307. Rozhovory v rámci Krystalografické společnosti - EXCURSIONS

„Crystallography in the Nuclear valley – from materials for nuclear safety to cultural heritage“

1) Analytical methods (not only) for cultural heritage

a. X-ray powder (micro-)diffraction, mobile FTIR and Raman spectrometers (ALMA)

The Academic Laboratory of Material Research of Painting Works (ALMA), a joint workplace of the Institute of Inorganic Chemistry of the Czech Academy of Sciences and Academy of Fine Arts in Prague, deals with natural science research in the field of heritage preservation and systematically focuses on deepening knowledge about art materials and painting techniques. ALMA then incorporates this knowledge into a comprehensive assessment of the work of art in terms of its origin, age and authorship. The laboratory develops methods of instrumental analysis and interprets the results in the context of the history of painting and the technology of materials.

X-ray powder diffraction (XRPD) has a long tradition at ALMA. As one of the first laboratories in the field of cultural heritage research, ALMA started using laboratory micro-XRPD enabling the analysis of heterogeneous micro-fragments or their cross-sections taken by restorers from easel or wall paintings or polychromies. This technique is non-destructive to the sample, and the sample can thus be analyzed by other microanalytical methods (SEM/EDS, FTIR, RAMAN, etc.). In some cases of artworks with smaller dimensions (e.g. portrait miniatures with the usual dimensions of approx. 10 cm x 10 cm), a programmable or manual sample stage can be used, and therefore powder diffraction is non-invasive with respect to these artworks. The XRPD laboratory is currently equipped with three diffractometers from the company (Malvern-)PANalytical, which cover different types of tasks – measurements in the reflection Bragg-Brentano arrangement or in transmission geometry, micro-diffraction measurements with different types of collimators, high-temperature measurements. Configurations with a copper or cobalt X-ray tube are available.

The shared mobile laboratory includes portable devices owned by both institutions. It enables measurements to be carried out in situ – in museums, galleries, churches, castles. Analytical screening is important in the first stages of restoration (e.g. probing), especially in cases where transporting of artworks is not possible and/or desirable. It can also be part of art-historical research independent of restoration. The Institute houses a mobile FTIR spectrometer and a Raman spectrometer with fiber optics and a mobile microscope. In addition to studying real works of art, the aforementioned methods are used to study the degradation of painting materials in model experiments.

During the excursion, examples of the methods described above will be demonstrated and questions about the methodology of analysis of samples from works of art will be answered.

b. Excursion to the facilities of the Laboratory of TANDETRON Accelerator (canam.ujf.cas.cz/lt)

The TANDETRON accelerator laboratory (canam.ujf.cas.cz/lt) is part of the Center for Accelerators and Nuclear Analytical Methods (CANAM). The Tandetron 4130 MC accelerator, commissioned in 2005, is used to produce ion beams with energies ranging from 400 keV to 30 MeV of almost all elements of the periodic

table. It is a medium current (MC) version of the accelerator with a terminal voltage variable from 200 kV to 3 MV. The produced ion beams are used for the analysis of trace elements using nuclear analytical methods and for the synthesis of new materials and structures, or for the modification of surfaces by irradiation with ion beams.

The main equipment of the laboratory are instruments for materials characterization by ion analytical methods (RBS, RBS-channeling, ERDA, ERDA-TOF, PIXE, PIGE, PESA and Ion-Microprobe with a lateral resolution of less than 1 µm, and scanning ion transmission microscopy STIM). Ion beams are used for targeted modifications of the composition, structure and morphology of surfaces and surface layers of materials using ion implantation, ion lithography and ion irradiation. A wide portfolio of methods is used in materials research in optics, nano-optics and electronics, bionics, sensors, radiation testing , dosimetry, semiconductor doping, space and nuclear energy technologies, wear-resistant materials, etc. The ion microprobe is effectively used for ion beam lithography and for 3D elemental mapping in combination with STIM, PIXE, PIGE and RBS.

Flexible instrumentation for ion implantation can be used for modifications, intentional introduction of defects into the microstructure, nanostructuring of materials as well as for testing ionizing radiation in dosimetry and detector development. An external beam is also available for analyzes of samples and artefacts that cannot be placed in a vacuum (e.g. archaeological artefacts, biological preparations, vacuum-unstable materials, etc.). Ion analytical methods are successfully and effectively used in many applications: ion beam interaction with solids, nanostructuring, synthesis and characterization of nano and microstructures, quantitative and qualitative elemental analysis and determination of depth distribution of elements with high resolution, characterization of trace elements, etc.

The parameters of the laboratory's experimental equipment are fully comparable with similar equipment in the world. The laboratory is unique in the Czech Republic and is able to meet specific requirements of Czech and foreign research organizations.

Restrictions: The workplace TANDETRON is located in the controlled area where ionizing radiation is used. Entry is prohibited to persons under the age of 18, pregnant and lactating women. It is recommended to come to the tour in closed shoes and long pants.

