Silicon-nitride-on-insulator (Si₃N₄) photonic circuits have seen tremendous advances in many applications, such as on-chip frequency combs, Lidar, telecommunications, and spectroscopy. So far, the best film quality has been achieved with low-pressure chemical vapor deposition (LPCVD) and high-temperature annealing (1200°C). However, high processing temperatures pose challenges to the cointegration of Si₃N₄ with pre-processed silicon electronic and photonic devices, lithium niobate on insulator (LNOI), and Ge-on-Si photodiodes. This limits LPCVD as a front-end-of-line process. Here, ultralow-loss Si₃N₄ photonics based on room-temperature reactive sputtering is demonstrated. Propagation losses as low as 5.4 dB m⁻¹ after 400°C annealing and 3.5 dB m⁻¹ after 800°C annealing are achieved, enabling ring resonators with highest optical quality factors of > 10 million and an average quality factor of 7.5 million. To the best of the knowledge, these are the lowest propagation losses achieved with low temperature Si₃N₄. This ultralow loss enables the generation of microresonator soliton frequency combs with threshold powers of 1.1 mW. The introduced sputtering process offers full complementary metal oxide semiconductor (CMOS) compatibility with front-end silicon electronics and photonics. This could enable hybrid 3D integration of low loss waveguides with integrated lasers and lithium niobate on insulator.

1. Introduction

Benefiting from the maturity, low cost, and scalability of complementary metal-oxide semiconductor (CMOS) manufacturing driven by the microelectronics industry, silicon integrated photonics has enabled significant advances in telecommunication,[1,2] Lidar,[3] integrated photonic sensors,[4] and quantum computing[5,6] over the past decade. Today, silicon photonics integrated circuits have been demonstrated with a wide diversity of optical functions via hybrid integration with different materials, such as III–V-on-Si integrated lasers, Ge-on-Si photodiodes, high-speed modulators, and (de)multiplexers. However, some intrinsic material properties of silicon pose challenges to the performance of silicon optical devices[7] in certain operation regimes. In particular, two-photon absorption of silicon at 1550 nm limits the ability of high-power handling and a high thermo-optic coefficient makes devices sensitive to temperature variations.

Silicon nitride (Si₃N₄) on insulator has attracted intensive interest as a dielectric material platform with ultralow propagation loss for photonic integrated circuits.[8] It has a wide transparency window covering visible to mid-IR, a relatively high nonlinearity (ten times higher than that of silica), and a low thermo-optical coefficient, as well as a CMOS-compatible manufacturing process. With its remarkable progress, it has enabled a wide range of applications in chip-scale frequency combs,[9] Lidar,[10] optical telecommunications,[11] spectroscopy,[12] on-chip delay lines,[13] sensing,[14] just to name a few. Among these applications—in addition to the requirement for low propagation loss—dispersion engineering and high confinement of optical modes is critical, which typically calls for film thicknesses of > 600 nm at telecom wavelengths. High Si₃N₄ thickness results in tight mode confinement in the waveguide core and offers various advantages, including negligible bending losses (small footprint), dispersion engineering capabilities and high effective Kerr nonlinearity. These characteristics are of great importance in nonlinear photonic applications. There are several methods to fabricate thick Si₃N₄ films, such as LPCVD,[15,16] plasma enhanced chemical vapor deposition (PECVD),[17–19] inductively coupled plasma chemical vapor deposition (ICP-CVD),[20] reactive sputtering[21,22] and atomic layer deposition (ALD).[23] So far, the best optical quality of Si₃N₄ films are deposited by high temperature (≥800°C) LPCVD.[8]
However, high film stress resulting from the LPCVD process leads to fatal cracks and prevents thick films (>400 nm) with high optical quality.\textsuperscript{[15]} As a result, several clever and sophisticated processes have been developed, such as patterning crack barriers,\textsuperscript{[15,24,25]} multi-step deposition,\textsuperscript{[15,24,25]} and a photonic Damascene process.\textsuperscript{[36]} In particular, prolonged high-temperature annealing (1200°C for >3 h) is indispensable to reduce the material loss and to achieve ultralow propagation loss (≈1 dB m$^{-1}$). This ultralow loss enables on-chip Si$_3$N$_4$ resonators with optical quality factors of >10 million.\textsuperscript{[24,27,28]} However, the high-temperature deposition and annealing processes can cause dopant diffusion and damages prefabricated temperature sensitive devices.\textsuperscript{[21,29,30]} Hence, LPCVD Si$_3$N$_4$ is very challenging to use for applications in back-end-of-the-line (BEOL) fabrication processes, co-integration with front-end-of-the-line (FEOL) silicon optoelectronic circuits, and foundry compatible processes.

