{3,30} and took into account our Fin-specific rectangular structures and the incurred volume expansion in reacted regions. As shown in Figure 3, the mass transport of Ni atoms during the



Figure 3. A schematic illustration of the rate-limiting processes involved in nickelide formation in InGaAs Fin channel. F_1 is the flux of Ni dissolved from the Ni reservoir to the Fin channel with an effective length, L_{bv} beneath the Ni pad. F_2 represents the flux of Ni diffusing through the reacted nickelide segment. F_3 is the flux of Ni that will react with fresh InGaAs at the nickelide/InGaAs interface. Because of volume expansion to the Fin channel, the height of InGaAs Fin increases from hto H after nickelide formation. Negligible lateral expansion was observed from AFM measurements, so the Fin width is fixed here as w.

nickelide growth involves three steps: (1) Ni dissolution across the Ni/nickelide interface, (2) Ni diffusion along the formed nickelide segment, and (3) Ni and InGaAs reaction at the nickelide/InGaAs interface. The fluxes of Ni atoms in the above three processes can be expressed as

$$F_{\rm l} = k_{\rm dissolve} (C_{\rm Ni/Nickelide}^{\rm eq} - C_0) (w + 2H) L_{\rm b}$$
(1)

$$F_{2} = -D_{\text{Ni}} \frac{C_{\text{L}} - C_{0}}{L_{\text{Nickelide}}(t)} X$$
$$X = \begin{cases} Hw & \text{Volume Diffusion} \\ 2(H+w)\delta & \text{Surface Diffusion} \end{cases}$$
(2)

$$F_3 = k_{\text{growth}} (C_{\text{L}} - C_{\text{Nickelide/InGaAs}}^{\text{eq}}) hw$$
(3)

where k_{dissolve} and k_{growth} are the interfacial reaction rate constants for Ni dissolution into nickelide and for nickelide growth at the reaction front with InGaAs, respectively. At these two interfaces, $C_{\text{Ni/Nickelide}}^{\text{eq}}$ and $C_{\text{Nickelide/InGaAs}}^{\text{eq}}$ denote the equilibrium Ni concentrations. C_0 and C_{L} are the equilibrium Ni concentrations inside the formed nickelide segment at zero-length position and at a reacted-length position, $L_{\text{Nickelide}}(t)$. The flux of Ni atoms diffusing along the formed nickelide segment, F_2 , depends not only on the diffusion coefficient of Ni species, but also on the diffusion cross-section X. The diffusion cross-section describes the diffusion path of Ni atoms, with Hw for volume (bulk-like) diffusion and $2(H + w)\delta$ for surface diffusion, where δ is the thickness of high-diffusivity surface layer, taken conventionally to be one atomic layer high.

By solving the steady-state equation, $F_1 = F_2 = F_3 = F$, F is given by

$$F = \frac{C_{\text{Ni/Nickelide}}^{\text{eq}} - C_{\text{Nickelide/InGaAs}}^{\text{eq}}}{\frac{1}{k_{\text{dissolve}}(w + 2H)L_{b}} + \frac{L_{\text{Nickelide}}(t)}{D_{\text{Ni}}X} + \frac{1}{k_{\text{growth}}hw}}$$
(4)

The three terms in the denominator represent three ratelimiting mechanisms. In order to solve this equation, the mass conservation of Ni atoms should be considered as follows:

$$Hw \frac{dL_{\text{Nickelide}}(t)}{dt} = F \frac{M_{\text{Nickelide}}}{N_{\text{A}}\rho_{\text{Nickelide}}}$$
(5)

If these three rate-limiting mechanisms are separately considered, the solutions are given in Table 1 (detailed

