

Microwave-induced transitions between weak localization and weak antilocalization in graphene

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Weak localization (WL) and weak antilocalization (WAL) of electrons in a disordered conductor refer to the decrease or increase of the conductivity caused by the quantum interference of the electronic motion. In graphene, the nontrivial Berry phase and the chiral nature of the charge carriers imply that both phenomena can be observed, as it has been firmly established in direct current (DC) magnetotransport experiments. However, studies of alternating current (AC) magnetoconductivity at frequencies comparable to, and much higher than, the dephasing rate (τ_ϕ^{-1}) remain unexplored. Using a combination of transport and spectroscopic measurements at low ($\omega/2\pi < 20$ GHz) and high ($\omega/2\pi = 90\text{--}350$ GHz) frequencies compared to the dephasing rate, we demonstrate a frequency dependence in the optical magnetoconductivity of graphene in the limit $\omega\tau_\phi \gg 1$. We prove that spectroscopic measurements at low frequencies are well reproduced by the WL theory in the static limit ($\omega = 0$), with no appreciable changes in the magnetoconductivity at different frequencies as long as the condition $\omega\tau_\phi < 1$ holds. At higher frequencies, the spectra exhibit transitions between WL and WAL induced solely by the external electromagnetic field, as they occur at constant temperature and carrier density. By analyzing the results with a WL model extended to finite frequencies, we observe that the usual decoherence mechanisms of inelastic scattering, in addition to the elastic processes of intervalley and long-range scattering, do not explain the observed behavior. Our results point to the need for a revised model for the decoherence dynamics in the limit $\omega\tau_\phi \gg 1$.

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I. INTRODUCTION

Coherent electronic transport is related to physical phenomena arising from the quantum interference of electrons in a conductor. As electron scattering with impurities is elastic, it does not break phase coherence [1,2], enabling quantum interference even after multiple scattering events. Decoherence, or dephasing, is reached through inelastic electron-electron or electron-phonon scattering, which are diminished as the temperature is reduced [2,3]. At sufficiently low temperatures, the characteristic time of dephasing, τ_ϕ , is much larger than the mean time between electron collisions with impurities, and quantum effects manifest in macroscopic physical observables. Besides the interest from a fundamental perspective, coherent transport has been proposed as a mechanism for detecting microwaves in the sub-THz and THz ranges [4–6],

where semiconductor detectors are ineffective, necessitating the investigation of alternative schemes. Hence, a deep understanding of the decoherence dynamics of electrons under microwave irradiation is necessary for the success of such technologies.

Weak localization (WL) arises in disordered one- and two-dimensional systems as a consequence of the constructive interference of electrons diffusing in opposite directions along a closed trajectory [7]. The constructive interference enhances the probability of backscattering and causes a decrease in the conductivity compared to the prediction of the classical Drude model [7–9]. In graphene, WL is distinct from that of conventional conductors because of the unique properties of its charge carriers [10,11]. For instance, carriers that complete a closed path acquire a Berry phase of π [12], leading to destructive interference of self-crossing trajectories and implying a weak antilocalization (WAL) behavior [10,11,13], similar as in conductors with strong spin-orbit coupling [14]. However, the chiral nature of the carriers and their additional valley degree of freedom introduces intervalley scattering originating from atomically sharp defects, which allows constructive interference of carriers at different valleys and tends to restore WL [10,11,13]. Generally, graphene samples fabricated by different methods and typical carrier densities exhibit WL behavior [15–20]; however, transitions between

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WL and WAL can be achieved by varying the carrier density and temperature [17].

Processes that break the time-reversal invariance affect WL, because they restrict the interference of electrons moving in opposite directions along closed trajectories. Thus an external magnetic field destroys the phase coherence and restores the classical Drude magnetoconductivity. WL and WAL manifest in DC transport as positive [7] and negative magnetoconductivity [14], respectively. They can also be observed spectroscopically by measuring the microwave absorption as a function of the magnetic field (B), as demonstrated in graphene [21,22], GaN/Al_xGa_{1-x}N heterostructures, and topological insulators [23] with microwaves of frequency $\omega/2\pi = 9.5$ GHz, ω being the angular frequency of the electromagnetic field. In these experiments, the microwave absorption is proportional to the real part of the longitudinal AC conductivity, $\text{Re}(\sigma_{xx}(\omega))$ [24]. This fact has been used in the contactless detection of transport properties of graphene, for example, in the observation of Shubnikov-de Haas oscillations with microwaves of sub-THz frequencies [25]. Considering that the typical dephasing times in graphene are of the order of $\tau_\phi \sim 1\text{--}10$ ps at temperatures in the range of $T \sim 100\text{--}1$ K [17–20], a microwave frequency of $\omega/2\pi = 9.5$ GHz ($\omega \approx 60$ ns⁻¹) is lower than the corresponding dephasing rates of $\tau_\phi^{-1} \sim 1000\text{--}100$ ns⁻¹. Indeed, it was found that WL probed by microwave absorption at low frequencies is well explained by the WL theory in the static limit ($\omega = 0$) [21]. Nevertheless, a time-dependent electromagnetic field breaks the time-reversal invariance, constituting an additional source of decoherence [26,27]. This dynamic decoherence was demonstrated in DC transport studies of WL under microwave irradiation in thin wires, and thin metallic and semiconducting films [28–33], evidencing that the effect is maximal when $\omega\tau_\phi \sim 1$ [30], in accordance with the theory [26]. In graphene, however, the dynamic decoherence was absent at frequencies up to 170 GHz, showing instead that the response under microwave irradiation is bolometric, that is, caused by microwave heating [34]. On the other hand, AC conductivity studies in thin copper films [35], AlGaAs/GaAs heterojunctions [36,37], and thin silver wires [38] have demonstrated a frequency-dependent WL/WAL in the limit $\omega\tau_\phi \sim 1$, without deviations from the existing theory [7,14]. Since the dynamic decoherence [26,27] is a nonlinear effect that depends quadratically on the amplitude of the alternating electric field E_ω , it does not manifest in the mentioned AC studies, which probe the linear response in E_ω [36].

Herein, we investigated the WL/WAL in graphene in the limit $\omega\tau_\phi \gg 1$, since the higher frequencies may result in an additional dynamic decoherence mechanism. We tested the current theory of WL/WAL by determining the characteristic rates and lengths of dephasing and elastic scattering (intervalley and long range), comparing them to those found in DC and AC conductivity at low frequencies as a gauge of the effect of dynamic decoherence. To this end, we used a combination of DC transport measurements, low-frequency microwave spectroscopy ($\omega/2\pi = 9.6\text{--}18$ GHz), and sub-THz spectroscopy ($\omega/2\pi = 90\text{--}350$ GHz) to characterize the WL in graphene samples prepared by chemical vapor deposition (CVD) and epitaxial growth on SiC. We demonstrate that WL probed by low-frequency spectroscopy gives comparable results to

transport measurements and is well-described by the WL theory in the static limit, justifying our spectroscopic approach at higher frequencies. For sub-THz spectroscopy, we employed an advanced method to continuously probe the optical conductivity across a wide frequency range, demonstrating that it exhibits a frequency dependence not explained by performing a simple extension of the WL theory to the dynamic case, where the microwave frequency enters via the replacement $\tau_\phi^{-1} \rightarrow \tau_\phi^{-1} - i\omega$ [14,36]. The spectra are characterized by numerous and continuous transitions between WL and WAL behavior induced exclusively by microwaves since they appear at fixed temperatures and carrier densities. Moreover, the spectral features are remarkably robust to temperature changes, indicating that their origin is not related to the usual decoherence processes of inelastic scattering, or to the elastic processes of intervalley and long-range scattering. These results point to the need for a new understanding of the decoherence dynamics in the limit $\omega\tau_\phi \gg 1$, where phenomena such as microwave-induced dephasing and a frequency-dependent diffusion coefficient may be relevant.

