

Formation of niobium hydride precipitates in superconducting qubits

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(Received 18 August 2025; accepted 19 November 2025; published 6 January 2026)

We report evidence for the formation of niobium hydride phase within niobium films on silicon substrates in superconducting qubits fabricated at Rigetti Computing. For this study, we combined complementary techniques—including room-temperature and cryogenic atomic force microscopy (AFM), synchrotron x-ray diffraction, and time-of-flight secondary ion mass spectroscopy (ToF-SIMS)—to directly reveal the existence of niobium hydride precipitates on the surface of superconducting qubits. Upon cryogenic cooling, we observed variation in the size and morphology of the hydrides, ranging from small (~ 5 nm) irregular shapes to large (~ 10 – 100 nm) domains within the Nb grains, which were fully converted to niobium hydrides. Since niobium hydrides are nonsuperconducting and can easily change in size and location upon different cooldowns to cryogenic temperature, our finding highlights a previously unknown source of decoherence in superconducting qubits. This contributes to quasiparticle losses, offering a potential explanation for changes in qubit performance upon cooldowns. Finally, by leveraging the RF performance of a 3D bulk Nb resonator, we quantify RF dissipation in a superconducting qubit caused by hydrogen concentration variation, and propose a practical engineering pathway to mitigate the formation of Nb hydrides for superconducting qubit applications.

DOI: [10.1103/mgnw-kjps](https://doi.org/10.1103/mgnw-kjps)

I. INTRODUCTION

Superconducting niobium has gained widespread recognition for transmon qubit applications in quantum computing. It presents the highest T_c among pure metals as a gapped superconductor and shows anisotropies regarding material properties [1–3]. Niobium thin films, typically ~ 100 – 200 -nm thick, are key components in many superconducting qubit architectures, used to form readout resonators, coupling lines, and capacitance pads present in qubits [4,5]. Therefore, any microwave dissipation mechanisms—arising from the niobium film, its native oxide layers, amorphous interface layers, or the substrate itself—can contribute to qubit decoherence. There have been intensive studies to understand the microscopic origins of the decoherence in the superconducting qubits [4,6], and several coherence-limiting mechanisms have been identified [7]. Those are amorphous oxides, which host a two-level system (TLS) at various interfaces [4,8–10], metal-air [3,11,12], metal-substrate [13–15], and substrate-air interface [3,14], which have been recently shown to be the primary coherence-limiting mechanism in the quantum regime

for 3D superconducting radio frequency (SRF) resonators (cavities) [12,16]. Additionally, non-equilibrium quasiparticles [17], including those produced by cosmic rays and radioactive sources in the environment [18,19], are another possible decoherence mechanism.

It has been previously discovered in bulk niobium SRF cavities that the formation of the nonsuperconducting niobium nanohydrides upon cooldown from room temperature to cryogenic temperatures (< 150 K) introduces a major dissipation mechanism, which limits the achievable quality factor [20–23]. The process is enabled by Nb's ability to absorb significant amounts of hydrogen (H) even at room temperature, whenever a layer of native niobium oxide, mainly Nb_2O_5 , does not passivate it [24,25]. Hydrogen atoms can occupy 12 tetrahedral sites in the body-centered cubic (bcc) Nb lattice, and they can induce several crystalline hydride phase formations linked to the occupied atomic position within the Nb lattice [26,27], particularly at temperatures below the saturation point of ~ 150 K [28]. The hydride phase can limit the achievable quality factors of the resonators [29] as significant microwave dissipation arises from the local suppression of superconductivity via proximity effects [22]. The possible presence and dissipation owing to niobium hydrides, well studied in bulk Nb SRF cavities, has not been extensively studied yet for the 2D superconducting qubits.

In this study, we apply the same techniques used to reveal the niobium hydrides in the bulk SRF Nb cavities to niobium film regions of the superconducting qubit test chips fabricated by Rigetti Computing [30]. We combine atomic force

