Complex conductivity measurements between 26 and 110 GHz using complex impedance bridges

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A method is described of measuring complex conductivity based upon the measurement of the impedance of a sample placed in a waveguide using a complex impedance bridge. From the observed changes in the bridge parameters, viz., attenuation and phase shift, the impedance and, hence, the complex conductivity are inferred. The principles and details of operation of three bridges covering the frequency range 26 to 110 GHz are discussed. The measurement technique was verified for lossless samples and for lossy resistive wires. An example of temperature and frequency dependent complex conductivity measurements on a linear chain material is presented.

INTRODUCTION

The millimeter wave frequency range has long been a relatively inaccessible spectral region for the study of materials. Conventional studies of the dynamics of transport are typically carried out in the microwave range (10 GHz or below) on the low-frequency side, and continue again in the optical range from the very far infrared (400 GHz) and above.

Recent developments in the broad area of interactions and disorder in materials indicates that the relevant energy scale for transport processes may, in certain cases, be orders of magnitude smaller than the relevant single particle energies, the bandwidth and [where a semiconducting state develops] the bandgap. Interactions in general lead to collective modes, such as charge-density waves (CDW) or spin-density waves (SDW). These collective modes can support electrical conductivity, resulting in strongly frequency dependent transport phenomena below infrared frequencies. Disorder leads to localization and to a dense set of localized electron states. Transitions between localized energy levels lead also to frequency dependent conduction at frequencies well below those which correspond to single particle energies. From experiments below 10 GHz, it is clear that studies in the millimeter-wave spectral range would throw considerable light on dynamical processes in these materials. Recent technological developments in the communication industry, particularly in the manufacture of solid state devices have reduced (somewhat) the hitherto prohibitive cost of broadband measurements above 26 GHz and have made this frequency range economically accessible.

In this paper we describe a very versatile technique utilizing a complex impedance bridge to measure the temperature and frequency dependent complex conductivity of thin long samples in the frequency range 26 to 110 GHz. We have employed this technique to study several quasi 1D materials and have obtained important information regarding the fundamental parameters that determine transport in the case of materials which display CDW dynamics. The technique is particularly suitable to needle shaped specimens, but can be easily extended to a variety of other materials and physical conditions.

These high-frequency measurements are supplemented by measurements at lower frequencies employing a variety of techniques. In the microwave frequency range, contactless cavity perturbation techniques have been utilized with a variety of high-Q cavity resonators at fixed frequencies. Here the samples are typically much smaller than cavity or waveguide dimensions. Consequently the perturbation caused by the samples is small and bridge configurations do not have the sensitivity required.

Our preliminary experiments at 94 GHz revealed that cavity perturbation techniques were impractical at high frequencies, mainly due to the very small cavity sizes. It was then decided to exploit the fact that at these frequencies, waveguide dimensions and sample lengths are comparable and, hence, a sample in a waveguide would cause a measurable change in waveguide impedance. This paper describes how this change, which is observed as a change in bridge parameters, is used to determine the complex conductivity of the sample.

This paper is organized as follows. Section I discusses the principles of operation of the bridge and of measurement of complex impedance. The impedance and equivalent circuit of needle shaped specimens is discussed in Sec. II, and the response of a bridge to such samples in Sec. III. Details of the experimental method and test measurements on lossless samples are described in Sec. IV. In Sec. V, we describe temperature dependent complex conductivity measurements on a lossy resistive material. Then, in Sec. VI, an example of a temperature and frequency dependent measurement of the complex conductivity of a charge-density wave material is discussed.

I. PRINCIPLE OF COMPLEX IMPEDANCE MEASUREMENT USING A BRIDGE

The bridges were used in a reflection-type configuration. The layout of the components is displayed in Fig. 1. Three such systems, covering the Ka (26–40 GHz), V (50–75 GHz), and W (75–110 GHz) bands are currently in operation.
A bridge is essentially an interferometric type of device. Power from the source is split into two components, one traversing the reference arm, and the other the sample arm, and recombined to provide a detected output, determined by the relative conditions of the two arms. The wave traveling the reference arm is reflected from a fixed short. In the sample arm, the wave is reflected from the unknown impedance which is to be measured. The bridge is nulled (zero detected output) by adjusting the attenuator \# 1 and the phase meter. At this condition, the attenuations in the two arms are equal and the phase shifts differ by an integral odd multiple of \( \pi \).

