Dajust/sgaid example

Powder diffraction data at high pressure

inorganic material

Inorganic Chemistry

Article

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Structural Phase Transitions on AgCuS Stromeyerite Mineral under Compression

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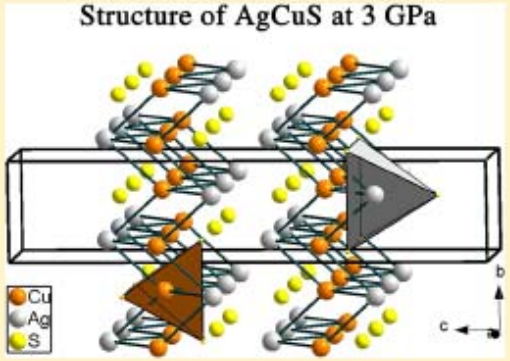
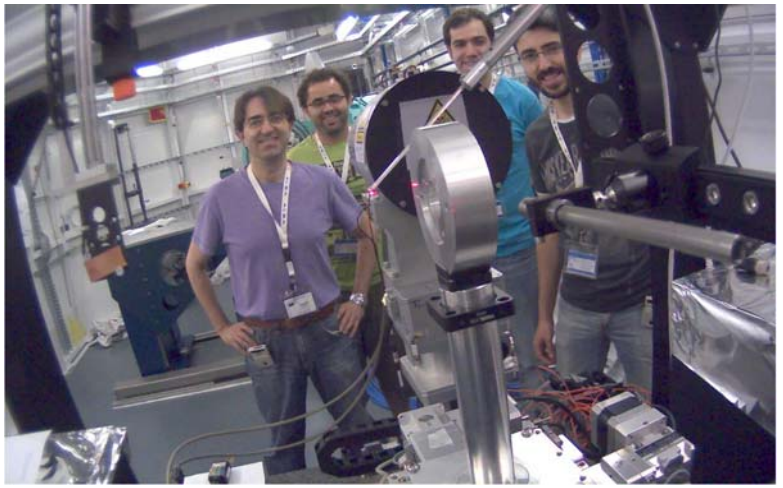
ABSTRACT: The structural behavior of mineral Stromeyerite, AgCuS, has been studied by means of angle-dispersive X-ray diffraction measurements up to 13 GPa and ab initio total-energy calculations. Two high-pressure phase transitions are found at 1.4 and 5.7 GPa, from the initial distorted Ni_2In-type phase (AuRbS-type, RP, space group $Cmc2_1$) through an anti-PbClF-type phase (HP1, space group $P4/nmm$) to a monoclinic distortion of this latter phase (HP2, space group $P2_1/m$). The collapse of the metal−metal interatomic distances at the RP−HP1 transition suggests a stronger metallic behavior of the high-pressure phase. The compressibility of the lattice parameters and the equation of state of the first pressure-induced phase have been experimentally determined. First-principles calculations present an overall agreement with the experimental results in terms of the high-pressure sequence and provide chemical insight into the AgCuS behavior under hydrostatic pressure.



