Role of non-linear effects and standing waves in microwave spectroscopy: Corbino measurements on superconductors and VO$_2$

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Broadband microwave spectroscopy can probe material properties in wide spectral and temperature ranges, including superconductors at cryogenic temperatures. The quality of such measurements crucially depends on the calibration, which also removes from the obtained spectra signatures of standing waves. Here we consider low-temperature reflection measurements in Corbino geometry, and we show that the non-linear response of superconducting samples close to the critical temperature can lead to strong signatures of standing waves even in a well-calibrated Corbino spectrometer. We demonstrate our findings with microwave measurements as a function of frequency and temperature for a variety of superconducting samples and for different length of the microwave transmission line. Finally we show that such non-linear effects extend beyond the case of superconductors by probing a VO$_2$ thin film at the insulator-metal transition.

I. INTRODUCTION

Optical spectroscopy can elucidate the electronic properties of a wide variety of materials.$^{11,12}$ Here the experimental spectral range has to match the phenomena of interest, and microwave spectroscopy, typically performed at frequencies in the range 1 GHz to 25 GHz, is suited to probe rather low energy scales in solids.$^4$ One material class of particular interest is superconductors: microwaves can probe the quasiparticle dynamics as well as the Cooper pair response$^{4,5}$ and for superconductors with very low critical temperature $T_c$ even the superconducting energy gap can be accessed.$^{5,6}$

Microwave spectroscopy on superconductors faces two main challenges: firstly, the microwave absorption of superconductors is typically very small, and therefore the sensitivity of the experiment has to be optimized. Secondly, due to the cm-range wavelength at GHz frequencies, typical dimensions of an experiment are such that partial reflections of the microwaves (e.g. at connectors/discontinuities of the microwave transmission line) cause pronounced standing waves that in frequency-dependent data show up as Fabry-Pérot oscillations and that overlap the signal of interest. Such standing waves are particularly difficult to keep track of for cryogenic experiments because the standing wave pattern depends on damping and phase shift of the microwave lines, and these strongly depend on temperature.

There are two quite different approaches to overcome these challenges. The first is employing as probe a microwave resonator that is perturbed by the superconducting sample of interest. If the intrinsic losses of the resonator are small, then the resonator reacts very sensitively to the sample properties, and also its bandwidth will be much narrower than the characteristic frequencies of the standing waves in the microwave setup. Therefore one can detect resonator frequency and bandwidth without being affected by parasitic standing waves.$^{10,11}$ The big disadvantage of such resonant setups is that one does not obtain any information about the frequency dependence of the sample response, unless one uses resonators with multiple frequencies.$^{12,13}$ If instead one aims at the full frequency dependence, then one has to turn to broadband microwave spectroscopy.$^{14-17}$ Here the most common probe geometry for the study of superconductors is Corbino reflectometry$^{22,23}$ where the flat sample terminates a coaxial transmission line, i.e. the sample shorts inner and outer conductors, and it thus reflects the microwave signal that travels on the line. If the superconducting sample is a very thin film, then its effect on the reflected microwave is strong enough to be detected,$^{24}$ and one can directly determine the frequency-dependent complex conductivity of the sample from the measured complex reflection coefficient $S_{11}.$$^{25}$ In such an experiment, standing waves cannot be avoided completely, and therefore appropriate calibration is needed.

Such broadband calibration of a cryogenic microwave setup is very demanding, and several strategies have been developed for both reflection$^{25-28}$ and transmission measurements.$^{29,30}$ For cryogenic Corbino spectrometers, a rigorous calibration involves three different standards (usually open, short, and load) that have to be measured at all frequencies and temperatures of interest.$^{29,30,31,32}$ and several groups have demonstrated successful, fully calibrated cryogenic Corbino measurements on highly conductive thin film samples.$^{29,30,31,32}$

As we will show, even a well-calibrated broadband microwave experiment can be prone to standing waves, namely when non-linear effects play a role. This is the case for superconducting samples close to $T_c$, when the sample properties depend strongly on temperature. Non-linear effects in superconductors have been studied previously by microwave experiments$^{15}$ but not in the context of broadband Corbino spectroscopy.

II. EXPERIMENT

We perform temperature-dependent Corbino reflectometry measurements, using several setups as listed in Table I. Setups #1, #2, and #3 employ a Hewlett-
Packard HP 85107B vector network analyzer (VNA) and cover the frequency range from 45 MHz to 40 GHz, \[39,56\] whereas setups #4 and #5 use an Agilent Technologies E5071C ENA series VNA for frequencies 300 kHz to 20 GHz. For all experiments the microwave output power of the VNA was set to a constant value throughout the full measured spectral range.

