The interpretation of broad diffuse maxima using superspace crystallography

Introducing disorder in superspace

Ella M. Schmidt and Reinhard B. Neder
Institute for Crystallography and Structural Physics, FAU Erlangen-Nürnberg
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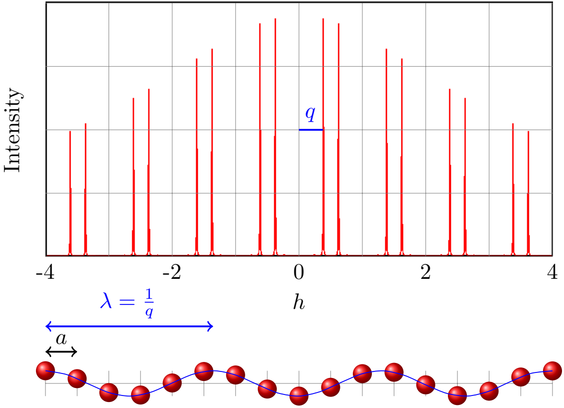
Diffraction pattern of a modulated structure



Bragg reflections: $h \in \mathbb{Z}$

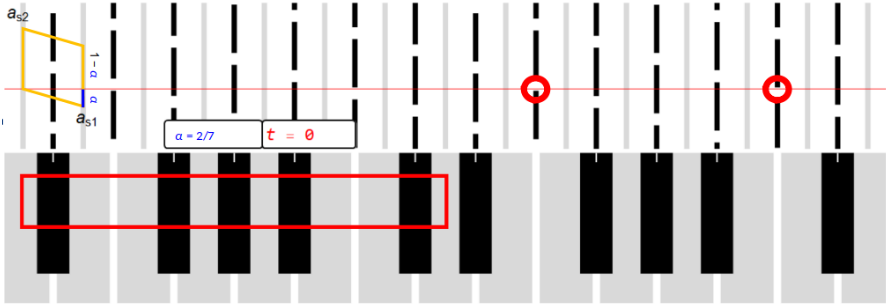
Satellite reflections: $h \pm mq \qquad h, m \in \mathbb{Z}$

Introduction to superspace crystallography

- Describe long-range ordered, modulated structure with few parameters
- Modulation wave vector $\boldsymbol{q} = (\alpha, \beta, \gamma)$

Paul B. Klar
FZU Prague

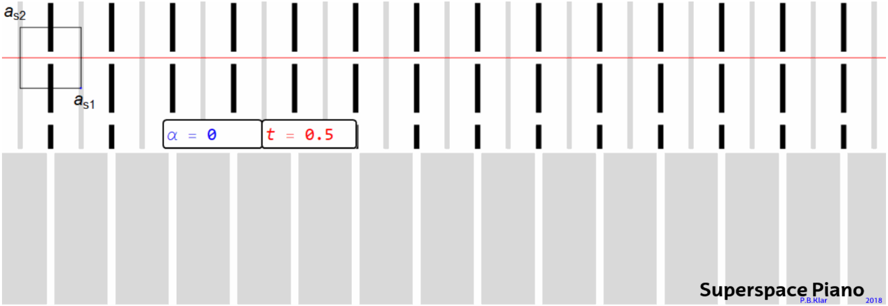
1-dimensional piano structure in two-dimensional superspace:



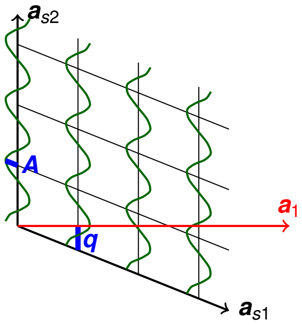
Introduction to superspace crystallography

Paul B. Klar
FZU Prague

Changing α $\leftrightarrow$ Changing real space periodicity



Introduction to superspace crystallography



Modulation function:

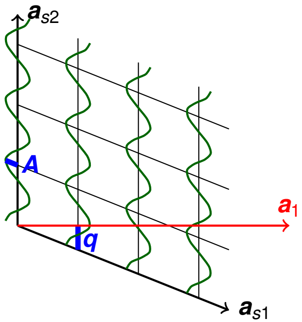
$u_{+}(\boldsymbol{a}) = \boldsymbol{A}\cos(2\pi \boldsymbol{q}\boldsymbol{a})$

- $\boldsymbol{q}$ determines periodicity of modulation function
- A determines the amplitude of the modulation function
- Modulation functions for occupancy and/or displacement
- Real space $\equiv$ cut through direct superspace
- Reciprocal space $\equiv$ projection of reciprocal superspace

[1] Van Smaalen, Sander. *Incommensurate crystallography*. Vol. 21. Oxford University Press, 2007.