Table 1. Nickelide Growth in InGaAs Fin Channels for	
Different Rate-Limiting Steps According to Equations 1-	-5

rate-limiting step	conditions	solution
Ni source supply limit	$k_{ m dissolve} \ll k_{ m growth}, D_{ m Ni}$	$L_{\text{Nickelide}}(t) = k_{\text{dissolve}} \left(\frac{1}{H} + \frac{2}{w}\right) L_{\text{b}} P t$
surface-diffusion limit($X = 2(H + w)\delta$)	$D_{ m Ni} \ll \ k_{ m dissolve}, \ k_{ m growth}$	$L_{\text{Nickelide}}(t) = \sqrt{4PD_{\text{Ni}}\delta\left(\frac{1}{w} + \frac{1}{H}\right)} \cdot t^{1/2}$
volume-diffusion limit (X = Hw)	$D_{ m Ni} \ll k_{ m dissolve}, \ k_{ m growth}$	$L_{\text{Nickelide}}(t) = \sqrt{2PD_{\text{Ni}}} \cdot t^{1/2}$
interfacial reaction limit	$k_{ m growth} \ll k_{ m dissolve}, \ D_{ m Ni}$	$L_{\rm Nickelide}(t) = k_{\rm growth} \frac{h}{H} P t$
[*] Here, $P = M_{\text{Nickelide}}$	$(C_{\text{Ni/Nickelide}}^{\text{eq}} -$	$C_{\text{Nickelide/InGaAs}}^{\text{eq}})/(N_{\text{A}}\rho_{\text{Nickelide}}).$

derivation in Supporting Information). From Table 1, we can find that in both Ni source supply limit and the interfacial kinetic reaction limits, the length of nickelide segment $(L_{\text{Nickelide}})$ is linearly proportional to the annealing time t, which contradicts the experimentally observed $t^{1/2}$ dependence in Figure 2. This suggested that the nickelide growth witnessed here in InGaAs Fin channels is dominated by the diffusion step of Ni along the formed nickelide segment. Because the volume-diffusion limited growth does not incur Fin geometrical terms in contrast to our observation of faster nickelide growth in narrower Fins (Figure 1), we conclude that Ni diffuses along the surface of the nickelide segment in the Fin channels, as precisely portrayed in the solution for surface-diffusion limited growth dependence in Table 1.

To further validate the surface-diffusion limited process, the length of the nickelide segment must follow a linear dependence as a function of $(1/w + 1/H)^{1/2}$ and was shown to be the case at 250 °C in Figure S3 (Supporting Information) for all annealing times (5, 15, 25, 45, 65, and 95 min) and for both Fin orientations. *H* here was fixed at 53 nm, which was measured as the average height of the reacted nickelide segment. However, for Fin widths larger than 100 nm in $\langle 110 \rangle$ orientation, a second slope



Figure 4. Combined plots of nickelide segment length in relation to annealing time and Fin geometrical factors, and extracted kinetic parameters. (a,b) $L_{\text{Nickelide}}$ versus $(t)^{1/2}(1/w + 1/H)^{1/2}$ according to the surface-diffusion limited model at three different growth temperatures and with Fin orientations of $\langle 110 \rangle$ and $\langle 100 \rangle$ respectively. The data points in black color are nickelide growth in $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ thin films, and they were normalized by the factor of $(1/H)^{1/2}$ by considering the planar film has an infinite width $(1/w \rightarrow 0)$. The extracted effective surface-diffusion coefficients D_{Surface} were plotted in semilogarithmic scale versus inverse temperature to calculate the activation energy of nickelide formation for (c) $\langle 110 \rangle$ and (d) $\langle 100 \rangle$ oriented In_{0.53}Ga_{0.47}As Fin channels. The extracted volume-diffusion coefficients D_{Volume} were plotted in semilogarithmic scale versus inverse temperature to calculate the activation energy of nickelide formation for (c) $\langle 110 \rangle$ and (d) $\langle 100 \rangle$ oriented In_{0.53}Ga_{0.47}As thin films. In these plots, the units for D_{Surface} and D_{Volume} are nm³/s and nm²/s, respectively.

Table 2. Calculated Effective Surface-Diffusion Coefficients D_{Surface} , Excluding the Geometric Factor $(1/w + 1/H)^{1/2}$, and Volume-Diffusion Coefficients D_{Volume} at Three Different Temperatures

