HIGHLIGHTS 2022

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Welcome to the 2022 issue of Institut Néel Highlights.

Institut Néel is a CNRS laboratory (Unité Propre de Recherche) with strong links with Université Grenoble Alpes. Our main stream research focuses on fundamental science in condensed matter physics and chemistry to push forward the frontiers of knowledge and look for new properties or systems. Since physics and chemistry are the basis of many societal issues, the laboratory also develops top level activities and engineering at different interfaces. The whole benefits from the historically strong technological skills of the laboratory which we are committed to maintain and further develop.

The theoretical and experimental activities of Institut Néel cover a wide variety of subjects. A major asset of the lab is to cover the entire range from the synthesis of materials, their patterning or functionalisation, to the study of their properties and the development of systems based on them (*pages 4, 5, 10*). Quantum science, which for us brings together quantum matter (*pages 8, 9, 12, 14*) and quantum technologies (*pages 6, 13*) is a second pillar of the laboratory. Eventually, physics at the interface with biology (*page 5*), astrophysics, very low temperature physics and its associated technological research (*pages 7, 11, 15*) and instrumentation - even in extreme conditions – illustrate how our developments are also of interest to other disciplines. Diversity in the topics but also in training and origins of the staff is a great strength of Institut Néel, which we also strive to preserve as well as possible.

We consider that valuing results is one of our missions. Beyond the strong links that we are developing with industrial partners (*page 11*) this year two more startups have been created, in quantum technologies. A total of six startups are now hosted by the laboratory. They exploit the research performed in the laboratory and also offer an interesting outlet for some of our PhDs.

Popularizing science, explaining what a scientific approach is, is a major challenge for our society. This is what the members of the laboratory who took part in the action that you will discover at the end of this booklet (*page 16*), attached themselves to. The result was acclaimed by the laboratory staff during our convivial day in September

Fundamental research as well as developing applications require time and we are happy to present in these few pages some of our 2022 achievements. Giving an exhaustive overview of NEEL activities in a few pages is not possible and further information can be obtained browsing our website https://neel.cnrs.fr.

On behalf of all the laboratory staff I wish you a pleasant reading !

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Laurence Magaud,
Director

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DIRECTOR OF PUBLICATION
Laurence Magaud

EDITORS
Jan Vogel, Virginie Simonet, Patricia Derango,
Benoît Chabaud, Hermann Sellier

PRODUCTION MANAGERS
Nathalie Bourgeat-Lami, Florence Fernandez

LAYOUT
Florence Fernandez

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Contents

Watch your steps while growing nanowires

Motivated by the emergence of new opto-electronics devices such as single-photon emitters, electron microscopes equipped with effusion cells have been developed in order to monitor *in situ* the molecular beam epitaxy of complex semiconductor nanostructures. Here we describe our results on the growth of II-VI nanowires obtained on the Nanomax setup in Palaiseau.

The development of semiconductor nanostructures has been achieved through a good control of their fabrication, for instance by Molecular Beam Epitaxy (MBE). For a long time, this technique has been limited to the growth of planar structures such as quantum wells, inducing a spatial confinement of charge carriers in the perpendicular direction. New challenges, such as the realization of single-photon emitters for quantum communications, require a stronger confinement in two or three directions, obtained by making nanowires and quantum dots.

The growth of nanowires is usually seeded by a liquid droplet of a few nanometers, which determines the diameter of the nanowire. During the growth, quantum dots can be inserted by changing abruptly the composition of the molecular beam. This "Vapor-Liquid-Solid" mechanism combines a vapor phase in the beam, a liquid phase in the droplet, and a solid phase in the nanowire. At the seed-nanowire interface, the growth is supposed to take place through the nucleation of a one-monolayer step and its propagation along this interface. For years, this was a mere assumption, but now this process can be observed *in-situ* in a modified electron microscope, such as the Nanomax setup operated in Palaiseau and first applied to GaAs nanowires by our colleagues from the Centre for Nanoscience and Nanotechnology.

In our group, we are interested in II-VI semiconductors combining a metal such as Zn or Cd (column II of the Mendeleev table) and a chalcogen such as Se or Te (column VI). These materials are particularly interesting as active optical elements, for light emission or photovoltaics. II-VI nanowires can be grown with a gold seed which is solid and crystalline instead of liquid: this is the "Vapor-Solid-Solid" growth, which is expected to provide sharper interfaces when inserting a quantum dot. Observing the growth of ZnTe nanowires at Nanomax revealed two original aspects: the role of the lattice mismatch at the nanowire-seed interface, and a self-regulation of the step dynamics.

Gold and ZnTe crystals feature the same Bravais lattice, but the ZnTe unit cell is 3/2 larger than that of gold. When the gold monolayer located at the interface is progressively replaced by a ZnTe monolayer, a strong mismatch strain appears at the step, which creates a barrier against the formation of such a step.

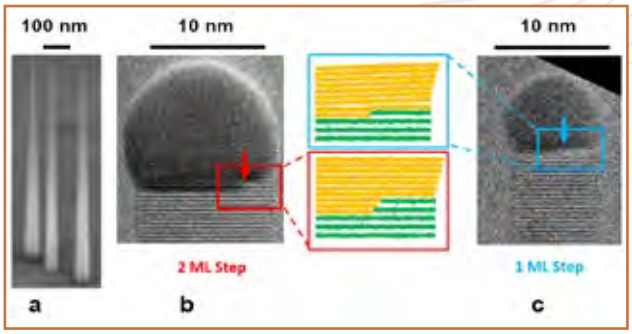


***Fig. 1:** a) ZnTe nanowires grown in our MBE cluster; (b) Nanomax image showing the gold seed at the top of the ZnTe nanowire, with a two-monolayer step at the interface; the step (indicated by the arrow) propagates from right to left; (c) idem, with a one-monolayer step. The schemes in-between display the Au atomic planes (in yellow) and the ZnTe planes (in green) for the two configurations.*

Alternatively, a different step configuration, made of 2 ZnTe monolayers facing 3 gold monolayers, shows practically no mismatch and a negligible strain energy, making this configuration more favorable. And indeed, our Nanomax investigation revealed that the growth proceeds essentially by the nucleation and propagation of such a two-monolayer step (*Fig. 1* and movie *in further reading*).

Another result concerns the growth rate, determined by the cycle time for step nucleation and step propagation. Since the nucleation is a random process, it could strongly affect the growth rate. Fortunately, a late nucleation induces an excess of Zn concentration in the seed, and thus a faster propagation. As a result of this compensation, fluctuations in the growth rate are efficiently reduced. And this is good news!

FURTHER READING

"Regulated Dynamics with Two-Monolayer Steps in Vapor-Solid-Solid Growth of Nanowires",
E. Bellet-Amalric, F. Panciera, G. Patriarche, L. Travers, M. Den Hertog, J.-C. Harmand, F. Glas & J. Cibert,
ACS Nano **16**, 4397 (2022).
Movies can be downloaded in the Supporting Information of this Reference

CONTACTS

Edith Bellet-Amalric
edith.bellet-amalric@cea.fr
Joël Cibert
joel.cibert@neel.cnrs.fr

Organic nanocrystals for fluorescence bioimaging

Among the powerful medical imaging techniques developed so far, fluorescence imaging appears as the one that may provide the best spatial resolution — close to the micrometer — without using hazardous radiations nor expensive equipment.

In particular, two-photon fluorescence imaging offers strong advantages, as the simultaneous absorption of two photons by the fluorophore (a fluorescent molecule or crystal injected in the biological tissue under observation) only occurs at the focal point of the excitation beam, which allows imaging in three dimensions. Furthermore, since two photons are absorbed simultaneously to induce a transition from the ground state to an excited state, the excitation wavelength with respect to one-photon absorption is shifted from the blue to the near infrared region. The lower photon energy reduces damage caused to, and autofluorescence emitted by, the tissue.

