

World's largest NaCl crystal model by Robert Krickl, exhibited in 2019 during the ECM32 in Vienna. Photo: Juergen Schreuer.



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Das Helmholtz-Zentrum Berlin betreibt die Synchrotronquelle BESSY II, die hochbrillante Röntgenstrahlung für die Forschung an unterschiedlichsten Materialien erzeugt. Die Fragestellungen reichen von der Festkörperphysik über die Werkstoffwissenschaften bis zu den Lebenswissenschaften. An den MX-Beamlines von BESSY II konnten bereits über 3000 Proteinstrukturen entschlüsselt werden. Zusätzlich bietet das CoreLab für Röntgendiffraktometrie ein breites methodisches Spektrum für kristallographische Untersuchungen an Pulvern und dünnen Schichten. Dieses Angebot steht Messgästen aus aller Welt zur Verfügung.

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DEAR READERS,

with this brochure, the German Crystallographic Society (DGK) would like to introduce itself to you. Using current examples, we would like to show you how crystallography can contribute to overcoming societal challenges in the areas of energy supply, sustainability and health.

Since the beginning of time, people have been fascinated by the sparkling stones and lustrous crystals they found in nature, even if they still did not understand what they were holding in their hands. The term crystal is derived from the Greek „krýstallos“, which means something like ice or frost. It is thought that in ancient times, people found quartz crystals in the Greek mountains and thought they were warm ice. Since then, attempts have been made to understand the crystals and their internal structure. Because, even if crystals do not have any miraculous healing powers, they still have fascinating properties that make our modern world possible in the first place. Almost all modern sciences, such as materials science or nanotechnology, are based on the findings of crystallography with its history of more than 2000 years. The often-used term structure-property relationships briefly summarizes what the task of modern crystallography is, because for a long time people have not only wanted to understand what they find in nature, they want to produce things in a targeted manner - from bronze swords to microchips. In general, materials should have desired properties: conduct electricity particularly efficient, generate light without too much waste heat or, as in a battery, incorporate charged particles but also release them again quickly, to name just a few examples. The basis for all these properties is the respective structure of the materials used, starting with the arrangement of the atoms in relation to one another, through the size and shape of the crystals to their distribution in the material. Because the term material often includes a combination of several crystalline or glassy materials.

All of this examines crystallography and thus provides the basic knowledge for many areas of physics, chemistry and engineering. Conversely, the natural sciences mentioned also provide valuable insights for modern crystallographers. This synergy enables a maximum gain in knowledge in order to finally recognize what holds the world together at its core and to find solutions for the challenges of our time. All the different approaches from the world of crystallography are brought together in this brochure to give a little insight into the work of modern crystallographers.

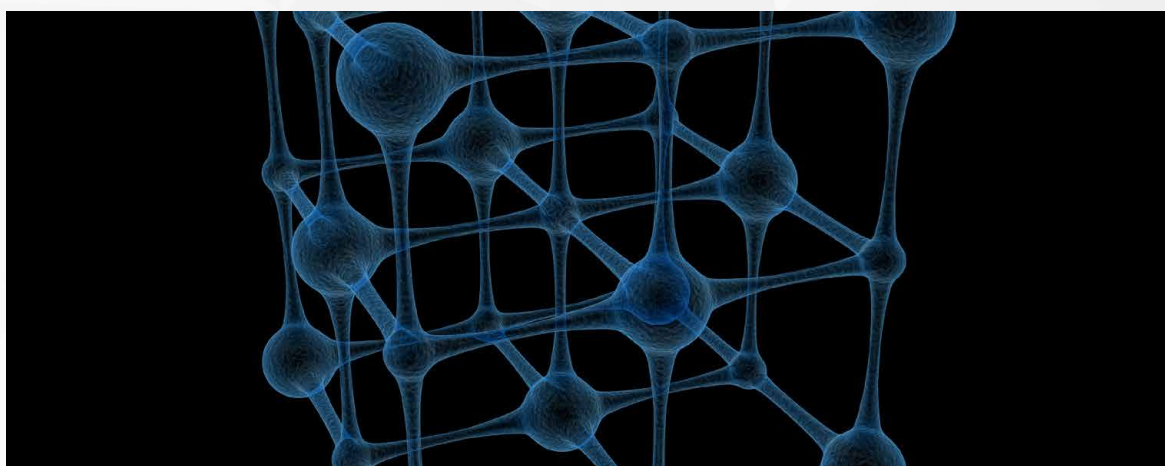
Further information and publications can also be found on our homepage: www.dgk-home.de

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THE GERMAN CRYSTALLOGRAPHIC SOCIETY



The working groups of the DGK conduct workshops, method courses and symposia on an on-going basis, each of which has a specific focus on crystallography. The German Crystallographic Society (DGK) was founded in Munich in 1991 through the consolidation of the West-German „working group crystallography“ and the East-German “Association for Crystallography”.

