Materials Growth and Measurement Laboratory

# ANNUAL REPORT 2019

# Publishing information

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# CONTENTS

# FOREWORD

It has been an honor for me to chair the Scientific Advisory Committee (SAC) of the Materials Growth and Characterization Laboratory (MGML) research Infrastructure since 2017. And it is my real pleasure to write the foreword of the first Annual report 2019, as I experienced first-hand the creation and development of this excellent facility many years ago. An impressive history of the evolution of MGML to its successful international status since the 90's is given by Pavel Javorský, the head of MGML, in his foreword.

The first meeting of the SAC with the scientific staff members of MGML took place in Prague in 2017 at the Department of Condensed Matter Physics, Charles University, where we intensively discussed suggestions to maintain and enhance MGML research infrastructure on high international level. At the following meeting in 2019, the SAC was extremely satisfied with the major scientific progress and performance of MGML. These include excellent purposefully upgrade of most of the instruments, improving the external parameters (lower temperatures, higher magnetic fields, and better stability), from which all users benefit. In particular, the extension of MGML by the laboratory of the Institute of Physics in Cukrovarnicka. The combination of two institutions is very important to sustain the excellence of the research infrastructure on international level. On the other hand, it is a pity that MGML did not reach the funding for the planned equipment that the SAC strongly supported in 2019. I, thus, support further projects or steps to get the planned equipment.

MGML in-house scientists and its international user community produced an impressive high quality publications covering a wide spectrum of research from fundamental science in modern condensed matter physics to technological applications. This is demonstrated in the following annual report which shows both the new technical developments for synthesizing new materials as well as for performing unique experimental measurements and some selected examples from the publications as scientific highlights.

Finally, I would like to emphasize that the scientific staff members of MGML have organized the international conference European High Pressure Research Group Meeting (EHPRG 19) in Prague, thereby promoting the scientific visibility of MGML. Such a success would not have been possible without the dedicated and hard work of all scientific and technical staff as well as students and users despite the limited financial situation.

I hope that the Annual Report 2019 inspires your interest in MGML and I take this opportunity to thank all scientific and technical staff members, students, users, and cooperating partners, who contributed to the successful research activities during recent years.

Mohsen Abd-Elmeguid

Chair of MGML Scientific Advisory Committee

It is my great pleasure to write foreword to the first Annual Report of our MGML Research Infrastructure. At this special occasion, I would like first to thank all who contributed to the success of our project and its ongoing realization. I should thank not only collaborators of the last year, but all those who contributed to the development of our laboratories in the last three decades without their effort MGML probably would not exist in the present form.

As this is the first Annual Report, I take the opportunity to summarize very briefly the history of our laboratories which resulted in the present (successful) state. Since nineties, the collaboration between the group of magnetic properties at the Faculty of Mathematics and Physics and the high pressure group at the Institute of Physics of Czech Academy of Sciences was naturally evolving. This collaboration finally led to an idea to establish internationally competitive joined laboratory providing possibilities of reliable measurements of physical properties of materials at low temperatures, high magnetic fields and high pressures dedicated not only to own needs but also to the needs of colleagues from less equipped Czech and foreign institutions. This was formally realized in 1998 by signing the agreement on the Join Laboratory for Magnetic Studies (JLMS). The first joint investment was realized – the PPMS14 became at disposal to the members of JLMS. In that time, it represented a huge step forward in experimental studies of condensed matter, namely magnetic, transport and thermodynamic properties of materials at low temperatures and high magnetic fields. It boosted the research in our laboratories including collaborations with other Czech and foreign institutions. The operation of JLMS was seriously affected by the disastrous flooding in 2002. The generous support by Charles University helped not only to restore the operation of JLMS within only six months after flooding but also to build a new specialized Cryogenics Laboratory in Troja (open in 2005) where you can find the main part of MPML now. Thanks to support of the Faculty of Mathematics and Physics and various funding opportunities, the laboratory was steadily upgraded by new instrumentation. Simultaneously, the technology of crystal growth and sample characterization has been progressively developed in the faculty laboratories reflecting the needs of users of high-quality and well characterized samples for the measurements. The unique complex of instrumentation and human resources enabled to establish the Research Infrastructure “Magnetism and Low Temperatures Laboratories” (MLTL) in 2010. MLTL was included in the Roadmap of Czech Research Infrastructures and financially supported by the Ministry of Education, Youth and Sports in the 2012 - 2016 period. The RI received more than 100 domestic and foreign users in this period. The experimental equipment was further intensively developed to satisfy the user needs. Although the continuation of MLTL as a funded RI was not approved in 2015, the user support and limited open access was maintained even in that period thanks to the faculty support.

In 2017, the new RI project was successfully introduced. Reflecting the significant development of the technology laboratories, with a new name - Materials Growth and Measurement Laboratory, consisting of two closely collaborating units: Material Growth and Characterization Laboratory (MGCL) and Material Properties Measurement Laboratory (MPML). The MGML laboratories are financed by Ministry of Education, Youth and Sports within the programme of Large Research Infrastructures since January 2019.

Although the financing had a one year delay, this was further boost in the development of our laboratories. A lot was changed – we run the full user programme, new instruments were installed, new postdocs arrived and I have a pleasant opportunity to write this Foreword to our first Annual Report. I am also very happy with the development of a collaboration between the two institutions participating in MGML: Faculty of Mathematics and Physics, Charles University as the host institution and the Institute of Physics of the Czech Academy of Sciences which became now a real partner institution. In this connection, one should mention mainly the integration of Cukrovarnická laboratory in JLMS and MGML.

Beside all the technical and scientific progress that is described further, we have also applied for new large investments in the OP VVV call. Although the project was considered for financing, the number of points received was just not sufficient to reach financing with the limited financial resources. We should try all possibilities to finance the instrumental development in future.

I would also like to thank the members of our Scientific Advisory Committee who did a great job by advising and consulting further steps. I also remember their suggestion to have such an Annual Report. So here is the first year!

Finally, I wish success to all our users because the service to the user community is our main mission. The users and their research make the laboratories alive and motivate further growth.

