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Lock-in Amplifiers up to 600 MHz





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ABSTRACT

The direct growth of III–V lasers on the industry-standard Si-platforms is the key for fully integrated Si-photonics. Conventional III–V hetero-epitaxy on Si substrates is plagued by crystalline defects generated at the mismatched interface, and thick buffers are often used to mitigate the issues. Here, we report the direct deposition of room temperature III–V micro- and nano-lasers on amorphous SiO₂ layers. Without the requirement of crystalline substrates nor having to cope with the long-lasting lattice-mismatch problem, we demonstrate the direct hetero-epitaxy of dislocation-free III–V nanopillars and micro-islands on amorphous SiO₂ layers. As the epitaxial InP crystals are embedded inside a low-index environment, we observed strong room temperature lasing from the as-grown InP crystallites under optical excitation.

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Targeting at Si-photonics manufacturing with lower cost and higher scalability, monolithic integration of III-V lasers on Si wafers via direct hetero-epitaxy has been extensively investigated in the last decade.^{1,2} The challenge lies at addressing the fundamental lattice/ polarity mismatches between III-V and Si to produce III-V single crystals with reduced dislocation densities and free of anti-phase boundaries (APBs).^{3,4} Promising laser results have been reported from III-V thin films grown on (001) Si using various defect management techniques.^{5,6} However, thick buffers needed for extended device lifetime and reliability post challenges in efficient coupling with Si-waveguides. Although these thick buffers are avoided in selectively grown III-V nanoridge lasers grown on pre-patterned Si substrates,⁷⁻¹¹ extending the III-V ridge dimension to micrometer scale for electrical injection may compromise the defect necking effect, leading to dislocation propagation into the active regions. While dislocation-free III-V nanowires can be selectively grown on Si, the use of metal catalysts, epitaxy on (111)-oriented substrates, and limited material volume complicate their integration with current Si-photonics.^{12,13} These aforementioned methods all adopt crystalline Si substrates for III-V hetero-epitaxy. The Si lattice guides the crystal phase and orientation of the epitaxial III-V alloys but, at the same

time, induces lattice mismatch and resultant crystalline defects inside the epitaxial III–V materials.³

Here, we present the deposition of III-V micro- and nano-lasers on amorphous SiO₂ layers using metal organic chemical vapor deposition (MOCVD). We discovered a catalyst-free growth scheme to circumvent the requirement for crystalline substrates and the inherent lattice/polarity disparity of III-V and Si and thereby enabled the selfassembly of dislocation/APB-free III-V crystals on amorphous SiO₂ through creation of preferential nucleation sites atop the SiO₂ layer. More significantly, these as-deposited III-V crystals are intrinsically embedded in a low-index environment and thus can sustain strong room temperature lasing behavior under optical excitation. Direct deposition of III-V lasers on the readily available SiO₂ layer greatly improves the flexibility in the design and epitaxy of III-V lasers on Si and facilitates light interfacing with Si-based optical components. With future growth site control, our preliminary results here offer an alternative approach for the integration of III-V lasers on Si and also highlight the possibility to integrate III-V functionalities on a variety of other substrates.

In the conventional selective area epitaxy of III-V compounds on pre-patterned Si substrates, the exposed crystalline Si surface acts as

preferential nucleation sites over the amorphous SiO₂ mask, and growth precursors diffuse from the amorphous oxide mask to the crystalline Si for hetero-epitaxy.^{14–16} We found that, with certain surface conditions combined with appropriate growth parameters, preferential III-V nucleation sites could be created on the SiO₂ surface. The key for growing crystalline III-V crystals on amorphous substrates is to generate well-separated nucleation sites and to prevent the aggregation of different nuclei into polycrystalline materials. Figures 1(a)-1(d) schematically depict the proposed growth mechanism of depositing III-V crystals on amorphous SiO2. Initially, the preferential nucleation sites on the SiO₂ surface capture the group III adatoms or nanoscale clusters during the surface diffusion process as illustrated in Fig. 1(a). The settled group III adatom/nanocluster then reacts with group V adatoms to form the primitive III-V nucleus on the amorphous SiO₂ surface [Figs. 1(b) and 1(c)]. This process should be performed at lower temperatures as a low surface diffusion rate of the group III adatoms eases the pivotal kink-capture process. Building from this III-V nucleus, III-V crystallites form atop the amorphous SiO₂ layer as shown in Fig. 1(d). Figure 1(e) schematically presents the formation of polycrystalline III-V on amorphous SiO2 when two adjacent III-V nuclei evolve and crystallites merge together. Continuous growth of these closely spaced crystallites eventually clusters into polycrystals with grain boundaries.