Today, the DGK has about 1000 members. The DGK promotes crystallography in teaching, research and industry as well as in public, especially through the exchange of experiences and ideas as well as advanced training in the national and international framework. For this purpose, the DGK annually organizes a scientific conference, the annual meeting.

Usually, the DGK awards several prizes each year in recognition of special scientific achievements: The Carl Hermann Medal is awarded to researchers for their outstanding scientific life's work and the Max von Laue Prize for outstanding young scientists. Furthermore, the Will-Kleber commemorative coin is awarded for excellent scientific work. The Waltrude and Friedrich Liebau Prize for supporting the interdisciplinary nature of crystallography is provided by the DGK on behalf of the Waltrude-und-Friedrich-Liebau-Foundation.

Since 2022, the Lieselotte Templeton Prize is awarded for very good bachelors, masters or similar theses. The DGK is a member of the international umbrella organizations, the European Crystallographic Association (ECA) and the International Union of Crystallography (IUCr), who organizes international conferences and workshops, respectively.



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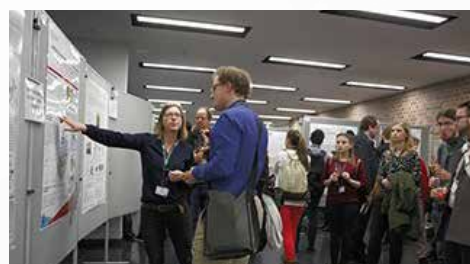
Crystallography

DGK ANNUAL MEETING



The DGK organizes an annual scientific conference at different locations. These annual meetings are organized by local members.

During the annual meeting, the latest scientific results will be presented in plenary lectures or lectures in thematically focused microsymposia.



During the poster sessions, current research results will be presented and discussed with the participants.

The annual conference offers space for the exchange of experiences and scientific discussion.



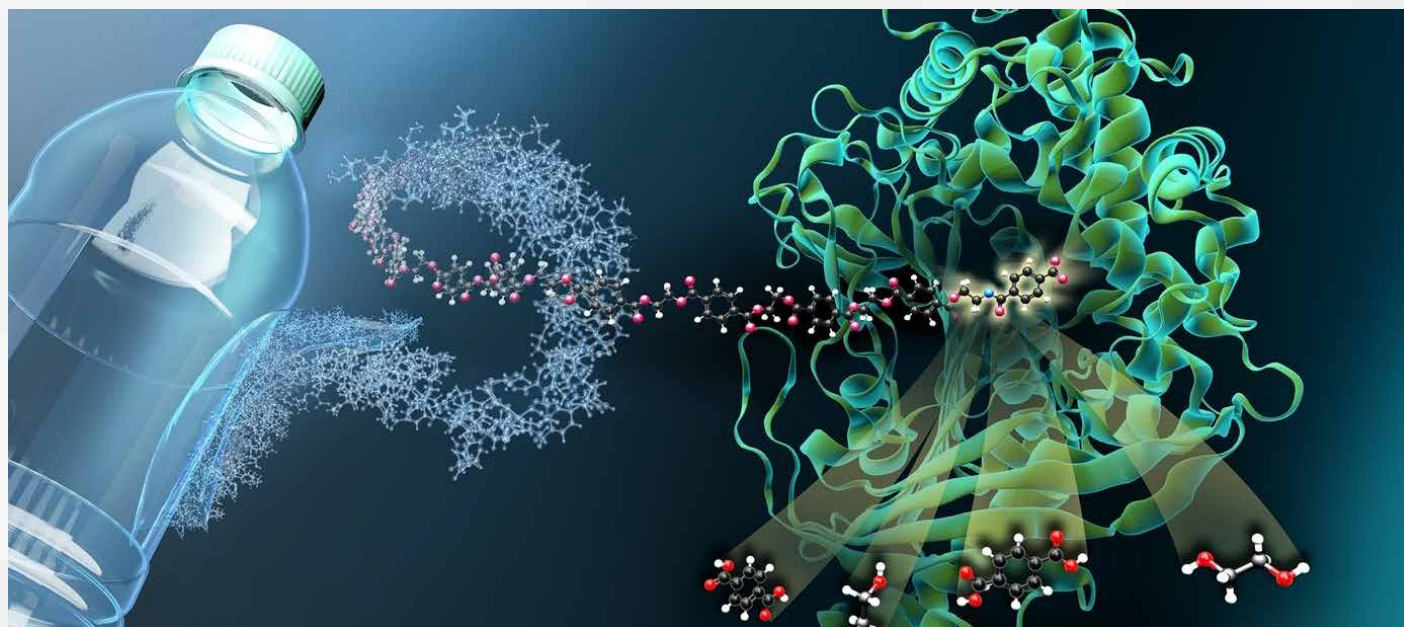
During the annual conference, various companies will provide information about their latest products and developments in the field of crystallography.