The overall brightness of two-photon fluorophores in general is low, which makes this technique difficult to implement with molecular compounds. Concentrating thousands of molecules into a single nano-object such as an organic nanocrystal should help solving this issue. However, aggregation of the fluorophores usually leads to quenching of the fluorescence, unless molecules are carefully designed to emit in the crystal state. Such molecules are engineered by our collaborators at the Laboratory of Chemistry of ENS Lyon. The aim of our research is to develop synthetic procedures to form organic nanocrystals, naked or embedded in a silicate matrix, to be used in two-photon fluorescence imaging.

We have developed an experimental setup to produce silica-coated organic nanocrystals in a single step, using a spray-drying technique (rapid drying of liquid droplets through a lamellar flux of hot gas). The obtained nanoparticles, initially polydisperse in size (40-400 nm), could be separated, selected and functionalized by poly(ethylene glycol) chains to enable a good dispersibility and a size distribution centered at 170 nm. Interestingly, the obtained colloid was stable in simulated body fluid and in the presence of proteins, which allowed for further use for in vivo imaging of the brain vasculature of mice. The nanoparticles could be seen as very bright spots by two-photon fluorescence imaging, under near infrared excitation (*Fig. 1*).

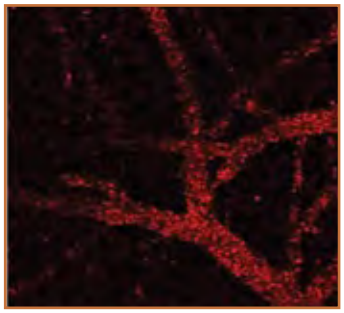


Fig. 1: *Two-photon fluorescence image of the vasculature of the brain of a mice using silica-coated organic nanocrystals covered with polyethylene glycol. Red dots arise from individual nanoparticles.*

To further develop this original type of nanoparticles, we have designed an original nanocrystallization setup, which uses the ability of ultrasounds to fragment a stream of organic solution which is injected in water, and to accelerate the nucleation of nanocrystals within micron-size droplets (sono-crystallization process, *Fig. 2*). Interestingly, we obtained micron-long needles for a particular organic fluorophore, whose crystallinity was characterized in detail by electron and X-Ray diffraction, and using the state-of-the-art technique of Dynamic Nuclear Polarization-enhanced Nuclear Magnetic Resonance developed at CEA Grenoble. Further mechanistic investigation revealed that the micron-long needles result from a fast evolution of smaller organic nanoparticles, which also feature an excellent crystallinity. This nanocrystallization technique is now being applied to obtain nanocrystals from a wide range of organic molecules for application in bioimaging or photodynamic therapy.

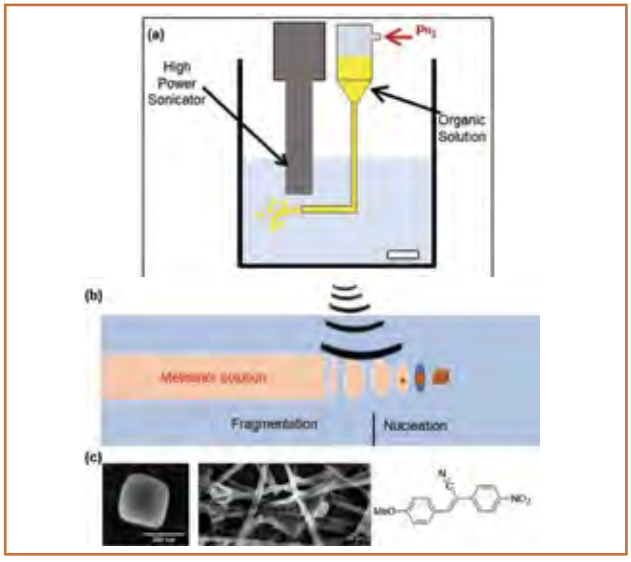


Fig. 2: *(a) Original setup developed for the nanocrystallization of organic compounds under the application of ultrasound (sonication); (b) Mechanism of sono-crystallization: The stream of organic solution injected in water is fragmented by the ultrasonic waves, which also favor the nucleation of the organic nanocrystals within the droplets. (c) Nanocubes and needles obtained from the CMONS dye (scalebars 200 nm).*

CONTACTS

Xavier Cattoën
xavier.cattoen@neel.cnrs.fr
Fabien Dubois
fabien.dubois@neel.cnrs.fr

PhD student:
Shridevi SHENOI PERDOOR

FURTHER READING

"Ultrabright Silica-Coated Organic Nanocrystals for Two-Photon In Vivo Imaging", S. Shenoi-Perdoor *et al*, ACS Appl. Nano Mater. **2020**, 3, 11933–11944. https://dx.doi.org/10.1021/acsanm.0c02499

"Sonocrystallization of CMONS Needles and Nanocubes: Mechanistic Studies and Advanced Crystallinity Characterization by Combining X ray and Electron Diffractions with DNP-Enhanced NMR ", X. Cattoën *et al*, Crystal Growth & Design **2022** 22, 2181-2191. https://dx.doi.org/10.1021/acs.cgd.1c01246

Quantum interferences reveal the 4e charge of Cooper quartets

Building Josephson junctions with three or more superconductors connected through the same contact opens prospects for drastically new physics. A recent experiment at Harvard University, inspired and interpreted by theorists in Institut Néel, brings decisive advances in the understanding and use of multi-terminal Josephson Junctions.

Cooper pairs of electrons, where two electrons are connected due to electron-phonon interactions, are the building blocks of superconductivity. Connecting two grounded superconductors with an insulating or metallic region forms a so-called Josephson junction that features a DC current induced by a phase difference between the Cooper pair wave functions at both sides of the junction. It also allows demonstrating the 2e charge of Cooper pairs in a SQUID (Superconducting Quantum Interference Device) geometry. Recently, it has been shown that connecting three superconductors together, biased with suitable voltages, induces new "quartet" modes within the junction. Those modes involve two entangled Cooper pairs and induce DC currents, contrary to Josephson AC currents in usual two-contact junctions when a bias voltage is applied.

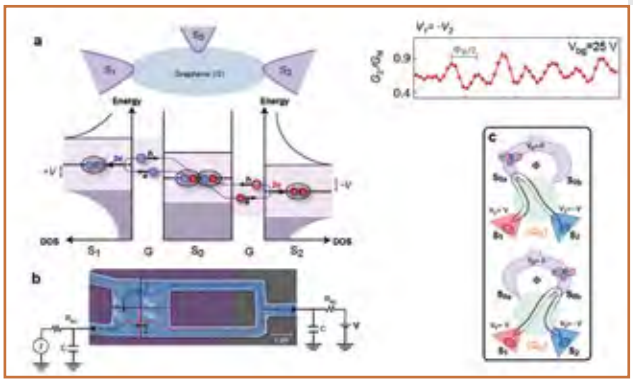
An experiment performed at Harvard University along the proposal of theorists in Institut Néel, has brought fundamental insights into this phenomenon. In its design, one of the terminals consists of a pierced loop, where periodically modulated interferences are made possible by a magnetic field. It also investigates for the first time the bias voltage dependence of the DC quartet current. Probing the voltage sensitivity would not be possible in the standard two-terminal DC Josephson effect that appears only at zero voltage. Both flux and voltage dependence of the quartet "critical" currents allow to draw the following conclusions:

First, the basic period of the flux modulation is half a flux quantum, hc/4e instead of hc/2e as expected for a Cooper pair current. This is a fingerprint of the fact that quartets cross the junction by connecting all three superconductors.

Second, the quartet critical current depends in a non-monotonous way on the voltage and its flux modulation may acquire a phase offset of π. This is counterintuitive, as it means that the quartet current can be larger when the loop is pierced by half a flux quantum than by zero flux. In a standard junction, destructive interferences would instead decrease the Josephson current in that case.

An explanation has been given to this anomalous flux and voltage variations by using field theory models suited to non-equilibrium transport. In a junction at zero bias, Andreev levels form within the superconducting gap. Any small applied voltage induces transitions between these levels, in a way similar to irradiation by a microwave: resonant transitions sharply reduce the current. The situation is more complex here, due to the coexistence of a DC mode and AC oscillations, but the physics is somewhat related.

