# Rapid Cryogenic Electrical Characterization of Materials and Devices Using Gifford-McMahon Cryocoolers

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Thin-film heterostructures are necessary building blocks for superconducting and phononic quantum computing devices. Many new generations of quantum hardware demand extensive materials research to optimize performances at cryogenic temperatures (below 10 K). Here, we demonstrate compact cryogenic measurement systems capable of reaching sub-10K temperatures in less than three hours with the ability to measure AC/DC resistance and dielectric properties of thin-film materials. Our platform utilizes Gifford-McMahon (GM) cryocoolers as effective tools for providing high throughput cooling-warming cycles. We successfully used the GM-based measurement systems to measure 1) the superconducting transition temperature for Nb thin films ($T_c$ ~7.8 K), and 2) the temperature dependence of the dielectric constant in $SiO_2$ thin films down to 10 K. The fast electrical characterization feedback will be critical in developing robust materials and components for cryogenic computing devices.

## Introduction

Cryogenic electronics have attracted a great deal of attention due to their great potential in high-speed classical and quantum processors [1], [2]. In recent years, the fast growth of superconducting quantum devices has revitalized the materials and device research for cryogenic logic applications research. In particular, discovering new materials that operate efficiently at cryogenic temperatures (< 77K) will be critical in realizing not only effective quantum electronics but the classical control circuits [3]. This demands high-throughput cryogenic characterization techniques that enable rapid monitoring of the electrical properties of samples (e.g., conductivity, dielectric constant, piezoelectric coefficient) with a broad range of processing conditions and device configurations.

Here, we use Gifford-McMahon (GM) cryocoolers to build high-throughput cryogenic test platforms for evaluating the electrical properties of superconductors and insulators with applications in quantum electronics. The use of GM cryocoolers allows for relatively rapid cool-down and warm-up cycles for small sample exchange volumes. This is evident from the cooling capacities of many common compact GM cryocoolers such as ARS DE-202SE (0.1W at 4.2K and 4W at 77K [4]). The cryocoolers follow the GM refrigeration cycle, which circulates $^4$He gas from a compressor to the coldhead in a closed circuit [5], [6]. The movement of the displacer allows for the expansion and cooling of the $^4$He gas, which removes heat from the coldhead as it moves through the regenerating material. The low cost, closed-cycle dry refrigeration, low vibrations, and simple operation procedure make GM cryocoolers a compelling platform for small-scale cryogenic laboratory equipment.

Our cryogenic characterization platform, equipped with GM cryocoolers, is capable of performing electrical measurements on solid-state materials and devices from room temperatures down to 7 K. The platform leverages the unique properties of GM cryocoolers in high cooling powers and versatility in geometry. Two electrical measurement functions are demonstrated on materials with applications relevant to superconducting and phononic quantum circuits: 1) AC/DC resistance measurements on metallic thin-film superconductors; 2) Frequency-dependent impedance measurements on dielectric thin films. The average cool-down time from room temperature (300 K) to 7 K is 2.5 hrs. This platform is ideal for applications where fast electrical feedback is critical in guiding the materials synthesis and device micro-/nanofabrication.

## Methods

**Cryogenic Measurement Systems: Fig.1** displays a schematic of our rapid cryogenic AC/DC resistance measurement platform with a GM cryocooler (Advanced Research Systems, DE-202 SE) that houses the samples. A compressor is connected to the cryocooler to provide high-pressure $^4$He (at 300 - 320 psi) for the cooling cycles. Throughout the cool-down process, the sample exchange space is evacuated to pressures as low as $1x10^{-5}$ mbar using a turbomolecular pumping station. Each cryocooler is equipped with two silicon diode temperature sensors (Lakeshore, DT-470,670) and one tape heater (Minco, HK6909), which are controlled with a cryogenic temperature controller (Lakeshore 340). The AC/DC measurement system is wired with 12 cryogenically-compatible phosphor bronze wires. A Dual-Phase Lock-In Amplifier (SRS 830) and a source measure unit (Keithley 2450) are used for AC/DC signal generation and processing.

