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# Characterization of process-related interfacial dielectric loss in aluminum-on-silicon by resonator microwave measurements, materials analysis, and imaging

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## ABSTRACT

We systematically investigate the influence of the fabrication process on dielectric loss in aluminum-on-silicon superconducting coplanar waveguide resonators with internal quality factors ( $Q_i$ ) of about one million at the single-photon level. These devices are essential components in superconducting quantum processors; they also serve as proxies for understanding the energy loss of superconducting qubits. By systematically varying several fabrication steps, we identify the relative importance of reducing loss at the substrate–metal and substrate–air interfaces. We find that it is essential to clean the silicon substrate in hydrogen fluoride (HF) prior to aluminum deposition. A post-fabrication removal of the oxides on the surface of the silicon substrate and the aluminum film by immersion in HF further improves the  $Q_i$ . We observe a small, but noticeable, adverse effect on the loss by omitting either standard cleaning (SC1), pre-deposition heating of the substrate to 300 °C, or *in situ* post-deposition oxidation of the film's top surface. We find no improvement due to excessive pumping meant to reach a background pressure below  $6 \times 10^{-8}$  mbar. We correlate the measured loss with microscopic properties of the substrate–metal interface through characterization with x-ray photoelectron spectroscopy, time-of-flight secondary ion mass spectrometry, transmission electron microscopy, energy-dispersive x-ray spectroscopy, and atomic force microscopy.

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## INTRODUCTION

Superconducting coplanar waveguide (CPW) resonators<sup>1</sup> in the microwave band are extensively used to read out the state of qubits in the circuit quantum electrodynamics (cQED) architecture.<sup>2–6</sup> They can also be used as tools to investigate the loss in superconducting qubits.<sup>5,7,8</sup> Several loss mechanisms can adversely affect the performance of quantum circuits, including charged two-level systems (TLS), spins, non-equilibrium quasiparticles, radiation, and magnetic vortices.<sup>9–11</sup> Among these, TLS

dominate the loss at millikelvin temperatures and single-photon-power excitations, i.e., under the operating conditions of a superconducting quantum processor.<sup>12</sup> TLS loss originates in dielectric amorphous materials,<sup>13</sup> mainly within the oxide layers located at the substrate–air (SA), metal–air (MA), and substrate–metal (SM) interfaces of the device; see Fig. 1. Coupling to a bath of TLS, or to spurious, individual TLS, results in qubit decoherence and resonator loss.<sup>12,14</sup> Extensive work on analyzing the relative importance of dielectric loss due to TLS at different interfaces has been done by the community.<sup>15–19</sup> In our previous work,<sup>20</sup> which includes the

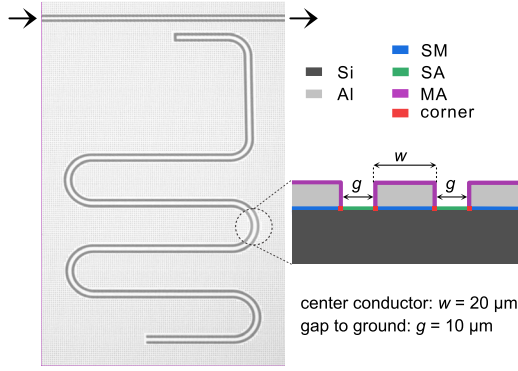


**FIG. 1.** The device and its materials' interfaces. The micrograph shows the quarter-wavelength CPW resonator, capacitively coupled to an input-output transmission line and surrounded by an array of  $2 \times 2 \mu\text{m}^2$  flux-trapping holes. The schematic illustrates a CPW cross section, highlighting the interfaces that contribute to the dielectric loss (not to scale). This CPW geometry is chosen to minimize dielectric loss, which is suppressed in larger geometries, while still keeping the geometry small enough to not exacerbate other losses related to large geometries, such as radiation.<sup>14</sup>

simulations of CPW resonators of identical geometry to the devices analyzed in this article, we find that a lossy SM interface dominates the total loss of the device. Once the SM loss is mitigated, losses at the MA and SA become dominant.

The TLS loss can be mitigated by refining the fabrication process. Different wet and dry etching processes have been employed to clean the Si substrate before the metal deposition to decrease the loss at the SM interface.<sup>21–24</sup> The choice of silicon (and sapphire) as popular substrates is due to their low loss tangent values,<sup>15,25,26</sup> which is essential in minimizing the loss in areas with high field concentrations. The bulk loss of the substrate then becomes negligible in comparison with the dielectric loss at the interfaces. Various etching methods, including trenching of the substrate, have been explored to transfer the resonator pattern onto the metal layer while maintaining a low-loss SA interface.<sup>27–31</sup> Both the SA and MA interfaces can be cleaned post-processing, before loading the sample into the cryostat for measurement.<sup>32,33</sup> In addition, the metal surface can be oxidized *in situ* after the deposition by filling the chamber with pure oxygen.<sup>8</sup> Passivation of the superconducting metal at the MA interface has also been explored.<sup>34,35</sup> We have combined some of these processes in a standard fabrication process for aluminum-on-silicon (Si–Al), discussed below, and it yields CPW resonators with TLS-limited internal quality factors  $Q_i \approx 1 \times 10^6$  and qubits with relaxation times  $T_1 \approx 100 \mu\text{s}$ .<sup>36–38</sup> The choice of aluminum as the superconducting material originates from its availability and the vast knowledge about its chemistry and fabrication techniques. A recent demonstration of Al transmon qubits with average relaxation time  $T_1 > 250 \mu\text{s}$  highlights the enduring potential of aluminum as a platform for superconducting circuits.<sup>20</sup>

