Journal of
Applied Crystallography
ISSN 0021-8898

Notes for Authors
Proceedings of the SAS 2002 Conference

1. Submission for refereeing

Authors should submit **four copies** of the paper, accompanied by a signed Transfer of Copyright Agreement form (Appendix *A*). Three of the copies should typed in double-spaced format, and one should be prepared in publication-ready format (see Appendix *B*). Contributions should conform to the general editorial style of *Journal of Applied Crystallography*.

Manuscripts should be submitted to:

SAS 2002 Conference Secretariat
Dipartimento di Chimica Fisica
Calle Larga S. Marta 2137
30123 Venice
Italy

Every effort should be made to ensure that the submission is made either on or before the opening day of the conference. **Papers received after 29 August 2002 will not be accepted for publication.**

At the acceptance stage authors will be required to submit an electronic publication-ready copy (see Appendix *B*).

2. Length of contributions

Contributed papers should be no more than **five** pages in publication-ready format. This equates to approximately 4000 words including figures and tables (a standard figure equates to 250 words). Invited papers are limited to **eight** pages.

3. Manuscript preparation

3.1. File formats

The manuscript should be prepared according to the instructions in Appendix *B*.

Authors should use the special SAS 2002 templates available from the Editorial Office by e-mail (**med@iucr.org**) or by ftp (see the sas2002 folder in the 'templates' directory at ftp.iucr.org). Figures should be prepared in PostScript, encapsulated PostScript or TIFF formats.

3.2. Title and authors

The title should be short and informative. The contact author should provide an e-mail address and a fax number. The e-mail address will be used for editorial communications and despatch of electronic reprints.

3.3. Abstract

The *Abstract* should state concisely the principal results obtained. Ordinarily 100 words will suffice. It should make no reference to tables, diagrams, or formulae contained in the body of the paper. Literature references in an *Abstract* are discouraged. If a reference is unavoidable, it should be sufficiently full within the *Abstract* for unambiguous identification, *e.g.* [Smith (2002). *J. Appl. Cryst.* **35**, 21–31].

3.4. Units

The International System of Units (SI) is used except that the angstrom (symbol Å, defined as 10^{-10} m) is generally preferred to the nanometre (nm) or picometre (pm) as the appropriate unit of length. Recommended prefixes of decimal multiples should be used rather than '$\times 10^n$'.

3.5. Figures

Figures should be prepared using one of the file formats listed in §3.1. Each diagram must also be provided in 'hard-copy' form, as detailed upon submission. Colour or greyscale figures should be provided as glossy prints; laser printer or photocopier output will generally be unsatisfactory for colour reproduction. Inclusion of colour will be at the discretion of the Editor.

3.6. Tables

Tables produced in Word should be prepared using the Word table editor.

3.7. Mathematics

Authors submitting in Word should use the Word equation editor to prepare displayed mathematical equations.

3.8. Multimedia

Multimedia additions to a paper are welcomed and will be made available *via* **Crystallography Journals Online**. Please contact the Editorial Office for submission details.

3.9. References

References to published work must be indicated by giving the authors' names followed immediately by the year of publication, *e.g.* Neder & Schulz (1998) or (Neder & Schulz, 1998). Where there are three or more authors the reference in the text should be indicated in the form Smith *et al.* (1998) or (Smith *et al.*, 1998) *etc.* All authors should be included in a full list at the end of the paper. This list should be arranged alphabetically and conform with the following style:

Andrews, M. D., Wright, H. E. & Clarke, S. A. (2001). In preparation.
Brünger, A. T. (1992). *X-PLOR. Version 3.1. A System for X-ray Crystallography and NMR*. Yale University, Connecticut, USA.
Collaborative Computational Project, Number 4 (1994). *Acta Cryst.* D**50**, 760–763.
Crowther, R. A. (1972). *The Molecular Replacement Method*, edited by M. G. Rossmann, pp. 173–178. New York: Gordon and Breach.

Note that **inclusive** page numbers must be given.

4. *Crystallography Journals Online*

Full details of author services can be found at http://journals.iucr.org/j/services/authorservices.html.

4.1. Electronic status information

After acceptance, authors may obtain information about the current status of their papers *via* the WWW at http://journals.iucr.org/services/status.html.

4.2. Reprints

After publication the correspondence author will be able to download the electronic reprint of the published article, free of charge. Authors will also be able to order printed reprints at the proof stage.

