

# PREFACE

## From Single Crystals and Powders to Polycrystals: Quantitative Texture Analysis



Over 100 years ago Röntgen discovered x-rays. And less than 20 years later Laue and his associates at Munich observed x-ray diffraction. Both events changed physics, chemistry, crystallography, mineralogy and medicine. X-ray diffraction was first documented for single crystals of copper sulfate in 1912 by Laue and his collaborators. But only 4 years later Debye and Scherrer had the ingenious idea of powder diffraction. Since then x-ray crystallography divided: Structure analysis relied largely on single crystals and phase identification on powders. Both avenues were lined with great successes. Structure determinations revealed unique features about ordered arrangement of atoms and bonding in the solid state. With the advent of computers in the sixties structure determination became more accessible and quantitative, and soon thereafter a routine. Phase identification benefited from the development of a universal catalog, based on a scheme introduced by Hanawalt in 1938. Today the JCPDS file contains 46 sets in 33 book volumes with more than 64,000 substances, and affordable PC computers find a match within a few seconds.

There have been remarkable changes in crystallography. From my predecessor I inherited boxes with Beavers-Lipson strips. I still have piles of punched cards with structure factors, tied together with rubber bands, which were carried to the computer center every morning. Today a PC, purchased for \$2000, is far more powerful than the mainframe computer in Berkeley, 25 years ago. Other aspects have not changed that much. Many computer programs are remarkably similar except that PUNCH has been replaced by WRITE. But it was not just changes in computer technology that advanced crystallography. Equally important were advances in diffraction instrumentation. I remember the days spent orienting a single crystal on a Weissenberg or precession camera, only to find out that it was not suitable to put it on a single crystal diffractometer. Quantitative diffractometers with sophisticated point and position sensitive detectors have put photographic cameras into the background. This development is continuing with 2d position sensitive CCD cameras, presently mainly at synchrotron facilities, but probably soon in every x-ray laboratory. Intensities are digitally recorded and immediately available for processing. Crystallography is one of the few fields where a textbook written in 1970 is today completely obsolete.

An important development occurred in 1969 when Rietveld proposed a new method to determine crystal structures from direct analysis of powder diffraction profiles. Not only did this simplify the experimental procedures, but enabled quantitative structure refinements on materials that were not available as large single crystals. Powders became respectable again in crystallography and today a majority of structure refinements rely on powders. Results, at least for materials with only moderate complexity, exceed the precision of single crystal work.

A single crystal and a powder are in many ways extremes. The real world consists of 'poly-crystals', aggregates of many crystals or crystallites, joined across grain boundaries. Examples are the steel body of

your car, an aluminum beverage can, a polyethylene garbage bag, a rock or a tooth. Polycrystals add enormous complexities to the solid state. Grain size, grain shape and microstructure are important. The orientation of crystals determines anisotropic properties. Interaction between these anisotropic crystals produces internal stresses. It is these aspects of solid state which have fascinated me for many years. And also here x-ray diffraction is the crucial tool for quantitative analysis. I would like to make a few comments about crystal orientation analysis.

Orientation patterns (or 'textures') change as a result of the specimen undergoing various physical processes. During preferential growth, e.g. in a film of high temperature superconductor deposited on a substrate, or in a coating of abrasion resistant nitrides on a tool steel, crystallites are aligned. When a polycrystalline material is deformed, crystals rotate into a specific pattern to maintain stress equilibrium and strain compatibility. Texture also forms during recrystallization. In this case some orientations grow at the expense of others, leading to a new overall orientation pattern.

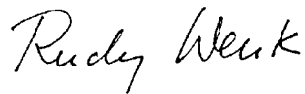
Best understood, at this point, are deformation induced textures. They are studied with polycrystal plasticity theory and can be predicted for a wide range of conditions. The very complicated patterns in an aluminum beverage can with heterogeneous strain and millions of interacting crystals, can be reproduced with a computer. Scientists have even made progress in explaining anisotropy due to texturing in such large systems as the Earth's mantle, during convection of crystalline rocks in cells extending over thousands of kilometers. Geologists can use texture patterns, measured in rocks, to infer the deformation history of a particular mountain belt. Textures indicate that the Earth's crust in the Western United States has deformed mainly by coaxial extension, rather than non-coaxial shearing. Metallurgists aim at producing workpieces with a characteristic texture which contributes favorable mechanical properties.

Textures are not restricted to inorganic materials. Biologists discovered that crystals of apatite in bones, calcite in egg shells and aragonite in mollusc mother of pearl are aligned in complicated patterns, generally with favorable mechanical properties for a particular environment. The growth direction is controlled by proteins.

While textures are becoming of interest and importance in a large variety of fields, there is growing need for quantitative characterization. Textures are typically measured by diffraction with x-ray, neutrons or electrons. The x-ray texture pole figure goniometer, an instrument combining a single crystal and a powder diffractometer, is most widely used to determine the orientation distribution of lattice directions and represent them in pole figures. Several features can greatly improve the quality of x-ray texture measurements. Most important is the collimation system to find the optimum compromise between angular resolution and defocusing. Use of a monochromator or an energy dispersive detector can greatly improve the peak-to-background ratio. For special applications position-sensitive detectors are advantageous. Rotating anode generators provide sufficient intensity for quantitative local analyses with a focused beam. Flexible software can give the users access to many functions of the instrument and let them customize it to their needs. Contrary to powder and single crystal diffractometers, pole figure goniometry is much less of a routine procedure and has to be tailored to specific applications which is a challenge for manufacturers.

Quantitative measurements are a first step. However, a pole figure (e.g.  $\{111\}$ ) is an incomplete and unsatisfactory representation of texture. Similar to a crystal structure determination from structure factors (hkl), quantitative methods exist to invert pole figures (hkl) and obtain the full three-dimensional crystal orientation distribution function (ODF). And as with crystal structures, these methods were once reserved to a few experts but are now becoming available to every applied user for a modest cost, such as in BEARTEX, the Berkeley texture package, or popLA, the preferred orientation package from Los Alamos which both cater to the applied user, rather than the texture expert. If one knows the ODF, one can then calculate physical properties of polycrystalline materials by orientation weighted averaging.

New developments are being advanced, following the example of Rietveld to use diffraction spectra to analyze polycrystals. So far applied to neutrons, but in the foreseeable future applicable to x-rays, combined information about crystal structure, texture, and phase proportions (contained in peak heights) and lattice parameters, internal stresses, grain size and microstructure (expressed in peak positions and peak shape) can be extracted simultaneously from profile analysis. This opens tremendous possibilities for characterization of the solid state and stimulates an interdisciplinary dialog and collaboration between crystallographers, mineralogists, chemists, physicists and materials scientists around an old topic: diffraction revisited.

A handwritten signature in black ink that reads "Rudy Wenk". The signature is written in a cursive, flowing style.

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