This experiment confirms that voltage is a new "knob" to control DC Josephson currents. It also suggests other ways of using interferences to probe quartet and other multimode currents in Josephson multi-junctions (as a quartet SQUID). More generally, it opens a new chapter in the Josephson effect, which in the future will contribute to quantum engineering.



(Top, left) *Sketch of the graphene three-terminal junction, and energy diagram showing the basic quartet process involving four Andreev reflections. Two pairs are emitted from the central (grounded) superconductor, one in each of the biased terminals.*
(Bottom, left) *Photograph of the real device containing a loop pierced by a magnetic flux in the grounded terminal.*
(Top, right) *Flux modulation of the circuit conductance showing the hc/4e periodicity.*
(Bottom, right) *Quartets emitted from each of the two grounded terminal branches interfere which reveal their charge 4e.*

FURTHER READING EXPERIMENTAL WORK

"Evidence for 4e charge of Cooper quartets in a biased multi-terminal graphene-based Josephson junction",
K.-F. Huang, Y. Ronen, R. Mélin, D. Feinberg, K. Watanabe, T. Taniguchi & P. Kim, Nature Communications, **13**, 3032 (2022).
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FURTHER READING THEORETICAL SUPPORT

"*Multiterminal ballistic Josephson junctions coupled to normal leads*",
R. Mélin, Phys. Rev. B **105**, 155418 (2022),
https://journals.aps.org/prb/abstract/10.1103/PhysRevB.105.155418

CONTACTS

Régis Melin
regis.melin@neel.cnrs.fr

"NewCooler" Demagnetization Refrigeration

Nuclear demagnetization refrigerators (NDR) can reach 100 picokelvin, which is the lowest temperature achieved in condensed matter.

Figure caption: *Aluminum is normally covered by a tenacious oxide that inhibits low resistance contacts with other materials. To obtain this pair of Al/Cu joints with total resistance of less than 3 nOhms, the Al oxide was plasma etched in an evaporator and then a gold layer was deposited without breaking vacuum.*

At present, only "single-shot" NDR are available, with a hold-time limited to weeks. Cryogen-free models, which are not immersed in a liquid helium bath and offer more experimental space, have even shorter autonomies. With support of the ERC (UNIGLASS) and CNRS *prématuration* (NewCool), the Ultra Low Temperatures research team and the Cryogenics, Bulk Crystal Growth and Nanofab technological groups, all at Institut Néel, are developing a cryogen-free NDR that will maintain temperatures in the microkelvin range indefinitely. This is well below the base temperature of other continuous refrigerators.

The refrigerant of a NDR is a metal, usually copper, containing nuclei with non-zero spin. First, a magnetic field is applied to the Cu while it is in contact with a precooler at ~10 mK, causing the spins to align with the magnetic field. Then the Cu is thermally isolated from the precooler and the applied field is decreased.

The thermal isolation implies that the degree of alignment of the spins remains constant. Thus the temperature of the spins ideally decreases in proportion to the field. Once the desired temperature is achieved, heat leaking into the refrigerator is balanced by further demagnetization. A conventional NDR must return to the precooler temperature for recycling when the applied field reaches zero. In contrast, our continuous NDR will cycle between two nuclear refrigerants so that the sample remains below 1 mK indefinitely.

A superconducting heat switch is used to thermally connect and disconnect each refrigerant to the sample. The heat switch relies on the fact that, well below the critical temperature T_c of a superconductor, only phonons contribute significantly to its thermal conductance. This is because the density of electronic quasiparticle excitations, which transport heat, decreases exponentially as the temperature is lowered below T_c. Thus, for $T << T_c$, the thermal conductance of superconductors can be "switched on" by applying a magnetic field that is large enough to destroy the superconductivity.

The cooling power of the continuous NDR is limited by the thermal resistance of the closed heat switch: the smaller this quantity, the more rapidly the refrigerator can be cycled. The equivalent electrical resistance must be in the nOhm range. Few heat switch designs achieve such a low resistance, and many employ materials that are difficult to obtain, have an inconveniently high critical magnetic field, or are mechanically weak. We have demonstrated a superconducting aluminum heat switch with a simple design and five times lower normal state thermal resistance than the previous state of the art (see Figure). This low thermal resistance will facilitate the construction of a powerful continuous NDR compatible with cryogen-free dilution precoolers, thereby extending the benefits of cryogen-free refrigerators to the microkelvin temperature range.

CONTACTS

Andrew Fefferman
andrew.fefferman@neel.cnrs.fr
Sébastien Triqueneaux
sebastien.triqueneaux@neel.cnrs.fr

Postdoc:
Matthias Raba

FURTHER READING

"Superconducting aluminum heat switch with 3 nOhm equivalent resistance", J. Butterworth, S. Triqueneaux, Š. Midlik, I. Golokolenov, A. Gérardin, T. Gandit, G. Donnier-Valentin, J. Goupy, M. Keith Phuthi, D. Schmoranzer, E. Collin & A. Fefferman, Review of Scientific Instruments (**2022**).

Does $(TaSe_4)_2I$ really harbor an axionic charge density wave?

The axion was first conceived in particle physics as a hypothetical particle, whose existence would explain why high accuracy measurements did not detect a neutron electrical dipole, within a limit 10 orders of magnitude lower than the expected theoretical value. It was later realised that if their mass was in a certain range, axions could also significantly contribute to dark matter.

Moreover, the axion is of interest by its own since it is a pseudo-scalar particle (a particle with no intrinsic spin and with a wave function that changes sign under parity inversion). If discovered it would be the first of its kind. It would therefore be more than appealing to find one of its formal analogues in condensed matter physics.

A number of theoretical scenarios were proposed to realize axionic-like quasiparticles in condensed matter. One of the most promising is that of a Charge Density Wave (CDW) induced by electron-electron or electron-phonon interactions in a so-called Weyl semi-metal. A Weyl semi-metal is a solid-state crystal whose valence and conduction electronic bands exhibit crossings at specific points in the reciprocal space. These Weyl nodes always appear by pairs owing to a lack of, or as an outcome of, a loss of spatial or time inversion symmetry, thus harboring quasi-particle excitations with a well-defined chirality. When the interactions correlate quasi-particles (Weyl fermions) of opposite chirality, an axionic CDW may emerge. The CDW itself can slide in the crystal giving rise to phase excitations (phasons). The dynamics of the phasons of this CDW under magnetic field is what would provide a solid-state analogue of an axion electrodynamics. Its distinctive experimental signature would be a strong variation of the conductance as a function of an applied magnetic field (magnetoconductance) through the coupling of the pseudoscalar axion field with the pseudoscalar invariant ($\vec{E}.\vec{B}$) of the electromagnetic field.

A recent publication in Nature claimed evidence for such an axion contribution to the magnetoconductance. It raised our interest since experiments were performed in the quasi-1D compound $(TaSe_4)_2I$ that we studied intensively three decades ago. The publication concerned the magnetoconductivity of the sliding CDW, a phenomenon we regularly studied in our laboratory. Therefore, in collaboration with a researcher from the Kotelnikov Institute in Moscow, we performed an experiment to measure the proposed excess of CDW velocity due to the axion field in this compound.

The CDW transition in (TaSe4)2I occurs at around 260K. Upon cooling, the resistance evolves from a semi-metal regime to a semiconducting one. The CDW sliding manifests itself by the appearance of a non-ohmic resistance beyond a threshold electric field. The experimental method to check the occurrence of a longitudinal magnetoconductance thus consists of setting the current above the threshold and measure the voltage change when a magnetic field is applied parallel to the current.

To our surprise we did not detect any additional contribution to the magnetoconductivity in the sliding state of $(TaSe_4)_2I$ that could reveal possible axion electrodynamics (see *Fig. 1*). A null result as we report is in its own an interesting result. This non-reproducibility with respect to results published in literature could be the source of controversy, but more importantly may open a scientific debate and lead to re-examine the experimental ways to investigate this new field. Anyhow, the novel mechanism for CDW formation, i.e. the breaking of a chiral symmetry with multi-bands of opposite chirality, goes beyond the traditional scenario provided by the nesting of parallel bands and remains an exciting possibility.

