JOURNAL OF
SYNCHROTRON
RADIATION

ISSN: 1600-5775
journals.iucr.org/s

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J. Synchrotron Rad. (2011). **18**, 938–941

Journal of
Synchrotron Radiation

ISSN 0909-0495

addenda and errata

On the calculation of the gauge volume size for energy-dispersive X-ray diffraction. Erratum

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An equation in the paper by Rowles [(2011), *J. Synchrotron Rad.* **18**, 938–941] is corrected.

In the paper by Rowles (2011), there is an error in equation (32): the equation should read

$$p = -\frac{h}{2\tan\gamma}. \quad (32)$$

References

Rowles, M. R. (2011). *J. Synchrotron Rad.* **18**, 938–941.

  doi:10.1107/S0909049511053878

Journal of
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Radiation

ISSN 0909-0495

Received 23 June 2011
Accepted 16 August 2011

On the calculation of the gauge volume size for energy-dispersive X-ray diffraction

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Equations for the calculation of the dimensions of a gauge volume, also known as the active volume or diffraction lozenge, in an energy-dispersive diffraction experiment where the detector is collimated by two ideal slits have been developed. Equations are given for equatorially divergent and parallel incident X-ray beams, assuming negligible axial divergence.

Keywords: energy-dispersive diffraction; gauge volume; active volume.

1. Introduction

Energy-dispersive diffraction has a range of applications in measuring engineering stress and strain (Korsunsky *et al.*, 2011), *in situ* experimentation in difficult environments (Scarlett *et al.*, 2009) and determination of reaction kinetics (Russenbeek *et al.*, 2011; Provis & Van Deventer, 2007).

The experimental arrangement for such an experiment is quite simple; it is often a parallel or divergent incident X-ray beam with an energy-dispersive detector fixed at a set angle, 2θ, to the incident beam, with the diffracted beam collimated using a pair of slits (Korsunsky *et al.*, 2010) or two parallel plates (Barnes *et al.*, 1998). The effect of a parallel-plate collimator is simply to produce several gauge volumes, effectively increasing the length of the diffraction volume along the incident beam (Häusermann & Barnes, 1992).

In order to correctly plan an experiment, or carry out a full analysis of the collected data, it is often important to know the exact dimensions of the volume from which the diffraction information is measured, *i.e.* the gauge volume. It is assumed that the X-ray beam is uniform in the axial direction and that the full three-dimensional representation of the gauge volume can be calculated by a simple projection of the cross section.

2. Gauge volume

2.1. General

The size and location of the gauge volume cross section is as shown in Fig. 1. The acceptance angle of an energy-dispersive detector, D, is defined by a pair of ideal slits along the diffracted beam path. The X-ray source is assumed to be an ideal line source with negligible axial divergence. The slits are assumed to be centred on, and perpendicular to, the diffracted beam path, which is at an angle φ ($\varphi \equiv 2\theta$) to the incident beam. The acceptance angle of the diffracted beam slits is given by 2α, where

$$\tan\alpha = \frac{a+b}{2c}, \tag{1}$$

where a and b are the widths of the primary and secondary slits, and c is the distance between them.

The two angles β and γ are given by

$$\beta = \varphi - \alpha, \tag{2}$$

$$\gamma = \pi - \varphi - \alpha, \tag{3}$$

where φ is the angle of the diffracted beam.

The distance, d, from the primary slit to the convergence of the diffracted beams, is given by

$$d = \frac{ac}{a+b}. \tag{4}$$

The lengths of the gauge volume along the centre of the incident beam before and after the centre of the goniometer is given by

$$f = (e+d)\,\frac{\sin\alpha}{\sin\beta}, \tag{5}$$

$$g = (e+d)\,\frac{\sin\alpha}{\sin\gamma}, \tag{6}$$

respectively, where e is the distance along the diffracted beam from the primary slit to the centre of the goniometer.