In recent years, low-temperature deposited Si$_3$N$_4$ films have attracted increasing interest in 3D hybrid integration of Si$_3$N$_4$ with other materials.\textsuperscript{[18,21,29–32]} Among different low-temperature deposition methods, PECVD and ICP-CVD are widely used as low-temperature (<400°C), and CMOS compatible processes, providing high-thickness Si$_3$N$_4$ films (>1 μm) without cracks. However, the optical loss within the deposited films at telecommunication wavelengths is usually high (typically 200 dB m$^{-1}$) due to the absorption of Si–H and N–H bonds.\textsuperscript{[13]} which poses a challenge for optical applications. To address this issue, deuterated silane (SiD$_4$) has been used as the deposition precursor, rather than conventional SiH$_4$.\textsuperscript{[29,30,34–35]} showing a 10 dB m$^{-1}$ propagation loss. However, SiD$_4$ precursors are not commonly available in foundries and expensive, which could make this deposition method less attractive for large scale low-loss waveguide fabrication. The state-of-the-art propagation loss with a PECVD process without post thermal annealing is 42 dB m$^{-1}$, which was achieved recently by including a chemical mechanical planarization step.\textsuperscript{[36]} Another potential BEOL method is low-temperature reactive sputtering of low-loss Si$_3$N$_4$ film with low stress.\textsuperscript{[21,32,37]} Waveguide losses of 80 dB m$^{-1}$ at 1550 nm and microring resonators with an intrinsic quality factor of 6×10$^{5}$ have been achieved with thermal annealing at 400°C in ambient atmosphere.

Here, we report the realization of ultralow-loss Si$_3$N$_4$ photonic devices with high film thickness by reactive sputtering at room temperature. The optical propagation loss of the sputtered Si$_3$N$_4$ film is investigated at O-, C-, and L-band wavelength ranges and at different annealing temperatures. After room-temperature sputtering and waveguide fabrication, the Si$_3$N$_4$ film loss is 32 dB m$^{-1}$ without thermal annealing and the intrinsic optical quality factor (Q) of the fabricated microresonators is 1.1 million at 1580 nm. After a CMOS-compatible annealing step at a temperature of 400°C, the propagation loss is significantly reduced to 5.4 dB m$^{-1}$ and the resonator Q is improved to 6.2 million at 1580 nm. By additional annealing at 800°C the film quality is further improved, enabling losses of 3.5 dB m$^{-1}$, with the highest Q-factors beyond 10 million and an average Q-factor of 7.5 million. To demonstrate the viability of the resonators for photonic applications, we show parametric oscillations at threshold powers down to 1.1 mW at 1310 nm. As a further verification of the optical properties of the silicon nitride films, we demonstrate the generation of dissipative Kerr soliton combs centered at both 1.3 μm and 1.5 μm wave-lengths. We believe the low-temperature sputtered Si$_3$N$_4$ with ultralow optical loss will have a significant impact on scalable foundry photonics and hybrid integration of 3D photonic circuits.

2. Film Deposition and Waveguide Fabrication

Figure 1a shows the fabrication flow starting with a 4-inch silicon wafer with a 3 μm thick thermally oxidized silicon dioxide (SiO$_2$) layer. The Si$_3$N$_4$ film is sputtered at room temperature by a commercial reactive magnetron sputtering system equipped with an asymmetric bipolar pulsed DC source. A P-doped silicon target with a diameter of 2 inch is used as the sputtering source. Pulse power (100-W DC ) is used for the Si$_3$N$_4$ deposition with a pulse frequency of 50 kHz and a duty cycle of 75%. The sputtering process takes place within a plasma environment containing a mixture of Ar (argon) and nitrogen (N$_2$). Argon, with a constant flow rate of 15 sccm serves as the sputtering gas, facilitating the ejection of silicon atoms into the environment through high-energy collisions with the target surface.

