# Preface

# THE KEY ROLE OF POWDER DIFFRACTION IN MATERIALS SCIENCE

The diffraction analysis of polycrystalline specimens, shortly called "powder diffraction", is one of the most important means for structural characterization in materials science and engineering. Recording a diffraction pattern yields a wealth of revealing data: the peak positions provide a "fingerprint" of the material under investigation and make phase identification possible as well as establishment of its state of (macro)stress; the (integrated) intensities allow determination of the amount of phase present and analysis of preferred orientation (texture), and, in combination with the peak positions, can even lead to crystal-structure determination; the line-profile shape (line broadening analysis) exhibits the effects of finite crystallite size and microstructural imperfections as microstrain, stacking faults and compositional inhomogeneities.

Hence, if we, the undersigned, would be asked to set up a laboratory for materials science, immediately after arranging for a light optical microscope and a hardness tester, we would advise anybody to obtain an X-ray generator and a diffractometer; only thereafter acquisition of (more trendy) sophisticated instruments as (high resolution) electron microscopes and surface analytical equipment (from Scanning Auger to Scanning Tunneling and Atomic Force microscopes) is in order.

Historical aspects of powder diffraction are dealt with in the first paper presented in these proceedings. The original, initiating work was performed in Europe and involved the development of photographic techniques performed with, for example, Debije-Scherrer and Guinier-de Wolff cameras. Although this has been the subject of even rather recent debate, it is now possible to state that the accuracy achievable with a modern diffractometer in principle surpasses that of the photographic techniques. Here we are indebted to A.J.C. Wilson[1] and, recently diseased, W. Parrish; the latter methodologist has been instrumental in the practical development of the famous, vertical (Philips) diffractometer of the fifties.

Without any doubt the advent of (personal) computers in the seventies has facilitated the actual, world-wide application of relatively advanced and in principle numerical methods proposed earlier: examples are the Stokes correction for instrumental broadening (1948)[2] and the Warren-Averbach analysis of size and microstrain (1950-1952)[3]. In this context a crucial role has been played by the pioneering work by H.M. Rietveld[4] who showed (in 1967-1969) how it is possible to arrive at the (refined) crystal-structure parameters from a measured powder diffraction pattern containing (many) overlapping reflections, on the basis of an (assumed) profile-shape function. These two developments have led to enormous revival of the interest in powder diffraction: "from ugly duckling to beautiful swan"[5]. This rise to maturity can for example be illustrated by recognizing that a first crystal-structure determination of the new class of superconductors was achieved by (neutron) powder (Rietveld) diffraction analysis[6].

We have gone a long way from the early days. Nowadays we (tend to) smile if we note that in 1928 the line-profile shape was described by a triangle[7]. On the other hand, the forthcoming generation of materials scientists will undoubtedly consider the (even split) pseudo-Voigt, Pearson VII and Voigt profile-shape functions as primitive, and right they are!

The present trend in powder diffraction involves development of methods for "total (diffraction) pattern analysis" (as is well illustrated by various review papers contained in Ref. 8), and methods for detailed, unprejudiced analysis of profile shape to characterize the microstructure[9].

In view of the past it is fitting that a series of conferences on Powder Diffraction in Europe has been initiated. The enormous attendance and participitation in Munich at EPDIC 1 ($14^{th}$ - $16^{th}$ March, 1991) illustrates that Europe still provides a focus for the field of powder diffraction.

Considering the contents of the proceedings, one may say that the really new (methodological and instrumental) developments concentrate on (i) the application of (the high intensity) synchrotron radiation (for example for structural analysis of thin films), (ii) unravelling of overlapping of clusters of peaks, (iii) selection of profile-shape functions for Rietveld analysis and linebroadening analysis, (iv) elimination of errors due to measurement limitations (as truncation of the range of measurement) and (v) analysis of structural change in non-ambient conditions.

We have been witness of and participant in the resurgence of powder diffraction since 1970 as a major tool of research in the physics and chemistry of the solid state. Materials science and engineering requires a multidisciplinary approach. Realizing that, we predict that powder diffraction analyses will play a dominant role in the progress of the field. These and following proceedings will show that.

R. Delhez
E.J. Mittemeijer

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Delft, The Netherlands


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3. B.E. Warren and B.L. Averbach, The effect of cold-work distortion on X-ray patterns, J.Appl.Phys. 21, 1950, 595-599.
4. H.M. Rietveld, A profile refinement method for nuclear and magnetic structures, J.Appl.Cryst., 2, 1969, 65-71.
5. J.L. Langford, The renaissance of powder diffraction: from ugly duckling to beautiful swan, Acta Cryst., A37 (Suppl.), 1981, C2-C3.
6. J.J. Capponi, C. Chaillout, A.W.Hewat, P. Lejay, M. Marezio, N. Nguyen, B. Raveau, J.L. Soubeyroux, J.L. Tholence and R. Tournier, Structure of the 100 K Superconductor $Ba_2YCu_3O_7$ between (5 + 300)K by Neutron Powder Diffraction, Europhys.Lett., 3, 1987, 1301-1307.
7. R. Brill, Teilchengrößenbestimmungen mit Hilfe von Röntgenstrahlen, Z. Kristallogr., 68, 1928, 387-403 .
8. R.A. Young (Editor), Proceedings International Workshop on The Rietveld Method ($13^{th}$-$15^{th}$ June, 1989, Petten, The Netherlands), International Union of Crystallography, in press.
9. R. Delhez, Th.H. de Keijser and E.J. Mittemeijer, Determination of crystallite size and lattice distortions through X-ray diffraction line profile analysis, Fres.Z.Anal.Chem., 312, 1982, 1-16.

## *Editorial Note*

The papers marked with * at the page number of the Table of Contents did not conform, as manuscripts, to the "Instructions for Authors" in some respect:

1. paper longer than 6 pages. Because of a misunderstanding some authors of noninvited papers thought it was allowed to submit papers longer than 6 pages. A considerable number of these papers have been shortened by us by using the space available more efficiently, e.g. by reducing the size of figures and tables and by rearranging these. However, this was not always possible. We apologize to the authors who did confine the length of their paper to 6 pages and now might have the feeling that they have been treated unfairly;
2. page length was not 24.5 cm (14.5 cm for title page) and/or page width was not 17 cm;
3. figures and tables were not of correct size and/or not fixed at the appropriate places in the manuscript;
4. coloured pictures were not allowed (although this was not explicitly stated).

All modifications have been performed as carefully as possible.

R. Delhez
E.J. Mittemeijer