LNCMI is formally associated with the “Université Grenoble Alpes”, the “Université Paul Sabatier”, Toulouse and the “Institut National des Sciences Appliquées”, Toulouse.
Dear Reader,

Please find here the 2021 annual report of the Laboratoire National des Champs Magnétiques Intenses (LNCMI). This report provides each year a quite complete overview of the in-house and collaborative scientific, as well as technical, activities of the LNCMI in 2021. LNCMI procures high magnetic fields (above the fields available in regular laboratories) to academic and industrial users from France, Europe and even abroad. In Grenoble, we can generate fields in continuous mode up to 36 T on a regular basis. In Toulouse, we can generate 90 T in the classical non destructive pulsed field installation and 200 T in the megagauss semi-destructive installation. In addition, we provide low temperature cryostats and many dedicated setups for optical, electrical, thermal measurements, and various spectroscopic techniques under magnetic field.

The LNCMI is a “Unité Propre de Recherche” of the Centre National de la Recherche Scientifique (CNRS- UPR3228) and is part of the French “Très Grands Instruments de Recherche” (Large Research Facilities). Located both in Grenoble and Toulouse, the laboratory is associated with the Université Grenoble Alpes, the Université Paul Sabatier de Toulouse and the Institut National des Sciences Appliquées de Toulouse. It is also one of the founding members of the distributed European Magnetic Field Laboratory, together with the High Field Magnet Laboratory in Nijmegen and the Hochfeld- Magnet Labor in Dresden. UK and Poland are official partners of EMFL, as well as the CEA.

Different upgrade projects are ongoing in LNCMI. The upgrade project to install a new 60 MVA power supply line and a dedicated transformer station at the Grenoble site is progressing, to be put into operation in 2023. The construction of the Grenoble hybrid magnet is now entering the (very) final assembly stage, to produce the first field in 2022.

As of 2021, users of the Toulouse facilities are finally able to take advantage of the upgraded 14 MJ and Megagauss capacitor banks as well as a new Helium liquifier, all of which are now fully operational. The commissioning of the new installations marks an important break after a 5-year period characterized by intense reconstruction work and the necessity to compromise between temporary shut-downs and the continuity of user operations. Incidentally, the latter aspect has also given rise to new technical solutions as the revised 14 MJ generator is now composed of mobile modules that will facilitate future maintenance work. In recognition for their commitment and the originality of their work the engineers and technicians involved in this project have been awarded the “Cristal Collectif du CNRS 2021”, a yearly prize that honors technical and administrative teams for outstanding achievements in their respective domains.

The upgrade of the Toulouse facility provides greatly increased reliability, availability and ergonomics. In addition, the world record of 100 T should be reached, and we have plan to continue up to 120 T in the coming years. A new big project is starting in Grenoble with the construction of a fully superconducting 40 T user magnet; the design study being financed by a European project SuperEMFL and the construction by an EQUIPEX funding of the French ANR.

Due to the COVID crisis, 2021 has been again, after 2020, an exceptional year, with many mail-in experiments due to travel restrictions that have often prevented our users to come to the LNCMI sites. A special word of thanks to our staff who have done their best to perform experiments and to keep the installations running under these difficult conditions. Despite these special conditions, the number of experiments, which were performed, was at the same level as before the crisis with many high level publications.

We hope that this report will convince you that high magnetic field science is a unique tool for exploring new materials which is often the key point of the development of new technologies.

Charles Simon
December 2021
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The LNCMI user facility

INTRODUCTION

The Laboratoire National des Champs Magnétiques Intenses (LNCMI) is one of the large scale facilities of the CNRS. It works as a research facility available both to its own researchers, as well as to external users. The high field facility is open to users from all over the world.

The LNCMI is also a member of the European Magnetic Field Laboratory (EMFL). EMFL is a legal entity (Association Internationale Sans But Lucratif, AISBL) in Brussels, Belgium, that provides the highest possible fields (both continuous and pulsed) for researchers. EMFL was founded in order to unite, coordinate and reinforce the three existing medium scale European high magnetic field laboratories - the Dresden High Magnetic Field Laboratory (Germany), the Laboratoires National des Champs Magnétiques Intenses in Grenoble and Toulouse (France), and the High Magnetic Field Laboratory in Nijmegen (The Netherlands) - within a single body in order to provide such an infrastructure. The British and Polish high magnetic field communities have also joined EMFL. The EMFL organization is the single entry point for users to obtain access to all European pulsed and DC field facilities. The access to superconducting magnets is treated separately by each facility.

HIGH MAGNETIC FIELDS AND INSTRUMENTATION AVAILABLE TO THE USERS

The LNCMI has resistive magnets at the Grenoble site, pulsed magnets at the Toulouse site as well as several superconducting magnets at both sites, achieving continuous magnetic fields up to 36 T and pulsed fields up to 90 T in which it is possible to install various experimental set-ups. The Toulouse site also operates a Megagauss facility producing pulsed fields up to 180 T in a semi-destructive magnet. Mobile generators and coils, designed for use in combination with other very large scale facilities, produce high fields for users of synchrotron, neutrons sources and intense lasers.

The technical and experimental environment also allows the user to combine very low temperatures and high pressures with very high fields. The laboratory offers a wide variety of instrumentation, allowing measurements in high magnetic fields in the following techniques:

- **Optical spectroscopy and magneto-optics**- optical microscope imaging, birefringence, dichroism and Faraday rotation, (micro-)photoluminescence spectroscopy, (micro-)Raman scattering, (far-)infrared spectroscopy.
- **Thermodynamic properties**- specific heat, thermopower and Nernst-Ettinghausen, DC/AC susceptibility, compensated-coil magnetometry, torque magnetometry, ultrasonic measurements (sound velocity and attenuation).
- **Magnetotransport**- magnetotransport with in-situ sample rotation, critical current of superconductors (wires, tapes and coils), contactless transport (TDO, PDO).
- **Magnetic resonance**- electron spin resonance, nuclear magnetic resonance.
- **Advanced sources**- X-Ray spectroscopies.
- **Environments**- $^4$He cryostats (1.5-300 K), $^3$He cryostats (down to 300 mK), dilution refrigerators (down to 30-100 mK), thermostats up to 300 °C, high pressure.
- **Other**- Megagauss facility (semi-destructive fields $> 170$ T), Mobile 1 MJ installation allowing X-rays, laser and neutron scattering under pulsed magnetic fields, levitation, thermometry.

ACCESS TO THE FACILITIES

Scientists who wish to obtain access to the resistive or pulsed magnets of the LNCMI must apply through the User Portal of the EMFL website (https://emfl.eu/SelCom/login.php). This application is drawn up according to a specific form and provides information demonstrating the relevance of the research work to be undertaken. The application is also used to ensure that the experimental conditions requested can be satisfied. Furthermore, scientists will find a large part dedicated to the user access on the EMFL website that includes relevant details about experimental possibilities with examples, instrumental set-ups and available magnets as well as practical information for users coming to the EMFL laboratories to perform their experiments.

For the use of the LNCMI mobile pulsed field facility, users must apply directly to the relevant very large scale facilities (ESRF, ILL, LULI, NIF).
Access to the superconducting magnets of the LNCMI as well as for technical projects using resistive magnets goes through an in-house procedure, which is open all year (http://lncmi.cnrs.fr/utilisateur/).

In the frame of the EMFL-ISABEL project, novel access procedures in order to satisfy the needs of all current and potential users are developed, implemented and evaluated. These procedures are based on feedback from the user community and on a study of ongoing practices at other research institutions (RIs). Thus, in 2021 a new dual-access method that will enable preliminary experiments in moderate fields at regional partner facilities, followed by high-field experiments at the EMFL high-field magnets was successfully introduced. These combined proposals have to be submitted during the regular calls, the EMFL selection committee will judge the pertinence of the low-field experiments and grant conditional access to the EMFL installations.

SUBMITTED PROPOSALS

A total of 127 proposals have been submitted in 2021. Figure 1 above shows on the left the distribution by proposal type (regular and dual-access proposals submitted to EMFL, proposals using advanced sources submitted to the very large scale facilities, proposals for superconducting magnets or instrumentation experiments going through the in-house procedure). The right part of figure 1 represents the affiliation country of the principal investigator (PI) by EMFL member countries, EMFL partners and other countries.

In the period 2017–2021, 749 projects have been submitted to the LNCMI, of which 358 proposals were requests for the different configurations of DC resistive magnets at the Grenoble facility, 288 demands for pulsed magnets at the Toulouse facility, 22 for using the mobile capacitor bank in combination with beam-lines at other RIs and 81 external projects submitted for superconducting magnets (see figure 2). The in-house projects performed on superconducting magnets are not considered here. A particular success of the possibility of working in different
bore diameters of the 24 MW DC magnets with fields of 36 T in a 34 mm bore, 31 T in a 50 mm bore and 20 T in a 170 mm bore must be underlined.

Furthermore, some users are particularly interested in large bore diameters such as 130 mm or 400 mm that use 12 MW which is half of the actual power available. These configurations are needed for the development of applied superconductivity, magneto-electrochemistry and magneto-hydrodynamics. The 12 MW magnets can also be used with the standard bore diameters of 34 and 50 mm achieving fields up to 25 T which can be of considerable interest for the preparation of very high field experiments or the development and testing of new experimental set-ups. This allows the LNCMI to offer access for very different types of experiments.

As a consequence of the Covid-19 crisis, with the lockdown and the travel restrictions, the number of submitted proposals at the first call of 2020 in spring (call 120) was very low. It had also been decided to extend the validity of proposals of the previous calls beyond 1 year. For the following call in autumn 2020, and both calls in 2021, the numbers returned to more normal levels. The laboratory does its best to offer magnet time to all users who had their proposals accepted by the selection committee. Nevertheless, the requested access is generally higher than the possibilities the laboratory can offer. Consequently, when submitting, the proposal quality is crucial.

SELECTION COMMITTEE

The EMFL regular and dual-access applications are examined and ranked by the EMFL selection committee, twice a year, in the course of December and at the end of June. The applications are ranked A, B or C. Projects rated A will be executed, those classified B are performed if the magnet time is available, and applications rated C are rejected. The ranking is made on the same basis for all applications, including those submitted by local scientists. The proposals for using the mobile pulsed field capacitor bank are evaluated directly by the relevant very large scale facility, the projects are rated A (accepted) or C (rejected). Figure 3 shows the high quality of the submitted proposals for the LNCMI sites for 2021, with 34% of the proposals rated A, 38% rated B+, 25% rated B and only 3% rated C.

Accepted experiments are subsequently programmed during the two semesters following the ranking of the selection committee. If the experiment has not been performed in this period, the principle investigator is required to resubmit the proposal mentioning the reasons why it could not be performed (issue with sample preparation, high pressure on the magnet time schedule, etc). Due to the Covid-19 crisis and the lockdown of the LNCMI facilities there was a considerable backlog of granted proposals in 2020. Therefore, accepted proposals of the previous calls 119 and 219 that could not be performed remained valid. Users were also invited to indicate special urgencies for performing their proposed experiment since 2020. The acceptance of an application means that for non-industrial users the entire cost of the experiment (scientific and technical support, electricity, cryogenic fluids, etc) will be covered by the budget of the laboratory, for industrial users a financial basis for the performance of the proposal will be defined.

The selection committee is divided into five groups, each with a different area of expertise (semiconductors, metals and superconductors, magnetism, applied superconductors, soft materials). In each area there are international experts from outside the EMFL consortium, as well as scientists from the different high magnetic field laboratories of the EMFL consortium, whose presence ensures that a project can technically be performed in the laboratory.
PERFORMING AN EXPERIMENT

Local contact

When the applicant is informed of the acceptance of his/her proposal, and of the number of pulses/shifts scheduled for the experiment, he/she is at the same time informed of the name of the local contact in charge of facilitating their work at the LNCMI. The local contact is a research scientist or an engineer of the laboratory. If an applicant has not identified a local contact, the laboratory management will appoint a scientist that the user can contact in order to define with him/her the conditions for the organization of the experimental work. The instrumentation groups of both sites of the LNCMI assure the technical support for the experiments.

Operation and magnet time

The periods of operation of the DC field facility, maintenance, works for upgrading and shut-down are fixed one year in advance. In general, the facility is stopped twice a year for maintenance with an additional short break of one week in spring. Further stops of the power installation can be decided for work, upgrading or setting up a new magnet.

The pulsed field facility is accessible the whole year except for the annual shutdowns, the first two weeks in August and one week for Christmas. The 14MJ generator is available to users for long pulse experiments. The 80T and 90T multi-stage coils are powered by the 14MJ and 3MJ generators and are fully available to users. The megagauss facility is open to users for optical spectroscopy and magnetization experiments up to 150T at $T = 10\,\text{K}$.

The magnet time schedule is drawn up for one semester and regularly updated. The demand for the facilities being high, visitors are strongly urged to get in touch with their local contact at the beginning of each semester, in January and in July, to discuss the scheduling of their experiment. LNCMI will do its maximum to satisfy the requests of its users and find the most suitable period for carrying out the experiments. The user support on the magnet sites is assured by the scientific local contacts and the instrumentation team.

The magnets operate on a schedule taking into account the availability of staff, magnets and instrumentation. For the DC field facility, the installation is operational up to 24 hours per day 7 days a week including weekends and bank holidays. Nevertheless, the experiments are scheduled during daytime or evening with two standard shifts from 9 a.m. to 4 p.m. and 4 p.m. to 11 p.m., and a weekly maintenance on Wednesdays from 9 a.m. to 4 p.m. If needed, the daily shift time can be flexible and adapted to the experiment’s needs. An LNCMI operator must be contacted during working hours for any operational request whereas outside normal working hours, a safety agent provides a presence in the laboratory.

At the DC field facility twice a month the technical teams (power facility group, magnet group, instrumentation group, operators etc.) and scientists of the currently scheduled projects come together in an interdisciplinary coordination meeting. This allows the exchange of information (planned experiments and special demands, observed incidents, technical improvements, organization issues etc.) and the coordination of technical interventions.

Regarding the superconducting magnets, the schedule is managed by the relevant scientific teams. The magnet time using the mobile capacitor bank in combination with beam-lines or lasers is directly attributed by the relevant very large scale facility.

ADMINISTRATION, FEEDBACK AND REPORTING

Users are invited to fill out and submit an online Admin form on the User Portal two weeks before the start of their experiment. The Admin form provides the facility with the information necessary to prepare the visit of the experimental team. The main proposer and all participants on the proposal are listed by default on the Admin form. The form can be modified and additional scientists added. Users will also indicate their travel and accommodation information for each participant. By submitting, the Admin form will be sent to the facility’s user support who will take care of accomplishing various formalities (declaring a presence to the central administration, authorizations and access to the campus, IT charter and safety formalities, logistics).

After performing the experiment, the user committee would like to receive feedback in order to constantly improve user access at the EMFL facilities. The feedback form can be filled out online. The data is handled confidentially by the User Committee (https://emfl.eu/emfl-user-organisation/).

Within two months following the experiment, users are required to submit a progress report. It can be filled online via the EMFL User Portal. This report is essential and is requested by the Selection Committee. It also is used to justify the use of the facilities.
ELECTRICITY COSTS & ENERGY MANAGEMENT AT THE DC FIELD FACILITY

Electricity costs

For the last 10 years the laboratory has to deal with steeply increasing electricity prices. Since 2020, the costs for electricity for the DC field facility are negotiated at the national level. The advantage is a more stable price over one or two years but with the drawback that with only a slight difference between low-tariff and high-tariff periods, the low-tariff periods are less interesting.

![Evolution of average electricity price.](image)

**FIG. 4.** Evolution of average electricity price.

Energy management

The DC field facility has set up different ways to optimize the relation of the number of scientific projects that can be performed with a more and more tight energy budget.

Firstly, users are invited to estimate the required field profile in advance with the help of the local contact and the user access manager. A special tool has been developed and can be made available to simulate the field profile and calculate the corresponding power consumption. An electricity budget is then allocated to each project. Furthermore, a thorough and detailed preparation of each experiment is essential in order to use the available magnet time efficiently.

Secondly, a new magnet operation mode has been developed this year and was implemented in September. It allows to reduce the mean power consumption by about 10 to 15%, taking advantages of the new materials available for magnets and of advanced piloting of the facility. The gain for a given experiment depends of course strongly on its field profile. Since September 690 MWh have been saved out of a total of 6120 MWh which has allowed more scientific projects to be performed (see figure 5).

![Energy saved since September 2021.](image)

**FIG. 5.** Energy saved since September 2021.

Finally, the facility has high degree of flexibility in power consumption. A collaboration with an energy aggregator has been started, and different modes to lower or cut the power for a short time during peak periods are under testing. These actions contribute to the optimal management of French and European energy resources by contributing to a better stability of the electrical network.
PROJECTS CARRIED OUT IN 2021

The statistics presented here summarize the use of the LNCMI facilities in 2021.

The left panel of figure 6 shows the country of origin of the projects that were performed on all magnets, the resistive, pulsed and superconducting magnets. Most of the projects were performed by scientists outside France. The users came mostly from Germany, Poland and the United Kingdom who are members or partners of EMFL, followed by users from the United States, Singapore, the Czech Republic and Canada. Due to the Covid-19 crisis and the travel restrictions, many of the experiments were performed remotely, with the commitment of the scientists of LNCMI, by mailing the samples and using video-conferences during the experiments.

Generally, most scientists who have already used our facilities come back regularly, introducing and progressively forming scientists who become themselves, in turn, new users. Nevertheless, every year the laboratory also welcomes completely new users. The projects realized at the LNCMI facilities are thematically distributed as shown in the right panel of figure 6 with focus on projects in the semiconductors, the metals and superconductors and the magnetism areas. Projects for instrumentation and technical developments go through an in-house proposal procedure.

FIG. 6. Performed proposals in 2021: by country of origin (left) and by research area (right).

FIG. 7. Distribution of the number of magnetic field shots at pulsed facility and magnet hours and power used at the DC field facility in 2021.
The top panel of figure 7 shows the magnet time and power used at the DC field facility. In total, the facility has provided 2035 hours of magnet time, mostly used on the 24 MW magnet sites (left) with a total power consumption of 11.35 GWh (right).

The bottom panel of figure 7 shows the distribution of the number of shots, depending on the power supply used, performed at the pulsed field facility or using the mobile capacitor bank in combination with the neutron source at the ILL. The long pulse field facility in Toulouse has provided 1760 shots at high field on a total of 3905 shots, 80 shots on the Megagauss facility and 797 shots using the mobile capacitor bank at ILL.

CONCLUSION

The technical and scientific environment of LNCMI, adapted to research in high magnetic fields, is highly appreciated by the international scientific community. A large majority of users shows great interest in the laboratory and they come regularly in order to perform their experiments, thereby strengthening collaborations between research groups. The number of projects submitted and performed at the LNCMI demonstrates the international scope of the laboratory. As the schedule of the Grenoble 24 MW magnets is extremely tight and the electricity costs high, it is essential to optimize the use of these magnets. Experiments may also be transferred from one facility to the other, after discussion with the scientists, if the schedule is too tight, or in case the another facility has a better adapted set-up for an experiment.

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Outreach activities

The on-going Covid-19 pandemic has led to the cancellation/postponement of almost every international scientific conference scheduled in 2021. The fluctuating and uncertain sanitary conditions make it almost impossible, and indeed imprudent, to organize any event with a large number of participants. In a similar manner, the outreach activities of LNCMI-G&T have been necessarily devastated in 2021.

LNCMI-Grenoble

Mock-up of superconducting levitation motorized by Laplace forces

To improve one of the demonstration tools for the visit of LNCMI-G, we have offered an internship for the realization of a mock-up. The aim of this mock-up is to have a mobile in levitation above a circuit made of permanent magnets and motorized by Laplace forces. Based on the work done by the students, the key points of this project are:

• the magnetic profile of the circuit
• the shape of the coil for the propulsion
• the electrical generator
• the production of the mobile

The circuit proposed is an oval track which is three magnets wide. This permits to stabilize the mobile in the center of the track. The final track is made of a total of two hundred permanent magnets.

The students have provide a functional software in Python. It is then possible to visualize the Laplace forces along a coil drawn on the screen. Thus, it is possible to determinate the most efficient shape of the coil for a specific track. An example of the result produced by this software is shown in figure 8.

FIG. 8. Visualization of Laplace forces along a coil.

The other achievement is the production of a mobile with the possibility to have an on-board battery (figure 9). It was made by 3D-printing at the LNCMI-G which can be used for further adaptations.

Currently, this mock-up is not fully functional. The motorized part does not permit for a self motion of the mobile. This project will be completed in a second internship, hopefully in 2022.

Visits

Several visits have taken place at the LNCMI-G this year:
The team from the “Lycée Saint-Denis - Annonay” of the “Olympiades de Physique France” has won a visit of the LNCMI-G. Another team from the “Lycée Pilote Innovant International - Jaunay-Marigny” has won the Trescal price with a subject on the magnetism entitled: “Descente magnétique”. Their presentation is visible at this url: https://www.youtube.com/watch?v=SEWv9vCORDs.

“Fêtes de la science 2021”: For this event, it has been possible to organize 4 visits during the week and a pack of 4 visits on Saturday (see figure 10).

This year, due to the successive waves of the Covid-19 pandemic, all our recurrent outreach activities (nuit des chercheurs, fête de la science, lumière sur le quai...) were cancelled due to circumstances beyond our control. Likewise, we have not been solicited for our usual general audience conferences in schools and colleges. Regrettably we were also forced to decline two class visits of the pulsed field installation. Nevertheless, the outreach team in Toulouse remain extremely motivated, and we hope to be able to resume our outreach activities as soon as possible in 2022.

Emerging Layered Materials
Topological Hofstadter butterfly in twisted bilayer graphene

Moiré superlattices in two-dimensional van der Waals heterostructures provide an efficient way to engineer electron band properties. Among them, twisted bilayer graphene (tBLG) has emerged as a rich platform to study strong correlations, superconductivity, magnetism and band topology. Stacking two graphene sheets with a slight rotation by an angle \( \theta \) creates a moiré super potential. The magic angles \( \theta_m \) in tBLG are a series of well-defined twist-angles for which the moiré bands are predicted to become ultra-flat, and have a helicity (and thus a topology) different from the one of \( \pi \) bands in monolayer graphene. While the tBLG at \( \theta_{m1} \sim 1.3^\circ \) has already been extensively studied, tBLG at well-defined smaller magic angles (e.g. \( \theta_{m2} \sim 0.5^\circ \)), in which inhomogeneity of moiré patterns is much more sensitive to twist angle fluctuations, has so far rarely been experimentally investigated. tBLG at \( \theta_{m2} \) offers an exceedingly large moiré unit cell, giving rise to a multitude of closely packed flat moiré bands. In this work [Lu et al., PNAS 118, 30 (2021)], we have investigated magneto-transport of tBLG devices, with \( \theta \) close to the predicted second magic angle \( \theta_{m2} \sim 0.5^\circ \). Our tBLG device is encapsulated with crystallographically non-aligned insulating layers of hexagonal boron nitride (hBN) and its carrier concentration \( n \) is capacitively controlled by an underlying graphite layer (see figure 11a).

As can be seen in figure 11b, at zero magnetic field, the longitudinal resistance \( R_{xx} \) exhibits small peaks and the Hall voltage sign changes (not shown) at equally spaced, integer multiples of \( n = s n_s \) (where \( n_s = 4/\Omega_m \) and \( \Omega_m \) is the area of the moiré unit cell) which marks the transitions between the individual flat bands. The resistance peaks are strongly enhanced by a small perpendicular magnetic field \( B_{\perp} \) and develop thermally activated, gapped behavior, consistent with the opening of the \( C_{\perp,T} \) protected Dirac nodes. When the magnetic field is increased further, Landau levels (LL) develop within the individual bands, and form gaps with nonzero Chern numbers \( C = 4, 8, 12, \ldots \) due to spin and valley degeneracy, which manifest themselves as dips in \( R_{xx} \) (blue regions in figure 11b). These dips trace back at \( B_{\perp} = 0 \) to the band-edges at integer fillings \( n/n_s \) and follow a well-defined slope \( \partial n/\partial B_{\perp} = Ch/e \), where \( e \) is the electron charge, \( h \) is Planck’s constant. Above \( B_{\perp} > 8 \) T robust quantum Hall states develop, with \( R_{xx} \sim 0 \) and quantized plateaus in the Hall resistance of \( R_{xy} = h/(Ce^2) \). In contrast, all trivial band gaps have \( C = 0 \) and emerge from \( B_{\perp} = 0 \) T without a slope. These gaps are identified by \( R_{xx} \) peaks (red) that follow straight vertical lines (for clarity, we highlight in the middle panel the most pronounced gaps as different colored lines). Hofstadter gaps (\( \pm 4, s \), \( \pm 8, s \), etc, are strikingly not confined within the moiré band from which they emerge at \( B_{\perp} = 0 \), but continuously extend into the higher lying moiré bands at high \( B_{\perp} \), and cross all the \( (0,s) \) band-gaps. This is qualitatively distinct from the Hofstadter butterfly emerging from of topologically trivial bands, where the spectrum is confined within the energy bandwidth of each band, defining an “energetically bounded” Hofstadter butterfly. The Hofstadter spectrum of a topological band is on the other hand not energetically confined to the bandwidth of the band and can propagate until it connects to the Hofstadter spectrum of another band. Such a topologically non-trivial, unbound and connected Hofstadter butterfly spectrum is clearly demonstrated in our \( \theta \sim 0.45^\circ \) tBLG device, and most-likely related to the single-particle fragile topology hosted by the two lowest moiré bands of each spin and valley in tBLG.

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Magneto-plasmons in graphene nanoribbons

Plasmons are usually optically inactive because the plasmon dispersion does not intersect the dispersion of light. Hence, the missing momentum must be provided, e.g., by a diffraction grating. The grating can be made of graphene itself (graphene nanoribbons), or the grating can be formed by metal stripes fabricated on top of the two-dimensional graphene sheet.

A disadvantage of nano-fabricated ribbons is contamination of graphene by the resist, which can alter the doping level or carrier lifetime. Hence, we also fabricated ribbons by protecting graphene by a 15 nm thick gold layer. Surprisingly, plasmons can also be observed on as-grown hydrogen intercalated graphene on SiC. The origin of the momentum matching in these as-grown graphene sheets is currently under investigation.