Pavel Javorský
Head of MGML

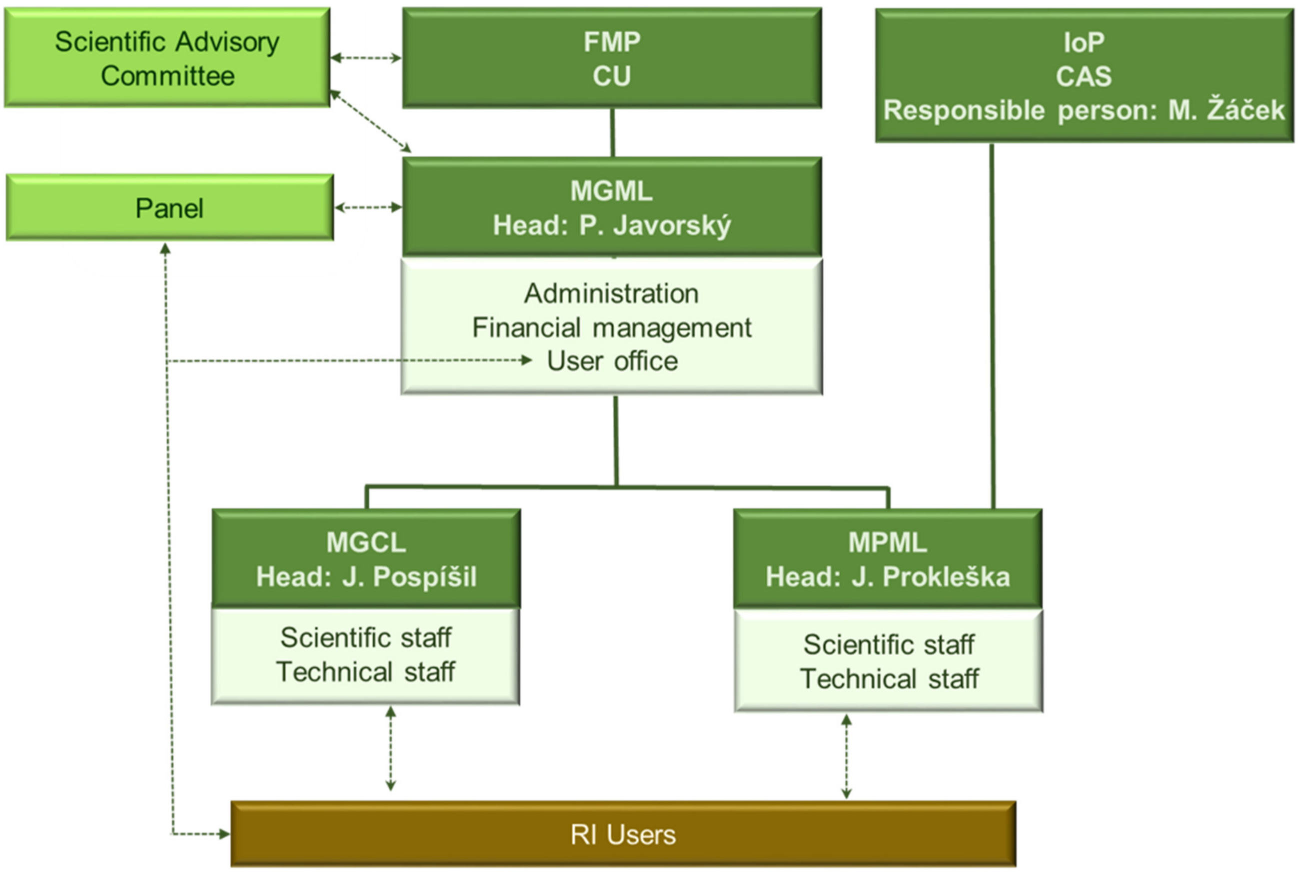
# What is MGML

MGML is a research infrastructure financed by the Ministry of Education, Youth and Sports within the program of Large Infrastructures for Research, Experimental Development and Innovation of CR (project No. LM2018096). It is an open access research infrastructure available to all users from CR and abroad.

MGML is hosted by the Faculty of Mathematics and Physics of Charles University with the Institute of Physics of the Czech Academy of Sciences as the partner institution.

MGML provides a laboratory base for advanced material research. Within its two closely co-operating units, Material Growth and Characterization Laboratory (MGCL) and Material Properties Measurement Laboratory (MPML), MGML offers open access for external users to a vast experimental instrument suite as well as high-level expertise of its scientists. MGCL has state-of-the-art facilities for metal refinement, synthesis of new materials, and the preparation of high-quality single crystals with several different techniques. The unique combination of different crystal growth methods allows users a great deal of flexibility and optimization of the technology of producing entirely new materials. Modern X-ray diffraction and electron microscopy instruments allow detailed

## Organizational chart of MGML



structural and phase characterization of samples. MPML offers the measurement of a wide portfolio of physical (magnetic, transport, thermal, acoustic and elastic) properties of materials through several complementary experimental methods. The extensive range of MGML instruments makes it possible to carry out the measurements in the temperature range from mK up to several hundred degrees Celsius, magnetic (up to 20 T) and electric (from -50V to + 50V) fields, hydrostatic and uniaxial pressures up to 15 GPa. Also important is the possibility of preparation, characterization and measurement of uranium materials, for which the institution has the appropriate license. Interconnection of this wide range of experimental techniques for the preparation, characterization and measurement of physical properties makes MGML a unique research infrastructure in the Czech Republic, fully comparable with the world's leading laboratories.

## Our laboratories are located at three places in Prague:

The MGCL technology laboratories are located in the building of the Faculty of Mathematics and Physics – Ke Karlovu 5. You can also find the administration unit here.

The measurements of material properties are performed mostly in the cryo-pavilion of the Faculty of Mathematics and Physics in Troja, V Holešovičkách 2. The helium liquefier located in this building supplies all the cryogenic needs of the laboratory.

Some instruments for measurements of material properties are situated also in the building of the Institute of Physics, Cukrovarnická 10.

# Scientific Highlights

## Ferromagnetic van der Waals materials for 2D spintronics

First isolation of graphene lead to the Nobel Prize in Physics in 2010 and opened a new avenue for application of numerous 2D layered materials having some interlayer bonds realized through weak van der Waals forces similar to graphite. Slabs of atomic layers of these 2D vdW materials can be easily exfoliated by breaking the vdW bonds (similar exfoliation of graphene sheets from graphite) and used to fabricate ultrathin devices exploiting their unique functional properties.

For fabrication of spintronic devices ferromagnetic and antiferromagnetic 2D vdW materials are essential [1,2]. Transition-metal trihalides (TX3) are promising for spintronic applications. Their crystal structure consists of a transition-metal layer sandwiched between two layers of halogen (see Fig. 1).

VI3 has received significant attention only in the past year [3-6]. It is known as a ferromagnet below TC ≈ 50K with complex magnetocrystalline anisotropy [4,6]. Limited unambiguous information on VI3 crystal structure, which has a decisive role for anisotropy, motivated us for a thorough structure study within the temperature interval 5 – 300 K. It was only known that VI3 has at room temperature a rhombohedral (trigonal) structure shown in Fig. 1 which transforms at Ts = 79 K to the low-temperature monoclinic crystal symmetry C2/c [3,4].

