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Structure analysis of new mineral species: current state and perspectives

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Abstract. A brief overview of X-ray diffraction techniques currently used for the structural investigation of new minerals is provided, focusing mostly on X-ray diffraction from single crystals, which remains the most widely used method, and on the emerging field of electron diffraction methods that are expected to overtake, to some extent, the role of X-ray diffraction methods.

Keywords: new minerals; crystal structures; structure determination; X-ray diffraction; electron diffraction; symmetry; nomenclature.

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Структурный анализ новых минеральных видов: современное состояние и перспективы

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Аннотация. В работе представлен краткий обзор методов рентгеновской дифракции, используемых в настоящее время для структурного исследования новых минералов, в основном, с акцентом на монокристальную рентгеновскую дифрактометрию, которая остается наиболее широко используемым методом, и на развивающиеся методы электронной дифракции, которые в некоторой степени вытесняют методы рентгеновской дифракции.

Ключевые слова: новые минералы, кристаллические структуры, определение структуры, рентгеновская дифракция, электронная дифракция, симметрия, номенклатура.

Благодарности. Выражаю благодарность 1) моим наставникам, без которых я бы не так заинтересовался кристаллическими структурами минералов: Иване Цисаровой (Карлов университет, г. Прага, Чехия), благодаря которой я получил первые представления о кристаллических структурах в магистратуре, покойному Иржи Чейке

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INTRODUCTION

In the past, the crystal structures of new minerals were only barely determined, or more specifically, determinations were limited only to “reasonably large” crystals that could be used for single-crystal X-ray diffraction studies. “Reasonably large” crystals are somewhat of an arbitrary term. Methods used in modern crystallography also employ reasonably large crystals. The reasonability correlates with the size of the crystal, with the characteristics of the incident beam used for diffraction (such as brilliance, flux, and diameter), and the properties of the detector used. Fifty years ago, with proportional or scintillation counters (point detectors) and a 1 mm-diameter beam from conventional X-ray tubes, the use of crystals, which can be called giants, compared to those used nowadays, was possible. And of course, the major factor is the experimenter’s impatience. This is, of course, much higher today than before. In the past, the calculation of the structure factors required ages, making the experimenter comfortable with waiting and humble enough to wait. Old times are gone, anyway, and since the early 2000s, when CCD detectors have massively outpaced CCDs in experimental devices (and the computational power of personal computers has advanced significantly), structure analysis has seen significant development. The mineral sciences (usually considered a Cinderella of Science) witnessed similar development. I refer to Burns (1998), which documents the coming revolutionary era. Today, the submission to the Commission of the New Minerals, Names and Nomenclature of the International Mineralogical Association, lacking the structure determined, has only a slim chance of success. In the following brief contribution, I will focus on the most recent developments in diffraction techniques for describing new minerals.

X-RAY DIFFRACTION METHODS

Single-crystal X-ray diffraction

Single-crystal X-ray diffraction has played a prominent role in structure determination in the past and remains the most widely used technique today (for fundamentals of crystallography, I refer to the book by Giacovazzo et al. 2011), even though numerous structure determinations are also carried out using electron-diffraction techniques. The structure determination benefits nowadays from the availability of extremely powerful in-lab sources that enable scientists to work with crystals as small as a few microns (~10–8 microns). The production of X-ray tubes by Incoatec (used in Bruker diffractometers) and the rotation anodes by Rigaku-Oxford Diffraction both expanded the possibilities. Each of those two types of sources (generalized) has advantages and disadvantages that will not be reviewed here; nonetheless, both have significant cons. And this is the fact that to obtain perfect diffraction data from a tiny microcrystal, you do not need to apply for the synchrotron beam time; plus one great advantage – in the case of the in-lab diffractometer, you are not usually limited by the queue of impatient users knocking at the beamline doors, but only your impatient colleagues, with whom you usually find a way how to negotiate about the beam-time schedule. The limits of what is nowadays possible can be shortened to the following: **1)** the ability of the crystal to diffract, and **2)** the ability of the experimenter to extract a small crystal. Ad 1) stemming from the size of the crystal and nature of the crystal – its diffraction ability, manifested by F_{000} *vs.* absorption and other factors – presence of stacking faults, defects, poor crystallinity, or general instability of the compound at given conditions.