We fabricated a series of graphene nanoribbons using different electron-beam lithography techniques, and we compared their magneto-optical response with as-grown graphene. We measured optical transmission by Fourier Transform Infrared spectroscopy (FTIR) in the far-infrared spectral range. The energy of the magneto-plasmon absorption exhibits scaling with the ribbon width, as seen in figure 12. We find that gold protection, although keeping graphene resist-free, depletes charge in the graphene ribbons, and, as a result, the plasmonic absorption is relatively weak. We show that hydrogen intercalation is a critical step towards the optical activity of plasmons in as-grown graphene [Paingad et al., Adv. Funct. Mater. 31, 2105763 (2021)]. Argon-grown graphene without hydrogen intercalation never shows measurable optical absorption. On the other hand, the plasmon absorption is observed even in argon-grown graphene if a regular array breaks the translation symmetry of lithographically made ribbons using polymethylmethacrylate resist. We also observe plasmon resonance in a continuous graphene sheet with gold grating made by the lift-off technique.

We also searched for topologically protected states formed by plasmonic excitations. Topologically protected states form in a class of crystalline topological insulators, and they can be observed in the surface states of the electronic spectrum of condensed matter. Besides the electronic spectrum, the topologically protected states are also predicted for quasi-particle excitations, such as plasmons. Here, the interplay between the plasmonic spectrum and spatial modulation of graphene sheets is the driving force. The hexagonal graphene antidot lattice results in the formation of the topologically protected states, and the graphene ribbons provide the missing momentum for optically observable plasmons. We optimized the fabrication of large-scale patterning of periodic structures on graphene. The requirement on the measurably high signal in FTIR absorption leads to the necessity of high-density ribbons on at least 2 mm² area. Here, we developed a novel nanopatterning technique that speeds up the lithography 4-5 times. In figure 13, we show an example of these new fast-patterned ribbons. However, as the ribbons were gold-protected, we assume much-reduced doping is responsible for the weak magneto-optical absorption observed in these ribbons.

![FIG. 12. Optical transmission in (left) 3 µm and (right) 0.35 µm wide graphene nanoribbons on SiC(0001). The dashed lines show a guide-for-eye expected magnetic-field dependence of the optically active plasmon absorption.](image1)

![FIG. 13. Scanning electron microscope image of graphene nanoribbons patterned by a hexagonal periodic antidot array.](image2)

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Wide critical fluctuations of the field-induced phase transition in graphite

A phase transition is accompanied by sharp discontinuities of the thermodynamic properties. Quantifying entropy by measuring the specific heat across the transition pins down the order of the transition and informs on the underlying microscopic interaction. Of particular interest is the critical regime of the phase transition, which allows the identification the universality class of the transition and provides direct information on the order parameter.

Critical fluctuations are important when their amplitude is comparable with the amplitude of the jump of the specific heat, $\Delta C$. In most cases, the critical fluctuations are located in the extreme vicinity of the transition, and therefore hardly observable. One notorious exception is the $^4$He superfluid transition for which the shape of the transition is determined by the critical fluctuation. Near a quantum critical point, thermal fluctuations are replaced by quantum mechanical zero-point fluctuations, which can produce new quantum phases.

Here, we report on the electronic specific heat ($C_{el}$) of graphite, using the state of the art of calorimeters, when all the carriers are confined in the lowest Landau levels. This situation corresponds to the so-called quantum limit, which can be easily achieved in dilute metals such as graphite. In presence of a large magnetic field on a 3D electron gas system, the kinetic energy, in the plane perpendicular to the magnetic field, is quenched. In that plane, the electrons circulate in quantized orbits while they are still propagating freely along the magnetic field. The resulting trajectories of the electrons are thus helices spiraling around the field lines. In the presence of a magnetic field, the almost isotropic three-dimensional conductor becomes a quasi one-dimensional conductor.

In the early 1980s, a phase transition was discovered in graphite, observed as a sharp increase in the in-plane magnetoresistance of graphite at $B=25$ T. The precise nature of this phase is still debated. Here we find that the quantum limit of graphite is marked by a steady field-induced enhancement of $C_{el}$, signaling the enhancement of electron-electron correlations (see figure 14). Deep in this regime, we detect a jump in $C_{el}$, unambiguously establishing a second-order phase transition induced by the magnetic field. As the magnetic field is reduced, the anomaly shifts to lower temperature and widens. It evolves from a BCS mean-field type transition at $33$ T to a cross-over regime below $25$ T.

At the lowest critical temperature ($T_c \sim 1$ K), fluctuations can be observed up to two times $T_c$. We identify them as critical fluctuations associated with an exceptionally large Ginzburg number due to a low energy condensation. Comparison with a number of other phase transitions shows what distinguishes this phase transition and its criticality. In graphite, the difference in the heat capacity between the normal and ordered phase within a coherence volume rapidly drops with decreasing magnetic field and becomes exceptionally low.

For more information please see [Marcenat et al., Phys. Rev. Lett. 126, 106801 (2021)].

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Exciton many-body states in monolayer MoS$_2$

The role of electron-electron interactions on the properties of two-dimensional (2D) electron and hole gases has been extensively studied in GaAs, AlAs and recently also in graphene. Typically the spin and valley susceptibility of the system is found to be enhanced by interactions. Signatures of collective phenomena are particularly pronounced in 2D materials such as semiconducting transition metal dichalcogenide (TMDC) MoS$_2$ owing to strong Coulomb interaction. The large variation of reported g-factors for excitons in MoS$_2$ and other 2D materials with arbitrary carrier concentration further highlights the necessity for studying valley Zeeman shifts with tunable and controlled carrier densities in 2D semiconductors.

In [Klein et al., Physical Review Research, 3, L022009 (2021)], we address the variation of g-factors in the literature which is a direct consequence of the many-body interaction with the fermionic bath. We control the carrier concentration $n$ in a dual-gate field effect device and study the magneto-optical response in high magnetic fields up to $B = 28$ T. We study the magnetic field dependence of neutral and charged excitons which encode the evolution of the total magnetic energy including carrier spin, valley magnetic moment and cyclotron phenomena arising from quantization of electrons and/or holes into discrete Landau levels (LLs). From our measurements, we directly observe that shape and magnitude of the valley Zeeman shift $\Delta E_{\text{VZ}}$ of excitons very sensitively depend on the spin and valley texture. Our results suggest that the interaction of the exciton with the Fermi-bath at low densities is driven by dipolar spin-interaction which differs from previous observations that have not taken into account the unique LL quantization in 2D TMDCs.

We excite the sample with unpolarized light at $\lambda = 514$ nm and an excitation power of 30 $\mu$W and detect $\sigma^-$ polarized PL at $T = 5$ K. Due to the robust optical selection rules in monolayer TMDCs, we only probe the emission from excitonic recombination in the K’ valley for positive and negative polarities of the $B$ field. We symmetrically tune the voltage applied to the top and bottom gates, which effectively counteracts Fermi level pinning due to the reduction of band tail states. This allows us to observe the $X^+$ transition in the p-charged regime I for the first time in MoS$_2$. The data reveal narrow emission lines of the $X^0$ due to the hexagonal boron nitride (hBN) encapsulation and LL oscillations in the intravalley $X^-$ at higher magnetic field.

An applied magnetic field lifts the K/K’ valley degeneracy by shifting time-reversed pairs of states in opposite directions in accord with the Zeeman energy $-\mu_B B$.

This effect will shift the exciton energy when the magnetic moment of conduction and valence bands are not equal. At charge neutrality, we measure a g factor of $X^0$, $g_{X^0} = -1.27 \pm 0.09$. While $E_{\text{VZ}}$ is completely linear at charge neutrality, it becomes nonlinear with electron doping. The nonlinear valley Zeeman shift in the $n$ charging regime is a direct consequence of a dipolar spin-interaction of the exciton with the local spin-polarized fermionic bath. Our reported results clearly show that the interaction is driven by few electrons occupying fully spin-polarized LLs, the effective magnetic moment originates from a magnetic field-induced net spin-polarization of excess carriers. We summarize the valley susceptibility of all features in our accessible density range in figure 15.

Our results show that all excitons in 2D materials are many-body correlated states that have a magneto-optical response that is sensitive to the local carrier density and related spin and valley textures. We explain the large variation of g factors observed in the literature as arising from lack of control of local doping. The findings of our study represent an important step toward studying and engineering many-body-related phases and novel interaction phenomena in atomically thin materials.

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FIG. 15. Carrier density-dependent g factors of $X^0$ (black and gray data), the negatively (red and blue data) and positively (green data) charged excitons $X^-$ and $X^+$, and the $X^0$ feature (yellow data).
Rydberg series of dark excitons and the conduction band spin-orbit splitting in monolayer WSe$_2$

Strong Coulomb correlations together with multi-valley electronic bands in the presence of spin-orbit interaction are at the heart of studies of the rich physics of excitons in monolayer transition metal dichalcogenides (TMDs). Those archetypes of two-dimensional systems promise a design of new optoelectronic devices. In intrinsic TMD monolayers the basic, intravalley excitons, are formed by a hole from the top of the valence band and an electron either from the lower or upper spin-orbit-split conduction band subbands; one of these excitons is optically active, the second one is dark, although possibly observed under special conditions.

Here we demonstrate the s-series of Rydberg dark exciton states in tungsten diselenide monolayer, which appears in addition to a conventional bright exciton series in photoluminescence spectra measured in high in-plane magnetic fields. The comparison of energy ladders of bright and dark Rydberg excitons is shown to be a method to experimentally evaluate one of the missing band parameters in TMD monolayers: the amplitude of the spin-orbit splitting of the conduction band.

Representative photoluminescence spectra of the investigated WSe$_2$ monolayer are presented in figure 16. Inspecting the low spectral range (figure 16a), one recognizes a characteristic set of multiple photoluminescence transitions, typical of WSe$_2$ monolayers. First, we examine the photoluminescence (PL) spectrum measured in the absence of magnetic field (B), and recognize the characteristic sequence of emission peaks related to the Rydberg series of $1s^B, 2s^B, 3s^B$, up to $4s^B$ states of bright excitons. The PL spectrum measured at B=0 shows also a weak transition related to the ground state, $1s^D$, of dark exciton.

When in-plane magnetic field is applied the intensity of the $1s^D$ emission grows progressively, and eventually dominates the PL spectrum in the limit of high magnetic fields. Importantly, new transitions driven by the application of the in-plane magnetic field appear also in the higher spectral range in the vicinity of the PL peaks associated to the excited states of bright excitons. These new transitions, labeled as $2s^D, 3s^D$ and $4s^D$ in figure 16, are attributed to the excited Rydberg states of the dark exciton.

The analysis of the energy ladders of bright and dark excitons allowed us to determine one of the missing band parameters, the amplitude of the spin-orbit splitting in the conduction band of this broadly investigated 2D structure. Its derived value, $\Delta_s = 14$ meV, is significantly lower than that commonly assumed, which calls for a revision of theoretical calculations of the electronic bands in TMD monolayers. Moreover, our results suggest that the difference between the binding energies of bright and dark excitons can be fully explained by the difference in the masses of electrons in the two spin-orbit-split conduction bands, without referring to exchange interactions.

For more details please see Kapuscinski et al., Commun Phys 4, 186 (2021).

![FIG. 16. Low-temperature photoluminescence (PL) spectra measured at selected in-plane magnetic fields in the energy region of both (a) ground and (b) excited states. Spectra are shifted for clarity and normalized to the $1s^D$ feature in the lower energy region and to the $2s^D$ feature in the higher energy region. Note that the intensity scales differ for these two regions.](image-url)
Excitonic complexes in an n-doped WS$_2$ monolayer

The origin of emission lines apparent in the low temperature photoluminescence spectra of n-doped WS$_2$ monolayer embedded in hexagonal BN layers was investigated using external magnetic fields and first principles calculations. We find that all the observed emission features are due to the negatively charged excitons, with the exception of the neutral A exciton line. Consequently, we identify emission due to both the bright (singlet and triplet) and dark (spin- and momentum-forbidden) negative trions as well as the phonon replicas of the latter optically-inactive complexes. Moreover, semi-dark trions and negative biexcitons are also seen.

We identify all emission lines apparent in the low-temperature PL spectra of n-doped WS$_2$ ML embedded in hBN layers using external magnetic field. An example of such an identification, for the case of the phonon replicas of dark trion ($T^D$) feature, is presented in figure 17. The energy difference between $T^D$ and the additional lines correspond well to the energies of phonons found in this material. Moreover, the circular-polarization of the emission lines indicates whether it is an electron (three lines that show strong circular-polarization) or a hole (one line that shows no circular-polarization) that is scattered by a phonon to the opposite valley. The evolution of the emission lines in the magnetic field further proves this assignment, as for the features related to the electrons scattered by a phonons we found a negative $g$-factor, and for a resonance related to scattering of a hole a positive $g$-factor.

Moreover, we found that the extracted $g$-factors of all transitions may be arranged in three groups revealing the nature of the electron-hole recombination; bright, intravalley, and intervalley dark. We explain their signs and magnitudes with the aid of first-principles calculations. The observed grouping may provide an opportunity to determine the origin of the so-called localized excitons in the emission spectra of the WSe$_2$ and WS$_2$ MLs exfoliated on Si/ SiO$_2$ substrates. The obtained $g$-factors of the spin-split subbands in both the conduction and valence bands are important for better understanding of the interlayer transitions in van der Waals heterostructures.

For more details please see Zinkiewicz et al., Nano Letters 21, 6 (2021).

FIG. 17. (a) Schematic illustration of possible recombination pathways of dark trions assisted by the emission of optical ($E''$) and acoustic (ZA, LA) phonons from the K or $\Gamma$ points of the BZ, which give rise to the PL of so-called phonon replicas. (b) The calculated phonon dispersion of WS$_2$ ML. (c) Low-temperature PL spectrum due to the intravalley TD dark trion and its phonon replicas measured on the studied WS$_2$ ML at $B_{||} = 10$ T. (d) Helicity-resolved low-temperature PL spectrum with the emission lines related to phonon replicas of the studied ML at zero magnetic field under circularly polarized excitation with the TS energy. (e) (left panel) False-color PL map as a function of out-of-plane magnetic field ($B_{\perp}$). (right panel) Transition energies of the $\sigma^\pm$ (red/ blue points) components of the studied line related to phonon replicas as a function of the out-of-plane magnetic field.

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Optical properties of MoS$_2$ on GaAs nanomembranes

We have investigated the optical properties of monolayer transition metal dichalcogenides (TMDs) on III-V nanostructures, specifically atomically thin MoS$_2$ placed on patterned GaAs substrate. The pattern is a high-quality, CVD-grown GaAs nanomembranes. The nanomembranes consist of an array of parallel, $\approx 100$ nm thick nanowires, separated by $1 \mu$m distance (figure 18b). The residual of the patterning process is a thin SiO$_2$ film ($\approx 20 \mu$m) on the GaAs substrate with the exception of the nanomembranes. The MoS$_2$ flake has been deposited on the patterned surface. Distinct areas of the flake can be highlighted: (i) part lying on the nanomembrane (ii) a small part outside of the nanomembrane, (iii) part in between two adjacent nanomembranes (figure 18a).

![Micrograph of MoS$_2$ deposited on GaAs nanomembrane.](image)

The photoluminescence (PL) spectrum of the investigated sample features pronounced emission peak around 1.91 eV attributed to the trion ($X_T$) and broad high energy peak at 2.07 eV attributed to B exciton ($X_B$). The A exciton ($X_A$) emission is only visible on a flat reference monolayer flake (lying approximately 100 $\mu$m from the nearest pattern) as a small high energy shoulder around 1.94 eV (figure 19a). The domination of the trion in the PL spectrum can be explained by charge transfer between GaAs/SiO$_2$ substrate and MoS$_2$ due to the alignment of the Fermi levels.

The presence of the nanomembrane is expected to result in redshift of PL emission energy due to tensile strain. Indeed, we have observed changes on the order of $\approx 5 - 15$ meV across different regions of the flake, with the lowest energy located in the area where flake lies on top of the nanomembrane (figure 19b). This result is corroborated by the shift of absorption energy in reflectivity measurements and by frequency shift of the Raman modes. All three experiments show very similar picture of inhomogeneous strain distribution across the flake with effective value on the order of $\approx 0.15\%$. The inhomogeneity of the optical properties could be explained by specific local morphology i.e. how well the flake adheres to the membrane/substrate and appearance of wrinkles on the flake. Those conditions influence both local strain and dielectric environment, which influences the electronic structure. Detailed understanding of the inhomogeneity and departure from a simple picture of on or off nanomembranes can be obtained by AFM mapping of the sample allowing for direct local correlation between morphology and optical properties.

![PL at $T = 4.2$ K.](image)

We also observe a non-zero degree of linear polarization (DOLP) of PL emission from the MoS$_2$ deposited on top of the membrane, meanwhile the emission from parts of flake lying outside of the membrane and the flat reference flake shows no DOLP (figure 20). The probable explanation for this phenomenon is the presence of effective uniaxial strain induced by the presence of the nanomembrane. The low value of $\approx 5 - 15\%$ DOLP might be due to averaging of the optical response from the sample, as the optically probed area $\approx 1 \mu$m is 10 times larger than the thickness of the nanowires. Therefore the DOLP at the tip of a nanowire might be larger.

![Polarisation resolved PL at $T = 4.2$ K.](image)
Interlayer excitons in MoSe₂/2D perovskite heterostructures

Here we present evidence for the formation of interlayer excitons (IX) in heterostructures (HS) composed of 2D perovskite and monolayer TMD. The structures consisted of \( n = 1 \) two dimensional PEA₂PbI₄ (PEPI) perovskite (where PEA is phenylethylammonium) or BA₂PbI₄ (BAPI) perovskite (where BA is butylammonium), and MoSe₂ monolayer. The stack was encapsulated in hexagonal boron nitride and placed on Si/SiO₂ substrate. Our DFT calculations show that the obtained HS has a type-II band alignment, where conduction band minimum is localized in MoSe₂ and valence band maximum is in perovskite layer. Charge transfer, often present in this kind of structures, thus seems possible, and is indeed observed in optical spectroscopy.

For more details please see [Karpinska et al., Appl. Mater. Interface 13, 33677 (2021)].

Figure 21 shows photoluminescence (PL) spectra measured at 5 K for (a)-PEPI/MoSe₂ and (b)-BAPI/MoSe₂ stacks. The comparison is made between bare TMD and perovskite flakes and HS region. Interestingly, in HS region on the low energy side, we observed an additional peak with intensity higher than that of charged exciton of MoSe₂. We ascribed this peak to interlayer exciton (IX). Similar results was observed in BAPI/MoSe₂ structure. To understand the origin of this peak we conducted additional time-resolved PL (shown in figure 22(a)) and PL excitation measurements (figure 22(b)). When comparing PL decays of charged exciton of MoSe₂ on bare TMD flake and in HS, we can clearly observe that its lifetime is drastically decreased in HS region. This is in agreement with charge transfer hypothesis, as PL decay sums up all the processes leading to reduction of exciton population. Moreover, PL excitation measurements show that IX emission intensity exhibits a resonance close to the energy of exciton B of the TMD, and of ground exciton of perovskite. This agrees well with our charge transfer hypothesis.

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Destructive photon echo formation in six-wave mixing signals of a MoSe$_2$ monolayer

Monolayers of transition metal dichalcogenides display a strong excitonic optical response. The possibility of encapsulating the monolayer with hexagonal boron nitride allows to reach the limit of a purely homogeneously broadened exciton system. On such a MoSe$_2$-based system, ultrafast six-wave mixing spectroscopy is performed and a novel destructive photon echo effect is found. This process manifests as a characteristic depression of the nonlinear signal dynamics when scanning the delay between the applied laser pulses. By theoretically describing the process within a local field model, an excellent agreement with the experiment is reached. By using an effective Bloch vector, we demonstrate that the destructive photon echo stems from a destructive interference of successive repetitions of the heterodyning experiment.

Using the strong excitonic optical response of transition metal dichalcogenides monolayers we explore the six-wave mixing (SWM) signals from a MoSe$_2$ monolayer in the low excitation limit, which here represents the $\chi$-regime. Our results show that the signal dynamics exhibit a characteristic temporary suppression depending on the delay between the pulses.

The measured SWM dynamics as a function of the real time $t$ after the third pulse and the delay $\tau$ are shown in figure 24. Here, the two pulses with $\varphi_1$ and $\varphi_2$ arrive at $t = -\tau$ and the pulse with $\psi_3$ at $t = 0$. The signal consists of a strong maximum at small $t \approx 0.5$ ps and $\tau \approx 0$. Moving to negative delays $\tau < 0$ (pulse 3 is arriving before 1 and 2), the signal is strongly damped. In correspondence with the previously described constructive signal enhancement in the photon echo, we call this pronounced signal reduction a destructive photon echo.

![Figure 23](image_url)  
**FIG. 23.** Sample structure consisting of a multilayer hBN, monolayer MoSe$_2$, multilayer hBN stack. (Left) Four-wave mixing (FWM) generated by two laser pulses with tunable delay $\tau$. (Right) Six-wave mixing (SWM) with three excitation pulses having a tunable delay $\tau$ between pulses 2 and 3. The FWM and SWM dynamics are measured in real time $t$.

We found that the SWM-relevant quasi-Bloch vectors are distributed along Lissajous curves which adds surprising aesthetics to the involved Bloch vector dynamics in SWM. We compare the traditional photon echo in FWM with the newly discovered destructive photon echo in SWM. We define three criteria that characterize the echo formation. Firstly, the timing of the photon echo formation is basically determined by the chosen delay $\tau$ between the two laser pulses. Slight deviations from the exact $t = \tau$ timing are used to learn about internal dynamics of the studied system. The same holds for the destructive echo in SWM. As discussed in detail the two main contributions compensate each other exactly at $t = \tau$. In the case of the destructive echo we have shown that we need two paths which interfere destructively to explain the novel echo effect.

Finally, the fundamental source of the photon echo is any sort of inhomogeneity, which might appear in space by a locally varying exciton energy, or in time via external noise that acts on the optical transition energy. For more details please see [Hahn et al., Advanced Science, 2103813 (2021)].

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Layered materials, such as transition metal dichalcogenides (TMDs), have attracted major interest thanks to their rich variety of ground states and the possibility of their exfoliation down to an atomically thin level. Recent observations of intriguing physics in artificially assembled heterostructures highlight the importance of interlayer interactions. Relevant aspects of the interplane coupling can be deduced by probing out-of-plane charge transport, even in bulk materials.

However, enforcing the current flow strictly along the c axis can be rather challenging due to the crystal’s common flake-like appearance and their propensity for delamination. Such a pitfall can distort measurement results by orders of magnitude, as demonstrated in our recent study of microstructured samples of 1T-TaS2 with a well-defined current flow [Martino et al. npj 2D Mater. Appl. 4, 7 (2020)]. This observation motivates a careful re-examination of the out-of-plane charge transport properties in this class of materials by adopting the latest state-of-the-art for quantum matter microfabrication.

In this research we investigated the out-of-plane electrical resistivity of bulk monocrystalline 2H-NbS2. This material is one of the three known structural variants of layered NbS2. 2H-NbS2 is a superconductor below 6 K, proposed to have a multiband character, and does not show any charge density wave (CDW) order, which is uncommon for metallic TMDs. The 1T polytype has been attracting interest recently as a candidate for realising a two-dimensional magnetic system.

Due to its non-trivial synthesis procedure, NbS2 crystals have up to 18% of pairs of neighbouring layers stacked in a 3R-like manner. Additionally, diffuse X-ray scattering experiments [Leroux et al. Phys. Rev. B 97, 195140 (2018)] revealed weak traces of the $\sqrt{13} \times \sqrt{13}$ CDW reconstruction, which appears as a triangular superlattice of David-star-shaped clusters defined by 13 Nb atoms. Earlier theoretical investigations have predicted 1T-NbS2 to be particularly prone to developing such a CDW order with a consequent magnetic ordering of the unpaired spin at the centre of each David-star superlattice cluster. One can therefore conclude that single crystals of 2H-NbS2 contain rare, atomically thin inclusions of the 1T polytype.

Our study of 2H-NbS2 revealed a remarkably strong low-temperature anomaly in the compound’s out-of-plane resistivity $\rho_c$ (see figure 25), manifesting as a minimum at around 40 K, followed by a pronounced upturn upon further cooling. The feature is simultaneously invisible in the in-plane resistivity (\rho_{ab}), and shows a highly anisotropic response to the magnetic field. Resistivity anisotropy measurements have been conducted at LNCMI thanks to the available 34 T static field (Grenoble) and 65 T pulsed field (Toulouse), on single crystals microstructured by focused ion beam.

This phenomenon is understood to be linked to the 1T-type of structural defects specific to 2H-NbS2. We argue that planes of magnetic moments, hosted by the inclusions of 1T-NbS2, cause a Kondo effect observable only when the current flows across these planes.

For more details please see [Martino et al. npj 2D Mater. Appl. 5, 86 (2021)].

![FIG. 25. Out-of-plane resistivity ($\rho_c$) of 2H-NbS2 as a function of temperature (T) for various longitudinal (main plot) and transverse (inset) magnetic fields (B). Solid lines and markers represent the measured data. Dashed lines in the main plot are fits according to the numerical renormalization group theory of Kondo effect. The fitted model includes a field-independent contribution due to the residual temperature-independent resistance as well as the electron-phonon scattering, described by the Bloch-Grüneisen formula (dash-dot line). Dashed lines in the inset are guides for the eye.](image-url)
Magnon-polarons in van der Waals antiferromagnet FePS$_3$

Spin waves and lattice vibrations are two relevant excitations in magnetically ordered systems. The coupling between magnon and phonon modes, called magnon-polarons, is best evidenced with the observation of the repulsion of the otherwise bare phonon and magnon excitations when they are brought to coincide. Such anti-crossing, if sufficiently strong, may offer a new playground when manipulating the magnetically ordered systems with light.

Here we report the observation of magnon-polarons in the quasi-two-dimensional antiferromagnet FePS$_3$ probed with magneto-Raman scattering and far-infrared/terahertz (FIR/THz) magneto-transition measurements. The results of magneto-Raman scattering measurements are illustrated in figure 26, where at low magnetic fields, the very first effect of application of magnetic field results in the splitting of the magnon peak into two, $M_+$ and $M_-$ components.

FIG. 26. False-color map of the evolution of the low-temperature ($T = 4.2$ K) magneto-Raman scattering response of FePS$_3$ with an applied magnetic field oriented perpendicular to the plane of the layers, together with a few selected spectra, in the spectral region between 60 to 180 cm$^{-1}$. Coupling between the lower magnon branch $M_-$ and three $P_i$ ($i = 1 \ldots 3$) phonon modes is clearly observed. An additional $R_1$ phonon mode is activated at high magnetic fields.

The work highlights the effects observed at high magnetic fields, when the $M_-$ magnon branch is tuned in the spectral range of three low energy $P_1 = 1, 2, 3$-phonons. As can be seen in figure 26, the $M_-$ magnon excitation does not intersect any of $P_1$, $P_2$ and $P_3$ phonons and instead a characteristic pattern of the avoided crossing events is observed. A simple inspection of the raw data leads us to conclude that the $M_-$ magnon effectively couples to all three $P_1$, $P_2$ and $P_3$ phonons.