The input impedance of an unknown device may be measured by first terminating the sample arm with a short and noting the attenuator and phase readings, \( A_0 \) (in dB) and \( \phi_0 \), respectively, when the bridge is nulled. Then the short is replaced by the device and the null settings \( A_1 \) and \( \phi_1 \) noted again. Let \( A_i = A_i - A_0 \) and \( \phi_i = \phi_i - \phi_0 \) be the differences in the settings. Then the impedance of the device normalized to the characteristic waveguide impedance, can be determined from

\[
Z = \frac{(1 + S) / (1 - S)}{\frac{i}{\phi_0}},
\]

where \( S = -10^{A/20} \) is called the scattering parameter. This impedance is referred to the plane of the initial short. If we wish to transform the reference plane by a distance of \( l \), then the scattering parameter relative to the new plane is \( S e^{|\phi_0 / \lambda_0} \), where \( \lambda_0 \) is the guide wavelength at the measurement frequency. This can be repeated at different frequencies and the frequency dependence of the impedance thus obtained. The bridge is actually a very versatile device and can yield the impedance of both active and passive devices.

For conductivity measurements on needle like specimens, the sample needs to be placed in a waveguide along the \( E \)-field lines, and at a position of maximum sensitivity. The sample holder was constructed with these features in mind. It consists of a short (-1 in.) waveguide section terminated by a metal shorting plate (Fig. 2). A small (0.025-in. diameter) hole enables the insertion of the sample. The sample position is at a distance of \( l \) away from the short and midway in the wide direction of the waveguide. At this position, the \( E \) field is a maximum for the frequency of measurement (for which \( l = 3 \lambda_0 / 4 \)). The samples we discuss here are quartz rods, a metal wire or quasi 1D needles attached to a quartz fiber.

The measurement technique consists of nulling the bridge with and without the sample and noting that changes \( A_i \) and \( \phi_i \) in attenuation and phase readings. Without the sample, the terminating impedance is a short [Fig. 2(b)]. As discussed later in Sec. II, the impedance due to very thin needle like samples is a pure shunt, which we call \( Z_s \). With the sample, the terminating impedance is \( Z_r \) referred to the plane \( P \) at the sample position. Since \( P \) is at a distance \( l = 3 \lambda_0 / 4 \) away from the short for the chosen measurement frequency, the short transforms into an infinite impedance in parallel with \( Z_r \), as depicted in Fig. 2(c). For this situation, \( S = 10^{-A/20} \) and \( Z_r \) can be determined from Eq. (1). (The situation for other frequencies within the band for which \( l \neq 3 \lambda_0 / 4 \) is discussed in Sec. IV.)

II. IMPEDANCE OF LONG, THIN SAMPLES

The sample which is placed along and at a maximum of the electric \( E \) field lines, acts as an obstacle in the waveguide. Several solutions\(^7\) for the impedance due to such an obstacle are available. In general, the equivalent circuit for such a configuration is a \( T \) circuit. However, the impedances in the \( T \) arms are smaller by a factor \( (d / \lambda)^2 \), where \( d \) is the sample diameter, than the shunt impedance. Therefore, when \( d < \lambda \) (as is always the case in the present measurements), the equivalent circuit is a pure shunt impedance which we call \( Z_s \). The shunt impedance of a sample with complex \( \sigma = \sigma_r + j \sigma_i \) can be written

\[
Z_s = Z_{s_0} + Z_{s_0}.
\]

(The expressions below can also be written in terms of the dielectric constant \( \varepsilon = 1 + \varepsilon_r - 1 - \varepsilon_j \), where \( \varepsilon_r \) is the permittivity of free space.) \( Z_{s_0} \) is purely reactive (\( = jX_{s_0} \)) and is independent of \( \sigma \). \( Z_{s_0} \) can in general be complex.