The calibration of the setups differs as follows: setups #1 and #2 (\(^4\)He bath cryostat) were fully calibrated with three standard samples (teflon as open, bulk aluminum as short, NiCr film as load) at each temperature of a sequence of measurements, following the established procedure with separate cooldowns.\[39,41\] Setup #3 (\(^3\)He bath cryostat) can also be fully calibrated this way\[55\] but for experiments as presented below (Figs. 2 and 3), with numerous finely-space measurements for different temperatures or powers, we use simpler normalization, which is sufficient to present the spectral features of interest in this study. The same holds for setup #4 (\(^4\)He flow cryostat; various coaxial cables) whereas its modification, setup #5 (\(^4\)He flow cryostat; single semirigid cable), was fully calibrated for all temperatures.

Table I provides an overview of the different microwave setups used in this study. The coaxial cables are different types of semirigid coax; setup #4 was used with several different additional flexible room-temperature coaxial cables to change the length of the microwave line. The electrical length \(L_\text{el}\) of the coaxial cables is calculated from the physical length \(L_\text{phys}\) via Eq. (6).

### III. BROADBAND MEASUREMENTS ON SUPERCONDUCTING THIN FILMS

#### A. Phenomenology: Corbino measurements at the superconducting transition

\[\text{Fig. 1(a)}\] shows the temperature-dependent dc resistance \(R_{dc}\) of an 8 nm thin aluminum film on sapphire with clear superconducting transition at \(T_c \approx 2.0\) K\[39\] which is substantially higher than \(T_c \approx 1.2\) K of bulk aluminum.\[55\] \[\text{Fig. 1(c)}\] shows Corbino spectra, in particular the real part \(\text{Re}(\bar{S}_{11})\) of the reflection coefficient \(\bar{S}_{11}\), obtained on this sample in setup #1 for several temperatures close to \(T_c\). Here we focus on rather low frequencies below 1 GHz although we can observe the feature of interest up to much higher frequencies. For the lowest temperatures, \(\text{Re}(\bar{S}_{11})\) is close to -1.0, corresponding to a microwave short, as expected for a superconducting sample. With rising temperature, \(\text{Re}(\bar{S}_{11})\) increases to an almost constant value around -0.6 for temperatures above \(T_c\). The rather flat spectra at temperatures well below and well above \(T_c\) demonstrate that the cryogenic three-standard calibration works well for this setup.\[55\] But for temperatures near \(T_c\), the reflection coefficient exhibits pronounced oscillations as a function of frequency.

In general, the complex sample impedance \(\hat{Z}\) is directly related to \(\bar{S}_{11}\) via\[56\]

\[
\hat{Z} = Z_0 \frac{1 + \bar{S}_{11}}{1 - \bar{S}_{11}} \tag{1}
\]

and can be calculated from the experimental \(\bar{S}_{11}\) (with \(Z_0 = 50\) \(\Omega\) the characteristic impedance of the coaxial line). The corresponding \(\hat{Z}\) spectra of the aluminum sample, shown in Figs. 1(d) and 1(e) for real and imaginary parts, also oscillate for temperatures close to \(T_c\). To characterize those oscillations, a Fourier transform...
is performed on the $\text{Re}(\hat{S}_{11})$ spectra and shown in Fig. 1(b). Here the x-axis quantity we call timefrequency $\tau_{\text{FT}}$, defined as the inverse of any period $p$ observed in the frequency domain, $\tau_{\text{FT}} = \frac{1}{p}$. Evolving from low to high temperatures, a peak in the Fourier transform at time-frequency $\tau_{\text{osc}} = 17.7$ ns first grows, but then vanishes again, which matches the occurrence of the oscillations in the frequency domain in Fig. 1(c) only near $T_c$.