Throughout the sputtering process, the chamber pressure is controlled at 3 mTorr and the temperature of the sample wafer is not stabilized and remains at ≈22°C (see Section S1, Supporting Information, for details). Note that the process is intrinsically hydrogen free, which avoids optical losses through N–H or Si–H bonds (see Section S2, Supporting Information, for details). Figure 1b shows the refractive index of the sputtered SiNx film at 636.4 nm as a function of Ar/N$_2$ ratio, together with a dashed line indicating the refractive index of stoichiometric Si$_3$N$_4$.\textsuperscript{[15]} Depending on the targeted refractive index, the Ar/N$_2$ ratio can be widely tuned, resulting in a variation of the SiNx refractive index from 2.07 (silicon rich) to 2.01 (nitride rich). The refractive index is measured at 636.4 nm with a prism coupler (Metronic 2010 m). For the fabricated samples presented here, the gas ratio is set to ≈2.7 for stoichiometric Si$_3$N$_4$, and the resulting refractive index is ≈2.04 at 636.4 nm with a deposition rate of 130 nm h$^{-1}$. Figure 1c shows the top surface roughness of the sputtered Si$_3$N$_4$ film with a thickness of 750 nm, measured by an atomic-force microscope. The root mean square roughness is 0.26 nm, which is on the same level as that of the substrate. The sputtered Si$_3$N$_4$ film is used to fabricate microring resonators to investigate the waveguide propagation loss by measuring the optical quality factor of the resonances and to explore potential applications in nonlinear photonics. A negative-tone photoresist (ma-N 2405) is used to pattern optical waveguides and microrings by electron beam lithography. After developing, the patterned samples are fully etched by inductive coupled plasma reactive ion etching with an etchant mixture of CHF$_3$ (50 sccm) and O$_2$ (5 sccm) at a pressure of 15 mTorr and DC bias of 240 V. Figure 1d shows a scanning electron microscope (SEM) image of a 100-μm-radius microring resonator with a waveguide cross-section of 750 nm × 1.8 μm. Figure 1e shows a zoomed-in SEM image of the ring waveguide with no visible sidewall roughness. The gap between the bus waveguide and ring is varied for different coupling conditions at different wavelength ranges. After the Si$_3$N$_4$ etching process, piranha solution is used to remove residual photoresist. Following this step, a 3-μm-thickness SiO$_2$ layer is deposited to encapsulate the samples via two steps. To fill the gap between the bus waveguides and microrings without air voids (see Section S3, Supporting Information, for SEM images of the gap region), a layer of SiO$_2$ with 400 nm thickness.
is deposited by ALD at a temperature of $45^\circ$C, followed by a 2.5-
$\mu$m-thickness SiO$_2$ layer deposited by PECVD at a temperature of
$300^\circ$C. Due to the tight mode confinement within the Si$_3$N$_4$
core, the PECVD SiO$_2$ layer does not interact significantly with
the optical mode, resulting in negligible losses while offering the
advantages of cost-effectiveness and a high deposition rate. The
samples are diced for end-fire coupling to characterize their optical
properties, such as optical $Q$, propagation loss, and dispersion of
microresonators.

3. Characterization of the Sputtered Silicon Nitride
Films and Microresonators

As shown in Figure 2a, two tunable continuous wave (CW) exter-
nal cavity diode lasers in the O-band and the C-/L-bands are used to
classify the optical transmission of the sputtered Si$_3$N$_4$
microresonators and generate dissipative Kerr micro-
combs. The polarization of the CW light is aligned by a fiber po-
arization controller to probe the quasi-transverse electric (TE) or
quasi-transverse magnetic (TM) modes of the Si$_3$N$_4$ resonators.
Light is coupled into and out of the resonators via two lensed
fibers. For low coupling loss from fiber to the waveguide, the
width of the Si$_3$N$_4$ waveguide is tapered down to $\approx$200 nm (see
Section S4, Supporting Information, for details). A coupling loss of
$< 2$ dB per facet is achieved in our experiments. For character-
ization of the resonators, both CW lasers are set to low optical
power to avoid resonance shifts induced by thermal effects
and the Kerr effect. The frequency of both CW lasers is widely
scanned across a range of 10 THz at a speed of $\approx 1$ THz $s^{-1}$.
To determine the laser frequency while scanning, we employ a
high-precision calibrated fiber loop cavity for providing frequency
markers.[38] The transmission spectra of the Si$_3$N$_4$ resonators and
reference fiber cavity are simultaneously recorded by an oscillo-
scope. Further data analysis is used to extract the optical quality
factors, propagation loss, and dispersion of the resonators.