Explicit expressions for \( Z_{s_0} \) and \( Z_{s_0} \) have been obtained by Schwinger,\(^7\) when the obstacle is a cylindrical rod placed across the waveguide and making electrical contact with the walls. Then \( Z_{s_0} \) is purely inductive (which we call \( Z_{s_0} \)). For a sample placed at the center, and for which \( d \ll \lambda \) the results are
\[
Z_{\infty} = j\left(\frac{a}{2\lambda_{g}}\right) \log\left(\frac{4a}{\pi d}\right) - 2 + 2\sum\left[\left(\frac{2a}{\lambda}\right)^{n}\right].
\]

(3)

\[
Z_{e} = j\frac{a}{2\lambda_{g}} \frac{J_{0}(Y)}{Y} \frac{1}{X J_{0}(Y) J_{1}(X) - Y J_{0}(X) J_{1}(Y)},
\]

where \(X = \pi d / \lambda, Y = (\sigma / \omega \epsilon_{0})^{1/2} \pi d / \lambda, \) and \(J_{n}\) is the Bessel function of order \(n\). The above form for \(Z_{e}\) is cumbersome for the evaluation of the sample parameter \(\sigma\). More useful are several limiting forms discussed below.

(a) Loss-less dielectric (\(\sigma = 0\))

If \(\sigma\) is small so that \(Y^{2} = \sigma / (\pi d / \lambda)^{2} / \omega \epsilon_{0} \ll 1\), the Bessel functions can be expanded for small \(Y\). Then

\[
Z_{e} = Z_{\infty} - j\frac{a}{\lambda_{g}} \left(\frac{\lambda}{\pi d}\right)^{2} \omega \epsilon_{0},
\]

(5)

The equivalent circuit is a shunt capacitance (for \(Y^{2}\) small, \(Z_{\infty}\) is negligible).

(b) Metallic wire (\(\sigma \rightarrow \infty\))

Here \(Z_{e} \rightarrow 0\) and \(Z_{e} = Z_{\infty} L\) and is given by Eq. (3), when the wire touches the walls. The equivalent circuit is a shunt inductance.

In both the above cases the samples present purely reactive impedances and do not lead to absorption. The next two cases have real resistive parts and do lead to losses.

(c) Skin Depth Regime (\(\sigma = 0\), skin depth \(\delta < d\))

If \(\sigma\) is large so that the skin depth \(\delta = (2 / \mu_{0} \sigma \epsilon_{0})^{1/2} < d\), where \(\mu_{0}\) is the permeability of free space, then the large \(Y\) limit of the Bessel functions applies

\[
Z_{e} = Z_{\infty} + (1 + j) \left(\frac{a}{2\lambda_{g}}\right) \left(\frac{\delta}{d}\right).
\]

(6)

In this skin depth regime, the apparent shunt inductance is larger than \(Z_{\infty} L\) due to the slight penetration of the fields into the sample. The equivalent circuit consists of a resistance and inductance in series. Both impedances in the second term are small compared to \(Z_{\infty} L\), since \(\delta < d\).

(d) Sample with complex \(\sigma (\delta > d)\)

The most useful case is where \(\sigma\) is complex

\[
Z_{e} = Z_{\infty} + \alpha \frac{1}{\sigma_{r} + j\sigma_{i}},
\]

(7)

where \(\alpha = \omega \epsilon_{0} (a / \lambda_{g})^{2} / \pi d^{3}\). The above expressions are valid when \(\sigma_{r} < 8(\lambda / \pi d)^{2} \omega \epsilon_{0}\), before the next term in the small \(Y\) expansion of the Bessel function becomes important, and for \(\delta > d\). The equivalent circuit is shown in Fig. 3 and consists of a parallel combination of a resistance \(R_{e} = \alpha / \sigma_{r}\) and reactance \(X_{e} = -\alpha / \sigma_{r}\) in series with \(X_{\infty}\). It should be noted that the intrinsic parallel reactance can be inductive or capacitive, being determined by whether \(\sigma_{r}\) is negative or positive.

