

Fermi surface and effective masses of IrO₂ probed by de Haas-van Alphen quantum oscillationsK. Götze^{1,2}, M. J. Pearce^{2,*}, S. Negi³, J.-R. Soh⁴, D. Prabhakaran⁵ and P. A. Goddard^{2,†}¹*Deutsches Elektronen-Synchrotron DESY, Notkestrasse 85, 22607 Hamburg, Germany*²*Department of Physics, University of Warwick, Coventry CV4 7AL, United Kingdom*³*Department of Physics, Faculty of Science, National University of Singapore, Science Drive 3, Singapore 117551, Singapore*⁴*Quantum Innovation Centre (Q.InC), Agency for Science Technology and Research (A*STAR),**2 Fusionopolis Way, Innovis #08-03, Singapore 138634, Singapore*⁵*Clarendon Laboratory, Department of Physics, University of Oxford, Parks Road, Oxford OX1 3PU, United Kingdom*

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Iridium-containing conducting materials are widely investigated for their strong spin-orbit coupling and potential topological properties. Recently the commonly used electrode material iridium dioxide was found to host a large spin-Hall conductivity and was shown to support Dirac nodal lines. Here we present quantum-oscillation experiments on high-quality IrO₂ single crystals using the de Haas-van Alphen effect measured using torque magnetometry with a piezoresistive microcantilever as well as density functional theory-based band-structure calculations. The angle, temperature, and field dependencies of the oscillations and the calculated band dispersion provide valuable information on the properties of the charge carriers, including the Fermi-surface geometry and electronic correlations. Comparison of experimental results to calculations allows us to assign the observed de Haas-van Alphen frequencies to the calculated Fermi surface topology. We find that the effective masses of IrO₂ are enhanced compared to the rest electron mass m_e , ranging from 1.9 to 3.0 m_e , whereas the scattering times indicate excellent sample quality. We discuss our results in context with recent ARPES and band-structure calculation results that found Dirac nodal lines in IrO₂ and compare the effective masses and other electronic properties to those of similar materials like the nodal chain metal ReO₂ in which Dirac electrons with very light effective masses have been observed.

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I. INTRODUCTION

5d transition-metal oxides are building blocks for a number of compounds whose properties are strongly influenced by spin-orbit coupling (see [1] and [2] for recent reviews). The dioxide IrO₂ in particular has received renewed attention after the discovery of a large spin Hall effect [3] and the subsequent prediction of Dirac nodal lines (DNL) [4]. ARPES measurements on IrO₂ [5–7] have confirmed the predictions about the band structure from [4] and revealed the importance of spin-orbit coupling and band topology for explaining the spin Hall effect. IrO₂ is commonly used for the production of electrodes, resistors, and capacitors in sensors [8–11] and is now discussed as a material for applications in spintronic devices [3]. It is also an important reference material for systems that comprise 5d transition-metal oxide octahedra like IrO₆ and that potentially host novel quantum phases [12,13].

Quantum oscillation results and band-structure calculations were first reported in the 1970s [14,15] but a complete analysis was not possible at the time as the magnetothermal oscillation measurements did not allow for the determination of effective masses and scattering rates.

In this paper we, therefore, present an update on the Fermi surface (FS) properties and electronic correlations of IrO₂ including the effective masses and Dingle temperatures. Our results are further compared with first-principle calculations of the band structure and are placed in context with previous calculations and with the FS and electronic properties of other topological Dirac materials.

Another aim of this work is to provide a reliable reference for experiments on the band structure of other materials that contain iridium oxide building blocks. Quantum oscillations are yet to be observed in, for example, pyrochlore iridates, and their unambiguous identification requires knowledge of the FS topology, corresponding quantum oscillation frequencies, and effective masses of closely related materials such as IrO₂ that might form in small quantities as a byproduct of the growth process.