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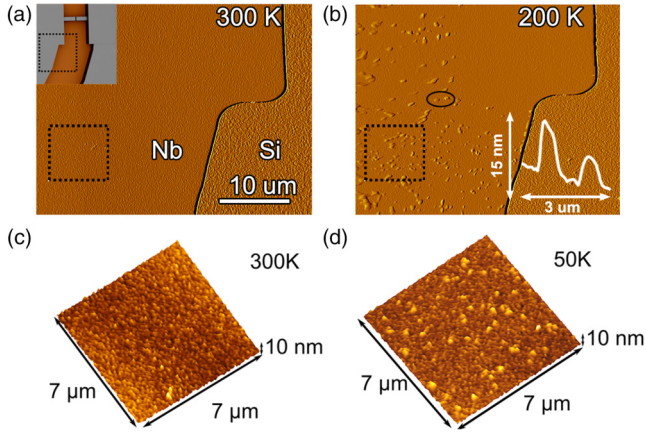


FIG. 1. (a) (Inset) Schematic of the Nb qubit pads area, cryo-AFM area of analysis is shown by dashed rectangle; AFM surface images of Nb films and Nb/Si boundary at room temperature, and (b) at $T = 200$ K (during warming up back to 300 K from initial cooling down to 2 K), with the formation of the unknown structures on the surface of Nb clearly observed. The line profile through the structures marked by the ellipse is shown in the inset on the right, indicating the height of the order of 15 nm. The higher magnification surface topology of Nb films from the dashed rectangles in (a) and (b) are shown in (c) RT and (d) 50 K (during initial cooldown to 2 K), respectively, with further smaller scale formations apparent.

microscopy (AFM), synchrotron x-ray diffraction, and the time-of-flight secondary ion mass spectroscopy (ToF-SIMS) at room and cryogenic temperatures to reveal the niobium hydrides' existence directly in the Rigetti superconducting qubits. In parallel, the microwave performance of a bulk SRF resonator is examined at a low RF field regime (<1 MV/m) and low temperature below 1.6 K after varying amounts of hydrogen loading content to assess the impact of Nb hydride on the extent of decoherence of superconducting qubits. These newly identified niobium hydrides may explain variability in qubit performance across cooldown cycles. Additionally, we discuss strategies to mitigate Nb hydride formation in superconducting qubit applications.

II. RESULTS

Superconducting qubit test devices with Nb resonators and Al/AlO_x/Al Josephson junctions were fabricated on a Si (100) substrate (float-zone $>10\,000\ \Omega\text{ cm}$) at Rigetti Computing following the published nanofabrication procedure [30]. The chip contains five superconducting qubits, readout resonators, and connecting lines. The Nb films on the test chips were deposited via high power impulse magnetron sputtering (HiPIMS) at room temperature, with a base pressure below 1×10^{-8} Torr.

In the inset of Fig. 1(a), a schematic of the superconducting qubit chip analyzed in this study is displayed with the Si substrate, Al/AlO_x/Al Josephson junction area, and Nb film pads labelled accordingly. The surface morphology of the Nb film is first analyzed using atomic force microscopy (AFM) at room and cryogenic temperatures, as shown in Figs. 1(a)–1(d). AFM scan was performed at every 50 K step from 300 K down to 2 K with a cooling rate of 20 K/min, followed

by a three-hour extended stay to avoid thermal fluctuation. At room temperature (RT), the Nb surface is smooth, displaying root-mean-square (RMS) roughness values <1 nm. However, we observed two distinct morphological features that arise during cooling down to 2 K and warming up back to 300 K. During the initial cooling from 300 K to 2 K, features with the size on the order of ~ 2 nm emerged in some of the Nb grains below 50 K, as shown in Fig. 1(d) and in the Supplemental Material [31]. Subsequently, during the warming from 2 K to 300 K, irregularly shaped structures with a lateral diameter of ~ 500 nm and a height of 10–20 nm appeared on the surface of the Nb films as the temperature approached 200 K, as shown in Fig. 1(b). These interesting morphological features disappeared entirely at 250 K, available in the Supplemental Material [31]. This implies that the irregular features should emerge below 200 K and persist through warming up to 250 K. Figures 1(a) and 1(c) show the surface topology at 300 K for comparison. In the case of bulk Nb SRF cavities, similar surface features have been previously detected at cryogenic temperatures, below 150 K, during the initial cooling from room temperature and have been shown to result from the precipitation of the Nb hydride [32].