II. THEORY OF WEAK LOCALIZATION AND WEAK ANTILOCALIZATION IN GRAPHENE AT FINITE FREQUENCIES

The correction to graphene's conductivity caused by the quantum interference of electron trajectories considers the relaxation rates of dephasing τ_ϕ^{-1} , intervalley scattering τ_i^{-1} , and the combined effects of intravalley scattering with trigonal warping of the graphene bands, accounted by the relaxation rate τ_*^{-1} [10,11,13]. The latter effect is caused by scattering processes with an interaction range longer than the lattice spacing [13], such that τ_*^{-1} may be called the long-range scattering rate, in contrast to intervalley scattering processes where the interaction range is shorter than the lattice spacing [13]. The theory of WL/WAL in graphene [10,11] is extended to finite frequencies via the replacement $\tau_\phi^{-1} \rightarrow \tau_\phi^{-1} - i\omega$, in analogy to the model of Hikami, Nagaoka, and Larkin [14], and as demonstrated by Vitkalov [36]. The resulting AC longitudinal magnetoconductivity, $\Delta\sigma_{xx}(\omega, B) = \sigma_{xx}(\omega, B) - \sigma_{xx}(\omega, B = 0)$, is given by

$$\Delta\sigma_{xx}(\omega, B) = N \left[F\left(\frac{\tau_B^{-1}}{\tau_\phi^{-1} - i\omega}\right) - F\left(\frac{\tau_B^{-1}}{\tau_\phi^{-1} + 2\tau_i^{-1} - i\omega}\right) - 2F\left(\frac{\tau_B^{-1}}{\tau_\phi^{-1} + \tau_i^{-1} + \tau_*^{-1} - i\omega}\right) \right], \quad (1)$$

$$= N \left[F\left(\frac{B}{B_\phi - iB_\omega}\right) - F\left(\frac{B}{B_\phi + 2B_i - iB_\omega}\right) - 2F\left(\frac{B}{B_\phi + B_i + B_* - iB_\omega}\right) \right], \quad (2)$$

with $N = e^2/\pi h$, $\tau_B^{-1} = 4eDB/\hbar$, D the diffusion coefficient, $B_\omega = \hbar\omega/4eD$, $B_{\phi,i,*} = \hbar\tau_{\phi,i,*}^{-1}/4eD = \hbar/4eL_{\phi,i,*}^2$ the characteristic magnetic fields associated with the dephasing, intervalley, and long-range scattering processes,

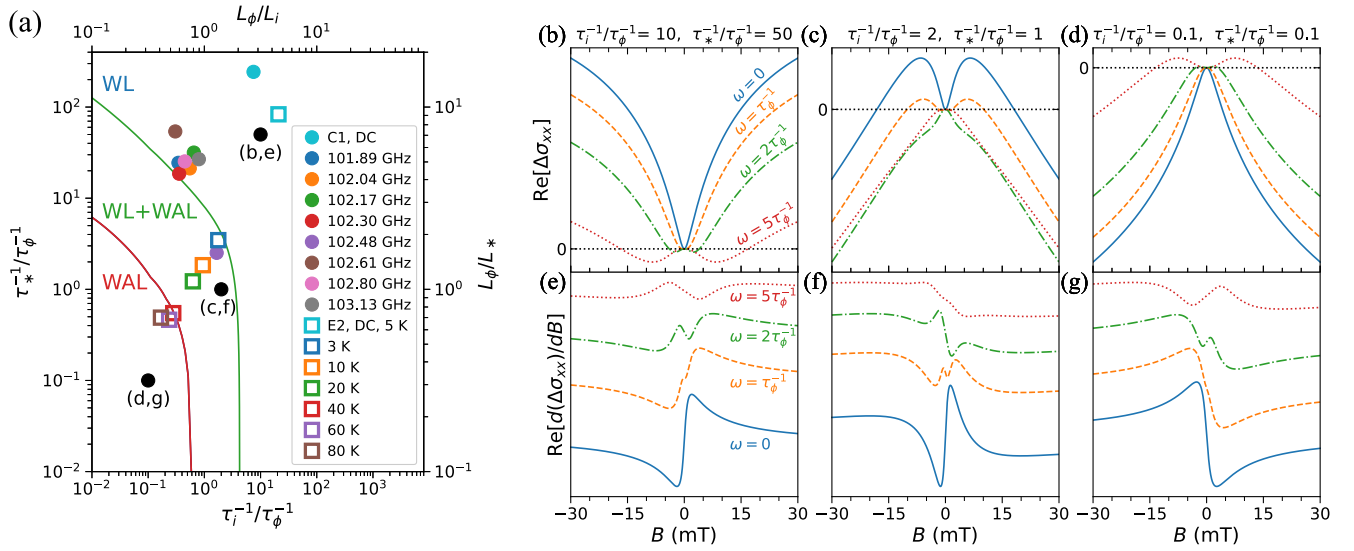


FIG. 1. Behavior of the magnetoconductivity at different localization regimes and its frequency dependence. (a) Diagram of the localization regimes [according to the static magnetoconductivity, $\Delta\sigma_{xx}(\omega = 0)$] as a function of the ratios $\tau_*^{-1}/\tau_\phi^{-1}$ and $\tau_i^{-1}/\tau_\phi^{-1}$ (L_ϕ/L_* and L_ϕ/L_i). Continuous lines separate different localization regimes. The solid circles indicate the parameters used for the plots in (b)–(g), as well as the fitted results for sample C1 in DC transport and in the frequency range 101–103 GHz, at $T = 5$ K, as displayed in Fig. 3(g). The hollow squares are fitted parameters for sample E2 in DC transport at $T = 5$ K, and at $\omega/2\pi = 320$ GHz at different temperatures, as shown in Fig. 4(f). [(b)–(g)] Behavior of the magnetoconductivity in Eq. (1) at different localization regimes and its frequency dependence. The top row [(b)–(d)] indicates the real part of the magnetoconductivity, $\text{Re}(\Delta\sigma_{xx})$, while the bottom row [(e)–(g)] corresponds to its magnetic-field derivative, displayed with a vertical offset for each ω . [(b) and (e)] Typical WL in the limit $\tau_\phi^{-1} \ll \tau_i^{-1} \ll \tau_*^{-1}$ ($L_\phi \gg L_i \gg L_*$). [(c) and (f)] Combination of WL and WAL for $\tau_\phi^{-1} = \tau_*^{-1} < \tau_i^{-1}$ ($L_\phi = L_* > L_i$). [(d) and (g)] WAL in the limit $\tau_*^{-1}, \tau_i^{-1} \ll \tau_\phi^{-1}$ ($L_*, L_i \gg L_\phi$). In all plots, it was considered that $D = 250 \text{ cm}^2/\text{s}$, $\tau_\phi^{-1} = 100 \text{ ns}^{-1}$, and $L_\phi = 500 \text{ nm}$.

$L_{\phi,i,*} = \sqrt{D\tau_{\phi,i,*}}$ are the characteristic scattering lengths, and

$$F(z) = \ln(z) + \psi\left(\frac{1}{2} + \frac{1}{z}\right), \quad (3)$$

with ψ the digamma function. The AC field introduces an additional characteristic length, $L_\omega = \sqrt{D/\omega}$, which acquires a value of $L_\omega = 200 \text{ nm}$ for an angular frequency $\omega = 100 \text{ ns}^{-1}$ and a typical diffusion coefficient of $D = 250 \text{ cm}^2 \text{ s}^{-1}$.

Tikhonenko et al. identified the conditions to find either WL or WAL in the DC conductivity [17], classifying the different localization regimes according to the ratios $\tau_i^{-1}/\tau_\phi^{-1} = L_\phi^2/L_i^2$ and $\tau_*^{-1}/\tau_\phi^{-1} = L_\phi^2/L_*^2$, as shown in Fig. 1(a). The real part of magnetoconductivity in Eq. (1) in different regimes and its frequency dependence is displayed in Figs. 1(b)–1(d), while Figs. 1(e)–1(g) displays its magnetic field derivative, corresponding to the observable quantity in field-modulated spectroscopic experiments. The typical WL behavior is shown in Figs. 1(b) and 1(e). WL is found when the first term within the square brackets of Eq. (1) is dominant, for which a sufficient condition is $\tau_\phi^{-1} \ll \tau_i^{-1}, \tau_*^{-1}$ ($L_\phi \gg L_i, L_*$). Since τ_ϕ^{-1} decreases as the temperature is decreased, while τ_i^{-1} and τ_*^{-1} have a weak temperature dependence, WL is expected at low temperatures and typical carrier densities ($\sim 10^{12} \text{ cm}^{-2}$). Focusing on the frequency dependence of Fig. 1(b), the magnetoconductivity decreases as the frequency increases, becoming negative with two local minima in the vicinity of $B = 0$. These local minima manifest as new peaks in $\text{Re}[d(\Delta\sigma_{xx})/dB]$ [Fig. 1(e)], as it is clearly seen at $\omega = 2\tau_\phi^{-1}$.

These peaks broaden and shift as the frequency increases; nevertheless, no new peaks emerge at higher frequencies.