This report presents a systematic evaluation of our CPW resonator fabrication process. We fabricate control resonators based on the standard recipe previously developed in our group<sup>8</sup> and also other resonators using recipes that deviate from the standard by only one step. We determine the resonators'  $Q_i$  and quantify the associated TLS losses in each fabrication step. In parallel, we

use atomic force microscopy (AFM) to study the film surface and transmission electron microscopy (TEM) to investigate the material structures at different interfaces of the resonators. Furthermore, we use a complementary set of techniques for compositional depth profiling, including x-ray photoelectron spectroscopy (XPS), energy dispersive x-ray spectroscopy (EDS), and time-of-flight secondary ion mass spectrometry (ToF-SIMS). XPS is suitable for exploring the chemical composition and the chemical state of the selected elements, while the ability of ToF-SIMS—a technique that has only recently gained popularity for investigating losses in superconducting devices<sup>39</sup>—to detect traces of individual elements and molecules with part-per-million accuracy can unfold the elemental depth profile of the device. We find that the most critical step to minimize the loss at the substrate–metal interface is the initial removal of the native oxide from the surface of the silicon wafer using hydrofluoric acid (HF). The substrate–air interface loss and, to a degree, the metal–air loss can also be reduced temporarily post-fabrication by the same acid treatment. This has previously been demonstrated on devices made from niobium<sup>10,40</sup> and tantalum.<sup>41</sup>

## METHOD

The internal quality factor ( $Q_i$ ) is used as a figure of merit to quantify the performance of a CPW resonator. We obtain  $Q_i$  of the resonator from the microwave forward transmission ( $S_{21}$ ) measurement of a quarter-wavelength resonator in a notch configuration relative to the feedline (see Fig. 1) using a routine circle fit.<sup>42</sup> The inverse of  $Q_i$  quantifies the contributing loss mechanisms,

$$1/Q_i = 1/Q_{\text{TLS}} + 1/Q_{\text{other}}, \quad (1)$$

where the first term is the loss due to the TLS and the second term,  $1/Q_{\text{other}} \equiv \delta_{\text{other}}$ , is the sum of other loss mechanisms. According to the TLS model,<sup>14</sup>  $\delta_{\text{TLS}}$  is written as a function of power and temperature,

$$\delta_{\text{TLS}} = F\delta_{\text{TLS}}^0 \frac{\tanh(\hbar\omega_r/2k_B T)}{(1 + \langle n \rangle/n_c)^\beta}, \quad (2)$$

where  $F$  is the filling factor, defined as the ratio of the electric field stored in the TLS material to the total stored electric field,  $\delta_{\text{TLS}}^0$  is the intrinsic TLS loss, and  $\omega_r$  is the resonator frequency.  $T$  represents the temperature,  $\langle n \rangle$  is the average number of photons stored/circulating in the resonator,  $n_c$  is the critical photon number to saturate a single TLS, and  $\beta \leq 0.5$  is the TLS saturation rate with photon power.<sup>43–45</sup>  $\hbar$  and  $k_B$  are the reduced Planck and Boltzmann constants, respectively. Combining Eqs. (1) and (2) and assuming a low operating temperature so that  $\tanh(\hbar\omega_r/2k_B T) \approx 1$ , we get

$$1/Q_i = F\delta_{\text{TLS}}^0 \frac{1}{(1 + \langle n \rangle/n_c)^\beta} + \delta_{\text{other}}, \quad (3)$$

which we use to quantify the TLS loss  $F\delta_{\text{TLS}}^0$  of the superconducting resonators. We assume the other, non-TLS-related losses  $\delta_{\text{other}}$  to be power-independent. This assumption is based on the expected power-independence of the other losses due to radiation, quasiparticles, magnetic flux noise, or packaging losses,<sup>46</sup> which is validated by experimental observations of power-independent  $Q_i$  at  $\langle n \rangle \gtrsim 10^7$ .

For our experiments, we used high-resistivity intrinsic Si (100) wafers and aluminum superconducting films. The standard fabrication process started with wafer cleaning, by submerging the wafer in a mixture of ammonium hydroxide, hydrogen peroxide, and deionized (DI) water ( $\text{NH}_4\text{OH}:\text{H}_2\text{O}_2:\text{H}_2\text{O}$ , 1:1:5)—known as standard clean 1 (SC1)—at  $80^\circ\text{C}$  for 10 min. The wafer was then rinsed with DI water and subsequently dipped in a 2% aqueous solution of hydrofluoric acid for 1 min, followed by another DI water rinse. Within a few minutes, we loaded the cleaned wafer into the load-lock of the evaporator *Plassys MEB550S* to minimize the re-oxidation of the Si surface.<sup>47</sup> Next, we heated the substrate, *in situ*, to  $300^\circ\text{C}$  for 10 min. After about 20 h, the wafer cooled down to room temperature ( $\sim 20^\circ\text{C}$ ) and the chamber base pressure reached  $4 \times 10^{-8}$  mbar. We evaporated 150 nm of Al at the deposition rate of  $1 \text{ nm s}^{-1}$ , followed by static oxidation of the Al surface at the pressure of 10 mbar for 10 min.

Following the film deposition, the resonators were fabricated by means of optical lithography and wet etching (in aluminum etchant, i.e., mixture of phosphoric, nitric, and acetic acids) and measured in a dilution refrigerator at about 10 mK. Besides the standard sample (No. 1), we measured five additional samples (Nos. 2–6), where for systematic evaluation, we fabricated each sample by varying only one step of the standard process. The list of the samples and the type of variation are summarized in Table I. We fabricated sample No. 1 using the standard process, while for sample No. 2, we did not remove the native oxide of the silicon substrate in HF prior to metal evaporation. For sample Nos. 3–5, we skipped the SC1 step, the substrate preheat, and the *in situ* oxidation of aluminum, respectively, and finally, we prepared sample No. 6 using a short (4 h) pumping time (reaching a pressure of  $6 \times 10^{-8}$  mbar) prior to metal evaporation.