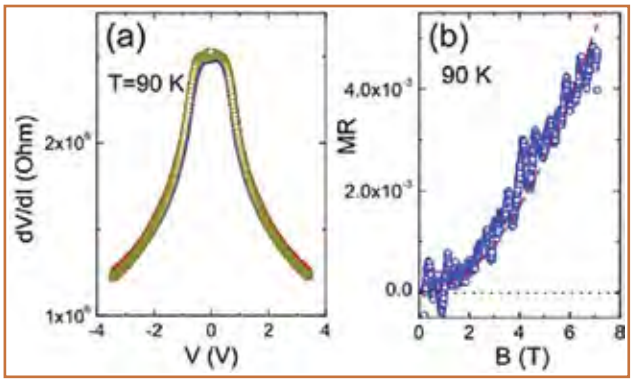

***Fig. 1: (a)** Differential current-voltage characteristics, dV/dI(V), at T= 90 K in magnetic field B = 0 T (dark yellow symbols), magnetic field B = 7 T parallel to the current (red symbols) and B = 7 T perpendicular to the current (blue symbols). **(b)** Magnetoresistance of the same $(TaSe_4)_2I$ sample at T = 90 K in a magnetic field parallel to the current I = 1.2 I_t , where It is the threshold current for the CDW sliding.*

FURTHER READING

"Does $(TaSe_4)_2I$ really harbor an axionic charge density wave?", A.A. Sinchenko, R. Ballou, J.E. Lorenzo, Th. Grenet & P. Monceau,
Applied Physics Letters **120**, 063102 (2022).

CONTACT

Pierre Monceau
pierre.monceau@neel.cnrs.fr

Bulk Twisted graphite: a room temperature superconductor

The discovery of superconductivity in twisted graphene multilayers opens the era of superconductors by design, as there is no theoretical upper limit for the value of their transition temperature. However, it is impossible to guess the optimal geometry for superconductivity, as the possibilities are innumerable. We show here a synthesis for testing all the geometries in the same bulk twisted graphite. Our samples show granular superconductivity at room temperature.

Recently, a new carbon material has been rendered superconducting: handmade stackings with several graphene layers twisted by a small angle. Such a small angle gives rise to a so-called Moiré pattern, an interference pattern produced by overlaying identical but slightly offset crystal planes. From this Moiré device emerges an arrangement of macromolecules, with a superposition of carbon atoms of different layers (*Fig. 1*). The resulting electronic structure has very flat bands that are held responsible for the superconductivity, presently with an empirical upper bound of the critical temperature of about 10K. Although there is no theoretical upper limit for the transition temperature, the number of Moiré candidates is inaccessibly large, as the stacking possibilities (number of layers, twisting angles) are practically infinite. Additionally, the manufacture of Moiré nano-devices is extremely difficult, rendering the search for higher transition temperatures an almost impossible lottery.

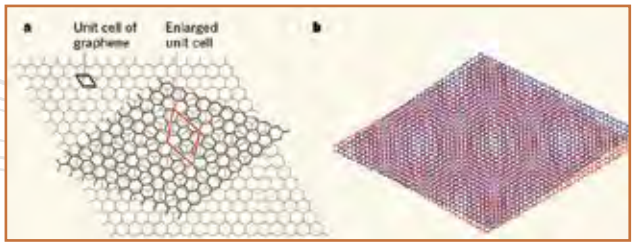

Fig. 1: *The lattice structure of graphene and a stacking of two layers twisted by an angle, leading to a Moiré pattern. The emerging macromolecules due to the regions of superposing carbon atoms and the new enlarged cell are shown on the left panel. A more pictorial image for a smaller angle is shown on the right panel.*

We have designed a method for constructing in the same bulk sample crystallites with a large number of possible twisting configurations. We first synthetize at 250°C KC_8, a potassium intercalated graphite compound where all the graphene planes are exactly one on top of the other. We then extract at room temperature the potassium by soft chemistry. The slow chemical kinetics of the reaction causes a disordered collapse of the KC_8 structure and a residual potassium content. X ray measurements performed at the European Synchrotron Radiation Facility show mainly six-fold stackings with arbitrary twisting angles, which depend on the local residual potassium concentration. The obtained bulk twisted graphite statistically contains a large number of different six-fold configurations in the same sample.

Magnetization measurements performed at Institut Néel and at the Laboratoire de Physique des Solides, show that at low temperature graphite's important diamagnetism is replaced by a ferromagnetic behavior. Furthermore, transitions to diamagnetic states are observed at 110K, 245K and 320K. The appearance of a given transition temperature depends on the synthesis protocol of the samples, i.e. starting material, synthesis time, etc. The magnetic field cycles show hysteresis, compatible with the superconducting character of the diamagnetic transitions. The cycles shrink with increasing temperature and disappear at the transition, as expected for a superconducting state.
For the 245K phase, we obtained almost zero resistance in electrical resistance measurements, which allowed the study of the effect of magnetic field on the transition temperature. It is extremely small, yielding a gigantic zero temperature critical magnetic field of 1200±200 Tesla, more than ten times larger than the largest presently known critical field. In fact, it is the value expected for a Moiré system with such a transition temperature.

Our measurements show the maximum possible transition temperatures, but do not tell us which is the exact Moiré configuration for these superconducting states. By construction, the number of crystals with the structure and doping necessary for superconductivity is tiny and hence, of granular character. More work is needed in order to increase their number and the superconducting volume, and to isolate the configuration of each superconducting phase.

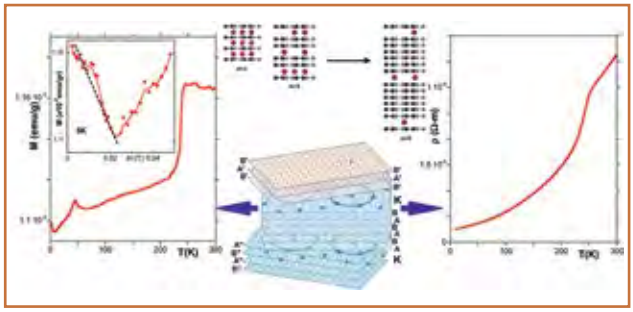

Fig. 2: *Upper middle panel: sketch of the de-intercalation reaction finishing in the six-fold intercalate. Lower middle panel: pictorial representation of an individual twisted crystal. Right panel: electrical resistance of a sample showing the superconducting transition at 245K. Left panel: magnetization measurement showing the superconducting transition at 245K. Inset: magnetization as a function of field at 5K showing the Meissner effect.*

CONTACTS

Manuel Núñez-Regueiro
manolo.nunez-regueiro@neel.cnrs.fr
Jean-Louis Hodeau
jean-louis.hodeau@neel.cnrs.fr
Marie-Aude Méasson
marie-aude.measson@neel.cnrs.fr

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"Possible high temperature superconducting transitions in disordered graphite obtained from room temperature deintercalated KC_8",
S. Layek, M. Monteverde, G. Garbarino, M-A. Measson, A. Sulpice, N. Bendiab, P. Rodiere, R. Cazali, A. Hadj-Azzem, V. Nassif, D. Bourgault, F. Gay, D. Dufeu, S. Pairis, J-L. Hodeau & M. Nunez-Regueiro, Carbon 201 (**2023**) 667–678.

First insight into the charge distribution at electrochemical interfaces

In electrocatalysis, reactivities and performances are crucially affected both by the atomic and electronic structure of the electrode surface and by the electrolyte, interacting at the electrochemical interface. While several techniques allow characterizing the atomic structure of the electrode surface, up to now an experimental technique able to give the electronic description was lacking.

Yet, the knowledge of the spatial charge distribution at the interface as a function of the applied potential is mandatory to fully describe the parameters influencing the electrochemical reactivity. Indeed, the sign and the extent of the interfacial charge largely impact the interactions of the electrolyte molecules/ions with the electrode surface.
We show here that by combining in situ Surface Resonant X-Ray Diffraction (SRXRD) experiments and ab initio calculations, we could obtain a quantitative description of the charge distribution at the electrochemical interface of the archetypal system Pt(111) in an acidic medium.