coefficient	orientation	250 °C	275 °C	300 °C		
$D_{\rm Volume}~(10^{-12}~{ m cm}^2/{ m s})$	film $\langle 110 \rangle$	2.59	7.97	28.9		
	film $\langle 100 \rangle$	2.24	6.36	23.8		
$D_{\rm Surface} (10^{-17} {\rm cm}^3/{\rm s})$	Fin $\langle 110 \rangle$	$2.88(1.54 \times 10^{-11} \text{ cm}^2/\text{s})^a$	$6.25 (3.33 \times 10^{-11} \text{ cm}^2/\text{s})^a$	24.9 $(13.3 \times 10^{-11} \text{ cm}^2/\text{s})^a$		
	Fin $\langle 100 \rangle$	$2.42 (1.35 \times 10^{-11} \text{ cm}^2/\text{s})^a$	$5.53 (2.95 \times 10^{-11} \text{ cm}^2/\text{s})^a$	20.2 $(10.8 \times 10^{-11} \text{ cm}^2/\text{s})^a$		
The values in parentheses are calculated diffusion coefficients for 30 nm fins at corresponding appealing temperatures						

^aThe values in parentheses are calculated diffusion coefficients for 30 nm fins at corresponding annealing temperati

with linear dependence was observed (Figure S3, Supporting Information). The slope differences became more eminent across the two segments of linear fitting for annealing times longer than 25 min suggesting that certain factors encumbered the nickelide growth in larger Fin widths that resulted in an overall slower growth than a purely surface-diffusion limited process.

In order to better understand the kinetic process accompanying the nickelide formation for all Fin widths, the length of nickelide segment is plotted against both the time and geometrical factors in Figure 4. Accounting for both geometric and time dependencies, all experimentally measured data for different annealing times and at a single temperature can be linearly fitted for $\langle 110 \rangle$ (Figure 4a) and $\langle 100 \rangle$ (Figure 4b) orientations validating the surface-diffusion dominant kinetic process. The nonzero intercept with the *x*-axis indicates an average incubation behavior of all Fins with 250 °C thermal treatment. However, larger Fin nickelide lengths (left-side data points of each data set, color-labeled) fell below the linear trend, indicating a deviation from the surface-diffusion limited model. Interestingly, these deflected data points gradually extend approaching nickelide lengths obtained from growth in In_{0.53}Ga_{0.47}As thin films (black squares in Figure 4a,b). Because metal diffusion in crystalline thin films occurs via interstitial diffusion process (volume-like diffusion),³⁵ the behaviors of nickelide formation in larger Fins could be described as a gradual deviation from surface-dominant to volume-dominant diffusion limited growth process.

The effective surface-diffusion coefficient with Fin-structure measurements and volume-diffusion coefficient with thin-film measurements were extracted from Figure 4a,b and summarized in Table 2. One can note from the equation of nickelide growth (Table 1) that the unit for effective surface-diffusion coefficient is cm³/s, which excludes the geometric factor, $(1/w + 1/H)^{1/2}$. For the surface-diffusion coefficients in 30 nm Fins, the values are about one-order of magnitude higher than volume-diffusion coefficients. Moreover, when calculating the surface-diffusion coefficients with infinite Fin width (i.e. $(1/w + 1/H)^{1/2} \rightarrow (1/H)^{1/2})$, the values are still twice as large as those in planar films, validating a distinct Ni diffusion mechanism between Fin structures and thin films. The activation energies were then extracted by using the Arrhenius relationship, $D \propto e^{-E_a/kT}$, for

both Fin structures and planar films and in both $\langle 110 \rangle$ and $\langle 100 \rangle$ orientations (Figure 4c–f), where E_a stands for the activation energy, k for Boltzmann's constant, and T is the diffusion temperature. In planar In_{0.53}Ga_{0.47}As films, the calculated activation energy is about 10% larger than in In_{0.53}Ga_{0.47}As Fins.

With the comprehensive analysis of the kinetics in nickelide formation in $In_{0.53}Ga_{0.47}As$ Fin channels, a deep understanding of the crystalline structure of formed nickelide phases and their interfaces with InGaAs and resultant strains can have far-reaching implications for device performance that utilize such compound contacts. The different crystal phases of compound or alloyed contacts can (1) alter the Schottcky barrier height and contact resistivity;^{36–38} (2) strongly influence the reliability of contact properties,^{39,40} and (3) induce strain during contact formation that significantly impact the band structure and carrier mobility of the channel material.^{41–43} The contact formation in nanoscale channels was proven to be different from that in planar devices^{3,32,44–46} and can be more sensitive to local microstructures, such as crystalline defects⁴⁷ and gate-dielectric layer coatings.^{45,48}

