Friedrich-Alexander-Universität
Naturwissenschaftliche Fakultät

FAU

DISCUS Workshop
Powder Diffraction

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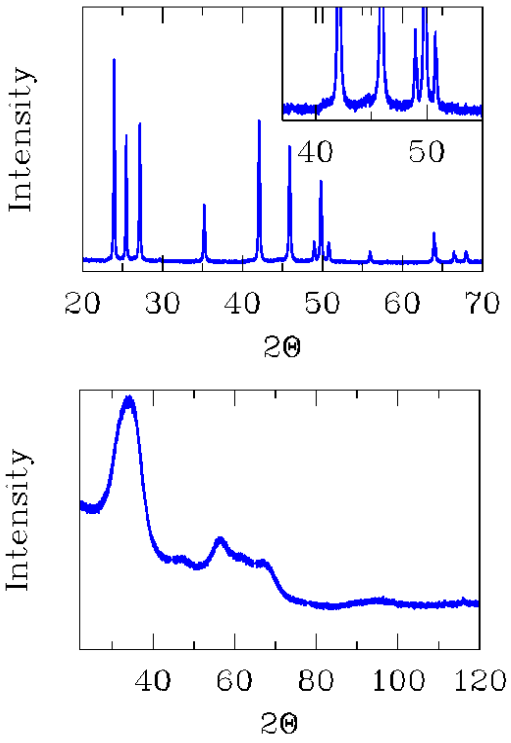
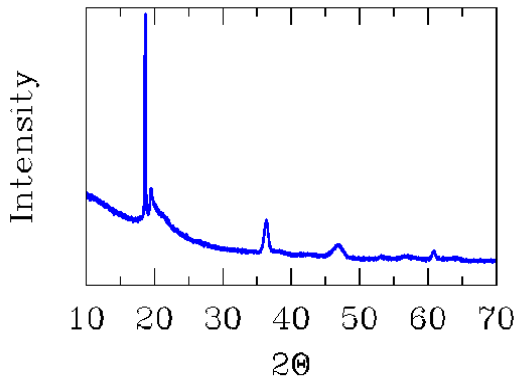
Powder diffraction pattern

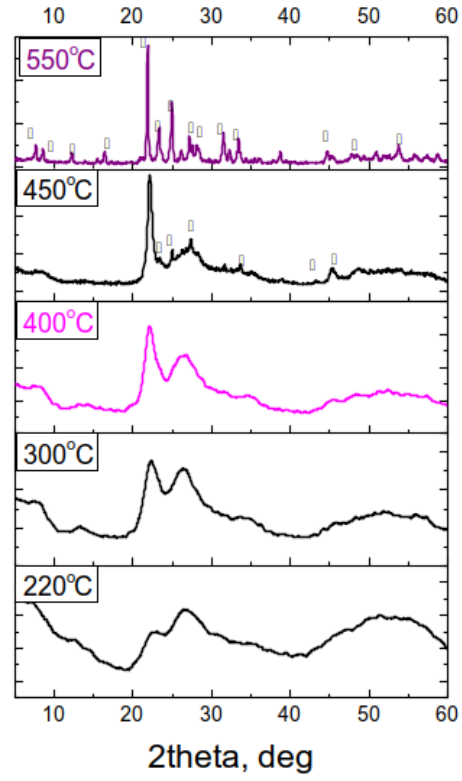
Crystalline material



Nano crystalline ZnO



Massive stacking faults
H_2TiO_3



Mo-V-Nb Oxide
Steps during synthesis

Calculating a Powder diffraction pattern

Rietveld

Complete Integration

Debye-Scattering-Equation

Calculating a Powder diffraction pattern

Rietveld

Complete Integration

Debye-Scattering-Equation

Sum over all integer Bragg reflections

Convolution by profile function

Repeat for all phases

Addition of a background

Calculating a Powder diffraction pattern

Rietveld

Complete Integration

Debye-Scattering-Equation

Sum over **a fine 3D-Grid in reciprocal space**

Convolution by profile function

Repeat for all phases

Addition of a background

Calculating a Powder diffraction pattern

Rietveld

Perform a spherical average of Intensity expression

Complete Integration

$$F(hkl) = \sum_{j=1}^{N} f_j e^{2\pi i(h x_j + k y_j + l z_j)}$$

Debye-Scattering-Equation

$$I(hkl) = F(hkl) * F^*(hkl)$$

$$\langle |F(|\boldsymbol{h}|)|^2 \rangle = \sum_i \sum_j f_i f_j \frac{\sin(2\pi|\boldsymbol{h}|\boldsymbol{r_{ij}})}{(2\pi|\boldsymbol{h}|r_{ij})}$$