![Fig. 3. Equivalent circuit for the impedance \(Z_{e}\) of a sample with complex conductivity.](image)

All of the above relations were derived for the case where the sample is in contact with the waveguide walls. In practice, it may be possible to make the sample long enough to traverse the waveguide but it is impractical to ensure contact with the walls. Thus gaps exist between the sample and the waveguide, which may be modeled as capacitive impedances \(Z_{m/c}\) in series with the inductive \(Z_{\infty} L\), and \(Z_{e} = Z_{\infty} L + Z_{m/c}\). The relative importance of \(Z_{m/c}\) and \(Z_{\infty} L\) is determined by sample size, with \(Z_{\infty} L\) dominating for long thin samples completely traversing the waveguide and \(Z_{m/c}\) dominating for short samples. We have experimentally verified this interplay of capacitive and inductive effects by varying sample size. Further, as our results described in Sec. IV demonstrate, for long thin samples, Eq. (3) for \(Z_{\infty} L\) describes the results very well.

An exact calculation of \(Z_{\infty}\) for arbitrary sample size is difficult, and at least for the present experiments, unnecessary. In the measurements on complex conductivity, \(Z_{\infty}\) is treated as an unknown quantity which is to be determined experimentally (Sec. V).

III. BRIDGE RESPONSE FOR LONG, THIN SAMPLES

It is useful to examine the response of a bridge in terms of the attenuations \(A_{e}\) and phase \(\phi_{e}\) differences caused by insertion of the samples discussed in the previous section.

For the purely reactive samples viz., dielectric or highly conducting wire, with reactance \(X_{e} = (Z_{e} / j)\) given by Eqs. (3) or (5), there is no absorption. Hence, \(A_{e} = 0\). However, there is a finite phase shift given by \(\phi_{e} = 2 \cot^{-1} (X_{e})\), where \(-\pi < \phi_{e} < \pi\). The insertion of the reactance is equivalent to lengthening or shortening the sample arm, depending on whether \(X_{e}\) is capacitive or inductive respectively. In the present notation, \(\phi_{e}\) is positive for inductive \((X_{e} > 0)\) and negative for capacitive \((X_{e} < 0)\) samples respectively.

An illustrative example of an absorptive sample is a lossy wire of diameter \(d < \delta\), and with \(\sigma_{r} = 0\). From Eq. (8), the equivalent circuit is a resistance \(R_{e} = \alpha / \sigma_{r}\) in series with \(X_{\infty}\). In general, \(X_{\infty}\) can be positive or negative. Then using Eq. (1) with \(S = 10^{-A/20} e^{j\phi}\) (corresponding to the sample configuration of Fig. 2)

\[
A_{e} = -10 \log_{10} (P^{2} + Q^{2}), \quad \phi_{e} = \tan^{-1} (Q/P),
\]

(8)

where

\[
P = \frac{R_{e}^{2} + X_{e}^{2} - 1}{(R_{e} + 1)^{2} + X_{e}^{2}}; \quad Q = \frac{2X_{e}}{(R_{e} + 1)^{2} + X_{e}^{2}}.
\]
In Fig. 4, $A_s$ and $\phi_s$ are plotted as functions of $1/R_s = \sigma_s/\alpha$ for $X_\omega = \pm 2, 0.5$. As the conductivity $\sigma_s$ increases, the attenuation initially increases with $\sigma_s$, reaches a maximum value at approximately $R_s \approx X_\omega$, and decreases for higher conductivities. In the low conductivity regime, $R_s \gg X_\omega$, the attenuation is proportional to the conductivity, i.e., $A_s \propto \sigma_s$. In the high-conductivity region, $R_s \ll X_\omega$ and $A_s \propto 1/\sigma_s$. Note that these results are valid only so long as $d > \delta$.