As shown in figure 27, our modelling reproduces the observed energy pattern of the avoided crossings of the $M_-$ magnon branch and three $P_1$, $P_2$ and $P_3$ phonons. The results demonstrate that several excitations among those traced with Raman scattering do also directly couple to light, giving rise to absorption resonances observed in FIR magneto-transmission measurements.

FIG. 27. Magnetic field dependence of the energy positions of hybrid magnon-phonon modes. Full circles represent the experimental data extracted from magneto-Raman scattering measurements. This data is reproduced with solid lines, following the theoretical modelling. Crosses account for the energy positions of transitions observed in FIR transmission spectra. Dashed lines show the field dependence of energy positions of magnon modes without coupling to phonons.

In conclusion, we have uncovered the efficient interaction between the magnon and selective phonon modes in bulk FePS$_3$. The hybrid magnon-phonon modes are efficiently traced with Raman scattering experiments but they also directly couple to light, raising the pronounced resonances in FIR transmission spectra. This can be expected to trigger further exploration of FePS$_3$, by probing the magnetization dynamics in this antiferromagnet with THz pulsed excitation, including the offered possibility to tune the strength of magnon-phonon with an applied magnetic field.

For more details please see [Vaclavkova et al., Physical Review B 104(13), 134437(2021)]
Semiconductors and Nanostructures
Electronic subbands in the $\alpha$-LaAlO$_3$/KTaO$_3$ interface revealed by quantum oscillations in high magnetic fields

Due to its fundamental complexity and potential applications in new-generation of semiconductor and spintronic devices, the two-dimensional electron gas (2DEG) formed at the interface between two insulating transition metal oxides has triggered significant attention in the past decade. The 2DEG at perovskite oxide interfaces with strong spin-orbit coupling was first discovered for LaAlO$_3$/SrTiO$_3$ (LAO/STO) heterointerface. 2DEG of higher mobility and stronger spin-orbit coupling with electrons originating from the Ta 5d orbitals has been revealed at interfaces based on KTaO$_3$ (KTO), a band insulator ($E_g \approx 3.6$ eV) and a polar oxide. Additionally, 2D-superconductivity with higher critical temperature has been reported at the KTO surface and KTO-based interfaces of high carrier densities ($n > 5 \times 10^{13}$ cm$^{-2}$). From the perspective of electronic band structure, the KTO-based 2DEG is similar to the STO-2DEG where electrons primarily occupy $t_2g$ orbitals of transition metal ions. Nevertheless, tight-binding calculations for the 2DEG at the KTO surface predict a strong mixing of $d_{x^2-y^2}$, $d_{xz}$, and $d_{yz}$ orbitals driven by spin-orbit coupling and a reconstruction of orbital symmetries of subbands.

To fully resolve the Shubnikov de Haas (SdH) oscillations corresponding to the closely spaced subbands of KTO-based 2DEGs, we have performed magneto-transport measurements on the amorphous $\alpha$-LAO/KTO interface in high magnetic fields ($B_{\text{max}} = 30$ T DC and 55 T pulsed) as shown in figure 28. Since the large lattice constant of KTO restricts the epitaxial growth of a hetero-interface, we create a conducting interface by growing an amorphous $\alpha$-LAO thin film on KTO. High-mobility carriers are induced at the interface using ionic-liquid (IL) gating at room temperature as reported before for $\alpha$-LAO/STO. The $\alpha$-LAO/KTO devices consist of 4 nm LAO thin film deposited on the patterned Hall bar on the surface of a 500$\mu$m-thick KTO(001) substrate using pulsed laser deposition. During deposition, the substrate temperature was kept at 25$^\circ$C and the oxygen partial pressure was $P_o \approx 10^{-4}$ mbar.

Simultaneous measurements of longitudinal and Hall resistance were carried out in high DC magnetic field ($B_{\text{max}} = 30$ T) and high pulsed magnetic field ($B_{\text{max}} = 55$ T with duration of 300 ms). The DC field measurements were performed in a $^3$He system and a dilution refrigerator, and the pulsed field measurements in a $^3$He system. For pulsed field measurements, we apply a dc current excitation of 5 $\mu$A, whereas we use a lower ac current of 100 nA for measurements in continuous fields. An in situ rotation mechanism was used to change the angle between the magnetic field orientation and the normal direction of the interface. Modulating the gate voltage applied on the IL gating the device switches from a high-conductance ($\approx 10^{-5}$ S) to a low-conductance ($\approx 10^{-10}$ S) state when $V_g$ sweeps from +2 V to -6 V.

The Shubnikov de Haas (SdH) oscillations show four frequencies corresponding to four 2D subbands with different effective masses in the range $0.20m_e < m^* < 0.55m_e$. Observation of multiple frequencies of 2D oscillations confirms the occupancy of multiple subbands with light and heavy electrons. However, additional SdH oscillations with an angle-independent frequency observed at high tilt angles indicate the coexistence of 3D charge carriers extending deep into the KTaO3.

The single-frequency oscillations originating from 3D electrons yields a larger effective mass of $\approx 0.70m_e$ corresponding to the heavy band. Overall, the inferred subbands are in good agreement with the theoretical calculations and angle-resolved photoemission spectroscopy studies on 2DEG at KTaO$_3$ surface.

![FIG. 28. Inverse field dependence of the second derivative of the longitudinal resistance ($-d^2R_{xx}/dB^2$) in (a) de field and (b) pulsed field applied perpendicular to the interface. Inset of panel (b) displays the longitudinal resistance as a function of magnetic field.](image-url)
Quasi-isotropic orbital magnetoresistance in lightly doped SrTiO$_3$

Magnetoresistance (MR) can be simply understood as a consequence of the Lorentz force exerted on mobile electrons by the magnetic field. This orbital magnetoresistance is largest when the magnetic field is perpendicular to the electrical current and expected to to saturate at high fields when $\mu_H B \approx 1$ where $\mu_H$ is the Hall mobility) for a closed Fermi surface. When the field and the current are parallel, we are in presence of longitudinal MR, expected to be negligibly small due to the cancelation of the Lorentz force. Here we report on the case of lightly doped SrTiO$_{3-x}$ which strongly deviate from this simple picture.

When the carrier density in SrTiO$_{3-\delta}$ is $n_H = 3 \times 10^{17}$ cm$^{-3}$, there is a single Fermi pocket at the center of the Brillouin zone (figure 29a). Nevertheless, upon the slope of a magnetic field of 54 T, there is a forty (twenty)-fold enhancement of resistance for the transverse (longitudinal) configuration (figure 29c). In both cases, the evolution with field is quasi-linear and there is no sign of saturation even if the high field regime ($\mu_H B >> 1$) is clearly attained. A polar plot of the normalised angular MR at a fixed magnetic field for another sample with a slightly lower carrier density ($n_H = 6.5 \times 10^{16}$ cm$^{-3}$) is shown in figure 29d). The magnetic field rotates from the transverse ($\theta = 0^\circ$) to the longitudinal ($\theta = 90^\circ$) configuration at two different temperatures. While at $T = 10$ K, the longitudinal magnetoresistance shrinks towards zero, at $T = 2$ K the magnetoresistance is quasi-isotropic and the relative direction of the magnetic field and the current injection barely affects its amplitude. Our doping and temperature study shows that this unusual regime of MR only occurs at low temperature (when resistivity is dominated by its elastic component), at low carrier concentration. It can be understood as a consequence of an isotropic field dependent mobility. The Hall mobility at a given magnetic field can be extracted using $\mu_H B = \sigma_{xy}/\sigma_{\perp} = \rho_{xy}/\rho_\perp$ (figure 29e). The deduced $\mu_H(B)$ can then be compared with the field dependence of the longitudinal conductance $\sigma_{//}$ (figure 29f).

Compared to zero-field counterparts, charge carriers following a cyclotron orbit are more vulnerable to shallow scattering centers. Such a picture has been invoked to explain the linear MR in 3D high mobility dilute semiconductors. One can invoke this picture to explain the quasi-linear transverse MR and field dependent mobility. However, the finite longitudinal MR remains unexplained, because only the plane perpendicular of the orientation to the magnetic field is concerned.

In a polar crystal, to which SrTiO$_3$ belongs, defects, by distorting the lattice, generate electric dipoles. The typical length for correlation between such dipoles is set by $R_c = v_s/\omega_0$ (where $v_s$ and $\omega_0$ are the sound velocity and the frequency of the soft optical mode, respectively). A possible solution to the mystery of the isotropic field reduction of the mobility is offered by this length scale, which does not depend on the orientation of magnetic field. In presence of randomly oriented mesoscopic dipoles, the charge current does not align locally parallel to its macroscopic orientation. Instead, it will meander along a trajectory set by dipoles’ electric field. The disorder affecting the whirling electrons will reduce mobility along different orientations. As the system is doped the distance between charge carriers decreases. When it becomes comparable with $R_c$ the charge carriers screened the dipolar moments and the longitudinal MR collapses in quantitative agreement with our observation.

For more details please see [Collignon, PRM, 5, 065002 (2021)].

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Brightening of dark excitons in 2D perovskites

2D perovskites constitute a hybrid material family obtained by dimensional reduction of the corresponding 3D crystal lattice. This is realized by incorporation of large organic molecules into the 3D crystal structure. The resulting organic-inorganic material adopts a new, layered structure often regarded as ideal quantum wells since they are not plagued by interface roughness or intermixing so characteristic for epitaxially grown quantum wells. Similar to fully inorganic quantum wells the thickness of the well can be altered, however, in 2D perovskites this is realized by changing the number of inorganic sheets $n$. Depending on the number of inorganic sheets $n$ 2D perovskites are considered as promising materials for light emitting applications $n \sim 1$ with outstanding quantum yield or as absorbers in light harvesting devices $(n \gg 1)$ with high efficiencies.

In 2D perovskites the large organic cations act as barriers providing a two-dimensional confinement for charge carriers but also serve as moderator of electrostatic forces between charges due to its different dielectric constant. Due to the high contrast of dielectric indices of organic/inorganic sublattices, the 2D perovskite materials are characterized by huge electron-hole binding energies with low effective Bohr radius. As a result, the excitonic effects can be readily observed even at room temperature. Furthermore, because of large binding energies, the exchange interaction between the electron and hole spins is greatly enhanced, exposing the exciton fine structure. The exciton fine structure results in the splitting of excitonic levels, and induces a splitting between the bright and dark states.

Optically inactive dark exciton states play an important role in light emission processes in semiconductors because they provide an efficient nonradiative recombination channel. Due to the lack of dipole-allowed recombination channel, the lifetime of dark excitons may extend above microseconds hence the dark state serves as an exciton reservoir. As a result, if the lowest excitation in the material system is the dark state. Understanding the exciton fine structure in materials with potential applications in light-emitting devices is therefore critical.

The energy structure of the band-edge excitons is qualitatively the same in 2D and 3D metal-halide perovskites [Yu et al., Sci. Rep. 6, 28576 (2016)]. We distinguish four band edge excitonic states which in the absence of exchange interaction are degenerate; the dark state ($\psi_1$), the excitonic state with out-of-plane dipole moment orientation ($\psi_2$, usually referred to as a gray exciton), and two excitonic states with in-plane dipole orientation ($\psi_3^-$ and $\psi_3^+$) which couple to left- and right-handed circularly polarized light. These excitonic states are built from s-like hole states and p-like electron states, each of them having total angular momentum $J_z/h = 1/2$. The $\psi_1$ with $J = 0$ is a spin-forbidden dark exciton state and the remaining three ($\psi_2$ and $\psi_{3\pm}$) are optically bright ($J = 1$) with different optical selection rules.

Studies of dark excitons are hindered by their absence in the optical spectra. However, the dark state is brightened in the presence of an external magnetic field, facilitating the observation of exciton fine structure. When the magnetic field is on, the intrinsic symmetry of the crystal is broken. The magnetic field applied in Voigt geometry mixes the in-plane excitonic states, $\psi_{3\pm}$ and $\psi_{3\pm}$, with the dark ($\psi_1$) and out-of-plane ($\psi_2$) excitonic states. With an increasing magnetic field the dark state gains oscillator strength and becomes observable in the optical spectra.

This situation is presented in Figure 30, where the absorption spectra is presented for selected magnetic field strengths. Clearly at the maximum magnetic field of 65 T an additional absorption signal is visible (labeled DX) at the low energy side of the bright exciton (BX) absorption. This new feature we identify as the magnetic field brightened (spin-forbidden) dark excitonic state. According to theoretical predictions, the absorption related to the brightened dark state shows a systematic increase of oscillator strength with increasing magnetic field.

For more details please see [Dyksik et al., Sci. Adv. 7, 1 (2021)].

![FIG. 30. Absorption spectra of (PEA)$_2$PbBr$_4$ measured in Voigt geometry. With increasing magnetic field strength an additional absorption signal (DX) at the low energy side of bright exciton (BX) is visible. This new feature is related to the brightened dark exciton.](image-url)
Bright and dark excitons in Mn-doped 2D Ruddlesden-Popper hybrid perovskites

Metal halide perovskites are of great interest largely due to their cheapness, ease of fabrication, and the wide range of optoelectronic applications such as solar cell and LEDs. The optoelectronic properties of lead-halide perovskites can be significantly altered by doping transition metal such as Mn-, Ni- and Cu-, leading to enhanced luminescence in Mn-, Ni- and Cu-doped nanocrystals, and modified growth, and solar cell performance in Mn-, Fe-, Co-, Ni-doped thin films. In this work, we investigate the magneto-optical properties of 2D Ruddlesden-Popper (RP) perovskites doped with manganese Mn:(PEA)$_2$PbI$_4$ (PEA = phenethylammonium, from now on Mn:PEPI and PEPI for the doped and undoped material, respectively). We extract the values of Landé $g$-factor and the diamagnetic coefficient. We also find that a dark exciton population isbrightened by state mixing with the bright excitons in the presence of a magnetic field, and the emission of the dark exciton in the magnetic field is significantly enhanced by doping PEPI with Mn.

In order to clarify the origin of the complex excitonic features of Mn:PEPI, low temperature circularly polarized transmission in high magnetic fields has been measured. Figure 31 shows the energy of the 1s exciton state shifts with the magnetic field. $E_+$ (red stars) and $E_-$ (black stars) correspond to $\sigma^+$ and $\sigma^-$ detection, respectively. Such excitonic spin split transition can be described by the following spin $s = 1/2$ Hamiltonian,

$$\Delta E_s = \alpha B^2 \pm \frac{1}{2} g\mu_B B,$$

where $\alpha$ is the diamagnetic coefficient, $g$ is the Landé $g$-factor, and $\mu_B$ is the Bohr magneton. Fitting the sum and the difference of the energies of the 1s exciton states allows us to extract the diamagnetic and Zeeman terms of the Hamiltonian (figure 31). We find a diamagnetic coefficient $\alpha = 0.338\mu eV/T^2$ and $g = 1.1$ for the Zeeman term. We notice that these values are quite close to the results for PEPI ($g = 0.96$ and $\alpha = 0.354\mu eV/T^2$).

In figure 32, we show magneto-photoluminescence properties of Mn:PEPI. At zero field, the bright exciton dominates the photoluminescence. With magnetic field applied in the Faraday geometry, the intensity of the bright exciton emission is continuously reduced with the increases field. A lower-lying peak dominates the PL spectrum at 14 T. We assign this peak to emission from the dark exciton, which the magnetic field mixes with the bright exciton state. Compared to zero field, the intensity of the dark exciton emission is approximately enhanced seven-fold at 14 T (figure 32 inset). It is worth noting that the increase in intensity of dark exciton emission is only four-fold at 14 T in PEPI, suggesting that the dark exciton emission is significantly enhanced in PEPI doped with Mn.

For more details please see [Neumann et al., Nature Communication 12, 3489 (2021)].

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Exciton fine structure of 2D CsPbBr$_3$-based nanoplatelets

Perovskite nanoplatelets are colloidal quantum wells, which consist of Pb-halide-based octahedra planes, surrounded by organic molecules. These, generally consisting of oleic acids, stabilize the stoichiometry of the quantum well material. These nanostructures have attracted a large interest in recent years due to their flexible chemical synthesis and the possibility of precisely controlling the number of octahedra planes which constitute the quantum well. Moreover, excellent optical properties such as high photoluminescence quantum yields and large exciton binding energy, make 2D perovskite nanoplatelets promising materials in lighting applications, LEDs or laser devices. For these applications, it is crucial to thoroughly understand the fine structure of the exciton manifold. In this work, we address this question by performing magneto-optical spectroscopy in pulsed magnetic field up to 65 T of CsPbBr$_3$-based perovskite nanoplatelets with a number of lead-halide octahedra planes ranging from 2 to 4. The magneto-optical measurements, performed in the Voigt configuration, allowed us to brighten the low energy dark exciton state. We thus extracted its energy separation with respect to the higher-energy bright state manifold.

In order to explore the fine structure of the exciton, we measured both magneto-photoluminescence (PL) and transmission in the Voigt configuration. Figure 34(a) shows the transmission and PL spectra of two monolayer thick (2ML) perovskite nanoplatelets at zero field and at 64.5 T, with the light polarized along to the magnetic field. In the zero field PL spectrum, we identify the weak, high energy peak as the recombination of the neutral exciton, only slightly Stokes-shifted with respect to the corresponding peak seen in the transmission spectrum. When the magnetic field is applied, the intensity of this peak decreases very rapidly, while a sharp, lower-lying peak dominates the PL spectrum at high magnetic field. We assigned this peak to the recombination of the originally dark exciton, which the magnetic field mixes with a bright exciton. We extracted the magnetic field dependence of the transition energies of both the dark and bright excitons, which we shown in figure 34(b). The black rectangles in figure 34(b) show the dependence of the emission energy of the dark exciton in magnetic field relative to the energy at zero field. For the bright branch, we took the absorption energy in magnetic field relative to the energy at zero field, shown as the red circles. We globally fitted both of dark and bright branches using,

$$E_{1,3L} = \frac{1}{2} \left( E_1 + E_3 \pm \sqrt{(E_1 - E_3)^2 + (g_L \mu_B B)^2} \right),$$

which allowed us to estimate the dark-bright splitting energies for different thicknesses, which we show in figure 34(c). As expected, the splitting energy increases with the decrease of thickness.

Figure 33(a-c) show the low temperature (4 K) absorption spectra of 2ML, 3ML and 4ML CsPbBr$_3$ nanoplatelets, respectively. Excitonic absorption peaks can be clearly observed in all these spectra. On the high energy side, the onset of the continuum absorption can be observed. To extract the exciton binding energy, we fit these absorption spectra using the 2D Elliott formula. A summary of the estimated exciton binding energy is shown in figure 33(d). As expected, the binding energy increases with decreasing number of lead-halide octahedra planes, which can be attributed to the stronger quantum and dielectric confinement.

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Revealing the full exciton fine structure in \((\text{PEA})_2\text{PbI}_4\) 2D perovskites

Two-dimensional perovskites, considered as an ideal quantum wells, with exciton binding energies significantly higher that their three-dimensional counterparts constitute very attractive playground for the exciton physics studies. Due to the low symmetry of their crystals lattice we can expect that the degeneracy (with respect to the angular momentum) of the excitonic states is completely lifted. Because of the enhanced exciton binding energy, the fine structure of excitons (resulting from exchange interaction between electrons and holes) should exhibit a significant state spacing. This not only provides an interesting opportunity for exciton fine structure studies, but can also have a dramatic impact on the performance of light emitters based on these 2D materials. Therefore, the understanding of the exciton fine structure is crucial from the potential application’s point of view.

![Figure 35](image1)

**FIG. 35.** Photoluminescence spectra in different magnetic fields for PEPI\(n = 1\). A low energy peak, attributed to the dark excitonic state appears in the spectra with increasing magnetic field. Dotted line represents a reflectivity spectrum at 0 T, showing the position of bright excitonic states approximately 15 meV above the dark state.

Fine structure in \((\text{PEA})_2(\text{MA})_{n-1}\text{PbI}_{3n+1}\) for \(n = 1\) (a compound with one layer of inorganic octahedra, abbreviated as PEPI) has been previously studied [Do et al., Nano Letters 20 5141 (2020)], however only bright states have been taken into consideration, leaving the excitonic picture incomplete. We examined the fine structure of excitons including the dark state for PEPI \(n = 1\) by using magnetic field, which induces transfer of the oscillator strength from bright states to the dark state, allowing its direct observation by optical methods.

We conducted a series of micro-photoluminescence measurements with applied magnetic field in Faraday configuration. The microscope objective used in this experiment has a high numerical aperture of 0.8. In this case this is crucial, because in the Faraday configuration, in the presence of a magnetic field, the dark state is coupled to the “out of plane” bright state, which emits photons in the plane of the quantum well, perpendicular to the detection axis [Yu et al., Scientific reports 6, 1 (2016)]. These measurements revealed the presence of an additional peak in the spectrum below bright excitonic states, absent at zero field conditions (figure 35). This feature is attributed to the dark excitonic state, gaining an oscillator strength with increasing magnetic field. This claim is supported by the fact that it’s intensity exhibits a quadratic dependence on the magnetic field (figure 36).

![Figure 36](image2)

**FIG. 36.** Dark exciton intensity as a function of the applied magnetic field for PEPI \(n = 1\). Solid line is a fit using \(I = I_0 + \alpha B^2\).

Results of the series of measurements for PEPI \(n = 1\) demonstrated the brightening of the dark state, unveiling its presence around 15 meV below the bright states (figure 35). In future we want to extend this investigation to structures with increasing thickness of the quantum well (\(n = 2, 3, 4\)). These studies can provide complete picture of the excitonic states in 2D perovskites which can help understand some of the unique properties of 2D perovskites.

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Landau level spectroscopy of the topological crystalline insulator Pb$_{1-x}$Sn$_x$Se

Lead-tin selenide Pb$_{1-x}$Sn$_x$Se exhibits an inverted bang-gap with conical 2D bands at the surface once the tin concentration exceeds $x \simeq 0.15$ at liquid helium temperatures. The material continues to be a subject of intense research, including the pursuit for its magneto-optical signature. We explored a series of Pb$_{1-x}$Sn$_x$Se samples using infrared magneto-spectroscopy in the Faraday configuration and observed a rich magneto-optical response arising from electronic excitations between pairs of bulk Landau levels.

The samples grown using the hot-wall-epitaxy method on a (111)-oriented BaF$_2$ substrate with the nominal tin content set at $x = 0.20, 0.25$ and 0.33. At $B > 2$ T, the transmission of particular samples becomes modulated by a series of excitations that follow a sub-linear dependence in $B$. These excitations may be straightforwardly identified as bulk inter-Landau level (inter-LL) transitions. We modelled those using a modified $4 \times 4$ band Hamiltonian [Liu et al., Phys. Rev. B 82, 045122 (2010)] which includes dispersive diagonal elements $Mq^2$. They account for the impact of remote bands as well as the band inversion. We then proceeded with the calculation of the dynamical magneto-conductivity that encodes the response in magneto-optical experiments.

We obtained very good agreement between our model and experiment data. The positions as well as the field dependence of all observed transitions are reproduced by using only three adjustable parameters $\Delta$, $M$ and $v$. We observe that the energy band gap ($E_g = 2\Delta$) increases roughly linearly with tin content. The same behaviour is observed for the absolute value of the inversion parameter $|M|$. To reproduce the measured magneto-transmission spectra accurately, the broadening parameter $\gamma$ was adjusted so that it increases linearly with the photon energy, $\gamma = \gamma_0 + \xi \hbar \omega$, where $\gamma_0 = 0.05$ meV and $\xi = 0.0125$. This latter choice resembles the empirical rule deduced for interband inter-LL excitations in graphene.

The shape of the conical band is given by the properties of the bulk velocity parameter. We have found no differences between longitudinal and oblique valleys. Therefore, we consider the velocity parameter $v$ to be isotropic in our samples. We applied high magnetic fields to our samples to drive surface electrons close to their quantum limit. At the same time, their fundamental CR mode is expected to move above the reststrahlen band of BaF$_2$. Hence, it should be traceable using infrared magneto-transmission technique (figure 37).

Despite expectations, and perhaps surprisingly for Pb-SnSe epilayers with a relatively high electronic quality, we do not find any clear signature of symmetry-protected surface states which is the most salient feature of topological crystalline insulators. Several phenomena might be at play; band-bending effect, surface disorder and hybridization between bulk and surface electronic states.


![FIG. 37. Magneto-transmission map with deduced and predicted electron transition spectra for the topological insulator Pb$_{1-x}$Sn$_x$Se with $x = 0.25$.](image-url)
Anomalous temperature dependence of effective mass in p-type Bi$_2$Te$_3$

Bi$_2$Te$_3$ is a layered narrow-gap semiconductor that has been extensively studied as an excellent thermoelectric material for energy conversion and cooling applications. It attracts even more interest as a three-dimensional topological insulators and as a candidate for spintronics applications.

An comprehensive infrared (IR) spectroscopic study is combined with transport properties on Bi$_2$Te$_3$ single crystals. The plasma edge measured in a broad temperature range (10 - 650 K) shows an anomalous non-monotonic shift. It decreases with rising temperature, passes through a minimum at room temperature and then it increases with temperature. Combining the IR and transport data such behavior is explained by the temperature dependence of the optical effective mass. We also study the influence of magnetic field on the IR properties of Bi$_2$Te$_3$ and Shubnikov-de Haas measurements. Both measurements provide coherent data with the low-temperature IR reflectivity.

![FIG. 38. (a) Energy scheme explaining the occupation of bands and their temperature dependence. (b) Temperature dependence of experimental DC conductivity and Hall coefficient (magnetic field 1 T, solid lines). The energy gap, $E_g = 0.13$ eV, is determined from the plot of the total carrier concentration versus 1/T. All quantities have log scales. The experimental DC conductivity is compared with static conductivity determined by fitting the reflectance spectra using the Drude model (solid blue symbols). Inset in panel (b) shows the temperature dependence of experimental mobility and its comparison with the fit $\mu_H \sim T^{-1}$.](image)

![FIG. 39. Temperature dependence of the relative optical effective mass ($m^*/m_0$) of charge carriers calculated from the plasma frequency and its damping.](image)

For more details please see [Železný et al., Phys. Rev. B 104, 165203 (2021)].

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Giant Seebeck effect across the field-induced metal-insulator transition of InAs

Lightly doped III-V semiconductor InAs is a dilute metal, which can be pushed beyond its extreme quantum limit upon the application of a modest magnetic field. We have conducted a study of its electrical and thermoelectrical properties, beyond its quantum limit \((B_{QL} = 4 \text{T})\) and up to a so-far unexplored range of temperature and magnetic field \([\text{Jaoui et al., NPJ Quantum Materials 5, 94 (2020)}]\). We report in figure 40 the two key results of this study.