To demonstrate that these oscillations are a generic feature and not restricted to a particular setup or sample, we show a quite different case in Fig. 2, namely spectra for the heavy-fermion superconductor UNi$_2$Al$_3$ [6] (with $T_c = 1.0$ K evident from the in-situ dc data shown in Fig. 2(a)) as a thin film on YAlO$_3$ [55] in stripe geometry [69] measured in a $^3$He Corbino spectrometer (setup #3). In Fig. 2(b) we show $|\hat{S}_{11}|$ spectra for several temperatures, normalized to $|\hat{S}_{11}|$ at 1.2 K visible in Fig. 1(b). Here the x-axis quantity we call timefrequency $\tau_{\text{FT}} = \frac{1}{p}$ as a function of temperature and power. Again, which matches the occurrence of the oscillations in the frequency domain in Fig. 3(c) for different applied microwave powers. As our third example, we discuss microwave spectra obtained in setup #3 on a strip-shaped $\approx 60$ nm thick lead film [77] on glass, with dc data and $T_c \approx 7.3$ K visible in Fig. 3(a). Microwave spectra obtained near $T_c$ are shown in Fig. 3(b) for several temperatures and in Fig. 3(c) for different applied microwave powers. As in the previous cases, pronounced oscillations are visible in the spectra only for certain combinations of temperature and power. Again we quantify these oscillations by their Fourier transform, which we evaluate at the time-frequency of this dominant oscillation, $\tau_{\text{osc}} = 24$ ns, and obtain the corresponding amplitude $|A_{\text{osc}}| = |A_{\text{FT}}(\tau_{\text{FT}} = 24 \text{ ns})|$ of the oscillations. Fig. 3(d) plots $|A_{\text{osc}}|$ as a function of temperature and power, and from the clear maximum in these data it is evident that the oscillations only occur in a narrow regime of combinations of temperature (near $T_c$) and power, with higher powers required for lower temperature. (E.g. the combinations {7.30 K, 6 dBm} and {7.23 K, 0 dBm} lead to maximal oscillations for either a given temperature or power.)
FIG. 3. Temperature- and power-dependent microwave measurements of a lead thin film near $T_c$, using setup #3. (a) Temperature-dependent dc resistance. (b) Spectra of Re($S_{11}$) for several temperatures, at fixed microwave power of -6 dBm. (c) Spectra of Re($S_{11}$) for different applied microwave powers, at the same nominal temperature 7.275 K. For certain combinations of temperature and power, (b) and (c) exhibit clear oscillations.

Dashed lines indicate the tuning of either temperature or power for the spectra shown in (b) and (c).

B. Cause of oscillations in spectra: sample heating due to standing waves

Observing regular oscillations in broadband microwave spectra usually calls for consideration of standing waves, but our present phenomenon of the oscillations appearing only near $T_c$ goes beyond conventional interference in microwave transmission lines. As evident from the spectra well below or well above $T_c$ in Fig. 1 that do not show oscillations, this nominal temperature is well calibrated, i.e. the calibration scheme effectively removes signs of standing waves from the raw data. Furthermore, some weak oscillations as remnants of a not-perfect calibration in spectra well below $T_c$ in Figs. 2b and 3b can have a very different oscillation period than the additional, strong oscillation that occurs only near $T_c$.

As we will demonstrate, the cause for the oscillation near $T_c$ is the combination of standing waves and non-linear behavior of the sample under study. Depending on the standing wave formation in the setup, the microwave power present at the sample position can oscillate as a function of frequency, even though the power submitted by the VNA into the setup is the same for all frequencies and the absorption properties of coaxial cables and sample only vary weakly with frequency. Oscillating microwave power at the sample means oscillating power dissipation in the sample, which causes heating of the sample, i.e. the actual sample temperature can differ from the nominal temperature measured by the nearby temperature sensor. Since the impedance of the sample strongly depends on temperature near $T_c$, the oscillating sample temperature means oscillating sample impedance, which in turn causes an oscillating reflection coefficient, which is the experimental signature that we observe.

To demonstrate that the oscillations near $T_c$ indeed are related to temperature changes of the sample, we perform in-situ dc measurements while the microwave frequency is swept. In Fig. 4, we show such data obtained in setup #4 on a 100 nm thick NbTiN film on silicon: the microwave spectrum (full line) obtained at sensor temperature 7.821 K (slightly below $T_c$) and normalized to a spectrum slightly above $T_c$ shows pronounced oscillations as discussed before, though with a more complicated frequency dependence than in the previous cases. The simultaneously measured dc resistance also oscillates substantially (full circles), and the observed oscillatory behavior closely matches the one of the $|S_{11}|$ spectrum, thus confirming that the two are directly related.

Next we evaluate the standing waves in more detail. As mentioned in the introduction, standing waves are a serious challenge for broadband microwave spectroscopy, in particular at cryogenic temperatures. A main goal of the different calibration schemes is to take the standing waves into account, i.e. spectra obtained with a properly calibrated setup should not feature standing-wave-caused oscillatory behavior. However, if the calibration is imperfect, then the calibrated spectra often contain standing-wave-type oscillations, which do not necessarily reflect the dominant standing waves physically present in the setup, but rather the dominant systematic error of the calibration in terms of standing waves. In contrast, for the present oscillation observed close to $T_c$ the cause...
is heating due to the standing waves physically present at the sample position, i.e. it is independent of the calibration scheme. A Corbino measurement on a superconducting sample has (at least) two elements that reflect the microwave signal on the line, namely the sample of interest and the VNA. If the coaxial line connecting VNA and Corbino probe does not feature any impedance mismatches, the standing wave pattern is governed by the total electrical length of this transmission line.