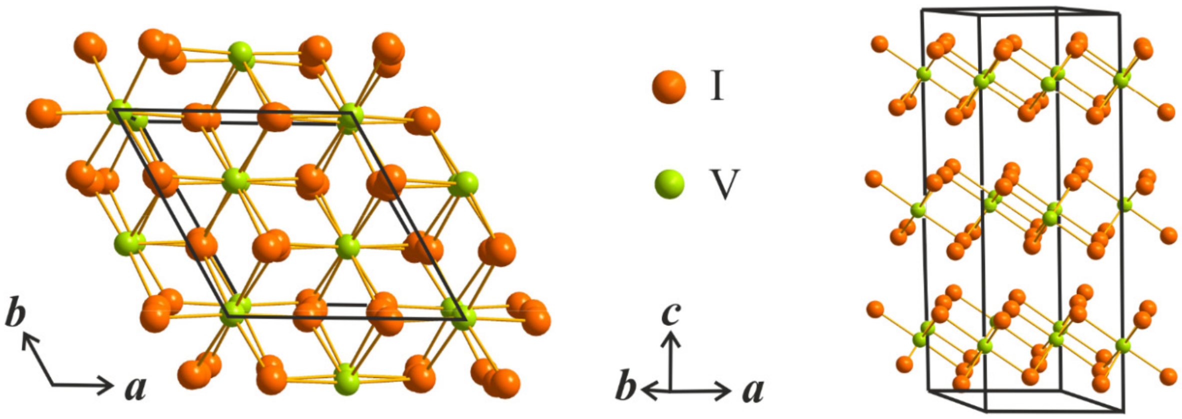



**Fig. 1.** Schematic picture of the room-temperature rhombohedral structure of VI3

Our detailed measurement and analysis of more than 2000 reflections from a VI3 single crystal at 250 K confirmed the existence of the room-temperature trigonal structure of the R-3 (148) space group. The common structural motif of TX3 compounds is a honeycomb net of transition-metal cations as shown in Fig. 1. The VI3 RT trigonal structure of the BiI3 type is characterized by the ABC layer stacking sequence. The subsequent layers are shifted along one of the V-V bonds. The honeycomb net is regular due to the three-fold symmetry.

When decreasing temperature, the (hhl) X-ray diffraction peaks suddenly split at temperatures between 79 and 78 K (see Fig. 2). This result unambiguously confirms that VI3 undergoes upon cooling a structure phase transition between the HT trigonal and the LT monoclinic phase at Ts = 79 K. In addition, the transition temperature Ts has been unexpectedly found by specific-heat measurements to decrease in magnetic fields applied along the trigonal c-axis.

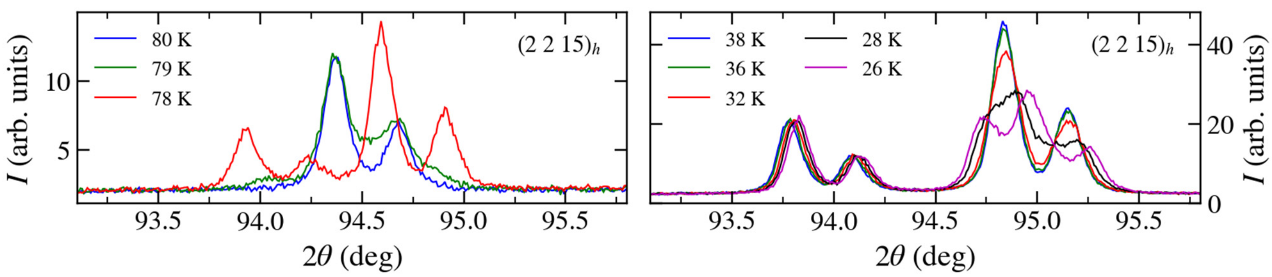



**Fig. 2.** Evolution of (2 2 15) diffraction peaks of the VI3 single crystal in the temperature intervals from 78 to 80 K (left panel) and 26 to 38 K (right panel).

Another surprise has been observed upon cooling VI3 across 32 K, in particular further splitting of some diffraction peaks indicating further lowering the crystal symmetry from monoclinic to triclinic. We associated this transition with the transformation of the ferromagnetic structure below 36 K, which has been reported by Gati et al. [5] from a thorough NMR study. The relations of structural transitions with magnetic phenomena in VI3 suggest a considerable role of magnetoelastic interactions in physics of this compound.

Our research was done in collaboration of the Department of Condensed matter Physics of the Faculty of Mathematics and Physics of Charles University with colleagues of the Institute of Physics of the Czech Academy of Sciences and Department of Physics and Astronomy, Seoul National University. The results of this VI3 structure study have been recently published as a Rapid Communication in Physical Review Materials [6] where all details of the structure study can be found.

This work is a part of the bilateral Czech–Korean research project which is financed by the Czech Science Foundation Grant No. GACR 19-16389J and by Grant No. IBS-R009-G1 provided by the Institute for Basic Science of the Republic of Korea. Most of the experiments were carried out in the Materials Growth and Measurement Laboratory MGML.

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## Magnetically doped topological insulators

The Nobel Prize in Physics was awarded for the discovery of the topological properties of solids in 2016. Our current research was focused on the first discovered three-dimensional (3D) topological insulator (TI) Bi2Se3, a material that acts as an insulator in the inside but contains free electrons on the surface.

The physics of TIs was first studied in two-dimensional (2D) HgTe/HgCdTe quantum well systems, which possess one-dimensional spin-polarized helical edge states. The corresponding class of 3D TIs were later found in melt-grown chalcogenide-type materials Bi1-xSbx, Bi2Te3 and Bi2Se3 with large spin-orbit coupling.

The significant manifestations of "topology" of the material is that surface electron states exhibit a characteristic helical spin texture [**look red arrows in Fig. 1 (up, left)**] and a linear dispersion without energy gap (Δ = 0) as shown in **Fig. 1 (up, left)**. For electrons residing in these so-called topological surface states 180 degrees backscattering is forbidden. Electron can travel around the Fermi circle with a quantum phase of π, called Berry phase, in the presence of disorder. As a result, the material exhibits greater electrical conductivity.