3.1. Devices without Thermal Annealing

Figure 2b shows a resonance spectrum of a 200-$\mu$m-radius Si$_3$N$_4$
resonator at a resonant wavelength of 1318.7 nm, and Figure 2c
shows a resonance spectrum of another 200-$\mu$m-radius Si$_3$N$_4$
Figure 2. Characterization of sputtered Si₃N₄ resonators without post thermal annealing. a) Experimental setup for characterization of Si₃N₄ resonator transmission spectra, resonator linewidth, optical quality factors, and dispersion. Normalized transmission spectra of Si₃N₄ ring resonators without thermal annealing at 1318 nm b) and 1578 nm c). d) Intrinsic Q measurement of all resonances within the laser wavelength ranges.

resonator at a wavelength of 1578.6 nm. Both resonators are directly fabricated from a room-temperature sputtered film and with silica cladding layers but without post-thermal annealing. The dashed black lines in Figure 2b,c are fitted resonance profiles using extended coupled-mode theory, showing that the resonance linewidths at 1319 and 1579 nm are ≈ 300 and 200 MHz, respectively, which correspond to loaded quality factors (Qₐ) of 0.75 million at 1319 nm and 0.94 million at 1579 nm. Considering that both resonances are undercoupled, the calculated intrinsic quality factors (Qᵢ) are 1 million at 1319 nm and 1.1 million at 1579 nm, respectively. The propagation loss α can be estimated from Qᵢ using α = f₀/(Qᵢ×R×FSR), where f₀ is the resonance frequency, R is the radius of the ring resonator, and FSR is the free spectral range of the microresonator. Without post-thermal annealing, the fabricated sputtered Si₃N₄ can achieve a propagation loss of 42 dB m⁻¹ in the O-band and 32 dB m⁻¹ in the L-band. This propagation loss is already lower than that of annealing-free LPCVD Si₃N₄ films deposited at 780°C. Figure 2d shows the distribution of Qᵢ of the resonances within the measured wavelength range, with an average Qᵢ of 0.79 ± 0.11 million in the O-band and 0.94 ± 0.13 million across the C- and L-band.

3.2. Devices after 400°C Thermal Annealing

To reduce the material loss, the Si₃N₄ devices after SiO₂ encapsulation are post-processed with thermal annealing. First, we anneal the devices in an ambient atmosphere at 400°C for 8 h. 400°C processing temperature is chosen because it is CMOS compatible and BEOL process compatible, and can be used for the integration of Si₃N₄ with pre-processed CMOS electronics, III–V materials, and prefabricated photonic circuitry, such as lithium niobate on insulator, Ge-on-Si photodiodes, and active silicon modulators. Figure 3a,b shows the normalized resonance transmissions of two fundamental TE modes at resonant wavelengths of 1328 and 1610 nm of a 200-μm-radius Si₃N₄ resonator together with fitted line shapes (dashed lines). The loaded resonance linewidth has been dramatically narrowed to ≈ 55 MHz after 400°C annealing, compared to Figure 2b,c. Considering that
the device is designed for undercoupling across the measurement range, the calculated intrinsic $Q$s at 1328 and 1610 nm are 4.6 and 6.2 million, respectively, corresponding to a propagation loss of 9.0 dB m$^{-1}$ at 1328 nm and 5.4 dB m$^{-1}$ at 1610 nm. This is approximately a fivefold improvement compared to the propagation loss without thermal annealing. Figure 3c shows the distribution of the intrinsic $Q$s of the fundamental TE mode of the resonator under test covering the O- and C/L-band wavelength ranges. The intrinsic propagation loss in the O-band stays roughly constant with an average $Q$ of 3.8 ± 0.7 million. We observe some low $Q$ modes, which are a result of mode crossings (see Section S5, Supporting Information, for details). In comparison, the intrinsic loss in the C-band is slightly higher than in the L-band, which we speculate is due to residual losses in the material (see Section S2, Supporting Information, for details). An average $Q$ of 3.6 ± 1.3 million is measured across the C- and L-band. To the best of our knowledge, this is the lowest Si$_3$N$_4$ propagation loss reported with a peak processing temperature of 400°C.