II. METHODS

IrO₂ crystallizes in a tetragonal rutile structure shown in the lower right inset of Fig. 1 with lattice parameters $a = 4.4990 \text{ \AA}$ and $c = 3.1546 \text{ \AA}$ [17]. Very similar values are

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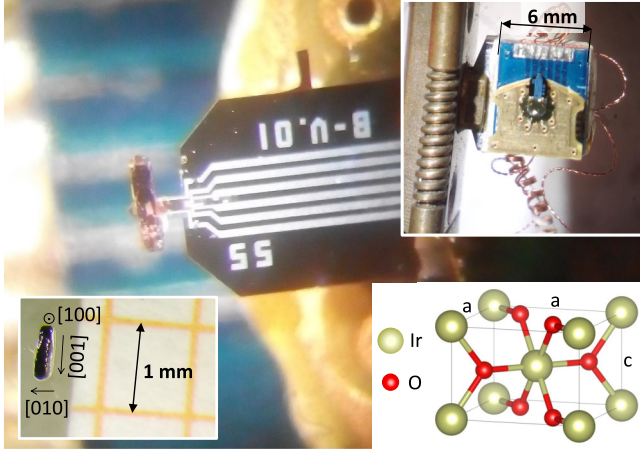


FIG. 1. Main figure: IrO₂ crystal on a microcantilever tip. Upper right inset: Microcantilever on PCB counterpart (blue) which, in turn, is glued to the rotator platform. Lower left inset: The same crystal from the main panel next to a piece of scale paper. Lower right inset: Rutile crystal structure of IrO₂. This representation of the structure was created using the program VESTA [16].

reported in [14,18,19]. IrO₂ single crystals were grown by chemical vapor transport as described in [20]. High purity iridium metal powder (99.9%) was kept in a tube furnace and the oxygen gas flow was set to 20 cc/min. The furnace was slowly heated up to 1050 °C and kept at this temperature for one week. Small crystals then formed near the cold end (950 °C) of the furnace.

We conducted magnetic torque measurements on IrO₂ single crystals in magnetic fields up to 15 T and at constant temperatures between 540 mK and 4.2 K. An IrO₂ crystal (lower left inset of Fig. 1) was attached to the tip of a SCL-Sensor.Tech. piezoresistive microcantilever using Apiezon N vacuum grease as shown in the main panel of Fig. 1. The crystal was oriented using natural growth faces with the crystallographic orientation indicated in the lower left inset of Fig. 1. The tip of the cantilever used in this study is 300 μm long, 100 μm wide and about 10 μm thick [21]. It is robust enough to allow for repeated mounting of a crystal with the help of a thin, grease covered hair and was found to carry crystals of a few 100 μm edge length without breaking. The cantilever chip is premounted on a PCB (upper inset of Fig. 1) customized to fit on our 7 mm × 7 mm rotator platform. The rotator mechanism allows for angle-dependent measurements with *in situ* change of angle. The piezoresistor that indicates the change of torque is incorporated into a Wheatstone bridge on the cantilever chip. Four-point resistance measurements were conducted with a standard lock-in amplifier, using an excitation current of ≈ 100 μA.

The Quantum Espresso package [22] was used for first principles calculations with a plane-wave basis set. A kinetic energy cutoff of 50 Ry was used along with fully relativistic ultrasoft pseudopotentials which accounted for the core electrons [23,24] together with the exchange-correlation functional of Perdew, Burke, and Ernzerhof [25]. The Brillouin zone (BZ) was sampled using a Γ -centered $6 \times 6 \times 6$ Monkhorst-Pack grid [26] for all calculations. The visualization of the FS was plotted with MATLAB and the lattice

parameters used for our calculations and for the drawing of the crystal structure in the inset of Fig. 1 are from [17].

III. RESULTS

We have observed de Haas-van Alphen (dHvA) quantum oscillations in IrO₂ which, at 0.54 K, start from about 6 T. At very high fields and very low temperatures, the oscillations acquire a sawtooth-like aspect indicative of magnetic and/or torque interaction [27,28]. (See also Appendix). In this paper we only consider those fields and temperatures at which purely sinusoidal oscillations are observed and where interaction effects can be excluded.