If there is a magnetic impurity in the topological material, this property could be lost. This is also associated with the loss of linearity of the dispersion spectrum and the creation of an energy gap (Δ >0), **as sketched in Fig. 1 (up, right)**. Recent theory [*Physical Review Letters* ***112 (2014)*** *146601*] on magnetically doped topological insulators predicts that quantum corrections Δκ to the temperature dependence of the conductivity

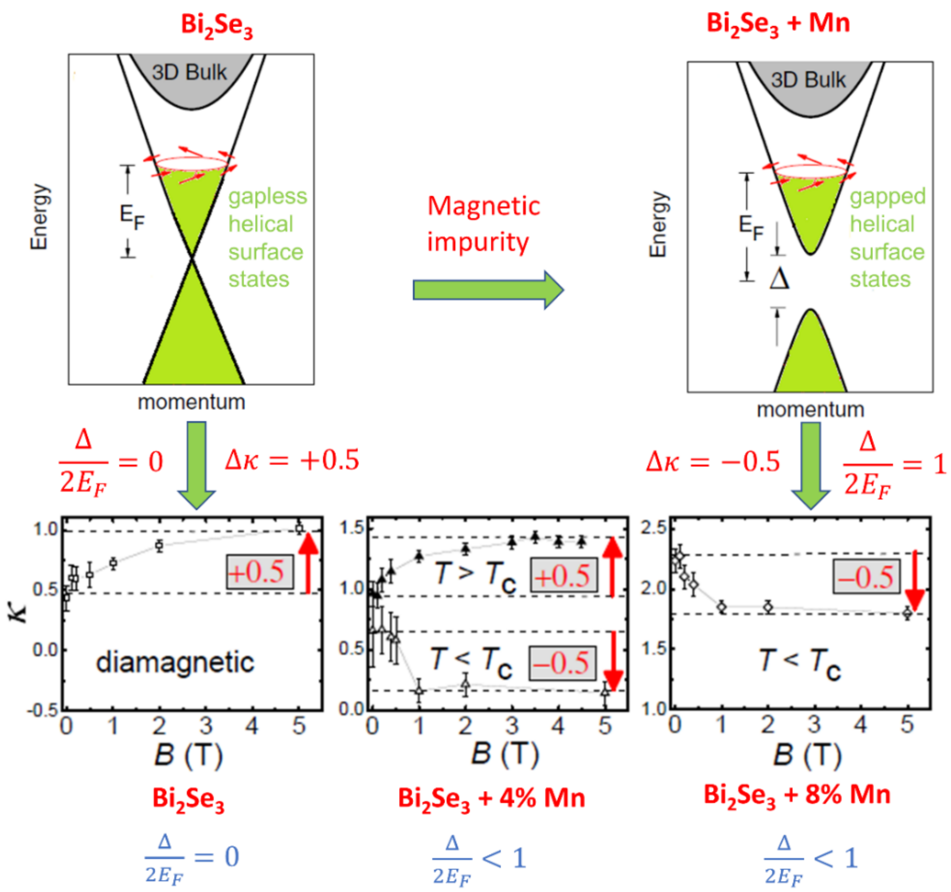



**Fig. 1 (up)** **S**chematic drawing of the band structure of the topological insulator with intact topological surface states (left) and topological surface states affected by magnetic doping produced the energy gap Δ (right), $E_F$ represents a Fermi energy **(down)** Respective experimental values of *k* at temperatures above and below the ferromagnetic Curie transition for the samples with different manganese concentrations.

can change sign across a ferromagnetic Curie transition TC. The change is attributed to a suppression of the Berry phase of the topological surface state, caused by a magnetically induced energy gap. The aim of our work was to experimentally test existing theoretical predictions of the magnetic field dependence of the parameter κ for the samples with different concentration of magnetic dopants down to very low temperatures, where ferromagnetic order sets in.

To that end, it was necessary to realize the measurement of electrical currents through a very thin layers of nanometer thickness and detect quantum transport properties at liquid Helium temperatures. Herby, so-called Hall-bar structures [**see Fig. 2**] had to be prepared by electron beam lithography using a scanning electron microscope.

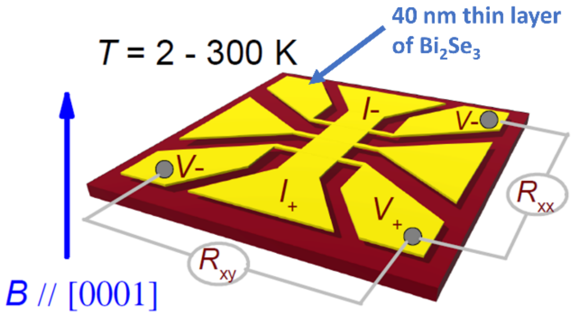



**Fig. 2.** Hall bar measurement geometry prepared by Electron Beam Lithography.

Our experiments have shown that theory describes the behavior of pure Bi2Se3 well, where κ changes by +0.5 is predicted for gapless topological surface states **[Fig. 1 (down, left)]**. However, contradictions with theory appear in the case of $Bi_2Se_3$ doped with magnetic manganese. At first glance, the opening of the energy gap brings the correct change of the parameter *κ* about -0.5. Unfortunately, the energy gap Δ presented in our system **[Fig. 3 (right)]** is not large enough to cause that big a change. There is a conflict with the existing theory, and thus another phenomenon must be responsible for *κ* changing by -0.5.

We demonstrate that a sign change can actually appear at a constant Berry phase and resolve contradictions by extending theory to a non-monotonic temperature scaling of inelastic scattering lengths showing a turning point at the Curie transition with $T_C$ = 5 and 6 K for Mn concentrations $x_{Mn}$ = 4 and 8%, respectively **[Fig. 3 (left and middle)]**. The results of our study were published in *Physical Review Letters* ***123*** *(2019) 036406*.