ToF-SIMS was used to probe the chemical identity associated with forming these surface features. Specifically, depth profile measurements performed at room temperature on several Nb contact pad regions from the same qubit device revealed an appreciable level of hydrogen at the surface [Fig. 2(a)]. We find that the H-/Nb-signal, which represents a measure of the free hydrogen concentration present in the parent Nb matrix, is maximum immediately beneath the niobium oxide layer Nb₂O₅, and then decays to a much lower level within the first ~ 10 -nm depth from the surface. The presence of this hydrogen at the surface is consistent with previous findings in the bulk Nb cavities and also potentially drives Nb hydride formation in Nb thin film geometries. Specifically, compared to a bulk Nb cavity, the H-/Nb-signal within the top 25 nm is more prominent in the contact pad [Fig. 2(b)]. In addition to hydrogen, significant concentrations of other impurities are observed. For example, the O-/Nb- and C-/Nb-signals, which also decay on a longer length scale than the H-/Nb-signal, are shown in Fig. 2(a).

Cryo-grazing incidence x-ray diffraction (GIXRD) was performed at the Spanish beamline BM25 (SpLine) of the European Synchrotron Research Facilities (ESRF) to identify Nb hydride precipitates in the Nb film. This film was cofabricated with the Rigetti qubit device using HiPIMS. Diffraction patterns were collected at energies up to 18.34 keV to avoid the Nb $K\alpha$ fluorescence (20–25 keV) [33]. The Nb film was mounted on a Cu cold finger under ultrahigh vacuum (UHV) conditions ($\sim 10^{-9}$ mbar), allowing cooling down to 5 K via a continuous liquid helium (He) stream. The x-ray penetration depth ($\lambda = 0.68\ \text{\AA}$) was estimated using Parratt's equation [34] for an incident angle range of 0.15° to 2° . In Fig. 3, the dominant crystallographic reflection corresponds to Nb (101) plane at $d = 2.40\ \text{\AA}$. Upon cooling down cycling, a new diffraction peak emerges at 150 K and remains present in the temperature range 150–100 K [Fig. 3(b)], in contrast, we highlighted the absence of this peak at room temperature (300 K), as shown in Fig. 3(a). This reflection corresponds to the (111) plane of the orthorhombic Nb-hydride phase,

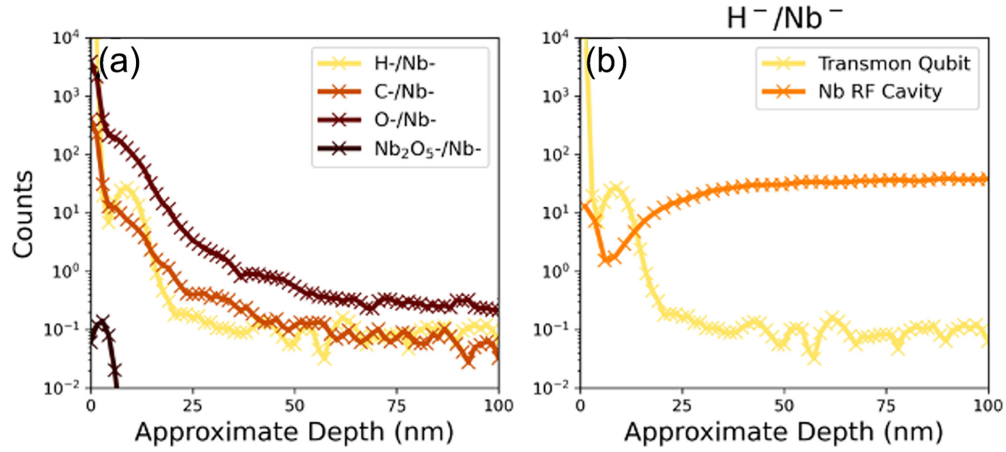


FIG. 2. (a) SIMS depth profiles of O, H, C impurities and the Nb₂O₅ surface oxide layer in Nb film in the superconducting qubit. (b) The comparison of hydrogen impurity in the transmon qubit and the bulk Nb surfaces.

NbH_{0.89}, which exhibits *Pnnn* space group with $d = 2.45$ Å. During a subsequent cooldown, this hydride-related peak shifted slightly by ~ 0.02 Å but retained its intensity between 100 K and 150 K. Nevertheless, below 100 K, the intensity of this hydride-related peak decreased substantially and is not clearly distinguishable at the base temperature of 5 K. Additionally, no other reflections related to hydrides were observed upon further cooling to 5 K, and the hydride reflections completely disappeared after warming back to 300 K. This behavior is analogous to previous observations in the bulk SRF Nb cavities, suggesting that the surface topographical features observed by cryo-AFM indeed correspond to hydride phases.