In general, when there is enough material and the crystal diffracts well, data collection is reduced to tens of minutes or a few hours to obtain high-quality datasets (in terms of redundancy and statistics, as re-

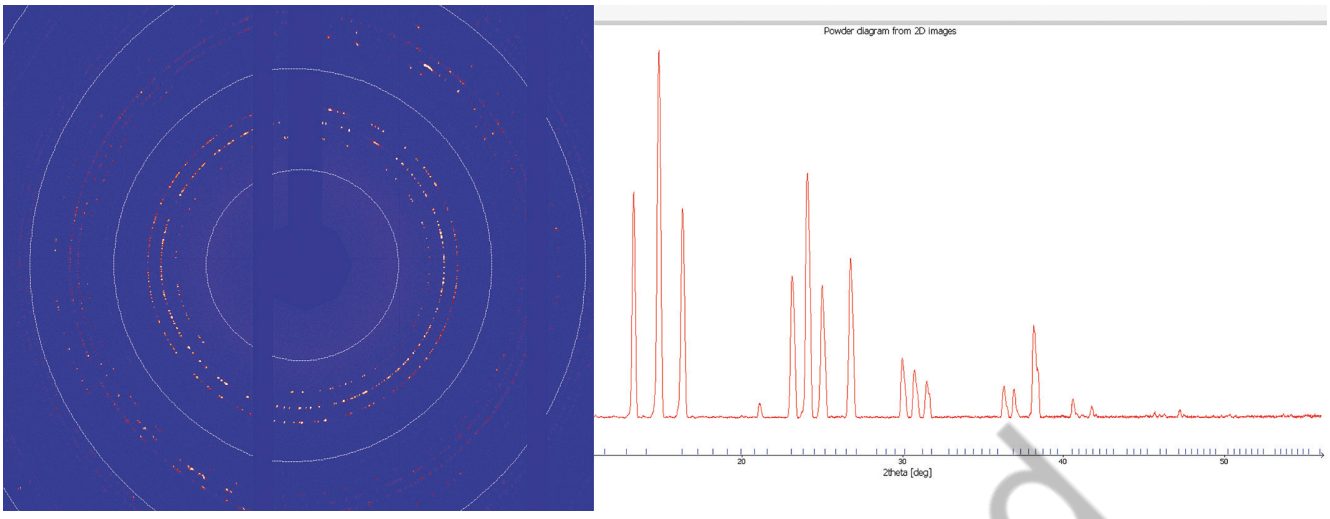

Fig. 1. Powder-like diffraction pattern (left) obtained by pseudo-Gandolfi movement of the kappa-goniometer from the 0.085 × 0.048 × 0.028 mm large crystal of theuerdankite (Plášil et al. 2024) and powder profile extracted from the data by routine in CrysAlis software. The average FWHM is ~0.11° 2θ. The diffractometer resolution was tuned by setting the primary optics (refining the beam divergence) to 0.3 mRad.

flected by [$I/\sigma(I)$]), namely when a new generation of X-ray sources is used. Nevertheless, the actual size of new minerals decreases over time, relatively to those in the past. We are mostly working with smaller amounts of material as most new minerals occurring in large crystals have already been discovered (with exceptions, such as saltonseaite; Kampf et al. 2012).