[2] Janssen, Ted, Gervais Chapuis, and Marc De Boissieu. Aperiodic Crystals: *From Modulated Phases to Quasicrystals: Structure and Properties*. Oxford University Press, 2018.

Introduction to superspace crystallography



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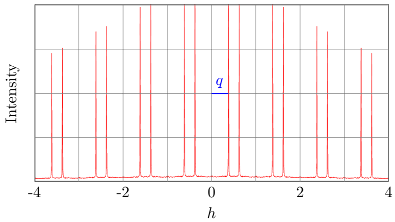

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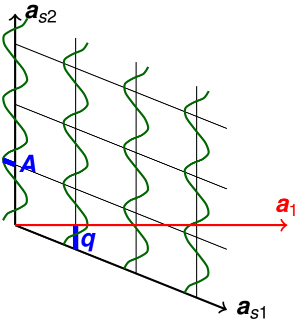
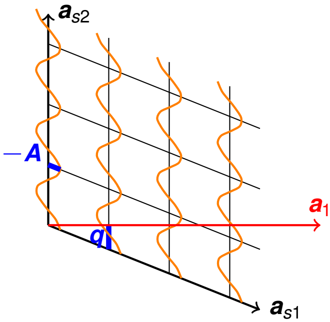
(1+1)D ordered superspace



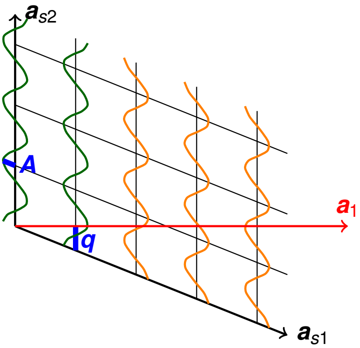


Modulation function:
$u_+(\boldsymbol{a}) = A\cos(2\pi \boldsymbol{q}\boldsymbol{a})$

Modulation function:
$u_-(\boldsymbol{a}) = -A\cos(2\pi \boldsymbol{q}\boldsymbol{a})$

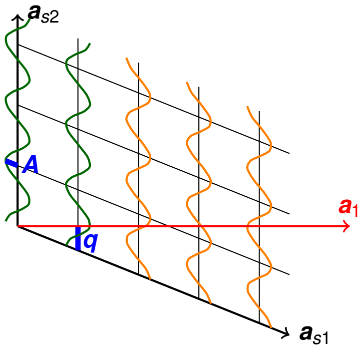
$\rightarrow$ Sharp satellite reflections at $h \pm q$.
$\rightarrow$ $u_+(\boldsymbol{a})$ and $u_-(\boldsymbol{a})$ cannot be distinguished experimentally.

(1+1)D disordered superspace



- Phase domains in superspace
- Positive correlation along $\boldsymbol{a}_{s1}$
- Warren-Cowley short range order parameter $\alpha_1^s > 0$

(1+1)D disordered superspace



- Phase domains in superspace
- Positive correlation along $\boldsymbol{a}_{s1}$
- Warren-Cowley short range order parameter $\alpha_1^s > 0$
- What happens to the sharp satellite reflections?

(1+1)D disordered superspace - Occupational disorder

- Au/Ag 1:1 crystal
- $q = \frac{\sqrt{7}}{7} \approx 0.378$
- Modulation functions:
  $p_+(a) = 0.5 + 0.5\cos(2\pi q a)$
  $p_-(a) = 0.5 - 0.5\cos(2\pi q a)$

- Create disordered super space structure with
  - positive correlation ($\alpha_1^s = 0.85$)
  - $m_{p_+} = m_{p_-} = 0.5$

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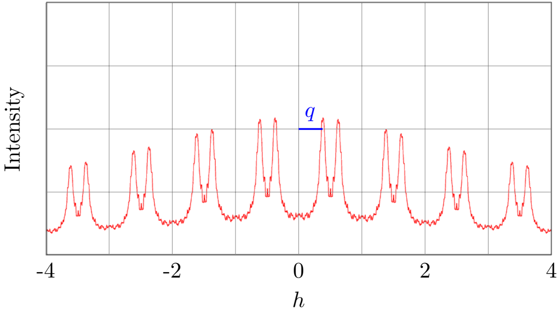