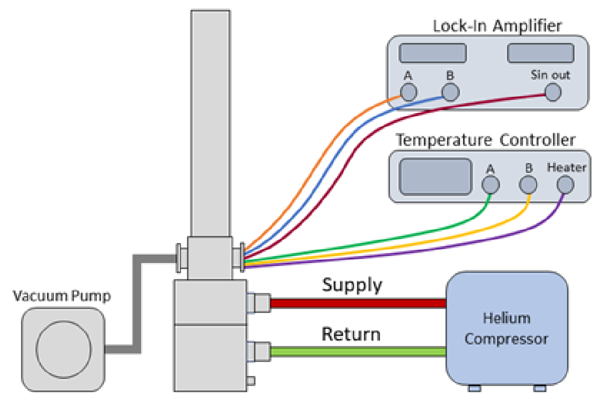


**Fig 1:** Diagram showing the GM cryocooler setup for AC/DC resistance measurements. The cryocooler is attached to a turbomolecular pump, a helium compressor, a Lakeshore temperature controller, and a lock-in amplifier. Electrical connections between the room-temperature electronics and the sample were made via a vacuum-compatible feedthrough on the cryogenic system.

A second GM cryocooler was configured for low-temperature characterization of dielectric materials using an LCR bridge (NF ZM2376). **Fig.2a** shows the cold finger attached to the second stage of the GM cryocooler with a PCB sample holder specifically designed for 4-terminal LCR measurements. The four terminals include high-potential (HPOT), high-current (HCUR), low-potential (LPOT), and low-current (LCUR). Cryogenically compatible coaxial cables were utilized to connect the terminals at room temperature to the four surface-mount SMP connectors on the PCB. **Fig.2b** shows a sample of $SiO_2$ metal-oxide-semiconductor capacitor (MOSCAP) mounted on the PCB sample holder using thin phosphor bronze wires attached to the circular pads and the back of the chip using In-Sn solder. Both cryogenic measurement platforms were automated using Exopy; an open-source Python-based library developed for automating experimental physics tools [7].

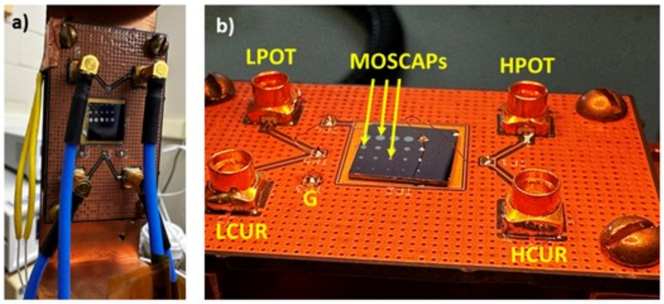


**Fig 2:** Cryogenic LCR measurement setup: a) Sample holder PBC mounted on the second stage of a GM cryocooler designed for LCR measurements: the four blue coax cables run between the PCB and the electrical feedthrough. b) An LCR test chip with arrays of metal-oxide-semiconductor capacitors (MOSCAPs) composed of Pd electrodes, $SiO_2$ dielectric, and heavily p-doped Si(001).

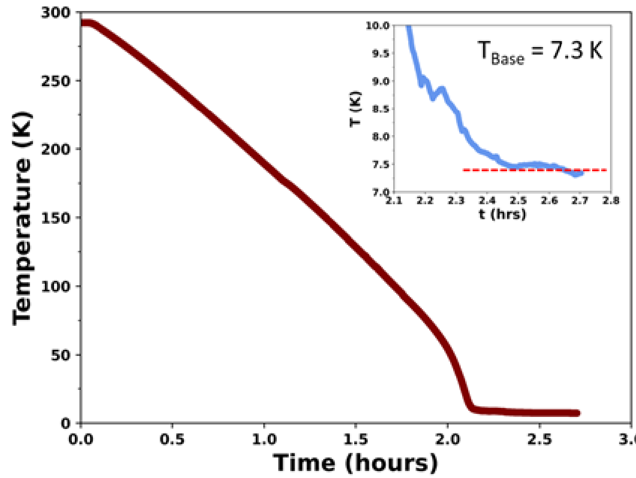


**Fig. 3:** Temperature vs. time for our AC/DC measurement system over a 2.8-hour cool-down period. The inset shows the cool-down trace below 10K. The red dashed line indicates the base temperature of the system (7.3 K).

**Superconducting Test Samples:** A Molecular Beam Epitaxy (MBE) system was used to prepare high-quality superconducting thin films for testing our cryogenic AC/DC measurement system. The samples were composed of thin niobium (Nb) films deposited on Si(001) substrates. The Si substrates were degreased by sequential sonications in acetone, isopropyl alcohol, and deionized water followed by $N_2$ dry. Once loaded into the MBE chamber, Si substrates were annealed to temperatures as high as 575 ˚C to remove the surface contaminants. The substrates were then cooled down to below -100˚C using a continuous $LN_2$ flow circuit. While cold, 30 nm thick Nb layers were deposited on the substrates via e -beam evaporation. By maintaining cryogenic temperatures for the substrate, we aim to minimize the Nb-Si intermixing throughout the growth.