Powder diffraction from single crystals: pseudo-Gandolfi method on four-circle diffractometers

The great advantage of nowadays laboratory high-flux sources and large sensitive area detectors is the possibility to obtain powder diffraction data from (the only) single-crystal (you may have), from which you obtained 3-D intensity data for the structure determination. At least, I have good experience with both the "old" Rigaku R-speed Axis, using an imaging plate detector, and the newer Rigaku/Oxford Diffraction devices, such as the SuperNova or Synergy XtLab diffractometers, both operating under the CrysAlis software (Rigaku). The acquisition of powder data via pseudo-Gandolfi movement is quick and reliable; the system offers several distinct algorithms for sampling (extraction) and correcting the 2D data to make a 1D powder profile (Figure 1). I refer to it as "pseudo" since the original Gandolfi camera allowed for even greater coverage; to some extent, it can be nearly achieved using modern analysis by involving both positive and negative θ positions and opposite κ settings.

Moreover, one can obtain powder data using pseudo-Gandolfi movements, even with non-ideal geometry. That means using irregular crystalline aggregates (of minute size) instead of loading the powder into a capillary. It must be emphasized that data quality is limited compared to conventional or dedicated powder diffractometers with focusing geometry (using curved Göbel mirrors and narrow Soller slits); the data is inferior. Nevertheless, with careful handling and an understanding of limits, some valuable results can be achieved (see, for instance, Kasatkin et al., 2023), particularly in situations where material is scarce for other methods or instrumentation.

Electron diffraction: an old method in a new coat

Electron diffraction has undergone a transition from the sparse niche-method to a "user-friendly and widely used" method (Palatinus et al. 2017). This has been enabled once due to the development of both instrumental and theoretical backgrounds. The evolution of the electron diffraction methods, including precession electron diffraction, PED (Palatinus et al. 2015), 3D electron diffraction, 3D ED (Gemmi and Lanza 2019), and the possibility to refine the structures from the electron data using dynamic theory of diffraction (Palatinus et al. 2015 a, b, 2019), all those made dreams come true for many crystallographers around the world. Along with the automation of the data-collection routines and user-friendly interfaces, such as the XtalLAB Synergy-ED electron diffractometer from Rigaku, the