First publication with Alba data

dx.doi.org/10.1021/ic302116b | Inorg. Chem. 2013, 52, 355−361

Summary of the experiment at bl04
Powder diffraction data vs. pressure



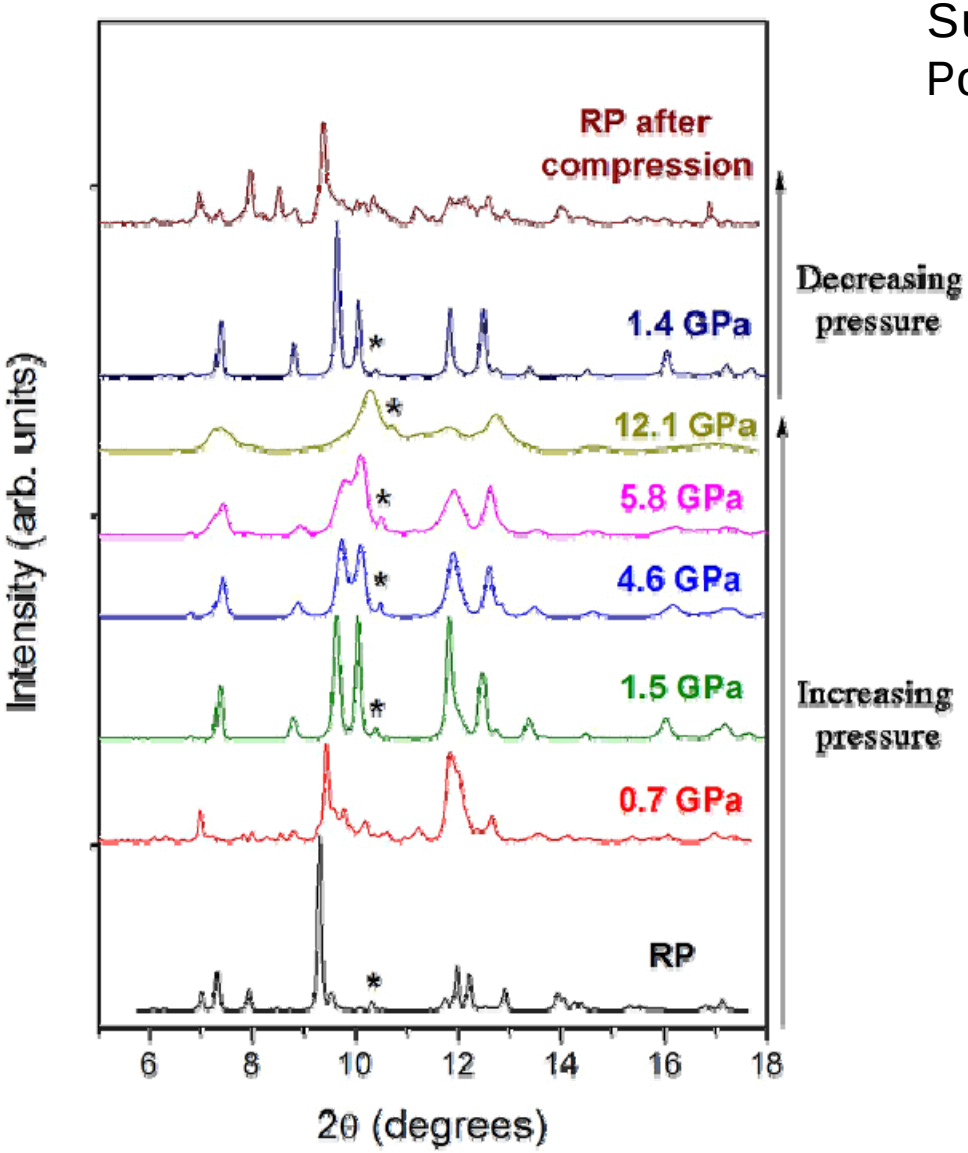


Figure 2. Powder XRD patterns of AgCuS at different pressure conditions. Upstroke and downstroke processes are indicated on the right-hand side. Asterisks denote the diffraction maxima of silver.

