V. K. Pecharsky & P.Y. Zavalij, "*Fundamentals of Powder Diffraction and Structural Characterization of Materials*", 2nd Ed., Springer (2009) p.385

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Fundamentals of Powder Diffraction and Structural Characterization of Materials

Second Edition

Springer

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Cover illustration, created by Peter Zavalij, follows the book content. It is inspired by Salvador Dali's painting "The Metamorphosis of Narcissus" where Narcissus (polycrystalline (Au,Ni)Sn4, courtesy Lubov Zavalij) falls in love with his own reflection (diffraction pattern), transforms into an egg (reciprocal lattice), and then into a flower (crystal structure in a physical space), which bears his name.

ISBN: 978-0-387-09578-3 e-ISBN: 978-0-387-09579-0
DOI: 10.1007/978-0-387-09579-0

Library of Congress Control Number: 2008930122

Printed on acid-free paper

springer.com

limits the search only to oxides of vanadium, while compounds containing other elements (e.g., vanadium hydroxides, vanadates, etc.) will not be considered and analyzed. On the other hand, "Inclusive AND" searches among all compounds containing both V and O in combination with any other chemical elements. The latter option may be useful, for example, when intercalates of vanadium oxides are suspected or studied.

Another example is found in EVA[36] search and match algorithm, which allows a user to specify the following parameters: quality marks or quality of a pattern in the database; sub-files or a class of the compound, for example, inorganic, mineral, etc.; 2θ window or tolerance; chemical composition, and others. The composition is set by selecting chemical elements and marking them as "must be present," "may or may not be present," and "cannot be present." The search can be conducted using positions of peaks or even the whole pattern.

When visually comparing potentially identical patterns, the following important issues should always be considered:

- When there are a few strong reflections in the database record, all should be present in the analyzed experimental pattern. When even one of the strong peaks is missing in the analyzed pattern, or it is present but has very low intensity, this match is likely incorrect, unless an extremely strong preferred orientation is possible in either pattern (but not in both), and there is a legitimate reason for the two to be different.
- Relative intensities should be analyzed carefully because significant discrepancies between experimental data and database entries may occur due to different wavelengths, diffractometer geometry, sample shape, or the presence and extent of preferred orientation. Preferred orientation is an important factor, and in many cases, it is unavoidable. Further, texture may be substantially different in different experiments, for example, yours, and that present in the database. Thus, the following rule should be applied when comparing intensities: a strong reflection in the database record should correspond to a strong peak in the analyzed pattern, and a weak reflection in the database record should correspond to a less intense peak in the analyzed pattern.

Even though all automatically found patterns are ranked according to certain matching criteria, visual analysis of at least several solutions (better yet, all that appear reasonable) is always recommended.

Once again, we consider experimental data used as an example throughout this chapter (Fig. 13.3). They were converted into a digitized pattern by background subtraction, $K\alpha_2$ stripping and smoothing, followed by automatic peak detection. The PDF search-match was restricted to phases containing Ni, Mn and O with the "Inclusive AND" option. Since relatively rigid restrictions were imposed on the chemical composition, search parameters were quite relaxed: the window was 0.06° of 2θ and only 2 Bragg reflections were required to coincide within the tolerance established by the window. Totally, about 20 matching patterns were found. One of

[36] Bruker AXS. EVA - DIFFRACplus Evaluation Package (2006).