

2.1. Instrumentation for laboratory X-ray scattering techniques

A. KERN

2.1.1. Introduction

X-ray scattering techniques are among the most essential means of characterizing materials, as they are the most direct analytical methods for providing structural information for a material. In particular, X-ray powder diffraction has become one of the most important techniques in materials science, since many materials are first formed or are only available or used as powders or other polycrystalline forms.

The ever-increasing need for materials characterization, from basic research to industrial quality control, has led to a multitude of evolutionary and revolutionary instrument and application developments. In the past two decades, the capabilities and thus the range of application of laboratory X-ray diffractometers have increased exponentially.

The present chapter covers the full range of commonly used instrumentation for home-laboratory X-ray scattering analyses as detailed in Section 2.1.2, with the focus on powder diffraction. The scope is limited to recent and commercial designs, available off-the-shelf from the major manufacturers. Neither technologically obsolete nor niche instrumentation will be discussed. A short description of the history of X-ray instrumentation is given in Section 2.1.3, illustrating the significant technological advances made since 1985. Sections 2.1.4 to 2.1.7 describe the components of the diverse range of currently available home-laboratory X-ray powder diffractometers. The most important concepts are discussed here; for technological details the reader is referred to the original literature or to textbooks.

In order to maintain neutrality as well as timeliness, the use of brand names and photos of real equipment has been avoided.

2.1.2. Scope and terminology

An X-ray (powder) diffractometer is by definition an instrument for measuring X-ray diffraction phenomena (from powders), where ‘diffraction’ is defined as elastic, coherent scattering of X-rays from a crystal lattice (in the crystallographic literature, the terms ‘diffraction’, ‘X-ray diffraction’ and ‘Bragg diffraction’ are frequently used synonymously). In recent texts, a ‘powder’ is frequently defined as a ‘solid containing small crystallites or particles that will flow when agitated’ in accordance to the usual sense of the word in colloquial speech.

Such definitions for ‘X-ray diffractometer’ and ‘powder’ are problematic, as their scope is too narrow and arbitrarily limited. They are probably the result of the historical development of the methodology and the lack of interaction between groups representing different application areas, such as X-ray scattering, emission or absorption techniques. The application range and thus capabilities of today’s instrumentation are neither comprehensively nor even appropriately described by their implicit limitation to measuring X-ray diffraction phenomena of crystalline solids in powdered form.

It is well known that scattering and thus interference phenomena will occur with any type of waves and obstacles, and are by no means restricted to X-rays or perfectly regular arrays of atoms exhibiting long-range order (X-ray diffraction). In general,

X-ray scattering can provide information on the arrangement of atoms or particles in materials with short-range order or no order at all, like gases, liquids and amorphous solids. For this reason it is obvious that X-ray diffractometers are intrinsically suited (and are actually used) for a wide range of X-ray techniques beyond X-ray diffraction as defined above. These techniques not only comprise X-ray scattering from any solids or liquids with any degree of order, but also X-ray absorption (radiography) or X-ray emission (XRF) techniques, see also Section 2.1.4.3. Consequently, the following terminology will be used throughout the remainder of this chapter:

A *sample* is the object or quantity of material to be investigated, while the *specimen* is the representative portion of the sample that is actually prepared and analysed. Specimen properties such as microstructure and packing density may differ from the properties of the sample as a result of specimen preparation. This must be taken into account for selection of the appropriate instrument configuration, data acquisition and evaluation.

A *powder* is defined in EN-1330-11 (2007) as a ‘large number of crystallites and/or particles (*i.e.* grains, agglomerates or aggregates; crystalline or non-crystalline) irrespective of any adhesion between them’ and thus can be a loose powder (in the sense of common language), a solid block, a thin film or even a liquid. An *ideal powder* is represented by a virtually unlimited number of sufficiently sized, randomly oriented and spherical crystallites.

The term *X-ray diffractometer* will refer to an instrument that, in principle, is capable of doing any of the X-ray techniques mentioned above, further detailed in Section 2.1.4.3. Instrument components will be described independently of applications, as they are not exclusive to any application area. Note that the term X-ray diffractometer also explicitly includes ‘film cameras’. This is worth mentioning, as even recent texts still differentiate between (i) cameras, originally characterized by the use of X-ray films, and (ii) diffractometers, originally defined as an instrument derived from a camera in which the film had been replaced by a point detector. In principle, any so-called cameras and any diffractometers can be equipped with any type of today’s point, linear and area detectors, so the former distinction between cameras and diffractometers, which arose from the historical development of X-ray instrumentation, is completely obsolete.

2.1.3. Historical overview

2.1.3.1. From film cameras to diffractometers

2.1.3.1.1. Film cameras

Powder diffraction analysis started with the development of simple film cameras, right after von Laue formulated his basic diffraction theory and the Braggs, father and son, laid down the foundations of crystal structure analysis, in the years 1912–1914. The first and simplest cameras were developed independently by Debye & Scherrer (1916) and Hull (1917), using a film to detect the scattered X-rays, with the instrument geometry termed ‘Debye–Scherrer geometry’. The basic drawback of Debye–Scherrer cameras was their lack of resolution. Consequently,