(1+1)D disordered superspace - Peak position tuning

1D occupational disorder

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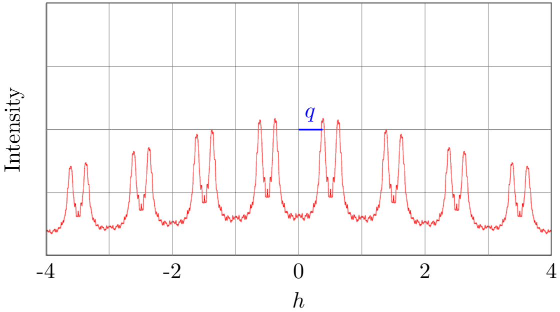
$q = \frac{\sqrt{7}}{7}$ $\alpha_1^s = 0.85$



(1+1)D disordered superspace - Peak width tuning

1D occupational disorder

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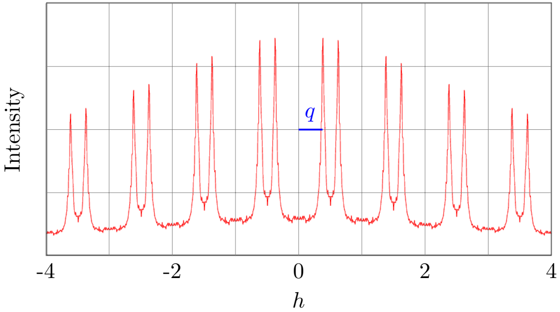
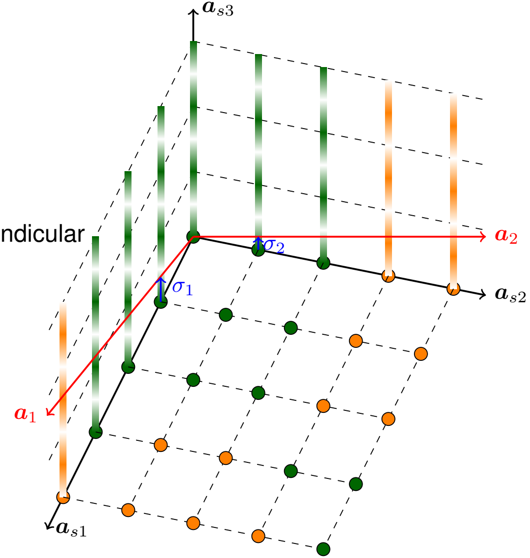
- $p_{-}(a) = 0.5 - 0.5\cos(2\pi q a)$

$q = \frac{\sqrt{7}}{7}$ $\alpha_1^s = 0.95$



(2+1)D superspace for occupational modulations

- 2D basic structure
- $\boldsymbol{q} = (\sigma_1, \sigma_2)$
- Internal dimension $\boldsymbol{a}_{s3}$ perpendicular to external, physical space spanned by $\boldsymbol{a}_1$ and $\boldsymbol{a}_2$



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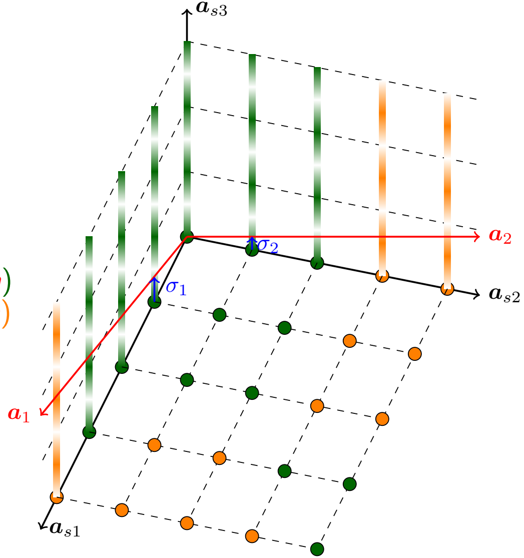

(2+1)D disordered superspace - Occupational disorder

- Au/Ag 1:1 crystal
- Plane space group *p*1
- One atom per unit-cell at $(0, 0)$