5. Contact information

Contact details for the IUCr Editorial Office in Chester are as follows:

The Managing Editor
International Union of Crystallography
5 Abbey Square
Chester CH1 2HU
UK

Telephone: 44 1244 342878
Fax: 44 1244 314888
E-mail: med@iucr.org
ftp: ftp.iucr.org
WWW: http://journals.iucr.org

APPENDIX *A*

International Union of Crystallography
Transfer of Copyright Agreement

Title of Article (*Please type or use capital letters*)

. . . . . .

. . . . . .

Authors (*Please type or use capital letters*)

. . . . . .

. . . . . .

Copyright to the above article is hereby transferred (except as otherwise required by national laws†) to the International Union of Crystallography, effective if and when the article is accepted for publication in *Acta Crystallographica*, *Journal of Applied Crystallography* or *Journal of Synchrotron Radiation*. In addition to reproduction in conventional printed form your article will be stored electronically. Your assignment of Copyright signifies your agreement to the journal making arrangements to include your paper in document delivery services, CD-ROMs and electronic databases, *etc*., worldwide. This transfer of copyright also applies to data submitted in machine-readable form. However, the authors reserve the following:

(1) All proprietary rights other than copyright, such as patent rights.
(2) The right to use all or part of this article in future works prepared by or on the behalf of the author.
(3) In the case of 'work for hire', the right of the employer to incorporate all or part of this article in future works.
(4) The royalty-free right of the employer to make copies of this article, without further permission, for his or her own use but not for resale. Any identification or notice appearing on the pages to be reproduced must not be deleted.

Signature . . . . . .

Name and position, if not author

. . . . . .

. . . . . .

Date . . . . . .

Signature . . . . . .

Name and position, if not author

. . . . . .

. . . . . .

Date . . . . . .

This Transfer of Copyright Agreement must be signed by at least one of the authors (who agrees to inform the others, if any) or, in the case of a 'work made for hire', by the employer.

By signing this form you certify that your contribution is your original work, has not been published before (in any language or medium) and is not being considered for publication elsewhere; that you have obtained permission for and acknowledged the source of any excerpts from other copyright works; and that to the best of your knowledge your paper contains no statements which are libellous, unlawful or in any way actionable.

The signed statement must be received before the article can be accepted for publication. Requests for further information should be sent to the Executive Secretary of the Union.

† In this case please give further details overleaf.

For use of the International Union of Crystallography only.

M. E. Reference	Coeditor No.	Issue	Journal

This form may be photocopied.

APPENDIX *B*
Instructions for electronic publication-ready papers

All papers will be printed in publication-ready format. Publication-ready copy should be prepared according to the following instructions. Please read these carefully, as publication-ready copy that does not conform to these instructions will be returned for correction.

(1) The paper should be submitted according to the SAS 2002 Notes for Authors. The publication-ready electronic version will not be needed until your paper has been **accepted** by the Editor.

(2) The maximum length of publication-ready copy permitted for these proceedings is **five** pages for contributed, **eight** pages for invited papers.

(3) Use the SAS 2002 electronic templates for the preparation of your publication-ready copy. Figures and tables should be included in their correct positions.

(4) The electronic templates (LaTeX and WORD) are available by ftp from the address ftp.iucr.org in the directory 'templates/j/sas2002'. The styles shown below are already set up in these templates.

(5) The style of a publication-ready paper for the SAS 2002 Proceedings is shown on the following pages. The detailed typographic specifications are as follows:

Layout details

Page size (text area):	183 × 238 mm
Column width:	88 mm
Space between columns:	7 mm

Fonts, spacing and WORD template styles

		WORD template styles
Title:	**12 pt bold Arial or Helvetica**	IUCr article title
Authors:	**10 pt bold Arial or Helvetica**	IUCr author section
Affiliations:	*9 pt oblique Arial or Helvetica*	IUCr affiliation text
Abstract:	9 pt Times Roman	IUCr abstract
Keywords:	**8 pt bold Arial or Helvetica**	IUCr keywords
Headings		
Level 1:	**8 pt bold Arial or Helvetica, aligned left**	IUCr heading 1
Level 2:	**8 pt bold Arial or Helvetica, aligned left**	IUCr heading 2
Level 3:	**8 pt bold Arial or Helvetica, aligned left**	IUCr heading 3
Text:	9 pt Times Roman	IUCr body text
References:	8 pt Times Roman	IUCr references
Figure captions:	8 pt Times Roman	IUCr figure caption
Table captions:	8 pt Times Roman	IUCr table caption
Table text:	7 pt Times Roman	IUCr table text
Footnotes:	8 pt Times Roman	IUCr footnote text

If you do not have Arial or Helvetica fonts available, please use another sans-serif font.