**LCR Test Samples:** Pd/$SiO_2$/Si MOSCAPs were fabricated as test devices for the cryogenic LCR measurement system. A Si wafer with 100 nm of thermally-grown $SiO_2$ was purchased from a vendor (MTI Corporation). The as-received wafer was diced into 1 cm x 1 cm chips that were cleaned by sequential sonication in acetone, isopropyl alcohol, and deionized water. The $SiO_2$/Si chips were then briefly exposed to an $O_2$ plasma to remove trace amounts of organic residues on their surfaces. Finally, arrays of electrodes were realized on each sample by electron beam evaporation of Pd through a physical shadow mask placed between the source and the chips. The Pd electrodes are about 100 nm thick.

## Results

The performance of each cryogenic measurement system was evaluated by measuring the temperature of the sample holder (wired but not loaded with a sample) vs. cooling time. **Fig.3** shows the cool-down characteristics of our AC/DC measurement system where the base temperature of 7.3 K was reached in 2.5 hours. The cooling rate varies throughout the cool-down process. In particular, a significant decline in the cooling rate is observed from 9 K down to the base temperature. Once the cryocooler reaches its base temperature, variations of only ±40 mK in the sample holder temperature are observed (see inset for Fig.3). Moreover, each measurement system has an average warm-up time of 1 hr. Therefore, the combined cool-down and warm-up time for our measurement systems is less than 3.5 hr.

**Fig.4a** shows the sheet resistance vs. temperature for a superconducting Nb test sample measured in our AC/DC measurement system. The Nb/Si (001) thin-film sample was measured in van der Pauw (VdP) configuration [8] from room temperature to 7.3 K. The sheet resistance vs. temperature near the critical temperature ($T_c$) of the Nb film is shown in **Fig.4b**. Based on this cool-down data the $T_c$ of the Nb thin film was estimated to be 7.88 K. After the Nb sample reached its base temperature, a controlled warm-up was performed to monitor the exact region of superconducting transition (inset in Fig.4b).

The performance of our cryogenic LCR measurement system was evaluated by measuring the dielectric constant of $SiO_2$ as a function of temperature. **Fig.5** displays the dielectric constant vs. temperature measured for a Pd/$SiO_2$/Si(001) MOSCAP from 300 K down to 9.5 K. To obtain these results, the capacitance of the MOSCAP was measured at each temperature at a drive frequency of 1.5 MHz and an AC drive voltage of 500 mV. The dielectric constant was then calculated using the equation used for conventional parallel plate capacitors (shown in **Fig.5**), where *d* is the thickness of $SiO_2$ film (d = 100 nm) and *A* is the area of the measured MOSCAP (A = 0.785 $mm^2$). At room temperature, the dielectric constant is ~3.83 consistent with previous reports on thermally-grown $SiO_2$/Si structures [9] [10]. Once below 200 K, the $SiO_2$ dielectric constant starts to decline at a steady rate reaching 2.43 at the base temperature of 9.5 K.

## Discussion and Conclusions

The cryogenic measurement platforms demonstrated here are capable of reaching sub-10 K temperatures in less than 3 hours, which makes them suitable for providing rapid feedback on materials and device fabrication processes. With a compact shape and flexible installation geometry, these cryogenic systems provide a cost-efficient solution for the rapid characterization of cryogenic classical and quantum electronics. The heaters on our cryostats allow the sample temperature to be controlled during cool-downs and warm-ups. This provides the possibility to focus on specific temperature windows above the base temperature to study the thermodynamics of phase transitions observed via electrical measurements. It should be noted, however, that the limitations on the size and types of heaters (e.g., polyimide tape heaters) can prevent precise temperature control above 100 K during warm-up measurements. Nevertheless, the rapid turn-around time of the GM-based cryocoolers allows our platforms to be efficient tools to characterize multiple samples daily.

The cryogenic AC/DC measurement system was successfully utilized for the characterization of superconducting behavior in Nb thin films. The transition temperature was evaluated in less than 2.8 hr. With such rapid cool-down times, we will be able to characterize multiple superconducting thin films (up to 4) each day. However, given the limited base temperature, the system will be limited to characterizing superconductors with Tc's above 7.5 K. Moreover, our cryogenic LCR system successfully demonstrated measurements of the dielectric constant for thermally-grown $SiO_2$ thin films integrated into MOSCAPs. The results showed a significant decline in the dielectric constant from 3.83 to 2.43 with an onset at about 200 K. In the future, we will investigate the dependence of this onset on the chemistry and thickness of the dielectric layer. This cryogenic LCR system is of particular interest for characterizing thin films of complex oxides with applications in phononic and hybrid quantum hardware [11].