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CRYSTALLOGRAPHY AND ENVIRONMENT

For about 70 years, humanity has been producing synthetic polymers (plastics). At the end of their lifetime, a large part of this material ends up as waste in the environment. This plastic waste, often broken down into micro- and nano plastic particles, can now be found all over the world and has severe environmental consequences. The effects on human health and on flora and fauna are largely unexplored. However, it is possible that nature provides a solution to this problem. A bacterium called *Ideonella sakaiensis*, recently discovered by Japanese researchers, is able to break down the plastic polyethylene terephthalate or PET for short. It does this with two enzymes, PETase and MHETase, which break down the polymer into its basic building blocks ethylene glycol and terephthalic acid.

Both substances can then be used again for a new synthesis of PET. In the near future, enzymes could thus replace the current energy-intensive and crude oil-dependent recycling processes for PET and other plastics. However, the enzymes are not yet efficient enough for technical large-scale applications. For the biotechnological improvement, it is of great advantage to obtain the three-dimensional structures of the enzymes. Researchers have now succeeded in using crystallography to analyse the structures of PETase and MHETase in detail. The aim is now to improve both enzymes to a point where they can be used on a large scale. Crystallography will also play a decisive role in this.



Annual global production of synthetic polymers or plastics has already exceeded 10 billion tons. The currently available recycling technologies are generally not sustainable, incur high energy costs and lead to a gradual loss of quality of the recycle. The enzymatic degradation of plastics has recently proven to be a viable alternative.

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RAVAGED BY TIME: HOW CRYSTAL STRUCTURE SOLUTION HELPS TO PRESERVE CULTURAL HERITAGE IN MUSEUMS

During centuries of storage, heritage objects are exposed to humidity and atmospheric gases like CO_2 and O_2 . In addition, wooden furniture and showcases, in particular oak, emits considerable amounts of formic and acetic acid or the respective aldehyde precursors. This triggers corrosion processes that can lead to severe damage of the historic object.

The occurrence of needle-like efflorescence crystals on the surface of heritage objects, also known as “Byne’s disease”, has been a frequently observed problem in museums and collections since the end of the 19th century (Fig. 1, a–c). Either these efflorescence crystals overgrow the entire surface of an object or they crystallize in cracks and pores.

Mainly carboxylate containing salts like

- > $\text{Ca}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$
 - > $\text{Ca}(\text{CH}_3\text{COO})_2 \cdot \frac{1}{2}\text{H}_2\text{O}$
 - > Calclacite ($\text{Ca}(\text{CH}_3\text{COO})\text{Cl} \cdot 5\text{H}_2\text{O}$)
 - > Thecotrichite ($\text{Ca}_3(\text{CH}_3\text{COO})_3\text{Cl}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$)
 - > Tennent’s Salt ($\text{Ca}(\text{CH}_3\text{COO})(\text{HCOO}) \cdot \text{H}_2\text{O}$)
 - > $\text{Ca}_2(\text{CH}_3\text{COO})(\text{HCOO})(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$
- occur as efflorescence phases

The crystal structures of all known efflorescence phases show striking similarities. Calcium is always coordinated by eight ligands, where four out of eight places in the coordination sphere are occupied by carboxylate anions, which leads to the formation of 1-dimensional calciumcarboxylate chains (Fig. 1, d, e). Hydrate water molecules, carboxylate or nitrate anions occupy the remaining places in the coordination sphere, with the latter often leading to a cross-linking of these chains. Additional hydrate water molecules or anions like chloride can be intercalated in-between the chains, as it can be found in the crystal structures of calclacite or thecotrichite. The calcium carboxylate chains are not always linear, they can also form collagen-like triple-helix motifs like in the structure of $\text{Ca}(\text{CH}_3\text{COO})_2 \cdot \frac{1}{2}\text{H}_2\text{O}$ (Fig. 1, f).