Subsequently, we investigated the influence of varying the conditions of the film deposition itself. For wafer No. 7 (includes sample Nos. 7a–7c), we followed the same conditions as for sample No. 1, with the exception that the SC1 cleaning was omitted and the Al film was sputtered instead of being evaporated: after cleaning the substrate using HF dip, the wafer was immediately transferred to the heated load-lock ( $80^\circ\text{C}$ ) of a sputter tool (*DCA MTD620*), which was pumped down for 40 min until a pressure below  $5 \times 10^{-7}$  mbar

was reached. Thereafter, the wafer was transferred to the deposition chamber. Here, the substrate was heated to  $300^\circ\text{C}$  for 10 min and pumped for 16 h, at which point the base pressure of the deposition chamber was reached at  $2.2 \times 10^{-8}$  mbar. Al was then deposited by direct-current (DC) magnetron sputtering in argon (Ar) plasma, followed by *in situ* oxidation of the film surface. In the [supplementary material](#), we also present an investigation into the effects on the resonator  $Q_i$  resulting from the deposition rate, substrate temperature during deposition, and high-temperature substrate preheating.

We used three samples, Nos. 7a–7c, to investigate the effect of a short dip in 2% HF post-fabrication, with the aim to remove the surface oxides of both the Al film and the Si substrate, and with them any adsorbates left behind by the fabrication process. This dip was only 15 s long due to the tendency of Al to be etched in this solution, and the samples were rinsed in DI water afterward. The effect of this procedure is shown on sample No. 7b, while No. 7a is kept as the control. Sample No. 7c underwent a vapor HMDS deposition at  $100^\circ\text{C}$  immediately after HF treatment, where the hydrophobic HMDS monolayer deposited this way could be expected to protect the Si from re-oxidizing.<sup>22</sup> More details on sample Nos. 7a–7c are given in Table II. Due to the tendency of both Al and Si to regrow oxides under ambient conditions, sample Nos. 7b and 7c were mounted inside the dilution refrigerator, with the pump-down preceding the cooldown started within 2.5 h after the HF dip.

The XPS experiment was performed using a *PHI 5000 VersaProbe III* system equipped with a monochromatic aluminum x-ray source (i.e., Al  $K\alpha$ ). To aid in depth profiling, we performed a step-wise iterative procedure of  $\text{Ar}^+$  ion sputtering to etch away the Al layer followed by an XPS measurement, until the SM interface was reached. We scanned selected binding-energy ranges based on the elements of interest, including oxygen (O 1s), aluminum (Al 2p), and silicon (Si 2p), in order to conduct a qualitative analysis of the chemical state(s) of individual elements.

TEM bright field (BF) imaging was performed using a *FEI Tecnai T20* microscope operated at 200 kV. TEM cross section samples were prepared using a *FEI Versa 3D* focused ion beam scanning electron microscope (FIB-SEM). Scanning transmission electron microscopy (STEM) and energy dispersive x-ray spectroscopy (EDS) analyses were carried out using a *JEOL Monochromated ARM 200F*

**TABLE I.** Summary of sample Nos. 1–6 (evaporated Al films). The table includes the average  $Q_i$  at  $\langle n \rangle = 1$  across all the resonators on a sample, the coupling quality factor  $Q_c$ ,  $\beta$ ,  $F\delta_{\text{TLS}}^0$ , and  $\delta_{\text{other}}$  as fit parameters to the TLS model, Eq. (3). Standard deviations are provided for the parameters of the TLS model.

| Process variation                 | Sample No. 1<br>Standard | Sample No. 2<br>No HF  | Sample No. 3<br>No SC1 | Sample No. 4<br>No preheat | Sample No. 5<br>No <i>in situ</i> oxidation | Sample No. 6<br>Short pumping |
|-----------------------------------|--------------------------|------------------------|------------------------|----------------------------|---------------------------------------------|-------------------------------|
| No. of resonators                 | 8                        | 6                      | 6                      | 7                          | 8                                           | 6                             |
| $Q_i$ ( $\langle n \rangle = 1$ ) | $1.1 \times 10^6$        | $0.24 \times 10^6$     | $0.95 \times 10^6$     | $0.8 \times 10^6$          | $0.92 \times 10^6$                          | $1.1 \times 10^6$             |
| $Q_c$                             | $1.1 \times 10^6$        | $1.2 \times 10^6$      | $1.2 \times 10^6$      | $1.0 \times 10^6$          | $0.84 \times 10^6$                          | $1.0 \times 10^6$             |
| $\beta$                           | 0.22<br>( $\pm 0.07$ )   | 0.29<br>( $\pm 0.04$ ) | 0.27<br>( $\pm 0.10$ ) | 0.20<br>( $\pm 0.07$ )     | 0.27<br>( $\pm 0.08$ )                      | 0.24<br>( $\pm 0.08$ )        |
| $F\delta_{\text{TLS}}^0/10^{-6}$  | 0.87<br>( $\pm 0.29$ )   | 4.4<br>( $\pm 0.79$ )  | 1.0<br>( $\pm 0.23$ )  | 1.4<br>( $\pm 0.70$ )      | 0.97<br>( $\pm 0.27$ )                      | 0.87<br>( $\pm 0.20$ )        |
| $\delta_{\text{other}}/10^{-7}$   | 2.3<br>( $\pm 0.53$ )    | 2.3<br>( $\pm 0.30$ )  | 2.0<br>( $\pm 0.72$ )  | 2.1<br>( $\pm 0.43$ )      | 2.1<br>( $\pm 0.51$ )                       | 2.4<br>( $\pm 0.76$ )         |