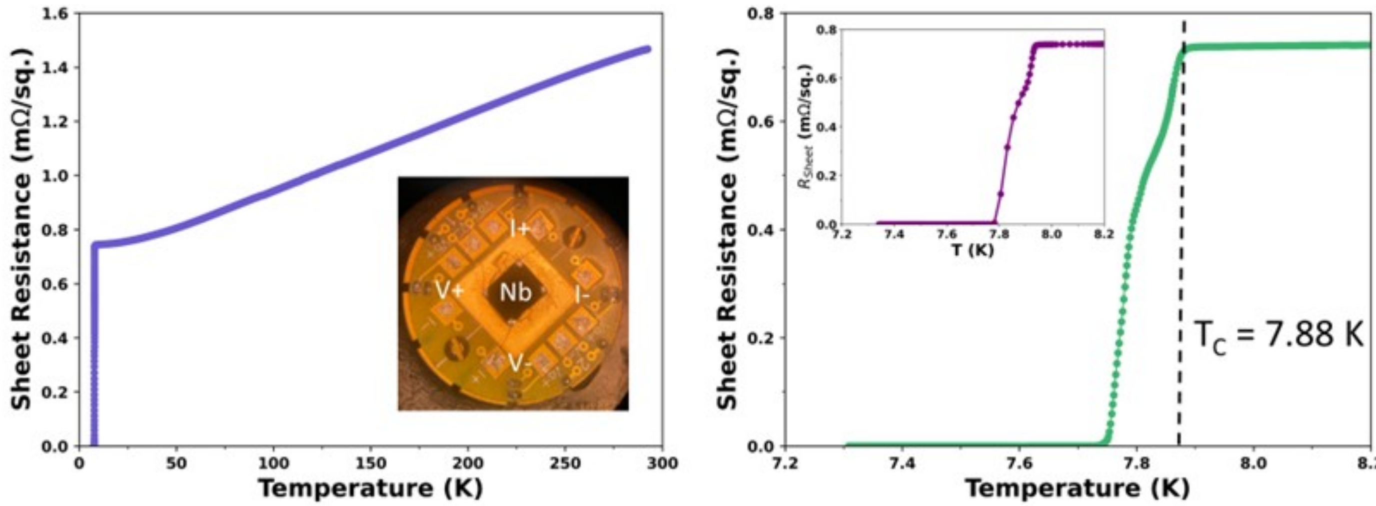


**Fig 4:** Sheet resistance vs. temperature for an Nb/Si thin-film test sample measured in our AC/DC cryogenic measurement system: (a) cool-down trace from 300K to 7.3K: the rectangular Nb sample was wired in the VdP configuration. (b) normal-to-superconducting transition observed below 8K with the dashed line indicating the critical temperature of the sample at 7.88 K. The inset on the right shows the superconducting transition during a controlled warm-up experiment on the sample.

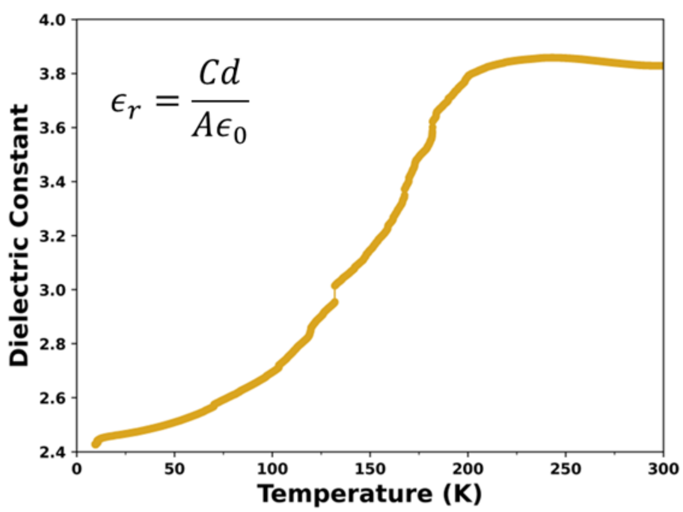


**Fig 5:** Dielectric Constant vs. temperature for a 100 nm thick $SiO_2$ film evaluated by our cryogenic LCR measurement system: throughout the cool-down process the capacitance for a Pd/$SiO_2$/Si MOSCAP was continuously measured via an LCR meter. The dielectric constant was then calculated from the recorded capacitance values using the parallel plate capacitance formula.


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## Notes and References

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