Poster

Single-crystal XRD: Black box vs experienced crystallographer

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The role of the single-crystal X-ray analysis (SC-XRD) in chemistry from 1990's as a '*fast, first resort analytical tool*' [1] is even more relevant nowadays, as the structural characterization of novel compounds becomes a pre-requisite for publication in the high-level chemical journals. On the one hand, fast automatic diffractometers and user-friendly software makes ground to a ' *black box*' strategy of SC-XRD. In the ideal case, researchers virtually without any knowledge in crystallography using fully automatic software expected to be able to solve most of the crystal structures. On the other hand, 'difficult cases' can be found even along so-called 'service XRD' directed to routine structure determination. For example, during last two years 270 crystal structures of boron- and silicon-containing organic compounds were solved for the group of Prof. M. Wagner in our university. Among them, 28 showed pseudo-merohedry twinning, and we found two cases of translational pseudo-symmetry, three examples of T-driven phase transitions, two structures revealed order-disorder (OD) effect and one compound was incommensurate.

Summarizing our dozens years of experience in SC-XRD, we would like to present here our ideas about a hypothetic 'perfect' black box software (BBS). that will take both *macro* factors, the non-coherent scattering from multiple domains (twinning, OD-effect), as well as *micro*-factors within the coherent dynamic scattering model (modulation, pseudo-symmetry, low crystallinity, disorder). To be on safe side, BBS better start not from the unit cell + F_{hkl} set, but rather from the primary 3D diffraction data (set of frames from a diffractometer). In this way, e.g., a tricky twinning would be interpreted before it causes an illusion of halving of c^* axis (Fig. 1a), or a translational pseudo-symmetry would be detected resulting in a correct 'halved' a^* axis (Fig. 1b). The next task for BBS is the space group determination ensuring the '*the highest symmetry causing the lowest disorder*' principle. Only after the correct symmetry is established (*including* the translational one), BBS can start the structure solution followed by assigning of the electron density peaks to the atomic sorts. It might be very challenging, as for example in the carbon-boron case. At this stage, some kind of AI approaches based on knowledge databases might be helpful. Then, the disorder can cause a lot of troubles for BBS, especially when it includes many or *all* atoms (Fig. 1c). Note that it can bring chemically essential information [2, 3]. Sometimes it is caused by the OD effect originated from stacking faults that results in appearance of 'ghost atoms' as the low-occupied Ru atoms (Fig. 1d).
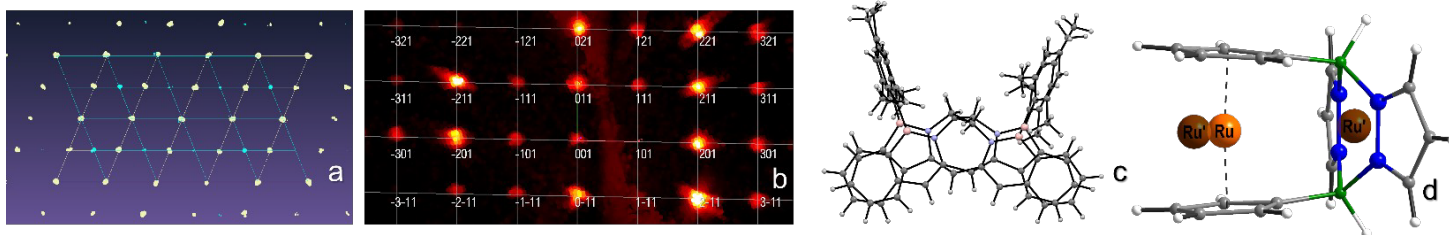

Figure 1. Examples of twinning (a), pseudo 'a/2' symmetry (b), full disorder (c) and manifestation of the OD-effect (d).

At the final stage, intellectual algorithms must be developed for checking the results of BBS to ensure that no false crystal structures will come out, even if an experienced crystallographer for some reasons is not available to evaluate the results.

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