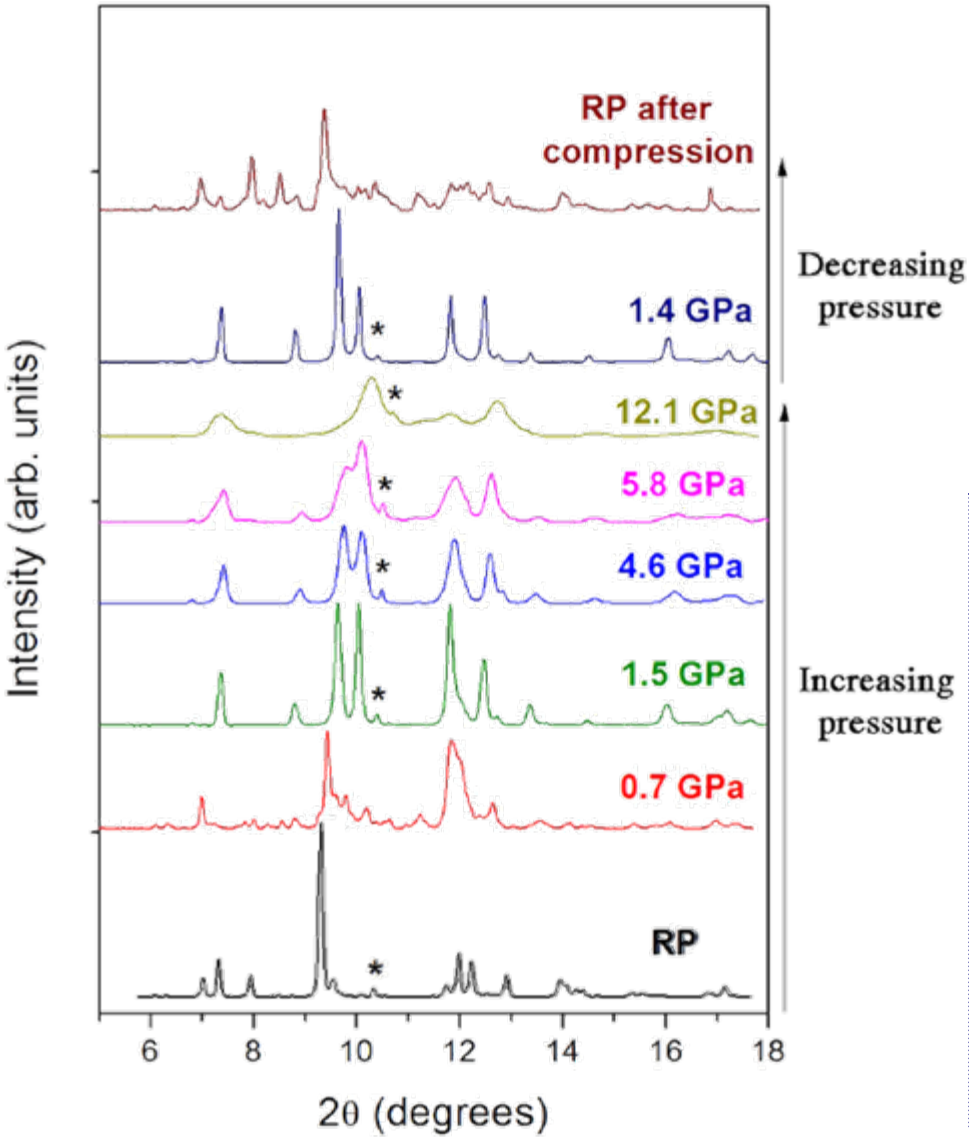


Figure 2. Powder XRD patterns of AgCuS at different pressure conditions. Upstroke and downstroke processes are indicated on the right-hand side. Asterisks denote the diffraction maxima of silver.

Starting point: room temperature phase
known structure

Room Pressure Phase of Stromeyerite, AgCuS
Cmc21, Z=4, a=4.06Å, b=6.64 Å anc c=7.97Å

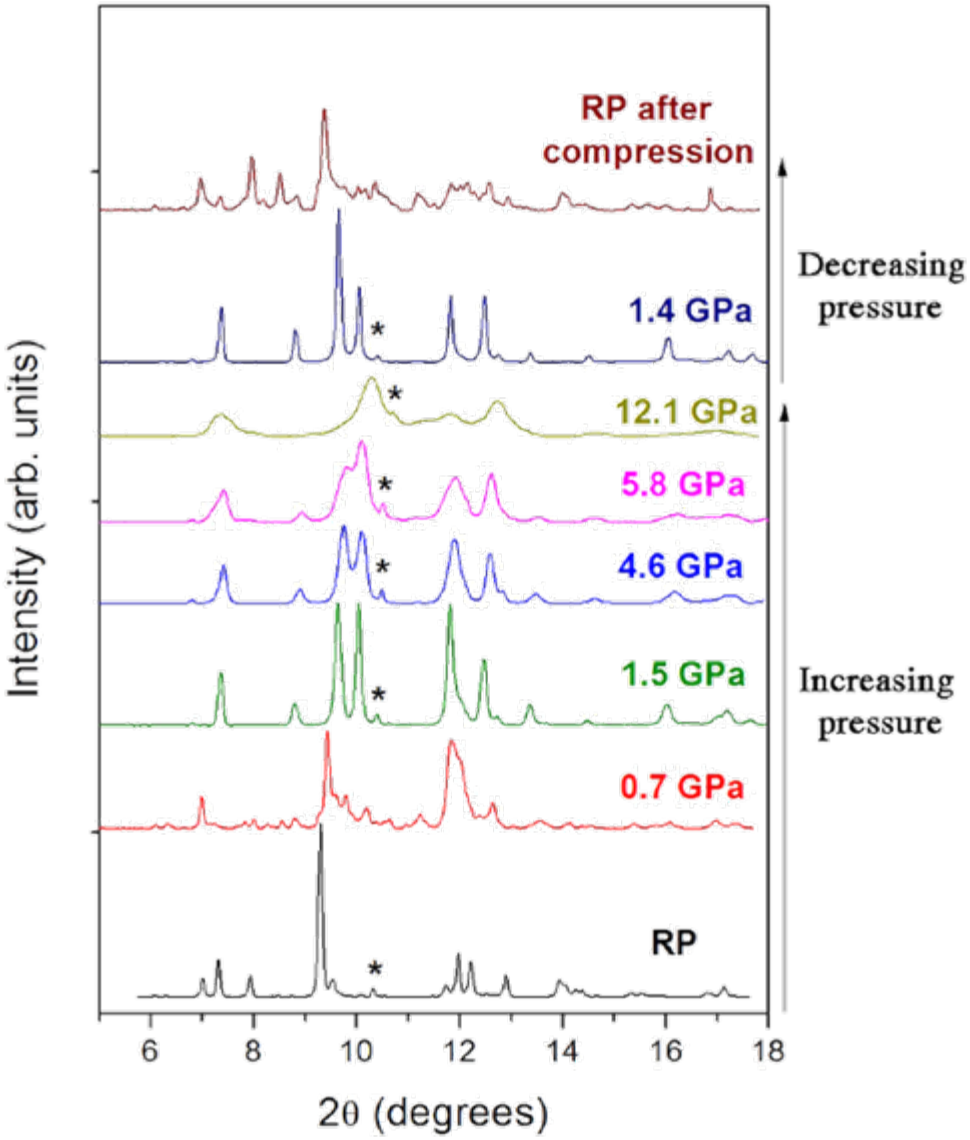
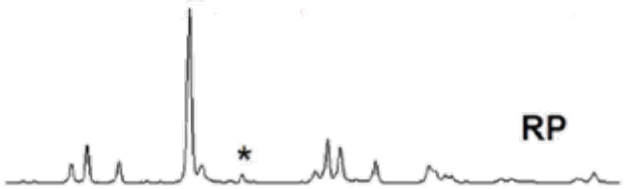


