X-RAY DIFFRACTION

DIFFRAC.EVA V6

New and Enhanced Features Explained

Version 6 of DIFFRAC.EVA makes the analysis of diffraction data faster, easier and more reliable than ever before. The current release does not only come with a fresh new design but brings a multitude of new and improved functionality, together with massive performance increases.

The native 64-bit architecture uses parallelized code for numerically demanding tasks. This exploits the full power of modern multi-core computers. Demanding visualizations that previously limited Cluster Analysis to a few hundred measurements, now benefit from new hardware accelerated graphics. That way the capability of EVA is extended to the simultaneous treatment of thousands of scans and the presentation of the results on high DPI graphics monitors.

New hardware that is supported by DIFFRAC.EVA covers the two-dimensional EIGER R 250K detector and the BioLogic potentiostat that is commonly used in the research of battery materials.

Ease-of-use is the key to mastering complex software. Different applications in EVA may benefit from individual screen layouts. This considers the user's personal habit to work in EVA, but also matches hardware constraints on monitor size, or the need for specific command or property panels that may differ among various analysis. Layouts may be saved as part of the EVA project. Upon re-opening the EVA document, the layout connected to the EVA file, is applied rather than the last active one.

Main features

- Heuristic auto species-identification
- TOPAS connectivity
- COD database maintenance
- Extended full-pattern matching
- Interoperability between EVA, other software and methods

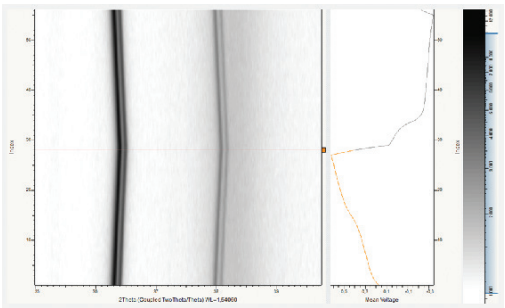

Fig. 1 Peak shift during charge and discharge cycles of in-operando battery work.

Phase-ID

Heuristic auto species-identification

In general heuristics combine practical problem-solving steps that are sufficiently useful for solving a complex task. In the context of species (or phase) identification from XRD, we revisited and brought together tools that were in the past already well accepted by our users of manual Search/ Match. Those tools include automated background subtraction and peak search, iterative phase search with residuals determination, signal to noise evaluations, and the use of match peaks in combination with smart search-database filters, element filters and match lists. All this, together with the new optional auto sample-displacement detection and correction, now allows to routinely apply auto phase-ID to moderately complex mixtures of 10 phases or even more, and extends over major, minor and trace phases.

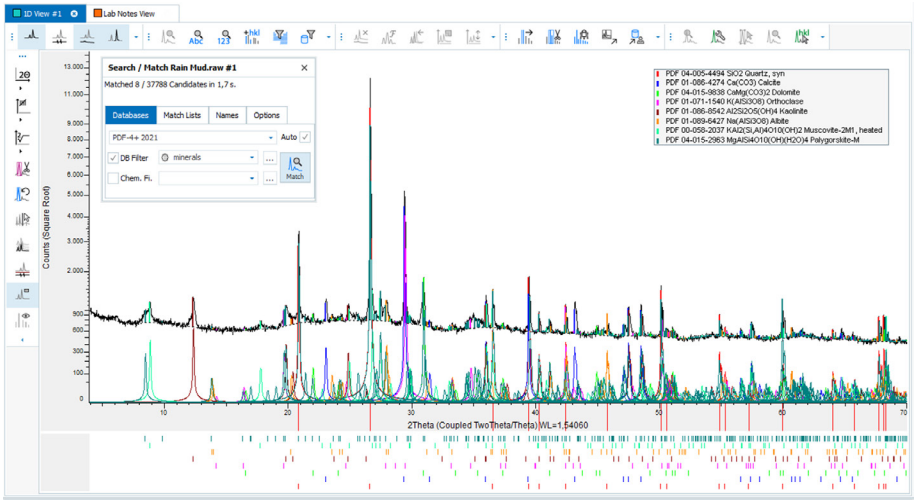

Fig. 2 Auto searching the ICDD PDF4+ database for mineral phases on a sample of dust sediments from the Sahara, collected in Crete. The eight phases found match with earlier results by Christidis et al. (2010) BGSG, 43(5), 2570-6

TOPAS

TOPAS Connectivity

This feature for the first time combines the results of an EVA species identification with the instrumental measurement settings (stored in the measurement data) that are needed in DIFFRAC.TOPAS to perform Rietveld analysis based on fundamental-parameters peak-profile calculation. Users no longer need to tediously search device data and copy information.