Here we carried out the structural analysis for nickelide phase in In_{0.53}Ga_{0.47}As Fin channels and their interfacial correlation. The TEM sample lamellas were prepared with conventional FIB milling with an in situ lift-out (INLO) process⁴⁹ (details in Supporting Information), followed by post Ar-ion cleaning that was found essential to remove the FIB damaged surface³⁰ for clearer imaging. The FIB cut was performed on a 500 nm (110)oriented Fin channel, which displayed a flat interface between nickelide and InGaAs segments. As shown in Figure 5a, the TEM lamellae of InGaAs was cut along the Fin channel, and the corresponding high-resolution TEM (HRTEM) image is shown in Figure 5b. The InGaAs phase with zinc blende (ZB) crystal structure was confirmed to be In_{0.53}Ga_{0.47}As (epitaxially grown on InP) by measuring the lattice constant from fast Fourier transform (FFT) patterns of the HRTEM images. The HfO₂ layer underneath the In_{0.53}Ga_{0.47}As was found to have monoclinic lattice structure after the thermal process of our wafer bonding at 400 °C. The polycrystalline nature of this HfO₂ layer did not seem to induce strain in the $In_{0.53}Ga_{0.47}As$ Fin channels, as no changes of lattice contact for In_{0.53}Ga_{0.47}As has been detected near the interface with HfO2, which is similar to the case of polycrystalline HfO₂ formed on Si⁵¹ and GaAs.⁵² In order to identify the unknown crystal structure of nickelide phase, the TEM lamellas were cut both along the Fin channel (Figure 5c) and perpendicular to the Fin channel (Figure 5d). The nickelide phase was identified as hexagonal Ni₄InGaAs₂ (the slightly stoichiometric difference between In and Ga is not denoted here), where the measured lattice constant agreed well with those reported for Ni reaction with (001) In_{0.53}Ga_{0.47}As film on InP wafer.^{53,54} This Ni to As stoichiometric ratio (2:1) was also reported in previous studies of Ni reaction with bulk GaAs.^{39,55} The measured lattice constants of In_{0.53}Ga_{0.47}As and Ni₄InGaAs₂ are summarized in Table S1 (Supporting Information), and the volume expansion after Ni₄InGaAs₂ phase formation based on mass conservation was calculated to be \sim 34.2%. This estimated volume expansion agrees well with the height change demonstrated by AFM measurements (Figures 1f,g) and further validates that the volume expansion is dominant in the vertical direction, the physical origin of which be discussed in detail next.

Unlike multiple silicide phases coexisting during the Ni reaction with Si nanochannels, the nickelide segment exhibited a single composition during the Ni and InGaAs solid-state reaction, as shown in Figure 6. Interestingly, the nickelide crystal



Figure 5. Crystal structure analysis of In_{0.53}Ga_{0.47}As and nickelide phases. (a) Schematic illustration of the relative positions of FIB cut lamellas for panels b–d. (b) HRTEM image at the cross-section of the channel for In_{0.53}Ga_{0.47}As/HfO₂ and corresponding FFT patterns. The In_{0.53}Ga_{0.47}As composition was confirmed by the lattice constant estimation from the FFT pattern, and the polycrystalline HfO₂ was found to be monoclinic structure. (c,d) HRTEM images of nickelide phase with FIB cut lamellas (c) along Fin orientation and (b) perpendicular to the Fin orientation. The nickelide phase was identified as Ni₄InGaAs₂ with (0001) plane perpendicular to the Fin orientation. Scale bars are 2 nm for all HRTEM images and 5 nm⁻¹ for all FFT images.

undergoes a gradual rotation of the Ni₄InGaAs₂ crystal from the reaction interface toward the Ni reservoir. At the nickelide/ InGaAs interface, the Ni₄InGaAs₂ [0001] direction is aligned with the In_{0.53}Ga_{0.47}As [111] direction. Far away from the interface, the Ni₄InGaAs₂ [0001] direction is parallel to the substrate surface and is in the same direction of the [011] Fin channel. The crystal rotation of Ni₄InGaAs₂ phase happens within about 200 nm near the nickelide/InGaAs interface, and undergoes a gradual change with defective crystallites (FFT pattern shows continuous arc for each diffraction spot rather than the paired sharp spots for twinned structures). Throughout the whole range of nickelide segment, the zone axis was fixed as $[\overline{1}2\overline{1}0]$, with Ni₄InGaAs₂ $[\overline{1}2\overline{1}0] \parallel In_{0.53}Ga_{0.47}As [0\overline{1}1]$. On the basis of these observations, we speculate that the Ni₄InGaAs₂ phase growth starts with the energy preferred epitaxial planar interface of Ni₄InGaAs₂ (0001) \parallel In_{0.53}Ga_{0.47}As (111) as is the case for zinc blende or wurtzite III-V semiconductor nanowire growth interfaces⁵⁶⁻⁶¹ and as is reported for Ni reaction with