Figure 2. Powder XRD patterns of AgCuS at different pressure conditions. Upstroke and downstroke processes are indicated on the right-hand side. Asterisks denote the diffraction maxima of silver.

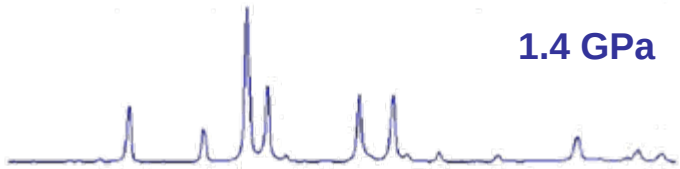


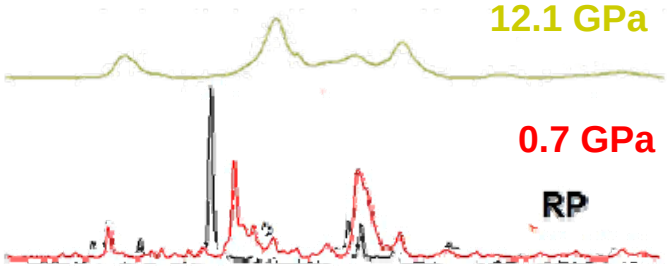
Room Pressure Phase of Stromeyerite, AgCuS

Cmc21, Z=4, a=4.06Å, b=6.64 Å anc c=7.97Å

P=1.4 GPa
(when decreasing the pressure)