EVA v6 creates the needed TOPAS input including

- Scan data
- Instrument parameters
- Phase lists and atomic coordinates based on previous searches of COD or ICDD and user databases. Occupancies of special atomic positions are automatically detected and corrected.

COD

COD database maintenance

The Crystallographic Open Datafile (COD) is a rapidly growing non-profit project that meanwhile hosts about half a million crystal structures. DIFFRAC.EVA provides access to COD data for phase ID and Rietveld based TOPAS quantitative analysis. Using an up-to-date database is a crucial requirement to those working in the field of species identification.

Only the EVA software, for the first time, enables users to maintain an up-to-data search database themselves, according to their own need for actuality and update pace. DIFFRAC.EVA provides:

- Initial database compilations of search tables and crystal structure data for minerals or the full set of structures
- Database compiler with update capability using the internet
- Duplicates detection
- Filtering and subfile creation for minerals, non-ambient data, inorganic and organic compounds
- Data mining tools that screen the database for specific compounds and access the respective structural information to provide EVA and TOPAS input.

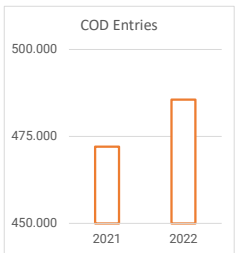

Fig. 3 From 2021 to 2022 the number of COD entries grew by 3%. This corresponds to about 13.000 structures.

Pattern Matching

Cluster Analysis

For cluster analysis general numeric data and correlation matrices complete the types of data that can be evaluated in EVA. Not only several numeric data sets, that even may have different number of columns, can be combined but also up to four sets of numeric, spectroscopic and XRD scan data can be evaluated together.

Also, with v6 comes the option, to seamlessly switch between big data in cluster analysis and detailed evaluation of selected individual candidates in EVA. The hardware accelerated dendrogram now allows to efficiently cluster thousands of scans. A script is provided for exporting all scans sorted by cluster membership

SQUALL

Additional preprocessing steps enhance the Semi Quantitative Analysis for all Materials (SQUALL). Added stability and improved convergence are achieved by Weighted Ridge Regression. Pattern-offset determination now may use derivatives for increased accuracy. In addition to the previously available RIR and MAC methods, a new quantification model based on chemical composition was added: the Direct Derivation Method (DDM).

The major user-benefits of DDM are:

- No crystal structure information is required,
- It works with crystalline and amorphous materials,
- It is a full pattern integration method, and therefore has a lower sensitivity to preferred orientation and even allows quantification of highly textured material.

PMI

A method new to XRD is Positive Materials Identification (PMI). It provides correlation analysis of materials (that can be pure compounds or mixtures of a given composition) and references. This is a quick classification method if there is no need to identify individual constituents of a mixture. Scans of unknown samples are matched against reference scans. The status "Identified" is based on the combined correlation and a user defined threshold value.

On-line EVA

On-line data evaluation in database mode allows to run SQUALL and PMI as automated push-button methods from starting the measurement, through the data evaluation, to the results presentation in the Results Manager.

