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Propagation losses are paramount to the performance of microphotonic devices. In silicon photonics, the expected contribution of known propagation-loss mechanisms is often insufficient to account for all the observed loss. Here, we identify a loss mechanism that we believe has not yet been reported in the literature. We observe loss reaching 70 dB/cm in silicon wire waveguides patterned in proximity of metals with low temperatures of silicide formation. The loss is attributed to formation of a dilute silicide at the waveguide sidewalls during reactive-ion etching. Sputtered metal atoms originate from exposed metal on the wafer surface or from the reactive-ion etcher chamber and react with the bare silicon of the waveguide sidewall being formed. © *2008 American Institute of Physics.* [DOI: 10.1063/1.2903714]

Low propagation loss[1] is critical to the performance of nearly all microphotonic devices and to the practical operation of microphotonic circuits of notable complexity. In silicon photonics, propagation loss is mainly attributed to scattering losses[2] and electronic absorption at surface states.[3] The absorption of the bulk material is considered negligible for single-crystal silicon waveguides. Comparing the expected contributions of known loss mechanisms with the commonly reported propagation losses of 2–3 dB/cm in single-crystal silicon waveguides,[4–6] one observes that it is often difficult to account for all the observed loss. For instance, one finds propagation losses reaching 2–2.5 dB/cm (Ref. 6) in waveguides designed to bring scattering losses below 0.5 dB/cm.[7] The loss contribution from surface states and the error on the scattering loss estimate are not expected to be sufficient to resolve this discrepancy. This indicates that other loss mechanisms may be present. In this letter, we identify a loss mechanism that we believe has not yet been reported in the literature.

We report on loss reaching 70 dB/cm in single-crystal silicon waveguides patterned in proximity of metals with a low temperature of silicide formation. The experimental data points toward formation of a dilute silicide at the waveguide sidewalls during reactive-ion etching (RIE). As shown in Fig. 1, the silicide forms without intimate contact between the metal and the silicon and without anneal. The temperature of the sample does not exceed 90 °C in the fabrication process. However, the metal atoms sputtered by the incident ions have more than sufficient energy to react with silicon on contact and are deemed responsible here for the observed silicide. In addition, typical metal contamination levels suggest that this loss mechanism can be observed without metal being present on the substrate during RIE. In fact, metal atoms sputtered from stainless-steel RIE chambers can be of sufficient concentration to produce observable loss.

We first observed this additional loss mechanism by applying the fabrication process reported in Ref. 8 to silicon waveguides. A unibond silicon-on-insulator wafer was thermally oxidized to form 40 nm of silicon oxide on 240 nm of single-crystal silicon and 2.5 μm of buried oxide. Next, 200 nm of poly(methylmetacrylate) (PMMA), a positive e-beam resist, and 50 nm of aquasave, a water soluble polymer used to prevent charging in e-beam lithography, were spun and exposed at 30 KeV in a Raith 150 scanning electron-beam lithography system. After the aquasave was removed, the PMMA was developed in a 2:1 solution of isopropanol and methyl isobutyl ketone. Then, 50 nm of nickel was e-beam evaporated and a lift-off performed by removing the nonexposed resist in 1-methyl-2-pyrrolidinone. To define the waveguides, the resulting nickel pattern was used as a hard-mask for RIE. This approach is commonly used for circumventing the weak etching resistance of most e-beam resists. Figure 1 illustrates the material stack during RIE. A Plasmatherm 790, a conventional RIE system, was used at a pressure of 10 mTorr and a bias of 500 V. A gas flow of 17 standard cubic centimeter per minute (SCCM) of CHF_3 was used for etching the top oxide layer and a gas flow of 13.5 SCCM of CF_4 and 1.5 SCCM of O_2 was used to
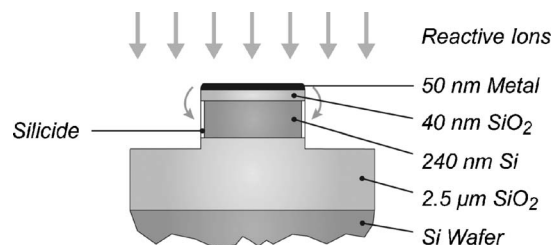

FIG. 1. Illustration of the mechanism to which the optical loss is attributed. During RIE, sputtered metal atoms react with the silicon sidewall to form a dilute silicide. The silicide is difficult to remove and induces an optical loss that is comparable to loss induced by a metal. The metal hard mask was thoroughly stripped before optical characterization.

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TABLE I. Transparency of waveguides etched with various metal hard masks.