Figure 40 shows the Seebeck effect \((S_{xx})\) across this MIT transition. At the lowest temperature \(-S_{xx}/T\) peaks at around 10 T. As the temperature increases the peak position is shifting to higher magnetic field while the amplitude of peaks is the largest at around 8 K. At this temperature \(S_{xx}\) is as large as 11.3 mV.K\(^{-1}\) comparable to the colossal thermopower of FeSb\(_2\) \([\text{Takahasi et al., Nature Comm. 7, 12732 (2016)}]\). Qualitatively the field dependence of \(S_{xx}\) is well captured by the Mott relation

\[
\frac{S_{xx}}{T} = \frac{\alpha^2 k_B^2}{3} e \frac{\partial \ln(\sigma(\epsilon))}{\partial \epsilon} |_{\epsilon=\epsilon_f}.
\]

This is in the region where \(\rho_{xx}\) and \(\rho_{xy}\) (and thus \(\sigma_{xx}\)) vary the most with both the magnetic field and temperature that \(S_{xx}\) is the largest. As a function of the magnetic field, it happens close to \(B_{MI}\) leading to a peak in the field dependence of \(S_{xx}\). However this relation fails to explain quantitatively the temperature and the two-hundred-fold enhancement of the Seebeck effect. Therefore another source of entropy has to be invoked such as the phonon bath. We find that the magnitude of this signal depends on sample dimensions and conclude that it is caused by phonon drag, resulting from a large difference between the scattering time of phonons (which are almost ballistic) and electrons (which are almost localized in the insulating state).

Our results show that the phonon drag effect is one road to boost the diffusive response of low carrier density metals across their field-induced MIT. Up to now this transition has been studied in a limited number of cases and the thermoelectric properties of dilute metals remains vastly unexplored. A large class of materials (ranging from well known doped semiconductors to new topological materials) remains to be studied, in particular at higher doping (and therefore at high magnetic field) where larger electron-phonon scattering rate can be attained, favoring even larger phonon drag effect.

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Giant increase of second harmonic light emission from wave-guiding core/shell ZnTe/ZnMgTe nanowires

Second harmonic generation (SHG) is a nonlinear optical process whereby photons with a frequency $\omega$ are converted into photons at twice the frequency, $2\omega$. A thorough understanding of the SHG efficiency in semiconducting nanowires (NWs) is not well established at present. In this work, we study the efficiency of SHG light emission intensity, $I_{\text{SHG}}$, from wave-guiding ZnTe/ZnMgTe core/shell NWs. As we tune the excitation wavelength from $\lambda_{\text{ex}} = 800 \, \text{nm}$ to $\lambda_{\text{ex}} = 1140 \, \text{nm}$, we observe a dramatic increase in $I_{\text{SHG}}$ by almost four orders of magnitude. We propose that the $I_{\text{SHG}}$ increase results from a decreased absorption of the second harmonic light as the exciton resonance is approached.

The nanowires were grown by molecular beam epitaxy applying a vapor-liquid-solid (VLS) mechanism (for details on the growth process and optical properties of the NWs. The morphology of the NWs was examined by a scanning electron microscope (SEM). Optical studies were performed at 4K with the sample mounted in a cold-finger helium cryostat. The low temperature allowed to study simultaneously SHG and emission of multiphoton luminescence (MPL). At room temperature only the SHG signal is observed while the MPL is quenched above about 100 K by a thermally activated non-radiative recombination process. In order to investigate single NWs, they were extracted from the as-grown sample by ultrasonication and drop-cast onto a Si wafer. A pulsed Ti:sapphire laser or frequency doubled optical parametric oscillator was employed as the excitation source. The beam was focused onto the sample surface with a microscope objective to a $\sim 2 \, \mu\text{m}$ spot. The excitation powers were on the order of tens of mW.

In figure 41(a) we show $I_{\text{SHG}}$ spectra excited at various wavelengths between 800 nm and 1140 nm. It is clearly seen that with increasing the excitation wavelength, $I_{\text{SHG}}$ exhibits a dramatic increase by almost a factor of $10^4$. In order to visualize this effect, in figure 41(b) the integrated second harmonic intensity and MPL intensity as a function of $\lambda_{\text{ex}}$ are shown. The highest SHG intensity occurs at $\lambda_{\text{ex}} = 1050 \, \text{nm}$. For $\lambda_{\text{ex}}$ higher than 1050 nm, $I_{\text{SHG}}$ intensity slightly decreases. On the other hand, the MPL intensity only fluctuates by a factor of $\sim 4$ over the relevant excitation range. To identify the origin of the $I_{\text{SHG}}$ enhancement, we compare the spectral dependence of $I_{\text{SHG}}$ with the one-photon excited PL spectrum presented in figure 41(b). We find that the exciton resonance occurs at an energy corresponding to $\sim 526 \, \text{nm}$. This value is very close to the wavelength corresponding to maximum SHG emission, which is observed at 525 nm. This comparison shows that SHG intensity enhancement is related to tuning of the excitation wavelength to resonance with the exciton energy in the NWs. We argue that the observed giant increase in the observed $I_{\text{SHG}}$ originates from a sum of three effects: (i) the absorption of second harmonic light by the NW bulk, (ii) the wave-guiding by the NW, and (iii) the leakage of light outside of the NW. We have also investigated the polarization anisotropy of $I_{\text{SHG}}$ and compare the results with a simple model taking into account different absorption cross sections for light parallel and perpendicular to the NW axis. We find that our results are substantially smaller than predicted by this model. The discrepancy is attributed to a bending of the NWs.

For more details please see [Szymura et al., Appl. Phys. Lett. 118, 192106 (2021)].
Evidence for nesting-driven charge density wave instabilities in the quasi-two-dimensional material LaAgSb₂

Charge density waves (CDWs) in solids are electronic ground states arising from an intrinsic instability of a metallic phase against a spatial modulation of the free carrier density, and are generally accompanied by a static distortion of the crystal lattice. They have originally been predicted and reported in low-dimensional metals, but since then they have been reported in a vast variety of compounds encompassing two-dimensional (2D) dichalcogenides, superconducting cuprates, or more recently in nickel pnictide superconductors. The existence of CDWs was proposed by Peierls who showed that a 1D chain is unstable due to a divergence of the static electronic susceptibility $\chi(q)$ at the wave vector $q = 2k_F$ that perfectly nests two parallel portions of the Fermi surface. Through electron-phonon coupling (EPC), the phonon spectrum softens at $2k_F$, ultimately resulting in a static distortion of the lattice as a mode’s energy vanishes. Unperfect nesting in real quasi-1D materials or in higher dimension compounds, on the other hand, rapidly suppresses the susceptibility divergence, and thereby invalidates the Peierls scenario in the vast majority of CDW materials. Even without resulting in a diverging electronic susceptibility, the presence of partial Fermi surface nesting in dimension $d > 1$ can enhance locally the EPC and eventually set the stage for the formation of a CDW. Depending on the system, alternative approaches have been proposed to account for the formation of CDWs, which encompass strong momentum or orbital dependence of the EPC (in combination with strong anharmonic effects), spin fluctuations, or exciton condensation.

Bulk LaAgSb₂ is a yet relatively unexplored compound of tetragonal structure (P4/nmm) that hosts two distinct CDWs with two critical temperatures of $T_{CDW1} = 207$ K and $T_{CDW2} = 186$ K as evidenced by x-ray diffraction, transport, thermal, and NMR studies. The two CDWs present at low temperature in LaAgSb₂ are aligned along the a and c axes, with a rather large real space periodicity of $\approx 17$ nm (CDW1, $\tau_1 \approx 0.026$ a*) and $\approx 6.5$ nm (CDW2, $\tau_2 \approx 1/6$ c*) and argued to be consistent with Fermi surface nesting. Interestingly, recent magnetotransport and angle-resolved photoemission spectroscopy (ARPES) investigations have indicated that electronic bands in the vicinity of the Fermi energy involved in the formation of CDW1 are of Dirac type, dispersing linearly as a function of momentum as in graphene, albeit with a band velocity twice smaller. The estimated nesting vector $\approx (0.09 \pm 0.04)\pi/a$, is large but relatively close to $\tau_1$. Ab initio calculations further suggest that CDW2 is also related to nested parts of the Fermi surface associated with a distinct electronic band. Time-resolved optical measurements revealed two low energy amplitude modes, suggesting that the CDW instability is triggered by the softening of a low-lying acoustic phonon mode. To date, however, the dispersion of the phonons in this system and their possible role in the formation of the two CDW states has not been investigated.

We have performed a series of temperature dependent diffuse scattering (DS) (see figure 42) and inelastic x-ray scattering (IXS) experiments in order to unveil the CDW formation mechanism in LaAgSb₂. The wave vectors of the CDW instability can be directly identified in the normal state DS intensity distribution, and IXS investigations confirm the soft-phonon driven nature of the CDW instabilities. Interestingly, we observe that the complex 3D momentum distribution of the DS intensity accurately follows that of the partial electronic susceptibility arising from the intraband contribution from the linearly dispersing electronic states. These states do not form a complete Dirac cone (as the dispersion is linear only along one k-space direction, and parabolic in the orthogonal direction), but their strong nesting locally enhances the EPC and yields the CDW formation. This provides a textbook example for the CDW formation in higher-dimensional materials and suggests possible routes for the design of such states through band structure engineering of metallic systems.

More information concerning this work can be found in [Bosak et al., Phys. Rev. Research 3, 033020 (2021)].

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High field ultrasound study of spin freezing in La$_{1.88}$Sr$_{0.12}$CuO$_4$

The coupling of electronic instabilities to the crystal lattice plays a significant role in shaping the phase diagram of some high-$T_c$ cuprate superconductors. The case of La-based cuprates is emblematic. Upon cooling, La$_{2-x}$Ba$_x$CuO$_4$ (LBCO) and rare-earth doped (Nd, Eu)$_x$La$_{2-x}$Sr$_x$CuO$_4$ ((Nd, Eu)-LSCO) evolve from a high-$T$ tetragonal (HTT) to a mid-$T$ orthorhombic (OMT) and finally to a low-$T$ tetragonal (LTT) crystal structure. The LTT order pins stripe order, a combination of mutually commensurate spin and charge modulations, initially found in Nd-LSCO. Within this context sound velocity and attenuation are particularly relevant quantities. Ultrasound measurements directly probe the lattice properties and they are sensitive to any strain dependent instability.

Among the La-based cuprate family La$_{2-x}$Sr$_x$CuO$_4$ (LSCO) appears peculiar. First, the OMT-LTT structural phase transition does not occur, although LTT-like distortions exist locally. Moreover, scattering evidence for charge ordering inside the pseudogap phase has remained elusive until recently. In LSCO around doping level $p = 0.12$, quasi-static charge modulation appears below $T_{CDW} = 70 \pm 15$ K with a maximal in-plane correlation length $\xi_0(T_c) \approx 30 \AA$, a value practically one order of magnitude smaller than in LBCO at the same doping.

In the same compound incommensurate antiferromagnetic (AFM) correlations are also found at low field for $0.02 \leq p \lesssim 0.135$. The temperature at which these correlations appear static depends upon the probe frequency, revealing the glassy nature of the magnetic state. However, as in other La-based compounds close to $p \approx 0.12$, one observes that the incommensurabilities of charge and spin density waves (respectively CDW and SDW) follow $\Delta \omega_{spin} = \delta_{charge}$, a relation reminiscent of charge-spin stripe ordering. Close to the hole doping level $p \approx 0.12$ elastic anomalies have been reported in both sound velocity and attenuation. Specifically, in single crystal studies and near the superconducting $T_c$, a broad sound velocity minimum has been observed in different acoustic modes. In a similar range of temperature, an attenuation maximum of longitudinal waves has been found in polycrystals. Different interpretations have been proposed to explain this peculiar behaviour such as magnetism, charge ordering, structural instabilities.

In this study, we show the anomalous sound velocity is caused by a coupling of the AFM glass to the lattice. Comparing ultrasound attenuation with NMR measurements on crystals from the same batch (see figure 43, we establish the link between the slowing down of magnetic fluctuations and the ultrasound anomalies observed in the $(c_{11} - c_{12})/2$ and $c_{11}$ elastic constants. Moreover, we show that the ultrasound properties of the $(c_{11} - c_{12})/2$ mode can be semi-quantitatively reproduced by a phenomenological dynamical susceptibility model initially developed for canonical spin glasses. Finally, by comparing different acoustic modes, we find that the spin freezing produces an enhanced susceptibility in the $B_{1g}$ channel, which is associated with nematicity in cuprates.

For more information please see [Frachet et al., Phys. Rev. B 103, 115133 (2021)].

![FIG. 43. Comparison between the ultrasound attenuation $\Delta \alpha$ (blue line, right scale) and the $^{139}$La NMR spin-lattice relaxation rate $1/T_1$ (green circle, left scale), at $\mu_0 H = 28$ T. NMR and ultrasound samples are from the same batch. Both physical quantities show a peak when the excitation frequency becomes equal to the frequency of the spin fluctuations. The dashed green line is a guide to the eye. $T_f$ ($T_a$) is the freezing temperature at the NMR (ultrasound) frequency.](image-url)
Normal state specific heat in the cuprates $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ and $\text{Bi}_{2+y}\text{Sr}_{2-x-y}\text{La}_x\text{CuO}_{6+\delta}$ near the critical point of the pseudogap phase

In the phase diagram of several organic, heavy-fermion or iron-based superconductors, superconductivity forms a dome around the quantum critical point, where an antiferromagnetic phase ends. The spin fluctuations associated with that quantum critical point are believed to cause both pairing and non-Fermi liquid behaviour. The thermodynamic signature associated with quantum criticality is a diverging electronic mass, which results in a logarithmic divergence of the electronic specific heat as $T \rightarrow 0$.

Specific-heat measurements in the normal state of Nd- and Eu-LSCO recently showed that the electronic contribution to the specific heat $C_e/T$, actually displays a pronounced peak as function of doping at $p \sim p^*$, the doping when the pseudogap ends. Moreover, for $p$ close to $p^*$, the electronic specific heat displays a logarithmic temperature dependence: $C_e/T = B\ln(T_0/T)$. Both behaviors are typical thermodynamic signatures of quantum criticality. It is important to investigate whether these characteristic features are also present, or not, in other cuprates. We, hence, report here a study of the temperature and doping dependence of the electronic specific heat $C_e$ in $\text{La}_{2-y}\text{Sr}_y\text{CuO}_4$ (LSCO) and $\text{Bi}_{2+y}\text{Sr}_{2-x-y}\text{La}_x\text{CuO}_{6+\delta}$ (Bi2201) single crystals.

As shown in figure 44, the specific heat of LSCO increases with the magnetic field in the mixed state and saturates at high fields, clearly indicating that the applied fields are large enough to suppress superconductivity (or are slightly below $H_{c2}(0)$ for $p \sim 0.22$ and 0.145). The field values above which $C/T$ saturates (at 2 K) are in very reasonable agreement with the $H_{c2}(0)$ values inferred from earlier transport measurements (shaded (colored) boxes in figure 44). Moreover, at higher temperatures, the crossover from the superconducting to the normal state shows up as a broad maximum in the field and/or temperature dependence of $C/T$. This smeared maximum (still visible at 2 K) for $p \sim 0.24$ is then reminiscent of the former specific jump occurring at the upper critical field, indicating the locus in the $H−T$ phase diagram where most of the change in the superconducting ordering energy occurs. All measurements were performed above this bump. By applying magnetic fields up to 35 T, we were, hence, able to determine the specific heat in the normal state for $p$ values as close as possible to $p^*$.

As seen in figure 44, large $C_e/T$ values (on the order of 15 mJ mol$^{-1}$ K$^{-2}$ at 2 K) are measured in the vicinity of the onset of the pseudogap phase at $p^* \sim 0.19$. However, in contrast to previous measurements in Nd/Eu-LSCO, $C_e/T$ remains large over an extended doping range, confirming the former indication for the presence of a broad maximum in the doping dependence of $C_e/T$ observed in Zn substituted samples.

Despite the presence of a large (hyperfine) Schottky contribution, we also show that a $B\ln(T_0/T)$ contribution has to be introduced in order to fit the temperature dependence of $C_e/T$. Similarly, a clear $B\ln(T_0/T)$ contribution to $C_e/T$ and concomitant large $C_e/T$ values (on the order of 13 J mol$^{-1}$ K$^{-2}$ at 0.65 K) are observed in Bi2201 for $p \sim p^*$, hence, confirming the universality of these features. Very similar values of the $B$ coefficient are observed in all compounds close to $p^*$.

For more information please see [Girod et al., Phys. Rev. B 103, 214506 (2021)].

FIG. 44. Magnetic-field dependence of the specific heat at $T \sim 2$ K in $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ after subtraction of the phonon contribution $C_{ph}$. The small overshoot observed for $x = p \sim 0.24$ is most probably reminiscent of the superconducting transition in presence of strong fluctuations. The shaded (colored) boxes indicate the locus of the previously estimated upper critical field $H_{c2}(0)$ values. Open circles are low-field data extracted from the literature. As seen, $(C − C_{ph})/T$ increases with $p$, reaching ~ 15 to 16 mJ mol$^{-1}$ K$^{-2}$ in the normal state for $p \sim 0.22 − 0.25$.

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Comparison of two superconducting phases induced by a magnetic field in UTe$_2$

Superconductivity induced by a magnetic field near metamagnetism is a striking manifestation of magnetically-mediated superconducting pairing. After being observed in itinerant ferromagnets, this phenomenon was recently reported in the orthorhombic paramagnet UTe$_2$. Here we have explored the phase diagram of UTe$_2$ (figure 45) under two magnetic-field directions: the hard magnetization axis $b$, and a direction tilted by $\approx 27^\circ$ from $b$ in the $(b,c)$ plane.

Zero-resistivity measurements confirm that superconductivity is established beyond the metamagnetic field $H_m$ in the tilted-field direction. We extract the full magnetic-field-temperature phase diagrams of UTe$_2$ for the two field directions. While superconductivity is locked exactly at fields either smaller (for $H \parallel b$), or larger (for $H$ tilted by $\approx 27^\circ$ from $b$ to $c$), than $H_m$, the variations of the Fermi-liquid coefficient in the electrical resistivity and of the residual resistivity are similar for the two field directions. From a Fermi-liquid analysis we also determine the field dependence of the residual resistivity and estimate the variation of the effective mass $m^*$. These quantities show striking similarities for the two field-directions in contrast with the very different superconducting phase diagrams. The resemblance of the normal states for the two field directions puts constraints for theoretical models of superconductivity and implies that some subtle ingredients must be in play.

For more details please see [Knafo et al., Communications Physics 4, 40 (2021)].

![Figure 45](http://example.com/figure45.png)

**FIG. 45.** Low-temperature electrical resistivity of UTe$_2$ versus magnetic field (a) for $H \parallel b$ and (b) for $H$ tilted by $\approx 27^\circ$ from $b$ to $c$. Magnetic-field-temperature phase diagram of UTe$_2$ (c) for $H \parallel b$ and (d) for $H$ tilted by $\approx 27^\circ$ from $b$ to $c$.
Transport signatures of the pseudogap critical point in the cuprate superconductor Bi$_2$Sr$_{2−x}$La$_x$CuO$_{6+δ}$

The fundamental nature of the pseudogap state and its critical point $p^*$ in cuprate superconductors remains the most puzzling piece of their phase diagram. Transport measurements achieved in magnetic fields high enough to suppress superconductivity and access the normal state down to $T \simeq 0$ in La$_{1.6−x}$Nd$_{0.4}$Sr$_x$CuO$_4$ (Nd-LSCO) revealed major insights on the electronic state of this pseudogap phase. First there is a sharp drop in carrier density at the doping $p^*$ where the pseudogap phase ends as revealed by the Hall number going from $n_H \simeq 1 + p$ above $p^*$ to $n_H \simeq p$ below $p^*$.

Secondly, the Wiedemann-Franz law is satisfied in the normal state of the pseudogap phase, supporting that the underlying ground state is metallic. The third signature, emblematic of quantum criticality, is a $T$-linear resistivity down to $T \to 0$. Moreover, the scattering rate corresponding to the linear regime is consistent with the Planckian limit $h/\tau \simeq k_B T$. On top of the linear resistivity, logarithmically diverging Seebeck coefficient $S/T \propto \log(1/T)$ as $T \to 0$ was measured close to $p^*$, adding to the typical signatures of quantum criticality observed near the end of the pseudogap phase. Finally, a large thermal Hall conductivity $\kappa_{xy}$ was observed inside the pseudogap phase, interpreted as phonons acquiring chirality as they enter this phase.

In this study, we revisit these five transport coefficients (resistivity, Hall, thermal conductivity, Seebeck and thermal Hall conductivity) in a completely different material in order to establish the universality of these signatures. We probe two overdoped Bi$_2$Sr$_{2−x}$La$_x$CuO$_{6+δ}$ single crystals with $T_c=18$ K ($x=0.2$) and $T_c=10$ K ($x=0$), very close to the pseudogap critical point ($T_c=8$ K according to NMR measurements). Resistivity and Hall measurements were performed under a pulsed field up to $H = 66$ T and under static field up to $H = 33$ T. Thermoelectricity was probed under a static field up to $H = 34$ T. Thermal coefficients were studied in a dilution cryostat down to 80 mK with an applied field up to $H = 15$ T.

These experiments allowed suppressing superconductivity down to low temperature leading to the findings of (i) a drop in the carrier density from the most to the less overdoped sample (figure 46(b)), (ii) the satisfaction of the Wiedemann-Franz law in both dopings, (iii) a quasi-linear resistivity for the doping closer to $p^*$, along with a slope that reaches the value given by Planckian dissipation, (iv) a diverging in temperature Seebeck coefficient (figure 46(d)) and finally (v) the thermal Hall effect becoming large and negative at low temperature. The observation of all these phenomena in a completely different material than Nd-LSCO consists in a key argument to the universality of these signatures of the pseudogap critical point.

For more details please see [Lizaire et al., Phys. Rev. B 104, 014515 (2021)]

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Locally commensurate charge-density wave in YBa$_2$Cu$_3$O$_y$

In recent years, charge order, a periodic modulation of the charge density and lattice positions, called charge-density wave (CDW), has been shown to be a universal property of hole and electron-doped cuprates, with possible connections to the pseudogap and superconducting phases. However, several fundamental questions remain unanswered such as why there are different CDW phases (with or without long-range order, with or without intertwined magnetic order) or what sets the CDW wave vector (and the associated periodicity in real space). More generally, the knowledge of the spatial profile of the charge modulation, which includes its periodicity but also its intra-unit-cell structure and the possible presence of topological defects or discommensurations, is important for understanding quantum oscillations, the coexistence of the CDW with superconductivity or the coupling between spin and charge degrees of freedom. For instance, the latter may depend on whether the CDW periodicity is odd or even, given that CuO$_2$ planes constitute a bipartite lattice of antiferromagnetic moments.

In this work [Vinograd et al., Nature Communication 12, 3274 (2021)], we performed detailed analysis and computer simulations of $^{17}$O and $^{63}$Cu NMR lineshapes in the long-range CDW phase of YBa$_2$Cu$_3$O$_y$ (YBCO), as observed by quenching superconductivity in high magnetic fields. Essentially, we find that all features of the lineshapes, that is, the presence of two peaks as well as their relative areas and positions (figure 47) are consistent with a 1D sinusoidal modulation of the charge density having a period three and phase 0°, over a broad range of doping and magnetic field values.

This result shows that the CDW is unidirectional with a commensurate period of three unit cells at the local scale probed by NMR. This implies that the incommensurate period found in X-ray scattering arises from the presence of phase slips (discommensurations). A regular array of at most three phase slips of $-2\pi/3$ per 100 unit cells is necessary to make a modulation locally commensurate with the lattice.

Taken together with recent scanning tunneling microscopy results in Bi2212 cuprates [Mesoros et al., Proc. Nat. Acad. Sci. 113, 12661 (2016)], our work shows that the spatial profile of the CDW generically results from the interplay between an incommensurate tendency at long length scales, possibly related to properties of the Fermi surface, and local commensuration effects, due to electron-electron interactions or lock-in to the lattice. Our observation of a period-3 contradiction the hypothesis of a universal period 4 and is most naturally explained by a locking of the CDW to the lattice.

Additionally, our analysis of the NMR spectra reveals that the CDW primarily modulates the oxygen, rather than copper, hole density, with the O sites in orthogonal directions being most likely in anti-phase ($d$ symmetry form factor).

At the broad level, these results provide suggestive evidence that superconducting cuprates are generically unstable towards the formation of a unidirectional, O-centered and locally commensurate CDW whose global periodicity is nonetheless incommensurate.

It turns out that the high-field phase of YBCO studied here offers a unique and pristine realisation of this CDW phase, whose observation is in general (in other cuprates as well as in YBCO at low fields) complicated by the conspiracy of disorder, magnetic ordering and competition with superconductivity.

FIG. 47. $^{17}$O NMR lineshape (second high-frequency satellite of O(2) sites lying in bonds oriented along the crystallographic a axis) in YBa$_2$Cu$_3$O$_{6.56}$ at $T = 2$ K and $B_z = 27.1$ T. The line shape is described by two peaks $O_A$ and $O_B$ and is fitted with asymmetric line shapes of equal widths, resulting in an area ratio of $1.95 \pm 0.06$ for the two peaks. Outside the CDW phase ($T \geq 60$ K), there is only a single, symmetric line. The asymmetry of the individual lines arises from an additional distribution of Knight shifts analysed in an earlier publication.

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Interplay between strong correlations and electronic topology in the underlying kagome lattice of Na$_{2/3}$CoO$_2$

Electronic topology in metallic kagome compounds is under intense scrutiny. Local Density Approximation (LDA) calculations in weakly correlated kagome lattices show the presence of Dirac or Weyl points as well as the presence of flat electronic bands. In compounds with 3d elements these states are expected to evolve due to strong electron correlations.

We present the study of the transport properties of the sodium cobaltate Na$_x$CoO$_2$. This compound has a layered structure with Co ions ordered on a triangular lattice (inset of figure 48). In earlier NMR/NQR experiments it was shown that the Na ions located between the CoO$_2$ layers display distinct orderings depending on the Na content, and the ionic order induces a charge disproportionation of the Co sites. In the case of the $x = 2/3$ phase the group of Co ions in Co1 positions are not involved in transport due to filled states, while the second Co2 group forms a kagome sub-lattice having delocalized charge carriers (inset of figure 48). The LDA+U computations demonstrate that a large coulomb interaction $U$ is required to induce that kind of disproportionation of the Co sites. Existence of the underlying well-conductive kagome sub-lattice and a large $U$ make the Na$_{2/3}$CoO$_2$ a model system to study the topological states of the kagome systems in the presence of strong electron interactions.