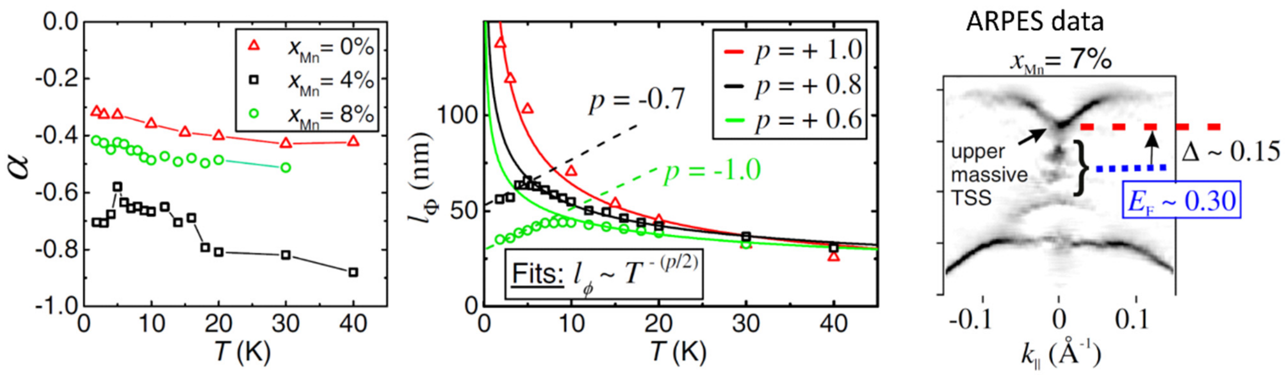


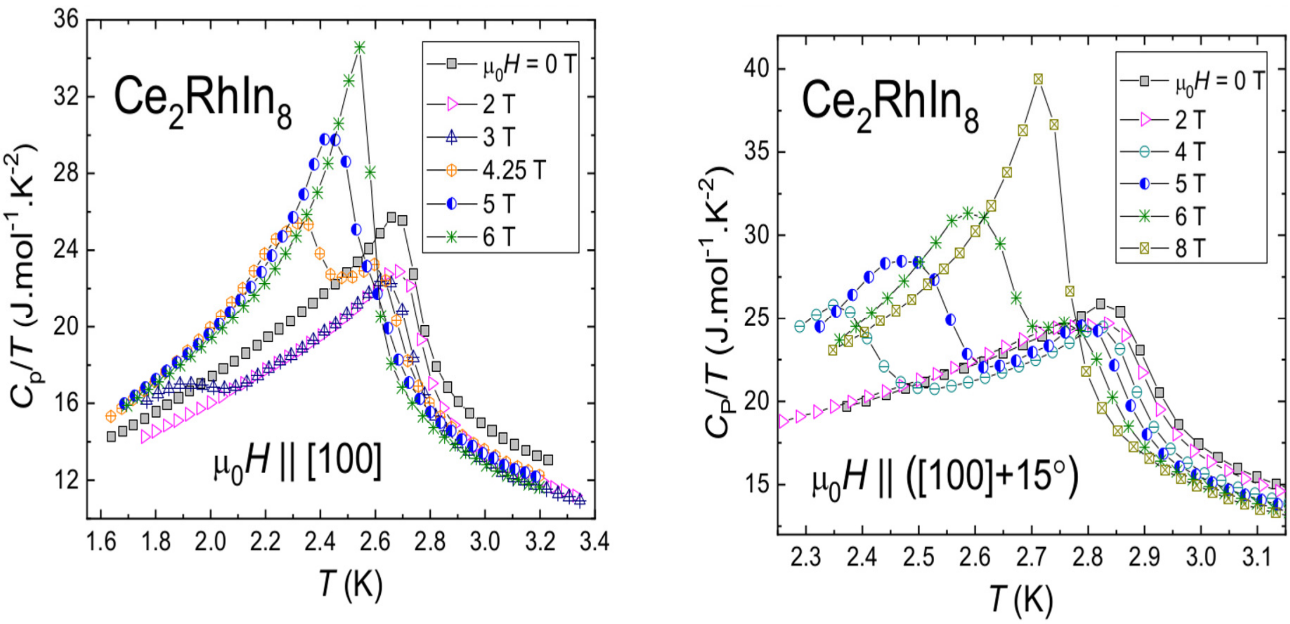

**Fig. 3** Temperature dependence of **(left)** average number of two-dimensional transport channels α(*T*) and **(middle)** phase coherence length $l_\phi(T)$ for different Mn concentrations $x_{Mn}$. Values were derived by fitting the magnetoresistance data by standard Hikami-Larkin-Nagaoka (HLN) theory and assuming lϕ(T) ~T-p/2 for T >TC (p > 0, solid lines) and T < TC (p < 0, dashed lines). (right) Surface state band structure visible in angle-resolved photoemission spectroscopy (ARPES) for xMn = 7% measured at hν = 18 eV and T = 100.

# Technical Developments

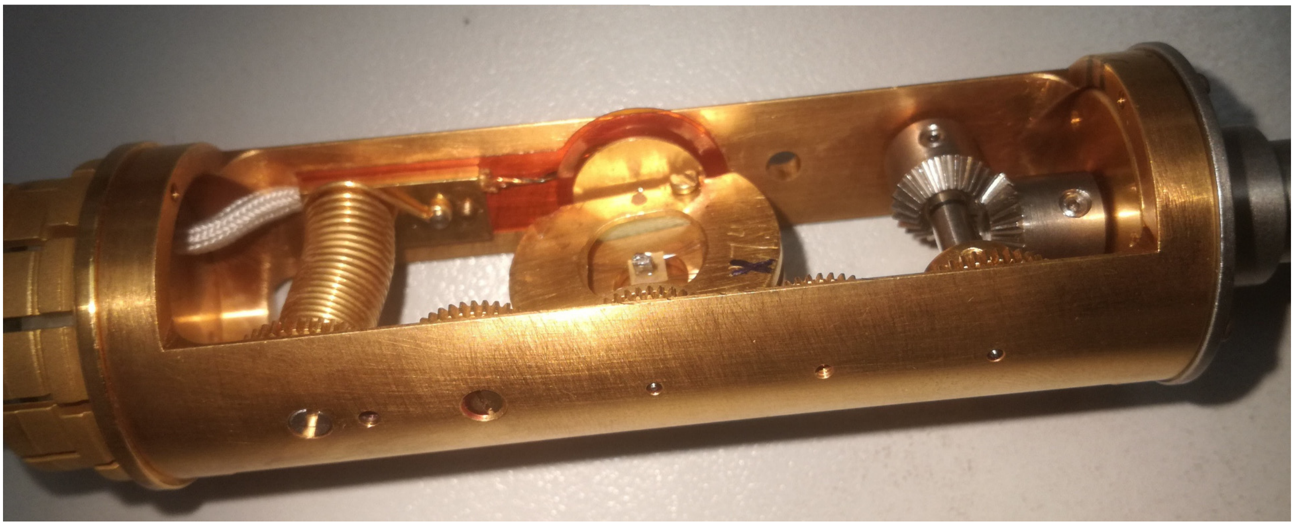
## Material Properties Measurement Laboratory (MPML)

In connection with the extension of the MGML by the Cukrovarnická site a thorough check of recovery lines in Cukrovarnická location was done in order to bring up the installation to current operational standards. This included identification of leaks and weak spots and disconnection of unused branches.

Due to increased user demands we implemented the measurement of heat capacity as the function of the angle between the sample and applied magnetic field. The proof of concept of this approach was the measurement of the (B, T, α) magnetic phase diagram of the $Ce_2RhIn_8$ single crystals at low temperatures (A. Bartha et al., Phys. Rev. B 100, 184425, 2019). The holder itself is designed to be used in wide range of temperatures (1.8 – 400K) and fields (0 – 14T).