An example of the measured torque signal at 1.37 K at $\Theta_{001} = 30^\circ$ is shown in Fig. 2(a). Θ_{001} is the angle for rotation from $B \parallel [100]$ to $B \parallel [110]$ defined as per the inset in Fig. 3. The frequency spectrum of the oscillating part of the signal obtained by fast Fourier transformation (FFT) can be seen in Fig. 2(b) for different temperatures. We observe a large number of peaks, some of which are harmonics or combination frequencies. The seven most prominent peaks between 225 and 2980 T were identified to be fundamental frequencies coinciding with results in [14], and were, therefore, labeled with the same Greek letters as in [14]. In addition, we observed a low frequency of about 50 T with a very low FFT amplitude, marked with an asterisk * in Fig. 2(b).

The angular dependence of the dHvA frequencies for IrO₂ from this work is displayed as black squares in Fig. 3 together with the quantum oscillation frequencies found from magnetothermal measurements [14] as open red squares. Semitransparent lines serve as guides for the eye to identify the different branches. We have not included the harmonics or combination frequencies in this display, as they likely stem from interaction effects and not from electronic properties of the sample.

We find that there is a very good correspondence between our experimental results and those from [14]. We observed all known frequency branches, with the exception of the high frequency τ branch, indicating comparable sample quality in both studies. At some angles, we also observed the low frequency F^* , with FFT peaks below the α branch at about 30–50 T. This frequency does not vary with angle but due to the small number of points and the limited frequency resolution, it is not possible to draw firm conclusions about the shape of this FS.

The energy band dispersion of IrO₂ as a result of density functional theory (DFT)-based band-structure calculations is displayed in Fig. 4(a) and we find that our results agree well with those in [4,6] and [7] including the DNL between X and M. There are two bands crossing the Fermi energy leading to two FSs: a holelike FS consisting of a cross around the Z point of the BZ and an ellipsoid around the M point [red in Fig. 4(b)], and an electronlike FS which occupies the perimeter of the BZ with holes around M and R [blue in Fig. 4(c)]. Following the assignments in [14], we highlight the extremal orbits of the observed dHvA frequencies in the bottom figures of Figs. 4(b) and 4(c). Frequencies β , ϵ , and $\rho_{1,2}$ originate from the cross, whereas λ is associated with the ellipsoid around M. The remaining frequencies α , $\gamma_{1,2}$, and τ stem from the electron FS: α comes from a small neck of this

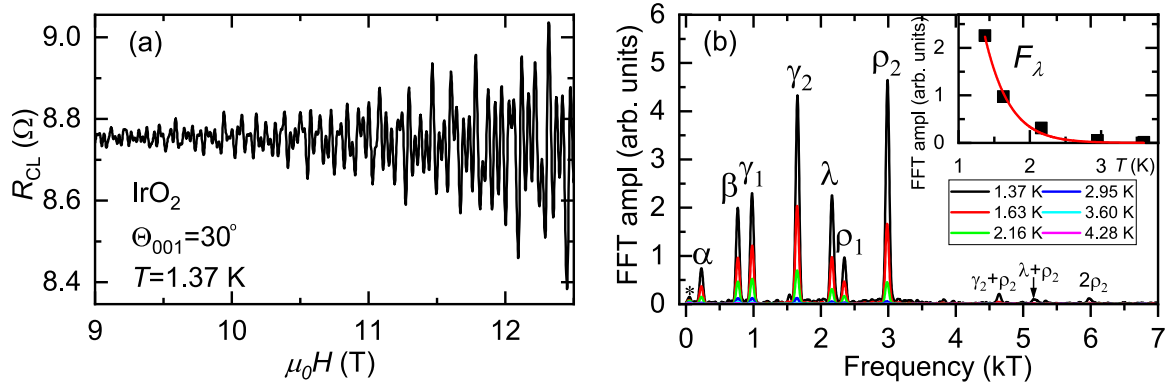


FIG. 2. (a) Field dependence of the microcantilever resistance for IrO₂ at 1.37 K. (b) Frequency spectrum obtained by fast Fourier transformation (FFT) of the oscillating part of the magnetic torque signal between 9 and 12.5 T at different temperatures between 1.37 and 4.28 K. Fundamental frequencies that were observed before are labeled with the same Greek letters as in [14]. We report frequency $F^* \approx 50$ T. Inset: temperature dependence of FFT amplitudes for $F_\lambda = 2.16$ kT and fit line determined by use of the Lifshitz-Kosevich formula yielding $m_\lambda^* = 2.3(5) m_e$, where m_e is the electron rest mass.