The peaks can be indexed with
Space group P4/nmm a=3.90Å, c=18.52 Å

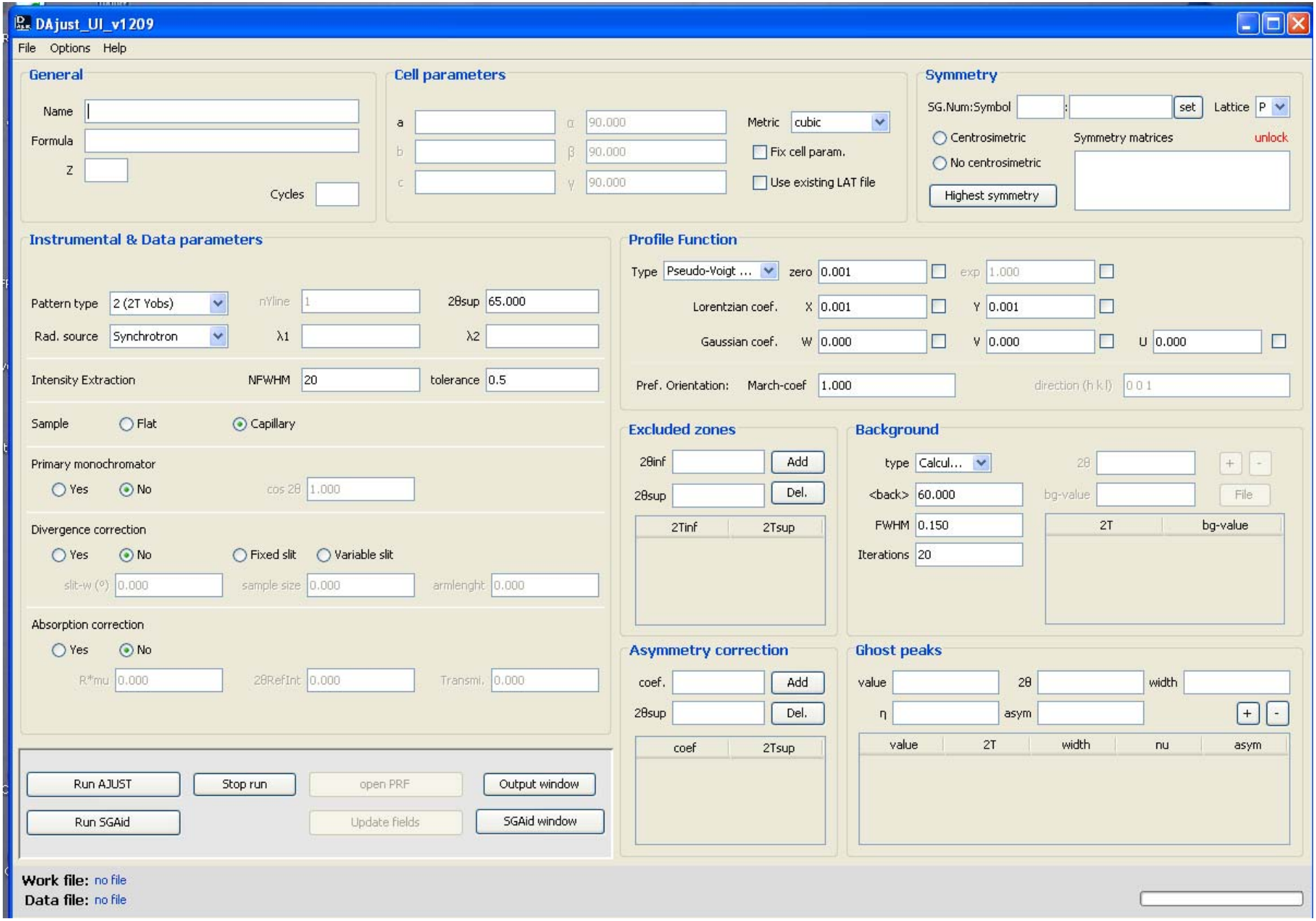




Room Pressure Phase of Stromeyerite, AgCuS
Cmc21, Z=4, a=4.06Å, b=6.64 Å anc c=7.97Å