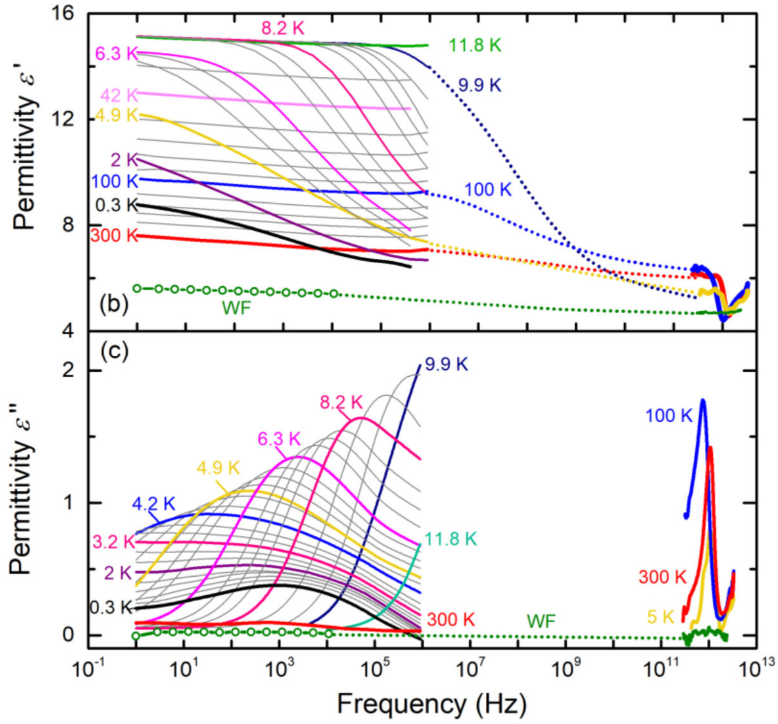



*Temperature dependence of heat capacity in different magnetic fields applied with various deflection from the c-axis.*

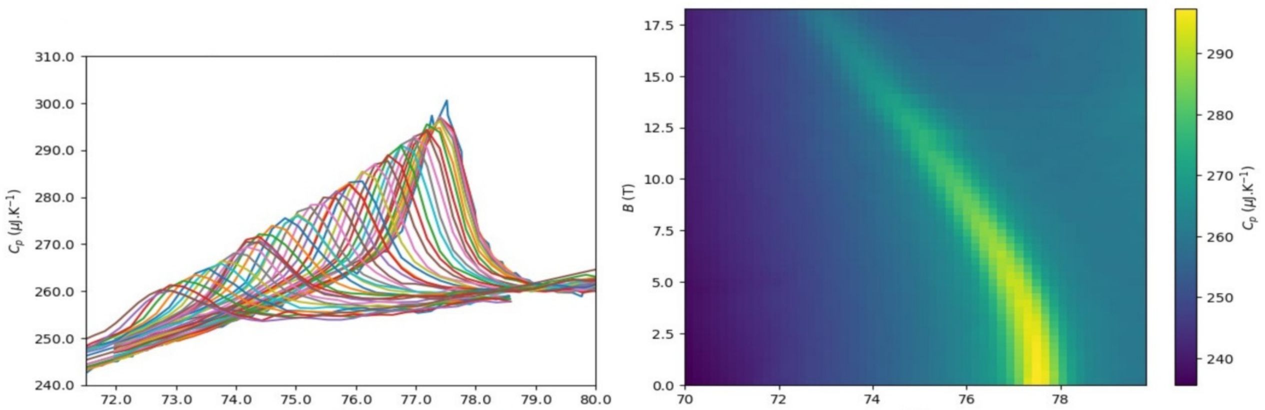


*The insert with the rotator and sample prepared for the measurement.*

Another often repeated request from the users was the possibility of the extension of the temperature range for the measurement of dielectric properties down to sub-kelvin temperatures. By modifying the $^{3}$He insert to the 20T cryostat we succeeded in the implementation of the measurement of wide band dielectric spectroscopy down to very low temperatures (below 2K, publication under review).




*Temperature evolution of relaxational absorption band in hydrous cordierite crystal for E || a polarization at different frequencies as indicated.*

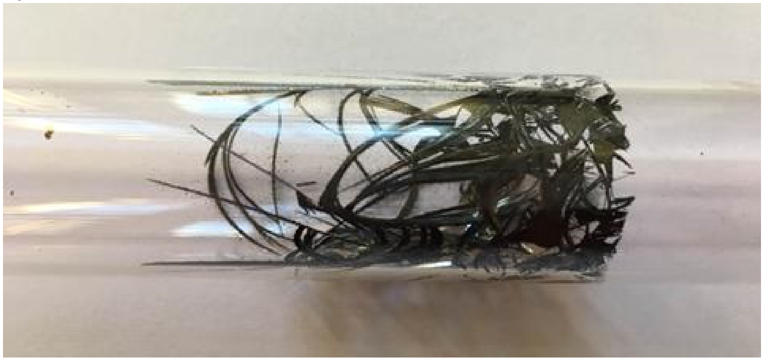



*LEFT: Temperature dependence of the heat capacity at field 0 to 18.3T. RIGHT: Resulting magnetic phase diagram of the given transition.*

We continue with step-by-step- extension of the 20T cryostat capabilities by implementation of measurement of heat capacity at very high static magnetic fields (up to 19.5T) and various temperatures.

## Material Growth and Characterization Laboratory (MGCL)

The user requests for technical instrumentation in MGCL laboratories copy the subjects of in-house running projects where we witness a clear increase of single crystals growth of non-metallic materials. Namely, the increase of usage and user interest were primarily in gradient furnace with implemented chemical vapour transport (CVT) technique for growth of transition metals halides. An example of produced crystal is shown below.



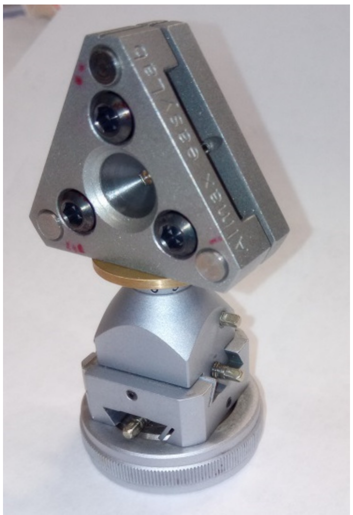
*Single crystals of $VI_3$ grown by CVT.*

The single crystal growth process in optical furnace was often seriously affected by vibration which originated from walking users in the laboratory when using other installed instruments. After installation of the new optical furnace we have performed re-arrangement of the instruments and both floating zone furnaces are located in the lab no.2, completely isolated from other users. The lab no. 2 is now dedicated for instruments for single crystals growth only. Moreover, we have invested effort to develop remote control for all instruments in the lab which was successfully implemented.

The digital microscope was installed to the glovebox. It allows handling of small samples in protective environment. Further improvement concerns installation of a new insert to vertical induction furnace for casting of precursor for single crystals growth by floating zone.