2) Excursion to the facilities of the CANAM Neutron Physics Laboratory at the LVR-15 research reactor (canam.ujf.cas.cz/npl)

Neutron Physics Laboratory (canam.ujf.cas.cz/npl) employs for its research the horizontal thermal neutron beams and the vertical irradiation channel of the light water research reactor LVR-15, which is operated by the Research Center Řež, s.r.o.. Neutrons are used for experiments using neutron scattering methods, and for nuclear analytical methods.

Neutron scattering enables to study the structures and microstructures of materials (e.g. advanced metals and ceramics, but also archaeological artefacts) in various size scales, from the arrangement of atoms in a crystal lattice to the study of heterogeneities in materials at the nano- and microscopic scales. The high neutron penetrability to the most of materials allows these tests to be performed non-destructively in the bulk of material and/or in a special sample environment (low and high temperatures, mechanical stress). Five neutron diffractometers specialize in powder diffraction (MEREDIT), residual stress mapping (SPN-100), small-angle neutron scattering (MAUD), high-resolution neutron diffraction (TKSN-400) and neutron optics development (NOD).

Nuclear reactions of neutrons with matter are used to analyze concentrations or concentration profiles of elements in solids and for fundamental nuclear physics. Neutron activation analysis (NAA), neutron depth

profiling (NDP), prompt gamma activation analysis (PGAA)) are techniques that can be used, for example, in the environmental, biomedical, geo- and cosmochemical fields, as well as in archeology and materials research.

Neutron Physics Laboratory also provides these experimental facilities and related support to external users in the open access mode.

Restrictions: The workplace is located in the controlled area where ionizing radiation is used. Entry is prohibited to persons under the age of 18, pregnant and lactating women. It is recommended to come to the tour in closed shoes and long pants.

3) Analytical methods for nuclear materials (max. 15 + 15 partcipants) – CVŘ

a. Excursion to the hot cells CVŘ (https://www.cvrez.cz/cs/vyzkum-a-sluzby/provoz-reaktoru/horke-komory-12202)

Hot gamma cells are intended for working with radioactive structural materials of nuclear power plant components with a maximum processed activity of ~300 TBq (converted to Co60 energy). The perimeter shielding structure consists of steel blocks with a maximum thickness of 500 mm and windows made of leaded glass, allowing operators to monitor the work inside the hot chamber. Inside the shielding blocks is located (for each hot chamber) a removable internal hermetic box made of stainless-steel sheet equipped with a window with cover glass.

The LVR-15 research reactor is used for irradiation of materials, production of radiopharmaceuticals and experiments using neutron radiography and other applications of neutron beams. After irradiation, the radioactive material is transported to a complex of hot cells in a transport container with sufficient shielding even for highly irradiated samples.

The instrumentation of the hot cells covers the entire material research process, i.e. from the production of samples from the basic irradiated material through mechanical tests, the preparation of metallographic samples to the final analysis of the microstructure. In recent years, a unique infrastructure for testing irradiated concrete and aggregates has been built in hot cells - accurate measurement of dimensional changes, changes in density and strength properties due to radiation and evaluation of changes at the structure level of individual components. The outputs of these analyses form the basis for numerical modelling, prediction and life extension of biological shielding of nuclear power plants.

The system hot cells is a supporting facility of the Merged LRI Czech International Centre of Research Reactors (CICRR) and within which we provide the possibility to use the experimental equipment also to external users in the Open Access mode (**https://www.cicrr.cz/**).

b. Laboratories of the Centre of Highly Sensitive Analytical Instruments (CVCAP)

In the laboratories of the Centre of Highly Sensitive Analytical Instruments (CVCAP), it is possible to prepare samples from non-irradiated and irradiated metallic and non-metallic materials for subsequent analysis on a high-resolution transmission electron microscope, a scanning electron microscope with a focused ion beam or X-ray diffractometer. The main focus of the Centre is research and development of materials used in

nuclear and conventional energy industry. It is primarily an assessment of the mechanical, physical and chemical properties of materials after their exposure to a working or extreme type of environment, such as humidity, pressure, chemical reactivity, mechanical tension, radiation.

The elemental and isotopic microanalysis group EIMA (Elemental and Isotopic Microanalysis), which is a member of the NWAL (Network of Analytical Laboratories) of the International Atomic Energy Agency (IAEA), carries out research and development in the field of nuclear forensic analysis. They specialize in the analysis of microscopic dust particles, especially their elemental and isotopic composition using the SIMS method. Another focus of the laboratory staff is the analytical support of governmental and international institutions during inspections of nuclear materials and police investigations of nuclear forensic incidents.

Restrictions: Both workplaces are located in the controlled area where ionizing radiation is used. Entry is prohibited to people under the age of 18, pregnant and lactating women. It is recommended to come to the tour in closed shoes and long pants.