In the low-conductivity regime, the reactive part is small and, hence, the phase shift $\phi_s$ is small. As $\sigma_s$ increases, $\phi_s$ increases monotonically reaching a limiting value $\phi_s = 2 \cot^{-1}(X_\omega)$ when $\sigma_s \to \infty$, i.e., when $R_s \to 0$. As mentioned earlier $X_\omega$ may be either inductive (positive) or capacitive (negative) and, hence, $\phi_s$ may be either positive or negative, though $A_s$ is independent of this sign, as is evident in Fig. 4.

For the two examples discussed in Secs. V and VI, one (TaSe$_4$) has conductivities increasing by nearly a factor 50 from 300 to 15 K. The raw data for $A_s$ displays a maximum at an intermediate temperature and $\phi_s$ decreases from nearly zero to a negative limiting value, thus possessing the qualitative features of Fig. 4. The other example, TaS$_3$, has a $\sigma_s$ which decreases from a low value at 300 K. In this case the low $\sigma_s$ part of Fig. 4 applies at all temperatures. $\phi_s$ is small (typically a few degrees) at all temperatures.

IV. EXPERIMENTAL METHODS AND CALIBRATION MEASUREMENTS

A variety of sources have been used to power the three bridges covering the 26-110-GHz frequency range. In the Ka band, a YIG tuned Gunn oscillator$^{9}$ covering 26-40 GHz was used. Initially in the $V$ and $W$ bands, voltage tuned Gunn oscillators$^{10}$ at 60 and 94 GHz with tuning bandwidths of 1 GHz were employed. None of the Gunn sources required external frequency stabilization. By the use of standard stable power supplies, the long-term frequency drift was less than 10 MHz over many hours, and did not affect the phase measurements.

For broadband measurements in the $V$ and $W$ bands, Impatt sweepers$^{10}$ were employed. However, since these sweepers have appreciable frequency drift (nearly 0.1 GHz/h when turned on), it was found necessary to use a source locking EIP 578 frequency counter,$^{11}$ by which means the long-term frequency variation was reduced to less than 1 kHz.

The power available from these sources (typically 1-10 mW) is entirely adequate and has never been a limitation to the sensitivity of the measurements.

The lengths of the sample and reference arm were made equal to within one guide wavelength (at the midband frequency). This helps to minimize effects of source FM noise and improves phase accuracy. This equalization was verified by noting that for a given frequency, there was only one phase meter setting for which the bridge was nulled.

A useful parameter characterizing the bridge operation is the ratio of the detected output voltage when the bridge is nulled or balanced and when it is unbalanced. The phase meter settings differ by 180° for these two cases. This is equivalent to a very high sensitivity of $10^{-5}$ in a VSWR measurement, and demonstrates that spurious reflections were small and negligible.

The detected ac voltage (the input power was square wave amplitude modulated at 1 kHz) was <1 $\mu$V, and the null setting was conveniently determined by a lock-in amplifier.

The attenuator and phase meter settings at null were determinable to within $\pm 0.025$ dB and $\pm 0.5°$, respectively. These limits are only due to the reading accuracies of the instruments.

A description of the sample holder has already been given in Sec. I. In order to validate the bridge measurement technique using this sample holder, test measurements were carried out on samples with known impedances. The samples used were quartz rods and metal wires, whose impedances are dominantly reactive. The phase shifts due to such samples were measured at different frequencies encompassing a waveguide band and compared with the calculated phase shifts based upon the expressions in the previous sections.