Crystal structure elucidation of the efflorescence phase does not only serve the fundamental scientific interest, it can also contribute to the preservation of cultural heritage. The crystal structure datasets are the basis for qualitative and quantitative phase analyses via X-ray powder diffraction. Moreover, only carboxylate anions are required for forming calcium carboxylate chains, which are present as the main motif in all crystal structures, whereas the presence of inorganic anions like chloride or nitrate does not appear to be required. Therefore, it becomes clear, why efflorescence crystals emerge on calcareous objects even after extensive desalination. Water molecules, which are intercalated in-between the chains show weak bonding to the crystals and can be released easily. Since the efflorescence phases exhibit a reversible de- and rehydration behavior, fluctuations in temperature or relative humidity induce shrinking and swelling of the crystals, which can lead to further damage to the cultural heritage objects.

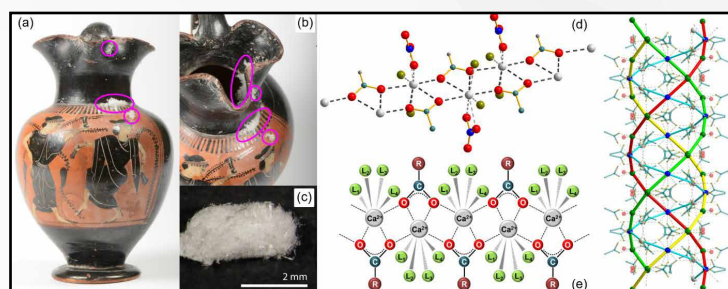


Illustration 1:

- (a, b) wine jug, ca. 500 BC, from the Prince of Canino collection in the National Museum in Leiden,
 (c) microscopic image of the efflorescence crystals,
 (d) calcium carboxylate chain in the crystal structure of the efflorescence phase $\text{Ca}_2(\text{CH}_3\text{COO})(\text{HCOO})(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$,
 (e) calcium carboxylate chain as characteristic main motif of the crystal structures of efflorescence phases on calcareous heritage objects
 (f) calcium carboxylate triple helix in the crystal structure of $\text{Ca}(\text{CH}_3\text{COO})_2 \cdot \frac{1}{2}\text{H}_2\text{O}$.



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WEIGHTLESSNESS AS AN IMPORTANT TOOL IN CRYSTAL GROWTH

Crystals or crystalline materials are essential and important in various fields of technological application due to their special physical properties. They are used in many fields like microelectronics for microchips or light emitting diodes, as well as materials for renewable energies like solar cells or thermoelectrics. The fabrication of these crystals happens mainly by so-called melt crystal growth. During such processes, convectional movements in the liquid and the related mass transport have a big impact on the quality of the growing crystals. A big part of these convectional movements is driven by density-dependent buoyancy flows. Other convection types have their origin at interfaces, like free melt surfaces. To understand the single phenomena and their interaction with each other, all of them have to be studied, qualified, and quantified separately. Afterwards, models of these convectional movements can be developed, which are used for the optimization of industrial growth processes, e.g. silicon single crystals.

Due to the omnipresent gravity with $1g$ (9.81 m/s^2), on earth no experiments without density-driven buoyancy convection can be conducted. The need to study other convections separately to understand the sum of phenomena completely is one reason to perform crystal growth experiments under weightlessness. Different platforms (drop tower, parabolic flights, sounding rockets, satellites, International Space Station) provide different durations of residual gravitational forces down to $10^{-6} g$ (so-called microgravity, μg), which allow experiments without convectional contribution under so-called diffusive conditions. These experiments deliver fundamental data, which enable an optimized description of theoretical models and adapt simulations to reality.

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CRYSTALS IN THE BODY...

HOW COME? WHY?

Living things take advantage of the properties of crystals, primarily to form their hard tissues such as teeth, bones, or shells. These hard tissues are composites of crystals of mineral matter and organic fibers of proteins (similar to those that make up silk) or polysaccharides, similar to chitin or cellulose. The mineral crystals contribute the hardness and resistance to compressive forces, the organic fibers provide the tensile strength and elasticity. The composite of both components compensates for the respective mechanical disadvantages of the individual components through a „hierarchical architecture“, i.e. there are many levels of structure formation, from the molecular chemical bond (scale 0.1 nm), through the nano scale (1 nm to 1 μ m), to fiber composite or laminate structures of the micro scale (1 μ m and up), to the macroscopic scale (mm to m). So there is e. g. a human tooth macroscopically from a hard, rather thin outer layer, the enamel, and the inner, softer dentin. Both layers consist of crystals of a mineral substance, which we call apatite, analogous to the macroscopic mineral with a similar composition. On the molecular scale, apatite is a compound with a defined structure of calcium and phosphate, which also contains ions such as carbonate and hydroxyl. The biological crystals reach dimensions of a few tens of nanometers and they are embedded in a polymeric matrix of fibrous protein molecules.