3.3. Devices after 800°C Thermal Annealing

To further reduce the material loss of the sputtered Si$_3$N$_4$ film, we anneal the devices at a higher temperature of 800°C. This processing temperature can enable future integration of sputtered Si$_3$N$_4$ with silicon integrated photonics, such as Si modulators\cite{43} and Ge-on-Si photodiodes.\cite{44} After encapsulation, the devices are post-annealed at 800°C for 8 h. Figure 4a shows a normalized resonance transmission of a 200-μm-radius Si$_3$N$_4$ resonator at a resonance wavelength of 1319 nm where the resonance is close to critically coupled but still undercoupled, together with a fitted line shape (dashed line). The loaded resonance linewidth is $\approx$ 37.5 MHz. The calculated loaded and intrinsic $Q$s are 6.1 million and 9.2 million, respectively, corresponding to a propagation loss of 4.5 dB m$^{-1}$. The losses at 1319 nm are approximately halved compared with the losses after 400°C thermal annealing. Figure 4b shows the distribution of the intrinsic $Q$s of the fundamental TE mode of the resonator in different wavelength ranges. From Figure 4b, we note that the intrinsic $Q$ of some modes in the L-band is beyond 10 million, and the corresponding propagation loss is $< 3.5$ dB m$^{-1}$. See Section S6 (Supporting Information) for the normalized resonance transmission in L-band. Similar to Figure 2c, the intrinsic propagation loss in the O-band stays nearly constant with an average intrinsic $Q$ of 7.0 ± 1.8 million, except for a few low-$Q$ modes resulting from mode crossings. The intrinsic $Q$ distribution in the C- and L-bands is significantly different from the $Q$ distribution in Figure 3c. After 800°C thermal annealing, the intrinsic losses in the C- and L-bands are roughly constant as shown in Figure 4b. This shows that the material loss in the C-band has been effectively reduced by 800°C annealing. The average intrinsic $Q$ crossing the C- and L-bands is 7.5 ± 2.2 million. In Figure 5, we compare our results with other state-of-the-art low-temperature Si$_3$N$_4$ deposition methods with high film thickness (> 600 nm). To the best of our knowledge, this is the lowest Si$_3$N$_4$ propagation loss reported at a peak processing temperature of 800°C. To further verify the ultrahigh optical $Q$ of our Si$_3$N$_4$ ring resonators, we test the threshold optical power for
Kerr-nonlinear optical parametric oscillations (OPO). The threshold power is determined by measuring the optical power in one of the two generated optical sidebands when varying the on-chip optical power. Figure 4c shows experimental results when pumping an optical mode at 1312 nm in a 130-μm-radius ring resonator with a loaded $Q$ of 5.5 million and an intrinsic $Q$ of 9 million, showing a threshold power of $\approx 1.1$ mW. With an effective area of the pump mode $A_{\text{eff}} = 0.99 \mu\text{m}^2$ (obtained from a COMSOL Multiphysics simulation) and the Kerr nonlinear coefficient $n_2 = 2.4 \times 10^{-19} \text{m}^2 \text{W}^{-1}$, the calculated threshold power is 1.06 mW, agreeing well with the observed threshold power of 1.1 mW.

In addition to the OPO threshold power measurement, we employ a recently developed method\textsuperscript{45} to explore the material-loss-limited $Q$ factors in the sputtered microresonators after 800°C annealing. We conduct measurements to investigate the resonance shift in response to changes in the intracavity circulating optical power and microresonator temperature change. Moreover, we measure the resonance shift ratio between Kerr effect and photothermal effects. Based on results using finite-element method simulations (COMSOL Multiphysics), we have determined the material-loss-limited $Q$ factors of our resonators to be $\approx 20.5 \pm 1.9$ million after 800°C annealing at a wavelength of 1550 nm. See Section S7, Supporting Information, for detailed experiments and simulations. This value exceeds the presented intrinsic optical quality factor. Thus, we expect that a reduction of surface roughness can further reduce optical losses in our Si$_3$N$_4$ structures. This aligns with findings from existing literature\textsuperscript{27,45}.