The cross-sectional area of the gauge volume can be calculated *via* Bretschneider's formula (Zwillinger, 2003, p. 322),

$$\text{Area} = \left[(s-\overline{12})(s-\overline{23})(s-\overline{34})(s-\overline{41}) - \overline{12}\,\overline{23}\,\overline{34}\,\overline{41}\cos^2\varphi\right]^{1/2}, \tag{7}$$

where s is given by

$$s = \frac{\overline{12}+\overline{23}+\overline{34}+\overline{41}}{2}, \tag{8}$$

where the construct '$\overline{yz}$' denotes the length of the line segment from point y to point z as numbered in Fig. 1. The lengths, $\overline{12}$, $\overline{23}$, $\overline{34}$ and $\overline{41}$ are defined in equations (11)–(14) and (25)–(28) for the divergent and parallel incident-beam cases, respectively.

The total length of the gauge volume is the distance between the projection of points 1 and 3 onto the centre of the incident beam, and the length of the central region is the distance between the projection of points 2 and 4 onto the centre of the incident beam. The central region is defined as the central quadrilateral sandwiched between two exterior triangles, the sum of which is the gauge volume. The size of the central region is of importance when examining, for example, thin flat plates oriented perpendicular to the incident beam, as the plate must be situated inside the central region to ensure uniform intensities across the thin dimension of the sample.

If the length of the central region, as given in equations (22) and (34) below, is negative, then the vertical extent of the gauge volume is defined by the diffracted beam optics, otherwise the vertical extent of the gauge volume is defined by the incident beam, as is the case in Fig. 1. The total and central length equations are not valid for $90^\circ - \alpha < \varphi < 90^\circ + \alpha$, as, in this range, the meaning of the points 1–4 is altered. Finally, the total and central lengths, as defined here, are valid up to a scattering angle of 90°, after which the equation for the total length yields the central length, and *vice versa*. As a guide, the total length is always greater than the central length.