Figure 6. Cross-sectional TEM image of a [110] oriented Fin with nickelide formation. The FFT patterns were collected from different regions along the Fin structure. The nickelide FFT and $In_{0.53}Ga_{0.47}As$ FFT are well aligned (details in Figure 7) at the interface. But away from the interface, there is a gradual rotation of the crystal structure to maintain an equilibrium [0001] nickelide axis parallel to the substrate surface. Scale bar is 200 nm for the TEM image and 5 nm⁻¹ for all FFT patterns.



Figure 7. TEM characterization and structural analysis of the Ni₄InGaAs₂/In_{0.53}Ga_{0.47}As interface. (a) TEM image of the crystalline structure at the interface. (b) EDX elemental mapping of Ni, In, Ga, and As near the interface. A false Ga signal appears in HfO₂ due to the overlap of Ga (K_{α}) and Hf (L_{β}) bands. (c) HRTEM image at the position indicated in yellow square in (a) at the nickelide (dark contrast) InGaAs (bright contrast) interface. The yellow arrows indicate the layered growth of nickelide on a (111) plane of InGaAs from top surface toward interface with HfO₂. Corresponding FFT diffraction patterns show that the (0001) plane of Ni₄InGaAs₂ lies in parallel with (111) plane of In_{0.53}Ga_{0.47}As. The diffraction spots along 111 direction (green line), indicating the nonuniformity of the in-plane strain and out-of-plane strain. Scale bars are 2 nm for the HRTEM image and 5 nm⁻¹ for all FFT images. (d) Schematic illustration of the simple hexagonal lattice structure of Ni₄InGaAs₂ plane in parallel with In_{0.53}Ga_{0.47}As. The I interface and In_{0.53}Ga_{0.47}As. The (0001) Ni₄InGaAs₂ plane in parallel with In_{0.53}Ga_{0.47}As (111) plane at the interface, while the Ni₄InGaAs₂ (0001) plane in parallel with In_{0.53}Ga_{0.47}As (011) plane far away from the interface. This crystal rotation happens through the formation of a defective transition region. (f) The atomic arrangement in the interfacial plane for Ni₄InGaAs₂ and In_{0.53}Ga_{0.47}As, respectively. The hexagonal arrangement of As and In (or Ga) atoms are preserved after nickelide formation with slight decrease of the distance between two As (or In, Ga) atoms.

(111) GaAs wafers where Ni₂GaAs (0001) || GaAs (111) interfacial correlation was observed.^{39,40} During the continual nickelide formation in InGaAs Fin channels, the Ni₄InGaAs₂ away from the interface rotated due to energy-minimization reasons as follows. The rotated Ni₄InGaAs₂ crystal away from the interface has its (1010) plane as top surface, which is the preferred facet^{57,62} for nanoscale semiconductors with hexagonal

structure due to their low surface energy. Further, the [0001] growth axis perpendicular to the growth interface cannot be maintained in such orientation with respect to the $In_{0.53}Ga_{0.47}As$ channel due to the pinned supply of Ni atoms on the substrate surface (which is not possible to be normal to the slanted nickelide/InGaAs interface). The [0001] growth direction and (1011) facet formation lead to the nickelide segment undergoing

a postgrowth crystal rotation. The slanted nickelide/InGaAs interfaces were observed in all the prepared TEM samples, and identical morphologies of interfaces were found when nickelide formed from both ends of the Fin channel (Figure S6 Supporting Information). From Figure 7c, we deduce that the nickelide phase growth follows a layer-by-layer growth sequence from the top surface toward the bottom, as indicated by the yellow arrows in Figure 7c. This layer-by-layer growth mechanism, which is deduced in experiment to be from Fin top to bottom, leads to an outward volume expansion as observed in Figure 7a. These observations suggest that Ni diffusion along the free top surface is faster than that along the nickelide/HfO₂, which leads to a protruded nickelide segment at the top of the In_{0.53}Ga_{0.47}As channel with respect to that near the HfO₂ interface (see Figure 7c).⁶³