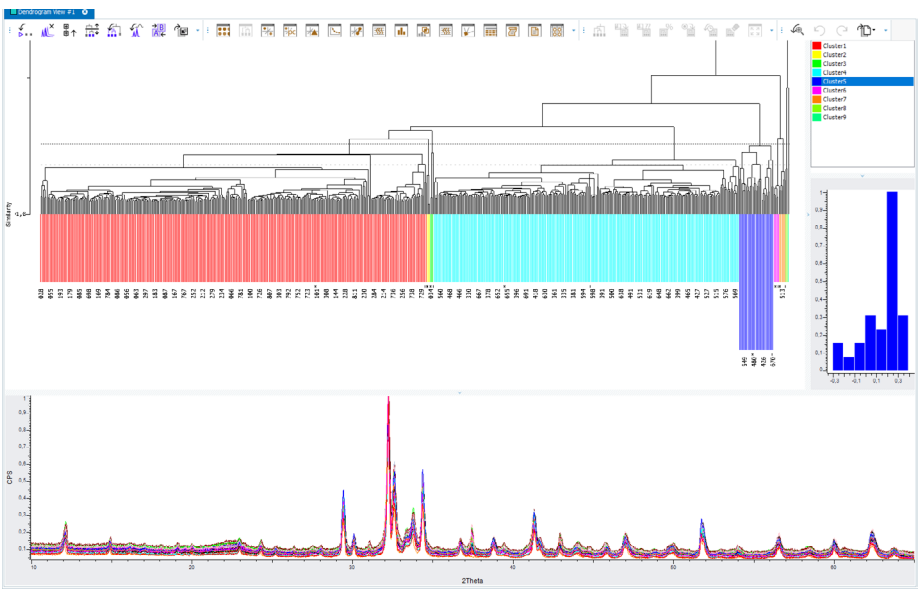

Fig. 4 Cluster analysis of a combined set of cement data consisting of 630 XRD scans and the same set of numeric data from DIFFRAC.TOPAS Rietveld analysis. A zoomed dendrogram view of the scan data is shown with one cluser selected for further evaluation.

Integration

Interoperability between EVA, other software and methods

The Results Manager of the DIFFRAC.SUITE database serves as a central hub for working with XRD data. The Results Manager creates and hosts filters and queries for measurement data that are stored in the instrument database. Scans can be opened directly with the respective evaluation software, and the analytical results are returned to the database. That allows to conveniently compare properties of multiple samples and to compile respective charts and tables for further reporting.

The following EVA results are stored to the database:

- Scan related properties: crystallinity, background, global fit curves (sum, difference)
- All area properties including the new signal/noise and peak/background evaluations
- Peak profile properties (intensity, shape, position, fit curves)
- Phase properties (crystallite size, sticks patterns, fit curves, search database meta-information, lattice parameters, space group, user color)
- Matching reference scans from on-line SQUALL and PMI evaluations, wt-% of phases and correlation coefficients

In times of constantly growing information, the visualization of data is of particular importance. The new EVA 6 offers color schemes for the consistent presentation of phase information across software packages (DIFFRAC.SUITE EVA, TOPAS, RESULTS MANAGER) and beyond.

To ease the reconciliation of XRD bulk phase results with e.g. surface analytical techniques such as mapping micro X-ray fluorescence or scanning electron microscopy EVA 6 provides predefined color schemes to work with Bruker AMICS or other automated minerals analyzers. Users may add their own color schemes that adapt to standards and commons of other industries and technologies.

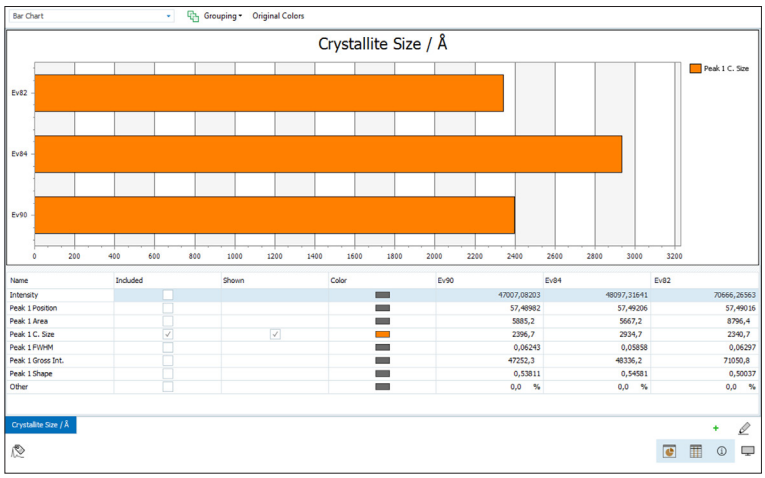

Fig. 5 Multi-sample reporting of peak properties in the DIFFRAC.SUITE RESULTS MANAGER. Area analysis was performed in DIFFRAC.EVA reading scans in database mode and the results were returned to the database.

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