This compound forms threads with a diameter of approx. 100 nm, which are then, like the fibers in a thread or a rope, connected or twisted to form larger units. The organisms determine the final shape of the tooth or bone via the organic matrix and thus create crystal forms that are completely different from those in the inorganic world. In the tooth enamel, this matrix is gradually degraded during the mineralization of the tooth, so that in the end almost only the apatite nanocrystals fill the space. The material is similar to ceramic. Dentin and bone, on the other hand, must be flexible and elastic to a certain degree. A little less than half of their material consists of apatite nanocrystals and the rest consists of soft tissue and also blood vessels.

Besides these biologically formed materials with a mechanical function, there are also biologically formed crystals with a sensory function. In the balance organ in our inner ear, e.g., there are tiny crystals of calcium carbonate at the ends of fibrous extensions of sensory cells. The crystals function as tiny „weights“ whose inertia causes the fibers to bend, which the cells use to register acceleration when we move. Calcium carbonate mineral (calcite) eye lenses are found in starfish, isopods, and trilobites.

Some bacteria have an organ for determining the orientation of the field lines of the earth's magnetic field. The microbes can use it to determine where „up“ and „down“ is in a lake. The sensory organ consists of a chain of nanocrystals of the mineral magnetite (iron oxide with the formula Fe_3O_4) held together in a tubular organic membrane.

Conversely, there is also harmful, pathological crystallization in the body. Kidney and urinary stones or arteriosclerosis are common examples.

The current research questions on biologically formed crystals or „biominerals“ are diverse. Tooth preservation, dentures, and replacement of bone substance after accidents or interventions are an important topic, since the hierarchical, hybrid structure of the biominerals results in properties that cannot be satisfactorily imitated with synthetic materials up to now. „Biomimetics“ is the science and art of converting what we can learn from observing or researching nature into improved synthetic materials or „devices“. The fossil skeletons (bones, shells) of living beings not only give us information about the evolution of life and its „materials“ on earth, they are also the materially preserved information carriers about the environmental conditions in which this life existed, because the chemical and the mineralogical composition of the material indicates, for example, ambient temperatures and nutritional principles.

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CRYSTALS AND COMMUNICATION

After discovering the piezoelectric effect on tourmaline crystals in 1880 by the brothers Jacques and Pierre Curie, it was initially more of a curiosity. The exploitation of its technological potential only began decades later, mainly related to the development of electronics. The direct piezoelectric effect, i.e., the generation of electrical charges through mechanical action on crystals, is the basis of pressure, force, and acceleration sensors, which are, for example, part of the safety technology in automobiles and are also of central importance for the development of autonomous driving.

The inverse piezoelectric effect has proven to be even more fruitful. Alternating electrical fields allow for the excitation of mechanical vibrations in piezoelectric materials and vice versa. Elastic natural oscillations of crystals are highly stable in frequency and are practically not subject to ageing effects. Accordingly, quartz oscillators (**Figure**) form the beating heart of almost all microchips. Frequency filters, which use the propagation of surface acoustic waves between comb-like electrodes on the crystal surface, are also widespread in communications technology.

Since the elastic properties depend on external conditions such as pressure and temperature, changes in the natural vibrations of crystals can be used to monitor the corresponding process parameters. Piezoelectric crystals are particularly appealing because random mechanical vibrations stimulate natural oscillations. In the case of proper orientation, shape, and dimension of the piezoelectric crystal, radio signals are generated offering contactless read-out, analogous to RFID technology. Their use in engines and turbines appears promising to reduce fuel consumption and increase service life by optimizing the combustion process. However, the harsh conditions exposed to the crystals are a major challenge, with temperatures in excess of 1000 °C, high centrifugal forces, and a corrosive atmosphere. Crystallography plays a crucial role in searching for or designing suitable new piezoelectric materials.

The elasticity and piezoelectricity of a crystal depend directly on the arrangement of the atomic building blocks and their interactions in its crystal structure. The relevant electromechanical structure-property correlations can be examined, for example, with the help of diffraction methods and resonant ultrasound spectroscopy.



Figure:

Left: Oriented quartz plates are the heart of commercial high-precision oscillators.

Right: Encapsulated quartz oscillator (red arrow) as clock generator in a clockwork.

Photo: Museum Wiesbaden/Bernd Fickert.