Metal hard mask	Ni	Pd	Co	Cr	Fe–Ti[c]	Fe
Silicide formation temperature	200 °C	200 °C	270 °C	400 °C	350 °C (Ti)	400 °C
Roughness variance[a]	3.0 nm^2	2.8 nm^2	4.8 nm^2	7.7 nm^2	3.1 nm^2	4.2 nm^2
Propagation loss[b]	~70 dB/cm	>70 dB/cm	>65 dB/cm	<15 dB/cm	<15 dB/cm	<15 dB/cm

[a]Results are for nonoptimized liftoff. Roughness could be improved by fabrication process optimization (Ref. 8).
[b]Loss on the sample etched with a Ni mask measured on ring resonators. Losses on the other samples measured on straight waveguides.
[c]Fe–Ti indicates that a 5 nm titanium cap was used on top of 45 nm Fe to prevent the potential corrosion of Fe at lift-off.

etch the Si layer and 60 nm into the buried oxide layer. The Ni was then thoroughly stripped in a nitric-acid-based commercial wet Ni etchant (Transene TFB) and a standard clean was performed in a 5:1:1 solution of $H_2O:H_2O_2(30\%):NH_4OH(29\%)$ at 80 °C.

The resulting waveguides showed propagation losses of about 70 dB/cm, which is orders of magnitude larger than what could be attributed to known loss mechanisms. This loss value was obtained from the spectral response of a microring resonator with a 30 μm radius and a 408 nm wide ring waveguide. To obtain an acceptable signal, the chip was cleaved about 300 μm from the resonator. The optical power was coupled into a bus waveguide with a lensed fiber at that cleaved edge. The through- and drop-port spectral responses were collected by contact-mode near-field optical microscopy, which involved sequentially contacting the top of the drop- and the through-port waveguides with a lensed fiber.

To better understand the origin of the loss, 360 and 450 nm wide waveguides were fabricated with various metal hard masks and the waveguide transparency was assessed by measuring the transmitted optical power through 2–4 mm long straight waveguides at wavelengths ranging from 1430 to 1610 nm. Table I summarizes the experimental results and shows the silicide formation temperatures for the metals investigated.[9–11] The attenuation of the fundamental mode was at least 50 dB/cm larger on waveguides fabricated using Ni, Pd, or Co hard masks than on waveguides fabricated using Cr, Fe–Ti, or Fe hard masks. The waveguide sidewall roughness was measured from top-down electron micrographs using the approach described in Ref. 12. The fabrication process was the same as the one described above but the metal stripping chemistry was tailored to the given metal employed. A 3:1:2 solution of $HCl(37\%):HNO_3(68\%):H_2O$ was used to strip Pd and Co, a commercial wet etchant (Cyantek CR-7) was used to strip Cr, and a 5:1 solution of $H_2O:HNO_3(68\%)$ was used to strip Fe.

The propagation loss in Table I correlates well with a low silicide formation temperature of the metal. On the other hand, the waveguide transparency does not show a correlation with the observed sidewall roughness indicating that roughness does not play a significant role in this experiment. In fact, the loss is significantly too high to be explained by roughness scattering. Based on Ref. 2, the variance of the sidewall roughness would need to be of the order of 70–100 nm^2 to explain the observed loss while the measured variance does not exceed 4.8 nm^2 on waveguides with high loss. A micrograph of the high-loss waveguide showing the largest sidewall roughness is presented in Fig. 2.

To confirm the silicide formation hypothesis, the presence of metal in the waveguide sidewalls needed to be di-rectly established. For this purpose, the waveguides fabricated with a Pd hard mask were inspected with a scanning transmission electron microscope (STEM) equipped with an energy dispersive x-ray spectrometer (EDS). The results are shown in Fig. 3. Pd was detected in the silicon waveguide at 1–2 nm from the sidewall but not at 8–9 nm from the sidewall. The Pd signal corresponds to about 5% atomic concentration of Pd in Si, indicating that a dilute silicide was present. Such dilute silicide was found difficult to remove. It did not respond to HF based solutions, which are commonly used for removing stoichiometric silicides. Moreover, the silicides of interest are not consumed but only displaced by oxidation so they cannot be removed by sacrificial oxidation of the outer silicon layer.