To investigate the growth mechanism of III–V hetero-epitaxy on amorphous SiO_2 layers, we prepared two samples. One sample is



FIG. 1. (a) Schematic showing the capture of group III adatom/nanoclusters by the preferential nucleation sites on amorphous SiO_2 . (b) Schematic showing the formation of the primitive III–V nucleus on amorphous SiO_2 . (c) Schematic showing the evolution of the III–V nucleus into crystals. (d) Schematic showing the direct growth of III–V crystals on the amorphous SiO_2 layer. (e) Schematic showing the coalescence of two InP crystals into poly-crystalline InP.

pre-patterned (001)-oriented Si substrates with crystalline Si openings and amorphous SiO2 growth masks. As shown by the schematic in Fig. 2(a), III-V hetero-epitaxy ideally only occurs atop the crystalline Si surfaces; however, under appropriate growth conditions, III-V growth might also initiate from some preferential nucleation sites on the amorphous SiO₂ mask. The other sample is the (001)-oriented Si substrate with a 500 nm thick thermal SiO₂ layer. In this case, III-V nucleates atop the SiO₂ layer and evolves into crystallites with different sizes and architectures as schematically depicted in Fig. 2(b). Before exploring the possibility to control the architecture and orientation of the deposited III-V crystals, we carried out a series of experiments to identify a growth scheme to create well-separated III-V nuclei, leading to single-crystalline III-V atop the amorphous SiO₂ layer. Prior to growth, the pre-patterned Si substrates with crystalline Si openings [the one in Fig. 2(a)] underwent a series of pre-treatments to obtain a fresh Si surface for III-V hetero-epitaxy.^{17,18} We dipped the sample into diluted HF solution (1%) for 30 s, then KOH solution (45% at 70 °C) for 90 s, and finally diluted HCl solution for 60 s. After being rinsed with DI water and blow-dried, the sample was immediately loaded into the MOCVD reactor (AIXTRON 200/4). Then, the sample was thermally annealed at 800 °C in a H₂ ambient for 15 min. Pre-treatments of the other sample with pure amorphous SiO₂ atop



FIG. 2. (a) Schematic showing the growth of single-crystalline InP on the amorphous SiO₂ mask during selective area growth. (b) Schematic showing the growth of single-crystalline InP on the pure amorphous SiO₂. (c) SEM image of InP nanoridges and crystals formed atop the pre-patterned Si substrates. The scale bar is $5 \,\mu$ m. (d) SEM image of InP crystals formed atop the amorphous SiO₂ layer. The scale bar is $5 \,\mu$ m. (e) Representative SEM images of InP pillars on amorphous SiO₂. The scale bar is 500 mm. (f) Representative SEM images of InP islands on amorphous SiO₂. The scale bar is 1.0 μ m.

[the one in Fig. 2(b)] are much simpler. The sample was cleaned with DI water, blown-clean by a nitrogen gun, and then loaded into the reactor for III-V hetero-epitaxy. We chose trimethylindium (TMIn) and tertiarybutylphosphine (TBP) as growth precursors and H2 as carrier gas. We selected the deposition of InP on SiO₂ to exemplify our proposed growth mechanism. For the pre-patterned Si sample with crystalline Si openings, we started by depositing an InP nucleation layer at 430 °C for 4 min with a V/III ratio of 1374. Then, the reactor temperature was ramped up to 630 °C in around 7 min. After reaching 630 °C, 1 min stabilization of the reactor temperature was incorporated before we continued the deposition of InP at 630 °C for 15 min, with a V/III ratio of 183. Next, we ramped the temperature down to 600 °C for the final InP growth for 45 min with a V/III ratio of 92. For the sample with pure SiO₂ atop, we began InP nucleation at 400 °C for 4 min with a V/III ratio of 917. A lower nucleation temperature was chosen to slow down the diffusion of the reactants and enhance the density of the InP crystallites. Then, we ramped up the temperature to 630 °C in a similar manner and subsequently continued the growth of InP at 630 °C for 40 min with a V/III ratio of 46. Note that, during temperature ramping and stabilization, keeping a TBP ambient without TMIn flow (material deposition) is essential. This two-step growth procedure, comprising a low temperature nucleation layer and a high temperature main layer, is critical for the formation of singlecrystalline InP on amorphous SiO2. Nucleating InP at lower temperatures often results in densely packed InP nuclei and subsequent coalescence into polycrystalline InP, while directly growing InP at high temperatures precludes InP nuclei formation atop the amorphous SiO₂ surface. Figure 2(c) displays a SEM image of InP crystallites grown on pre-patterned Si substrates with crystalline Si openings and amorphous SiO₂ masks. As expected, InP nanoridges grow from the exposed Si surfaces, along with InP crystallites formed atop the SiO₂ mask layers sparsely at various locations. This phenomenon suggests a strong affinity between the indium adatoms/nanoclusters and the preferential nucleation sites on the SiO2 surface. The nice faceting of the InP individual crystallites on SiO₂ signals the formation of a singlecrystalline semiconductor. Figure 2(d) presents a SEM image of InP crystallites grown on pure uniform SiO₂ layers. Well-separated and faceted InP crystals form atop the amorphous SiO₂ layer. We observed two different types of InP crystallites on the amorphous SiO₂ layer as illustrated by the SEM images in Figs. 2(e) and 2(f). One type is hexagonal InP nanopillar as showcased by the representative SEM images in Fig. 2(e). Interestingly, no tapering was detected from the deposited InP pillars. The other type is InP micro-island with various dimensions and architectures, and some typical ones are presented in Fig. 2(f). As expected, these self-assembled InP crystallites exhibit a random position and orientation. In general, the density of the InP islands with myriad shapes is larger than that of InP nanopillars with hexagonal cross sections. The primitive nucleation sites of the InP islands and pillars might be different and could somehow regulate the crystallite size and structure during the initial nucleation process.