SRXRD is a site sensitive spectroscopic technique probing both atomic and electronic surface structures. It combines the sensitivity of Surface X-Ray diffraction to the atomic structure of the surface layers and the sensitivity of X-Ray Absorption Near Edge Spectroscopy to the atomic oxidation states. A synchrotron x-ray source is needed to perform such experiments: a high brightness of the X-ray beam is mandatory in order to measure the tiny surface signal. We performed in situ SRXRD experiments on Pt(111) in an acidic sulfate medium, using a dedicated electrochemical cell, at the French CRG D2AM beamline at the ESRF. The recorded spectra show a strong dependence on the applied potential value. In order to relate these experimental data to the surface properties we used ab initio calculations.

Calculations were performed using the home-made FDMNES software, which relies on the Density Functional Theory. Recently extended to the simulation of surface resonant diffraction experiments, it has now been developed to describe the electrochemical interface, taking into account the presence of the electrolyte facing the crystal and the effect of the applied external electric field.

When a (charged) metal is in contact with a liquid, a charge exchange occurs between the two phases, due to the initial gradient of the electrochemical potential. The simplest model to describe this mechanism was proposed by Hermann von Helmholtz. It states that a double layer of opposite polarities develops at the electrochemical interface. In the absence of specific ion adsorption on the electrode surface, it is described by a charged plane at the metal surface and a second plane in the solution containing the counter ions.

In this context, FDMNES models the electrochemical interface by adding a potential V(z) (where the z coordinate is perpendicular to the crystal surface) to the surface atomic structure potential. This additional potential simulates the presence of a non-ordered ionic layer at a distance z0 from the outermost surface atomic plane (*Fig. 1*).

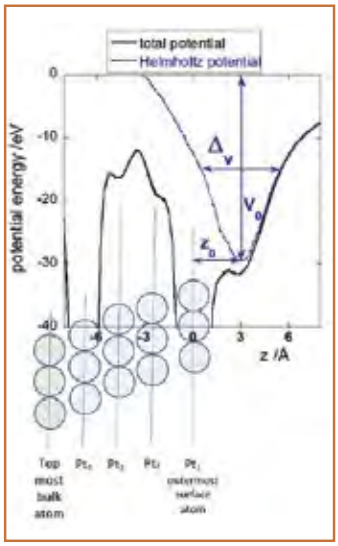

***Fig. 1:** Total potential (continuous black line) as a function of the z coordinate perpendicular to the crystal surface. The Helmholtz contribution (V_0=-30 eV, z_0=3 Å, Δ_V=5 Å) due to the ionic plane at z0 from the crystal surface is represented by the blue dotted line. The mathematical expression for V(z) in shown, where α=0.2859. A schematic representation of the Pt surface structure (four surface layers on the Pt bulk) is also given.*

In the potential region where no adsorption is present, we could determine the charge distribution on each of the metal surface layers and the distance to the oppositely charged ionic plane in the electrolyte (*Fig. 2*). We thus revealed the presence of an electric dipole over the two outermost platinum layers. Our results demonstrate the capacity of this original approach to unveil the electronic densities at the electrochemical interfaces, a challenging topic for the understanding of the electrochemical reactivity.

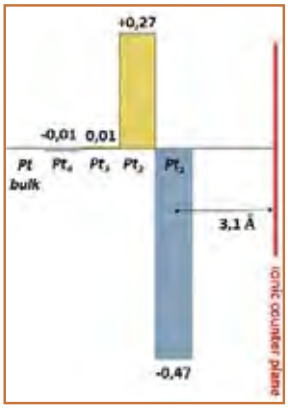

***Fig. 2:** Schematic representation of the charge distribution at the electrochemical interface: charge per platinum atom on each of the four atomic planes constituting the electrode surface (Pt_1, Pt_2, Pt_3, Pt_4). The oppositely charged ionic counter plane is placed at 3.1 Å from the electrode surface.*

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CONTACTS

Yvonne Soldo-Olivier
yvonne.soldo@neel.cnrs.fr
Yves Joly
yves.joly@neel.cnrs.fr

Chilling with light for a new generation of space cryocoolers

Optical cooling is a phenomenon where a crystal emits more optical power than it absorbs, leading to its cooling. In collaboration with Air Liquide Advanced Technologies (ALAT, Sassenage), we are studying its potential for a new kind of space cryocooler embedded in a satellite. It will pave the way to contact-less, vibration-free cooling of sensitive equipment down to about 100 K. To test this possibility researchers and technical staff of Institut Néel have built and successfully operated a first prototype of a fiber-coupled lasercooler.

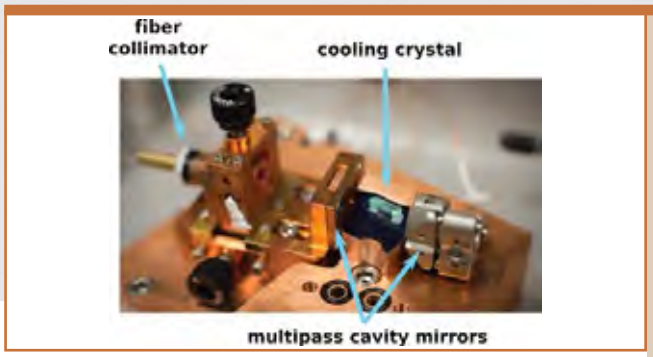

Fig. 1: *Photograph of the laser cooling experimental bench. The Yb:YLF crystal sits on 2 silica aerogel pads. The laser power is coupled through a fiber collimator inside a multipass cavity to be fully absorbed in the crystal. The green light in the crystal comes from the fluorescence of Er impurities.*

The principles of optical cooling derive from the very first studies on the interaction of light with matter. In 1852 Stokes explains the phenomenon of fluorescence and observes that in some materials the wavelength of the emitted light is different from the one of the absorbed light. Most interestingly, this 'Stokes shift' can be negative, re-emitting light with a shorter wavelength. This observation leadw Pringsheim to assert in 1929 that repeated cycles of absorption and fluorescence (with a higher-energy photon) extract energy from the material, hence leading to its cooling. The first clear experimental demonstration of cooling took place in 1995 after progress in lasers and in growth science that allowed producing good enough materials. Indeed, cooling is possible if and only if each absorption event is followed by a fluorescent photon escaping the crystal. Any other phenomenon leads to a net heating two orders of magnitude larger than the cooling process!

Laser cooling is considered by ALAT as a potential breakthrough technology in the market of space cryocoolers which are required for infra-red detectors in Earth-observation missions. It offers many advantages over existing solutions based on pulsed-tube coolers: no vibrations, solid-state cooling with no moving parts, no electromagnetic perturbation and a dramatic reduction of volume. Preliminary studies in a scientific collaboration with Institut Néel in the frame of a CIFRE thesis (Rémi Vicente) compared the requirements of a laser cooler and a pulsed-tube cooler in terms of size, weight and power. In spite of a significantly higher use of power, a laser cooler requires less volume and up to 20% less weight.

A practicable implementation of a laser cooler requires separating the cooling head from the laser source. We designed an original setup where a high-power single mode polarization maintaining fiber connects the two systems. Colleagues from the University of Pisa provided a Yb^{3+} doped $YLiF_4$ crystal (Yb:YLF), which is the best material for laser cooling so far. In order to minimize the thermal load on the crystal, the setup is pumped to reduce convection, the crystal stands on low-conduction silica aerogel pads and is mounted inside a 'clam-shell' (top cover not shown in *Fig. 1*) whose surfaces are coated by a low-emissivity Tinox layer in order to reduce radiative transfers and efficiently absorb the crystal fluorescence. The experimental setup was designed with the support of the Experimental Engineering technical group, which also machined and assembled its inner core.

Contact-free thermometers are used to monitor the crystal temperature. First a carefully calibrated thermal camera (*Fig. 2*) is used, down to a temperature of 160 K. Below this limit, a fluorescence spectroscopy method is used, which allowed us to record a minimum temperature of 125K for 11 W of absorbed power. After the laser is switched off, the return to equilibrium at room temperature allows to evaluate the conductive and radiative thermal loads on the crystal and hence the cooling power, which is in good agreement with a simple model. Our collaboration with ALAT continues. It now concerns the thermal link between the cooling crystal and the object that is to be cooled. Another objective is to design a compact cooling experiment.