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STRUCTURE-BASED DRUG DESIGN

At modern Synchrotron sites, it takes only minutes to collect diffraction data from crystals of macromolecules and determine their molecular structure. This led to the almost routine usage of macromolecular crystallography as a screening technique to determine starting points for drug design. As a start, libraries of low molecular weight compounds, called fragments (approx. 100–300 Dalton) are screened. Binding fragments identified from the crystallographic data of usually 100–500 samples reveal molecular details of interaction with the protein surface and serve as “foot in the door” for further improvement. To arrive at a molecule with drug-like properties and biological effectiveness, numerous rounds of optimization are necessary. While later stages always involve dedicated custom medicinal chemistry, the first steps from a fragment to a better binder can be achieved using catalogues of already available molecules (Fig. 1.). Such optimizations can already result in improvement of the molecule’s binding affinity by one to three orders of magnitude.

While structure-based design of nearly optimal molecules is well established, crystallographic fragment screening is a relatively new development of the last decade that is currently transforming the pharmaceutical industry, as such workflows increase the speed of early-stage drug discovery projects. Future synchrotron capabilities for macromolecular crystallography with even higher throughput and automation will render fragment screening of 1000 conditions an every-day experiment and enable academic groups to kickstart drug discovery on neglected biological targets, i.e. for development of urgently needed new antibiotics.

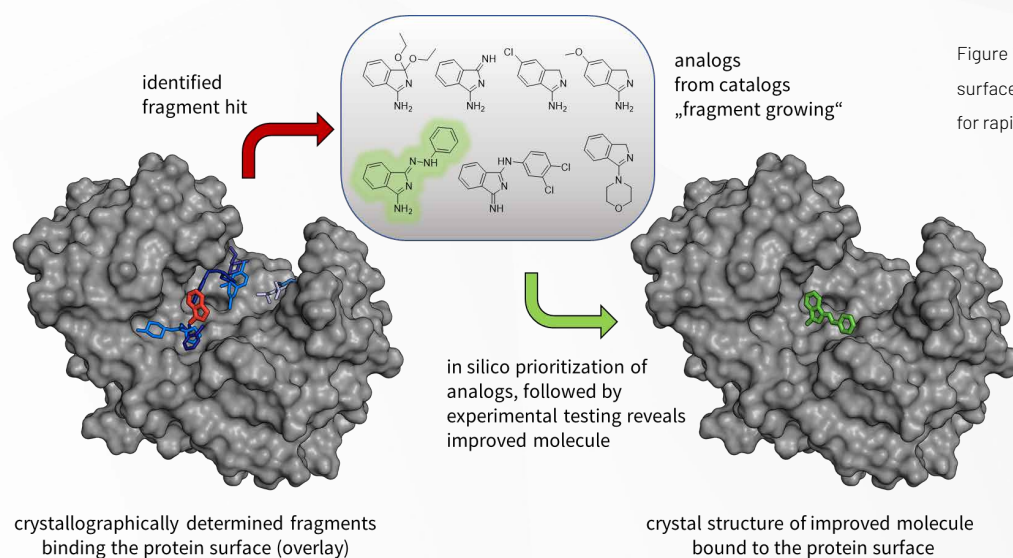


Figure 1 – Example of how fragments identified on protein surfaces by crystallography can be used as starting points for rapid structure-based molecule improvement.



Anna-Lena Hansen

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BATTERIES – MORE THAN JUST MOVING ELECTRONS



It is not just since John B. Goodenough, M. Stanley Whittingham and Akira Yoshino were awarded the Nobel Prize in Chemistry in 2019 that batteries, more precisely accumulators, have become an integral part of our lives. Batteries are so easy to handle and so indispensable to our daily life, but the processes that take place inside a battery can be just as complex. This is especially true when we take a look into the sustainable battery of the future. Because the requirements for such a battery are almost endless.

They should be efficient, charge extremely quickly, be light, safe, but also economically friendly and of course cheap. Crystallography helps us to approach these goals. Because batteries contain a whole zoo of crystalline and non-crystalline materials that enter into an exciting interplay.

For example, it is important to understand the stability of novel anode and cathode materials during battery cycling – which phases are formed? Does it make a difference whether the battery is completely discharged or in which voltage range it is used?

How can the ionic conductivity of solid electrolytes be optimized, and how can one ensure that no undesirable side reactions occur? The so-called real structure of the materials is also receiving more and more attention. All, including non-periodic, features of the structure should be recorded so that the influence of, for example, local lattice distortions or chemical segregation on the physical properties can be understood. All of this can be clarified using crystallography before you can hold a working battery in your hands.