To test this influence of the length of the coaxial cable, we take advantage of the substantially different cable lengths of the setups listed in Table I. The characteristic timefrequency, i.e. the time the signal needs for one round trip between the reflecting points on the coaxial line that cause the Fabry-Pérot-type oscillations, is the inverse of the distance between the resonance frequencies, \( f_k \) and \( f_{k+1} \), of two adjacent standing-wave modes (with mode number \( k \) and \( k+1 \)) and depends on \( L_{\text{el}} \) as follows (with \( c_0 \) the speed of light in vacuum):

\[
\tau_{\text{osc}} = \frac{1}{p_{\text{osc}}} = \frac{1}{f_{k+1} - f_k} = \frac{1}{\frac{(k+1)c_0}{2L_{\text{el}}} - \frac{k c_0}{2L_{\text{el}}}} = \frac{2}{c_0} L_{\text{el}}
\]

For the measurements with setups #4 and #5, which share the same VNA and cryostat, we observed two distinct timefrequencies, and both are plotted in Fig. 5 (These two separate oscillation frequencies are also the reason for the 'beating' pattern visible in Fig. 4). Based on Eq. 3, we expect linear behavior for Fig. 5 with slope \( 2/c_0 \), but with an unknown offset because our \( L_{\text{phys}} \) does not contain the part of the transmission line within the VNA. Indeed we find that the data can be classified as three groups with different offsets (setups #1 to #3 with HP 85107B VNA, first oscillations of setups #4 and #5 with ENA E5071C VNA, and their second oscillations, respectively) but all with the expected slope \( 2/c_0 \), as indicated by the straight lines in Fig. 5.

These experiments confirm the interpretation that the pronounced oscillations in the Corbino spectra near \( T_c \) are caused by the combination of standing waves on the coaxial line and pronounced non-linear response of the sample in the vicinity of the superconducting transition. This also explains the very 'inharmonic' oscillation spectra that we have observed in several cases e.g. in Fig. 1 c)-(e) (blue curves), where the sample resides in the superconducting state for certain frequencies with low local microwave power, but if the standing waves induce a local microwave power that is strong enough to cause a temperature increase of the sample into the steep section of the \( R_{dc}(T) \) curve, then the sample impedance will increase drastically as evidenced in the observed spectra. The observed oscillations in the \( S_{11} \) spectra (e.g. Fig. 1 c)) thus indeed stem from oscillations of the sample impedance as shown in Figs. 1(d) and (e), but this is not an intrinsic frequency dependence of the sample but a measurement artifact caused by the oscillating sample temperature caused by the physical standing waves at the sample.

IV. BROADBAND MEASUREMENTS ON VO\(_2\)

While the superconducting transition might be the most prominent case of a steep \( R_{dc}(T) \) curve, the above explanation can apply to any material with strong impedance changes as a function of temperature. Therefore, we now consider a very different case, namely thin-film VO\(_2\) with its well-studied transition around 340 K from a low-temperature insulating phase to a high-temperature metallic phase. Here we use setup #5 to study a 200 nm thick VO\(_2\) film grown on sapphire with the insulator-metal transition evident from the dc data shown in Fig. 6(a). Microwave spectra of Re(\( S_{11} \)) obtained for several temperatures near the transition are shown in Figs. 6(b), (c), (d) and (e). Here, the oscillations of interest are less visible than for the superconducting examples, but the Fourier transform in Fig. 6(f) indicates two pronounced peaks at 9 ns and 14 ns, which are strongest for temperatures 337 K to 338 K, consistent with the steepest slope in Fig. 6(a). Furthermore, when investigating the oscillations as a function of power, shown in Figs. 6(g) and (h), we can clearly see that the oscillations strongly increase with power.