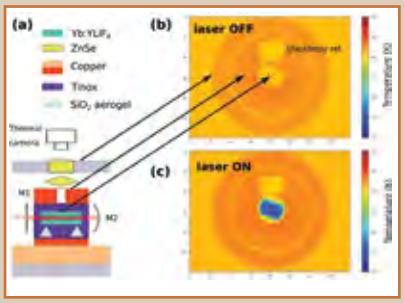

Fig. 2: (a) *Cut of the experiment. A set of ZnSe optics allows to record its image on a thermal camera.* ***(b)*** *Thermal image with no cooling laser. The different temperature readings are due to the different emissivities of the materials and finite transmission of the lenses.* ***(c)*** *Same image when the cooling system is on.*

CONTACTS
Gilles NOGUES
gilles.nogues@neel.cnrs.fr
Laurent Del-Rey
laurent.del-rey@neel.cnrs.fr
Julien Jarreau
julien.jarreau@neel.cnrs.fr

PhD student:
Rémi Vicente (CIFRE contract 2018-0065)

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The enigmatic pseudogap phase of high temperature superconductors

Superconductivity is a macroscopic quantum state characterized by the absence of electrical resistance below the critical temperature T_c, and the ability to fully expel an applied magnetic field.

In 1986, the discovery of superconductivity in "cuprates" (complex copper oxides, most famously yttrium barium copper oxide "YBCO"), with a critical temperature exceeding 100K, triggered huge interest. However, despite three decades of intensive studies, unraveling the mystery of high-temperature superconductivity remains a fundamental issue in modern solid-state physics. One of the most intriguing features is that superconductivity emerges in the vicinity of an enigmatic, so-called "pseudogap" phase which shows up below a critical chemical doping p^*. This phase exhibits several unusual behaviors that go against the laws generally observed in classical metals. Unveilling the nature of this pseudogap phase is hence a fundamental issue in order to understand the physical mechanism leading to this unconventional superconductivity.

The emergence of high T_c superconductivity around p^* however hinders any direct measurements of the ground state of the pseudogap state for T approaching zero. To get access to this phase it is hence necessary to "destroy" the superconducting state, which is in principe feasible by applying a magnetic field. However, superconductivity is extremely robust in cuprates and the field required to do so exceeds hundreds of teslas in the case of YBCO, well beyond the values accessible in laboratories. Still, the normal state is reachable by applying "only" ~ 30 teslas in some compounds such as $Bi_{2+y}Sr_{2-x-y}La_xCuO_{6+\delta}$ (Bi2201), $(Nd/Eu)_{0.4}La_{1.6-x}Sr_xCuO_4$ (Nd/Eu-LSCO) or pristine LSCO. Although very large, this field value is accessible in high magnetic fields facilities, such as the Laboratoire National des Champs Magnétiques Intenses (LNCMI) in Grenoble.

The determination of the specific heat C is very challenging in those extreme temperature and field conditions but we have developed a high sensitivity microcalorimeter which enabled us to measure C/T down to 0.3K and up to 36T in all of the above-mentioned systems, in collaboration with Louis Taillefer (University of Sherbrooke, Canada). As expected in metals, the electronic contribution to the specific heat (the so-called Sommerfeld coefficient) C_e/T was of the order of 5-6 mJ/molK² and temperature-independent far from p^*. But our measurements show that C_e/T displays a strong peak for $p \sim p^*$ and T approaching zero in all of the measured systems, reaching ~ 22 mJ/molK² at 0.5K in Nd/Eu-LSCO. Moreover, those large values are associated with logT temperature dependence at odds with the classical behaviour of metals, suggesting that the particles that get coupled into pairs in the superconducting phase are some kind of "highly entangled mixture" of wave functions forming a so-called quantum crital state. Recent calculations showed that no symmetry is broken when the system enters the pseudogap phase and the onset of the pseudogap phase occurs at a Widom line emanating from a (finite-temperature) critical endpoint of a first-order transition between this strongly correlated phase and a standard metal. Those calculations showed that a temperature dependent specific heat maximum is expected to appear at p^*, in good agreement with our experimental observations (see *Fig. 1*). Hopping of quasiparticles between adjacent Cu and O atoms (hindered by on-site repulsion on the Cu atoms) is also expected to generate so-called "superexchange" spin–spin correlations leading to intense electron pairing, hence being a very promising candidate for the unconventional mechanism leading to high T_c superconductivity: solving this longstanding riddle may finally be within reach.

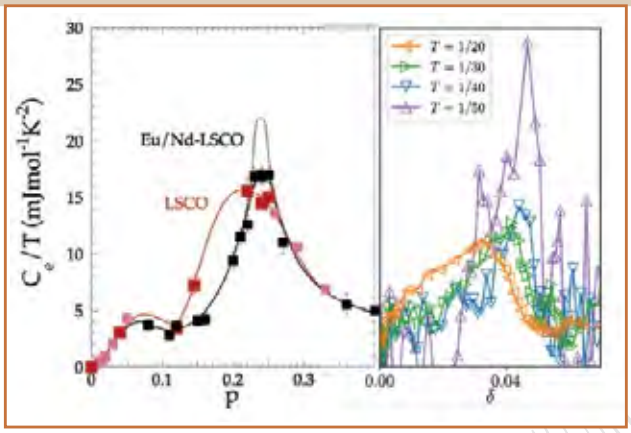

Fig. 1: *Left panel: Electronic specific heat of two cuprate materials "Eu/Nd-LSCO" (black squares) and pristine-LSCO (red squares), as a function of hole-doping p (holes per crystal lattice unit) at 2K. Very large magnetic fields (up to 35T) had to be applied in order to destroy the superconducting state to get access to the normal state properties. As shown, C_e/T displays a pronounced peak at a doping content corresponding to the onset of the pseudogap phase p* ~ 0.23 and ~ 0.21 in Eu/Nd-LSCO and LSCO, respectively. The peak is stronger at 0.5K (dotted black line in Eu/Nd-LSCO) due to a logT divergence of C_e/T for p ~ p* (not shown here). Right panel: cluster-DMFT calculations of C_e/T as a function of hole-doping (here noted δ) for various temperatures (from G. Sordi, C. Walsh, P. Sémon, and A.-M. S. Tremblay, Phys. Rev. B **100**, 121105(R) (2019)) showing that a temperature dependent specific heat maximum is expected to be observed at the onset of the pseudogap in the two-dimensional Hubbard model.*

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CONTACT
Thierry Klein
thierry.klein@neel.cnrs.fr

PhD student:
Clément Girod

Amplifying signals at the quantum limit with graphene

The precise measurement of weak signals is of extreme importance for various applications in quantum engineering, such as quantum computing and quantum sensors. Standard solutions based on circuits built with superconductors, that can carry a charge current without resistance below a critical temperature, lack the possibility to be controlled by an electric field. We have demonstrated that by inserting graphene, a single-layer of carbon atoms, inside a superconducting circuit we get the best of both worlds: absence of dissipation, one key property of superconductors, and electrical tunability, that is the hallmark of semiconductors.

Realizing hybrid electrical circuits that combine different materials is often a real challenge as individual properties can be degraded in the integration processes. In this work, an international team led by researchers at Institut Néel, has succeeded to integrate graphene into a special kind of amplifier, a Josephson parametric amplifier.

For that purpose, we have fabricated a Josephson junction, i. e. two superconductors, here the aluminum contacts, weakly connected by a non-superconducting material, here the graphene, which is itself encapsulated in hexagonal boron nitride for protection. In such junctions, the graphene layer inherits the properties of the superconductor. For instance, an electrical current can flow in graphene without dissipation, which is mandatory for a low noise amplification. In addition, graphene keeps some of its own key properties, in particular its gate-tunability (capability to tune the electrical conductivity of the material by applying a voltage).

The interest of Josephson junctions is that their behavior depends on the signal power, a phenomenon known as nonlinearity (i.e. that the output signal is not proportional to the input signal). That nonlinearity is the key property that allows signal amplification.