Strategies to index the new phase

- Group subgroup relationships (*Bilbao crystallographic server*)
- Ab initio calculations
- Already known phases of the same family of compounds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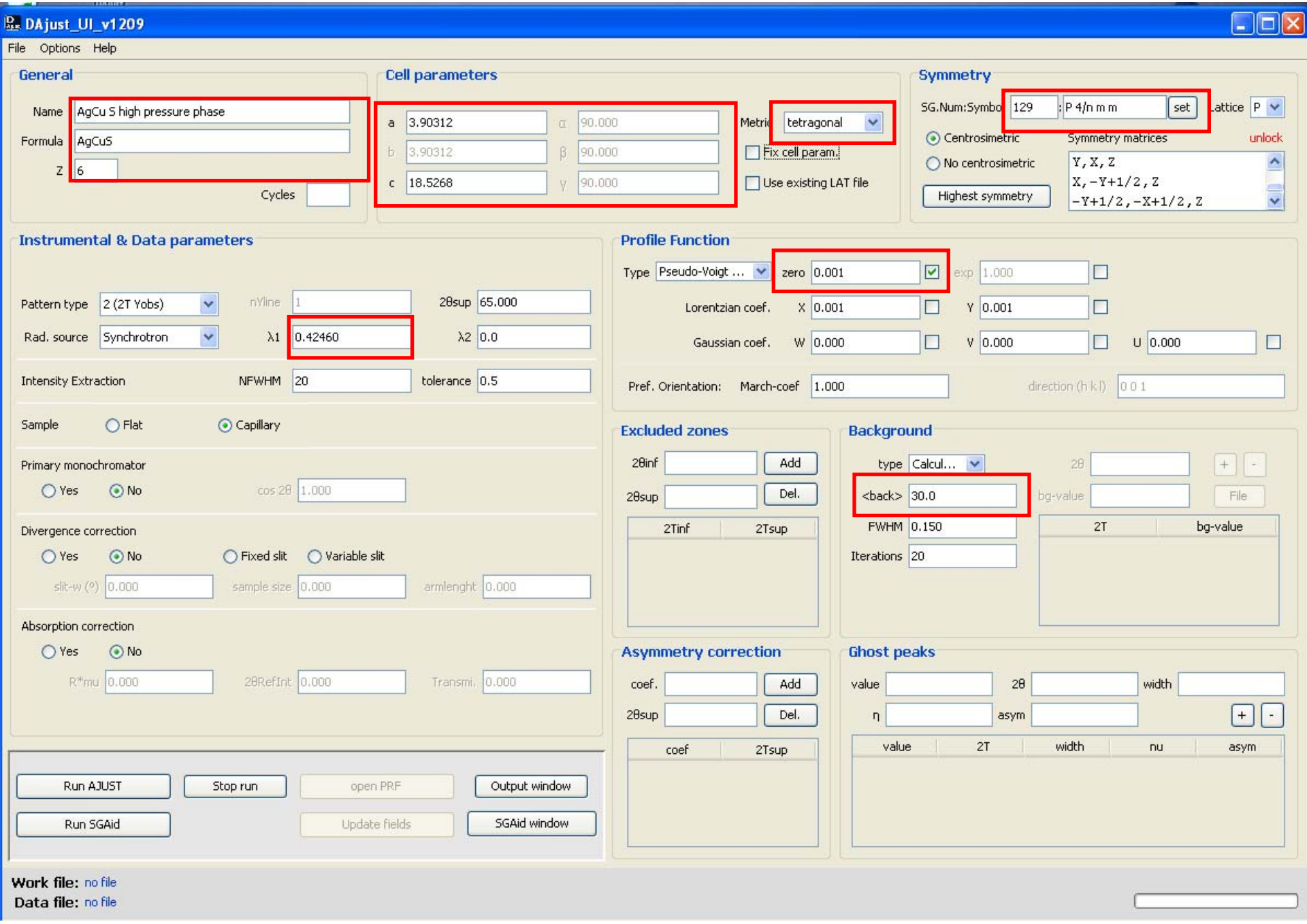
Name
Formula
Z
Cycles
Cell parameters
a b c
α β γ
90.000
Metric cubic
Fix cell param.
Use existing LAT file
Symmetry
SG.Num:Symbol
set
Lattice P
Centrosimetric
No centrosimetric
Highest symmetry
Symmetry matrices
unlock
Instrumental & Data parameters
Pattern type 2 (2T Yobs)
nYline 1
2θsup 65.000
Rad. source Synchrotron
λ1
λ2
Intensity Extraction
NFWHM 20
tolerance 0.5
Sample Flat Capillary
Primary monochromator
Yes No
cos 2θ 1.000
Divergence correction
Yes No Fixed slit Variable slit
slit-w (°) 0.000
sample size 0.000
armlenght 0.000
Absorption correction
Yes No
R*mu 0.000
2θRefInt 0.000
Transmi. 0.000
Profile Function
Type Pseudo-Voigt ...
zero 0.001
exp 1.000
Lorentzian coef. X 0.001 Y 0.001
Gaussian coef. W 0.000 V 0.000 U 0.000
Pref. Orientation: March-coef 1.000
direction (h k l) 0 0 1
Excluded zones
2θinf
2θsup
Add
Del.
2Tinf 2Tsup
Background
type Calcul...
<back> 60.000
FWHM 0.150
Iterations 20
2θ
bg-value
File
2T bg-value
Asymmetry correction
coef.
2θsup
Add
Del.
coef 2Tsup
Ghost peaks
value 2θ width
η asym
value 2T width nu asym
Run AJUST
Stop run
open PRF
Output window
Run SGAid
Update fields
SGAid window
Work file: no file
Data file: no file