An intermediate scenario where the magnetoconductivity transitions from positive to negative is achieved when the rates τ_ϕ^{-1} , τ_i^{-1} , and τ_*^{-1} are comparable with each other, indicating that the system is in a combination of WL and WAL, as depicted in Figs. 1(c) and 1(f). To define the ratios $\tau_*^{-1}/\tau_\phi^{-1}$ and $\tau_i^{-1}/\tau_\phi^{-1}$ that characterize such behavior, we found the values that produce a negative magnetoconductivity above a given magnetic field, chosen arbitrarily as 40 mT; the parameters that fulfill this condition for a coherence rate of $\tau_\phi^{-1} = 100 \text{ ns}^{-1}$ are displayed in Fig. 1(a). At $\omega = 0$, the magnetoconductivity has two local maxima in the vicinity of $B = 0$ [Fig. 1(c)]. In this static limit, $d[\Delta\sigma_{xx}]/dB$ shows two local maxima as well [Fig. 1(f)], but as the frequency increases, a new emerging peak implies that there are three such maxima for $\omega > \tau_\phi^{-1}$.

Finally, WAL is found at low ratios $\tau_*^{-1}/\tau_\phi^{-1}$ and $\tau_i^{-1}/\tau_\phi^{-1}$, following the condition depicted in Fig. 1(a) [17]. WAL can be achieved by increasing τ_ϕ^{-1} through the temperature increase, provided that the sample already has a short intervalley scattering rate (large L_i), characteristic of samples with a low density of crystal defects. τ_i^{-1} can be decreased further by reducing the carrier density [16,17]. Figures 1(d) and 1(g) display a hypothetical WAL scenario in the limit $\tau_*^{-1}, \tau_i^{-1} \ll \tau_\phi^{-1}$ ($L_*, L_i \gg L_\phi$). Due to the negative magnetoconductivity at $\omega = 0$, the behavior is opposite to the WL case of Figs. 1(b) and 1(e), although sharing similar features like the emergence of a new peak at $\omega \gtrsim \tau_\phi^{-1}$, and the broadening and shift of the peaks at higher frequencies.

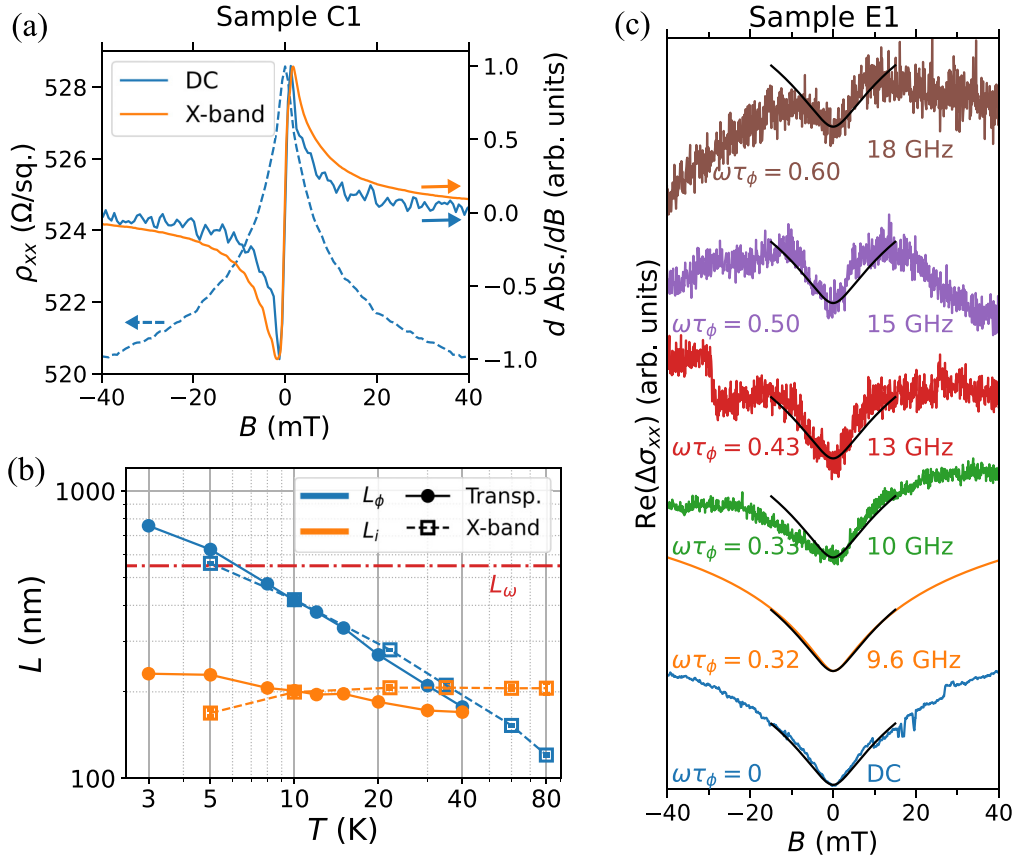


FIG. 2. Weak localization in DC transport, X-band spectroscopy, and AC conductivity at low frequencies. (a) Magnetoresistivity and X-band spectrum ($\omega/2\pi = 9.6$ GHz) of CVD graphene on quartz at $T = 5$ K. The DC magnetoresistivity (ρ_{xx}) is shown in a dashed line, and its normalized magnetic field derivative multiplied by -1 , proportional to $d\sigma_{xx}/dB$, is shown in a continuous line. (b) Fitted characteristic lengths of CVD on quartz samples found from DC transport (filled circles, continuous lines) and X-band spectroscopy (open squares, dashed lines) as a function of the temperature. The lines are guides to the eye. $L_\omega = \sqrt{D/\omega} = 550$ nm for sample C1 and at the X-band frequency is indicated. (c) DC conductivity, integrated X-band spectrum, and AC conductivity ($\omega/2\pi \geq 10$ GHz) of epitaxial graphene samples at $T = 5$ K. The data were normalized and vertically offset for clarity. The superimposed black lines are fittings to Eq. (2) with $\omega = 0$ of the DC conductivity.

III. RESULTS

A. Weak localization in DC and AC transport at low frequencies

We investigated the WL in DC and AC transport at frequencies lower than, and comparable to, the dephasing rate. We studied two epitaxial samples grown on (0 0 0 1)6H-SiC with an approximate area of 4×4 mm² and thickness 0.5 mm, and a commercial monolayer CVD graphene sample on a quartz substrate (Graphenea S.A.), with an area of 5×5 mm² and a thickness of 0.5 mm. Details of the sample preparation and characterization by DC and AC transport are found in “Methods.” We focus our analysis on the CVD graphene on quartz sample, denoted C1, and on an epitaxial sample, denoted E1. The remaining samples showed similar results, which are found in Ref. [39], Fig. S3 (see also Refs. [40–50] therein).

For sample C1, Hall measurements at $T = 5$ K revealed hole doping with a carrier density of $n = (14.45 \pm 0.03) \times 10^{12}$ cm⁻², a Hall mobility of $\mu = (822 \pm 2)$ cm² V⁻¹ s⁻¹, a mean-free-time of $\tau = (36.49 \pm 0.12)$ fs, and a diffusion coefficient of $D = v_F^2 \tau / 2 = (182.4 \pm 0.6)$ cm² s⁻¹, with $v_F = 10^6$ m/s the Fermi velocity of graphene. For sample E1 at

$T = 2.5$ K, we found $n = (8.76 \pm 0.02) \times 10^{12}$ cm⁻² (hole doping), $\mu = (1366 \pm 9)$ cm² V⁻¹ s⁻¹, $\tau = (47.2 \pm 0.4)$ fs, and $D = (236 \pm 2)$ cm² s⁻¹.