**TABLE II.** List of sample Nos. 7a–7c (sputtered Al films). No. 7a is a control sample. Sample Nos. 7b and 7c were treated with HF post-fabrication, with the addition of an HMDS step in No. 7c. The table includes the average  $Q_i$  at  $\langle n \rangle \sim 10$  across all the resonators on a sample, the coupling quality factor  $Q_c$ ,  $\beta$ ,  $F\delta_{\text{TLS}}^0$ , and  $\delta_{\text{other}}$  for the TLS model fit for every sample, as shown in Fig. 6(a). Standard deviations are provided for the parameters of the TLS model.

| Process variation                     | Sample No. 7a<br>Sputter reference | Sample No. 7b<br>HF    | Sample No. 7c<br>HF + HMDS |
|---------------------------------------|------------------------------------|------------------------|----------------------------|
| No. of resonators                     | 6                                  | 5                      | 7                          |
| $Q_i$ ( $\langle n \rangle \sim 10$ ) | $1.0 \times 10^6$                  | $2.2 \times 10^6$      | $2.1 \times 10^6$          |
| $Q_c$                                 | $1.1 \times 10^6$                  | $1.1 \times 10^6$      | $1.2 \times 10^6$          |
| $\beta$                               | 0.36<br>( $\pm 0.07$ )             | 0.33<br>( $\pm 0.09$ ) | 0.36<br>( $\pm 0.07$ )     |
| $F\delta_{\text{TLS}}^0/10^{-6}$      | 0.86<br>( $\pm 0.29$ )             | 0.27<br>( $\pm 0.05$ ) | 0.39<br>( $\pm 0.16$ )     |
| $\delta_{\text{other}}/10^{-7}$       | 1.5<br>( $\pm 0.24$ )              | 2.0<br>( $\pm 0.37$ )  | 1.7<br>( $\pm 0.49$ )      |

TEM, which is equipped with a Schottky field emission electron source, a double-Wien monochromator, a probe Cs corrector, an image Cs corrector, and a double silicon drift detector (SDD) for EDS.

In addition, we performed the ToF-SIMS measurement using an *IONTOF 5* system, with a bismuth ion ( $\text{Bi}^+$ ) gun for imaging and a cesium ion ( $\text{Cs}^+$ ) sputter gun for depth profiling. The mass spectrum of the secondary ions emitted from the sample was collected at each sputtering step and converted to an elemental depth profile. Note that the XPS and ToF-SIMS measurements were performed either on chips from the same wafers as the resonators were fabricated on, or on chips from wafers that were prepared following identical cleaning and film growth procedures.

## RESULTS AND DISCUSSION

Each sample contains up to eight resonators (Fig. 1), whose frequencies range between 4 and 8 GHz. The frequencies are varied by changing the length of the resonators, while keeping the cross-sectional dimensions  $w$  and  $g$  identical. We measured the power-dependent  $S_{21}$  of the feedline using a vector network analyzer (VNA) and calculated  $F\delta_{\text{TLS}}^0$  by fitting the results with the TLS loss model described in Eq. (3). Figure 2(a) presents  $Q_i$  as a function of  $\langle n \rangle$  for a representative resonator of a comparable frequency, one resonator each for sample Nos. 1–6. At low  $\langle n \rangle$ ,  $Q_i$  becomes scattered. This is likely due to the temporal instability of TLS, as the TLS can switch in and out of resonance with the device on timescales comparable to the measurement time due to the large number of averages required at low  $\langle n \rangle$ .<sup>36,48,49</sup> For samples fabricated with the standard process, on average, we find  $Q_i = 1.1 \times 10^6$  when  $\langle n \rangle = 1$ , and  $F\delta_{\text{TLS}}^0 = 0.87 \times 10^{-6}$ . For the other samples, the values of  $Q_i$  together with the fitting parameters of the TLS loss model [Eq. (3)] are shown in Table I. The parameter  $F\delta_{\text{TLS}}^0$  from the fit is presented as a box plot in Fig. 2(b), where each point in this plot is from an individual resonator. The bounds of the box represent the upper and lower quartiles of the statistical distribution, and the whiskers extend to the farthest data points lying within  $1.5 \times$

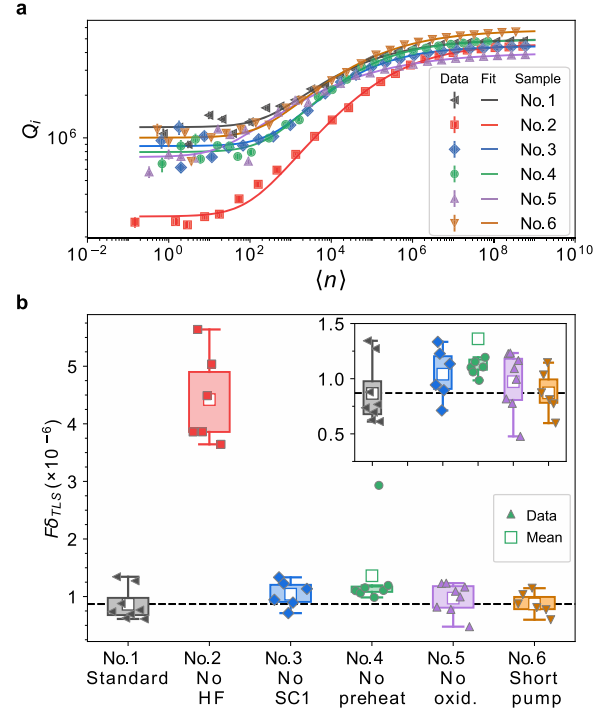


**FIG. 2.** Resonator loss as fitted to the TLS model [Eq. (3)] for sample Nos. 1–6, with process variations listed in Table I. (a)  $Q_i$  vs circulating power. The resonance frequency is  $\sim 4.4$  GHz. (b) Box plot of TLS loss. Each data point represents the loss of an individual resonator. The inset shows a close-up of the area of the figure where most data points for sample Nos. 1 and 3–6 are located.

the interquartile range from the box bounds. The loss due to other parameters  $\delta_{\text{other}}$ , also presented in Table I, is about  $2 \times 10^{-7}$  for all samples.