## Sample environment

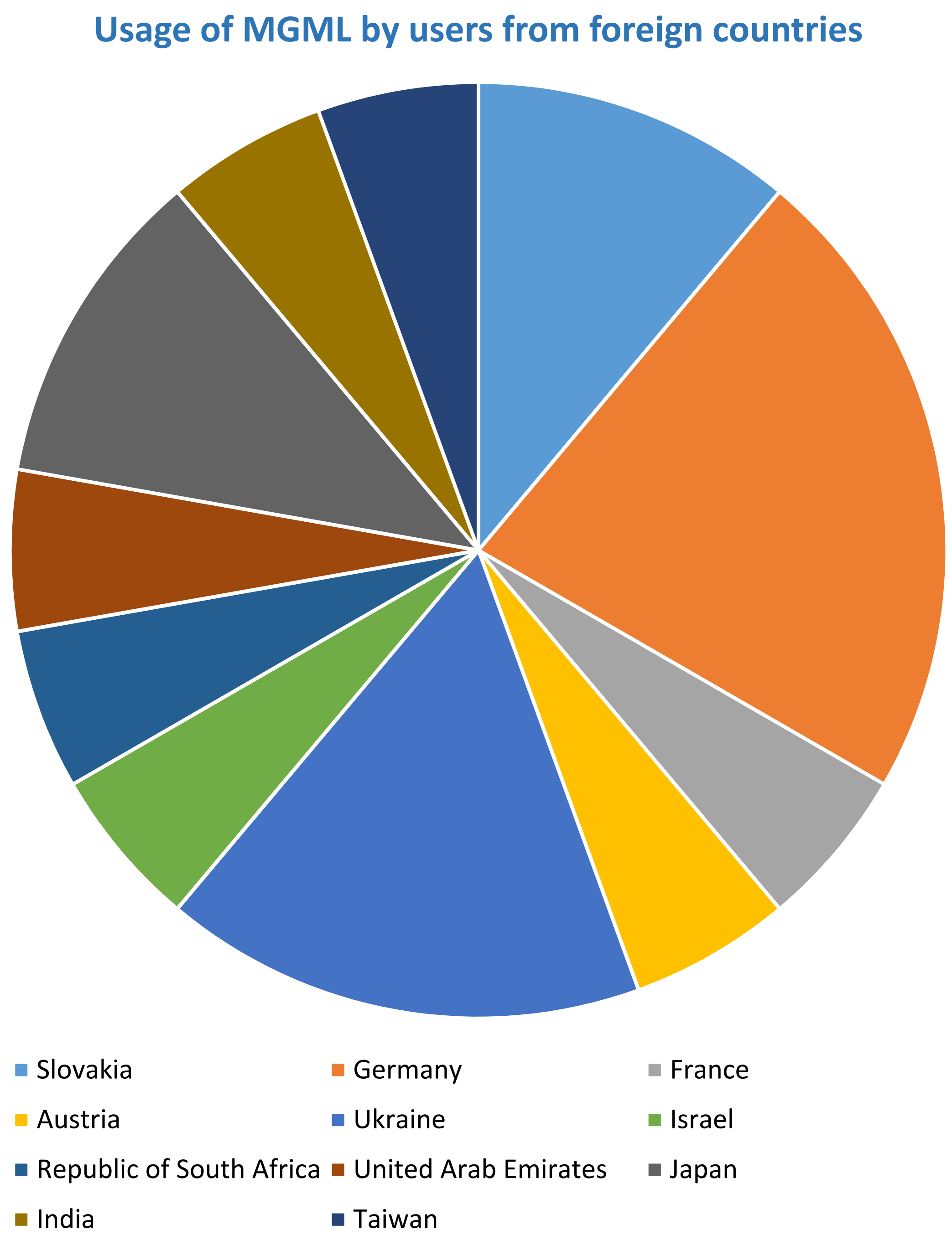
A new diamond-anvil high-pressure cell was delivered in December 2019 to our laboratory. The cell is dedicated for room temperarture High-pressure XRD experiments with pressure limit up to about 20GPa. The sample is accessible by the X-ray beam through anvils seated in a tungsten carbide support with the open angle of 85°. The available sample space ranges from 0.2 to 0.5mm in diameter. We are running the test measurements. After implementation it will be used for determination of the lattice changes upon pressure application, e.g. the compressibility of the studied material to the high pressure.



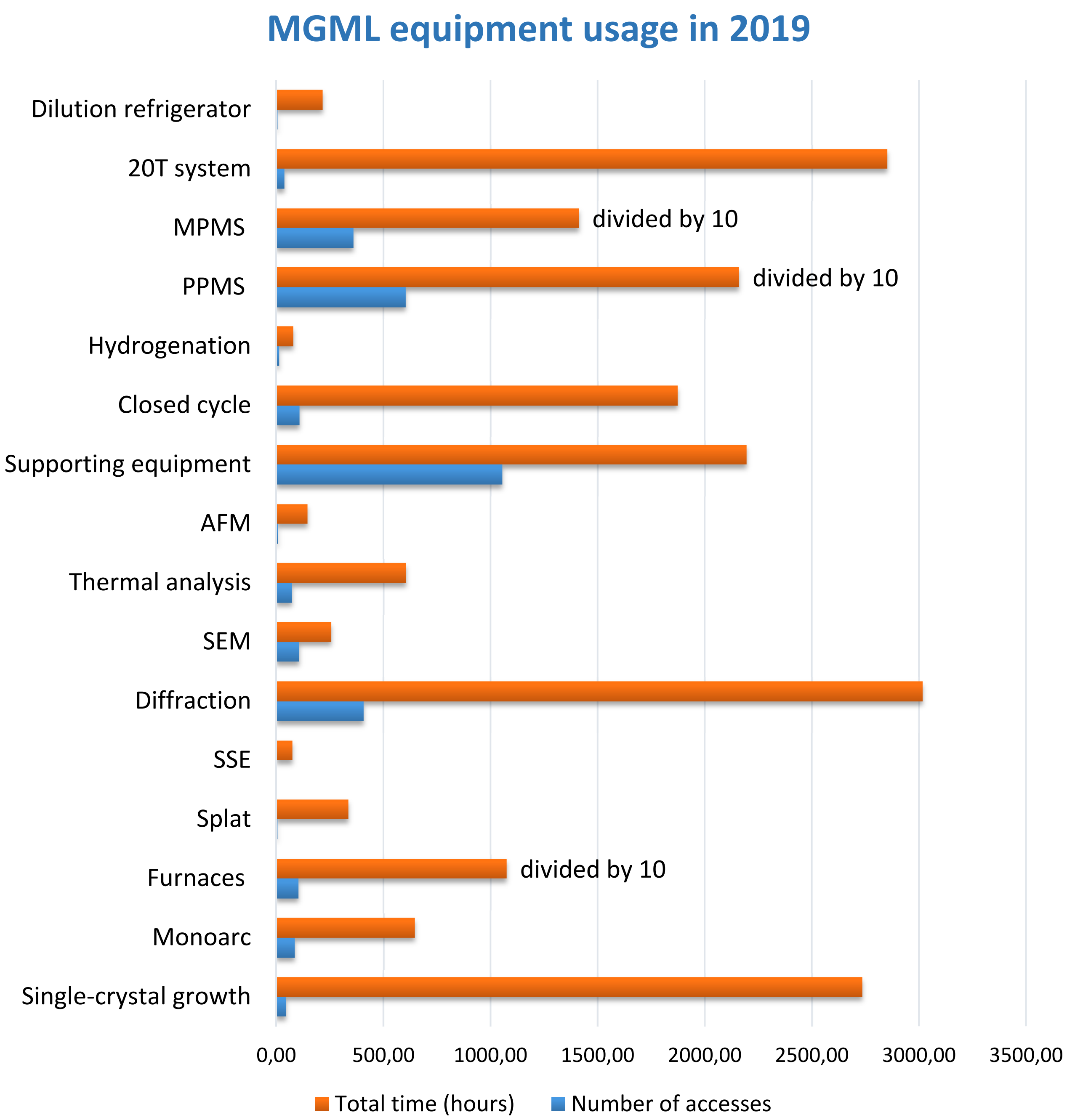
*The high-pressure cell for the room-temperature XRD experiments, mounted on a diffractometer holder adjusted for the cell.*