The composition of nickelide phase was characterized with the energy-dispersive X-ray (EDX) spectroscopy (Figure S7 Supporting Information) and with the elemental mapping under scanning TEM (STEM) mode (Figure 7b). Ni shows an abrupt composition change at the nickelide/InGaAs interface. HRTEM image of the interface (Figure 7c) further validates the Ni₄InGaAs₂ (0001) \parallel In_{0.53}Ga_{0.47}As (111) interface. Figure 7d,e shows the lattice structures for Ni₄InGaAs₂ and In_{0.53}Ga_{0.47}As and their crystallographic interfacial relationship. The Ni₄InGaAs₂ (0001) and In_{0.53}Ga_{0.47}As (111) interfacial planes have hexagonal atomic arrangements of both As and In (or Ga) atoms for both phases, and the distance between two As atoms in Ni₄InGaAs₂ is 5.6% smaller than that in In_{0.53}Ga_{0.47}As. This suggests that during the Ni reaction with In_{0.53}Ga_{0.47}As, the As and In (or Ga) atoms keep the close-packed (hexagonal) inplane arrangement, however, along the Ni₄InGaAs₂ [0001] || In_{0.53}Ga_{0.47}As [111] direction, the bonds between As and In (or Ga) are broken and those atoms are separately encaged by Ni atoms. Because of the minimal changes of atomic distances in the interfacial plane, while large separation of atoms is necessary to be perpendicular to the interfacial plane, the volume expansion mainly happens in the vertical direction normal to the substrate rather than in the lateral direction of Fin channels, as experimentally observed in Figure 1.

We turn now our focus to the in-plane $(\varepsilon_{\parallel})$ and out-of-plane (ε_{\perp}) strains that have direct influence on the electronic properties of the InGaAs channel. The magnitude and spatial distribution of strain near the InGaAs/nickelide interface shown in Figure 8a-d were obtained from HRTEM utilizing the geometrical phase analysis (GPA) tool⁶⁴ within the DigitalMicrograph(Gatan) package. For Figure 8a-d, the xaxis is selected to be along [111] direction, so the y-axis will be in parallel with the InGaAs (111) interfacial plane. Both of the two strain-tensor components of In_{0.53}Ga_{0.47}As, ε_{xx} and ε_{yy} exhibit negative values near the InGaAs/nickelide interface, indicating a quasi-hydrostatic compressive stress exerted to the In_{0.53}Ga_{0.47}As channel upon nickelide formation. This profile is also validated with another strain mapping, performed along the [011] channel direction (Figure S9, Supporting Information). The measured interplane spacing along the In_{0.53}Ga_{0.47}As [111] direction from FFT patterns (Supporting Information Figure S10) was used to calibrate the ε_{\parallel} by comparing these values with those measured far from the interface, and the interplane spacing along the $[\overline{1}11]$ direction was used to calibrate the ε_{\perp} . Both the ε_{\parallel} and ε_{\perp} were measured in multiple locations from top to bottom surface (with the averaged value shown as one data point in Figure 8e) and from the nickelide/InGaAs interface deep into the In_{0.53}Ga_{0.47}As channel. The ε_{\perp} , introduced by volume expansion, shows a clear



Figure 8. Strain induced band-edge shift in In_{0.53}Ga_{0.47}As Fin channels. (a) HRTEM image of a typical InGaAs/nickelide interface with the In_{0.53}Ga_{0.47}As (111) || Ni₄InGaAs₂ (0001) interfacial plane perpendicular to the x-direction. Scale bar is 10 nm. (b) The FFT pattern for In_{0.53}Ga_{0.47}As segment (left-side to the interface), and the highlighted two diffraction spots were used for strain mapping. (c,d) The mapping of two strain tensor components, ε_{xx} and ε_{yy} , respectively in In_{0.53}Ga_{0.47}As segment. (e) Out-of-plane and in-plane strains of In_{0.53}Ga_{0.47}As as a function of distance from the interface. Those values were calculated by comparing the interplane spacing from FFT diffraction patterns (as illustrated in Figure S10, Supporting Information) with the unstrained one. (f) The calculated band-edge energies as a function of distance along x-axis based on elastic theory that incorporates deformation potentials obtained from local density functional theory.⁶⁷ The estimated bandgap of In_{0.53}Ga_{0.47}As near the InGaAs/nickelide interface is ~1.26 eV in this case.