The highest level of TLS loss is observed in sample No. 2 (no HF), a fivefold increase compared to that of the standard sample (No. 1); see Fig. 2(b). Using TEM, we find a clean SM interface of the standard sample, whereas there exists a layer of  $\sim 1.5$  nm-thick oxide at the SM interface of No. 2 [Figs. 3(a) and 3(b)]. The XPS spectra for O 1s, Al 2p, and Si 2p at the SM interface of sample Nos. 1 and 2 are shown in Figs. 3(c)–3(e), respectively. Although the oxide layer at the SM interface of No. 2 was supposedly comprised of native oxide atop the Si substrate ( $\text{SiO}_2$ ), the oxidation–reduction reaction at room temperature between Al and  $\text{SiO}_2$  has turned the oxide into an aluminum oxide type.<sup>50</sup> The signature of this oxide is observed through the XPS spectra of O 1s and Al 2p. The Al 2p spectrum shows that metallic Al, with the binding energy position at 72.8 eV, is the dominating chemical state, while a small shoulder positioned at 75.3 eV corresponds to the oxidized Al(III) state. The major peak of O 1s at 532 eV gives an energy difference of 456.8 eV with Al (III). This implies that the interface oxide is mostly in the form of aluminum oxide. Still, as the oxygen content is rather low and close to the XPS detection limit, i.e., 1.0 at.%, it is difficult to determine exactly in which form the oxide has developed; see Figs. 3(c) and 3(d). The characteristic peak of Si 2p at 99.4 eV indicates that silicon is mostly in its elemental state. The secondary peak at 104.0 eV

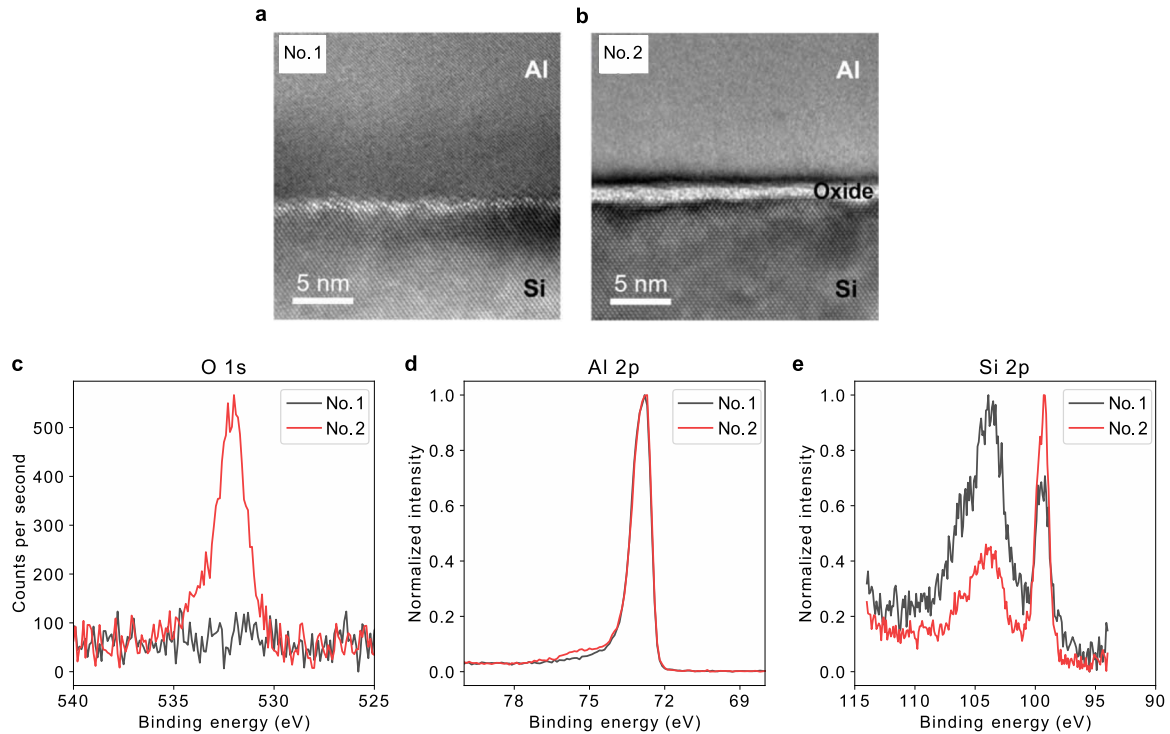


**FIG. 3.** TEM images and XPS spectra comparing the SM interface of the standard sample (No. 1) and the sample without the HF clean (No. 2). (a) The TEM BF image of sample No. 1 shows a clean, oxide-free interface. Al and Si lattices directly contact each other at the interface at the atomic scale, indicating a clean interface without an amorphous interface layer. The bright contrast at the interface is due to Fresnel fringe contrast. (b) The TEM of sample No. 2 shows an oxide layer at the SM interface with a thickness of about 1.5 nm. (c)–(e) XPS spectra of O 1s, Al 2p, and Si 2p at the SM interface resulting from the standard-process (black) and no-HF (red) samples.

refers mostly to the Al 2p plasmon loss peak instead of the overlapping Si(IV) state, with reference to both the energy difference and the area ratio compared to the metallic Al 2p peak; see Figs. 3(d) and 3(e).