The negative magnetoresistance (positive magnetoconductance) typical of WL was observed in four-point measurements in sample C1 [Fig. 2(a)], and two-point measurements in sample E1 [Fig. 2(c)]. Fittings to Eq. (2) in the static limit ($\omega = 0$) for both samples corroborated a coherence length decreasing with temperature, while L_i and L_* remained temperature-independent, in agreement with previous studies of WL in graphene [18,19,51]. For sample C1, we found a coherence length of $L_\phi = 750$ nm at $T = 3$ K [Fig. 2(b)], which is larger but of the same order of magnitude as the reported values of $L_\phi \sim 500$ nm for CVD graphene at $T = 1.5$ K [19]. It decreases to $L_\phi = 176$ nm at $T = 40$ K, while the intervalley and long-range scattering lengths were approximately constant, taking the values of $L_i = (200 \pm 20)$ nm and $L_* = (40 \pm 3)$ nm, averaged over temperatures up to 40 K (Fig. 2(b), the results for L_* are displayed in Ref. [39], Fig. S3 (g)). For sample E1, the coherence length varied from $L_\phi = 367$ nm at $T = 5$ K, to $L_\phi = 200$ nm at $T = 20$ K. The intervalley and long-range scattering lengths

were $L_i = (141 \pm 22)$ nm and $L_* = (48 \pm 10)$ nm, averaged over the temperature range $T = 2.5$ – 20 K (Ref. [39], Fig. S3 (h)). These results are in agreement with previous reports on epitaxial graphene [18,19,51]. At $T = 5$ K, the coherence rates are $\tau_\phi^{-1} = D/L_\phi^2 = 46$ ns $^{-1}$ and $\tau_\phi^{-1} = 175$ ns $^{-1}$, for C1 and E1, respectively, establishing a frequency limit of $\omega/2\pi > 7.6$ GHz and $\omega/2\pi > 28$ GHz necessary to fulfill the condition $\omega\tau_\phi > 1$ in each sample. Therefore the condition is achieved for sample C1 at the frequency $\omega/2\pi = 9.6$ GHz, which is used in X-band spectroscopy.

AC magnetoconductivity studies at frequencies up to 18 GHz were conducted using two methods. The first was X-band magnetospectroscopy ($\omega/2\pi = 9.6$ GHz), performed using a Bruker EMX plus spectrometer, routinely employed for electron spin resonance (ESR) spectroscopy. Here, the magnetic-field derivative of the microwave absorption (proportional to $d\sigma_{xx}/dB$) was recorded employing the magnetic-field modulation technique. The second method employed a coplanar microwave resonator based on a superconducting YBCO transmission line [52], probing the frequency range $\omega/2\pi = 10$ – 18 GHz. Further experimental details can be found in “Methods.” We studied a CVD graphene on quartz sample similar to C1, and we assume that both have the same transport properties. Figure 2(a) displays the normalized X-band spectra of the CVD sample at $T = 5$ K, exhibiting a sharp signal with its maximum located at $B = 1.7$ mT. At $\omega/2\pi = 9.6$ GHz, the length scale introduced by the frequency is $L_\omega = \sqrt{D/\omega} = 550$ nm, being lower than $L_\phi = 625$ nm for sample C1 at $T = 5$ K, and indicating that at this temperature the microwave frequency is comparable to the dephasing rate, with $\omega\tau_\phi = L_\phi^2/L_\omega^2 = 1.3$. In view of this, we compare the X-band spectrum with the field-derivative of the DC magnetoresistivity multiplied by -1 , which is proportional to $d\sigma_{xx}/dB$, shown in Fig. 2(a). Even considering that $\omega\tau_\phi$ is of order one, a good agreement is found in both quantities, with the X-band spectrum being broader than the derivative of the DC magnetoconductivity, consistent with the prediction of the frequency-dependent WL model displayed in Fig. 1(e). Nevertheless, the theory predicts the emergence of a peak in the vicinity of $B = 0$ for $\omega\tau_\phi \sim 1$, not observed in the X-band spectrum. Such peak emergence, however, is weak for $\omega\tau_\phi \sim 1$, which could explain the discrepancy. Fitting the X-band spectra at different temperatures to Eq. (2) with $\omega = 0$ results in practically the same values of L_ϕ and L_* , and similar ones for L_i , as those found in DC transport, as shown in Fig. 2(b) (the fitted DC magnetoresistance and X-band spectra at different temperatures are found in Ref. [39], Fig. S3). The small deviation of L_ϕ at $T = 5$ K can be explained by the limit $\omega\tau_\phi > 1$ ($L_\phi > L_\omega$) being reached, as displayed in Fig. 2(b). Hence, we infer that spectroscopic measurements in the limit $\omega\tau_\phi < 1$ are well described by the WL theory in the static limit, in agreement with previous reports in graphene and semiconductor samples [21,23].

Studies at various frequencies were performed on sample E1, as displayed in Fig. 2(c), which compares the DC and AC magnetoconductivity at $T = 5$ K. Considering that the dephasing rate found from DC transport is $\tau_\phi^{-1} = 175$ ns $^{-1}$, the condition $\omega\tau_\phi \leq 0.6$ holds for all the frequencies, with $\omega\tau_\phi$ indicated for each spectrum in Fig. 2(c). The figure shows the integrated X-band spectrum, proportional to $\text{Re}(\Delta\sigma_{xx})$.

Since the absolute magnetoconductivity is not measured in AC transport experiments using a superconducting resonator, a direct comparison with the DC magnetoconductivity is not possible. Instead, the data were normalized with respect to the peak between $|B| < 30$ mT. A good agreement was found between the DC and X-band conductivity, with fittings to Eq. (2) in the static limit resulting in $L_\phi = 367$ nm and $L_\phi = 350$ nm for DC transport and X-band spectroscopy at $T = 5$ K, respectively. (Fitted results for sample E1 are reported in Ref. [39], Fig. S3.) For measurements with a superconducting resonator, corresponding to frequencies $\omega/2\pi \geq 10$ GHz, the positive AC magnetoconductivity is superimposed on a background signal that varies with frequency, characteristic of the resonator (Ref. [39], Fig. S4). This fact, combined with the relatively weak WL signal, permits only a qualitative comparison with the DC and X-band results. We observe that the width of the signal around $B = 0$ presents slight variations compared to the DC conductivity, whose fitted result is overlapped on each spectrum. Additionally, as the frequency increases, the AC magnetoconductivity appears to transition from positive to negative at a certain magnetic field, similarly to the WL + WAL scenario of Fig. 1(c). This would imply a change in the scattering lengths L_i and L_* induced by the frequency not observed in X-band spectroscopy; nevertheless, the nontrivial signal changes in the resonator, displayed in Ref. [39] (Fig. S4), do not permit us to arrive at a clear conclusion. Since all frequencies follow $\omega\tau_\phi < 1$, we do not observe the emergence of two local minima in the vicinity of $B = 0$, as the theory predicts for $\omega\tau_\phi \gtrsim 1$ [Figs. 1(b) and 1(c), with $\omega > 0$], indicating that the behavior of the conductivity is still described by the WL theory in the static limit.

B. Weak localization at sub-THz frequencies

The conductivity at the sub-THz frequencies $\omega/2\pi = 90$ – 350 GHz (denoted as optical conductivity to distinguish it from the AC conductivity at lower frequencies) was investigated on a home-made high-frequency rapid scan electron spin resonance spectrometer (THz-FRASCAN-ESR) [53,54]. As in X-band spectroscopy, the use of magnetic field modulation implied that the magnetic field derivative of the microwave absorption was measured, proportional to $\text{Re}[d\sigma_{xx}(\omega, B)/dB]$. We employed two ways of recording the absorption spectra: spectral maps and continuous-wave (CW) measurements. In spectral maps, absorption is probed as a function of both microwave frequency and magnetic field, simultaneously. In contrast, CW measurements record the magnetic field dependence at a fixed frequency. Further experimental details in “Methods.” Due to the higher uniformity of CVD graphene compared to epitaxial graphene, we focus our analysis on a sample of CVD graphene on quartz from the same provider as C1, assuming that both samples have the same transport properties. The optical conductivity of the epitaxial samples exhibited a similar behavior; the results are reported in Ref. [39], Figs. S6 and S7. The measurements were done at $T = 5$ K, unless stated otherwise.

