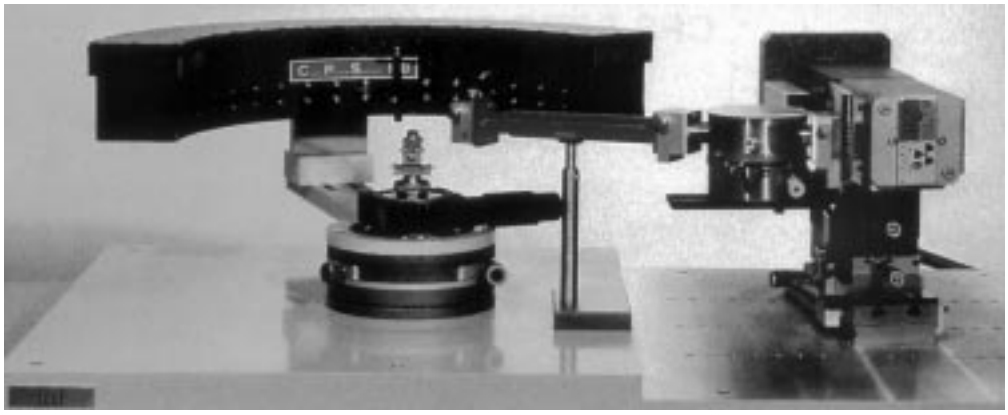
1. Diffraction and the X-Ray Powder Diffractometer



1.1 Diffraction

1.1.1 Introduction to Diffraction

Materials are made of atoms. Knowledge of how atoms are arranged into crystal structures and microstructures is the foundation on which we build our understanding of the synthesis, structure and properties of materials. There are many ways for measuring chemical compositions of materials, and methods based on inner-shell electron spectroscopies are covered in this book. The larger emphasis of the book is on measuring spatial arrangements of atoms in the range from 10^{-8} to 10^{-4} cm, bridging from the unit cell of the crystal to the microstructure of the material. For measurements over this broad spatial scale there are many different experimental techniques, but most of them involve diffraction. To date, most of our knowledge about the spatial arrangements of atoms in materials has been gained from diffraction experiments. In a diffraction experiment, an incident wave is directed into a material and a detector is typically moved about to record the directions and intensities of the outgoing diffracted waves.

"Coherent scattering" preserves the precision of wave periodicity. Constructive or destructive interference then occurs along different directions as scattered waves are emitted by atoms of different types and positions. There is a profound geometrical relationship between the directions of waves that interfere constructively, which comprise the "diffraction pattern," and the crystal structure of the material. The diffraction pattern is a spectrum of real space periodicities in a material.[1] Atomic periodicities with long repeat distances cause diffraction at small angles, while short repeat distances (as from small interplanar spacings) cause diffraction at high angles. It is not hard to appreciate that diffraction experiments are useful for determining the crystal structures of materials. Much more information about a material is contained in its diffraction pattern, however. Crystals with precise periodicities over long distances have sharp and clear diffraction peaks. Crystals with defects (such as impurities, dislocations, planar faults, internal strains, or small precipitates) are less precisely periodic in their atomic arrangements, but they still have distinct diffraction peaks. Their diffraction peaks are broadened, distorted, and weakened, however, and "diffraction lineshape analysis" is an important method for studying crystal defects. Diffraction experiments are also used to study the structure of amorphous materials, even though their diffraction patterns lack sharp diffraction peaks.

In a diffraction experiment, the incident waves must have wavelengths comparable to the spacings between atoms. Three types of waves have proved useful for these experiments. X-ray diffraction (XRD), conceived by von Laue and the Braggs, was the first. The oscillating electric field of an incident x-ray moves the atomic electrons and their accelerations generate an outgoing wave. In electron diffraction, originating with Davisson and Germer, the charge of the incident electron interacts with the positively-charged core of the atom, generating an outgoing electron wavefunction. In neutron diffraction, pioneered by Shull, the incident neutron wavefunction interacts with nuclei or unpaired electron spins. These three diffraction processes involve very different physical mechanisms, so they often provide complementary information about atomic arrangements in materials. Nobel prizes in physics (1914, 1915, 1937, 1994) attest to their importance. As much as possible, we will emphasize the similarities of these three diffraction methods, with the first similarity being Bragg's law.

[1] Precisely and concisely, the diffraction pattern measures the Fourier transform of an autocorrelation function of the scattering factor distribution. The previous sentence is explained with care in Chap. 9. More qualitatively, the crystal can be likened to music, and the diffraction pattern to its frequency spectrum. This analogy illustrates another point. Given only the amplitudes of the different musical frequencies, it is impossible to reconstruct the music because the timing or "phase" information is lost. Likewise, the diffraction pattern alone may be insufficient to reconstruct all details of atom arrangements in a material.