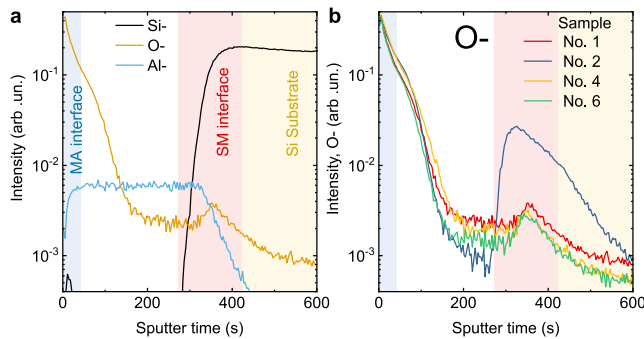


**FIG. 4.** ToF-SIMS. (a) Depth profile of the standard-process sample (No. 1) characterized by ToF-SIMS. The depth is proportional to the time during which the sample was etched by the Cs<sup>+</sup> sputter beam. The MA and SM interfaces are highlighted with blue and red, respectively, while the Si substrate is in yellow. (b) The depth profile of O<sup>-</sup> from sample No. 1 (standard process), No. 2 (no HF), No. 4 (no preheat), and No. 6 (short pumping).

The XPS spectra indicate no trace of O 1s in sample No. 1 at the Si–Al interface; see Fig. 3(c). However, the depth profile of No. 1 from the ToF-SIMS measurement in Fig. 4(a) features a small peak from oxygen at the SM interface. Since the oxide was not observed in TEM either, we speculate that the trace oxide in No. 1 is either local or in the form of a discontinuous layer along the grain boundaries.<sup>20</sup> The oxygen signal at the SM interface in No. 2 is more intense and agrees well with the TEM and XPS results; see Fig. 4(b). The intensity of the O peak in No. 2 is almost an order of magnitude higher than that of the other samples, in agreement with the relatively higher concentration of O at the SM interface of this sample and its correspondingly high TLS loss level; see Fig. 2.

Surface treatment with SC1 and HF has been a prominent cleaning method in the semiconductor industry for several decades.<sup>51</sup> The SC1 solution cleans the surface of the Si substrate from most organics and some metallic contamination by trapping them into an oxide layer on the surface, which is then removed by HF. If SC1 is not performed, some contamination remains on the substrate surface, resulting in 15% higher  $F\delta_{\text{TLS}}^0$  in No. 3 compared with No. 1 [Fig. 2(b)], even though the oxide layer, as the main source of TLS loss, has already been removed by the HF solution.

Another process extensively used to ensure a good quality SM interface is the heating of the substrate under vacuum.<sup>52–54</sup> The elevated temperature of the substrate can desorb moisture and volatile molecules from the surface.<sup>55</sup> In some cases, the substrate undergoes

annealing temperatures of above 700 °C for surface reconstruction to achieve a better atomic transition between the metal and substrate.<sup>24,56</sup> In our experiments, the sample with no preheating process (No. 4) showed a 61% increase in  $F\delta_{\text{TLS}}^0$  compared with sample No. 1. However, one of the resonators on sample No. 4 shows a significantly higher loss than the remaining resonators. Discarding this outlier resonator, the mean  $F\delta_{\text{TLS}}^0$  of sample No. 4 falls to  $1.1 \pm 0.07 \times 10^{-6}$ —a 26% increase in TLS loss compared to the standard sample (No. 1). We observe no discernible difference in the oxygen concentration at the SM interface of sample No. 4 compared to the standard sample (No. 1) in the ToF-SIMS measurement shown in Fig. 4. It might be that the change in the oxygen level is below the observable limit, or it might not be the desorption of oxidizing species behind the increased performance of the devices on sample No. 1 when the preheat step is included.

The TLS loss of sample No. 5 is just marginally higher (10%) than the standard sample; see Fig. 2(b), and one may conclude that *in situ* oxidation of the Al film is unnecessary. However, upon SEM inspection of No. 5, we found dark spots distributed over the Al film—resembling voids—as shown in Fig. 5(a). The TEM image of Fig. 5(c) shows the cross section of one of these voids. For further investigation, we prepared a second Al film on the Si substrate, where we skipped the *in situ* oxidation after evaporating the film. We observed no voids in the Al immediately after deposition. Next, we heated a sample to 160 °C for 5 min to create a similar condition to that of baking the photoresist during resonator fabrication.

We inspected the sample daily; the voids appeared on the piece after about two days.

The SEM image of sample No. 5 shows residues on the silicon substrate within the CPW gap where Al was wet etched; see Fig. 5(a). These residues remain on the silicon surface after wet etching of the Al layer using aluminum etchant, even if we etch a sample that is not exposed to heating after metal deposition. A longer etch time does not remove the residues either. The lateral size of these residues varies from a few tens of nanometers up to 200 nm, and the thickness is measured to be ~2 nm, as shown in the AFM image of Fig. 5(b). Cross-sectional EDS analysis in TEM [see Figs. 5(d) and 5(e) and the [supplementary material](#)] shows the composition distribution of a residue flake in the etched Al area. There is an intermixing of Al, Si, and O in the flake. A ToF-SIMS analysis of the etched Al areas of sample Nos. 1 and 5 shows a significantly stronger presence of the residues of Al and Al oxide on sample No. 5; see Fig. S7 in the [supplementary material](#).