To estimate the optical loss that should result from a silicide layer at the sidewalls, we performed a vectorial mode solver analysis using the published optical properties of metal silicides.[13–17] We found that for the silicides of interest with reported optical properties, the optical loss would be of the order of 2000–20 000 dB/cm per nanometer of silicide at the waveguide sidewalls. Hence, a silicide layer with an average thickness of the order of 0.01 nm would account for the observed loss. Such thickness is below an atomic layer and is not expected to be accurate as dilute films are unlikely to share the optical properties of stoichiometric films. Nonetheless, it indicates that traces of silicide could account for the experimentally observed loss.
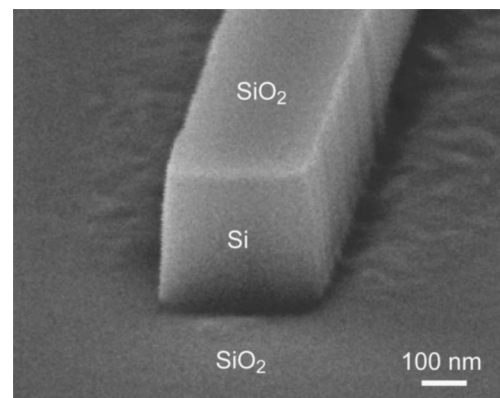

FIG. 2. Scanning electron micrograph of the high-loss waveguide with the largest sidewall roughness observed in this study. This order of roughness is not sufficient to account for the observed loss. The waveguide was fabricated with a Co hard mask. The roughness on the top surface of the thick SiO$_2$ under-cladding is due to thin lift-off imperfections partially masking the waveguide surroundings during RIE until they were sputtered away.
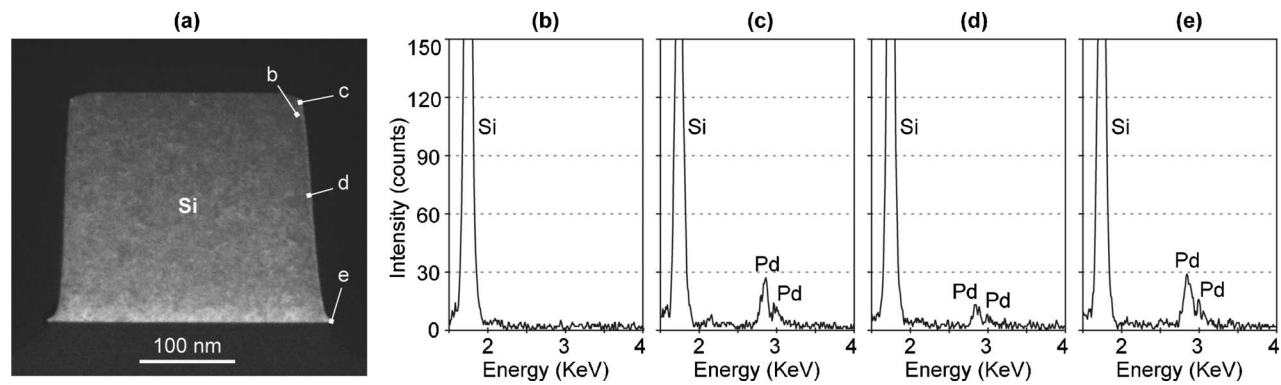
FIG. 3. STEM-EDS analysis of a Si waveguide etched in proximity of Pd and thoroughly cleaned with wet chemical processing. (a) Cross-sectional micrograph indicating where the EDS spectra were acquired. (b) EDS spectrum acquired 8–9 nm from the waveguide sidewall showing no detectable Pd. [(c)–(e)] EDS spectra acquired 1–2 nm from the waveguide sidewall showing, in average, about 5% atomic concentration of Pd in Si. The distribution of Pd along the waveguide sidewall is consistent with sputtering from the top metal mask.

To evaluate the importance of this loss mechanism beyond waveguides fabricated with metal hard masks, we compare the metal concentration observed by EDS to typical metal contamination levels. Our data show that a 5% atomic Pd concentration over about 20 atomic Si layers results in loss in excess of 70 dB/cm. By comparing this to Ni contamination on bare silicon wafers resulting from the stainless-steel electrode of a RIE chamber,[18] we obtain an expected loss in excess of 1 dB/cm. This indicates that typical RIE contamination levels can be of sufficient order to produce measurable optical loss without the presence of a metal hard mask on the wafer. In contrast, the metal contamination levels in state-of-the-art front-end-of-the-line microelectronic processing are sufficiently low for the reported loss mechanism not to be observable.

We reported on loss in silicon microphotonic waveguides due to metallic contamination. The loss is attributed to formation of a dilute silicide on the waveguide sidewalls during RIE. We believe this to be the first reported demonstration of this loss mechanism. The loss can be severe when a metal hard mask is used. Moreover, typical RIE contamination levels can be of sufficient order to produce measurable loss without the presence of a metal hard mask. If one is to use a metal hard mask in fabrication of silicon photonics, a metal with high temperature of silicide formation (such as Cr or Fe) is preferred.

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