3.10. Accuracy in Rietveld quantitative phase analysis with strictly monochromatic Mo and Cu radiations

L. León-Reina, A. Cuesta, M. García-Maté, G. Álvarez-Pinazo, I. Santacruz, O. Vallcorba, A. G. De la Torre and M. A. G. Aranda

3.10.1. Introduction

Most industrial materials are multiphase systems and the accurate determination of their phase assemblage is key to understanding their performances. There are different approaches to carrying out quantitative phase analysis (QPA; see Chapter 3.9); however, nowadays, the Rietveld method is the most widely employed methodology for QPA of crystalline materials (Madsen *et al.*, 2001; Scarlett *et al.*, 2002), including cements (Stutzman, 2005; León-Reina *et al.*, 2009; Chapter 7.12).

The factors affecting the accuracy and precision of Rietveld quantitative phase analysis (RQPA) results can be gathered into three main groups: (i) instrument related, (ii) sample-preparation related and (iii) data-analysis protocol(s). The Rietveld method is a standardless methodology which uses the crystal-structure descriptions of each crystalline component to calculate its powder pattern. For this reason, the correct choice of crystal-structure description for each phase in multiphase materials is key (Zevin & Kimmel, 1995; Madsen *et al.*, 2001, 2011). The influence of the instrument type on RQPA has previously been evaluated (Madsen *et al.*, 2001) and the main conclusion was that neutron and synchrotron powder diffraction yielded the best results owing to larger irradiated volumes and also to the minimization of microabsorption effects.

High-energy (short-wavelength) X-rays contribute (i) to minimize absorption and microabsorption effects, (ii) to the measurement of a higher number of Bragg peaks and (iii) to increase the irradiated volume of the specimen. Figs. 3.10.1(*a*) and 3.10.1(*b*) show the irradiated volumes bathed by X-rays when using flat samples for Mo and Cu radiations in transmission geometry, and Fig. 3.10.1(*c*) shows the irradiated volume for Cu in reflection mode (Cuesta *et al.*, 2015). Mo radiation combined with a flat sample in transmission geometry allows an irradiated volume of close to 100 mm^3; meanwhile, for Cu radiation (flat samples in reflection and transmission geometries) the irradiated volumes are close to 5 mm^3 (Cuesta *et al.*, 2015). In this context, it is worth mentioning that the absorption correction for flat-

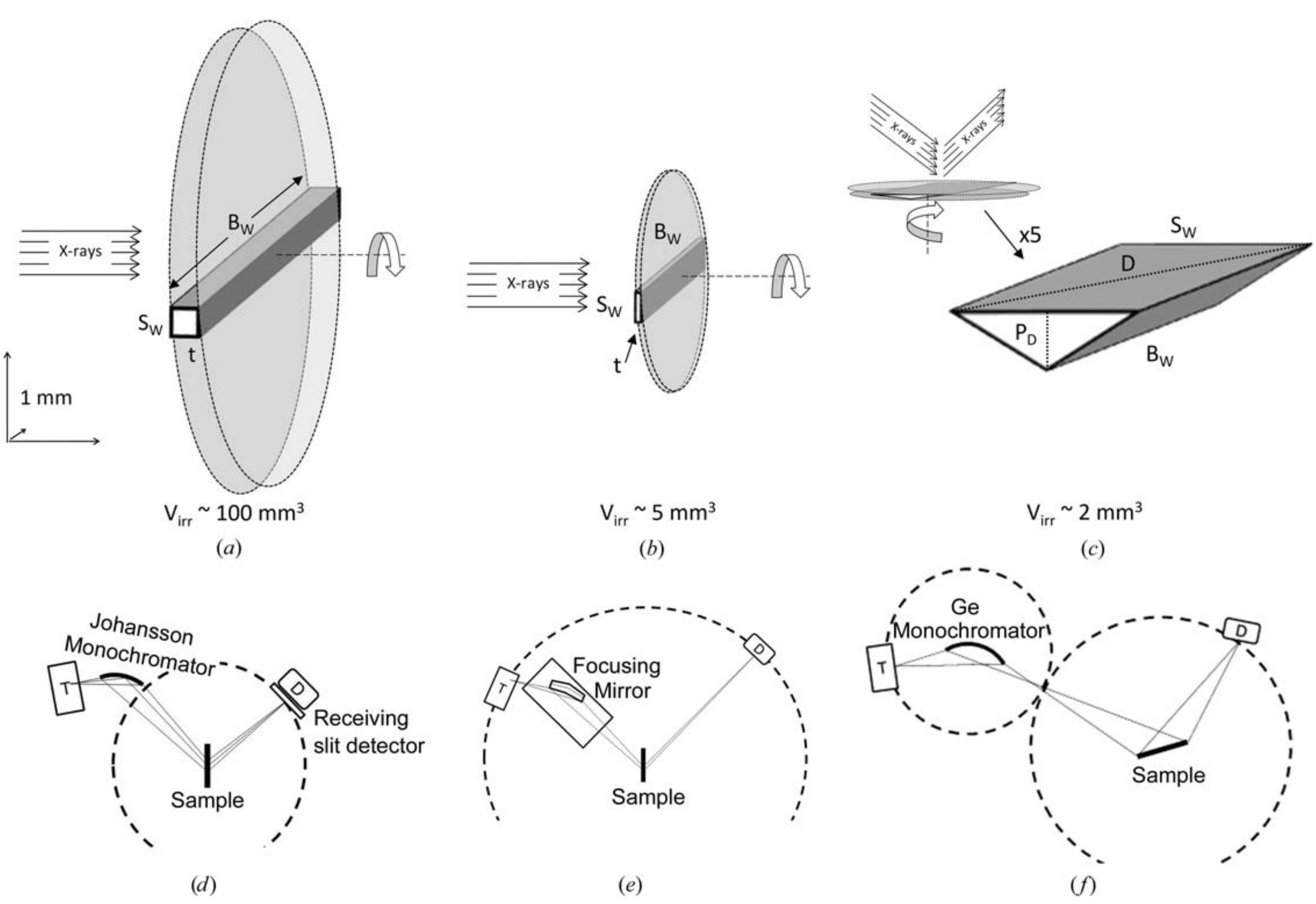

Figure 3.10.1
Irradiated volume for a flat sample holder in transmission mode using (*a*) Mo radiation and (*b*) Cu radiation, and (*c*) reflection mode using Cu radiation. Diffraction-geometry sketches: (*d*) transmission geometry with primary monochromator, (*e*) transmission geometry with focusing mirror and (*f*) reflection geometry with primary monochromator. [Reprinted from Cuesta *et al.* (2015) with permission from Cambridge University Press.]