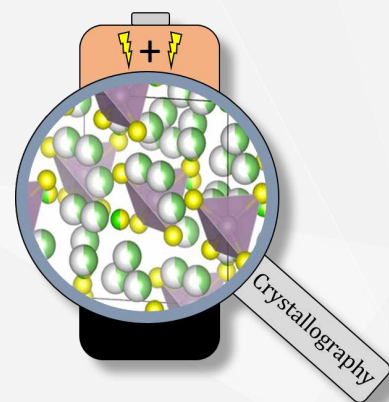


Figure: Crystallographic methods enable us to look insight into structural changes in a battery.

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POINT DEFECTS IN COMPOUND SEMICONDUCTORS: CURSE OR BLESSING FOR THE SOLAR CELL?

Problems concerning environmental and energy policy belong to the greatest societal challenges these days. Within the renewable energies photovoltaics (PV), the direct conversion of sunlight into electrical energy within a solar cell, plays a key role.

The most commonly known solar cell configuration is a p-n junction, realized by a p-type and an n-type semiconductor. The PV market is dominated by solar modules based on silicon, which has to be doped by boron or phosphorous to become p-or n-type. Ternary and quaternary compound semiconductors, used as absorber materials in thin film solar cells, the most promising and cost-efficient PV technology, contain intrinsic (or native) defects which cause the type of conduction (p or n). There are "good" and "bad" structural point defects in the material, the first are useful for device operation the second can make the device useless. Thus, defects have become a key ingredient in solar cell device technology.

The Chalcopyrites $\text{Cu}(\text{In,Ga})(\text{Se,S})_2$ (CIGSe) are the most advanced and most efficient absorber materials in thin film solar cells, presenting record lab efficiencies of >22%.

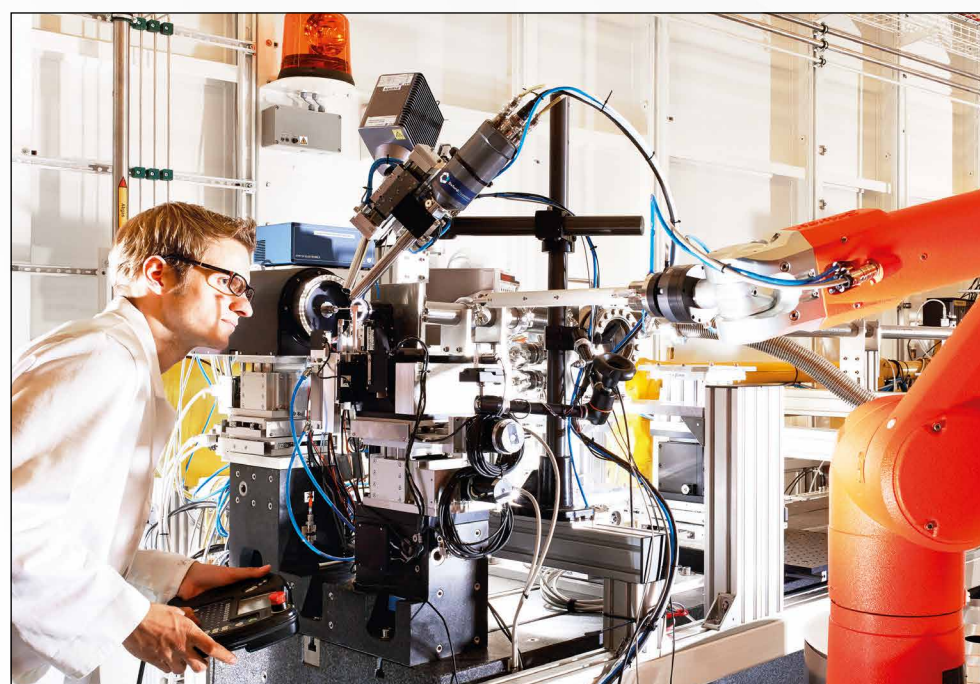
Since the availability of indium is an object of concern regarding the large scale production of solar modules, its replacement with Zn and Sn is beneficial in this sense. Solar cells based on Kesterite type $\text{Cu}_2\text{ZnSn}(\text{S,Se})_4$ (CZTS, CZTSe) are considered as the only critical raw material free PV technology. In high efficient record devices these materials generally show a non-stoichiometric composition which is possible due to a remarkable flexibility of its crystal structure. This ability to accept deviations from stoichiometry is correlated to the formation of intrinsic point defects like vacancies, anti sites or interstitials. Copper vacancies cause the p-type conductivity, acting as "good" defect. Anti sites connected with tin cause charge carrier recombination, thus acting as "bad" defect.