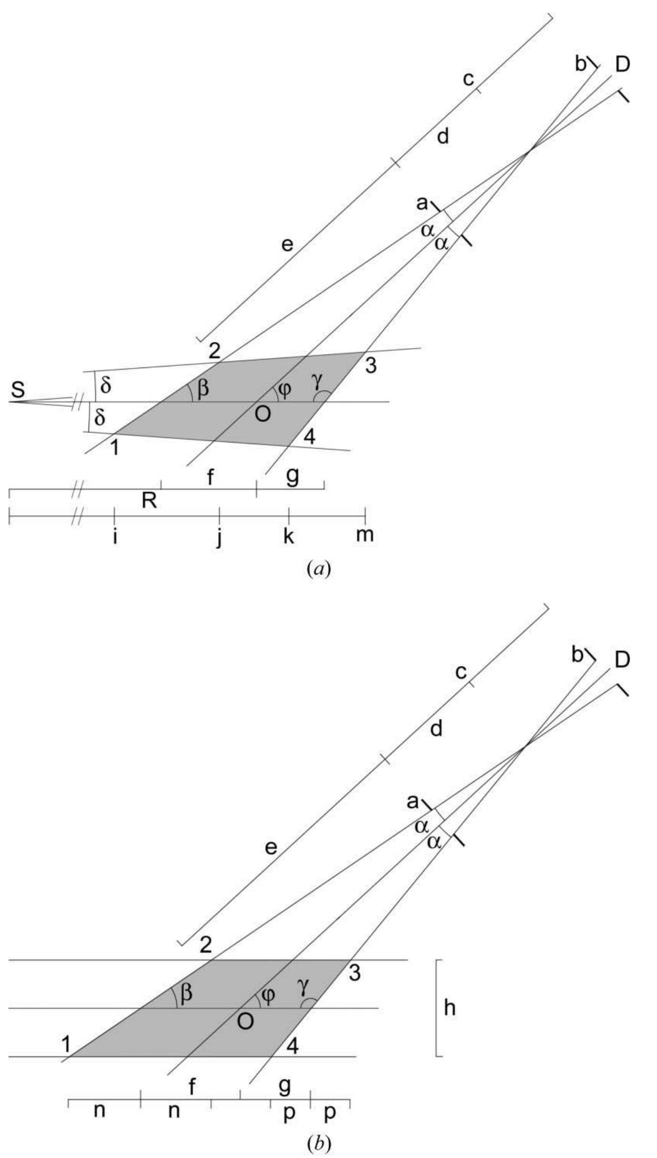

Figure 1
Schematic diagram of the experimental layout of an energy-dispersive diffraction experiment. The shaded regions denote the gauge volume; the region from which diffraction information is measured. An energy-dispersive detector, *D*, is collimated by the primary, *a*, and secondary, *b*, slits, which are perpendicular to the beam diffracted at an angle φ. The two slits are a distance *c* apart, and the primary slit is a distance *e* from the centre of the goniometer, *O*. The acceptance angle of the slits is given by 2α. The distances *f* and *g* denote the upstream and downstream lengths of the gauge volume at the centre of the incident beam from the centre of the goniometer. The total length of the gauge volume is given by the distance between the projection of points 1 and 3 onto the centre line of the incident beam. The length of the central region of the gauge volume is given by the distance between the projection of points 2 and 4 onto the centre line of the incident beam. (*a*) Divergent beam. The apparent source, *S*, is a distance *R* from the centre of the goniometer, and has a divergence of 2δ. (*b*) Parallel beam. The incident beam has a height of *h*.

2.2. Divergent incident beam

The divergent beam case is presented in Fig. 1(*a*). The apparent source radius is given by *R*, the distance from the apparent source, *S*, to the centre of the goniometer, *O*. The apparent incident beam divergence is given by 2δ.

For an ideal line source of finite height *q* at a distance $R_{\rm inst}$ from the centre of the goniometer with a divergence controlled by a slit of height *t* at a distance *u* from the line source, the apparent source radius, *R,* and apparent incident beam divergence, 2δ, are given by

$$R = \frac{qu}{t-q} + R_{\rm inst}, \tag{9}$$

$$\tan\delta = \frac{t-q}{2u}. \tag{10}$$

The lengths of the four sides of the gauge volume are given by

$$\overline{12} = (R-f)\sin\delta\left[\frac{1}{\sin(\beta-\delta)} + \frac{1}{\sin(\beta+\delta)}\right], \tag{11}$$

$$\overline{23} = \sin\alpha\left[e + d - \frac{R\sin\delta}{\sin(\varphi-\delta)}\right]\left[\frac{1}{\sin(\beta-\delta)} + \frac{1}{\sin(\gamma+\delta)}\right], \tag{12}$$

$$\overline{34} = (R+g)\sin\delta\left[\frac{1}{\sin(\gamma+\delta)} + \frac{1}{\sin(\gamma-\delta)}\right], \tag{13}$$

$$\overline{41} = \sin\alpha\left[e + d + \frac{R\sin\delta}{\sin(\varphi+\delta)}\right]\left[\frac{1}{\sin(\beta+\delta)} + \frac{1}{\sin(\gamma-\delta)}\right], \tag{14}$$

and the lengths of the diagonals are given by

$$\overline{13}^2 = \overline{12}^2 + \overline{23}^2 + 2\,\overline{12}\,\overline{23}\cos(\beta-\delta), \tag{15}$$

$$\overline{24}^2 = \overline{23}^2 + \overline{34}^2 + 2\,\overline{23}\,\overline{34}\cos(\gamma+\delta). \tag{16}$$

The lengths *i*, *j*, *k* and *m* refer to the distance from the source to the projection of points 1, 2, 4 and 3, respectively, onto the centre line of the incident beam. These lengths are given by

$$i = \frac{(R-f)\sin\beta\cos\delta}{\sin(\beta+\delta)}, \tag{17}$$

$$j = \frac{(R-f)\sin\beta\cos\delta}{\sin(\beta-\delta)}, \tag{18}$$

$$k = \frac{(R+g)\sin\gamma\cos\delta}{\sin(\gamma-\delta)}, \tag{19}$$

$$m = \frac{(R+g)\sin\gamma\cos\delta}{\sin(\gamma+\delta)}. \tag{20}$$

The total length of the gauge volume is given by the difference of (20) and (17); the length of the central region of the gauge volume is given by the difference of (19) and (18),

$$\text{Length}_{\rm total} = m - i, \tag{21}$$

$$\text{Length}_{\rm centre} = k - j. \tag{22}$$

The beam heights at points 2 and 4 are given by

$$h_2 = \min\left[-\frac{\overline{23}\sin(\gamma+\delta)}{\cos\gamma}, 2j\tan\delta\right], \tag{23}$$

$$h_4 = \min\left[\frac{\overline{41}\sin(\beta+\delta)}{\cos\beta}, 2k\tan\delta\right], \tag{24}$$

where $\min(x, y)$ denotes choosing the minimum value of x or y as the beam height.