Our measurements reveal that scattering losses impose a limitation of $\approx 10$ million on the optical quality factor. Given that the annealing process at lower temperatures does not effectively mitigate scattering losses, especially considering the relatively mild thermal treatment ($<400$°C) employed in our study, which is significantly lower than the Si$_3$N$_4$ softening temperature (1900°C), we assume that the quality factors at temperatures $<400$°C are primarily limited by material loss.
4. Soliton Frequency Comb Generation in Sputtered Si$_3$N$_4$

In this section, we use the fabricated Si$_3$N$_4$ microresonators for soliton frequency comb generation. We first measure the dispersion of a 100-μm-radius resonator with a cross-section of 750 nm × 1.8 μm. The resonance frequencies of a mode family in a resonator can be described with a Taylor series as

$$\omega_\mu = \omega_0 + D_1 \mu + \frac{D_2}{2!} \mu^2 + \cdots$$

where $\mu$ is the mode number offset from the center mode at $\mu = 0$ and $\omega_0$ are the resonance frequencies. $D_1/2\pi$ is the FSR of the resonator at the center mode ($\mu = 0$), and $D_2$ is the coefficient of second-order dispersion. $D_{\text{int}}$ is the integrated dispersion, depicting the deviation of resonance frequencies from the equidistant grid spaced by $D_2$. Figure 6a shows the measured integrated dispersion profile (blue circles) at a center mode of 1310 nm together with a second-order polynomial fitted curve (blue curve). The dispersion profile shows anomalous dispersion with mode crossings at 1280 and 1326 nm. Figure 6b shows the transmission traces when scanning the 1.3-μm pump laser frequency from the blue-detuned to the red-detuned side of the resonance. Different soliton steps, corresponding to different soliton numbers, are observed in the red-detuned regime (green shaded area).

Using the passive temperature stabilization with the auxiliary laser at 1.55 μm, bright solitons at 1.31 μm can be accessed by manually tuning the laser frequency into the red-detuned soliton regime. Pumping with 80 mW on-chip power at 1.3 μm wavelength leads to the generation of a bright soliton, shown in Figure 6c together with a fitted sech$^2$ envelope (black dashed). Note that the mode crossing at 1326 nm (c.f. dispersion profile in Figure 6a) induces a dispersive wave (marked with an arrow) at 1326 nm in Figure 6c. In addition, we also demonstrate a bright soliton frequency comb at 1.55 μm. Figure 6d shows an optical spectrum of a bright single soliton when pumping a mode at 1574 nm. See Section S8 (Supporting Information) for the measured integrated dispersion profile at 1574 nm. In this case, the 1.3-μm laser is used as the auxiliary laser to thermally stabilize the resonator.

5. Conclusion

In summary, we report the fabrication of ultralow-loss Si$_3$N$_4$ photonic devices with high film thickness using room-temperature reactive sputtering. The measured propagation loss is 32 dB/m without post-thermal annealing. After thermal annealing at a CMOS-compatible processing temperature (400°C), the film loss is dramatically reduced to 5.4 dB/m, enabling the fabrication of Si$_3$N$_4$ ring resonators with an intrinsic Q of 6.2 million in the L-band. The deposition process is intrinsically hydrogen free, which prevents the formation of optically lossy hydrogen bonds. Ultralow loss integrated Si$_3$N$_4$ devices with low temperature...
annealing enable fully monolithic back end of line integration with pre-processed CMOS electronics, silicon integrated photonics,[7] and LNOL.[49] After further thermal annealing at 800°C, the film losses are further reduced to 3.5 dB m⁻¹ corresponding to attainable optical quality factors of microresonators beyond 10 million. This performance is also the lowest Si₃N₄ propagation loss reported at a peak processing temperature of 800°C. We believe that our low-temperature sputtered Si₃N₄ is a significant step toward the hybrid monolithic integration of silicon nitride with silicon photonics for applications in telecom systems, nonlinear optics, frequency comb generation and scaling of quantum computing systems.[50]

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

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

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hybrid photonic integration, low-temperature sputtering, silicon nitride, soliton frequency comb

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