Next, we employed a 3D 1.3 GHz superconducting radiofrequency (SRF) niobium resonator (cavity) as a simplified, single-interface system to quantify RF dissipation caused by varying hydrogen concentrations in the RF penetration layer. Initially, the baseline test of the 1.3 GHz Nb cavity was performed after following an empirical engineering process of the cavity surface treatments. The cavity was

tested with power balance RF measurement to extract the unloaded quality factor, Q_0 [35], and applied to zero-span decay measurement as discussed in Refs. [10,16] to probe the quality factor at the lower-field regime. After the baseline testing, hydrogen incorporation was performed by mechanical grinding the cavity's exterior with a vibrational sander under continuous deionized water flow, maintaining the resonator's inner surface integrity. To further enhance hydrogen uptake and elevate hydrogen concentration within the cavity inner wall, an additional H-loading procedure was executed with electrochemical treatment. The details of cavity preparation are described in the Supplemental Material [31]. Figure 4(a) summarizes the RF test results using Q (quality factor) vs E_{acc} (on-axis electric field) plot. Q is the figure of merit presenting how far the stored energy decays as a function of the on-axis electric field. To verify successful hydrogen incorporation, two representative cavity cutouts underwent identical H-loading procedures, and their hydrogen concentrations were analyzed using ToF-SIMS. These results, also displayed in Fig. 4, confirm increased hydrogen concentrations.

Our findings indicate that increasing free hydrogen concentration in the cavity walls by approximately a factor of 2.5 results in a nearly tenfold reduction in the low-field quality factor, likely due to an increased volume fraction of niobium hydrides. These normal conducting inclusions inherit a diminished level of superconductivity via proximity-coupling to the niobium matrix and remain superconducting up to a breakdown field. Above this breakdown field, the hydrides become normal conducting and introduce significant loss, which increases with the field, a phenomenon known as the high-field Q slope (HFQS) [22]. We observe that significant hydrogen loading reduces the onset of HFQS from 25 MV/m to 0.4 MV/m. As the breakdown field inversely correlates with normal-metal inclusion size, distribution, and frequency (density), this lower onset field suggests significantly larger niobium hydride inclusions.

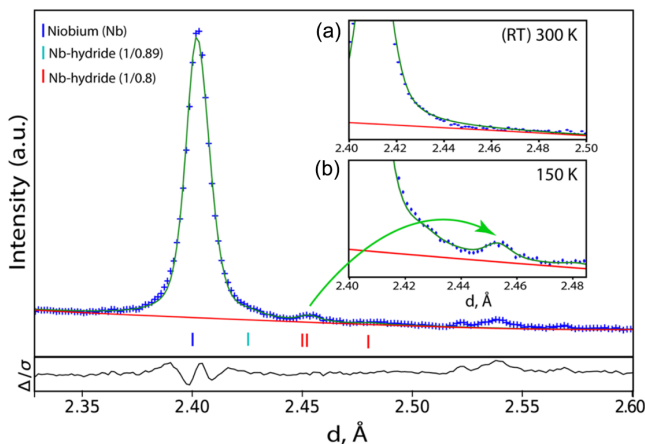


FIG. 3. Rietveld refined the synchrotron x-ray diffraction pattern of the Nb film measured at 150 K. The insets show the interest region at (a) 300 K and (b) 150 K. At 150 K, we observed the presence of weak peaks associated with Nb-hydride precipitations.

III. DISCUSSION

Through the use of complementary characterization techniques, we revealed that nanometric-scale hydrides locally