(2+1)D disordered superspace - Occupational disorder

- Au/Ag 1:1 crystal
- Plane space group $p1$
- One atom per unit-cell at $(0, 0)$
- $\boldsymbol{q} = \left(\frac{1}{2}, \frac{\sqrt{7}}{7}\right)$
- Modulation functions:

$$p_+(\boldsymbol{a}) = 0.5 + 0.5\cos(2\pi\boldsymbol{q}\boldsymbol{a})$$
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- Create disordered super space structure with
  - positive correlations along $\boldsymbol{a}_{s1}$ and $\boldsymbol{a}_{s2}$:
    $$\alpha^s_{(1,0)} = 0.7$$
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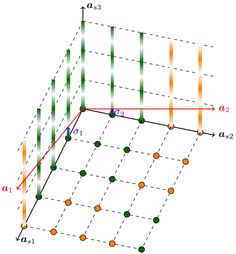
  - $m_{p_+} = m_{p_-} = 0.5$



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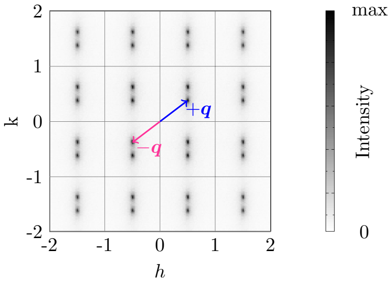

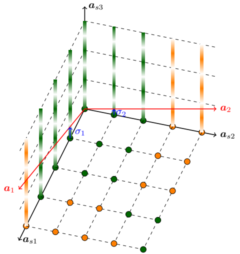

(2+1)D disordered superspace - Diffuse rods

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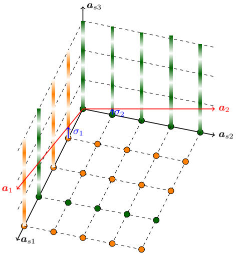

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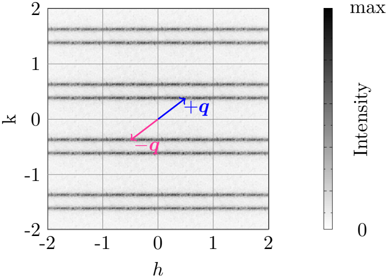

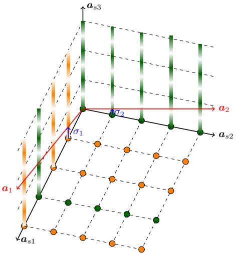

(2+1)D disordered superspace - Reflection condition

- Basic structure shows symmetry
- Superspace group $pg1(\frac{1}{2}, \beta)$
  Site A: $(+0.1, 0.0)$
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- Modulation functions:

$$\text{Site A: } p_A(\boldsymbol{a}_A)=0.5\pm0.5\cos(2\pi(+\sigma_1 a_{1A}+\sigma_2 a_{2A}))$$
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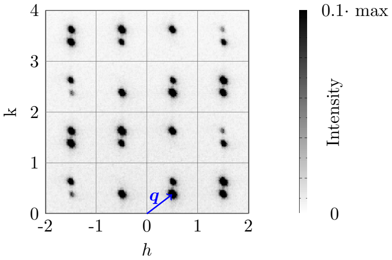

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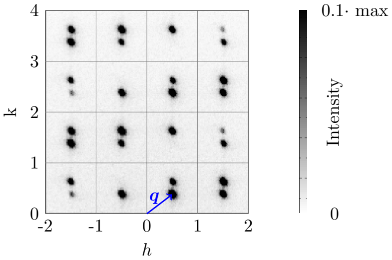

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$\rightarrow$ Diffuse satellites obey reflection condition as well

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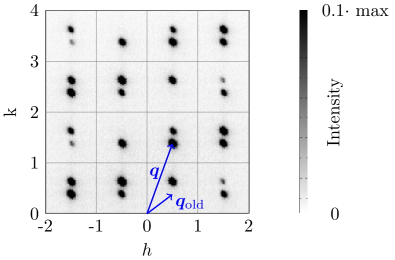

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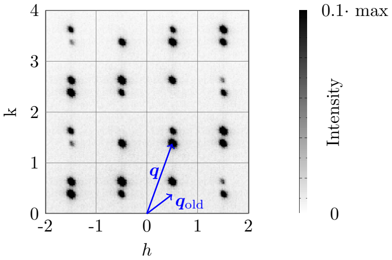

$\rightarrow$ Diffuse satellites obey reflection condition as well

$\rightarrow$ "Virtual" satellite reflection condition $0k: \quad k = 2n+1$

(2+1)D disordered superspace - Reflection condition

- Basic structure shows symmetry
- Superspace group $pm1(0,\beta)s0$ with internal translation
  Site A: $(+0.1, 0.0)$
  Site B: $(-0.1, 0.0)$
- Satellite-reflection condition[a]:
  $hk: \quad h \neq 0$
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[a] For occupational modulation functions with only one cosine-term.