## User Programme

The new user portal was put into operation. It enables all the users to submit a proposal, report or book a time on individual instruments. We run the proposal-based user system starting this year. In total, 46 experimental proposals were accepted in 2019, among them 21 long-term proposals, 22 standard and 3 test proposals. Experiments were performed by 69 individual users from 12 different countries. Namely, Czech Republic, Slovakia, Germany, France, Ukraine, Austria, Israel, Republic of South Africa, United Arab Emirates, Japan, India and Taiwan. Majority of experiments (86 %) were performed by users from the Czech Republic. The usage by users from other countries is represented in Figure below.



The usage of individual equipment is depicted on the following Figure. The PPMS, MPMS, 20T system, furnaces and crystal-growth equipment together with the X-ray diffraction analysis are the most used ones. The Figure also illustrates somewhat different manner of use: while the time per access is relatively very long for the 20 T system, PPMS or MPMS, significantly shorter time per access is found for characterization techniques (diffraction, SEM). This is a natural difference reflecting different nature of use of these instruments.

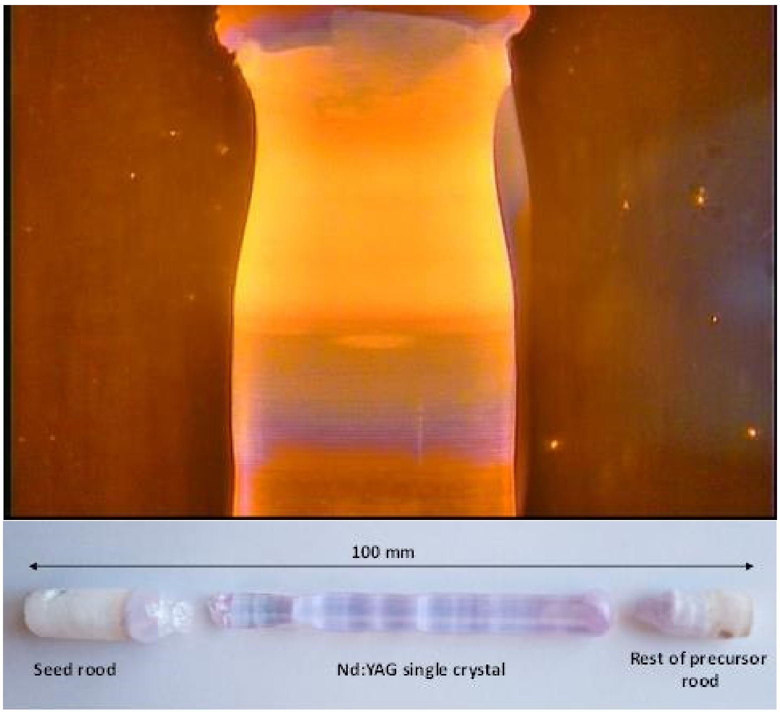


# Collaboration with industrial sector

## Crytur Company – development of new photonic single crystal

The unique mixture of available single crystals growth techniques in MGML has induced interest not only in users from academic institutions but also in industrial companies. MGML has established collaboration with the Crytur Company in Turnov which is the global leader in industrial growth of photonic single crystals based on yttrium-aluminum garnets, perovskites and wide series of their substituted derivate. MGML was implemented to the company development programme to investigate new compositions of photonic single crystals to improve their properties for laser applications and detectors.

The primary technique for single crystals growth of photonic crystals is Czochralski method in industrial scale and mPD method in laboratory scale. Both methods are limited by the properties of material of the crucible, typically W, Mo, Re, Ir in which the batch is melted. The limits of crucible usage are its melting point in contrast to high melting point of garnets and perovskites, price, lifetime, extreme sensitivity on residual oxygen at high temperatures and possible contamination of the melt by material of the crucible. All these obstacles can be solved by floating zone method. In floating zone method, melt is trapped levitating in between the feeding polycrystalline precursor and the single crystal by surface tension, see the Figure. This method is thus crucible free, that brings several fundamental advantages. Here, the selection of the material is not limited by melting point of material of the crucible, melt is not contaminated by material of the crucible and growth process can be realized in any type of environment, particularly oxygen even at high pressure. The application of the floating zone method installed in MGML has allowed one to investigate the compositions of photonic single crystals which were impossible to grow by traditional techniques. The company program is realized through a bachelor thesis of a student at the Faculty of Mathematics and Physics, Charles University. A bilateral contract with the Crytur Company has been prepared as well as joined project to the Czech Science Foundation.




*Single crystals growth process by floating zone method of Nd:YAG for laser applications(top) and an example of the grown single crystal (bottom).*

## Conferences, Workshops, Events

The international conference **European High Pressure Research Group Meeting** (EHPRG, https://www.ehprg2019.org) was organized by several members of MGML.

EHPRG conference is an essential event joining experts from various scientific fields by the method used - i.e. high pressure. The conference counted more than 280 contributions covering wide range of topics including Geosciences, Material Research, Chemistry, Bio- and Food sciences, Theory upon high-pressure application, New phenomena under pressure and - for high-pressure studies naturally essential topic - High pressure instrumentation. About 270 participants were present at the conference, coming from 26 countries. (The success of the meeting was clearly evident from the above-average number of arrived participants, also from the response of the participants themselves, which was confirmed by the satisfactory result of the survey filled by some number of the participants after the conference.)

**Delegates of MGML involved in organization of the EHPRG**
(*members of the EHPRG Organizing committee*):

- Jiří Prchal (conference chair, Charles University)
- Jan Prokleška (Charles University)
- Jaroslav Valenta (Charles University)
- Martin Míšek (Programme committee chair, Institute of Physics CAS)
- Jiří Kaštil (Institute of Physics CAS)


*Opening of the EHPRG 2019 conference (left) and the photo of conference participants (right).*