To examine the crystalline structure of the InP crystallites and explore the nucleation mechanism, we prepared InP lamellas using focused ion beam (FIB) milling and inspected the specimens using transmission electron microscopy (TEM). A thin Pt layer was deposited atop the examined crystal to protect the epitaxial InP during the FIB process. Figure 3(a) shows a SEM image of the probed InP nanopillar, and the yellow dotted line denotes the direction of the TEM



FIG. 3. (a) SEM image of the inspected InP pillar. The scale bar is 500 nm. (b) Cross-sectional TEM image of the InP pillar. The scale bar is 500 nm. The inset shows the intermixing of zinc blende and wurtzite phases. The scale bar in the inset is 5 nm. (c) Zoomed-in TEM image of the InP pillar base, revealing an apparent surface kink. The scale bar is 10 nm. (d) SEM image of the inspected InP island. The scale bar is 500 nm. (e) Cross-sectional TEM image of the InP pillar base, revealing an apparent surface bar is 500 nm. (e) Cross-sectional TEM image of the InP island, showing a pure zinc blende phase. The scale bar is 500 nm. (f) Zoomed-in TEM image of the InP island base, revealing an apparent surface kink. The scale bar is 10 nm. (g) SEM image of the inspected InP micro-sheet. The scale bar is 5.0 μ m. (h) Cross-sectional TEM image. The scale bar is 200 nm. The inset presents a high resolution TEM image. The scale bar in the inset is 5 nm. (i) Zoomed-in TEM image of the InP sheet base, revealing an apparent surface kink. The scale bar is 5.0 μ m. (h) Cross-sectional TEM image of the InP sheet bar is 200 nm. The inset presents a high resolution TEM image. The scale bar in the inset is 5 nm. (i) Zoomed-in TEM image of the InP sheet base, revealing an apparent surface kink. The scale bar is 10 nm.

lamella. Figure 3(b) displays a global view of the InP pillar, and the insets reveal the detailed crystalline structure. The majority of the pillar exhibits a mixture of zinc blende and wurtzite phases, which is commonly observed in the selective area growth of III-V nanowires.¹⁹ Figure 3(d) presents a SEM image of the probed InP island, and the yellow dotted line indicates the direction of the TEM lamella. Figure 3(e) shows a global-view TEM image of the probed InP island. Despite a few stacking disorders, the majority of the InP island features a pure zinc blende phase. Figure 3(g) displays a SEM image of one InP micro-sheet deposited on the SiO₂ layer, and the yellow dotted line marks the direction of the TEM lamella. Figure 3(h) shows a globalview TEM image of the probed InP micro-sheet, and the inset presents a high-resolution TEM image. Similarly, the majority of the InP micro-sheet exhibits a pure zinc blende phase with a few stacking disorders originating from the oxide surface. Without the limitations of lattice mismatch, we observe no threading dislocations in the self-assembled InP nanopillars and micro-islands. Close inspection of the base of the InP crystallites reveals apparent surface kinks on the SiO₂ surface as illustrated by the high resolution TEM images in



FIG. 4. (a) Room temperature emission spectra of one InP nanopillar laser. (b) L–L curve of the probed nanopillar laser. The lower inset shows a SEM image of the probed nanopillar laser, and the upper inset shows an image of the device above threshold with distinctive speckle patterns. (c) Room temperature emission spectra of one InP micro-island laser. (d) L–L curve of the probed micro-island laser. The lower inset shows a SEM image of the probed micro-island laser, and the upper inset shows an image of the device above threshold with distinctive speckle patterns.