For the sample holder (Fig. 2), $l = 3\lambda_s/4$ only for one frequency within the band, and for this case the discussion of Sec. I applies. At any frequency the short transforms into an impedance $j\tan(2\pi l/\lambda_s)$ in parallel with the sample impedance $Z_s$. Thus with the sample, the terminating impedance referred to the sample position is $Z_s = [1/jX_s + 1/\tan(2\pi l/\lambda_s)]^{-1}$, where we have taken the sample to be purely reactive ($Z_s = jX_s$), while without the sample the impedance is a short at a distance $l$ from the sample position. Then using Eq. (1) with a transformed scattering parameter $S = -e^{\phi_s}$, $e^{\phi_s l/\lambda_s}$, the phase shift can be calculated

$$\phi_s = -2 \left[ \cot^{-1} \left( \frac{1}{X_s} + \frac{1}{\tan(2\pi l/\lambda_s)} \right) - \frac{2\pi l}{\lambda_s} \right].$$
The materials discussed here typically crystallize in long thin fibers or needles. Under a microscope, very thin (approx. 1 x 1 \( \mu \)m) needles were selected and cut to lengths of 2–4 mm. The samples tend to attract electrostatically to dielectric materials and they were so attached to a quartz fiber 0.010-in. diameter, which acted as a mechanical support. The quartz fiber was suspended into the waveguide so that the sample traversed or nearly traversed the waveguide.

The sample was then cooled and the attenuation and phase meter settings at null noted for each temperature. Later after warming up, the sample was detached from the quartz and the measurement repeated with only the quartz in the waveguide. The differences, \( A_t \) and \( \phi_t \), in the readings were then used to determine sample impedances, and hence the complex conductivity, as discussed in Sec. V.

In the temperature-dependent measurements without a sample, the attenuation changed slightly (less than 2 dB) between 300 and 15 K, while the phase change was quite large (nearly 70 degrees, depending on frequency). These results are consistent with thermal contraction of the cooled portion of the waveguide.

A very thin (0.00025-in.-thick) Mylar disk was used as a window at the junction of the SS waveguide and the directional coupler, to prevent gas flow into the bridge system. When the gas flow enclosure was connected to the gas flow tube from the Dewar, initially the air in the waveguide was replaced by \( \text{He}_2 \) gas. The phase reading change due to the flushing with the gas was reproducible to within a degree.

In principle the best method of measuring the perturbation due to the sample may seem to be to move the sample in and out of the waveguide at each temperature. This is quite difficult because of the very small sizes involved. By repeated temperature runs, we have verified that our results are consistently reproducible to within the measurement accuracy. In particular the phase measurements, which are particularly sensitive, are reproducible to within one degree.

The quartz fiber introduces a small perturbation (less than five degrees at the highest frequency) and appears as a large reactance in parallel with \( Z_t \). A simple analysis shows that our procedure for determining \( A_t \) and \( \phi_t \) as the difference of the measurements with and without the sample but with the quartz always present, is insensitive to the reactance.

V. RESULTS: COMPLEX CONDUCTIVITY OF A TEST MATERIAL

The bridge technique was tested at room temperature as discussed in Sec. IV, however, it was still necessary to test for any systematic errors introduced by varying the temperature. In addition, the technique had to be tested on a lossy material. The requirements for a good test material were that the test material be available in fibers with small enough cross section in order to achieve \( \delta > d \), and that the material should be a normal metal over the temperature range of the measurements. In normal metals the conductivity is independent of frequency until optical frequencies, and this implies that the dc conductivity would not differ from the millimeter-wave conductivity. The material selected for the test...
was TaSe₃. It has a conductivity of $2 \times 10^3 (\Omega \text{ cm})^{-1}$ at room temperature which increases by almost two orders of magnitude at $T = 20$ K.

The measurements were performed primarily in the limit, $d < \delta$. This limit is ideal when sufficiently small samples are available because both the real and imaginary parts of the conductivity may be measured. From Eq. (7),

$$\sigma_r(T) - j\sigma_i(T) = \frac{\alpha}{Z_r(T) - jX_w},$$

(10)

where $Z_r(T)$ is determined at different temperatures from the measured $A_r$ and $\phi_r$ at each temperature as described in Sec. II. Thus the $T$ dependence of $\sigma_r$ and $\sigma_i$ can be determined from $A_r$ and $\phi_r$ at each $T$ if $\alpha$ and $X_w$ are known. Since $\alpha$ and $X_w$ are purely geometric, being determined only by sample size and, hence, are temperature independent, it suffices to determine them at any convenient temperature.