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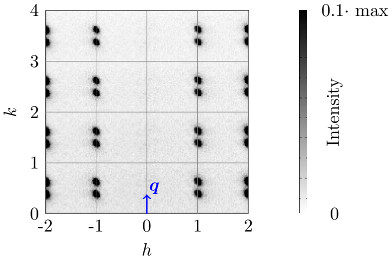

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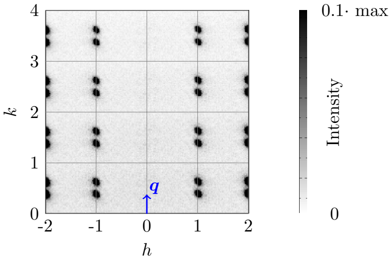

[a] For occupational modulation functions with only one cosine-term.

Possible applications

Diffuse rods showing 'extinction conditions'

research papers

Acta Crystallographica Section B
Structural Science
ISSN 0108-7681

Symmetry analysis of extinction rules in diffuse-scattering experiments

R. L. Withers,[a] M. I. Aroyo,[b]* J. M. Perez-Mato[b] and D. Orobengoa[b]

Structured diffuse-scattering intensities, whether of compositional or of pure displacive origin, static or dynamic, contain important information about the symmetry of the individual compositional and/or displacive modes responsible for the observed intensities. However, the interpretation of the

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research papers

IUCrJ
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PHYSICS | FELS

Exploiting superspace to clarify vacancy and Al/Si ordering in mullite

Paul Benjamin Klar,[a]* Iñigo Etxebarria[b] and Gotzon Madariaga[a]

[a]Departamento de Física de la Materia Condensada, Facultad de Ciencia y Tecnología, Universidad del País Vasco UPV/EHU, Apartado 644, Bilbao 48080, Spain, and [b]Departamento de Física Aplicada II, Facultad de Ciencia y Tecnología, Universidad del País Vasco UPV/EHU, Apartado 644, Bilbao 48080, Spain. *Correspondence e-mail: paulbenjamin.klar@ehu.eus

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Possible applications

Diffuse rods showing 'extinction conditions'

research papers

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Symmetry analysis of extinction rules in diffuse-scattering experiments

R. L. Withers,[a] M. I. Aroyo,[b]* J. M. Perez-Mato[b] and D. Orobengoa[b]

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Phase transitions: Diffuse scattering turns into sharp satellite reflections

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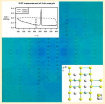
The Mystery of the AuIn 1:1 Phase and Its Incommensurate Structural Variations

Laura C. Folkers,*,[†] Arkadiy Simonov,[‡] Fei Wang,[§] and Sven Lidin[†]

[†]Centre for Analysis and Synthesis, Lunds Universitet, Naturvetarvägen 14, Lund 222-61, Sweden
[‡]Inorganic Chemistry Laboratory, University of Oxford, South Parks Road, Oxford OX1 3QR, United Kingdom
[§]Department of Chemistry, Missouri State University, 901 S. National Avenue, Springfield, Missouri 65897, United States

Supporting Information

ABSTRACT: In this communication, the AuIn 1:1 phase (*Naturwissenschaften*, **1953**, *40*, 437, DOI: 10.1007/BF00590353), and its ordering behavior at various temperatures is investigated. To enable the growth of a X-ray suitable specimen, a tempering routine was established by the interpretation of a differential scanning calorimetry (DSC) study. In this way, good quality single crystals were grown and measured at the Crystal beamline at Synchrotron SOLEIL. From the acquired data, three variations of this structure could be found at temperatures of 400 °C and 300 °C and room temperature, with differing degrees of incommensurate modulation. Diffuse scattering found at 400 °C was interpreted with the help of a three-dimensional difference pair distribution function (3D-ΔPDF) study.



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- Unified model for diffuse maxima at different positions in reciprocal space
- Superspace symmetry allows direct access to extinction rules

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- Expansion to $(3 + d)$D

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- Superspace symmetry allows direct access to extinction rules

Outlook

- Application to different disordered systems
- Expansion to $(3+d)$D
- Possible peak shape tuning by introducing size-effect like relaxations?
- Possibility of curved diffuse features?

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