The phenomenology and explanation described above...
for superconductors thus also applies to other materials. But the amplitude of the oscillations for the case of VO₂ is much weaker than for the superconducting examples, and this can be explained as follows: firstly, the impedance change due to heating is stronger for steeper temperature dependence. While the superconducting transitions above have width of order 100 mK, the transition in our VO₂ measurement extends over several K. Secondly, if the standing waves induce an impedance change in a superconductor via heating, then the impedance will increase, leading to even more absorption; i.e. the process is self-enhanced. For our VO₂ case, Fig. 1(d) with substantially smaller |S₁₁|, this effect will be weaker and even self-inhibiting for somewhat higher temperatures (for Z smaller than Z₀ and negative R(T) slope). Thirdly one has to consider the thermal properties of the materials involved, i.e. how much the sample temperature increases for a given absorption of microwave power. The rather low specific heat of many materials at cryogenic temperature thus might further enhance the sensitivity of superconductive samples with respect to the standing-wave oscillations.

V. IMPLICATIONS FOR CORBINO SPECTROSCOPY

We have shown that broadband Corbino measurements are prone to oscillatory artifacts in the microwave spectra that stem from standing waves combined with non-linear sample response due to sample heating. This phenomenon can easily occur in Corbino measurements on superconducting thin films, but is much more general both in terms of materials under study as well as probe geometry, i.e. it could occur in any frequency-dependent microwave measurement on any non-linear sample. Considering the typical three-standard reflection calibration it is thus important that the calibration standards do not exhibit non-linearity.

In spectroscopy, one is typically interested in the intrinsic material response and thus wants to avoid extrinsic contributions that affect the measured spectra. We thus briefly discuss possible strategies to minimize the unwanted oscillations. One obvious step is reducing standing waves in the coaxial line as much as possible, which will also help for the different calibration schemes. But as mentioned, reflection can neither be avoided for the sample nor for the VNA. In principle one can suppress standing waves on the coaxial line by increasing the damping, e.g. using attenuators, but increased attenuation leads to even stricter requirements for the calibration, which already is the limiting factor for cryogenic Corbino spectroscopy. Thus there are no simple hardware perspectives to reduce the oscillations.

Concerning the actual microwave measurements, an obvious strategy is applying as low VNA output power as possible such that the non-linear response does not set in. In many instances this will be possible, in particular if the frequency range of interest is small. But if one is interested in wide frequency ranges, e.g. up to 20 or even 40 GHz as already demonstrated, then one faces the problem that the attenuation in the microwave line increases with frequency. Thus, if one applies the same output power for all frequencies, then the power of the reflected signal reaching the VNA detector will be comparably low for highest frequencies, and thus the output power has to be high enough to allow reliable detection. The lower damping at lower frequencies then can lead to much higher power at the sample for low frequencies, which then could induce the non-linear response. A possible solution could be an additional amplifier for the reflected signal before being detected by the VNA.

Another approach could be a frequency-dependent VNA output power chosen such that the microwave power at the sample is frequency-independent. Such a ‘power flatness’ procedure can be applied for room-temperature testing using VNAs and is implemented by measuring the microwave power at the position of interest using a dedicated power sensor. In our case, this strategy is not viable for two reasons: firstly, since damping and phase shift of the microwave line strongly depend on temperature, the power-flatness procedure would be needed.
for cryogenic temperatures and for each temperature of interest separately. Secondly, and more fundamental, the relevant standing wave pattern depends on the sample impedance and thus would not be properly reproduced during a power measurement at the sample position with a dedicated microwave sensor.

With no ideal solution at hand, our approach to address this experimental problem will be careful consideration of the applied microwave power and inspection of the measured spectra for possible signatures of this unwanted effect.

VI. CONCLUSION

We have shown that experimental microwave spectra of superconducting samples near $T_c$ can contain signatures (‘oscillations in the spectra’) of standing waves in the setup even if a full low-temperature calibration is successfully applied. These oscillations are caused by the combination of non-linear response of the sample and frequency-dependent microwave power at the sample position, with the latter caused by the standing wave pattern of the setup. This effect is generic to any broadband microwave measurement and non-linear sample, but is particularly relevant for the study of superconductors. Considering the diverse research activities with microwave spectroscopy studying phenomena related to superconductivity, we expect that the ‘standing-wave non-linear oscillations’ have to be taken into account carefully in numerous instances to optimize experimental procedures that then reveal the fundamental material properties of interest.

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From independent measurements we know that $T_c$ of the studied NbTiN sample is several K higher than suggested by the data in Fig. 4. This is due to the distance between sample and temperature sensor and the large temperature gradients in the probe of setup #4 when operated at such low temperatures.

Flexible Pasternack Precision Cables, 095 Series PE35611. The used cables have a physical length of 16 cm, 31 cm, 46 cm, and 77 cm, respectively.

Manufacturer specifications are for room temperature, and we neglect the temperature dependence of $L_{el}$.