Transport data was taken on a series of distinct samples. The small residual resistivity ($\rho \approx 2 \mu\Omega \cdot cm$) measured in our single crystals proves the low level of disorder for this $x = 2/3$ phase. Experiments have revealed intriguing properties. The $ab$-plane magnetoresistance (MR) switches from positive, with an initial linear $B$ dependence at low $T$, towards a negative $B^2$ behavior at high $T$ (figure 48). At low temperature the Hall effect switches from negative value to positive for fields $B > 30$ T while beyond $T = 5$ K it remains negative for all fields. This gives evidence for a multiband character of the Fermi surface at all fields and temperatures. We established the coexistence of light and heavy carriers. At low $T$ the dominant light carrier conductivity at $B = 0$ is suppressed by a $B$-linear MR suggesting Dirac like quasiparticles. Lifshitz transitions induced at large magnetic field $B > 20$ T and $T > 5$ K unveil the carriers having lower mobility. These carriers display negative $B^2$ MR due to scattering from magnetic moments likely pertaining to a flat band. We underline an analogy with heavy Fermion physics.

Intense magnetic fields allowed us to separate hole and electron charge carriers with markedly different mobilities, but we cannot unambiguously explain at this stage all the features of the band structure. Further work is needed to determine if there is a phase shift of Shubnikov - de Haas oscillations (SdH) due to existence of the Berry phase, which is predicted for kagome systems. We also need to study the field dependence of the MR well above 30 T that is in the range where the Hall effect becomes positive. If a simple two band model applies above 30 T that might allow to separate fully the heavy carriers from the light carrier contributions and then to dispose of serious guidelines for the understanding of the band structure. Such studies should help us to assign the distinct carriers to the theoretically expected Dirac cone bands and flat bands. They will also allow us to associate the detected SdH oscillations to specific orbits on the Fermi Surface. Such a thorough characterization of the Fermi Surface pockets is essential to build theoretical models of the band structure and to understand its incidence on the electronic properties.

For more details please see [Gilmutdinov et al., Phys. Rev. B 104, L201103 (2021)]

\[ \text{FIG. 48. } \rho(B) \text{ curves at different temperatures showing magnetoresistance switch from positive } B\text{-linear dependence at low } T, \text{ towards a negative } B^2 \text{ behavior at high } T. \]

\[ \text{In the inset - 2D arrangement of the two types of sites in the Co plane. The Co1a and Co1b (yellow and brown) are non-magnetic Co}^{3+} \text{ sites. The 3d orbitals of the Co2a and Co2b sites (light and dark blue) arranged in a kagome sub-lattice are both nearly identically involved at the Fermi level.} \]

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**I.F. Gilmutdinov, D. Vignolles, C. Proust**

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Robust Fermi-surface topology of CeRhIn$_5$ in high magnetic fields

Rare-earth-based materials are now widely recognized as an ideal playground for exploration of the fascinating physics that develops around a quantum critical point (QCP), a second-order phase transition at zero temperature. In Ce-based compounds, such a QCP can be induced by hydrostatic pressure, chemical doping, or magnetic field, where it typically separates an antiferromagnetic (AFM) state from a nonmagnetic ground state. In spite of numerous experimental investigations of such systems in the vicinity of a QCP, the details of what drives the QCP remain the subject of much theoretical debate.

CeRhIn$_5$ is one of the best-studied heavy-fermion (HF) materials. This tetragonal AFM compound with $T_N = 3.8$ K can be tuned to a QCP by pressure, chemical substitution, and magnetic field. Several de Haas-van Alphen (dHvA) experiments evidence that the $f$ electrons of CeRhIn$_5$ are localized at ambient pressure. As the critical pressure for the suppression of antiferromagnetism, $P_c = 2.3$ GPa, is reached, all dHvA frequencies observed at $P < P_c$ change discontinuously, signaling an abrupt Fermi-surface (FS) reconstruction as a consequence of the $f$-electron delocalization. A similar discontinuous change of the dHvA frequencies was observed upon substituting Rh by Co in CeRh$_{1-x}$Co$_x$In$_5$. However, the FS reconstruction does not occur at the critical concentration $x_c \approx 0.8$, where the AFM order is suppressed, but deep inside the AFM state at $x \approx 0.4$, where the AFM order alters its character and superconductivity emerges to coexist with antiferromagnetism.

Recent results obtained at high magnetic fields suggested a unique behavior in CeRhIn$_5$. A field-induced QCP was reported to occur at the critical field $B_c \approx 50$ T applied along both the $c$ and $a$ axes. Furthermore, an electronic-nematic phase transition was observed at $B^*$, and attributed to an in-plane symmetry breaking. Finally, the emergence of additional dHvA frequencies was observed at $B^*$, and interpreted as a field-induced FS reconstruction associated with the $f$-electron delocalization. This result is surprising given that magnetic fields are generally expected to localize $f$ electrons.

To resolve this controversial issue, we performed a comprehensive dHvA study of CeRhIn$_5$ using both static (up to 36 T) and pulsed (up to 70 T) magnetic fields. Several dHvA frequencies gradually emerge at high fields as a result of magnetic breakdown, as shown in figure 49. Among them is the thermodynamically important $\beta_1$ branch, which has not been observed so far. Comparison of our angle-dependent dHvA spectra with those of the non-$4f$ compound LaRhIn$_5$ and with band-structure calculations evidences that the Ce $4f$ electrons in CeRhIn$_5$ remain localized over the whole field range. This rules out any significant Fermi-surface reconstruction, either at the suggested nematic phase transition at $B^* \approx 30$ T or at the putative quantum critical point at $B_c \approx 50$ T. Our results [Mishra et al., Phys. Rev. Lett. 126, 016403 (2021)] rather demonstrate the robustness of the Fermi surface and the localized nature of the $f$ electrons inside and outside of the antiferromagnetic phase.

It was previously reported that the $f$ electrons also remain localized in CeIn$_3$ above its critical field $B_c \approx 60$ T. Whereas CeIn$_3$ is an isotropic HF compound with an almost spheroidal FS, CeRhIn$_5$ is a prototypical example of a strongly anisotropic material with quasi-2D FSs. The continued localization of the $f$ electrons well above $B_c$ in both compounds is not consistent with either of the two existing theoretical models of AFM QCPs. This implies that magnetic field, which itself tends to localize $f$ electrons, should be treated differently from such control parameters as pressure or chemical doping.

FIG. 49. FFT spectra of the static-field dHvA oscillations in CeRhIn$_5$. The inset shows the evolution of the dHvA frequencies with field obtained from pulsed (circles) and static (triangles) field measurements.

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CeRhIn$_5$ is one of the best-studied heavy-fermion compounds. It crystallizes in the tetragonal HoCoGa$_5$ structure (space group $P4/nmm$), which can be viewed as a stack of alternating layers of CeIn$_3$ and RhIn$_2$ along the $c$ axis. The electronic specific heat coefficient, $\gamma \approx 400 \text{mJ/K}^2\text{mol}$, makes CeRhIn$_5$ a moderate-heavy-fermion material. At ambient pressure and zero magnetic field, it undergoes an antiferromagnetic (AFM) transition at $T_N = 3.8\,\text{K}$. Within the AFM phase, the Ce moments are antiferromagnetically aligned within the CeIn$_3$ planes. The moments spiral transversally along the $c$ axis with a propagation vector $\mathbf{Q} = (0.5, 0.5, 0.297)$ incommensurate with the crystal lattice.

When a magnetic field is applied along the $c$ axis, $T_N$ monotonically decreases until it is completely suppressed at $B_c \sim 50\,\text{T}$. Surprisingly, the critical field for this orientation is approximately the same as along the $a$ axis in spite of a considerable crystallographic and magnetic anisotropy of CeRhIn$_5$. On the other hand, the critical field was extrapolated from specific heat measurements in pulsed magnetic fields, while there is a difference between the results obtained in pulsed and static fields.

The most interesting feature was observed in various measurements at $B^* \simeq 30\,\text{T}$ for field applied either along or slightly tilted from the $c$ axis. While it was interpreted as a transition into an electronic-nematic state, the exact origin and nature of this anomaly is still under debate. Surprisingly, specific heat measurements have so far failed showing a direct indication of this anomaly. It is thus still unclear whether the anomaly corresponds to a real thermodynamic phase transition or a crossover.

To address this issue, we performed high-field low-temperature specific heat measurements on a single crystal of CeRhIn$_5$ in static fields up to $35\,\text{T}$ applied along the $c$ axis. For this orientation, we observed a weak but distinct anomaly at $B^*$, as shown in the inset of figure 50.

The presence of the specific heat anomaly at $B^*$ implies that it is likely a real thermodynamic phase transition rather than a crossover, contrary to what was previously suggested. The latter suggestion, however, was based on magnetostriction measurements performed in a magnetic field applied at $20^\circ$ from the $c$ axis. Furthermore, the anomaly we observe at $B^*$ does not have the characteristic $A$-like shape of a second-order phase transition. Therefore, the anomaly observed at $B^*$ most likely corresponds to a first-order phase transition. Moreover, it is thermodynamically forbidden that three second-order phase boundary lines meet at a triple point. This further supports our hypothesis that $B^*$ is a first-order phase transition. Finally, the anomaly at $B^*$ is observed only within the AFM state, in agreement with previous reports. Based on this, the most natural explanation of the phase transition at $B^*$ is a change of magnetic structure. Previous high-field NMR measurements unambiguously suggest that the AFM phases both below and above $B^*$ are incommensurate. Therefore, $B^*$ should correspond to a transition from one incommensurate phase, AFM1, to another phase incommensurate along the $c$ axis, AFM4, with a propagation vector $\mathbf{Q} = (0.5, 0.5, l)$, where $l$ is different from 0.297 of the AFM1 phase. This hypothesis is consistent with previous reports. Indeed, the previously observed resistivity jump at $B^*$ can be naturally accounted for by a metamagnetic spin-reorientation, as we suggest here.

Figure 50 shows the revised magnetic phase diagram of CeRhIn$_5$ for field applied along the $c$ axis. The field dependence of $T_N$ obtained from our static-field measurements is consistent with that previously reported, based on the pulsed-field data. The transition at $B^*$ is weakly temperature-dependent in agreement with previous measurements. As was already discussed above, we suggest that this first-order transition separates two different incommensurate magnetic phases [Mishra et al., Phys. Rev. B 103, 045110 (2021)].

**FIG. 50.** Magnetic phase diagram of CeRhIn$_5$ for the field applied along the $c$ axis established from relaxation (circles) and AC (triangles) specific heat measurements. Closed symbols correspond to second-order transitions from AFM to PM phase. Open symbols indicate the anomaly at $B^*$, which presumably corresponds to a weakly first-order transition. The inset shows an example of the specific heat curve measured at $T = 2.42\,\text{K}$ for a magnetic field applied along the $c$ axis.
The heavy-fermion compound CeRhIn$_5$ undergoes an antiferromagnetic (AFM) transition at $T_N = 3.8$ K. Within the AFM phase, the Ce moments are antiferromagnetically aligned within the CeIn$_3$ planes. The moments spiral transversally along the $c$ axis with a propagation vector $Q = (0, 0.5, 0.297)$ incommensurate with the crystal lattice.

**Phase diagram of CeRhIn$_5$ for field applied along the $a$ axis**

Figure 51(a) shows specific heat divided by temperature, $C/T$, obtained from relaxation measurements for a magnetic field applied along the $a$ axis. For this field orientation, apart from the AFM transition at $T_N$, there are two additional field-induced transitions at $T_1$ and $T_2$, as shown in figure 51(b). The transition at $T_1$ manifests itself by a sharp $\delta$-like peak characteristic of a first-order transition. The transition at $T_2$ appears as a $\lambda$-type anomaly typical for a second-order transition. This transition is observed only at low fields, as shown in figure 51(b). In agreement with previous reports, $T_N$ initially increases up to about 10 T, and then decreases monotonically up to the highest field of our measurements. The transition temperature $T_1$ shows a similar trend. Above 3 T, $T_1$ increases up to about 12 T, and then starts to decrease. Its suppression rate, however, is slower than that of $T_N$. With increasing field, the two transitions approach each other. At 28 T, the two transitions are barely distinguishable, and at 30 T only the transition at $T_N$ remains, as shown in figure 51(c). All the transitions are also observed in measurements using the AC technique [Mishra et al., Phys. Rev. B 103, 045110 (2021)].

The resulting $B$ – $T$ phase diagram for field along the $a$ axis is shown in figure 52. It contains three different antiferromagnetic phases labeled AFM1, AFM2, and AFM3. The magnetic structure of all three phases was previously determined by neutron diffraction. All three phases meet at a triple point inside the AFM phase at ($3$ T, 3.4 K). The AFM2 phase exists only in a narrow temperature range close to $T_N$. This range shrinks with increasing magnetic field until the AFM2 phase is completely suppressed at $\sim 30$ T, giving rise to yet another triple point. Above 30 T, only the commensurate phase AFM3 exists up to the complete suppression of the AFM order.

**FIG. 51.** Specific heat divided by temperature, $C/T$, of CeRhIn$_5$ for a magnetic field applied along the $a$ axis. (a) $C/T$ as a function of $T$ obtained from relaxation technique for several values of a magnetic field. Curves are vertically shifted according to the magnetic field scale shown in the right axis. A zoom at low and high fields is shown in (b) and (c), respectively.

**FIG. 52.** Magnetic phase diagram of CeRhIn$_5$, obtained from relaxation (circles) and AC (triangles) specific heat measurements for a field applied along the $a$ axis. Closed and open symbols correspond to second- and first-order transitions, respectively.
Angular-dependent magnetic phase diagram of CeRhIn$_5$

CeRhIn$_5$, although discovered barely two decades ago, is one of the best studied heavy-fermion materials. It undergoes an antiferromagnetic (AFM) transition at $T_N = 3.8$ K. The zero-field magnetic structure is commensurate. At low temperatures, when a magnetic field $B$ is applied in the basal plane, a first-order transition occurs at $B_m \simeq 2$ T. The transition corresponds to a change of magnetic structure from incommensurate to commensurate. When the magnetic field is tilted away from the basal plane, $B_m$ shifts to higher fields and follows the $1/\cos \alpha$ dependence (where $\alpha$ is the field angle from the basal plane) up to about $75^\circ$ – $80^\circ$. At higher angles, however, $B_m$ deviates from a $1/\cos \alpha$ dependence towards lower fields.

The exact origin of the transition at $B^*$, especially in bulk crystals, remains, therefore, an important open question. To shed more light on the origin of $B^*$, we performed angular-dependent ultrasound-velocity measurements in high magnetic fields [Mishra et al., Phys. Rev. B 103, 165124 (2021)]. The angular dependence of the transition fields $B_m$ and $B^*$ was obtained from measurements of the $C_T$ mode, where both anomalies are most pronounced.

The field dependence of the ultrasound-velocity variation, $\Delta v/v$, for different angles $\theta$ from [001] towards the [110] direction is shown in figure 53(a). At $2^\circ$, both anomalies manifest themselves as sharp steps (figure 53(b)). With increasing angle, both features progressively become smaller. While the anomaly at $B_m$ remains sharp, the one at $B^*$ broadens with increasing angle. This probably implies that the transition at $B^*$ at small angles changes to a crossover at larger angles. The anomaly at $B_m$ is easily traceable all the way up to $40^\circ$, while its counterpart at $B^*$ is still present at $30^\circ$, but is no longer visible at $40^\circ$, where the curve is completely dominated by strong low-frequency quantum oscillations (figure 53(c)). The angular dependence of the anomaly at $B^*$ is strikingly different from that observed in resistivity measurements on FIB-fabricated microdevies. The difference is probably due to uniaxial stresses or strains inevitably present in FIB-fabricated devices.

The resulting angular dependence of both $B_m$ and $B^*$ is shown in figure 54. In agreement with previous results, $B_m$ is strongly angle dependent. It is well fitted by $1/\cos(90^\circ - \theta)$ down to about $10^\circ$ from the $c$ axis but deviates at lower angles. $B^*$, on the other hand, is only weakly angle dependent. It increases approximately linearly all the way from $2^\circ$ to about $30^\circ$, above which it either disappears or shifts to beyond $36^\circ$ T. The remarkable difference between the two angular dependencies is probably due to different magnetic moment arrangements in the vicinity of the two transitions.

Recent results obtained in high magnetic fields applied along or close to the $c$ axis suggest a remarkably novel behavior in CeRhIn$_5$. The AFM order is suppressed at $B_c \approx 50$ T giving rise to a field-induced quantum critical point. Furthermore, a hysteretic jump in the in-plane resistivity of CeRhIn$_5$ microstructures fabricated by focused ion beam (FIB) was observed at $B^* \approx 30$ T. Later on, these experiments were advanced, allowing for simultaneous measurements of the in-plane resistivity along the [100] and [010], as well as along the [110] and [1T0] directions. These measurements revealed a strong in-plane resistivity anisotropy, which emerges above $B^* \approx 30$ T. This electronic anisotropy was interpreted in terms of an electronic-nematic transition. To the best of our knowledge, these results, however, have not been reproduced on bulk samples so far.

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Magnetic Systems
Revealing three-dimensional quantum criticality in Han Purple

Quantum spin-dimer systems constitute a particularly valuable class of materials for the study of quantum criticality. In an applied magnetic field, these systems undergo a quantum phase transition (QPT) – at a critical field $H_c$ – from a quantum disordered phase with $XY$ symmetry to a phase with transverse magnetic order that breaks the $XY$ symmetry and which can be described as Bose-Einstein condensate for 3D-coupled materials. However, quasi-2D systems are special because of Berezinskii-Kosterlitz-Thouless physics, which has recently been argued to give rise to “unconventional” behavior at the dimensional crossover. Thus the question arises if “conventional” emergence of 3D physics can really be expected at their QPT.

A prominent quasi-2D spin-dimer material is Han Purple (BaCuSi$_2$O$_6$), which consists of stacked bilayers hosting spin dimers and was reported to show apparent 2D scaling close to the QPT interpreted as “dimensional reduction”. However, below 90 K, this system undergoes a phase transition from a tetragonal structure with equivalent bilayers to a weakly orthorhombic structure that contains three inequivalent bilayers. These inequivalent bilayers cause an anomalous effective scaling regime [Allenspach et al., Phys. Rev. Lett. 124, 177205 (2020)], with the 3D critical behavior remaining experimentally unobservable. Remarkably, the simple high-temperature tetragonal structure can be stabilized down to lowest temperatures by stoichiometric substitution of Sr for Ba ions. We have performed inelastic neutron scattering (INS) experiments for Ba$_{0.9}$Sr$_{0.1}$CuSi$_2$O$_6$ to determine its magnetic model and verified that all bilayers are equivalent at 1.5 K.

In addition, we have extracted the phase diagram of this compound using various high-field methods supplemented by quantum Monte Carlo (QMC) simulations based on the INS results.

To investigate the quantum criticality, phase-boundary points were extracted from $^{29}$Si NMR spectra of Ba$_{0.9}$Sr$_{0.1}$CuSi$_2$O$_6$ measured for various magnetic fields and low temperatures. In the critical regime, the phase boundary takes the form $T_c(H) = \alpha(H - H_c)\phi$, where $\phi = 2/d$ is inversely proportional to the dimensionality $d$ of the system. The phase boundary extracted from the NMR spectra and simulated by QMC was analyzed using Bayesian inference, thereby determining the posterior distribution of the model parameters. Figure 55(a) displays the phase-boundary points and the posterior distribution of the phase boundary obtained by including all points up to a maximal temperature $T_{\text{max}} = 0.43$ K. Because the width of the critical regime is non-universal, the Bayesian analysis was repeated for different $T_{\text{max}}$ and the resulting posterior distributions of $\phi$ are shown in figure 55(b). A convergence of $\phi$ to 2/3 (3D scaling) is visible for both NMR and QMC when $T_{\text{max}}$ is reduced. Due to the large uncertainties of the NMR estimates, the width of the critical regime was determined from the QMC estimates and used for the data ranges shown in figure 55(a). We conclude that the structural alteration caused by the small 10% Sr-substitution of Han Purple fully restores the originally envisaged properties of 3D quantum critical scaling at the field-induced QPT of a quasi-2D spin-dimer system [Allenspach et al., Phys. Rev. Research 3, 023177 (2021)].

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Neutron diffraction of field-induced magnon condensation in the spin-dimerized antiferromagnet Sr$_3$Cr$_2$O$_8$

Sr$_3$Cr$_2$O$_8$ is a spin-1/2 dimerized antiferromagnet. The spin dimers consisting of $S=1/2$ Cr$^{3+}$ ions are coupled antiferromagnetically by an intra-bilayer exchange interaction $J_T$ along the $c$ axis. At room temperature, the single 3$d^4$ electron of the Cr$^{3+}$ ion has a twofold orbital degeneracy, which makes the system Jahn-Teller active. The Jahn-Teller transition is related to a structural phase transformation from hexagonal $Rar{3}m$ to monoclinic $C2/c$ at $T_{JT} = 285$ K. The magnetic frustration due to the hexagonal structure is lifted because of this distortion, leading to spatially anisotropic magnetic interactions. The magnetic phase diagram of this compound is characterized by a lower critical field for the magnon condensation at $B_{c1} = 30.9(4)$ T and an upper critical field at $B_{c2} = 61.9(3)$ T [Aczel et al., Phys. Rev. Lett. 103, 207203 (2006)]. The maximum temperature of this domelike phase is $T_c \approx 8$ K, at approximately 45 T. The extracted critical exponent of the ordering temperature as a function of reduced field around $B_{c1}$, $T_c \sim (B - B_{c1})^\nu$, was found to be $\nu = 0.65$, very close to $\nu = 2/3 \approx 0.67$ predicted for a three-dimensional Bose-Einstein condensation (BEC) of magnons. Recent experiments of dilatometry and ultrasound have revealed a strong spin-lattice coupling with the shrinkage of the unit cell in the ordered phase [Nomura et al., Phys. Rev. B 102, 165144 (2020)].

In this work, magnetostriction, inelastic neutron scattering, and neutron diffraction have been used to establish the magnetic phase diagram of the spin-dimerized quantum antiferromagnet Sr$_3$Cr$_2$O$_8$, shown in figure 56. Inelastic neutron scattering in static magnetic fields up to 15 T reveal linear field dependency of the spin singlet-triplet excitations in this material, in agreement with the reported results of electron spin resonance measurements. The magnetic structure in the Bose-Einstein condensation phase was probed by neutron diffraction in pulsed magnetic fields up to 39 T. The observed magnetic signal confirms the field-induced phase transition starting at $B_{c1} =30.9$ T.

A representation analysis of the obtained data allowed us to derive two possible spin structures at 2 K above $B_{c1}$, both of which confirm an antiferromagnetic configuration of the XY components that are perpendicular to the field direction. However, we cannot address the ordering of the longitudinal spin components due to the limited reciprocal space available in our experimental techniques. The resolved XY-AFM configuration of the magnon condensation phase is consistent with the theoretical description. Additionally, our zero-field diffraction results indicate that the monoclinic structure is only fully developed below 60 K, the same temperature range as the intradimer interaction. This suggests that not only do the orbital and lattice degrees of freedom play a role in the stabilization of the crystallographic symmetry breaking transition, but that dimerization also has an important role. More details about this work can be found in [Gazizulina et al., Phys. Rev. B 104, 064430 (2021)].

**FIG. 56.** Zeeman splitting of the triplet modes in Sr$_3$Cr$_2$O$_8$. The $\Gamma$-point data are taken from the inelastic neutron scattering experiments. The $M$-point data are from zero-field inelastic neutron scattering and pulsed-field neutron diffraction measurements. All data were measured at $\approx 2$ K. ESR data points are taken from Ref. [Wang et al., Phys. Rev. B 89, 174406 (2014)]. Critical fields are extracted from the magnetostriction results. The red and blue arrows below the $\chi$ axis show a schematic illustration of the direction of the spins in a dimer. On the inset, the magnetic structure in the XY-AFM phase $\Gamma_2$ and $\Gamma_4$ are presented. The magnetic unit cell is $2a \times 2b \times c$, but only $a \times b \times c$ is shown for clarity. The red solid line represents the dominant AFM intradimer interaction $J_0$. The interdimer interactions are shown by dotted lines.

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Biology and Molecular Magnetism
Helicene-based ligands enable strong magneto-chiral dichroism in a chiral ytterbium complex

Magneto-Chiral Dichroism (MChD) represents a fascinating manifestation of light-matter interaction in chiral magnetized systems. It features a modification of the absorption or emission of unpolarized light that depends on the relative orientation of the magnetic field, the direction of light propagation vector, and the absolute configuration of the system. MChD has been studied on a limited number of chiral molecular systems. Renewed interest in this phenomenon has allowed its detection in 2D chiral perovskites, and in a 2D molecular ferrimagnet at temperatures as high as 40 K. More recently, MChD investigations on a molecular magnetic helix based on Jahn-Teller elongated Mn$^{II}$ ions have revealed the crucial role of spin-orbit coupling in MChD intensity.

These results call for a study of MChD in chiral lanthanide systems because they possess a stronger spin-orbit coupling than first row transition metals and allow for impressive single-molecule magnet (SMM) properties. Indeed, long-range ordered chiral magnets or SMMs showing strong MChD can be envisioned for optical readout of magnetic data with unpolarized light.

In this study we have investigated for the first time MChD through NIR light absorption on enantiopure Yb$^{III}$ complexes [Yb$^{III}((X)-L)(hfac)_3$] ($X = P, M$; $L = 3-(2$-pyridyl)-4-aza[6]-helicene; Hhfac = 1,1,1,5,5,5-hexafluoro-acetylacetone) (1-(P), 1-(M)) (figure 57).

MChD measurements performed at $T = 4.0$ K with an alternating ($\Omega = 1.5$ Hz) magnetic field $B$ applied along $k$ in the $0.0 - 1.86$ T range reveal strong MChD signals originating from the difference in the absorption of the $^{2}F_{5/2} \leftarrow ^{2}F_{7/2}$ transition split by crystal field and vibronic coupling, with equal intensities and opposite signs for the two enantiomers.

The extensive temperature range explorable for this system has allowed to observe an unprecedented feature in the band-shape of the main MChD signal. At low temperatures, it has an absorption-like line-shape. When the temperature increases, the intensity becomes lower as a result of the lower magnetization, but more importantly, the line-shape changes, becoming of derivative-type (figure 58).

An absorption-like band-shape is associated with B and C terms (Barron and Vrbanech microscopic model), the latter expected when the population of the ground state levels changes, whereas the derivative-like band-shape is associated with A terms, related to the lifting of the degeneracy of ground- or excited state levels due to the magnetic field. The A and C terms contributions are unraveled by spectral multiple regression fit over the whole temperature range and their temperature dependence evidences a $1/T$ dependence for the C term, whereas the A term is essentially temperature independent, as predicted by the theory.