Figures 3(a) and 3(b) display the spectral maps in the range 90–150 and 180–240 GHz, respectively, while Fig. 3(c) shows CW measurements at selected frequencies in the range 195–211 GHz. Considering a dephasing rate of

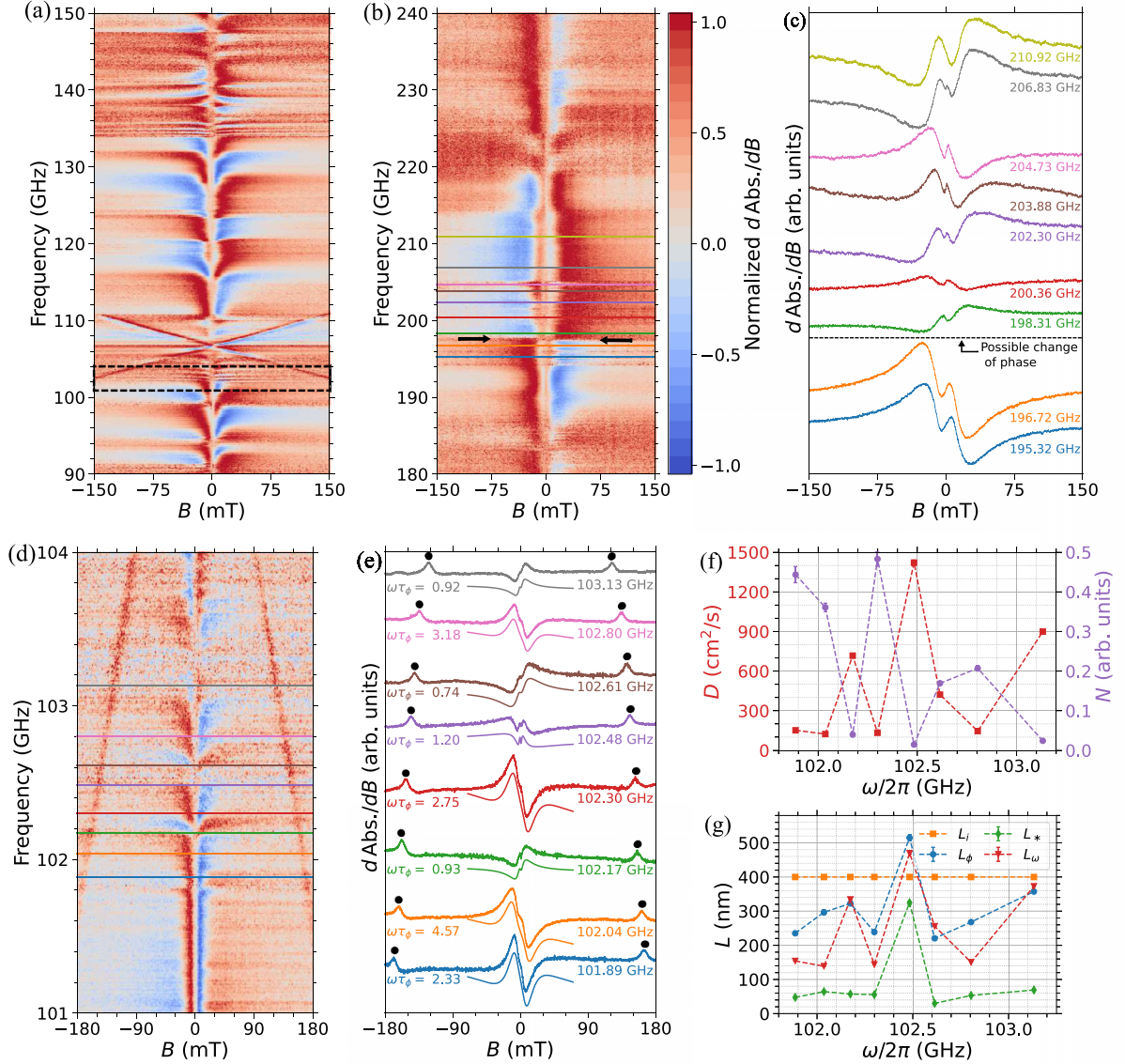


FIG. 3. Sub-THz spectroscopy measurements on CVD graphene on quartz. (a) Spectral map in the frequency range 90–150 GHz. In all maps, the signal at each frequency was normalized with respect to the highest peak. The color scale is the same as in (b). The box in dashed lines is the region plotted in (d). (b) Spectral map in the range 180–240 GHz. The horizontal colored lines correspond to the frequencies of the CW spectra in (c). The feature indicated by the black arrows is a phase change in the spectra, as discussed in the text. (c) CW spectra at selected frequencies in the range 195–210 GHz. Unnormalized data. (d) Spectral map in the range 101–104 GHz. The horizontal colored lines correspond to the frequencies of the CW spectra in (e). (e) CW spectra at selected frequencies in the range 101–103 GHz exhibiting rapid signal changes. Unnormalized data. Fittings according to Eq. (2) are shown below each spectrum. The peaks indicated by black circles are the ESR resonance of the zero-field energy splitting of molecular oxygen adsorbed on graphene. (f) Fitted values of the diffusion coefficient and the normalization factor according to Eq. (2). (g) Fitted results of the characteristic lengths.

$\tau_\phi^{-1} = 46 \text{ ns}^{-1}$ found for sample C1 at $T = 5 \text{ K}$, it follows that $\omega\tau_\phi = 12\text{--}33$ for the probed frequency range, meaning that the system is in the limit $\omega \gg \tau_\phi^{-1}$, where, in accordance with Eq. (1), the WL correction is significantly reduced (or modified). In such a wide frequency range, the optical conductivity exhibits peaks consistent with WL/WAL behavior, which continuously emerge, broaden, and shift as the frequency and magnetic field increase.

Observing the CW spectra of Fig. 3(c), it is evident that their behavior is fundamentally different from that of low-frequency spectroscopy [e.g., the X-band spectra of Fig. 2(a)];

rather, they resemble frequency-dependent spectra in different WL/WAL regimes shown in Figs. 1(e)–1(g). For instance, at 198.31 and 210.98 GHz, the behavior is consistent to WL with frequency $\omega = 2\tau_\phi^{-1}$ [Fig. 1(e)], while at 195.32, 196.72, and 200.36 GHz, it is consistent with WAL with frequency $\omega = 2\tau_\phi^{-1}$ [Fig. 1(g)]. Furthermore, at the frequencies 202.30, 203.88, and 206.83 GHz, the optical conductivity exhibits three local maxima, similar to those in the intermediate WL + WAL scenario of Fig. 1(f). In those spectra, a small peak is located at very low magnetic fields, of just 1 mT for $\omega/2\pi = 206.83 \text{ GHz}$. Remarkably, such a feature

is resolved in the spectral map of Fig. 3(b). We have verified that changes of one order of magnitude in the microwave field power affect the intensity but not the shape of the spectra (Ref. [39], Fig. S5), demonstrating that the optical conductivity is in the linear response regime. The frequency interval characterizing the changes in the optical conductivity is not uniform, varying from tenths of GHz, as seen in the range 101–104 GHz [Figs. 3(d) and 3(e), discussed in subsequent paragraphs], to a few GHz, as evident in Fig. 3(b) for the range 180–240 GHz. This has been corroborated in the epitaxial samples as well (Ref. [39], Figs. S6 and S7). Given the strong frequency variations of the signal observed in the CW spectra, we conclude that the spectral maps are necessary to obtain a clear picture of the WL/WAL behavior in the limit $\omega\tau_\phi \gg 1$.

A disadvantage of the heterodyne detection and quasi-optical bridge employed in these spectroscopic measurements is the introduction of changes in the signal shape at unpredictable frequencies, known as phase changes. The phase changes are measurement artifacts, and look like a sudden inversion of the signal shape, for example, at 197.5 GHz in Fig. 3(b), as indicated by the black arrows. These changes make the identification of WL or WAL according to their shape difficult since the spectra are inverse with each other, e.g., as in Figs. 1(e) and 1(h). Nevertheless, the phase changes do not cause the appearance of new peaks in the spectra, such as those observed in Fig. 3(c). To demonstrate that the changes in the optical conductivity are a real effect, we take as a reference the signal evolving linearly with the frequency and magnetic field observed in Fig. 3(a) in the interval 102–111 GHz. It crosses $B = 0$ at 107.5 GHz (3.586 cm^{-1}), corresponding to the electron spin resonance (ESR) of the zero-field energy splitting of molecular oxygen with spin $S = 1$ [55], indicating the presence of such paramagnetic impurities adsorbed on graphene. Figures 3(d) and 3(e) display the spectral map and CW measurements in the range 101–104 GHz. Pronounced changes in the signal around $B = 0$ are seen as the frequency increases by just 0.13 GHz, as seen at the frequencies 102.04–102.17 and 102.48–102.61 GHz of the CW spectra in Fig. 3(e). The oxygen peaks, indicated by filled black circles in the CW spectra, have a constant shape and amplitude in the probed range, indicating no measurement-induced artifacts. Although phase changes may occur at different frequencies, we infer that the observed frequency dependence of the signal is a fundamental property of the optical conductivity of graphene.

Attempting to fit the optical conductivity of Fig. 3(e) to Eq. (2) using constant values for N , $L_{\phi,i,*}$, and D was not possible; therefore, they were set as fitting parameters. The large parameter space makes the quantitative analysis difficult because of the high correlation between D and L_ϕ , as well as between L_i and L_* , meaning that they have similar effects on the optical magnetoconductivity of Eq. (2). To reduce the parameter space, we considered that L_i is frequency-independent, as it originates from atomically sharp crystal defects that are not affected by the external electromagnetic field. Successful fittings at fields $|B| < 40 \text{ mT}$ were obtained by setting $L_i = 400 \text{ nm}$, which, although higher than $L_i = (200 \pm 20) \text{ nm}$ found in DC transport, was necessary to capture the changes in the magnetoconductivity. The fitted spectra are displayed in Fig. 3(e), with the fitted parameters

of the normalization factor and diffusion coefficient shown in Fig. 3(f), and the fitted characteristic lengths in Fig. 3(g).