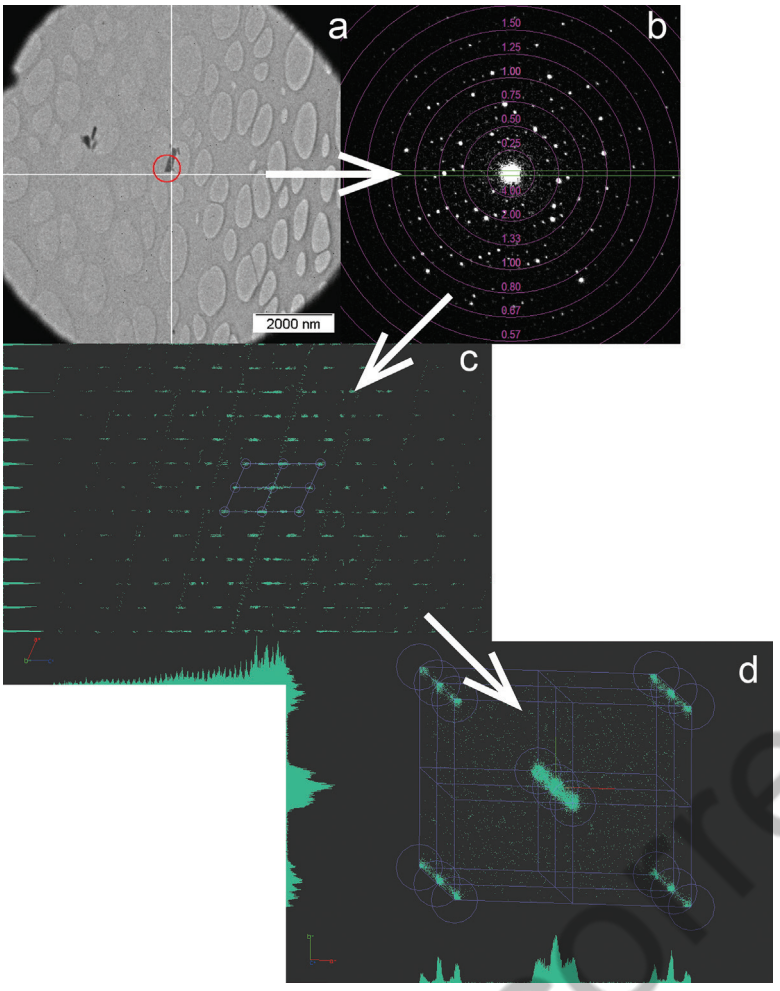

Fig. 2. Diffraction data from 3D electron diffraction and the workflow (arrows): a – the lath-like arsenuranylite crystal used for data collection on a carbon grid; b – the typical "single-crystal" frame obtained from that crystal; c – the indexing routine in PETS 2.0 software (showing the outline of the unit-cell and all the reflections, including those unindexed); d – the somewhat inclined projection of the reflections with the arsenuranylite unit cell (4 × 4).

electron diffraction will become an even more accessible and widely used technique. Nowadays, the determination of unit-cell parameters benefits from applicable corrections, thereby improving their accuracy (Brázda et al., 2022). Nonetheless, we still should be aware of several disadvantages or limitations of the method: 1) measurements are done in a high vacuum; 2) the data are usually collected at 100K; 3) the *R*-indices, or general, the statistical agreement factors of the refinements (for most structures) are comparable to those obtained, e.g., from X-ray diffraction, but still are higher namely for the crystals of the large mosaicity, i.e. far-from-perfect crystals (and it does not mean that the structure models obtained from them are incorrect). However, despite the challenges posed by beam-sensitive materials or highly hydrated compounds, such studies can be conducted (e.g., Banihashemi et al. 2020; Steciuk et al. 2021; Gurung et al. 2025) (Figure 2). The limitation of the 100K lies mostly in a possible phase transition that the studied material can undergo. The main benefit of the technique is the ability to obtain accurate structural parameters or the entire crystal structure from a material/mineral of minute size, otherwise inaccessible by other techniques. When employing SEM-FIB devices as well, the lower limit is about 100 nm to micrometer-sized lamellae (e.g., Mikuš et al. 2023; Majzlan et al. 2024). I, in person, can speak to a true jump in the method's accessibility, when comparing the suite of experimental and computational techniques in statu nascendi during the structure study of uranyl carbonate mineral widenmannite (Plášil et al., 2014), one of the first complex mineral structures determined using PED, and the methods used nowadays (e.g., Plášil et al., 2025a, b). Personally, I expect that the number of new minerals whose structures will be determined by electron diffraction methods will increase steadily.

FUTURE… (REMAINS UNWRITTEN)

As nobody, forty, but maybe even ten years ago, had any idea of the current state and development of computational power and experimental possibilities, we cannot predict how science, mineralogy, or miner-

alogical crystallography will develop. It is quite normal to consider the current level of development the highest achievable, but this is simply a matter of our position in time and space.

I wish mineralogical crystallography a long, prosperous life, of course. However, I am realistic and, with a bit of sadness, witness a slight decrease in the interest of decision-makers responsible for education and research management, and, consequently, a corresponding disrespect for this traditional and still valuable solid-state science, at least, in Europe. Nevertheless, what makes every discipline forth is being less formalistic and more provocative. Mineralogical crystallography and structure analysis can be like that. We should focus our efforts on difficult topics, and we should not waste time on solving every structure of a new mineral, just because it is a new mineral (and the structure is, for instance, known, at least for the synthetic counterpart, or apparently related phase). The theoretical background and fundamentals of chemistry and physics describing chemical bonds are well understood and accessible: see, for instance, a great recent paper by Hawthorne (2026). Modern structure analysis offers us extremely powerful methods and tools that a handy scientist can turn into a highway to success (heaven).

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