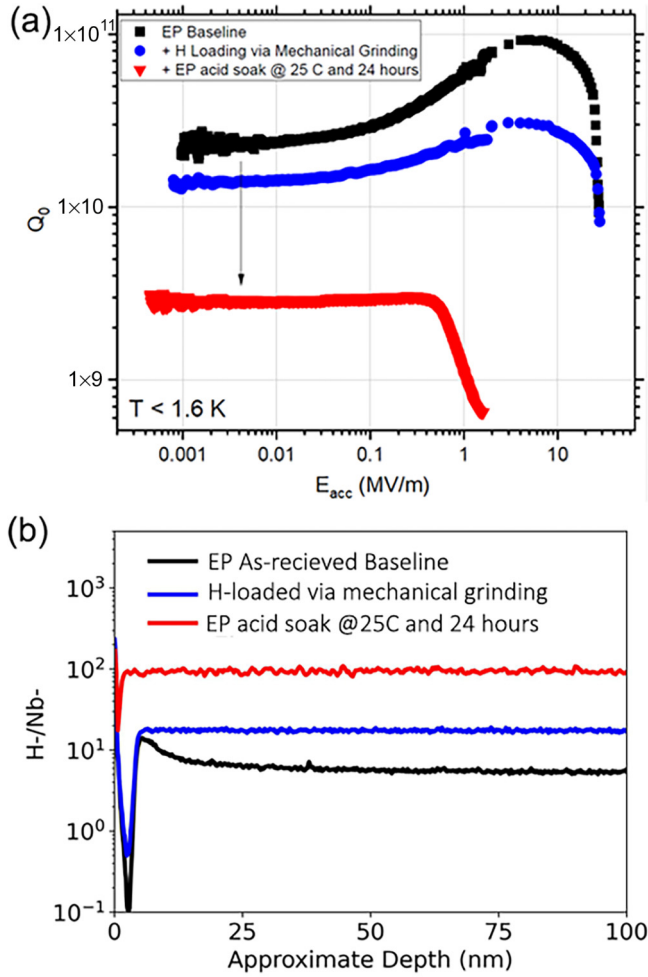


FIG. 4. (a) Q (quality factor) vs E_{acc} (accelerating field) plot from the RF measurements at a temperature less than 1.6 K on a 1.3 GHz niobium SRF resonator subjected to various hydrogen loading processing techniques. (b) ToF-SIMS acquired data on cavity cutouts of EP as-received, after H-loading via mechanical grinding, and EP (electropolishing) acid soaking for 24 hours, subjected to the hydrogen loading processing techniques, respectively.

precipitate in the niobium pads of the superconducting qubit chip. Topographical variations observed via cryo-AFM during cooling suggest that hydride precipitation in the Nb film follows a trend similar to that observed in bulk Nb [20,22,32]. The solid solubility of hydride becomes thermodynamically significant below 150 K [26,28], as confirmed by temperature-dependent grazing incident x-ray diffraction data presented in Fig. 3. Free hydrogen atoms located beneath the passive oxide layer are believed to be the primary source for hydride phase formation, as indicated by ToF-SIMS depth profile.

Recent finite-element modeling [36] has shown that niobium hydride phases migrate to the free surface of the Nb film under a hydrogen-rich environment, which is consistent with our results. Several possible explanations exist for the causes of the hydrogen incorporation and subsequent hydride formation in the qubit Nb films. First, hydrogen atoms may be incorporated during deposition because of residual hydrogen in the sputter chamber [9,37]. Similar to bulk Nb, any chemical treatment [38], annealing [39], or mechanical polishing

[40,41] steps in the manufacturing process performed in a hydrogen-containing environment, which remove the protective Nb_2O_5 layer from Nb, can lead to hydrogen incorporation in the underlying Nb. A recent study [25] highlights the critical role of the Nb_2O_5 layer in hydride formation in Nb films, linked to degradation in performance of a superconducting resonator. In addition, a Cottrell atmosphere at dislocations or within dense dislocation networks may provide a favorable environment for hydride nucleation [42,43]. Therefore, further investigation is needed to identify the specific fabrication processes contributing to hydrogen incorporation.

Given that ToF SIMS results confirm the presence of significant impurities such as oxygen, nitrogen, and carbon in the niobium-impurities that are known to act as effective hydrogen traps and thus suppress hydride formation [44–46]—the observed formation of Nb hydrides in certain regions of the qubit chip indicates that concentrations of these impurities may not be sufficient to trap hydrogen. Indeed, prior studies have demonstrated that increasing the oxygen concentration progressively reduces the H/Nb-ratio and gradually eliminates hydride-related losses such as HFQS [47]. Therefore, strategically introducing higher concentrations of oxygen into the niobium films could further mitigate hydride formation, offering an effective pathway to enhance superconducting qubit performance.

A slight discrepancy in cryo-AFM topological features is observed between the Nb film and bulk SRF Nb. As presented in Fig. 1, unlike in the bulk [32], distinct Nb hydride-related features did not appear during the initial cooldown but instead emerged after warming back from 2 K. Topographical features of ~ 2 nm in size were observed only during the initial cooldown below 50 K, and they persisted at 2 K. These features were more distinguishable at 150 K during the warming cycle, as shown in the Supplemental Material [31]. This behavior suggests that the film’s intrinsic columnar crystalline structure, as shown in the Supplemental Material [31], may hinder hydrogen diffusion into defect sites within the Nb matrix, thereby suppressing hydride formation. Interestingly, our cryo-GIXRD indicates that the hydride morphology can evolve with repeated cooling cycles, consistent with findings in a previous study [20]. This observation may offer a possible explanation for changes in qubit coherence times following each cooldown and the effect of “aging” from the accumulated number of cooldowns.