Our graphene-based amplifier has allowed us to observe a large amplification, of the order of 100. In addition, the frequency of the amplified microwave signal could be tuned, thanks to the properties of graphene, by as much as 1 Gigahertz with a simple electric field (see *Fig.1*). This allows to envision a large number of applications. Finally, the noise added during the amplification process was limited to half a photon, that is the minimal amount set by the laws of quantum mechanics.

Such performances pave the way to the use of such devices in future quantum computers, as well as the exploration of new amplification mechanisms, unique to these kind of hybrid structures.

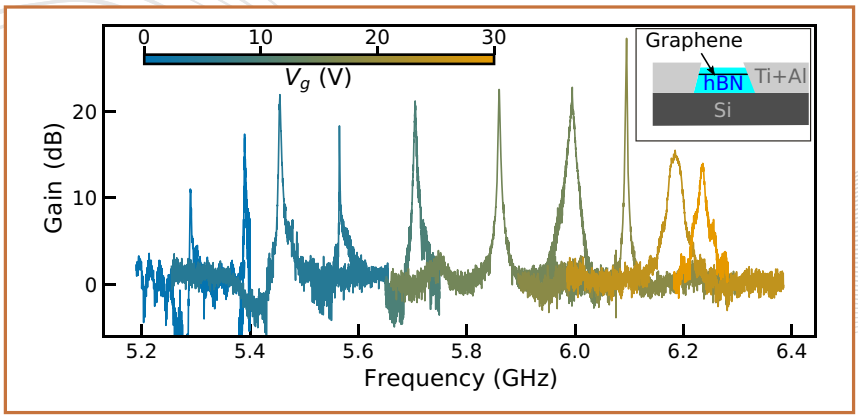

Fig. 1: *Gain of the device (in dB) measured for different gate voltages (V_g). We see that changing the gate voltage allows to amplify a signal at another frequency. The full tunability of the device goes from about 5.2 to 6.2 GHz. The inset illustrates the concept of a graphene Josephson junction: a layer of graphene (encapsulated in hexagonal boron nitride, h-BN, here) is connected by titanium (Ti) and aluminum (Al). The device lies on a silicon substrate (Si).*

CONTACT
Julien Renard
julien.renard@neel.cnrs.fr

PhD student:
Guilliam Butseraen

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Discovery of a novel superconducting iron-based silicide: $LaFeSiO_{1-\delta}$

Below a critical temperature called T_c, some materials become superconducting, transporting charge carriers without dissipation and expelling external magnetic fields. A lot of different superconductor families exist and among them the iron-based superconductors (IBS), discovered in 2008, constitute the family with the second highest T_c at normal pressure, next to copper oxides. But, such Fe-based compounds contain quite toxic elements like arsenic or selenium. In the last years new IBS were discovered with silicon or germanium in substitution of As/Se. Here we present a new member of the Fe-based silicide family, $LaFeSiO_{1-\delta}$, which is also found to be superconducting.

In the last years new IBS were discovered with silicon or germanium in substitution of As/Se. Here we present a new member of the Fe-based silicide family, $LaFeSiO_{1-\delta}$, which is also found superconducting.

Since their discovery in 2008, iron-based superconducting pnictides (As, P...) and chalocgenides (Te, Se...) are now a well-established class of unconventional superconductors, spanning multiple structural families, with Tc up to 55 K in bulk materials. This category of superconductors has recently been extended to other layered materials where pnictogen/chalcogen atoms are replaced by crystallogen elements (i.e. belonging to the carbon group : C, Si, Ge...): either with Ge in YFe_2Ge_2 (T_c ~ 2 K) or Si in LaFeSiH (T_c ~ 10 K).

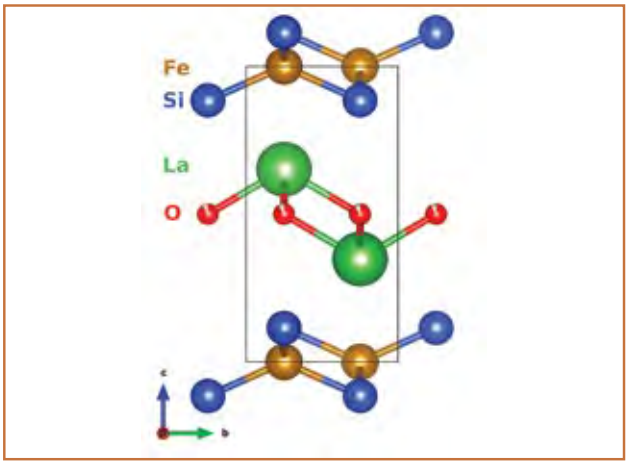

***Fig. 1:** Crystal structure of the novel layered iron oxy-silicide $LaFeSiO_{1-\delta}$.*

In this work, we present the discovery of a new compound obtained by topochemical oxygenation of LaFeSi. Such topotactic reaction allows the insertion of oxygen atoms between the double layer of La atoms, at the empty tetrahedral La_4 sites, preserving the remaining crystal structure. The properties of the resulting and unexpected crystallogenide, $LaFeSiO_{1-\delta}$ were probed by combining complementary experimental techniques: X-ray-, neutron- and electron diffraction, energy dispersive X-ray and nuclear magnetic resonance (NMR) spectroscopy, thermogravimetric analysis, resistivity and magnetization measurements. Our detailed study reveals that this crystallogenide is superconducting with a relatively high T_c ~ 10 K, despite its strongly squeezed Fe Si anion height, below 1 Å, challenging the usual relationship between crystallographic features and superconductivity in Fe-based compounds.

This unique crystal structure has a strong impact on the electronic properties of $LaFeSiO_{1-\delta}$. Just above T_c, resistivity shows a non Fermi liquid temperature scaling, i.e. a deviation from the T^2 temperature dependence, a signature of enhanced electronic correlations. This correlated behavior is also visible in the NMR data which evidences weak antiferromagnetic fluctuations. The calculated electronic structure of LaFeSiO is significantly changed compared to the one of the canonical LaFeAsO oxy-arsenide. In the oxy-silicide, the Fe related Fermi surface, consisting almost uniquely of hole pockets, suggests another kind of electronic correlations and then a different related superconducting mechanism than the usual s±-mechanism. Finally, the location of $LaFeSiO_{1-\delta}$, LaFeSiH and partially fluorinated $LaFeSiF_x$ superconductors in the phase diagram points towards the existence of a new emerging superconducting dome related to Fe silicides.

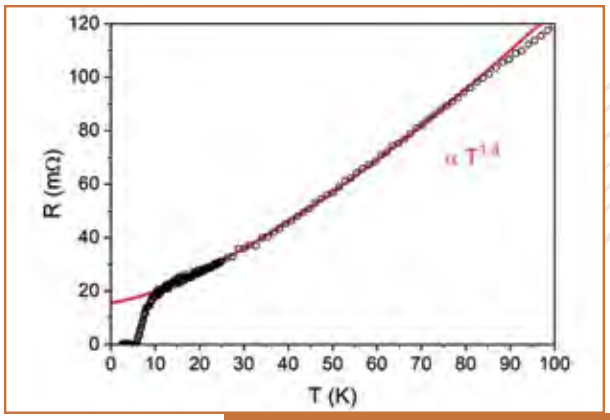

***Fig. 2:** Resistivity measurement of $LaFeSiO_{1-\delta}$ evidencing the superconducting transition at T_c ~ 10 K.*

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CONTACTS

Pierre Toulemonde
pierre.toulemonde@neel.cnrs.fr
Andrés Cano
andres.cano@neel.cnrs.fr

PhD student:
Mads Fonager Hansen

A new probe to measure temperature at the nanoscale

Determining the local temperature of micro- or nanostructures is of increasing importance in nanoscience, since the dimensions of objects in for instance microelectronics continue to be reduced. The notion of temperature on a very small scale of space has intrigued the scientific community for many years. Indeed, on which length scale does thermalization take place? How to measure a temperature at the atomic scale, using which probe? These are all questions we can address by the development of our state-of-the-art nanoscale temperature AFM probe.