In conclusion, a fine-structured strong MChD signal associated to the $^{2}F_{5/2} \leftarrow ^{2}F_{7/2}$ transition has been detected. The analysis of the temperature and magnetic field dependence of the MChD spectra has revealed (i) the major role of crystal field and vibronic coupling in defining the shape of the MChD spectrum, and (ii) the temperature dependence of the MChD lineshape confirms the importance of the A and C terms of the MChD theory, representing a first unambiguous identification of the underlying mechanisms. For more details please see [Atzori et al., J. Am. Chem. Soc. 143, 2671-2675 (2021)].

Validation of microscopic magnetochiral dichroism theory

Although magneto-chiral dichroism (MChD) has now been experimentally observed in different types of materials, no quantitative comparison of experimental results with the microscopic theory developed by Barron and Vrbancich (BV) has been made so far. From the experimental side, the determination of accurate MChD spectra in the presence of the generally much stronger natural circular dichroism (NCD) and magnetic circular dichroism (MCD) effects is challenging. Some MChD calculations, based on the BV theory, have been reported for small molecules, but they have not been compared to experiments, the predicted values being much below current experimental sensitivity.

To bring our quantitative understanding of MChD to the same level as that of NCD and MCD and to underpin the potential of MChD as a chiral spectroscopy tool, we have selected two well-characterized paramagnetic chiral materials, with clearly identified optical transitions, tris-(1,2-diaminoethane)metal(II) nitrate \([M^{II}(dae)_3]_2(\text{NO}_3)_2\) \([M^{II} = \text{Ni}^{2+} (1), \text{Co}^{2+} (2); \text{dae} = 1,2\text{-diaminoethane}]\). These tris-chelated octahedral enantiopure complexes (figure 59) are obtained by spontaneous resolution during crystallization and crystallize in the chiral \(P6_322\) space group.

We have measured their low-temperature absorption and MChD spectra in the \(4 - 16\) K range, both parallel and perpendicular to the optical axis (figure 60). In this temperature range, the experimental MChD spectra turn out to be entirely dominated by the so-called \(C_1\) terms, stemming from ground state population changes and identified by their temperature dependence, thereby greatly reducing the computational effort required to calculate the spectrum and increasing the accuracy of the results.

The BV theory expresses the electric dipole-magnetic dipole \(C_1\) term and the electric dipole-electric quadrupole \(C_2\) terms as a sum over transition moments between the electronic wave functions of the ions, perturbed by their ligands. We have calculated these transition moments, including the vibronic coupling (VC) contributions, for the nickel(II) derivative with state-of-the-art quantum chemical methods, whereas, at present, the computational cost for the cobalt(II) analog remains out-of-reach.

The results of the calculations of the MChD spectrum for 1 for both axial and orthoaxial orientations are shown in figure 60, with and without the VC. Very good agreement with the experimental MChD spectra is obtained only when the VC is included.

In summary, we have experimentally and theoretically investigated the MChD of two model systems, tris-(1,2-diaminoethane) nickel(II) and its cobalt(II) analogue. Very strong MChD signals have been experimentally observed and associated with the metal ions absorption bands. Their temperature dependence is characteristic for \(C\) terms in the BV theory. For the nickel(II) derivatives, good agreement is found with calculations of such \(C\) terms, thereby confirming this part of the BV theory, while the unexpected importance of vibronic coupling was evidenced. For more details please see [Atzori et al., Sci. Adv. 7, eabg2859 (2021)].

FIG. 59. View of the molecular structure of \((\Delta)[M^{II}(dae)_3]_2^{2+}\) (left) and \((\Lambda)[M^{II}(dae)_3]_2^{2+}\) (right) \((M^{II} = \text{Ni, Co})\) complex cations. Color codes: red, \(M^{II}\); blue, \(N\); gray, \(C\); white, \(H\). Nitrate anions are omitted for clarity.

FIG. 60. (a) Experimental \(\Delta A_{MChD}\) spectra for a single crystal of 1 in axial and orthoaxial configuration. (b) Corresponding calculated \(\Delta A_{MChD}\) spectra. (c) Calculated \(\Delta A_{MChD}\) spectra with only \(C_2\) terms (note change in scale).

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Robust magnetic anisotropy of a hexacoordinate Fe(II) complex

One of the current challenges in the field of molecular magnetism deals with the ways these molecules can be integrated in solid state devices. In this frame, the understanding of the changes of their magnetic properties when deposited on a surface is crucial to minimize them. Here we report on the study of an Fe(II) complex [Kelai et al., Inorg.Chem.Front.8, 2395(2021)] possessing a relatively rigid organic ligand, which precludes structural deformation and ensures some robustness of the magnetic anisotropy: [Fe(3,5-(CH$_3$)$_2$Pz)$_3$BH$_2$] where Pz = pyrazolyl (1). The complex undergoes only a partial spin cross-over (SCO) when deposited on a surface but a complete one in bulk form. Thus the study of the bulk magnetic anisotropy was performed on the parent complex: [Fe(3,5-(HPh)Pz)$_3$BH$_2$] where Ph = phenyl (2).

Multifrequency HF-EPR study performed on a microcrystalline powder sample of 2, pressed into a pellet to reduce torquing effects, revealed several signals for frequencies $\nu > 200$ GHz (figure 61). The resulting spectra suggest that molecules with different anisotropies (resulting probably of slight geometrical changes) are present with a distribution exhibiting two maxima (a main one and a secondary one, with population ratios 4:1). Indeed, as can be seen in the 662.4 GHz spectra, the strong and well-resolved low field signals (between 4 and 6 T) correspond to forbidden transitions (off-axis turning points) whereas the signals appearing at higher field which correspond to allowed transitions are much broader, extending for some of them over 1 T. To analyse the spectra recorded at all frequencies we used a model Hamiltonian to obtain a set of parameters defining the magnetic anisotropy for the molecules responsible for the strongest signals (e.g. 5.5 T at 662.4 GHz): $D_{EPR} = 12.1 \text{ cm}^{-1}$, $E_{EPR} = 0.61 \text{ cm}^{-1}$ and $g_x = g_y = 2.1$, $g_z = 2.2$, allowing to reproduce several observed transitions (figure 61).

$$H = \mu_B S \cdot g \cdot B + D(S_z^2 - S(S+1)/3) + E(S_z^2 - S_x^2),$$

where $D$ is the axial zero-field splitting term, $E$ the rhombic one, $\mu_B$ the Bohr magneton and $g$ the Landé matrix.

These values are in good agreement with the ones issued from the magnetic data analysis (susceptibility and magnetization). The $g_x$ value is the most uncertain one as no well-defined transition could be associated to the $z$ orientation. Unfortunately, it is not possible to obtain a full set of parameters for the magnetic anisotropy of the molecules leading to the secondary maximum (e.g. 4.2 T signal at 662.4 GHz). An axial magnetic anisotropy $D'_{EPR} = 14.1 \text{ cm}^{-1}$ is found considering that the rhombicity is unchanged (i.e. $E'_{EPR} = 1.14 \text{ cm}^{-1}$) as well as the $g$ values ($g_x = g_y = 2.1$ and $g_z = 2.25$).

Alongside, the magnetic anisotropy of the high spin S=2 state of 1 assembled as an organic monolayer on Cu(111) was measured by X-ray Magnetic Circular Dichroism (XMCD) spectroscopy. The results indicate that the easy plane magnetic anisotropy ($D > 0$) of the assembled molecules is not affected when they are present at the substrate/vacuum interface.

FIG. 61. (a) Experimental and (b) calculated EPR powder spectra of 2 at three different frequencies and at 5 K (black) and 15 K (red).

$ab initio$ wave functions based theoretical calculations are then used to compare these results with those of a reported complex having an almost identical FeN$_6$ coordination sphere but a negative $D$ value (easy axis of magnetization). They show that the nature of the magnetic anisotropy (easy axis versus easy plane) is governed by the torsion angle defined by the relative orientation of the pyrazolyl five-membered rings to the pseudo three-fold axis of the molecules. The rigidity of the (Pz)$_3$BH tridentate ligands, where the BH group holds the three pyrazolyl moieties, allows only very slight changes in the torsion angle and is at the origin of the robustness of magnetic anisotropy of this family of compounds.

A.L. Barra
New cyanido-bridged heterometallic 3d-4f 1D coordination polymers

The discovery of slow relaxation of the magnetization phenomena for discrete metal complexes (Single Molecule Magnets, SMMs) and 1D coordination polymers (Single Chain Magnets, SCMs) has stimulated the development of an intensive interdisciplinary research field. Beyond their relevance in fundamental Physics and Chemistry, spectacular applications in quantum computing and high-density information storage from these molecules are expected. The SMMs as well SCMs can be assembled using homo- or heteronuclear spin carriers. (3d-3d', 3d-4f, 2p-3d, and 2p-3d-4f). Recently we reported on a new family of 1D coordination polymers, which are assembled from \([\text{LnL}]^{3+}\) and \([\text{M(CN)}_6]^{3-}\) ions (L = bis-semicarbazone ligand, M = Fe, Co).

Three new 1D cyanido-bridged 3d-4f coordination polymers, \([\text{Ln(L(H}_2\text{O})_2\text{Fe(CN)}_6\cdot\text{H}_2\text{O})_n}\) (1GdFe), \([\text{Dy(L(H}_2\text{O})_2\text{Fe(CN)}_6\cdot3\text{H}_2\text{O})_n}\) (2DyFe), and \([\text{Dy(L(H}_2\text{O})_2\text{Co(CN)}_6\cdot\text{H}_2\text{O})_n}\) (3DyCo) were assembled following the building-block approach (L = pentadentate bis-semicarbazone ligand resulting from the condensation reaction between 2,6-diacetylpyridine and semicarbazide). The crystal structures consist of crenel-like LnIII-MIII alternate chains, with the LnIII ions connected by the hexacyanido metalloligands through two cis cyanido groups. The evolution of the temperature dependence of DC magnetic susceptibility for 2DyFe and 3DyCo is defined by the presence of strongly anisotropic DyIII ions. Variable-temperature and variable-field AC susceptibility measurements show that two DyIII-containing representatives (2DyFe, 3DyCo) exhibit field-induced slow magnetic relaxation. A two distinct relaxation processes are detected for 2DyFe and 3DyCo. The AC susceptibility data were analyzed with generalized Debye equations including a two-step relaxation process. In both compounds, the first relaxation process at low frequency (LF) is well-defined with \(\alpha_1 = 0.3 - 0.5\). Three different mechanisms of relaxation, namely, quantum tunneling of magnetization (QTM), direct, and mechanisms were discussed. The QTM and Orbach mechanisms are dominant over the investigated range of temperature.

These results open interesting perspectives for the synthesis of new cyanido-bridged 3d-4f complexes, using not only homoleptic, but also heteroleptic cyanido tectons, as well as other types of metalloligands.

For more details please see [Dragancea et al., Magnetochemistry 7, 57 (2021)].

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Applied Superconductivity
Insert repair and new probe fabrication for a HTS user magnet

Some outer/inner electrical junctions of our metalas-insulation (MI) high temperature superconducting (HTS) insert were damaged following a quench at 32.5 T and an unanticipated shutdown of the background magnetic field ($B_{ext}$) from 19 T. To repair the insert, 7 MI double-pancake (DP) coils were re-wound with the former REBCO tapes and only the damaged HTS pieces were replaced by new ones at the inner junctions. However, 2 DP coils had to be fully fabricated with new REBCO tapes because their conductors were deformed. Note, that although the electro-magnetic performance of a DP coil rewound with a deformed conductor at 77 K was found similar to that of the original one, we did not want to take any risk and decided to replace the deformed tapes.

**TABLE I. Specifications of the repaired MI HTS insert.**

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Conductor:</strong> GdBaCuO tape</td>
<td></td>
</tr>
<tr>
<td>Width: total thickness</td>
<td>6; 0.075 mm</td>
</tr>
<tr>
<td>Critical current at 77 K</td>
<td>&gt; 210 A</td>
</tr>
<tr>
<td><strong>Repaired MI insert</strong></td>
<td></td>
</tr>
<tr>
<td>Coil i.d.: average o.d.</td>
<td>50; 110.8 mm</td>
</tr>
<tr>
<td>Number of DP</td>
<td>9</td>
</tr>
<tr>
<td>Average number of turns per pancake</td>
<td>286</td>
</tr>
<tr>
<td>Stainless steel overband turns</td>
<td>45</td>
</tr>
<tr>
<td>Calculated coil constant</td>
<td>44.3 mT/A</td>
</tr>
<tr>
<td>Magnet inductance</td>
<td>0.82 H</td>
</tr>
<tr>
<td>Initial characteristic resistance</td>
<td>295 mΩ</td>
</tr>
<tr>
<td>Initial contact surface resistivity</td>
<td>14.7 mΩ·cm$^2$</td>
</tr>
</tbody>
</table>

This probe has two HTS current leads of high capacity (maximum 1000 A at 4.2 K) to reduce the liquid helium (LHe) consumption and avoid Joule heating near the HTS magnet. In addition, 12 Jaeger connectors (144 pins in total) were installed on top flange of the probe for measuring the many signals of tested HTS inserts. To estimate the field strength and stability of the insert, an NMR coil with 1 mm$^3$ of $^{27}$Al metallic foil sample (gyromagnetic ratio $\gamma = 11.1122$ MHz/T) as well as some Hall sensors were placed on a support that can be displaced from outside in the 34 mm bore size tube. Figure 65 shows images of the repaired MI insert, new probe and homemade 3D printed insert supporting several sensors.

FIG. 64. Measured values of critical current, resistance and coil constant of original, dismantled and repaired DP coils in bath of liquid nitrogen at 77 K in self-field.

The outer junctions were all remade as well as the over-banding. During the repair of the DP coils, the thin copper plate insulated by G10 sheets, used as an interlayer between the single-pancake (SP) coils, was replaced by a sapphire plate combining electrical insulation with thermal conduction to enhance the cooling condition inside each DP coil. Figure 64 shows the test results of the original, dismantled and repaired MI DP coils at 77 K in self-field (SF). The resistance values of DPs 2 to 8, with only the inner joints rewound, were 0.32, 0.17, 0.23, 0.6, 0.33, 0.12 and 0.12 $\mu$Ω, respectively. They were significantly reduced by making new inner junctions.

After the 77 K tests, the nine repaired DP coils were stacked again on the insert support structure and an axial preload pressure of 10 MPa was applied using Belleville washers. For a better cooling, 0.3 mm thick copper plates insulated by 0.2 mm G-10 sheets, one on each side, were inserted in between the DP-DP coils. Each pancake coil was then over-banded by about 45 turns of 75µm thick 316L stainless-steel (SS) tape. 3 turns of REBCO tape were wound onto the middle of the over-banding to eventually reduce induced voltages, currents and unbalance forces that could be generated by an quench event. Table I lists the specifications of the repaired insert. A new probe was designed with a 34 mm bore size tube to access to the insert bore for NMR characterization, and eventually for use by real end-users.

**FIG. 65. Photographs of the repaired MI insert, new probe and homemade insert supporting five Arepoc Hall sensors and an NMR coil with an Al sample.**
Characteristic resistance change of a metal-as-insulation HTS insert

The characteristic resistance ($R_c$), the sum of resistances within a no-insulation (NI) variant magnet, is mainly dominated by the turn-to-turn contact resistance, which is an important factor in determining the current distribution within the magnets [Wang et al., Supercond. Sci. Technol. 26, 035012 (2013)]. Many research institutes for development of NI variant magnets have estimated the $R_c$ values through several sudden discharge tests at low operating current level in self-field (SF) just before their first operation [Kim et al., Rev. Sci. Instrum. 91, 023314 (2020)]. However, there are no reports about $R_c$ change of NI variant magnets according to long-term normal operation and change of $B_{ext}$. Since 2018, we have measured $R_c$ value of the metal-as-insulation (MI) insert by sudden discharge (SD) at low current level (10-20 A) in SF without any specific test interval. In addition, to investigate $R_c$ change with respect to increasing with respect to increasing $B_{ext}$, we also performed this time SD tests from 0 to 15 T. Figure 66 shows the measured charging time constant ($\tau$) and estimated $R_c$ values in SF at 4.2 K. $R_c$ varies continuously ranging from 185 to 683 mΩ. The initial $R_c$ value of the insert increased probably because the turn-to-turn contact resistivity increased along with the cryogenic work hardening of the copper stabilizer under repetitive magnetic pressure load [Song et al., IEEE Trans. Appl. Supercond. 30, 4701806 (2020)]. Meanwhile, after repair, the range of the $R_c$ change is slightly larger, but does not deviate significantly.

FIG. 66. The measured $\tau$ and estimated $R_c$ values of the insert in self-field at 4.2 K.

Figure 67 shows $R_c$ values estimated from the measured $\tau$ at $B_{ext} = 0, 5, 10, 15$ T. The $R_c$ values in SF and 15 T were 185 and 339 mΩ, respectively, indicating that the $R_c$ value increased by 83%. One of several possibilities for this phenomenon might be the magneto-resistance (MR) of the REBCO tape. We estimated magneto-resistance of SuperPower, Theva REBCO and Durnomag tapes under $B_{ext}$ from 0 to 16 T at 4.0 K as reported in figure 68. Samples were measured with an ac-current of 5 mA amplitude and a frequency of 35-110 Hz at 4.0 K. In figure 67, the relative resistance change (right y-axis) of Durnomag is changing only by a little (4.5% increase) while the resistances of THEVA and SuperPower REBCO tapes increase from 0 to 15 T by 50% and 114%, respectively. This result suggests that the $R_c$ of the REBCO insert is influenced by magneto-resistance. Since $R_c$ is largely depending on the surface condition, contact pressure and intrinsic properties of the conductor, it varies continuously upon repetitive operations and change of operating conditions. This implies that simulation work using a single value for $R_c$ obtained by SD tests at SF struggles to reflect the reality. Moreover, we can expect that the local turn-to-turn resistance varies also depending on the position in the coil.

FIG. 67. $R_c$ of the MI insert and normalized magneto-resistance of durnomag SS tape, THEVA and SuperPower REBCO tapes from $B_{ext} = 0$ to 15 T.

FIG. 68. Durnomag SS tape, THEVA and SuperPower REBCO tapes mounted on sample holder for MR measurement.

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Charging tests of the repaired metal-as-insulator HTS insert

Although resistances of the repaired metal-as-insulated (MI) double-pancake (DP) coils are similar to those of original coil at 77 K in self-field (SF), this does not guarantee that reliable operation under high magnetic fields ($B_{ext}$) and high charging currents at 4.2 K. Therefore, the repaired insert was submitted to a set of new tests at 4.2 K at various charging currents under $B_{ext}$ from 0 to 18 T. Figure 69 shows the resistance value of each DP coil under $B_{ext}$ = 8 and 16 T. Before repair, all DP coils, except DP5, were highly resistive ($1.1 - 14.1 \mu \Omega$) compared to the original values at the operating current $I_{op} = 300$ A under $B_{ext} = 8$ T.

After repair, the DP coils show a much lower resistance value (210-520 nΩ) under both $B_{ext} = 8$ and 16 T except DP7, indicating that most of repaired DP coils are operating well at 4.2 K under very high magnetic field. The damage that occurred during the quench are mainly localized at the inner and outer electrical junctions. The DP coils themselves are well protected by our passive HTS protection scheme comprising the MI winding technique and the over voltage mode of our power supply. We even removed the external dump circuit and the breaker prone to create signal spikes. The high resistance of the repaired DP7 seems to be linked to a damage of the conductor during the joining process at the outer DP-DP electrical junction. As it is very localized, it can be repaired with sufficient care.

Figure 70 shows the central magnetic field of the HTS insert with various $I_{op}$ in SF at 4.2 K. The field production performance of the original and repaired inserts are very close. Figure 71 shows a charging test of the repaired insert at a current ramp rate of 0.5 A/s under $B_{ext} = 18$ T at 4.2 K. It produced a field of 10.2 T at 235 A under $B_{ext} = 18$ T, which means a total field strength of 28.2 T as indicated by our central Hall sensor. The coil constants of the original and repaired inserts were 44.0 and 43.4 mT/A, respectively, implying that there is no by-pass current within the repaired DP coils. Note, that the coil constant of the refurbished insert is slightly smaller than that of original one because a few turns of tapes had to be removed for repair at the inner and the outer junctions. Above 27.7 T, some instrumentation wires of the insert suddenly showed abnormal signals suggesting a short circuit. This behavior occurs only at high field and may be linked to an insulation default of our voltage taps as the mechanical pressure increases with field.

It should be noted that at high field, the cooling condition for the repaired insert were more unstable than those for the original one. This might be due to the probe modification and its additional sample tube which leaves much less space for liquid helium circulation in the inner bore of the insert. A more stable condition is achieved by pumping on the helium bath. However, the temperature of the top of the repaired insert increased up to 9 K probably due to trapped helium bubbles as already observed during other HTS insert tests. Therefore, taking all these issues into account, we decided not to charge the insert to higher fields. The insert will be tested again for 30 T operation after solving these issues.

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Field stability estimation of the resistive and HTS hybrid magnet

An NMR coil with 1 mm$^3$ of $^{27}$Al metallic foil sample (gyromagnetic ratio $\gamma = 11.1122 \text{MHz/T}$) as well as Hall sensors were located at center position of our hybrid (HTS insert and resistive outsert) magnet. The metal-as-insulator (MI) HTS insert was charged up to 10 T under background magnetic field $B_{\text{ext}} = 10 \text{T}$ at 4.2 K. The central magnetic field and stability were measured at the total magnetic fields ($B_{\text{tot}} = 12.0, 14.2, 17.1, 20.4 \text{T}$). For NMR characterization, 200 single shot NMR spectra were taken at each field during 7-8 minutes. Figure 72 shows the deviation of the average field between Hall and NMR measurement. This deviation is within 1.2% of the field measured by NMR, indicating a good agreement between both measurements and a good accuracy of our Hall sensors, the NMR measurement being the reference.

![Figure 72. Differences of average field between Hall and NMR measurements at total magnetic fields of 12.0, 14.2, 17.1, and 20.4 T ($B_{\text{ext}} = 10 \text{T}$).](image)

Figure 73 shows the measured field stability as a function of time measured by Hall sensor and NMR at $B_{\text{tot}} = 20 \text{T}$ at 4.2 K. In the case of the NMR measurement, the central magnetic field fluctuation and drift were 5-10 ppm and 5 ppm/min, respectively. Apart from an offset, both measurements show the same trend until we started to pump on the helium bath to stabilize the cryogenic condition as the top temperature of the insert was increasing. We observed on this occasion that, while the NMR remains stable, the Hall sensor signal is affected by the change of cryogenic condition. Hall data is thus less reliable to estimate the field stability if a change of temperature occurs concurrently. Therefore, the field stability of our resistive-HTS hybrid magnet was only estimated by NMR under high magnetic field. Figure 74 shows field deviation ($\Delta B/B$) measured by NMR as a function of time at $B_{\text{tot}} = 28.2 \text{T}$ at 4.2 K. After 4 minutes, the central magnetic field drift of the hybrid magnet was 7 ppm/min. In addition, the standard deviation ($\sigma$) of field fluctuations after subtracting a second order drift term (inset in figure 74) was 2.6 ppm. The field drift of 7 ppm/min is mainly due to magnetic field induced by transient screening currents, as the NMR record started immediately after reaching the target field. Weijers et al. from NHMFL, Tallahassee reported that central magnetic field drift changed from 10 ppm/min to 0.5 ppm/min (their power supply drift level) after one hour at 32 T. Therefore, the field drift of the MI HTS insert will be estimated again after staying a longer time ($\geq 1 \text{ hour}$) at target field.

![Figure 73. Estimating result of field stability measured by Hall sensor and NMR characterization at total magnetic field of 20 T.](image)

![Figure 74. Field deviation measured by NMR as function of time at $B_{\text{tot}}$ of 28.2 T at 4.2 K. Inset: Remaining fluctuations after subtraction of second order drift term.](image)

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Investigating magnetic shielding effect and quench current of the THEVA-SuperPower magnet to develop quench protection

In 2019, we tested a high temperature superconducting (HTS) mock-up magnet wound with THEVA and SuperPower REBCO tapes to evaluate quench current of the magnet under various background magnetic fields ($B_{ext}$). The magnet was protected by (i) metal-insulation (MI) winding technique, (ii) over voltage protection (OVP) mode and (iii) magnetic shielding (MS) technique comprised of 3 turns of REBCO tape within the over-banding (OB). During the tests, despite around 40 quench events under ($B_{ext}$) from 0 to 19 T, the magnet survived without any damage. These results demonstrate that the MI winding technique with OVP mode completely protects the HTS magnet. However, it was difficult to investigate the MS effect because no results were available for comparison. Therefore, the 3 turns of REBCO tape were removed from the OB of the MI mock-up magnet. The magnet was suddenly discharged at charging current $I_{op} = 20$ A at 4.2 K in self-field (SF) to investigate its field decay behavior without MS. Furthermore, the quench currents of the magnet were also estimated at $B_{ext} = 0, 5, 10, 15$ T to check degradation of the magnet because it was left outside exposed to air over 2 years.

FIG. 75. Sudden discharge test results of the MI magnet with (a) and without (b) MS at 4.2 K in SF.

Figure 75 shows normalized currents and central magnetic fields versus time during a sudden-discharge of the MI magnet with and without MS. Unlike the MI magnet without shielding, the MI magnet with shielding showed two charging time constants. A fast decay ($\tau_f = \tau$) due to rapid decrease of azimuthal current within the coil, and a slow decay ($\tau_s \approx 3\tau$) due to radial current circulation within the coil). In addition, the field decay of the MI magnet with MS was faster than that of the MI magnet without MS. This indicates that the MS is helping to avoid; (i) high local heating due to discharge of non-uniform azimuthal current within the coil and (ii) mechanical damage generated by unbalanced forces due to very fast discharge of magnetic field. Figure 76 shows the quench test results of the MI magnet under various $B_{ext}$ from 0 to 19 T at 4.2 K. The critical current values and total magnetic field values of the magnet in 2021 test are almost the same as those of the magnet in 2019 test. Figure 77 shows the resistance values of the HTS coil, each DP coil and inner/outer electrical junctions at $I_{op} = 150$ A in SF after quench tests. Even after the magnet experienced over 50 quench events, the resistance values did not increase. These results demonstrate that our protection techniques comprised of MI winding, OVP and MS are perfectly reliable for very high field HTS magnets.

FIG. 76. Quench, charging currents and total center magnetic fields of the magnet under various $B_{ext}$ at 4.2 K.

FIG. 77. The resistance values of HTS coil, each DP coil and inner/outer electrical junctions at 4.2 K in self-field.