For samples which attain a high conductivity at low temperatures $A_r \rightarrow 0$ and $\phi_r$ tends to a limiting value, $\phi_w$, then $X_w = \cot(\phi_w / 2)$. This method was used for TaSe₃, and it is the preferred method when a material has a highly conducting limit.

The quasi 1D materials typically¹ have a high-temperature phase which is metallic, for which $\sigma_r = 0$ up to optical frequencies. Thus, $X_w$ can be determined from the measured $A_r$ and $\phi_r$ at room temperature. This method was used for TaS₅, for which $\sigma_r$ is always low and the previous method is inapplicable. For TaSe₃ this method resulted in the same value of $X_w$ as the technique in the previous paragraph.

The quantity $\alpha$ enters multiplicatively and if one is interested only in the relative $T$ dependence, e.g. $\sigma_r(T)/\sigma_r(300 \text{ K})$, it need not be determined. In order to determine absolute magnitudes $\alpha$ can be evaluated from $\alpha = (\alpha / 2\lambda_a) (\lambda / \pi d)^2 \omega \epsilon_0$, if $d$ is known. The diameter was measured by means of an optical microscope. Note also that when the conductivity is low, so that $Z_r > Z_w$, and the sample diameter $d$ can be precisely determined, it is sufficient to use Eq. (10) along with Eq. (3) and the defining equation for $\alpha$, to determine $\sigma_r$ and $\sigma_i$.

Thin needles of approximate cross section $1 \times 1 \mu m$ were selected. The measurements were performed as discussed in Sec. IV. The sample did not extend across the entire height of the waveguide. The geometric impedance, $X_w$, and the corresponding shift, $\phi_w$, were negative (capacitive). The phase shift and attenuation due to the sample, $\phi_r$ and $A_r$, respectively, are shown in Fig. 6(a). The attenuation rises smoothly to a peak at 45 K then decreases sharply at low temperatures. The phase shift at high temperatures is almost constant and zero to within the scatter. At around 100 K the phase shift starts to decrease slowly then sharply below 70 K. At the lowest temperatures $\phi_r$ is beginning to saturate. The saturation may not be clear from the figure, but a plot of $\text{Im}(Z)$ vs $\text{Re}(Z)$ at all temperatures can be extrapolated to $\text{Re}(Z) = 0$ leading to $\text{Im}(Z) = X_w = -1.09$ and $\phi_w = -85$°.

The calculated resistivity, $\rho$, is displayed in Fig. 6(b). The solid line is a four-probe dc resistivity on a sample from the same preparation batch. The dc resistivity changes by a factor of 50 and the 109-GHz resistivity follows it to within a scatter of ± 10%. At the lowest temperatures the classical skin depth, $\delta$, is less than $1 \mu m$ and, therefore, $\delta = d$, but the agreement indicates that Eq. (10) still applies.

The imaginary part of the conductivity can also be calculated from Eq. (10). At all temperatures it is zero to within the scatter of the measurement. The scatter is $± 500$ $(\Omega \text{ cm})^{-1}$ at room temperature and increases to $± 20 \times 10^4$ $(\Omega \text{ cm})^{-1}$ at low temperatures.

These results act as a confirmation of the technique for lossy samples.

VI. RESULTS: COMPLEX CONDUCTIVITY OF A CHARGE-DENSITY WAVE COMPOUND

The main focus of the present work has been the study of the dynamics of electrons in linear chain quasi 1D materials at high frequencies. Typically these materials show a metallic behavior at high temperatures [at room temperature $\sigma \sim 10^2 (\Omega \text{ cm})^{-1}$]. The metallic conductivity is independent of frequency up to optical frequencies. At low temperatures, they commonly undergo a transition to a charge-density
wave (CDW) state, characterized by a strong frequency dependent complex conductivity.