(6) Once your paper has been accepted, the Editor will ask you to provide (*a*) an electronic version of the final publication-ready paper as a LaTeX or WORD file including figures in their correct positions, (*b*) originals of all figures and (*c*) electronic versions of all figures in PostScript, Encapsulated PostScript or TIFF format. Please send the material to the Editor (not the Editorial Office) in a rigid card envelope to ensure it is not damaged in the post.

(7) When the publication-ready copy has been approved by the Editor, it will be forwarded to the Editorial Office in Chester for publication.

Treatment of grazing-incidence small-angle X-ray scattering data taken above the critical angle

A. Martorana,[a,b]* A Longo,[b] F. d'Acapito,[c] C. Maurizio,[d] E. Cattaruzza[e] and F. Gonella[e]

[a]Dipartimento di Chimica Inorganica, Università di Palermo, Viale delle Scienze, I-90128 Palermo, Italy, [b]ICTPN-CNR, via Ugo La Malfa, 153, I-90146 Palermo, Italy, [c]INFM and European Synchrotron Radiation Facility, GILDA-CRG, BP 220, F-38043 Grenoble, France, [d]INFM and Dipartimento di Fisica, Università di Padova, via Marzolo, 8, I-35131, Padova, Italy, and [e]INFM and Dipartimento di Chimica Fisica, Università di Venezia, Dorsoduro 2137, I-30123 Venezia, Italy. E-mail: nino@ictpn.pa.cnr.it

The equations taking into account refraction at the sample surface in grazing-incidence small-angle X-ray scattering (GISAXS) when the angle between the incoming beam and the sample surface is slightly larger than the critical angle are d... and discussed. It is demonstrated that the refracti... incoming and the scattered beam at the sam... GISAXS pattern and that, when a... perpendicular to the inco... on the azimuthal ... depending... incidenc... different ... intensities ... making the ... simulations ... interacting h... As a case study, the refraction correction is ap... to the investigation of a Cu–Ni implant on silica glass.

Keywords: grazing incidence; small-angle X-ray scattering; critical angle.

1. Introduction

Grazing-incidence small-angle X-ray scattering (GISAXS) (Levine *et al.*, 1989) is a powerful tool for the structural characterization of thin (micrometre scale) superficial layers containing nanosized particles. Samples can be obtained by ion implantation (Babonneau *et al.*, 1999; d'Acapito *et al.*, 1998; Cattaruzza *et al.*, 2000), sol–gel synthesis (Kutsch *et al.*, 1997) or vapour deposition (Naudon & Thiaudière, 1997). Investigation of particles deposited on the surface is usually performed at the critical angle α_c; however, when dealing with buried nanostructures, the working angle must be slightly higher than α_c to permit a controlled limited beam penetration in the matrix. In the latter geometry, the refracted beam acts as the effective primary beam of the SAXS experiment and the scattered radiation is likewise refracted on leaving the sample. Thus, correction is required in order to retrieve the actual scattering angle $2\theta'$ from the measured 2θ between the incoming beam and the outgoing X-rays.

The use of an experimental setup consisting of a planar detector perpendicular to the incident beam assures better counting statistics, with respect to linear devices, and also allows the investigation of anisotropic implants (Babonneau *et al.*, 1999). On the other hand, the scattered beams recorded at the same 2θ but at various azimuthal φ angles strike the sample surface with different inclination, thus requiring suitable correction for refraction. In the literature, the correction is given only on the plane containing the incoming beam and the surface normal of the sample, that is, at $\varphi = 0$ (Kutsch *et al.*, 1997). The equations allowing one to obtain the internal angles $(2\theta', \varphi')$ from the detector angles $(2\theta, \varphi)$ are derived in the next section of this paper.

Despite the small vertical size of the incident beam, its projection on the sample surface is very long when working in grazing incidence (typically a few centimetres), so that the limits of the illuminated sample region are actually determined by the overall sample length. The smearing of the r... ed pattern arising from the sample size increase... ttering angle (becoming noteworthy in the... decreases with the distance of t... ment. An estimate of t... ring can affect... has been ... y scat- ... widths. ... the refraction ... pair of detector angles. ... ations involving the scattering ... se interacting hard spheres (Guinier & ... hat for isotropic implants a simplified procedure