It is observed that to reproduce the spectra, the diffusion coefficient must experience strong, essentially discontinuous changes in a short frequency range. For instance, it increases from $130 \text{ cm}^2 \text{ s}^{-1}$ at 102.30 GHz, to $1420 \text{ cm}^2 \text{ s}^{-1}$ at 102.48 GHz, which is an order of magnitude higher than $D = 182 \text{ cm}^2 \text{ s}^{-1}$ obtained by DC transport. The characteristic scattering lengths also exhibit a frequency dependence, albeit weaker than the diffusion coefficient. From them, it is possible to determine the localization regime in each frequency by computing the ratios $\tau_*^{-1}/\tau_\phi^{-1} = L_\phi^2/L_*^2$ and $\tau_i^{-1}/\tau_\phi^{-1} = L_\phi^2/L_i^2$. The results, displayed in Fig. 1(a), indicate that the conductivity in the majority of frequencies is near the limit between WL and WL + WAL regimes, except at $\omega/2\pi = 102.48 \text{ GHz}$, which is in WL + WAL. The spectrum at 102.48 GHz is particular because it lies at the transition between two spectra with opposite shape, which, in order to be captured by the model, requires order-of-magnitude changes in L_* : from 55 nm at 102.30 GHz, to 325 nm at 102.48 GHz, and then to 30 nm at 102.61 GHz. These large, discontinuous changes in D and L_* are not caused by the rather arbitrary choice of a constant $L_i = 400 \text{ nm}$, because, as mentioned, a much lower value does not fit the spectra, while higher ones would not be justified in view of the results of DC transport and X-band spectroscopy.

The characteristic length $L_\omega = \sqrt{D/\omega}$, displayed in Fig. 3(g) alongside the scattering lengths, permits a comparison with L_ϕ and to establish the value of $\omega\tau_\phi = L_\phi^2/L_\omega^2$, indicated for the spectra at each frequency in Fig. 3(e). Note that L_ϕ and $L_\omega = \sqrt{D/\omega}$ are both fitted parameters, with $\omega\tau_\phi \sim 14$ in frequency range 101–104 GHz if we consider D and τ_ϕ^{-1} found by DC transport. The spectra can be classified by those with $\omega\tau_\phi \sim 1$ ($\omega/2\pi = 102.48 \text{ GHz}$, and others similar to 102.17 GHz) and those with $\omega\tau_\phi > 2$ (similar to 101.89 GHz). The spectra in the latter category have a similar shape to the theoretical WAL examples in Fig. 1(g); however, according to the fits, they fall within the WL category. A careful observation of the predicted magnetoconductivity in the WAL regime explains this result. Fig. 1(g) shows that at $\omega = 0$, the peaks located at $B \sim \pm 2 \text{ mT}$ decrease slowly as $|B|$ increases, in contrast to the rapid decrease of the experimental spectra with $\omega\tau_\phi > 2$. Such a rapid decrease of $\text{Re}[d(\Delta\sigma_{xx})/dB]$ cannot be captured by Eq. (1) because it requires a transition from negative to positive magnetoconductivity as $|B|$ increases, which cannot happen because of the dominant prefactor of 2 in the third term of Eq. (1). In other words, a behavior inverse to that of the WL + WAL regime in Figs. 1(c) and 1(f), necessary for a sharp decrease of $\text{Re}[d(\Delta\sigma_{xx})/dB]$ in the WAL limit, is not predicted by the theory. As a consequence, large variations in $\omega\tau_\phi$ are necessary to adjust the spectra, resulting in the discontinuous changes in the diffusion coefficient D and the characteristic lengths L_ϕ and L_* .

The magnetic field dependence of the microwave signal displays changes in the shape with the frequency, revealing features that appear and disappear in relatively short frequency intervals. These changes are consistently observed in all samples, irrespective of the substrate or fabrication method (Ref. [39], Figs. S6 and S7). A direct comparison with Eq. (1)

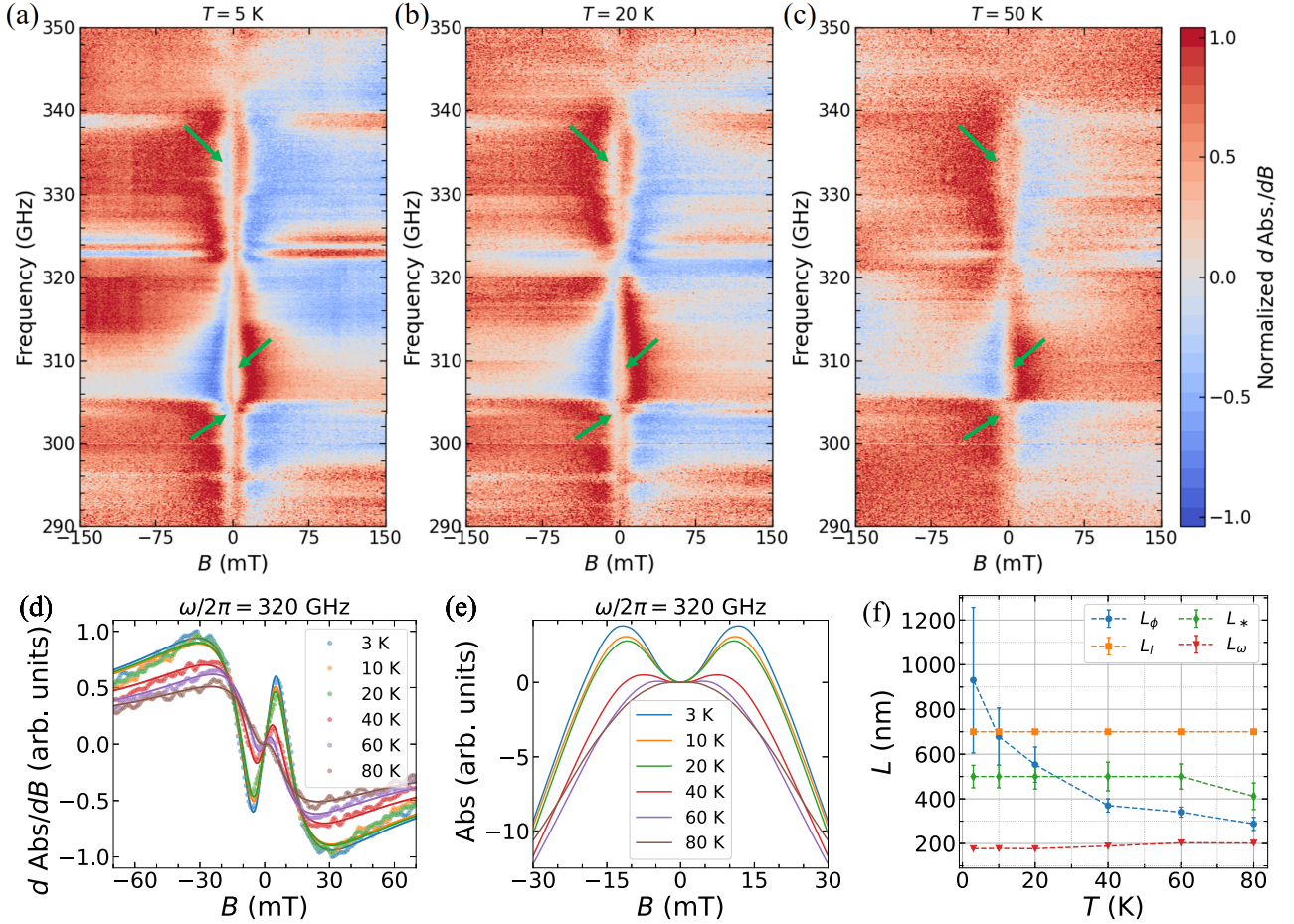


FIG. 4. Temperature dependence of the optical conductivity of sample E2. [(a)–(c)] Spectral maps at frequencies $\omega/2\pi = 290$ – 350 GHz and temperatures $T = 5, 20$, and 50 K, as indicated on top of each panel. The green arrows indicate features mentioned in the text. (d) Temperature dependence of the CW spectra at $\omega/2\pi = 320$ GHz. Continuous lines are fittings to Eq. (2). (e) Integrated CW spectra. (f) Fitted results of the characteristic lengths. The dashed lines are guides to the eye.

leads to drastic variations of the fitting parameters, which are found to be far from what is expected from DC measurements. An explanation of the observed behavior requires further investigation. In particular, the observed signal could be a mix between the real and imaginary parts of Eq. (1). This mix can be quite sophisticated since in Eq. (1) there are three complex functions (contributions) with different phases.