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The BMV experiment

2021 was devoted to the commissioning of the new coil dedicated to the BMV experiment called foil coil. The principle of this polarimetric vacuum birefringence search is illustrated in figure 78. Due to the presence of a Fabry-Perot cavity of finesse $F$, as it propagates through the region of birefringent vacuum, the polarization of the light exiting the cavity acquires a total ellipticity $\Psi$ amplified by a factor $2F/\pi$:

$$\Psi = \frac{2F}{\lambda}K_{CM}B^2L_B\sin(2\theta).$$

The polarization components are then separated by the analyzer (power extinction ratio $\sigma^2$) at the output of the optical cavity. Then, the signal is recorded as the power at extinction of the second polarizer and compared to the total power exiting through the cavity end mirror,

$$P_{ext} = \left[\sigma^2 + (\Gamma + \Psi)^2\right]P_t,$$

where, taking into account that Fabry-Perot mirrors are also birefringent, one has to introduce a corresponding ellipticity generated by the cavity itself, $\Gamma$.

The cavity is formed by two interferential mirrors of radius of curvature of 2 meters. We have achieved a cavity finesse as high as $537\,000$ and an extinction ratio as low as $\sigma^2 \sim 1.5 \times 10^{-9}$ enabling us to have a static birefringence as low as $\Gamma \sim 5 \times 10^{-5}$ rad.

A notable feature of the new apparatus is that the vacuum tube is directly in contact with the coil and it is mechanically isolated by two bellows from the vacuum chambers.

Another novelty of these series of pulses is that we have placed at critical places of the optical set-up some absorbing materials to reduce stray light in the vacuum tanks.

All the pulses were performed in high vacuum with pressure ranging from about $10^{-6}$ to $10^{-7}$ mbar thanks to two ionic pumps that work continuously during the magnetic pulse.

We have investigated the impact on the optics of different pulse rise times, ranging from 2.9 ms to 5.8 ms, and different field amplitudes i.e. the energy injected into the system.

This coil can deliver a field transverse to the light propagation of more than $10\,T$ over about 0.8 meters operating without cryogenic equipment.

The results have been published in [Béard et al., Review of Scientific Instruments 92, 104710, (2021)]. They have been obtained from 578 pulses distributed over 14 sets with different pulse lengths (5.8 ms, 4.4 ms and 2.9 ms) and different pulse strengths.

Our best value has been obtained with the short pulses at $5.5\,T$ and an analysis time of $2.7\,ms$ where we reached a maximum field similar to 2014 and obtained $K_{CM} = (0.2 \pm 1.0) \times 10^{-20}\,T^{-2}$, which is about the same as the value obtained in 2014, if we consider that this has been obtained with half the number of pulses, but much more accurate. This value is shown with other reported results in figure 79. Indeed, unlike in the 2014 results, we do not observe a systematic effect.

The insertion of the new magnet in the apparatus has been very successful. The magnet commissioning and the first results look encouraging. We are currently working to diminish the overall noise by better acoustically insulating the apparatus from the coil. On a long term perspective we are also studying how to suspend in vacuum the optical tables holding mirrors and polarizers. The new generation of the BMV experiment has just begun, and there is very much room for improvement.

FIG. 78. Illustrative example of a vacuum magnetic birefringence experiment. The laser light is polarized before entry into the cavity surrounding the magnetic field region, after the cavity light is analyzed by another polarizer at extinction. Two photodiodes PD-t and PD-ext monitor respectively the ordinary beam $P_t$ and the extraordinary one $P_{ext}$.

The cavity is formed by two interferential mirrors of radius of curvature of 2 meters. We have achieved a cavity finesse as high as $537\,000$ and an extinction ratio as low as $\sigma^2 \sim 1.5 \times 10^{-9}$ enabling us to have a static birefringence as low as $\Gamma \sim 5 \times 10^{-5}$ rad.

A notable feature of the new apparatus is that the vacuum tube is directly in contact with the coil and it is mechanically isolated by two bellows from the vacuum chambers.

Another novelty of these series of pulses is that we have placed at critical places of the optical set-up some absorbing materials to reduce stray light in the vacuum tanks.

FIG. 79. Measurements of vacuum magnetic birefringence across the years, errors bars are represented with a coverage factor $k = 3$. 

First results from the Grenoble axion haloscopes (GrAHal)

Particle physics is not only confined to the high energy frontier. There are unexplored territories at ultra-low energies, i.e. sub-eV, which are also promising for major discoveries. The emblematic particle of this physics is the axion, a pseudo-scalar particle predicted independently by Weinberg and Wilczek in 1978 to solve the fundamental problem of the apparent non-violation of the CP symmetry by the strong interaction. Standard axion at the electroweak scale has been excluded after extensive experimental searches but leaving fully open the case for “almost” invisible axion, i.e. with mass and coupling to other particles extremely weak. If the axion mass is typically in the range 1-1000 µeV, this particle could also be the main dark matter component of our universe and is one of the rare non-supersymmetric candidates. In this context, we have built several Sikivie’s haloscopes for Axion search with unprecedented sensitivity. The Grenoble hybrid magnet will offer a unique opportunity to detect in the laboratory axion DM particles or any other axion like particles (ALPs) within the mass range 1-150 µeV.

The haloscope principle relies on the following two main assumptions; (i) the major part of the DM galactic halo is made of weakly interacting axions or ALPs, (ii) they decay into photons in strong magnetic fields. If a resonant RF cavity is added to amplify the signal, the power increase due the axion conversion into photon can be written as,

\[ P = g_{a\gamma\gamma}^2 \left( \frac{\rho_{\text{halo}}}{m_a} \right) B^2 V C Q \]

where \( g_{a\gamma\gamma} \) is the unknown axion-diphoton coupling constant, \( \rho_{\text{halo}} \approx 450 \text{ MeV cm}^{-3} \) is the assumed halo DM density, \( m_a \) the probed axion mass (\( m_a \approx \omega \) in units with \( c = \hbar = 1 \)), \( B \) the magnetic field, \( V \) the cavity volume, \( C \approx 0.5 \) the coupling factor of the electromagnetic mode and \( Q \approx 10^4 - 10^5 \) the quality factor of the cavity. The instrumental challenge lies in the weakness of the awaited signal (of the order of \( 10^{-23} \text{ W} \)) requiring specific ultra-low noise amplifiers as well as ultra-low temperature to minimize thermal noise.

Preliminary results have been obtained using a haloscope prototype working at liquid He temperature. It consists of an oxygen free copper cylindrical RF cavity (inner diameter 36 mm, length 150 mm) without tuning system as a first step and with a TM010 mode frequency close to 6.373 GHz at low temperature (axion mass close to 26.37 µeV). It was maintained at liquid He temperature inside a 52 mm bore 14 Tesla magnet. Its unloaded quality factor \( Q_0 \) is 37000 ± 10%, the main antenna coupling constant is \( \beta = 1.2 \) and we take \( C = 0.69 \) for the TM010 mode coupling factor. The signal emerging from the cavity main port was amplified a first time using a cryogenic high-electron mobility transistor. The total gain of the receiver chain was calibrated using the power emitted by a heated load connected at the location of the RF cavity. A few tuning steps have been obtained by increasing the He temperature and then decreasing it to 1.7 K to minimize thermal noise. No signal above the noise was detected. From a statistical analysis, the axion-diphoton coupling constant \( g_{a\gamma\gamma} \) can be constrained at 95% confidence level to be less than \( 2.2 \times 10^{13} \text{ GeV}^{-1} \), i.e. \( g_{a\gamma\gamma} < 22g_{\text{KSVZ}} \), around 6.373 GHz corresponding to axion mass of 26.37 µeV. This new result has been inserted as a thin exclusion line inside figure 80 showing exclusion limits obtained from worldwide axion search experiments as well as highlighting GrAHal perspectives.

For more details please see [Grenet et al. arxiv:2110.14406].

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Magnetohydrodynamic (MHD) studies the behaviour of conductive fluids subjected to a magnetic field. In these fluids, the so-called Alfvén waves can occur. These waves combine transverse oscillations of the magnetic and velocity fields which propagate along the magnetic field. They were first predicted by Alfvén in 1942, in the case of perfectly and inviscid fluids [Alfvén, Nature, 150 (1942)], the theory was further extended to the case of a viscous and resistive fluid. Shortly after the prediction of their existence, these waves were observed by Lundquist using a vessel filled with liquid sodium. More recently, from experimental tests using a gallium alloy, we provided evidence of Alfvén wave propagation [Alboussiere et al., Phys. Fluids, 23 (2011)]. However, the dynamics of these waves and especially their interaction with the turbulence of a fluid are still not fully understood.

One of the main results of the model is the evolution of the velocity amplitude ratio between the top plate and the bottom plate as a function of the excitation frequency of the wave, for different Lundquist number $S$ (figure 82). The Lundquist number refers to intensity of the magnetic field $B_0$. For instance, in our set-up a value of $S = 53$ is equivalent to a magnetic field of 10 T. In this figure, one can note it appears so-called “resonant peaks” at specific frequencies, where the higher is the magnetic field, the higher are the peak values and the resonant frequencies. These markers will be determinant to confirm the existence the Alfvén waves during experimental trials.

An experimental device is under development. Figure 83 shows the vessel’s interior, where the liquid metal will be located. In particular, the velocity fluid will be measured next to the bottom and top plates using the electric potential velocimetry. In addition, the magnetic oscillation will be generated at the bottom or the top plate, by injecting an ac-current via 10 mm diameter electrodes.

The first part of this investigation was to develop a semi-analytical model of these wave in the resistive MHD assumption i.e. when the advection of the magnetic field is much weaker than its diffusion. In this model, Alfvén waves are studied in a axisymmetric 10 cm high vessel filled with a conductive fluid and subjected to vertical uniform magnetic field $B_0$. The forcing of the wave is carried out at the bottom of the vessel by imposing a magnetic field perturbation. In fact, the purpose of this model is to show the evolution of the propagation of this wave, either regarding the magnetic disturbance or the velocity disturbance, along the magnetic field. In particular the model gives access to the velocity of the field next to the bottom and the top plate. This model was designed to be consistent with the experimental setup that will be presented below. The figure 81 shows a simplified diagram of the experimental device. In this figure, one can see a qualitative representation of Alfvén waves, which are vortex oscillations along the magnetic direction.

**FIG. 81. Diagram of the Flowcube device with vortex oscillations called Alfvén waves.**

**FIG. 82.** Amplitude ratio of velocity versus the frequency of the injected current for different Lundquist numbers at 10 cm from the electrode.

**FIG. 83.** Close-up view of the vessel’s interior. (1) injection electrode, (2) potential probes, (3) ultrasound transducers.
Instrumentation
60 GHz electron cyclotron resonance ion source using radially cooled polyhelices

The following experiment description concerns the SEISM (Sixty gigahertz Ion Source using Megawatt magnets) compact ECR ion source operating at 60 GHz. The prototype uses a magnetic CUSP to confine the plasma. This simple magnetic geometry was chosen to allow the use of polyhelixes coils to generate a strong magnetic confinement featuring a closed ECR surface at 2.14 T. The plasma is sustained by a 300 kW microwave pulse of 1 ms duration and with a 2 Hz repetition rate. Previous experiments using a resistive magnet have successfully demonstrated the establishment of the nominal magnetic field and the extraction of ion beams with a current density up to $\simeq 1 \text{ A/cm}^2$. The presence of “afterglow” peaks was also observed, proving the existence of ion confinement in a CUSP ECR ion source.

During 2021, two coils (among four), damaged during a previous campaign, were rebuilt and a new analysis beam line was installed. All the equipment were aligned and tested before the general startup. Figure 84 shows a picture of the overall experiment installed at a resistive magnet site. The transport line is composed of a quadrupole triplet with 150 mm gap to ensure focusing of the ion beam, a dipole spectrometer with a gap of 90 mm, a horizontal aperture of 300 mm and beam diagnostics at the end of the line (Faraday cup and a pepperpot emittance measurement device (figure 85).

During this year, through several commissioning experimental campaigns, the nominal magnetic field configuration was obtained with a current of 23 kA applied on the newly rebuilt polyhelixes. Furthermore, the Gyrotron was restarted and set up to deliver reliably pulses of 150 kW of HF power (figure 86). The combination of the magnetic field and the injection of HF pulses inside the source allowed the production of the first ion beam pulses analysed directly after the extraction. This important milestone paves the way for further investigations and a detailed characterisation of the ion source, which should start imminently.
**Field modulation of resistive high field magnets at the ppm level**

High resolution nuclear magnetic resonance (NMR) studies require a precise control of the external magnetic field, at least at the level of parts per million (ppm). Commercial superconducting NMR magnets provide field stabilities at the parts per billion (ppb) level up to 28.3 T. At dedicated high field facilities, superconducting magnets up to 32 T operate at the ppm level. Resistive high field magnets, however, have limited field stability due to fluctuations and field drifts. The former mainly originate from current instabilities, whereas the field drifts are mostly due to thermal processes in the magnet that are invisible for the current regulation of the power converters. LNCMI high field magnets show field fluctuations of about 20 ppm and field drifts up to 40 ppm/h.

In order to compensate the drift, an external control circuit based on an NMR field-lock can be used. For this purpose the new LNCMI power converters are equipped with an external field modulation entry. An external voltage \( U_{\text{mod}} \) changes the current of the power converters up to a limit of 1 % of their nominal current of 33 kA. The total magnetic field \( B_t \) is the sum of a constant field \( B_0 \), which is set by the magnet console, and a variable field \( \Delta B \), which originates from the application of \( U_{\text{mod}} \) so that \( B_t = B_0 + \Delta B(U_{\text{mod}}) \). The relation between \( \Delta B \) and \( U_{\text{mod}} \) is supposed to be linear. The objective of our study was the validation of the linearity and the determination of the resolution of the modulation entry. This is important for user experiments as well as for the development of an NMR field-lock device.

For this purpose we performed \(^1\)H NMR measurements of \( \text{H}_2\text{O} \) at a 50 mm bore resistive magnet site. In order to overcome the low homogeneity of the resistive magnets, and to obtain a sufficient resolution, we only used the external Bitter part that provides a better field homogeneity. Furthermore, we took a small \( \text{H}_2\text{O} \) sample volume (1 mm\(^3\)), which was accurately positioned at the center of the magnet (better than 0.2 mm) in order to avoid line broadening due to off-center effects (second order gradient term of 6 ppm/mm\(^2\)). We measured \( B_t \) at constant set field \( B_0 = 6 \text{ T} \) as a function of \( U_{\text{mod}} \), which we first increased and decreased in steps of 10 mV from 0-100 mV and back. In a second experiment we applied steps of 1 mV from 0-10 mV and back. Using the information on the modulation entry from the power supply manufacturer (33 A/V) and the field factor of the bitter magnet (\( 3.408 \times 10^{-4} \text{T/A} \)), a modulation voltage step of 1 mV should correspond to a field step of 11 \( \mu \text{T} \).

At each value of \( U_{\text{mod}} \), we recorded the \(^1\)H NMR free induction decay (FID). We then calculated the spectral NMR line position \( f(U_{\text{mod}}) \) after Fourier transformation by a model-free approach using the first moment of the spectrum. The magnetic field values \( B(U_{\text{mod}}) \) were then determined by the relation \( B = f(U_{\text{mod}})/\gamma \), where \( \gamma \) is the gyromagnetic ratio of the \(^1\)H nucleus (42.574 MHz/T for \(^1\)H). For each value of \( U_{\text{mod}} \) we performed 16 measurements, and we averaged the spectrum 1024 times in order to eliminate the influence of field fluctuations on the first moment.

Selected spectra for the 1 mV steps are shown in figure 87(top panel), as function of a relative magnetic field axis. The spectra clearly show that 1 mV steps of \( U_{\text{mod}} \) are well resolved despite a spectral linewidth that exceeds the voltage step. This linewidth originates from field fluctuations, field drifts and the stability of the modulation power supply. In figure 87(bottom panel), we show the extracted first moments of the spectra as a function of \( U_{\text{mod}} \). The data clearly confirm the linear relation between \( U_{\text{mod}} \) and \( B \) with a slope \( k = 2.6 \text{ ppm/mV} \). Our results further show that the resolution limit of the modulation entry is at least 2.6 ppm at 6T. This resolution will be sufficient for the use of the modulation entry for an NMR field-lock. Our obtained slope value corresponds to an absolute field step of 16 \( \mu \text{T} \) upon applying a voltage step of 1 mV, which is 50 % above the expected value. This considerable difference requires a careful analysis of the modulation command chain.

![Figure 87](image)

**FIG. 87.** Top: \(^1\)H NMR spectra for different modulation voltages \( U_{\text{mod}} \) from 0-10 mV. A voltage step of 1 mV is clearly resolved in the NMR spectrum (thick solid and dashed lines). Bottom: Linearity and resolution of field modulation, the solid line is a linear fit providing a slope of 2.6 ppm/mV.

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Development of a $^3$He cryostat for measurements in DC magnetic fields up to 37T

Here we report the commissioning of a cold finger $^3$He insert developed in Toulouse and intended for specific heat and thermoelectric measurements under steady magnetic fields up to 37 T available in Grenoble.

![Figure 88](image)

**FIG. 88.** (Left) The $^3$He insert. (Right) With the insertion tube and the internal vacuum chamber removed showing (1) the 1K pot, (2) the spacer, (3) $^3$He chamber, and (4) the sample holder.

The cryostat (see figure 88) is equipped with an inner vacuum chamber containing the $^3$He injection and pumping line. In its upper part, this line is placed in thermal contact with a pot containing $^4$He liquid supplied from the main bath via a needle valve. The temperature of the pot is lowered to 1.4 Kelvin by pumping. The $^3$He gas injected into the line condenses on contact with the pot and accumulates in a chamber located at the lower end of the pumping line. Placed in the vacuum, the cold finger of the cryostat consists of a 6 mm diameter and 64 mm length silver rod M6 threaded on the vacuum side. At the wet side a 1.2 mm thick layer of silver powder is sintered over a length of 30 mm in order to increase the area immersed in the cold liquid and, therefore, enhance the heat exchange between liquid $^3$He and cold finger. The cold finger is then soft welded, at its middle, through the bottom end of the chamber.

A thermally insulating spacer made of fiberglass placed between the 1.4 K pot and the $^3$He chamber allows precise centering of the latter in the inner vacuum chamber.

As shown in figure 89(a), tests in Toulouse where performed in “one shot” mode with the help of a 18 m$^3$/h vane pump connected to the pumping line of the $^4$He pot allowing 0.15 mole of $^3$He to be condensed at 1.4 K in around 15 minutes. The liquid $^3$He bath was then pumped with the help of a 65 m$^3$/h vane pump down to 0.3 K. The temperature, measured using a calibrated Germanium sensor, was maintained over a period of 31.5 hours until the $^3$He bath dried up. The cooling power of the system, when pumped with the 65 m$^3$/h vane pump and through a 50 mm diameter and 3 m length pumping line is displayed in figure 89(b).

Delivered in July 2021, the system was tested once again in the Grenoble facility, and is now available for experiments on the resistive magnets. This project is an example of the type of synergy developed between the teams of the Grenoble and Toulouse sites.

![Figure 89](image)

**FIG. 89.** (a) Hold time of the $^3$He insert. Arrows indicates the end of condensation (start of pumping the $^3$He bath) and the drying up of the $^3$He bath. (b) Cooling power from 0.3 to 0.78 K.
Instrumentation developments for the Megagauss generator

Since the renewal of the capacitor bank in 2019, activities around the Megagauss (MG) installation have focused on the development of experimental techniques and a general improvement of measurement conditions, in particular as far as low temperatures, noise rejection and a better characterization of the field are concerned. The use of 30–40 T oscillating fields produced by reinforced single-turn coils in non-destructive mode has permitted an extensive testing and optimization of these new developments, while still providing realistic conditions as far as microsecond timescales and electromagnetic perturbations are concerned.

**THz-spectroscopy**: Figure 90 shows the result of preliminary cyclotron resonance measurements on a GaAs multiple quantum well using a 3.5 THz quantum-cascade laser, specially designed non-metallic hollow waveguides and a fast Ge:Ga detector. While the new setup is currently too noise-sensitive to resolve signal changes against perturbations caused by the MG generator’s high-voltage trigger on the initial up-sweep of the field, it clearly shows a single well-defined absorption line on the down-sweep and all further field oscillations. The principal technical objective for this experiment will therefore be an improved screening of electromagnetic perturbations similar to those already implemented for electric transport measurements.

**Electrical transport**: Electrical measurements of any kind are difficult to implement in MG fields, as signals are distorted by magnetic induction in uncompensated current loops, baseline shifts and noise originating from sparks and high-voltage trigger pulses. A consequent minimization and geometrical optimization of electrical circuitry, modulation frequencies around 800 MHz, and the design of high-voltage compatible metallic screens are the principal ingredients of an ongoing project to implement electric transport measurements in MG field. Figure 91 gives an impression of the new measurement site with improved screening and a $^4$He bath-type cryostat for temperatures down to 1.5 K which represents a novelty in MG fields.

**Field generation and characterization**: In addition to new experimental techniques, a numerical tool for simulating the electromagnetic, thermal and mechanical dynamics of single-turn coils (STC) has been developed since 2020. This project has given rise to 3 notable results; firstly, the field homogeneity in a $10 \times 10 \text{mm}^2$ (diameter × axial width) coil is estimated to be better than 1% in a 1.5 mm diameter sphere throughout the discharge; secondly, a 30% reduction of the axial width of a coil may produce 8% more field while still providing 1% homogeneity in a 1 mm sphere which is sufficient for most experiments; thirdly, the destructive effects observed in fields exceeding 150 T could be due to an effect similar to superheating. The increase of magnetic pressure on the current-carrying inner surface of a STC thus delays the sublimation of Cu during the rise of the magnetic field. Once the maximum field is crossed the pressure is released and sublimation takes place explosively. The effect becomes destructive at higher fields, as more conductor material sublimates at the moment when the coil has not yet sufficiently expanded to accommodate the vapor.

**FIG. 90.** Preliminary measurement of cyclotron resonance in a GaAs multiple quantum well during the down-sweep of the first and the up-sweep of the second field oscillation

**FIG. 91.** MG generator frontend featuring the new $^4$He bath-type cryostat. The white panel in the back is part of the aluminum screening that envelopes the current leads and separates the measurements site from the capacitor bank.

Magnet Development
The DC high magnetic field installation

We summarize important events which occurred this year for the 24 MW facility in Grenoble.

**Replacement of a 15 kV/550 V transformer**

In April, a leak in the tank of one of the two new 9 MW transformers has been confirmed (figure 92). As a result the control command chain was modified in order to pilot magnets with 3 out of the 4 AC/DC converters usually in operation. The transformer has then been replaced by one of the two old transformers that were kept as spare parts. The spare transformer was put back into operation in July, allowing researchers to take advantage of the maximum magnetic field in early summer.

**Hydraulics**

We have focused on improving our pump fleet and on optimizing the maintenance planning of our measuring instrument park. On our internal de-ionised water loop, we have installed on the pumps an automatic motor bearing lubrication injection system that should increase the life span. This system allows the injection of grease continuously during operation, with great precision on the quantity.

**Differentiated piloting mode**

For 5 months with 3 AC/DC converters in operation, we have implemented an energy efficient piloting mode (ecoNRJ) characterized by a differentiated piloting of each of the two sub-magnets. Namely, when a ramping of the field is requested, we first increase the electrical current that feeds the inner most magnet. An example of this new mode is given in figure 93 for a sweep to 36 T. We have added real time energy saving by using a new converter control mode. Since November 2021 this new operating mode has been set as the default mode on the high field magnets.

We have also upgrade the user interface as shown in figure 94. Among the changes, as the new control mode expends less energy while achieving the same field value, the interface shows the difference in energy between classical and new mode (power saved using eco mode 1.56 MW, near top right of figure 94).

![FIG. 92. Departure of the 9 MW 15 kV/550 V transformer for repair after the detection of a weakness in May 2021.](image1)

![FIG. 93. Evolution of the currents injected into each of the two sub-magnets during a linear sweep of the magnetic field up to 36 T on a resistive magnet site. As compared to the classical mode operation in which the two current ramps are kept equal, a reduction of electrical power of \( \approx 12\% \) is obtained.](image2)

![FIG. 94. Improved user magnet interface implemented to take into account the new piloting mode. On the upper left corner one can see the difference in the feeding currents of the two sub-magnets. Near the top right, the power saved in eco mode of 1.56 MW is indicated.](image3)

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Magnetic field quality under ecoNRJ operation of resistive coils

The energy consumption of resistive high field magnets is an ever growing concern. Recently, we have developed the ecoNRJ mode, that reduces energy consumption by up to 20% using the special architecture of the high field magnets. Namely, the two concentric Helix and Bitter sub-magnets can be independently powered and exhibit different contributions to the total field \( \vec{B}(r) \). Thus, for a given field value at the center, the current in each sub-magnet can be optimized to minimize the total electrical power. However, a possible drawback of this operation mode is the potential degradation of the magnetic field quality.

The total magnetic field \( \vec{B}(r) \) of a 24 MW high field magnet is the sum of the contributions of the Bitter and Helix sub-magnets, given by

\[
\vec{B}(r) = f_{\text{Hel}} I_{\text{Hel}} \vec{b}_{\text{Hel}}(r) + f_{\text{Bit}} I_{\text{Bit}} \vec{b}_{\text{Bit}}(r),
\]

where \( f_{\text{Hel}}, I_{\text{Hel}}, \vec{b}_{\text{Hel}}(r) \) and \( f_{\text{Bit}}, I_{\text{Bit}}, \vec{b}_{\text{Bit}}(r) \) denote the field factors, currents and normalized spatial field maps of the Helix or Bitter parts. Under normal operation both currents are equal to within a few percent. However, both coils have almost identical resistances, but considerably different field factors: \( f_{\text{Hel}} > 2 f_{\text{Bit}} \), depending on the Helix configuration. Therefore, a reduction of the total power is possible by operating the magnet with a current ratio \( R = I_{\text{Hel}}/I_{\text{Bit}} > 1 \), up to a certain threshold field \( B \), that is mainly defined by lifetime considerations of the Helix magnets, subject to very high currents and fields. At the very highest fields, the relative contribution of the Bitter coils increases, and the power saving is lost. Using data from two years of user operation for optimization, we have chosen \( R = 2.5 \), up to 80 % of the maximum Helix current in the ecoNRJ mode.

As the ecoNRJ mode changes the contribution of Bitter and Helix magnets to the total magnetic field, it might affect (i) the value of the magnetic field at the center of the coil, (ii) the spatial field distribution as well as (iii) the ramp rate \( dB/dt \). These parameters are very important for experiments that rely on precise values of the magnetic field, like nuclear magnetic resonance, or on spatial field distributions, such as field gradient magnetometers. A change of ramp rate at the threshold might induce spurious signals in high precision voltage measurements due to induced voltages. In order to quantify these effects we performed NMR studies of field values and field homogeneity as well as magnet supervision records for ramp rates.