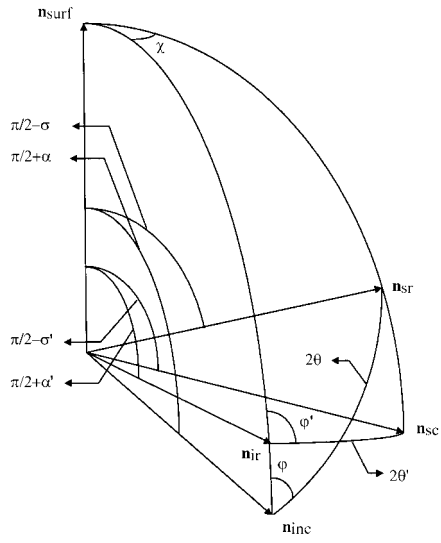

Figure 1
Scheme of directions and angles for refraction correction. $\mathbf{n}_{surf}$ is the unit vector normal to the sample surface; $\mathbf{n}_{inc}$ is the unit vector in the direction of the incoming beam; $\mathbf{n}_{ir}$ is the unit vector in the direction of the refracted incoming beam; $\mathbf{n}_{sc}$ is the unit vector in the direction of the scattered beam; $\mathbf{n}_{sr}$ is the unit vector in the direction of the refracted scattered beam; χ is the angle between the planes $(\mathbf{n}_{surf}, \mathbf{n}_{inc})$ and $(\mathbf{n}_{surf}, \mathbf{n}_{sc})$; 2θ is the angle between $\mathbf{n}_{inc}$ and $\mathbf{n}_{sr}$, measured at the detector; $2\theta'$ is the scattering angle between $\mathbf{n}_{ir}$ and $\mathbf{n}_{sc}$; α and α' are the angles of the incoming beam at the sample surface before and after refraction, respectively (notice that the angle $\pi/2 + \alpha$ is evaluated between the positive directions of $\mathbf{n}_{surf}$ and $\mathbf{n}_{inc}$, so that the incoming beam actually strikes the sample travelling from left to right in the figure); σ' and σ are the angles of the scattered beam at the sample surface before and after refraction, respectively; φ is the azimuthal detector angle between the planes $(\mathbf{n}_{surf}, \mathbf{n}_{inc})$ and $(\mathbf{n}_{inc}, \mathbf{n}_{sr})$; φ' is the azimuthal scattering angle between the planes $(\mathbf{n}_{surf}, \mathbf{n}_{ir})$ and $(\mathbf{n}_{ir}, \mathbf{n}_{sc})$.

238 mm

88 mm

183 mm

Table 1
Fitting parameters within the LMA of the scattering curve (*a*) after and (*b*) before correction for double refraction of the X-ray beam impinging on the sample.

η_{HS} is the hard-sphere volume fraction in the structure factor; *a* and *b* are the parameters that define the Weibull function $W(R) = (a/b)(R/b)^{a-1}\exp[-(R/b)^a]$.

Experimental scattering curve	*a*	*b* (Å)	Mean cluster radius (Å)	Mean intercluster distance (Å)	η_{HS}
(*a*)	2.48	21.5	19.1	87	0.24
(*b*)	2.35	21.5	21.0	68	0.39

is allowed, consisting first of azimuthal integration of the recorded intensities and then correction for refraction according to an average φ angle. An application to the actual case of a Cu–Ni-implanted layer on silica glass is also described.

2. Refraction correction

The correction for refraction at the sample surface of both the incident and the scattered beams is based on the scheme dr… Fig. 1. The angles $(2\theta, \varphi)$ are measured at the det… $(2\theta', \varphi')$, the actual scattering angles, are… detector placed within the implant… the refracted incoming be… tion of $(2\theta', \varphi')$ a… sional tri…

si… $\ldots s(2\theta)$, (2)

$\ldots(\sigma)/(1-\delta)$, (3)

$$\cos\chi = (\cos 2\theta + \sin\alpha\sin\sigma)/\cos\alpha\cos\sigma, \quad (4)$$

$$\cos 2\theta' = \cos\alpha'\cos\sigma'\cos\chi - \sin\alpha'\sin\sigma', \quad (5)$$

$$\cos\varphi' = (\sin\sigma' + \sin\alpha'\cos 2\theta')/(\cos\alpha'\sin 2\theta'), \quad (6)$$

where $(1-\delta)$ is the real part of the refractive index.