C. Temperature dependence

In graphene, the weak electron-phonon coupling implies that the main source of decoherence at low temperatures is inelastic electron-electron scattering, resulting in a decrease of the coherence length (increase of the dephasing rate) as the temperature increases [16,18], which indeed was observed in DC transport and X-band spectroscopy [Fig. 2(b)]. We analyze the temperature dependence of the optical conductivity of the epitaxial sample E2; the results for sample E1 are found in Ref. [39], Fig. S8. Two-point DC transport characterization of E2 by Shubnikov-de Haas (SdH) oscillations revealed a mobility of $\mu_{\text{SdH}} = (820 \pm 70) \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, a diffusion coefficient of $D = (140 \pm 1) \text{ cm}^2 \text{ s}^{-1}$, and a decoherence rate of $\tau_\phi^{-1} = 100 \text{ ns}^{-1}$ at $T = 5$ K (characterization details in Ref. [39], Fig. S2).

Figures 4(a)–4(c) show the spectral maps of sample E2 at the temperatures $T = 5, 20$, and 50 K, and in the frequency range $\omega/2\pi = 290$ – 350 GHz, corresponding to $\omega\tau_\phi = 18$ – 22 at $T = 5$ K. The signal consistent with WL/WAL was clearly present at temperatures as high as 50 K, highlighting the robust coherence properties of graphene. The most notable observation is that the frequencies at which new peaks emerge, indicated in the figures by the green arrows, do not depend on the temperature. In particular, as the temperature increases, the spectral maps retain their shape, although the peak intensity decreases. To illustrate this, we examined the temperature dependence of the CW spectrum at the fixed frequency of $\omega/2\pi = 320$ GHz, displayed in Fig. 4(d), as well as its numerical integration representing the microwave absorption, shown in Fig. 4(e). The signal shape is consistent with the theoretical WAL behavior of Figs. 1(d) and 1(g), with the central peak in $d\text{Abs.}/dB$, as well as the local maxima in the microwave absorption, clearly vanishing at high temperatures. These results are precisely what is being depicted in the WAL prediction of Figs. 1(d) and 1(g): the peak around $B = 0$ vanishes as τ_ϕ^{-1} increases, i.e., as $\omega\tau_\phi$ decreases.

We analyzed the spectra by fitting them to Eq. (2) in the field range $|B| < 70$ mT, considering N, L_ϕ, L_* , and D as free

parameters, and a constant $L_i = 700$ nm ($L_i = (140 \pm 13)$ nm found by DC transport, Ref. [39], Fig. S3 (i)). The fitted spectra are shown in Fig. 4(d), and the obtained characteristic lengths are displayed in Fig. 4(f). The errors in the parameters were taken as one standard deviation. As with the previous fittings, the high correlation between L_* and L_i implies that the uncertainties in these quantities are larger than those shown in the figure. The spectra are well described by almost temperature-independent L_i , L_* , and L_ω , with L_ϕ decreasing with temperature similarly to the DC and X-band results of Fig. 2(b). Except for the lowest temperature of $T = 3$ K, the dephasing length is lower than L_i in the probed temperature range, and becomes lower than L_* around $T \sim 25$ K. The determination of the localization regimes by computing the ratios L_ϕ/L_i and L_ϕ/L_* confirms that the system is in WAL for $T \geq 40$ K, and in WL + WAL for $T \leq 20$ K, as shown in Fig. 1(a).

The fitted diffusion coefficient showed an increase with temperature, from $630 \text{ cm}^2 \text{ s}^{-1}$ at $T = 3$ K, to $820 \text{ cm}^2 \text{ s}^{-1}$ at $T = 80$ K (not shown in Fig. 4), considerably higher than that $D = (140 \pm 1) \text{ cm}^2 \text{ s}^{-1}$ obtained in DC transport. This implied a slight increase in L_ω , from 180 to 200 nm for $T = 3$ –80 K [Fig. 4(f)], higher than $L_\omega = 83$ nm predicted by DC transport. From Fig. 4(f), it is observed that L_ϕ approaches L_ω at high temperatures, confirming that the central peak in the spectra vanishes when $L_\phi \sim L_\omega$ ($\omega\tau_\phi \sim 1$).

The temperature dependence of the quantum corrections at the high frequency of 320 GHz demonstrates the behavior expected from Eq. (1) with fitting parameters that are of the same order as those obtained in DC measurements. However, the model does not explain the observed sharp features [marked by green arrows in Figs. 4(a)–4(c)], which require discontinuous changes in the fitting parameters at different frequencies, in a similar manner as the results for the CVD sample displayed in Figs. 3(e)–3(g). Additional efforts are necessary to understand such behavior.

IV. DISCUSSION AND CONCLUSIONS

We presented a study of the quantum corrections to the optical magnetoconductivity of graphene caused by WL/WAL, in the limits $\omega\tau_\phi \lesssim 1$ and $\omega\tau_\phi \gg 1$. In the former limit, we demonstrated that the WL theory of Eq. (1) in the static limit ($\omega = 0$) reproduces the spectroscopic measurements with fitting parameters comparable to DC transport measurements, in agreement with previous reports in graphene [21,22].

In the limit $\omega\tau_\phi \gg 1$, we found that the optical magnetoconductivity exhibits numerous and continuous transitions between WL and WAL. Such behavior is consistently observed in CVD and epitaxial samples, proving that it originates from the electronic properties of graphene. Fitting the spectra using the WL model of Eq. (1) results in high values of L_i and L_* compared to those obtained by DC transport, as well as in discontinuous changes in the fitting parameters at different frequencies. This forces us to conclude that the frequency-dependent WL/WAL model of Eq. (1) has inconsistencies in the regime $\omega\tau_\phi \gg 1$. The model captures several qualitative features of the spectra, such as the emergence of peaks in $d\text{Abs.}/dB$ in the vicinity of $B = 0$ as the frequency is increased, and the decrease of the coherence length as the

temperature increases, with the remaining scattering parameters being approximately temperature-independent, in agreement with DC transport and X-band spectroscopy. However, the significantly high values of L_i and L_* needed to adjust the spectra, which are not justified by the DC transport results, indicate that the elastic processes of intervalley and long-range scattering are not responsible for the observed variations, and suggest the existence of additional unidentified mechanisms affecting electron coherence. It is assumed that the decoherence process related to spin-orbit coupling is not relevant since the substrates (quartz, SiC) are composed of light elements, in which the spin-orbit coupling is weak. On the other hand, the presence of paramagnetic oxygen impurities with spin $S = 1$ suggests that electron scattering through spin centers may be an additional decoherence channel; however, their presence does not modify qualitatively the decoherence dynamics described by the WL/WAL model in the static limit, as observed by the successful fittings of the DC resistivity. On the other hand, the dynamic decoherence mechanism described in [26,27] cannot be the source of those features because of its nonlinear origin, which is not compatible with the linear response regime probed in our experiments. Furthermore, the dynamic decoherence effect is maximal at $\omega\tau_\phi \sim 1$ and decreases significantly when $\omega\tau_\phi \gg 1$ [26,27], in contradiction with our experiments.

The experiments give a few clues about the origin of the mechanism affecting electron coherence. First, it must manifest in the linear response regime, implying that it is independent of the magnitude of the microwave electric field. This is supported by the invariance of the signal shape over changes of an order of magnitude in the microwave power (Ref. [39], Fig. S5). Furthermore, the frequency dependence of the spectra points to the dynamic origin of the process, requiring further investigation to be confirmed. Another possibility is that the signal could be a combination of the real and imaginary parts of the complex magnetoconductivity of Eq. (1), which can be quite elaborate due to the mix of three terms with different complex phases. Finally, we find that changes in the diffusion coefficient compared to the DC transport results are necessary to adjust the spectra at higher frequencies. This fact, despite originating from an inadequate model applied in the analysis, suggests that WL/WAL studies at sub-THz frequencies are an attractive route for investigating the frequency dependence of the diffusion coefficient, as long as an accurate theoretical model for the decoherence dynamics is found.