In superconducting RF Nb resonators, hydrogen incorporation within interstitial sites leads to the so-called “Q-slope,” where the NbH phase, proximity coupled to the surrounding Nb matrix, causes significant degradation of the quality factor at high RF fields [21,22]. Furthermore, because these hydride regions exhibit a smaller superconducting gap (or Δ , order parameter) than the surrounding Nb, they may act as sinks for quasiparticles and, in turn, diminish relaxation times near the operating temperature. The widespread presence of these precipitates throughout the film suggests that simply modifying film thickness is unlikely to mitigate this decoherence mechanism.

Figure 4 shows that significant Q_0 degradation can also arise even in a low accelerating field (<1 MV/m) at a lower temperature (<1.6 K), comparable to the quantum regime, as the precipitates of nanoscale hydride phases are coupled

with the proximity effect of the superconducting to normal state. The extent of suppression of the quality factor depends on the amount of H loading. In this study, the application of a 3D bulk Nb cavity clarifies the effects of hydride formation on the level of RF dissipation, compared to the study for the complex architecture of a superconducting quantum device. Surface property variation by engineering chemical and thermal processes [48,49] has been sufficiently studied in the bulk Nb SRF cavity for optimal RF performance, and may be adapted to more complex device architectures. This suggests that the substantially reduced decoherence time observed in the superconducting transmon qubit could be linked to the emergence of normal-conducting hydride regions. However, our bulk Nb study does not address the potential involvement of two-level system (TLS) associated with the hydride phase. Further work should include detailed RF characterization using Nb film-based resonators.

In summary, our findings suggest that the Nb hydride precipitates in Nb thin films are pervasive and present a previously unaccounted-for source of decoherence in superconducting qubits and planar Nb resonators. Niobium hydrides likely contribute to quasiparticle dissipation and may contribute to cooldown-to-cooldown variability and long-term “aging” effects.

ACKNOWLEDGMENTS

This work was supported by the U.S. Department of Energy, Office of Science, National Quantum Information Science Research Centers, Superconducting Quantum Materials and Systems Center (SQMS), under Contract No. 89243024CSC000002. Fermilab is operated by

Fermi Forward Discovery Group, LLC under Contract No. 89243024CSC000002 with the U.S. Department of Energy, Office of Science, Office of High Energy Physics. We acknowledge the Spanish Ministerio de Ciencia, Innovación y Universidades and Consejo Superior de Investigaciones Científicas for financial support through the Projects 2010 6 0E 013 and 2021 60 E 030, and for the provision of synchrotron radiation facilities at BM25-SpLine at the ESRF. This work made use of the EPIC, Keck-II, and/or SPID facilities of Northwestern University’s NUANCE Center, which received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-1542205); the MRSEC Program (NSF DMR-3541121262) at the Materials Research Center; the International Institute for Nanotechnology (IIN); the Keck Foundation; and the State of Illinois, through the IIN. We thank Rigetti Computing and, in particular, Cameron Kopas and the Rigetti chip design and fabrication teams for the development and manufacturing of the qubit devices used in the reported experimental study. We are also grateful to Carlos Gerardo Torres Castanedo, Drs. Paul Chandrica Masih Das, Dominic Pascal Goronzy, Prof. Mark Hersam, and David N Seidman. We would also like to thank Dr. Germán R. Castro and all the technical staff from the SpLine beam station for their valuable support during our experiments.

DATA AVAILABILITY

The data that support the findings of this article are not publicly available because of legal restrictions preventing unrestricted public distribution. The data are available from the authors upon reasonable request.

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- [31] See Supplemental Material at <http://link.aps.org/supplemental/10.1103/mgnw-kjps> for preparation and RF test of the Nb resonator, Annular Dark Field (ADF) - scanning transmission electron microscope (STEM) image of Nb/Si cross-section of Nb resonator on Si (Fig. S1), cryogenic-atomic force microscope (AFM) intensity and topography images of the Nb pad region of the Rigetti transmon qubit device during a cooling and warming cycle (Fig. S2).
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