Convolution by profile function

Repeat for all phases

Addition of a background

Calculating a powder diffraction pattern

Open in Windows Explorer: Lectures\
03_Diffraction\

Start DISCUS_SUITE

You should see:

...
User macros in ...
System macros in ...
Start directory ...

suite >

suite > cd Lectures\03_Diffraction

suite > **@complete.mac nickel**

Try: silicon; lab6; kaolinite

POWDER calculate a powder diffraction pattern

Main steps

Radiation:	**xray**;	**neutron**;	**electron**
Wavelength:	**set wvle**;	**set energy**	
Range:	**set tthmin**;	**set tthmax**;	**set dtth**
Algorithm:	**set calc, complete**;	**set calc, debye**	
Profile:	**set profile, pseudo**;	**set profile, off**	
Debye-Waller:	**set temp, use**;	**set temp, ignore**	
LP correction:	**set lpcor, bragg, angle**		

For complete algorithm

Steps in rec.: **set dh**; **set dk**; **set dl**

POWDER calculate a powder diffraction pattern

complete.mac

```
powder
reset      ! Ensure clean start up conditions
neutron                  # Select radiation
set wvle,CU12            # Set the wavelength
set axis,Q               # Perform calculation on equaly spaced Q grid
set calc,complete        # Use complete integration algorithm
```

POWDER calculate a powder diffraction pattern

complete.mac

```
powder
reset      ! Ensure clean start up conditions
neutron                  # Select radiation
set wvle,CU12            # Set the wavelength
set axis,Q               # Perform calculation on equaly spaced Q grid
set calc,complete        # Use complete integration algorithm
#
set dh, 1.0              # Steps in reciprocal space,
set dk, 1.0              # here integer for Bragg only
set dl, 1.0              #
```

POWDER calculate a powder diffraction pattern

complete.mac

```
powder
reset     ! Ensure clean start up conditions
neutron                  # Select radiation
set wvle,CU12            # Set the wavelength
set axis,Q               # Perform calculation on equaly spaced Q grid
set calc,complete        # Use complete integration algorithm
#
set dh, 1.0              # Steps in reciprocal space,
set dk, 1.0              # here integer for Bragg only
set dl, 1.0              #
#
set tthmin, 1.0          # Start/end/step angle in degrees
set tthmax, 90.          # Adapt to experiment
set dtth  , 0.005        # Or: qmin; qmax; dq
```

POWDER calculate a powder diffraction pattern

complete.mac

```
powder
#
set profile, off        # Switch convolution by profile function off
set profile, pseudo     # Use Pseudovoigt (or Pearson VII, or TOF)
set profile, uvw, 0.000, 0.000, 0.005 # Cagliotti u,v,w values
set profile, eta, 0.500   # Mixing parameter 1=Lorenzian 0=Gaussian
set profile, asym, 0.00, 0.00, 0.00, 0.00 #Asymmetry parameters
```

Calculating a powder diffraction pattern

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Start directory ...

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Modify:

Radiation; Wavelength
2Theta range
Profile parameters

Try: silicon; lab6; kaolinite

Debye Scattering Equation

Double sum over all atom pairs

$$\langle |F(|\boldsymbol{h}|)|^2 \rangle = \sum_i \sum_j f_i f_j \frac{\sin(2\pi|\boldsymbol{h}|\boldsymbol{r_{ij}})}{(2\pi|\boldsymbol{h}|r_{ij})}$$

No constraints on:

periodicity; defects; shape

Calculating a powder diffraction pattern

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03_Diffraction\

Start DISCUS_SUITE

You should see:

...
User macros in ...
System macros in ...
Start directory ...

suite >

suite > **cd Lectures\03_Diffraction**

suite > **@debye.mac 10**

Try: different sizes
WARNING:
TIME = SIZE^6

Calculating a powder diffraction pattern

Open in Windows Explorer: Lectures\
03_Diffraction\

Start DISCUS_SUITE

You should see:

...
User macros in ...
System macros in ...
Start directory ...

suite >

suite > **cd Lectures\03_Diffraction**

suite > **@debye.mac 10**

Modify:

Different materials (fixed or as parameter)
Different shapes

Try: different sizes
WARNING:
TIME = SIZE^6