V. METHODS

A. CVD graphene

Commercial monolayer graphene samples grown by chemical vapor deposition and transferred onto a quartz substrate were supplied by Graphenea (San Sebastian, Spain). The size of the samples as purchased was $10 \times 10 \text{ mm}^2$ with a thickness of 0.5 mm, and they were cut to $3 \times 3 \text{ mm}^2$ for spectroscopic measurements using a diamond pen. For transport measurements, the sample was laser-cut to the size $5 \times 5 \text{ mm}^2$, after a resist polymer (PMMA) was spin-coated onto the graphene surface as a protective layer, and then removed

using acetone and then rinsed with isopropyl alcohol (IPA). The samples were characterized by Raman spectroscopy using a WITec confocal Raman imaging system alpha300 R, with excitation wavelength of 533 nm and an incident power of 7 mW. We verified the ratio between the intensity (I) of the Raman peaks as $I(G)/I(2D) < 1$; $I(D)/I(G) < 0.1$.

B. Epitaxial graphene preparation

Nominally monolayer graphene was synthesized from $8 \times 8 \text{ mm}^2$ semi-insulating, (0 0 0 1)6H-SiC in an Aixtron/Epigress VP508 horizontal hot-wall reactor. The sample is etched in 5 standard liters per minute (SLM) of high-purity H_2 (using a laminar flow for this and all of the following) at 200 mbar during a temperature ramp to 1570 °C followed by graphene synthesis in 100 mbar high purity Ar at 1580 °C for 20 min. The reactor is cooled to 950 °C–1150 °C in Ar and H-intercalation of epitaxial graphene is carried out under a flow of 80 SLM of H_2 and a chamber pressure of 900 mbar for 15–75 min. Optimal temperature and time was determined to be 1050 °C for 60 min using Raman mapping and scanning electron microscope to confirm material uniformity and Hall measurements for sheet density and carrier mobility [56–58]. Reactor temperature calibration using Si melt tests yielded a temperature uniformity of about $\pm 2 \text{ K}$ over 100 mm at 1410 °C under a 90 SLM H_2 flow. For transport and spectroscopic measurements, the samples had an approximate area of $4 \times 4 \text{ mm}^2$, with a thickness of 0.5 mm. Further details on epitaxial graphene preparation and characterization are found in Ref. [59].

C. DC transport measurements

DC transport was investigated using two setups. The first is a cryogen-free magnet system (Cryogenic, London, UK), employed to study sample C1. The sample was glued to a chip expander using low-temperature varnish, and aluminum wires were contacted in the van der Pauw configuration using silver paint. The sample was located in a variable temperature insert operating in the temperature range 1.9–300 K, with a magnetic field up to 9 T applied by a solenoid cryomagnet. A Keithley 2400 was employed to source a DC current of 5 μA , and a Keithley 2182A nanovoltmeter was used to measure the voltage and resistance. The magnetic field sweep rate was 0.5 mT s^{-1} for measurements in the range of $|B| < 1 \text{ T}$, and 8.5 mT s^{-1} for fields above 1 T.

The second setup is a home-made high frequency rapid scan electron spin resonance spectrometer (THz-FRASCAN-ESR) [53,54], equipped with a sample holder appropriate for electrical measurements [54]. Samples E1 and E2 were studied with this setup. The samples were glued to a chip expander using low-temperature varnish (SCBltv02, Cryoandmore Budzylek GbR), and gold wires were contacted in the van der Pauw configuration using silver paint. The samples were located in a variable temperature insert operating in the temperature range 1.9–300 K, with a magnetic field up to 15 T applied by a solenoid cryomagnet (Cryogenic, London, UK). The magnetic field sweep rate was 1 mT s^{-1} for measurements in the range of $|B| < 1 \text{ T}$, and 3 mT s^{-1} for fields above 1 T. A Keithley 2450 was employed to source and measure voltage

and current. Figures S1(a)–S1(c) within Ref. [39] show the optical microscope images of samples C1, E1, and E2 as prepared for DC transport measurements.

D. X-band spectroscopy

Spectroscopic measurements at X-band frequencies were performed with a Bruker EMX plus spectrometer, routinely used for electron paramagnetic resonance (EPR) experiments, operating at 9.2–9.8 GHz. An Oxford Instrument ESR900 cryostat was used for temperature measurements in the range 5–70 K with stability $\pm 0.05 \text{ K}$. The sample was located in a resonant cavity, and the magnetic-field derivative of the microwave absorption (proportional to $d\sigma_{xx}/dB$) was recorded employing the magnetic-field modulation technique, with a modulation frequency and amplitude of 100 kHz and 6 G.

E. AC conductivity measurements

AC conductivity measurements in the frequency range $\omega/2\pi = 10\text{--}18 \text{ GHz}$ employed a coplanar microwave resonator [52] based on a superconducting YBCO transmission line. Epitaxial graphene samples were mounted on the resonator structure using vacuum grease and inserted into a variable temperature insert (VTI), enabling temperature-dependent measurements from 1.7 to 300 K and magnetic-field-dependent measurements up to 8 T. Microwaves tuned to various resonating frequencies were generated by a vector network analyzer (VNA), and were coupled to the coplanar resonator via coaxial cables. The microwave absorption by the sample was evaluated by measuring the transmitted on-resonance power using the VNA.

F. Sub-THz spectroscopy

The conductivity at the sub-THz frequencies $\omega/2\pi = 90\text{--}350 \text{ GHz}$ was investigated on a home-made high frequency rapid scan electron spin resonance spectrometer (THz-FRASCAN-ESR) [53,54]. The microwaves were generated by a synthesizer (8–14 GHz, Virginia Diodes, Charlottesville, USA) and subsequently tuned to the desired frequency by amplifier-multiplier chains (Virginia Diodes, Charlottesville, USA). They propagate in free space and are focused on the sample by a quasi-optical system (Thomas Keating, Billingshurst, UK) and then through a corrugated waveguide. Heterodyne detection with magnetic field modulation is employed, superseding the use of a resonant cavity and enabling the study of the microwave absorption in a wide frequency range. Magnetic-field modulation with a modulation frequency and amplitude of 3.01 kHz and 25 G was employed. We estimate that the average power is 1 mW across the frequency range of 90–350 GHz, although it varies for different frequencies. We employed two ways of recording the absorption spectra: spectral maps and continuous-wave (CW) measurements. In spectral maps, the absorption is probed as a function of the microwave frequency and magnetic field, simultaneously, while in CW measurements, its magnetic field dependence is recorded at a fixed frequency. In spectral maps, the microwave frequency was swept in intervals of 10 GHz with a total sweep time of 5 s between the initial and final frequency. For instance, individual maps were obtained

at 180–190 GHz, 190–200 GHz, and successive frequency ranges, which were subsequently post-processed to generate the map shown in Fig. 3(b). The magnetic field sweep rate was 0.18 mT s^{-1} in spectral maps and 0.32 mT s^{-1} in CW measurements. Since the power output of the microwave signal generators is not uniform across the whole frequency range, the intensity of the WL signal at different frequencies cannot be compared reliably; thus, it does not give significant physical information. For this reason, the spectral maps are normalized with respect to the highest peak at each frequency. In CW measurements, the data were not normalized.

The temperature-dependent measurements at $\omega/2\pi = 320 \text{ GHz}$ [Figs. 4(d) and 4(e)] were done in a home-built high frequency electron paramagnetic resonance (HFEP) spectrometer [60]. The linearly polarized microwave radiation is generated by a synthesizer (Virginia Diodes Ltd) multiplied in an amplifier-multiplier chain to frequencies of 80–1100 GHz. The microwave is propagated through free space and then focused on the sample through a corrugated waveguide. The microwave power is typically less than 1 mW. Fields up to 15 T and temperatures down to 2 K are accessible. Magnetic-field modulation was employed, with a field modulation amplitude and frequency of 20 G and 1.2 kHz.

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J.N.-G. performed the DC transport and sub-THz spectroscopy experiments with the support of V.T.S. and O.L., analyzed the measurements, and prepared the initial manuscript. O.L. and V.L. performed the X-band spectroscopy experiments. D.B. carried out the sub-THz experiments at 320 GHz [Figs. 4(d) and 4(e)]. A.B. and M.S. characterized the samples by AC conductivity at $\omega/2\pi = 10\text{--}18 \text{ GHz}$. D.K.G. fabricated the epitaxial graphene samples and conceptualized the experiments. P.N. supervised the experiments. All authors discussed the results and commented on the manuscript.

DATA AVAILABILITY

The data that support the findings of this article are openly available [61].

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