FS (between M and R). $\gamma_{1,2}$ and τ can be traced back to the FS portion around X and the dog-bone-shaped section whose orbit extends along k_z in the corners of the BZ, respectively, and show a stronger angular dependence [14].

A fit function based on the Lifshitz-Kosevich (LK) formula [27,29,30] with the temperature damping factor $R_T = X/\sinh(X)$ with $X = \alpha m^* T/\bar{B}$ and $\alpha = -14.69$ T/K is applied to the FFT amplitudes of a certain frequency for different temperatures T yielding the effective mass m^* as one of the fit parameters [27]. Here $\bar{B}^{-1}$ is the center of the inverse field window used in the FFT. The inset of Fig. 2(b) shows the FFT amplitudes and the curve of the corresponding LK fit function for $F_\lambda = 2.16$ kT with an effective mass of $2.3(5) m_e$. For the other fundamental frequencies we find effective masses between 1.9 and $3.0 m_e$. F^* has a mass of about $1 m_e$. The effective masses of the harmonics and combination frequencies in Fig. 2(b) could not be determined reliably due to the limited

field and temperature range. The effective masses of all main frequencies observed at $\Theta_{001} = 30^\circ$ are listed in Table I.

As the FFT amplitude of F^* is much lower than the amplitude of the other frequencies, its temperature dependence is shown in more detail in Fig. 5. The main panel shows the FFT amplitudes and the curve of the corresponding LK fit function as a function of temperature, whereas the inset shows the frequency spectrum around 50 T for different temperatures. We find that the amplitudes of F^* are bigger than the noise floor for all temperatures. In addition, we tried different methods for the background subtraction to verify that the peak in the frequency spectrum is not an artifact that arises from the subtraction of a higher order polynomial. It proved to be robust for different polynomials. The FFT amplitude decreases with increasing temperature and follows the LK prediction reasonably well, given that the amplitude of F^* is close to the noise level. We, therefore, conclude that F^* most likely originates from dHvA quantum oscillations and that there is another FS pocket that was previously not detected.

The LK formula can also be applied to the field dependence of the quantum-oscillation amplitudes measured by the torque technique via the so-called Dingle factor $R_D = \exp(\alpha m^* T_D/B)$ with $\alpha = -14.69$ T/K [31] which depends on the effective mass m^* and the Dingle temperature T_D . The

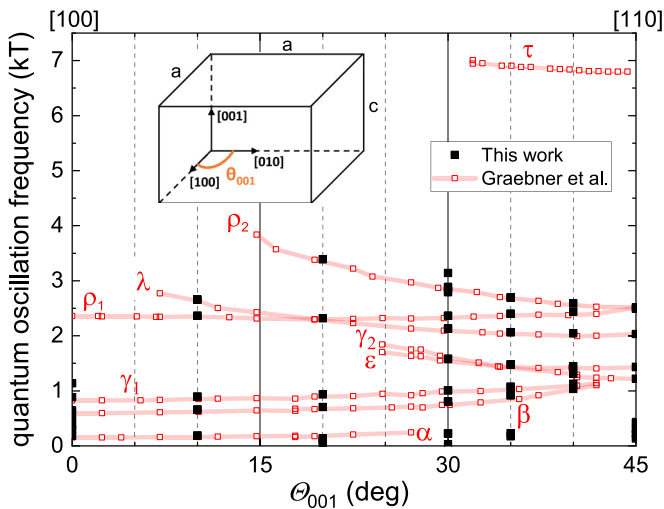


FIG. 3. Angular dependence of quantum oscillation frequencies of IrO₂ for rotation from $B \parallel [100]$ toward $B \parallel [110]$. Full black squares: this work; empty red squares: experimental results from [14]. Semitransparent lines are guides to the eye to better illustrate the different branches. The inset defines the rotation angle Θ_{001} .