2.3. Parallel incident beam

The parallel beam case is shown in Fig. 1(*b*). The lengths of the four sides of the gauge volume are given by

$$\overline{12} = h/\sin\beta, \tag{25}$$

$$\overline{23} = \sin\alpha\left(e + d - \frac{h}{2\sin\varphi}\right)\left(\frac{1}{\sin\beta} + \frac{1}{\sin\gamma}\right), \tag{26}$$

$$\overline{34} = h/\sin\gamma, \tag{27}$$

$$\overline{41} = \sin\alpha\left(e + d + \frac{h}{2\sin\varphi}\right)\left(\frac{1}{\sin\beta} + \frac{1}{\sin\gamma}\right), \tag{28}$$

where h is the height of the beam. The lengths of the diagonals are given by

$$\overline{13}^2 = \overline{12}^2 + \overline{23}^2 + 2\,\overline{12}\,\overline{23}\cos\beta, \tag{29}$$

$$\overline{24}^2 = \overline{23}^2 + \overline{34}^2 + 2\,\overline{23}\,\overline{34}\cos\gamma. \tag{30}$$

The distances $2n$ and $2p$ refer to the distance from the projection of points 1 and 2, and 3 and 4, respectively, onto the centre line of the incident beam. These lengths are given by

$$n = \frac{h}{2\tan\beta}, \tag{31}$$

$$p = \frac{h}{2\tan\gamma}. \tag{32}$$

The total length, and the length of the central portion, of the gauge volume are given by the combination of equations (5), (6), (31) and (32),

$$\text{Length}_{\text{total}} = (f + n) + (g + p), \tag{33}$$

$$\text{Length}_{\text{centre}} = (f - n) + (g - p). \tag{34}$$

The beam heights at points 2 and 4 are given by

$$h_2 = \min\left(-\overline{23}\tan\gamma, h\right), \tag{35}$$

$$h_4 = \min\left(\overline{41}\tan\beta, h\right), \tag{36}$$

where $\min(x, y)$ denotes choosing the minimum value of x or y as the beam height.

3. Application

At a recent experiment at beamline I12-JEEP at the Diamond Light Source, it was necessary to calculate the gauge volume dimensions to enable the correct positioning of the sample. All the relevant instrument dimensions are given in Table 1. The calculated parameters, along with the gauge volume lengths, are given in Table 2. As the main interest in this experiment was the investigation of surface layers, it was necessary to ensure that the sample was located in the central region of the gauge volume. This made certain that there was an equal intensity distribution across the thickness of the sample, ensuring that the resultant data analysis would not be biased. The calculation of the length of the central region of the gauge volume gave an indication as to the tolerances in which our sample alignment was effective.

Table 1
Measured instrument parameters.

Measured parameters	Dimension (mm or °)
a	0.15
b	0.20
c	1455
e	553
h	1
φ	5

Table 2
Calculated instrument parameters using the parallel incident beam model.

Calculated parameters	Dimension (mm or °)
d	623
f	1.63
g	1.62
n	5.72
p	5.71
α	0.00689
β	4.99
γ	174.99
Total length	14.7
Central length	−8.18

4. Conclusion

The equations for the calculation of the dimensions and area of a gauge volume in an energy-dispersive diffraction experiment where the detector is collimated by two slits are given. Equations are given for both equatorially divergent and parallel incident X-ray beams with negligible axial divergence.

Example values are given for an experiment carried out at beamline I12-JEEP at the Diamond Light Source. For synchrotron sources the parallel beam case should be sufficient for most applications. Implementation of laboratory sources may require the use of the divergent beam case.

I would like to acknowledge Mark Styles (University of Melbourne), Daniel Riley (ANSTO), Ian Madsen and Nikki Scarlett (CSIRO) for their helpful discussions during the experiment. Travel funding provided by the International Synchrotron Access Program (ISAP) managed by the Australian Synchrotron is also acknowledged. The ISAP is an initiative of the Australian Government being conducted as part of the National Collaborative Research Infrastructure Strategy. This research was undertaken on the I12-Joint Engineering, Environmental and Processing beamline at the Diamond Light Source, Oxfordshire, UK. I would like to thank beamline scientist Drs Thomas Connolley, Christina Reinhard and Michael Drakopolous for their assistance during the experiment.

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