Advanced Materials Analyses Using the Powder Diffraction File

T. G. Fawcett, F. Needham, C. Crowder, S. Kabekkodu
International Centre for Diffraction Data
Newtown, Square, Pennsylvannia, USA

Abstract: The Powder Diffraction File (PDF) in Release 2009 contains 674,139 entries in two major databases. Release 2009 PDF-4+ contains predominately inorganic materials and has 291,440 material data sets and PDF-4/Organics contains organic and pharmaceutical materials and has 370,844 material data sets. These databases grow every year as nearly 50,000 material analyses are currently being edited and reviewed from global sources on an annual basis. This provides a constant source of new high quality data. The higher quality is a result of recent advances in instrumentation and data analysis software, applied prior to publication, as well as a vigorous editing and statistical review of compiled data after publication, and before inclusion in the PDF. These developments means that materials can be identified, characterized and analyzed with an array of new data mining tools backed by an extensive classified and standardized database. The database itself can be data-mined to provide insights into the materials state of matter, crystallinity, solid solution behavior, polymorph composition and multi-phase composition.

Introduction: The Powder Diffraction File is the collaborative effort of several international database organizations. The collaboration results in comprehensive material databases that are used to identify and quantitate unknown solid state materials. A major and continuous effort of the ICDD, ICDD members and ICDD grantees has been to edit, classify and standardize all the data from international sources. This enables the database to be analyzed as an interrelated collection of materials enabling data mining and many different types of analyses. This provides global researchers with the capability to analyze all the significant data present in a diffraction pattern such as phase identification, purity, structure, quantitative content, crystallite size and crystallinity.

Experimental: All the data shown in this presentation were made with the PDF databases and the embedded software contained in the PDF-4 product line. Similar data, but with a smaller interface can be produced with PDF-2 products in combination with the viewing software DDView. The data content and the number of search and display options are shown in Table 1.

Release	2005	2006	2007	2008	2009
PDF-4					
Searches	38	44	48	48	53
Display Fields	48	65	66	70	85
Entries:					
PDF-4+	240,050	254,873	272,232	285,402	291,440
PDF-4/Minerals	17,826	19,254	25,861	29,607	34,212
PDF-4/Organics	265,208	286,464	312,355	341,540	370,844
PDF-2					
Searches	33	39	42	46	49
Display Fields	8	8	8	8	24
Entries	174,699	186,107	199,574	211,107	218,610

Table 1. Search and display options for PDF products during the last 5 years.

Results and Discussion: Through continued bibliographic efforts and a series of strategic collaborations the Powder Diffraction file has dramatically increased in size (Figure 1) over the last decade. This results in changes to the basic nature of material identification as it was practiced from 1940-1990 in that users now have a powerful library of material references and they will always get a candidate match using modern unknown identification software. The challenge for global researcher today is to have sufficient knowledge and analysis tools so

that they can choose the best and appropriate solution from among many candidates.

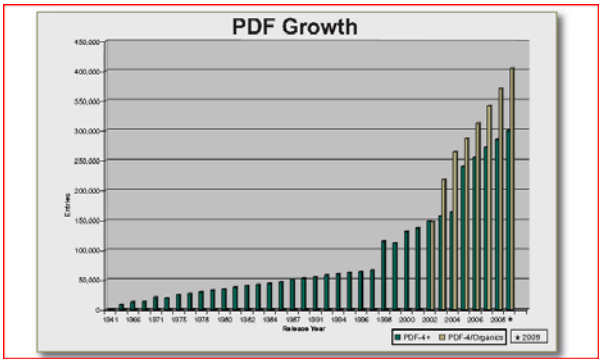

Figure 1. Growth of the Powder Diffraction File

ICDD in cooperation with worldwide software developers have produced a series of tools for data analysis. The ICDD has converted all data in its material data sets to a relational database with JAVA interfaces. The material data sets contain diffraction patterns, chemical nomenclature, structure classifications, crystallographic data, physical properties and bibliographic information which now can be searched, sorted, graphed and plotted in nearly endless combination. An example of a material entry in PDF-4+ is shown in Figure 2 additional analysis tools have been developed so that the basic data can be shown as neutron, synchrotron or electron diffraction, spot patterns or ring patterns as well as electron diffraction backscatter patterns. For 125,319 entries in PDF-4+ there is also a capability for molecular drawing, visualization and calculations for bond angles, distances and nearest neighbor atoms.