TABLE I. Experimental values for fundamental frequencies F , effective masses $m^*(m_e)$, Dingle temperatures T_D , relaxation times τ , and quantum mobilities μ_q of dHvA oscillations observed in IrO₂ for $\Theta_{001} = 30^\circ$.

F (kT)		$m^*(m_e)$	T_D (K)	τ (ps)	μ_q ($\frac{\text{cm}^2}{\text{Vs}}$)
0.05	F^*	1.0(2)	4.5(5)	0.27(3)	500(100)
0.225	α	2.15(5)	0.95(3)	1.28(4)	1044(41)
0.77	β	2.1(3)	0.30(1)	4.0(1)	3390(500)
0.98	γ_1	1.9(1)	0.95(5)	1.35(7)	1250(100)
1.64	γ_2	2.1(2)	1.10(5)	1.10(5)	920(100)
2.16	λ	2.3(5)	1.0(1)	1.2(1)	930(220)
2.34	ρ_1	2.0(4)	1.3(1)	0.93(7)	820(175)
2.98	ρ_2	3.0(3)	0.7(1)	1.7(2)	1020(180)

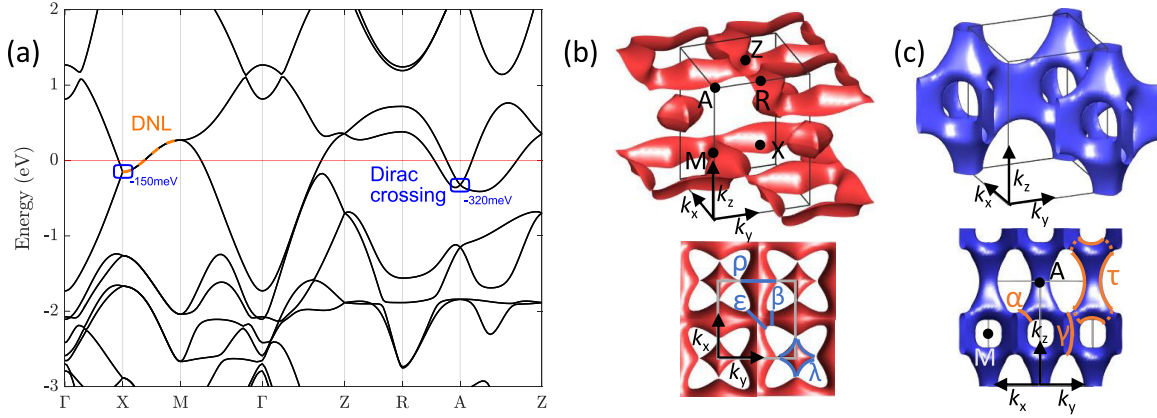


FIG. 4. (a) Band dispersion of IrO₂ with spin-orbit coupling. One of the DNLs (between X and M) is highlighted by an orange dashed line and the two Dirac crossings around X and A are marked with blue rectangles. (b) Top: The holelike Fermi surface and the Brillouin zone including high symmetry points. Bottom: View along k_z for a better visualization of the crosslike structure. (c) Top: The electronlike Fermi surface and the Brillouin zone. Bottom: View along the [110] direction which corresponds to $\Theta_{001} = 45^\circ$ in our experimental results. For both (b) and (c) bottom figures, the extremal orbits for the dHvA frequencies described in the text are drawn and labeled. In both top figures a quarter of the FS in the BZ has been omitted for clarity.

Dingle temperature is inversely proportional to the relaxation time τ by $T_D = \hbar/(2\pi k_B \tau)$ and is, through its relation to the relaxation time, a measure for sample quality. The Dingle plot in Fig. 6 displays the amplitude A of the dHvA oscillations of frequencies α , γ_2 , and ρ_2 at 1.63 K as $\ln(A/[R_T B^{3/2}])$ vs $1/B$. The Dingle temperature can then be determined from the slope of a linear fit to the data. We have limited the fits to a field range of 8.3 to 12.5 T, similar to the range for the effective mass fits. The dHvA amplitudes in the Dingle plots of α , γ_2 , and ρ_2 show the expected linear dependence and the low Dingle temperature values of 0.7 to 1.1 K indicate very high sample quality. The Dingle temperatures and relaxation times are included in Table I for all observed frequencies.