With the use of X-ray and neutron diffraction, it was possible to determine the defect type and the concentration of point defects in chalcogenide compound semiconductors of the Chalcopyrite type and Kesterite type and to correlate them with the synthesis conditions of the material. This laid the foundation for successful defect engineering for a further increase in solar cell efficiency.

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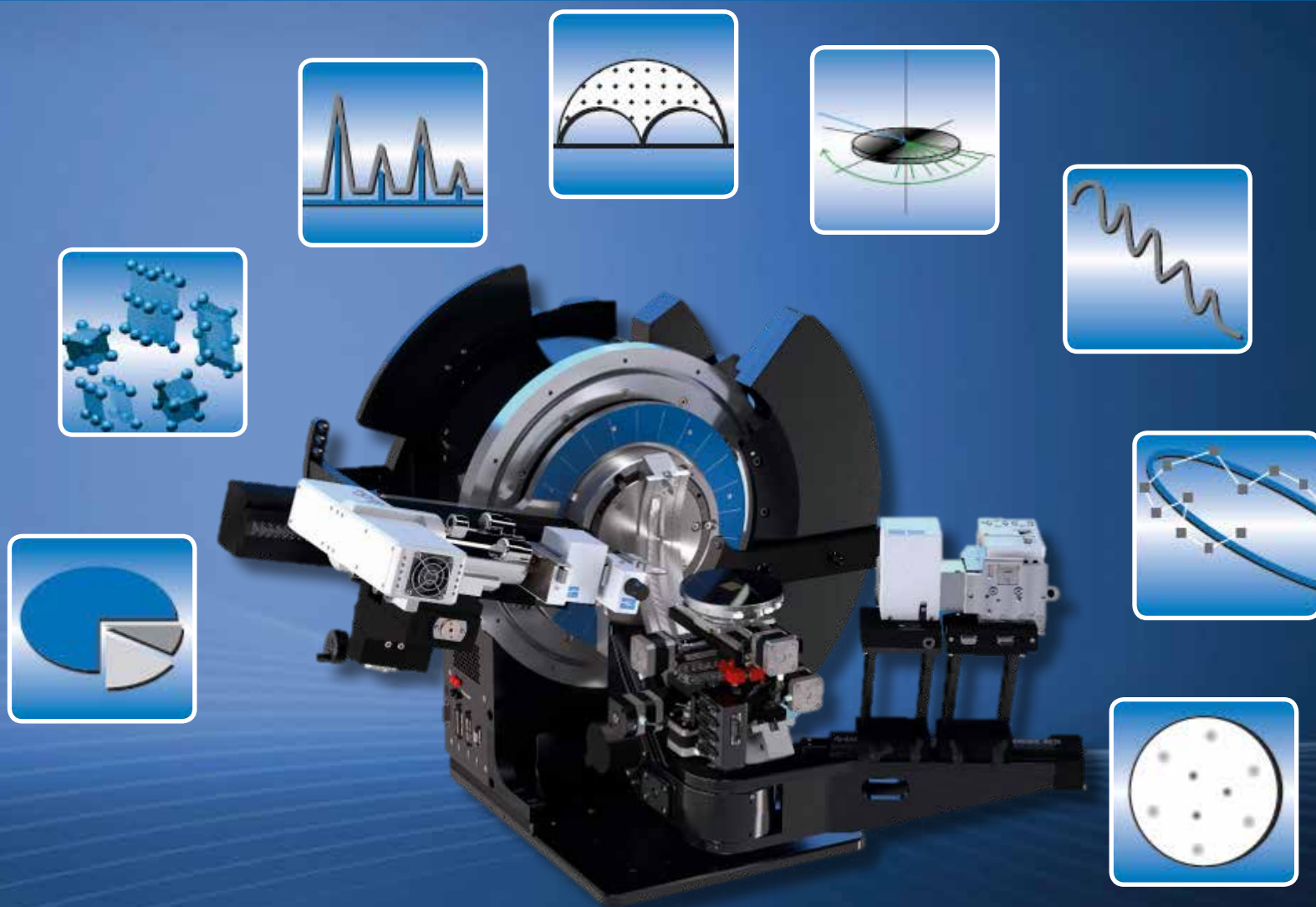


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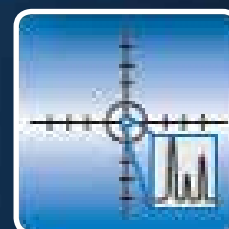
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