Orthorhombic TaS$_3$ displays a metal—CDW transition at $T_c = 225$ K. From a room-temperature value of 2600 (Ω cm)$^{-1}$, the dc conductivity decreases rapidly below 225 K. The raw data at 60 GHz for $\sigma$, and $\phi$, vs $T$ are shown in Fig. 7(a). Unlike the data for TaSe$_3$, $\phi$, remains small at all $T$ and never reaches a large limiting value. This is because $\sigma$, is low at all temperatures. $X_{\infty}$, which is inductive, was determined by assuming $\sigma$, = 0 at 300 K, since TaS$_3$ is a metal at room temperature. From the room-temperature measurements, $X_{\infty} = 2.8$, $X_{\infty}$ (300 K) = 10.07. $d$ was evaluated from optical measurements, which yielded $d \sim 1$ μm. From $d$, $\alpha = 2.4 \times 10^6$ (Ω cm)$^{-1}$, $\sigma$, (300 K) = $\alpha/R$, (300 K) = 2357 (Ω cm)$^{-1}$. Within the measurement accuracy, $\sigma$, (60 GHz) = $\sigma$, at 300 K, so that there is no frequency dependence at this temperature, as appropriate for this temperature. The normalized conductivities $\sigma$, ($T$)/$\sigma$, (300 K) and $\sigma$, ($T$)/$\sigma$, (300 K) evaluated from the data of Fig. 7(a) are shown in Fig. 7(b). Below $T_c$, $\sigma$, is orders of magnitude larger than the dc conductivity [not shown], indicating a strong frequency dependence in the CDW state. From nearly zero in the metallic phase above $T_c$, $\sigma$, is negative in the CDW state. At low temperatures, $\sigma$, changes sign and becomes positive. The insets in Fig. 7(b) show the sample impedance in different temperature regions as evaluated from the raw data of Fig. 7(a).

These measurements, when carried out at different frequencies, yield the full frequency dependence of $\sigma$, and $\sigma$, in the range 26–110 GHz. The bridge measurements were supplemented by cavity perturbation methods at 9 GHz. The results for the CDW contribution $\sigma_{CDW}$ [Re $\sigma_{CDW}(\omega) = \sigma, (\omega) - \sigma, (\omega = 0)$, Im $\sigma_{CDW}(\omega) = \sigma, (\omega)$] vs $f = \omega/2\pi$ at a representative temperature of 160 K are shown in Fig. 8. The data are normalized to $\sigma_{max} = \text{Re} \sigma_{CDW}$ (9 GHz) measured by means of a cavity perturbation method. The data agree very well with a Drude-like behavior, $\sigma_{CDW} = (ne^2/\tau/m^*) (1 + i\omega\tau)^{-1}$, obtained from considering the damped inertial response of the CDW condensate, and shown in Fig. 8 as the solid line. Here $\tau$ is the damping parameter, $m^*$ the effective mass of the CDW, and $n$ the number of electrons of charge $e$. These measurements have enabled a complete characterization of the fundamental CDW parameters $\tau$ and $m^*$ for the first time.

VII. DISCUSSION

We have described a technique for measuring complex conductivity using complex impedance bridge at millimeter wave frequencies between 26 and 110 GHz. The technique has proved to be quite sensitive and has hitherto yielded important information regarding the dynamics of electrons in quasi 1D materials. The experimental configuration is well suited for needle-like specimens but is quite versatile and can be easily extended to a variety of samples types and physical conditions, such as lower or higher temperatures, magnetic fields, applications of dc bias, etc.

The millimeter wave frequency range seems to be optimal for this measurement technique. The bridge size scales with wavelength and can get uncomfortably large at lower frequencies. At higher frequencies, the waveguide sizes become very small, requiring greater mechanical precision. In the submillimeter wave range, quasi-optical techniques will probably be much more appropriate.

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5. The bridge components were purchased from Hughes Aircraft Co., Torrance, CA 90509.
10. Manufactured by Hughes Aircraft Co., Torrance, CA 90509.
11. Manufactured by EIP Microwaves, Inc., 2731 N. 1st Street, San Jose, CA 95134.