We note here that the Dingle plots in Fig. 6 for α and γ_2 display linear behavior up to the highest measured field of 15 T. The amplitudes of ρ_2 show a slight deviation from linear

behavior at high fields possibly due to the onset of interaction effects at high oscillation amplitudes.

We also calculated the quantum mobilities $\mu_q = q\tau/m^*$ from the electron charge q , relaxation time τ and the effective mass m^* . They are listed in the final column of Table I and range from 500 to 3390 cm²/(Vs). Those are typical values for nontopological electrons in line with the masses and Dingle temperatures observed in this study and they are comparable to values of 3000 cm²/(Vs) at 1.8 K obtained by transport measurements of epitaxial thin films of IrO₂ reported in [32]. Much higher values would only be expected for very light Dirac electrons (e.g., $\mu_q \approx 15000$ cm²/(Vs²) for two

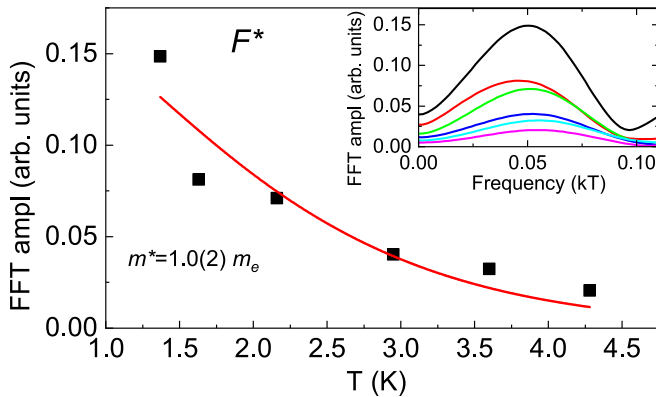


FIG. 5. Temperature dependence of FFT amplitudes for $F^* \approx 50$ T and fit line determined by use of the Lifshitz-Kosevich formula yielding $m^* = 1.0(2) m_e$, where m_e is the electron rest mass. Inset: FFT amplitude at 50 T at different temperatures between 1.37 and 4.28 K. The color coding is the same as in Fig. 2(b).

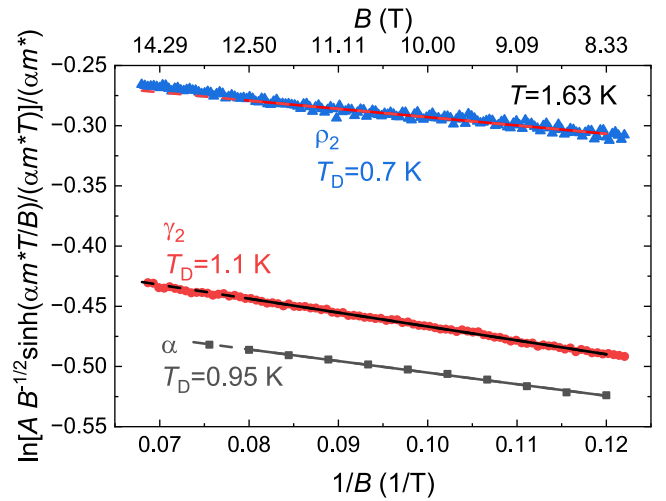


FIG. 6. Dingle plot of the field dependent FFT amplitudes at 1.63 K for frequencies α , γ_2 , and ρ_2 between 6.3 and 15 T. Fit functions were applied between 8.3 and 12.5 T (solid lines). Dashed lines are extrapolations to higher fields (lower $1/B$) to illustrate that the amplitudes of frequencies α and γ_2 continue to follow the linear behavior with the same slope whereas the slope for ρ_2 shows small deviations.

frequencies in the Dirac node arc semimetal PtSn₄ [33]). We will discuss in the following why such light electrons have not yet been observed in quantum oscillation studies of IrO₂ despite the prediction of DNLs and their observation in ARPES experiments.