Due to the thermal expansion mismatch between Al and Si, heating or cooling can generate stress in the Al film ( $\alpha_{\text{Al}} = 23.1 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$  and  $\alpha_{\text{Si}} = 2.6 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ <sup>57</sup>). The textured (polycrystalline) form of evaporated Al and its low melting point make it prone to recrystallization. Stress can promote the recrystallization and formation of defects in the film. The development of hillocks and voids in pure Al films on Si (or even silicon oxide) has been a known challenge in the CMOS industry.<sup>57</sup> The oxide on the surface of Al might help keep the grains together and partially

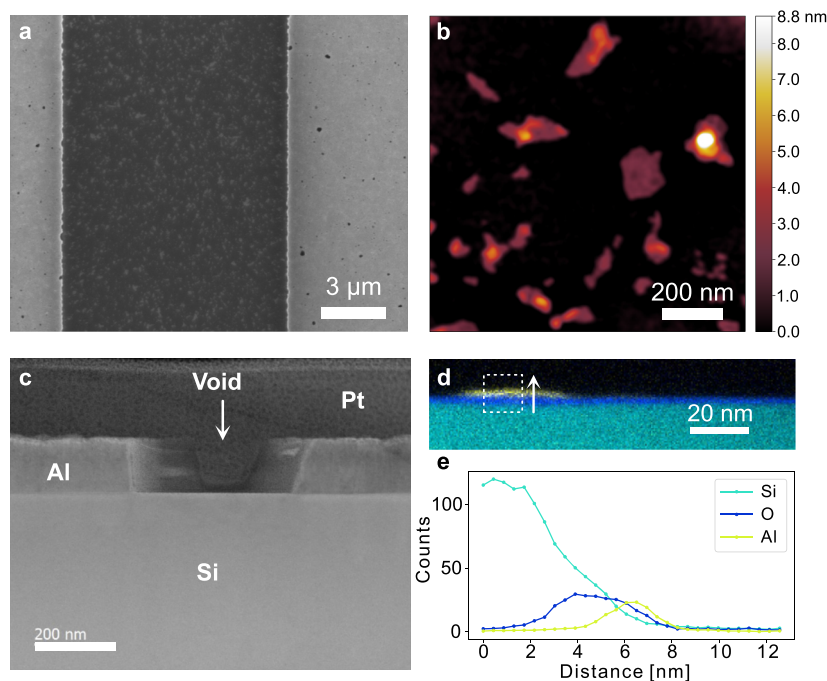


**FIG. 5.** SEM, AFM, TEM, and EDS of sample No. 5 (no *in situ* oxidation of Al). (a) In the SEM, voids appear as dark spots in the Al film (light gray). In addition, flakes are observed in the etched Al area on Si (gray). (b) AFM image of the flakes with the lateral size ranging from a few tens of nanometers to 200 nm and thickness of about 2 nm. (c) TEM BF image showing a void in the Al film. The Pt film was deposited on the sample during TEM cross section sample preparation to protect the structure. (d) Composite EDS composition map of Al (yellow), O (blue), and Si (cyan) in an area where the Al layer has been etched. The map was obtained using Al-K, O-K, and Si-K EDS signals. The dashed-square line and arrow indicate the area and direction where the EDS intensity profiles were extracted. The profiles are shown in (e).

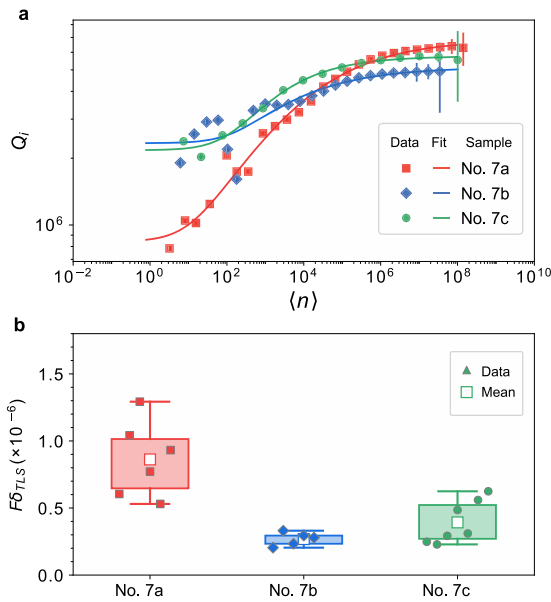


**FIG. 6.** Internal quality factor ( $Q_i$ ) and losses determined from the TLS model fits for the samples with a sputtered Al film. (a)  $Q_i$  vs the average number of photons for the resonators fabricated with the process variations listed in Table II. The resonance frequency is  $\sim 4.8$  GHz. (b) Box plot of TLS loss from the fits to the TLS loss model for all of the resonators on each sample. Each data point represents the loss of an individual resonator.

prevent the creation of defects. We speculate that different types of aluminum oxide grow atop the Al film as a result of *in situ* oxidation (exposure to pure oxygen at low pressures) or ambient air oxidation (exposure to oxygen and moisture at atmospheric pressure). Oxidation in the ambient air might introduce more defects and porosity in the aluminum oxide, making it less effective in protecting the film. This can result in the formation of voids and defects in the Al film.

With the shorter pumping time used for sample No. 6, the base pressure and the substrate temperature prior to deposition were  $6 \times 10^{-8}$  mbar and  $50^\circ\text{C}$  (passively cooled down from  $300^\circ\text{C}$ ), respectively. The  $Q_i$  and TLS loss of the resonators on sample No. 6 are comparable to those of No. 1 (Fig. 2). The ToF-SIMS results of No. 6 show only a negligible change of O intensity at the SM interface in Fig. 4(b), meaning that longer pumping time is not necessary.