The standardization and classification editorial processes are also powering tools being developed by OEM's and software developers such as Rietveld and LeBail refinements, pattern fitting, and cluster analysis tools.
Today, very few Chinese laboratories are using these enhanced capabilities of the PDF-4 product line which are seeing widespread usage in North America and Europe. However, Chinese scientists are major contributors to these product developments both from data submitted by grantees and published data extracted and edited from the global literature including Chinese scientific journals.

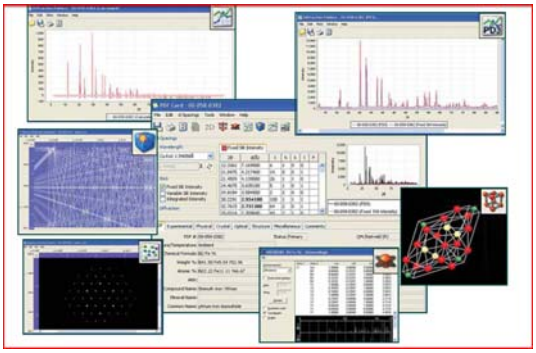
Figure 2. Example of a material dataset for entry PDF 00-058-0382. There are several other functions and nested display data not shown.

PDF products now include the contributions of thousands of Chinese scientists. These contributions include key reference data on energy materials, electronic materials, minerals, and both natural and synthetic pharmaceuticals.

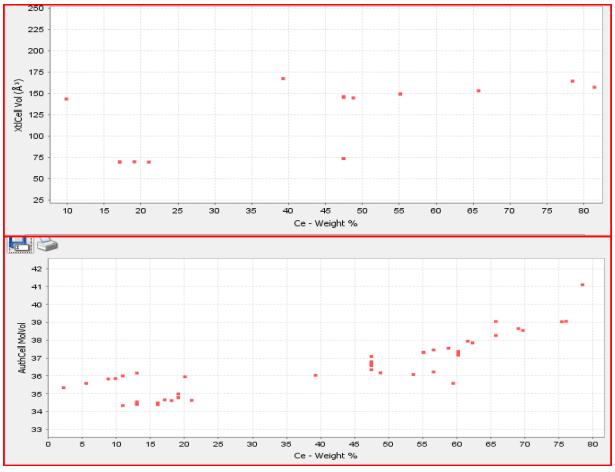

Figure 3. Unit cell volume versus Cerium weight % for Ce doped zirconia. Pre 1990 data (top) and all current data (bottom).

The data shown in Figure 3 demonstrates data mining on ceria stabilized zirconia using data parameters stored in the database. By comparing top and bottom graphs one can see that a large amount of data has been entered into the PDF since 1990. Every point on the

graph represents a separate material analysis. Ce doped zirconia is a material now used in a wide variety of commercial applications including fuel cells, cutting tools and automotive catalysts. In the bottom graph one can see clusters of data that weren't as apparent in the top graph. These correspond to tetragonal and orthorhombic modifications of the structure used in commercial applications. It the analysis of a modern catalytic converter the researcher would identify that the material was a zirconia, but also identify that the dopant was ceria, the crystal symmetry of the stabilized zirconia as either orthorhombic or tetragonal, and the exact concentration of the ceria in the commercial material.

Examples of how to combine data mining tools with enhanced graphics to characterize solid solutions, minor and trace phase identification (below 10 wgt. %), polymorph analyses, cluster analyses and nanomaterial analyses are too extensive to be shown here. However a wide range of specific examples are publically available for free download and review on the website www.icdd.com/resources/tutorials.

Conclusion: The Powder File is very dynamic and new tools are being continuously added and developed to enhance materials analysis.