IV. DISCUSSION

In our dHvA quantum oscillation experiments we have reproduced almost all of the frequencies and, therefore, the FS cross sections observed earlier in [14]. In addition, we measured the temperature dependence of the oscillation amplitudes at $\Theta_{001} = 30^\circ$ and derived the effective masses, Dingle temperatures, and quantum mobilities. Our experimental study is complemented by DFT band-structure calculations that allow us to place the experimentally observed dHvA frequencies in context with the calculated FS topology.

The effective masses are enhanced by a factor of 2–3 compared to the electron rest mass. Those values are comparable to the effective masses in elemental iridium where the highest experimentally observed mass is $2.99 m_e$ [34]. Ir has a Sommerfeld coefficient of $\gamma = 3.19 \text{ mJ}/(\text{mol K}^2)$ [35], whereas IrO₂ has a γ value of $5.51 \text{ mJ}/(\text{mol K}^2)$ [36]. This indicates that even higher masses could be expected in IrO₂. A candidate frequency for high masses is the τ frequency ($> 7 \text{ K}$) which we have not observed, possibly due to an unfavorable combination of enhanced mass and a curvature factor that causes an additional dampening of those high frequency oscillations. Also, the ratio between the FFT amplitudes for different frequencies can differ for different techniques [37] which could explain why the τ frequency was observed in the magnetothermal oscillations of Ref. [14] and not in our torque magnetometry data. While some higher masses could be expected from the specific heat coefficient, very low masses of the order of $0.1 m_e$ or lower are typically associated with Dirac electrons. DNLs have been predicted for the IrO₂ band structure and have also been experimentally observed in ARPES measurements. Such low masses have not been observed in our experiment, and in the following we will discuss the most likely reason why this was not the case and consider further experiments that might help to reveal or exclude the existence of Dirac electrons in IrO₂.

For comparison, we will review the case of ReO₂, a close relative of IrO₂ with a similar γ value of $3.0 \text{ mJ}/(\text{mol K}^2)$ [38], which was predicted to host Dirac nodal chains [39] and which has been studied using the dHvA and Shubnikov-de Haas (oscillations in electrical transport) effect [40].

ReO₂ was found to have four large FS sections with quantum oscillation frequencies of 100–3600 T. Their respective effective masses are of the order of 0.67 to $1.74 m_e$. So far, those properties are comparable to our findings for IrO₂. However, ReO₂ also has a very small FS with frequencies of 8–12 T and an extremely low mass of $0.06 m_e$. This FS is located inside a Dirac loop and, therefore, its low mass is very likely related to the hourglass nodal chains with Dirac points and linear band dispersion. As mentioned, such low masses have not been observed in our experimental study of IrO₂. The low frequency F^* at about 30–50 T marked in Fig. 2(b) might be close to the low frequency in ReO₂ and was observed up to 4.3 K but the result of the LK fit gave an estimated effective

mass of the order of $1.0(2) m_e$ which is significantly heavier than the very light mass observed in ReO₂.