Figs. 3(c), 3(f), and 3(i). The depth of the surface kinks is around 4 nm, and the one at the pillar base appears to be slightly deeper and sharper than that at the island base. We detected this type of surface kink in most of the examined InP crystallites. We suspect that the surface kinks serve as preferential nucleation sites on the SiO₂ surface during the nucleation process and play a vital role in the shape and size of the final single crystalline InP. A copious amount of works in the literature have shown that surface terraces, textures, and kinks can function as preferred deposition sites in various CVD processes.^{20–25} Our preliminary result here suggests the possibility to control the position, orientation, and size of the single crystalline InP through deliberately introducing and manipulating the surface kinks atop the SiO₂ surface as nucleation sites.

Since the as-grown InP crystallites on SiO₂ are intrinsically embedded inside a low-index environment, spontaneously emitted light is tightly confined within the III-V crystals, amplifies with reflection from facets, and builds up into strong stimulated emission. We probed the lasing characteristics of the as-grown InP nanopillars and micro-islands using a home-built micro-photoluminescence system. Excitation was provided by a mode-locked Ti/sapphire laser with a wavelength of 800 nm, a pulse width of 100 fs and a repetition rate of 76 MHz. The light emission of the examined micro-lasers was collected and analyzed using a thermoelectric-cooled InGaAs detector. Figure 4(a) presents a representative emission spectrum of an InP nanopillar laser, and Fig. 4(b) shows the light-light (L-L) plot of the optically pumped device and emission intensity as a function of the excitation density. The lower inset of Fig. 4(b) displays a SEM image of the probed nanopillar laser. Under low excitation, we detected a broad spontaneous emission spectrum with a peak wavelength at around 900 nm, which further attests the phase mixture of the InP pillars. At high excitation levels, a sharp peak at 845 nm reaches threshold, intensifies, and protrudes from the background emission. The linewidth of the lasing peak is around 1.6 nm. The clear S-shape of the L-L curve manifests a strong lasing behavior, and a threshold of 58 μ J/cm² is extracted. The upper inset of Fig. 4(b) shows an image of the InP nanopillar laser above threshold with evident speckle patterns. Figure 4(c) plots a representative emission spectrum of an InP microisland laser. Under low excitation, we observed a broad spontaneous

emission spectrum with a peak wavelength around 920 nm, which corroborates the pure zinc blende phase of the InP islands. Under high excitation levels, a sharp peak at 895 nm reaches threshold, strengthens, and overshadows the background emission. The linewidth of the lasing peak is around 1.7 nm. Figure 4(d) shows the L–L curve of the micro-island laser, with a SEM image of the probed laser in the lower inset. The apparent S-shape of the L–L curve indicates a strong lasing behavior, and a threshold of 106 μ J/cm² is obtained. The upper inset of Fig. 4(d) shows an image of the InP micro-island laser above threshold with distinctive speckle patterns. We observed room temperature lasing behavior from a majority of the InP crystallites directly deposited on amorphous SiO₂ layers. Lower lasing thresholds are generally observed from the nanopillars because of the rabry–Pérot cavity and stronger carrier confinement induced by phase intermixing.²⁶

In conclusion, we demonstrated room temperature lasing from self-assembled InP nanopillars and micro-islands directly deposited on amorphous SiO₂ layers. Extensive TEM inspection suggests that the surface kinks atop the SiO₂ surface relate to the formation of the primitive III-V nuclei and guide the phase/shape of the epitaxial III-V crystals. The direct growth of III-V lasers on the easily accessible SiO₂ layers without the need of thick buffers and crystalline substrates resolves the inherent III-V/Si lattice mismatch issues that plague the community for decades. Our preliminary results unleash a strategy for integrating III-V lasers with Si-photonics. Deposition of crystalline III-V on thin amorphous layers also signals the potential to integrate III-V functionalities on a variety of substrates (with thin amorphous layers atop) otherwise unsuitable for III-V hetero-epitaxy. Future work includes controlling the position and structure of the surface kinks for steering the nucleation, architecture, and crystalline orientation of the single crystal III-V lasers on amorphous layers.

AUTHOR'S CONTRIBUTIONS

Y.H. and Z.Y. contributed equally to this work.

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The data that support the findings of this study are available from the corresponding author upon reasonable request.

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