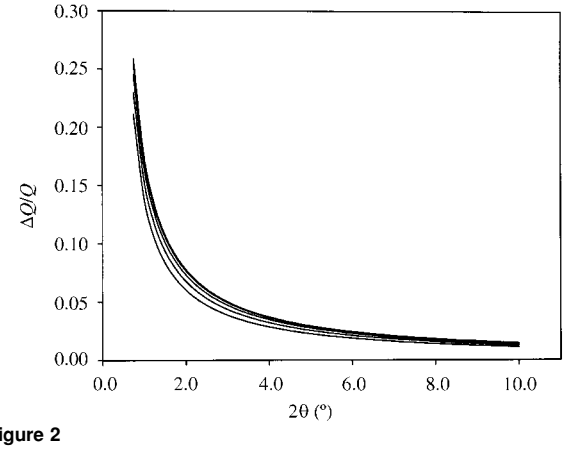

Figure 2
$\Delta Q/Q$, as a function of the detector angle 2θ, for the GISAXS parameters: $\alpha = 0.25^\circ$, $\delta = 7.7 \times 10^{-6}$, $\lambda = 1.49$ Å. The different curves, going downwards from the topmost, are relative to $\varphi = 0$, 10, 20, 30 and 40°.

In Fig. 2, the values of $\Delta Q/Q$ [$Q = (4\pi\sin\theta)/\lambda$, $Q' = (4\pi\sin\theta')/\lambda$, $\Delta Q = Q - Q'$] are drawn as a function of the detector angle 2θ, for the α, δ and λ values ($\alpha = 0.25^\circ$, $\delta = 7.7 \times 10^{-6}$, $\lambda = 1.49$ Å) of a previously reported experiment (Cattaruzza *et al.*, 2000). From inspection of Fig. 2, the $\Delta 2\theta$ correction is seen to be slightly dependent on φ and more effective at the smaller 2θ values; it is negative (that is, $2\theta' = 2\theta - |\Delta 2\theta|$) and decreases in absolute value with 2θ, whereas the correction concerning the azimuthal angle φ is positive, decreasing with 2θ and increasing with φ. This behaviour can be understood qualitatively by inspection of Fig. 1. The correction for refraction of the scattered beam can be regarded, on the detector plate, as a shift parallel to $-\mathbf{n}_{surf}$ of the intensity recorded at a given pixel. On a fixed scattering ring (that is, at constant 2θ) the shift at $\varphi = 0$ concerns… 2θ, whereas at increasing φ values it tends to become… the scattering ring, thus giving rise to incre… ng $|\Delta 2\theta|$ values.

From equ… sample of a sca… $\theta, \varphi)$ can … (7)

… for absorption of the … approximation $\varphi' \simeq \varphi$ (Kutsch *et*

3. Conclusions

The correction for refraction of the incoming beam and of the scattered beam at the sample surface in GISAXS patterns has been derived under the assumption of a point-like sample and for an experimental setup consisting of a planar detector perpendicular to the incoming beam. It has been demonstrated by the simulations reported in §3 that the smearing effects arising from the finite size of the illuminated sample are negligible in the small-angle scattering region. The parameters of the simulations are those of a GISAXS experiment previously carried out by the authors (Cattaruzza *et al.*, 2000), but for a shorter sample-to-detector distance, thus ensuring an overestimation of the sample smearing effects. Therefore, equations (5) and (6) can be directly applied to the experimental data to obtain the corrected intensity values.

References

Acapito, F. d', Thiaudière, D., Zontone, F. & Regnard, J. R. (1998). *Mater. Sci. Forum*, **278–281**, 891–896.
Babonneau, D., Naudon, A., Thiaudière, D. & Lequien, S. (1999). *J. Appl. Cryst.* **32**, 226–233.
Cattaruzza, E., d'Acapito, F., Gonella, F., Longo, A., Martorana, A., Mattei, G., Maurizio, C. & Thiaudière, D. (2000). *J. Appl. Cryst.* **33**, 740–743.
Guinier, A. & Fournet, G. (1955). *Small-Angle Scattering of X-rays*. New York: Wiley.
Kutsch, B., Lyon, O., Schmitt, M., Mennig, M. & Schmidt, H. (1997). *J. Appl. Cryst.* **30**, 948–956.
Levine, J. R., Cohen, J. B., Chung, Y. W. & Georgopoulos, P. (1989). *J. Appl. Cryst.* **22**, 528–532.
Naudon, A. & Thiaudière, D. (1997). *J. Appl. Cryst.* **30**, 822–827.
Pedersen, J. S. (1994). *J. Appl. Cryst.* **27**, 595–608.
Yoneda, Y. (1963). *Phys. Rev.* **131**, 2010–2013.

238 mm
88 mm
183 mm