The Dirac points in ReO₂ lie rather close to the Fermi energy E_F (calculated at -105 meV and 136 meV , respectively [40]) and one of the associated nodal chains encircles the aforementioned small FS around the U point of the BZ, making the Dirac electrons observable in quantum oscillation experiments. In IrO₂, in turn, our calculations yield Dirac crossings at -150 and -320 meV below E_F at X and A , respectively [see Fig. 4(a)], whereas the DFT calculation in [4] place them at -250 and -400 meV . The Dirac crossings were observed experimentally by ARPES at about -150 and -400 meV in Ref. [6] and at about -200 and below -400 meV in Ref. [7]. The DNLs run along the edges of the BZ (see Fig. 2 in [7] for a clear illustration). Their position further below E_F makes the observation of very light Dirac electrons by dHvA oscillations less likely in IrO₂. Another indicator for topological properties, such as DNLs, is the observation of a nontrivial π Berry phase manifest in a phase shift in Shubnikov-de Haas and dHvA oscillations [41] which was experimentally observed in a number of systems [42–44]. ReO₂, too, is reported to have a nontrivial Berry phase for one of its (larger) electron pockets [45]. An analysis of the oscillating signal of IrO₂ by applying a fit function based on the LK formula that is comprised of all frequencies or by generating a Landau level fan diagram, both methods that reveal the Berry phase, was inconclusive due to the high number of frequencies and the observation of harmonics or combination frequencies. Therefore, no conclusions about the existence of DNL can be drawn from our quantum oscillation data. However, the material's very low residual resistivity at low temperatures attributed to the ultrahigh mobility of Dirac electrons [40] hints toward the existence of light electrons that contribute to the material's properties at low temperatures. As a reference, $\rho(2\text{K})$ in ReO₂ is $206 \text{ n}\Omega\text{cm}$ [40]. Exact values for IrO₂ are not published but can be estimated to range between 145 and $220 \text{ n}\Omega\text{cm}$ from the data in [46] and [47]. This low residual resistivity implies that Dirac electrons and their topological properties play a role for the electronic characteristics of IrO₂ and indicates that further experimental effort is warranted. The low masses of Dirac electrons are typically associated with low frequencies. As the magnetic background of the torque signal requires the subtraction of a higher order polynomial at some angles, there is the risk of unintentionally subtracting low frequency portions of the signal. When searching for low frequencies it might, therefore, be necessary to use different experimental techniques with less pronounced background like magnetoresistance measurements or different magnetometry techniques. The lowest frequency in ReO₂, for example, was only detected in Shubnikov-de Haas oscillations of magnetoresistance and not in the torque signal [40]. In addition, such experiments need to be conducted at intermediate temperatures where high frequencies are less dominant. A number of studies have used DFT in different implementations of increasing complexity with the inclusion of spin orbit coupling, Hubbard U , dynamical mean field theory, or hybrid functionals to compare the calculated band structure to ARPES results ([5–7]) or optical spectra ([48,49]) but there are still discrepancies between the measured and calculated results suggesting that there are fea-

tures of the band structure which *ab initio* calculations cannot capture.

V. SUMMARY AND CONCLUSION

We have performed dHvA experiments on single crystals of the DNL material IrO₂ using torque magnetometry with a piezoresistive microcantilever and have performed DFT-based calculations on the electronic band structure of this material. We find very good correspondence between our experimental data and results in [14] for the angular dependence of the dHvA frequencies, as well as very good agreement for the band dispersion and FS from our calculations and [4,6,7]. The temperature and field dependence of the oscillation amplitudes revealed enhanced effective masses of 2–3 times the electron rest mass and low Dingle temperatures indicating excellent sample quality. A low frequency of the order of 50 T and a mass of about 1 m_e was observed, but we did not detect any orbits with the very low electron masses expected to be associated with DNLs, probably because the corresponding band-crossing points sit well below the Fermi energy. However, the low temperature transport properties of IrO₂ hint toward the contribution of low mass electrons to the electronic properties of this material and further experiments with complementary techniques are warranted.

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DATA AVAILABILITY

The data that support the findings of this article are openly available [50].

APPENDIX: MAGNETIC AND TORQUE INTERACTION

Magnetic interaction (MI) is the result of an induced magnetic field in the sample caused by the oscillating magnetization. The effective field inside the sample would then be $B_{eff} = \mu_0(H_{ext} + M_{osc})$. This can lead to a stronger contribution from harmonics and modulate the amplitudes of oscillations. If more than one frequency is present, a feedback effect causes the occurrence of combination frequencies and their harmonics [27].

Torque interaction (TI) occurs due to feedback effects of the oscillating magnetic moment on the position of the lever in the field. This can alter the shape of the oscillations and lead to a stronger contribution of harmonics and side bands [28]. It is highly likely that either MI or TI or a combination of both is responsible for both sawtooth shaped oscillations and for the observation of prominent combination frequencies in the quantum oscillation spectrum of IrO₂ at high fields and very low temperatures.

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