(i) Absolute value of magnetic field; the magnetic field value for the user is provided by a precise current measurement by four direct current-current-transformers (DCCT, 10 ppm accuracy) and the field factor that is determined by a pick-up coil experiment (200 ppm accuracy versus NMR). The field is then calculated using the equation above. In order to check the influence of the ecoNRJ mode on the absolute field values, we performed \( ^{23}\text{Na} \) NMR experiments on a NaCl solution at 19.174 T in the 50 mm bore of a 24 MW resistive magnet. NMR spectra were subsequently recorded at identical nominal fields for normal (\( R = 1 \)) and ecoNRJ mode (\( R = 2.5 \)). The magnetic field values were determined from the NMR spectral line positions \( f_0 \) using the relation \( B = f_0/\gamma \), where \( \gamma \) is the gyromagnetic ratio of the nucleus (11.2625 MHz/T for \( ^{23}\text{Na} \)). The NMR field values for the ecoNRJ mode were found up to 5 mT lower than the field obtained by DCCT records and the field factors (figure 95). These deviations are within the precision of the command chain.

(ii) Spatial field distribution; Bitter and helix magnets have different geometries and, hence, different spatial field maps \( \vec{b}_{\text{Hel},\text{Bit}}(r) \). Therefore, the homogeneity of the field near the center changes when using the ecoNRJ mode. Using the known field maps, the leading second order term of the Taylor series expansion of the axial field profile near the center increases by 10 % for a 34 mm bore 24 MW resistive magnet for an ecoNRJ mode with \( R = 2 \). This behavior was confirmed by an experimental axial field profile at 24 T using an NMR experiment. This induced an increase of the field inhomogeneity across a standard sample (1 mm thickness) from 22 ppm to 24 ppm. This is negligible in comparison to other effects such as field fluctuations and drift or intrinsic line broadening by demagnetization fields.

(iii) Ramp rates; during a sweep the ramp rate above the threshold increases by 1.5 %. This is mainly due to the numerical approximation in the communication between the power supplies and the user interface. So far, it does not seem to be noticeable for users, but it can still be an issue for specific experiments and should hence be carefully monitored.

FIG. 95. Magnetic field values measured by DCCT and \( ^{23}\text{Na} \) NMR at 19.1974 T on a 50 mm bore resistive magnet for normal and ecoNRJ mode. An error of 5 mT between the DCCT and NMR fields is seen in the ecoNRJ mode, which is essentially due to the precision of the command chain.

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Materials for DC high magnetic fields

During the last few years, CuAg0.1wt.% has been replaced by CuAg5.5wt.% for cold sprayed polyhelix construction. Indeed, the CuAg0.1wt.% alloy was presenting a fatigue weakness which has led to damage during magnet operation. In the framework of the material development, other CuAg alloy grades have been investigated, namely CuAg2.75wt.% and CuAg11wt.%.

The powder to be sprayed has been bought from the Safina company. The distribution has a d10 of 15 \( \mu \text{m} \) and a d90 of 55 \( \mu \text{m} \). The production of tubes for characterization is done using the parameters for cold spray projection previously used for the CuAg5.5wt.% alloy. The oxygen level measured in the depositions are 150 ppm and 120 ppm for the CuAg2.75wt.% and CuAg11wt.% respectively.

Slices of 1.4 mm thickness are collected by wire cutting in the raw deposition. The slices are subjected to different heat treatments to investigate the mechanical and electrical properties reachable. Based on the micro-hardness evolution, a first series of heat treatments has limited the range of temperature to investigate: from 250°C to 325°C for the CuAg2.75wt.% and from 300°C to 375°C for the CuAg11wt.% alloy. The temperature for annealing is investigated by 25°C steps and the temperature is held during 4 h under vacuum.

![FIG. 96. Evolution of engineering yield strength and conductivity of CuAg2.75wt.% and CuAg11wt.% deposited by cold spray with different thermal treatments.](image)

As expected, the yield strength decreases with the increase of the temperature. At 20°C the yield strength of the materials are approximately 505 MPa and 575 MPa for the CuAg2.75wt.% and CuAg11wt.% respectively. Up to 150°C, the difference between these two alloys slightly decreases with a 50 MPa gap at this temperature. However, at 225°C the yield strength of both alloys are approximately the same with a value of 400 MPa. Thus, the slope of the decrease of the yield strength is higher for the CuAg11wt.% than for the CuAg2.75wt.% and this is particularly evident above 150°C. Such behaviour with an inflexion point of this curve has also been noted with the CuAg5.5wt.% alloy.

The evolution of the electrical conductivity and the yield strength in function of the temperature of the heat treatment is shown in figure 96. For both alloys, the couple of properties (yield strength;electrical conductivity) gradually evolves as expected: with an increase of the annealing temperature, the yield strength decrease and the electrical conductivity increase. The electrical conductivity is ranging from 51.6 to 55.2 MS.m\(^{-1}\) for the CuAg2.75wt.% and from 50 to 55.6 MS.m\(^{-1}\) for the CuAg11wt.% alloy. The yield strength is ranging from 420 to 560 MPa for the CuAg2.75wt.% and from 450 to 640 MPa for the CuAg11wt.%.

Based on previous calculation, the engineering yield strength should be above 500 MPa with a minimum of 50 MS.m\(^{-1}\). Indeed, with these properties, the integrity of the magnet should be ensured during operation. Thus, the selected heat treatments are the following: 4 h at 300°C for the CuAg2.75wt.% and 4 h at 325°C for the CuAg11wt.% alloy. The evolution of the yield strength of these heat treated materials has been evaluated by tensile test at high temperature, figure 97.

![FIG. 97. Evolution of the engineering yield strength with the temperature for the CuAg2.75wt.% [HT 4 h at 300°C] and the CuAg11wt.% [HT 4 h at 325°C].](image)

The CuAg11wt.% alloy is highly promising with the best couple of properties (yield strength;electrical conductivity). However, this material needs other qualifications before approval for polyhelix production. Indeed, this higher amount of silver requires a modification of the oxidation treatment before gluing and possibly the type of glue used between the turns of the helix.

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The Grenoble 43+T hybrid magnet - progress towards commissioning

The Grenoble Hybrid magnet combining resistive and superconducting technologies will be a modular user platform with the objective to deliver to the scientific community various continuous high magnetic field and flux configurations. For the first step, they range from 43 T in 34 mm diameter using 24 MW of electrical power down to 9 T in 800 mm diameter, when the superconducting outsert magnet is used alone. The ongoing upgrade of the electrical power installation first up to 30 MW, and possibly up to 36 MW later on, has been anticipated since the first design phase of the hybrid magnet with target to reach a magnetic field of at least 46 T in 34 mm diameter. Further studies are ongoing for this challenging step considering realistic worst-case scenarios to calculate ultimate forces for dimensioning the support structure. It will also be based on the first years of experience gained with the hybrid magnet operation up to 43T.

Strain and temperature gauges have been installed in the most critical parts of the structure and the superconducting coil to develop a precise understanding of the mechanical and thermal behavior of the system up to 43 T and define the optimum path for the generation of fields well above 45 T. In 2021, major milestones have been achieved for the final assembly phase of the hybrid magnet. At the beginning of the year, the superconducting outsert coil of 1100 mm aperture weighing 18 tonnes has been equipped with instrumentation wires, thermometers, heaters for the warm-up and strain gauges. Lifting tests were also performed and the measured elastic deformation of the flanges was found to be in agreement with mechanical calculations. After a thorough preparation phase including several insertion trials with a mockup in wood of the same dimensions, the outsert superconducting coil has been successfully inserted inside the He vessel the 5th of May. The closure welds of the He vessel containing the large bore superconducting coil have been made by the company SDMS following a strict procedure to avoid damage to the coil, the instrumentation wires and the electrical ground insulation.

The cryogenic line connecting the magnet cryostat to the cryogenic satellite (figure 98) has been delivered by Cryo Diffusion on the 30th of June. Its positioning in its final location on its support for the acceptance test has been successfully achieved the 7th of July after delicate lifting and handling operations.

The 1.8 K cold mass assembly, i.e. the closed He vessel containing the superconducting coil, was then placed on the magnet support ferrule. Unfortunately, the mounting of the outer vacuum chamber had to be performed several times from September to November 2021. The reason was a leak detected on the upper part of the internal radius connection between the top plate and the warm central tube. It required a detailed investigation to be properly located, identified and was then successfully repaired.

The next steps of the Grenoble hybrid magnet project can be summarized as follows. The pumping of the vacuum vessels of the magnet cryostat and cryogenic satellite is expected to start in February 2022. The beginning of the cooling of the superconducting magnet with a strict control of thermal gradients is planned for April or May 2022 and the superconducting magnet will be powered up in June or July 2022. This will then be followed by an evaluation program of the electromagnetic interactions between insert and outsert and the definition of the operating parameters that will allow safe operation of the hybrid magnet as a first step up to 43T and later, to fields well above 45T.

FIG. 98. Site dedicated to the hybrid magnet with the magnet cryostat assembly containing the large bore superconducting coil outsert, the cryogenic line ready for busbar insertion prior to its interconnection, and in the back, the cryogenic satellite producing the superfluid He.
The hybrid magnet combining resistive and superconducting technologies in construction at LNCMI-Grenoble has reached important milestones in 2021. After a thorough preparation phase, the outsert superconducting coil of 1100 mm aperture has been successfully inserted inside the He vessel (figure 99(a)). Then the closure welds of the He vessel have been realized by the company SDMS (figure 99(b)) following a strict procedure to avoid damage to the coil, the instrumentation wires and the ground electrical insulation. The dye penetration testing of the welds was successful as well as the pressure and leak tests.

The cold mass assembly weighting 23.4 tones, i.e. the enclosed large bore superconducting coil inside the He vessel, was then covered by the first set of superinsulation layers of the 4K thermal shield, before being installed on the support ferrule of the cryostat (figure 99(c)) for the next cryogenic steps.

FIG. 99. (a) Successful insertion of the superconducting coil in the He vessel. In the most constrained part at the bottom, the mechanical clearance with respect to the radius is equal to 0.4 mm. (b) Closure welding of the He vessel with the superconducting coil inside requiring a strict control of the temperature to not damage the electrical insulation (contractor SDMS). (c) Installation of the cold mass with its first layers of superinsulation on the support ferrule of the cryostat.
The Grenoble hybrid magnet - delivery of the cryogenic line and assembly tests

The cryogenic line of the Grenoble hybrid magnet connects the superconducting magnet cryostat to the cryogenic satellite producing the pressurized superfluid He. It is made by a super-insulated vacuum transfer line for cryogenic fluids of about 5.5 meter long, which contains also the superconducting busbars to power the superconducting magnet as well as about 700 instrumentation wires travelling either in the central helium pipe or in the vacuum chamber. The insulation vacuum of the cryoline is segmented by a vacuum barrier separating the vacuum of the magnet cryostat from the one of the cryogenic satellite. The cryogenic line has been design by CEA Saclay and built by Cryo Diffusion. It was delivered on the 30th of June. To be able to position the cryoline weighting 1.4 tones to its final location in between the cryogenic satellite and the magnet cryostat, a dedicated lifting tool (trepuchet) has been design and built by Barclet Rhône Alpes (figure 100(a)).

Assembly tests for bolts connections to the cryogenic satellite and magnet cryostat took about 2 days and allow some minor mechanical adjustments. For this, the cryoline was installed and fixed on its support foot bolted to the ground (figure 100(b)). Figure 100(c) shows the cryoline connected to the cryogenic satellite. Once the trial assembly was achieved, both ends of the cryoline was disconnected. It was then installed temporary in such a way to not interfering with the mounting of the magnet cryostat. The final assembly of the cryoline with its welded and bolt connections to the magnet cryostat and cryogenic satellite is expected starting in January 2022.

FIG. 100. (a) Delicate handling operation of the cryogenic line in the hybrid hall with dedicated lifting tool. (b) Fixation of cryogenic line to its support foot. In the background, the connection to the magnet cryostat. (c) View of the cryogenic line with in the background its connection to the cryogenic satellite.

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Grenoble hybrid magnet - assembly of the magnet cryostat and leak tests

The cryostat of the Grenoble hybrid magnet outsert is fitted with three thermal shields at 100 K, 40 K and 4.2 K, to intercept the heat transfer from 300 K to 1.8 K. They have been successfully assembled (figure 101(a-b)) together with the outer vacuum chamber (OVC) (figure 101(c)) prior to performing extensive leak testing with a He leak detector from Pfeiffer Vacuum.

Unfortunately, a leak was detected in the warm part of the vacuum chamber. It was precisely localized on the upper circular junction of the OVC with the internal warm bore. It was decided to disassemble the OVC in order to repair the identified defective part. The leak arose from a poorly glued assembly on the top plate of the OVC, which has now been replaced by a welded one. The OVC was then put back in place and the pumping restarted.

The last leak test still reveals a tiny leak signal $\leq 3 \times 10^{-8}$ mbar.l/s, which is three times larger than the technical specification. This value was nevertheless judged to be acceptable. To reduce this measured value, a much longer pumping duration would have been required. It was therefore decided to go ahead with the cryostat assemblage, and the welding of the quench line.

FIG. 101.  (a) Cryostat of the hybrid magnet showing the assembly of the 40 K thermal shield. The cryogenic line connecting the magnet cryostat to the cryogenic satellite is visible in the background.  (b) Last superinsulation layers covering the 100 K thermal shield installed prior to the mounting of the outer vacuum chamber.  (c) Mounting of the outer vacuum chamber (OVC).

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Pulsed magnets

The majority of the nondestructive magnetic fields pulses are realized thanks to 60 and 70 T magnets that always equipped four or five of the seven experimental cells in Toulouse. The design of these magnets has evolved during the last decade and is now mature. All the coils produced each year for a given category of field has the same dimensions, wire properties and section, reinforcement quantity and a very reproducible lifetime. In order to decrease the cost and the duration of production we have decided to redesign these two magnets to simplify the mechanical subparts and the winding process. In figure 102 are shown the two standardized mechanical assembly of the 60 and 70 T coils. The next step will be the progressive replacement of the old systems by the new one at each coil failure.

The new magnetic dipole made with a copper foil designed for the vacuum magnetic birefringence experiment (BMV) has been commissioned this year and used for preliminary tests. Connected to one module of the 6 MJ capacitor bank this coil can generate more than 10 T transversally to the 82 cm optical path. About 1000 pulses between 1 and 10 T have been realized in 2021. Three values of capacitance of the bank, 10, 5.5 and 2.8 mF have been used to check the effect of the pulse length on the measurements in the range 5.8 to 2.9 ms of rise time. The first measures tend to show that shorter pulses could be better for the measurement sensitivity. We are currently studying the feasibility of a connection of this magnet to the 1 MJ capacitor bank that can allow to generate pulses with 4.3 ms rise time instead of 5.8 ms with the 6 MJ generator.

Capacitor banks

The three new capacitor banks are in operation since July 2019 for the 6 MJ and October 2020 for the 14 MJ and the 1 MJ and more than 12000 pulses have been made without any major bug. Magnetic fields above 70 T and thus, generated with dual coil systems are available since November 2020. The increase of the total available energy from 14 to 21 MJ, planned in 2021, has been delayed, the time to evaluate the possible modifications of the capacitor banks routing necessary to connect the BMV coil to the 1 MJ capacitor bank.

In the framework of our collaboration with the Laboratoire pour l’Utilisation des Lasers Intenses started in 2013 we pursue our effort to provide high magnetic fields that can be used in high power lasers for laboratory astrophysics experiments. In addition to the pulsed magnets that were energized originally by a capacitor bank provided by the HLD we decided to build a new generator with 2.5 times more energy. While continuing experiments at LULI2000 facility at the École Polytechnique, the commissioning of the multi-terawatt laser Apollon at the “Orme des meurisiers” offers new opportunity for laboratory astrophysics and having a second generator will avoid transporting the HLD bank between the two facilities. This new capacitor bank, visible in figure 103, stores 86 kJ that can be released in less than 40 μs every minute. Thank to this high power, up to 2.21 GW and current, up to 220 kA, split coils with axial and radial accesses of about 1 cm in diameter will generate 60 T at room temperature.

FIG. 102. CAD view of the new mechanical assembly of, on the left, the new 70 T coil and, on the right, the new 60 T coil. Greenish yellow pieces are made with FR4 or G11 glass fiber/epoxy composite and are machined in the laboratory on a dedicated numerical milling machine.

FIG. 103. Photo of the new capacitor bank dedicated to laboratory astrophysics experiments.
Upscaling the production of Ag-Cu composite wires for non-destructive pulsed magnetic field applications

The aim of this work is the development of new high-strength conductors for the production of the next-generation of non-destructive electro-magnets. The research consortium is composed of one industrial partner and four academic laboratories. Each partner provides complementary expertise in different areas:

- powder metallurgy process, powder mixing, spark plasma sintering (CIRIMAT)
- powder atomization, cold spray (LERMPS)
- severe plastic deformation (LNCMI-T)
- fine microstructural and mechanical characterization (Institut Pprime)
- high stress and high strain rate environments (I-Cube Research)
- testing of magnets (I-Cube Research, LNCMI-T, LNCMI-G).

We aim to produce high-strength nano-structured Ag-Cu conductors, with ultimate tensile strength over 1 GPa at room temperature and electrical conductivity higher than 90% of the pure copper value. Indeed, these properties fulfill industrial and scientific challenges;

- long lifetime magnets/coils for industrial Magnetic Pulse Forming and Magnetic Pulse Welding,
- high pulsed non-destructive magnetic field beyond 100 Tesla with long pulse duration, beyond 10 milliseconds, for scientific users,
- steady magnetic (DC) fields beyond 38 Tesla for scientific users.

In the case of high pulsed magnetic field, coils are wound with high strength conducting wires. In our previous studies we have developed a preparation method of Ag-Cu composite wires by powder metallurgy, spark plasma sintering (SPS) and wire-drawing. We applied wire-drawing to SPS precursors with 8 mm diameter to obtain samples of Cu-Ag wires with diameters ranging from 0.2 to 1 mm for the characterization and the understanding of the deformation mechanisms. However, in order to produce coil prototypes a change of scale is necessary. A twenty-fold increase in the volume of the cylinder would lead to the following dimensions: 24 mm in diameter and 60 mm in length.

The main risk is that the macroscopic properties will be modified during the upscale, no longer fulfilling the initial specifications. Increasing the volume of production needs some adaptation in the process compared to the preparation of smaller cylinders. In order to facilitate the scale up, we decided to increase the sample volume in two steps. About 150 g of powder is necessary to sinter 24 mm in diameter and 30 mm in length cylinder (figure 104). The powder is heated at 160 °C in flowing H₂ to reduce any copper oxide present at the surface of the Cu grains and also to obtain a cohesive, pre-sintered powder because this was found to be favourable for the subsequent consolidation. The powder is consolidated by SPS at 400 °C during 5 minutes. A uniaxial pressure of 25 MPa is applied to help the densification (96.2%). These values were found convenient because a too high density hampers the deformability of the cylinder, resulting in sample breakage during wire-drawing. The SPS precursor cylinder diameter is reduced from 24 mm to 0.2 mm by wire-drawing at room temperature. This is representing a cross sectional reduction of 99.993%. At the final diameter (0.2 mm) it is possible to obtain a length of 432 meters of wires. Samples with diameter ranging from 0.2 to 3 mm were taken for future mechanical, electrical and microstructural characterizations.

FIG. 104. Photograph of a 24 mm precursor cylinder after the first scale up step (on the left) and a 8 mm precursor cylinder having been used for the preliminary work on the composite Ag/Cu wires by SPS and wire drawing (on the right).
Composite versus alloyed silver-copper conductors for high magnetic field applications

The aim of this research is to prepare and characterize high-strength, high-conductivity silver-copper (Ag-Cu) composite wires obtained by powder metallurgy, spark plasma sintering (SPS) and room-temperature wire-drawing. These wires will then be compared to Ag/Cu alloyed conductors.

Ag nanowires (length 30 µm, diameter 200 nm) were prepared at CIRIMAT and add to a commercial Cu powder (1 µm). The 1 vol. % Ag-Cu powder was heated at 160°C under H₂ to reduce any Cu oxide present at the surface of the Cu grains and also to obtain a cohesive, pre-sintered powder because this was favourable for the following consolidation. The 1 vol. % Ag-Cu powder was sintered by SPS at 400 or 600°C.

According to the Cu-Ag phase diagram, the sintering of the 1Ag-Cu powder at 600°C (the solubility of Ag in Cu is about 2.4 vol. % Ag) leads to the formation of an Ag/Cu alloy. By contrast, the sintering at 400°C, (the solubility of Ag in Cu is below 0.1 vol. %), allows to obtain a composite microstructure with pure Ag nanowires dispersed in a pure Cu matrix (figure 105).

Even if this change of microstructure seems tiny, the formation of Ag/Cu alloy during SPS at 600°C, although it did not really affect the ultimate tensile strength of the wires, significantly degraded their electrical resistivity compared to the composite wires prepared from a cylinder sintered at only 400°C (figure 106). We have demonstrated that the formation of an Ag / Cu alloy, although very limited and with a low Ag content, leads to a significant reduction in electrical resistivity. This confirms the importance of preventing alloying during preparation of Ag-Cu samples in order to avoid a dramatic effect on electrical resistivity and thus meet the requirements for high magnetic fields.

Thus, Ag-Cu composite materials could also find use in DC magnetic field magnets. Some resistive DC magnets are made up of Ag/Cu alloy (prepared by Cold Spray) containing 5.5 vol. % Ag in the case of polyhelex magnet or Ag/Cu sheets (prepared by melting of Cu and Ag followed by solidification) in the case of Bitter magnet. For an equivalent Ag content and an equivalent deformation rate, Ag/Cu alloy wires and sheets present similar properties (965 MPa / 2.4 µΩ.cm and 1050 MPa / 2.3 µΩ.cm). The resistivity of our 1 vol. % Ag-Cu composite wires is ranging from 1.93 to 2.02 µΩ.cm. Reducing the resistivity of Ag/Cu materials used in DC magnets, without compromising the mechanical properties is a major issue in view of reducing the electricity consumption as well as increasing significantly the attainable magnetic field of DC facilities.

For further details please see [Tardieu et al. Journal of Materials Science 56, 4884 (2021)].

FIG. 105.  SEM image of the transverse section showing Cu (grey), Ag grains (white) and porosity (black) for: 1 vol. % Ag-Cu cylinder sintered at (a) 400°C and (b) 600°C.

FIG. 106.  Ultimate tensile strength versus electrical resistivity at 293 K for Cu SPS, 1Ag-Cu/400 and 1Ag-Cu/600 composite wires, Ag-Cu alloy wires (21 - 25 vol. % Ag), Ag-Cu cold spray alloy (5.5 vol. % Ag), Ag/Cu alloy sheet (5.7, 12 and 21 vol. % Ag).

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Development of mechanically robust magnetoforming coils with a long lifetime

Since 2013, the laboratory has been collaborating with the Toulouse company Bmax / I Cube Research to design magnetoforming coils with an ever longer lifetime. This is a crucial feature for the use of electromagnets on an industrial scale, that is, the manufacture of parts in large quantities with extreme precision, and at a controlled cost.

To generate very strong magnetic fields, it is necessary to use conductors that combine good electrical conductivity with very high mechanical strength. For this purpose, the LNCMI therefore has its own wire drawing workshop and is able to manufacture in-house the conductors necessary for the production of these coils which can withstand non-destructive and long-lasting magnetic fields (several tens of milliseconds) of up to 98.8 T. It is this expertise that attracted Bmax / I Cube Research, specialist in magnetoforming.

Magnetoforming is a technique that makes it possible to manufacture parts with shapes that could not be obtained by other methods, extremely precisely and at reasonable costs. The pulsed electromagnets that we design generate very short and strong magnetic field pulses, which induced a current in the metal part to be shaped. This part is then projected at very high speed onto a mold of which it takes the form.

This is direct magnetoforming, which differs from the indirect process where a hammer set in motion under the effect of magnetic forces hits the material to be modeled and then sent to the mold. The operation takes place in a few microseconds, and offers a level of precision in the shape of the parts that can meet the expectations of very demanding sectors such as luxury, aeronautics and automotive industry.

However, with such pulsed electric currents, typically a few tens of kiloamps in about one hundred microseconds, the electromagnets that make the process possible are subjected to enormous magnetic, mechanical and thermal stresses. The challenge for Bmax / I Cube Research is to use coils made from conductive materials sufficiently resistant to these stresses, to be able to be used on an industrial production line, without having to be frequently replaced. From a few hundred pulses realized a few years ago, electromagnets such as the one shown in figure 107 are able to withstand now more than 30,000 pulses, which is about a 50-fold improvement in the lifetime.

Bmax / I Cube Research will use long-life electromagnets developed by LNCMI for industrial applications, and intends, thanks to these tools, to conquer new markets.

FIG. 107. Photo of a prototype flat magnetoforming coil. During operation, an aluminium hammer of about one kilogram, placed on the top face of the coil, is projected at a speed of 14 m/s in less than 100 μs onto the piece to form, which is then pressed onto the mold.

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G. Avrillaud, R. Pecquois, P. Thouêt (Bmax / I-Cube Research, Toulouse)
PhD theses 2021

1. Banan Kerdi
   *Transport quantique des trous dans une monocouche de WSe$_2$ sous champ magnétique intense*
   Doctorat de l’Université Paul Sabatier Toulouse III
   Thèse soutenue le 5 février 2021

2. Sanu Mishra
   *Experimental investigation of Ce-115 and related compounds in high magnetic fields*
   Doctorat de l’Université Grenoble Alpes
   Thèse soutenue le 24 mars 2021

3. Piotr Kapuściński
   *Fine structure and Rydberg series of excitons in transition metal dichalcogenides*
   Doctorat de l’Université Grenoble Alpes, Wroclaw University of Science and Technology et Ecole Polytechnique de Wrocław, Pologne
   Thèse soutenue le 21 décembre 2021
Habilitations à diriger la recherche 2021

1. William Knafo
   *Magnetism of heavy-f-electron metals*
   Université Toulouse III
   Soutenue le 9 avril 2021

2. François Debray
   *Contribution au développement des aimants pour champs magnétiques intenses dans le domaine des matériaux, de la thermique et de la gestion de l’énergie*
   Université Grenoble Alpes
   Soutenue le 12 juillet 2021
List of Publications 2021


[62] Lorenzo Poggini, Erik Tancini, Chiara Danielli, Andrea Luigi Sorrentino, Giulia Serrano, Alessandro Lunghi, Luigi


List of patents 2021

1. *Procédé et dispositif de caractérisation de propriétés magnéto-chirales d’un échantillon par spectroscopie de résonance paramagnétique électronique*
   
   French patent n° FR2105909, first deposed 04/06/2021
   
   Inventors: Geert Rikken, Manuel Donaire

2. *Copper-silver composite material*
   
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