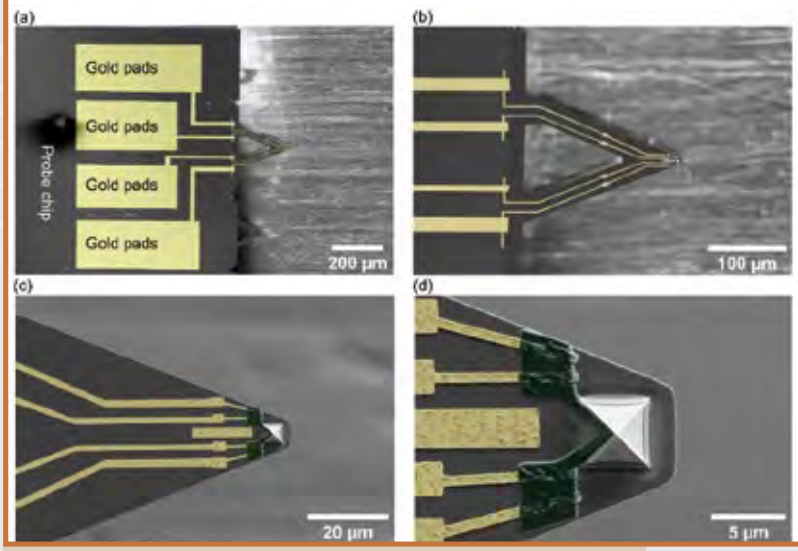

Fig. 1: *AFM cantilever equipped with a NbN thermometer. The SEM images illustrate the integration of the thermal sensitive niobium nitride thin film on an AFM cantilever. (a) and (b) show the top view of the four-terminal electrical leads of Ti/Au in yellow on the AFM chip. (c) and (d) show the integration of the NbN resistive thermometer in green at the apex of the non-planar 3D pyramidal shaped AFM tip.*

Reducing the size of components in microelectronics involves that heating by the Joule effect can become preponderant when current flows in these components. The rise of temperature on a very small scale can cause premature aging of the components or eventually their destruction, generating significant additional costs for the manufacturers. Thus, being able to probe such hot spots is becoming essential for the detection of physical failure in all kind of microchips. Unfortunately, a precise measurement of the temperature at this close-to-nanometer scale is not trivial, especially when one wants to determine this temperature on a very precise position on an electronic integrated circuit.

In the research work presented here, we have developed a sub-micron sized temperature probe by affixing a thermometric thin film to the apex of the tip of an atomic force microscopy (AFM) lever. Cantilevers for AFM are devices that are able to probe the surface of matter with a spatial resolution of a few nanometers. Our work consisted in adapting these levers by adding an ultra-sensitive thermometer close to the usual zone of physical contact between the tip and the material to be probed, namely directly on the measurement pyramid *(see Fig. 1(d))*. This was made possible by the original micro and nano-lithography techniques we developed with the technical group Nanofab at Institut Néel. These unique techniques, which are not achievable through conventional clean room methods, consist in our ability to perform lithography on non-planar 3D objects, such as a pyramid.

With these methods, we have succeeded in fully integrating a temperature probe on the pyramid of an AFM tip *(see Fig. 1)*. The thermometer is made of a thin film of niobium nitride, which can be ten times more sensitive than its metal competitors. The performance of our probes has been demonstrated through calibration on reference systems (Si, sapphire, glass), allowing temperature measurements at the scale of a few tens of nanometers. A substantial improvement in performance has been demonstrated with respect to existing probes, making our device much better than the state of the art.

The development of this new tool, which is both ultra-sensitive in temperature and shows a high spatial resolution, opens up tremendous prospects for both fundamental science and industrial applications. Valorization of this unique technological development is underway with the SATT of Grenoble Linksium through the NANOTIP maturation project. In addition, research on heat exchange at the nanometer scale will greatly benefit from this new tool, which is in strong demand from the micro and nanothermal scientific community, especially for thermal scanning microscopy.

CONTACTS

Olivier Bourgeois
olivier.bourgeois@neel.cnrs.fr
Gwénaëlle Julié
gwenaelle.julie@neel.cnrs.fr
Simon Le-Denmat
simon.le-denmat@neel.cnrs.fr

PhD student:
Rahul Swami

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"Point de Bascule" (Tipping Point): a play about climate crisis

In the last years, many efforts have been made in order to inform the general public about the climate crisis of our planet. Front-page news, radio and television programs regularly alert about the situation, report GIEC publications and provide explanations about the involved physical processes. Despite this, the knowledge and awareness of the population on these questions remains largely inadequate in order to curb significantly the current trends. Scientific terms regularly employed such as "greenhouse effect", "infrared radiation" or "greenhouse gases" refer to complex phenomena which effective meaning is not always clear in the people's mind or, even worse, is sometimes misunderstood. In this context, a group of scientists from Institut Néel, IGE and UGA, has decided to collaborate with the theatre company "La Compagnie du Gravillon".

The goal of this collaboration was to create a play which deals with the scientific issues of climate crisis in a humoristic and poetic way. In the "Point de bascule" show, the meta-researcher Barthélémy Champenois, who lies somewhere "beyond the CNRS", decides to plant the trees necessary to compensate the CO_2 emissions generated by his flight to New York. He rapidly realises that this is not a realistic way to achieve the goal. While he faces full of doubts and incoherence in his every-day life, the audience lives with him all the situations he goes through and is led to think about all of this. Scientific information dispatched throughout the show are made accessible to all with the help of body live performances and with the tale of Barthélémy's personal adventures. The audience laughs, sometimes even to tears, but the seriousness of the situation is never far off, which leads to an intense emotional experience.

The success of the collaboration between Barthélémy and a group of physicists from Institut Néel and UGA was already proofed with the previous spectacle "Lumière, histoire d'une hors la loi" (Light, history of an outlaw). This play tells the discovery of the nature of light and its laws from the ancient Greeks to Einstein, always mixing scientific truth with fun. The group of scientists who worked on the light history enlarged for the project on climate, to cover other scientific domains, such as climatology, hydrology and oceanography. Scientists and artists regularly met during two years. We defined together the content of the future play by sharing and broadening our knowledge about the physical or chemical principles underlying the climate processes. Then, the actor Nicolas Prugniel transcribed the various concepts in his own way with the help of the artistic team. These sequences were finally presented to the scientific team and open to public scrutiny during artistic residencies. The final play is the result of these rich ongoing scientific and artistic exchanges.

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The first official public performance of "Point de bascule" (Tipping point) took place in September 2022, during "Merci, Bonsoir !" festival in Grenoble. Since that date, the show meets with strong success, with many performances in high schools and junior high schools, festivals, theatres, municipal halls. Barthélémy was also invited to Institut Néel day in Saint-Jean de Chepy! The feedbacks received from the public underline the strong emotional impact of the chosen art form.

In some occasions, the public is welcome to exchange with the artistic and/or scientific team involved in the project. These discussions give the opportunity to broaden up the debate and to address other environmental issues such as the collapse of biodiversity or the exhaustion of natural resources. The play will now live his life, and you can follow the upcoming show dates on the Compagnie du Gravillon web site: *https://compagniedugravillon.fr/*

ARTISTIC TEAM

"La Compagnie du Gravillon"
Nicolas Prugniel, actor /// Jérémy Brunet, director
Florian Lyonne, light and audio designer /// Myriam Dardard, creation assistant

SCIENTIFIC TEAM

Emmanuel Cosme (IGE), Julien Delahaye (Institut Néel), Thierry Grenet (Institut Néel), Jean-Louis Hodeau (Institut Néel), Didier Mayou (Institut Néel), Gérémy Panthou (IGE), Yvonne Soldo (Institut Néel), Théo Vischel (IGE), Sylvie Zanier (UGA).

CONTACTS
Julien Delahaye
julien.delahaye@neel.cnrs.fr
Yvonne Soldo
yvonne.soldo@neel.cnrs.fr

HIGHLIGHTS N°16

Institut Néel - CNRS
25 rue des Martyrs - BP166
38042 Grenoble cedex 9 - France

neel.cnrs.fr