For the resonators etched out of a sputtered Al film, we achieved a comparable quality factor to those on the evaporated film; see Fig. 6 and Table II. The average TLS loss  $F\delta_{\text{TLS}}^0$  obtained for sample No. 7a is about  $0.86 \times 10^{-6}$ , which is comparable to that of No. 1. This is despite the increased surface roughness of the sputtered film compared to the evaporated film, which may increase the loss at the MA interface. The other, non-TLS related loss mechanisms are lower for the sputtered film No. 7a (smaller  $\delta_{\text{other}}$ ). Since  $\delta_{\text{other}}$  is a combination of several loss mechanisms, pinpointing the exact cause of the lower loss needs further study. A fraction of this may be an effect of lowered quasiparticle loss due to an increased superconducting gap in the sputtered film, as the superconducting critical temperature  $T_c$ , collected from 4-point probe DC transport measurements, is 1.2 K for the sputtered film and 0.91 K for the evaporated (standard) film. In contrast, the residual-resistance ratio RRR is 3 for

the sputtered film and 8 for the evaporated one, indicating a higher crystalline quality of the evaporated film. We remark that the quality factors in sample No. 7 were obtained after optimizing the sputtering process—for details of the samples, also their quality factors, surface roughness and atomic force micrographs, see the [supplementary material](#).

A short dip in HF post-fabrication reduces the TLS loss, doubling the  $Q_i$  achieved at low photon numbers, as shown in Fig. 6 and Table II. Since it took us about 2.5 h to place the HF-treated samples in vacuum (at the mixing chamber inside the dilution refrigerator), we anticipate the regrowth of aluminum oxide at the MA interface, while it takes longer for the silicon oxide on the substrate to fully regrow.<sup>40</sup> Therefore, we think that the improvement in the loss after the HF treatment is mainly due to the reduced contribution of the SA interface, while a minor improvement comes from the reduced loss at the MA interface: as discussed above, the partial etch of Al can help with removing the adsorbates on the aluminum surface.

A subsequent passivation of the silicon substrate by HMDS in sample No. 7c slightly degrades  $Q_i$  compared to No. 7b, although  $F\delta_{\text{TLS}}^0$  is still below one-half that of the untreated sample No. 7a; see Table II. We note that due to the decreased resonance linewidth of the higher-quality resonators, single-photon levels are not reached in the measurement power span. Therefore, in Table II, we compare the  $Q_i$  of the resonators at  $\langle n \rangle \sim 10$ . The decreased TLS loss of the HF-treated samples becomes comparable to the other losses  $\delta_{\text{other}}$ , resulting in the low-power  $Q_i$  being limited by the other loss mechanisms as well.

## CONCLUSION

In conclusion, our investigations show that concerning the loss at the substrate–metal interface, the surface treatment with SC1 and HF is the most effective method to reduce the associated TLS loss in superconducting Al-based CPW resonators fabricated on Si substrates. ToF-SIMS characterization shows one order of magnitude higher intensity of oxygen at the SM interface if the native oxide is not removed from the substrate. In comparison with the standard sample, this leads to an increase in the TLS loss by five times. In addition, we found that avoiding preheating of the substrate prior to deposition can increase the TLS loss by about 60%.

The *in situ* oxidation of the Al film led to a marginal increase in the loss; however, observation of voids in the Al film and residues in the gap suggests that this step is necessary for not facing random and unwanted open circuits in the device. Finally, despite the higher pre-deposition base pressure and temperature, the TLS loss of the sample with short pumping was still comparable to the standard process, reaffirming the insignificance of the bulk material quality and composition compared with the surfaces and interfaces as the source of TLS loss.<sup>58</sup>

With regard to the substrate–air interface, and, to a lower degree, the metal–air interface, a post-fabrication HF dip of resonator chips reveals that removing the surface oxides of Al and Si can decrease the TLS loss by about three times. As the effect of the oxide strip is temporary, until an oxide has regrown, it is desirable to extend this effect for practical applications. We show that immediate passivation of the Si by HMDS using vapor deposition can slightly degrade the  $Q_i$  achieved after the HF dip, although still keep-

ing the loss well below that of an untreated sample. Further studies into the longevity of the protective effect of the HMDS layer are necessary.

## SUPPLEMENTARY MATERIAL

The [supplementary material](#) includes details of the Q factor extraction, a discussion of the sputtering parameter optimization based on microwave measurements and materials analysis, a demonstration of the post-fabrication treatment by HF + HMDS on a sputtered sample, and additional materials characterization of the sample that has not undergone *in situ* oxidation (sample No. 5).

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## AUTHOR DECLARATIONS

### Conflict of Interest

The authors have no conflicts to disclose.

## Author Contributions

Janka Biznárová and Lert Chayanun contributed equally to this work.

**Lert Chayanun:** Formal analysis (equal); Investigation (equal); Visualization (equal); Writing – original draft (equal); Writing – review & editing (supporting). **Janka Biznárová:** Formal analysis (equal); Investigation (equal); Visualization (equal); Writing – original draft (equal); Writing – review & editing (equal). **Lunjie Zeng:** Investigation (supporting); Writing – review & editing (supporting). **Per Malmberg:** Investigation (supporting); Writing – review & editing (supporting). **Andreas Nylander:** Investigation (supporting); Writing – review & editing (supporting). **Amr Osman:** Investigation (supporting); Writing – review & editing (supporting). **Marcus Rommel:** Investigation (supporting); Writing – review &

editing (supporting). **Pui Lam Tam:** Formal analysis (supporting); Investigation (supporting); Visualization (supporting); Writing – review & editing (supporting). **Eva Olsson:** Investigation (supporting); Writing – review & editing (supporting). **Per Delsing:** Formal analysis (supporting); Funding acquisition (equal); Writing – review & editing (supporting). **August Yurgens:** Formal analysis (supporting); Writing – review & editing (supporting). **Jonas Bylander:** Conceptualization (equal); Formal analysis (supporting); Funding acquisition (equal); Supervision (equal); Writing – original draft (equal); Writing – review & editing (equal). **Anita Fadavi Roudsari:** Conceptualization (equal); Formal analysis (supporting); Investigation (supporting); Supervision (equal); Visualization (supporting); Writing – original draft (equal); Writing – review & editing (equal).

## DATA AVAILABILITY

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

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