trend of relaxation when far from the interface. The ε_{\parallel} , caused by lattice mismatch, exhibits several fluctuations before decreasing to zero. From the trend of ε_{\parallel} , a compressive strain below 3% can be predicted at the nickelide/InGaAs interface. However, this value is smaller than 5.6% (the theoretical lattice mismatch between In_{0.53}Ga_{0.47}As and Ni₄InGaAs₂), which is likely to be relaxed by the sawtooth like steps present at the nickelide/ InGaAs interface and the three free surfaces of the In_{0.53}Ga_{0.47}As channel. Under compressive stress from both in-plane and outof-plane directions, the band structure of $In_{0.53}Ga_{0.47}As$ is significantly altered,^{65,66} and the strain-induced shift in bandedge energies can be calculated using well-known elastic theory that incorporates deformation potentials.⁶⁷ Figure 8f shows the calculated band-edge energies as a function of distance from the nickelide/InGaAs interface (detailed calculations in Supporting Information). The heavy hole (E_{hh}) and light hole (E_{lh}) bands split under the compressive strain with the $E_{\rm hh}$ slightly higher than $E_{\rm lh}$, which is different from the conventional cases of uniaxial and biaxial stresses where band anticrossing occurs. The E_{σ} gradually increases from the relaxed center of the channel (0.75)eV) toward the strained interface with a peak value of \sim 1.26 eV at the interface. Such an increase of $E_{\rm g}$ could lead to an increase in the electron effective mass and reduction of the injection velocities in ultrascaled sub-10 nm channels and may be detrimental for ohmic contact formation with InGaAs Fin channels due to the increased Schottcky barrier height (SBH) with a larger stress-induced bandgap of InGaAs. Possible strategies to mitigate these effects may include the development of heteroepitaxial layers⁶⁸ that exert tensile stresses on the Fins/

nanowires to zero-balance the strain, and through contact engineering that can allow the formation/piling of higher In composition in InGaAs near the contact region to compensate the stress effects and to lower $E_{\rm g}$.

In summary, this work presents the first detailed study of solidstate reaction between metal (Ni) and ternary III-V semiconductor $(In_{0.53}Ga_{0.47}As)$ nanochannels. Upon nickelide formation, the Fin channel showed a $33\% \pm 5\%$ height increase with negligible lateral expansion with a flatter interface for $\langle 110 \rangle$ oriented Fins compared to $\langle 100 \rangle$ ones. Characterization of the nickelide segment lengths as a function of time, temperature, and geometrical factors of InGaAs Fin channels revealed a sizedependent Ni surface diffusion dominant process during the nickelide formation in InGaAs Fin channels that gradually departs to volume diffusion as the Fin width increases. Detailed structural analysis identified the nickelide phase as Ni₄InGaAs₂ with Ni₄InGaAs₂ $[\overline{1210}] \parallel In_{0.53}Ga_{0.47}As [0\overline{11}]$ and Ni₄InGaAs₂ (0001) || $In_{0.53}Ga_{0.47}As$ (111) with a peculiar rotation of Ni₄InGaAs₂ [0001] axis away from the nickelide/InGaAs interface. The nickelide formation led to both in-plane and out-of-plane compressive strains on the Fin channels that opened up the InGaAs energy bandgap. We believe that these detailed timely observations are likely to benefit the development of selfaligned compound contacts to future electronic devices and inspire new compound contacts and device architectures that can enhance their performance.

ASSOCIATED CONTENT

Supporting Information

Additional details on In_{0.53}Ga_{0.47}As Fin structures fabrication and TEM samples preparation are described, together with diffusion model derivation, volume/surface diffusion component ratio, volume expansion ratio, and strain-induced energy band-edge shift after Ni₄InGaAs₂ phase formation. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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