DAjust_UI_v1209
File Options Help
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Formula AgCuS
Z 6
Cycles
Cell parameters
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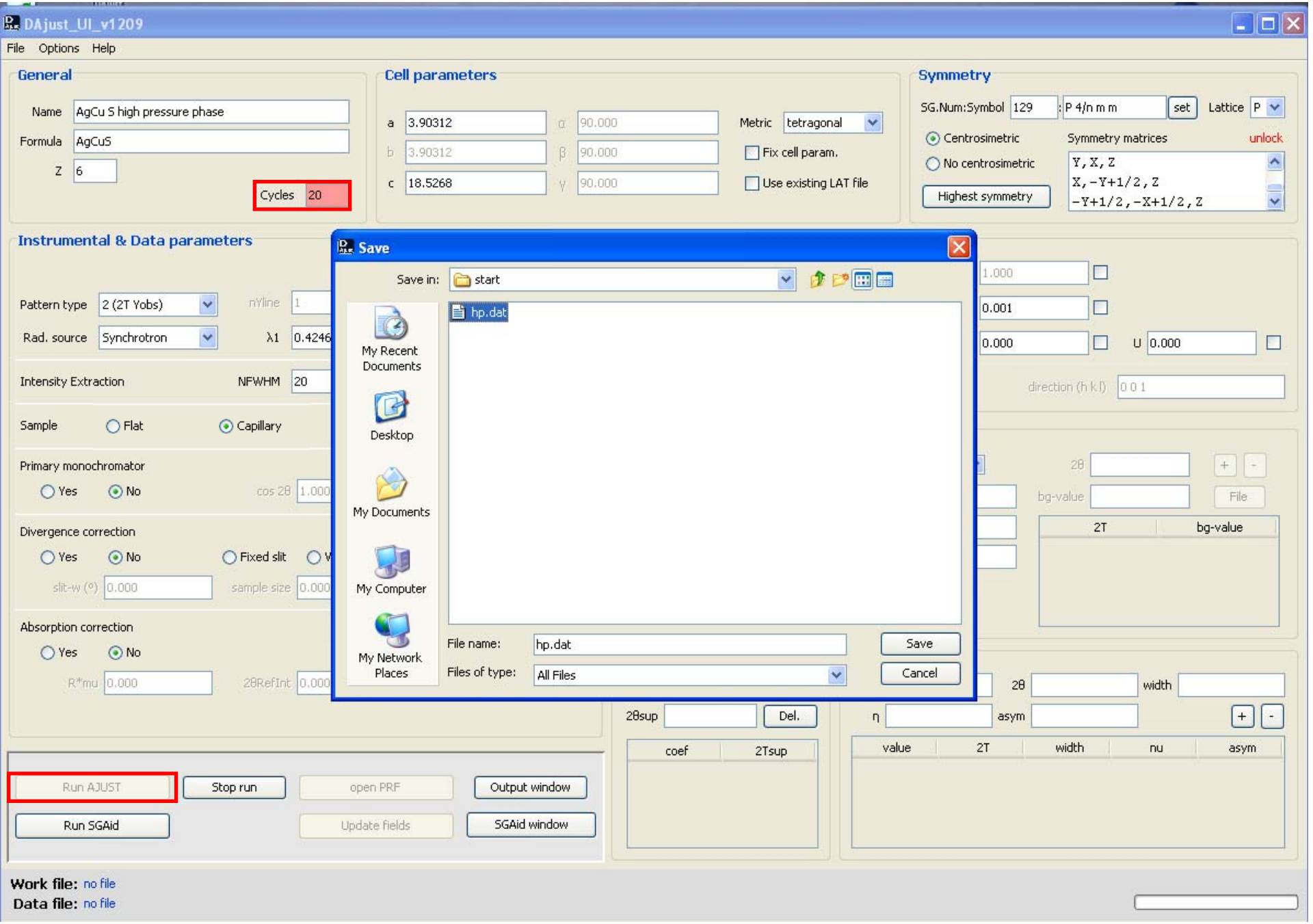
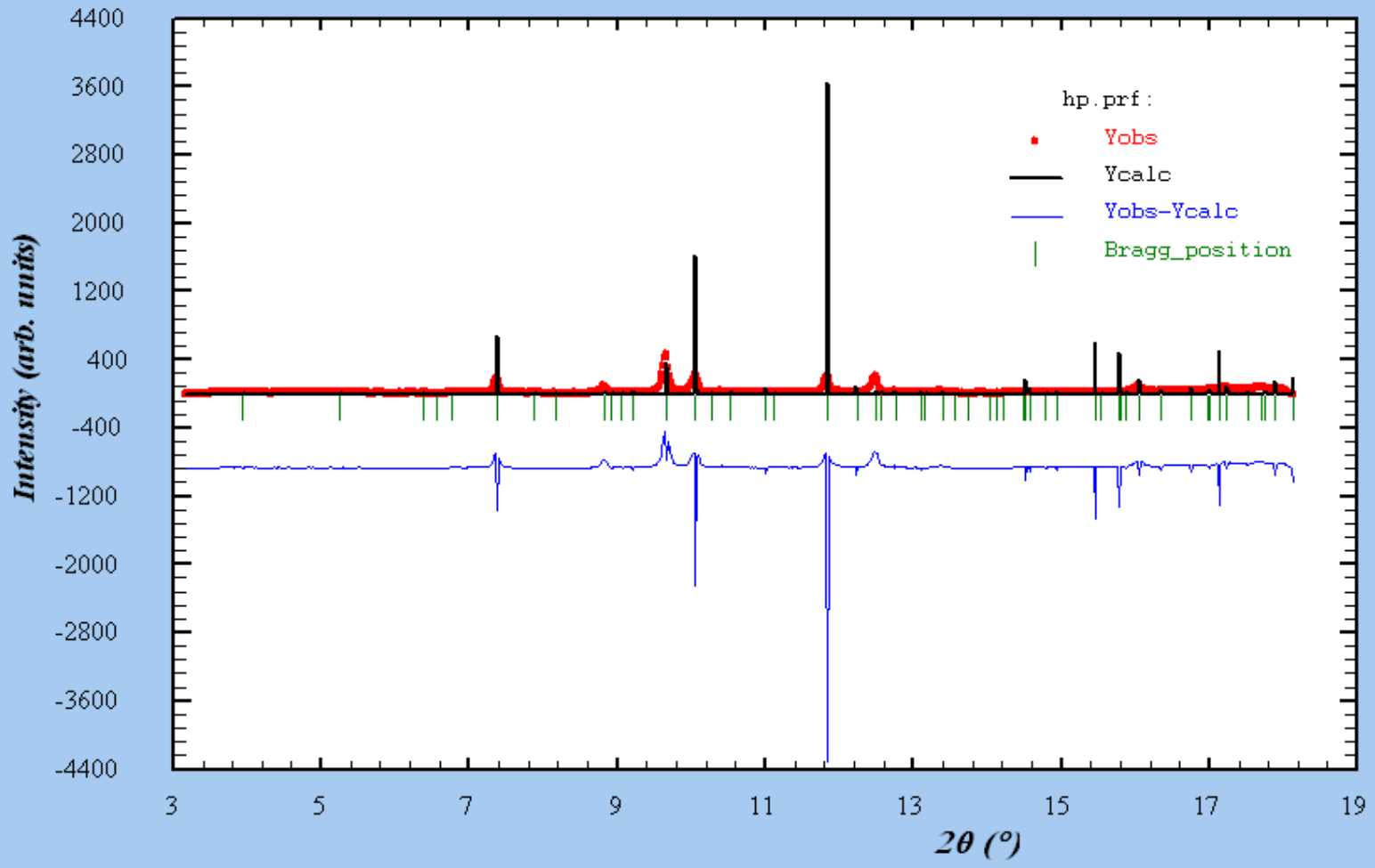
b 3.90312 β 90.000
c 18.5268 γ 90.000
Metric tetragonal
Fix cell param.
Use existing LAT file
Symmetry
SG.Num:Symbo 129 : P 4/n m m set
Lattice P
Centrosimetric
No centrosimetric
Highest symmetry
Symmetry matrices
unlock
Y,X,Z
X,-Y+1/2,Z
-Y+1/2,-X+1/2,Z
Instrumental & Data parameters
Pattern type 2 (2T Yobs)
nYline 1
2θsup 65.000
Rad. source Synchrotron
λ1 0.42460
λ2 0.0
Intensity Extraction
NFWHM 20
tolerance 0.5
Sample Flat Capillary
Primary monochromator
Yes No
cos 2θ 1.000
Divergence correction
Yes No Fixed slit Variable slit
slit-w (°) 0.000
sample size 0.000
armlenght 0.000
Absorption correction
Yes No
R*mu 0.000
2θRefInt 0.000
Transmi. 0.000
Profile Function
Type Pseudo-Voigt ...
zero 0.001
exp 1.000
Lorentzian coef. X 0.001 Y 0.001
Gaussian coef. W 0.000 V 0.000 U 0.000
Pref. Orientation: March-coef 1.000
direction (h k l) 0 0 1
Excluded zones
2θinf
2θsup
Add
Del.
2Tinf 2Tsup
Background
type Calcul...
<back> 30.0
FWHM 0.150
Iterations 20
2θ
bg-value
File
2T bg-value
Asymmetry correction
coef.
2θsup
Add
Del.
coef 2Tsup
Ghost peaks
value 2θ width
η asym
value 2T width nu asym
Run AJUST
Stop run
open PRF
Output window
Run SGAid
Update fields
SGAid window
Work file: no file
Data file: no file

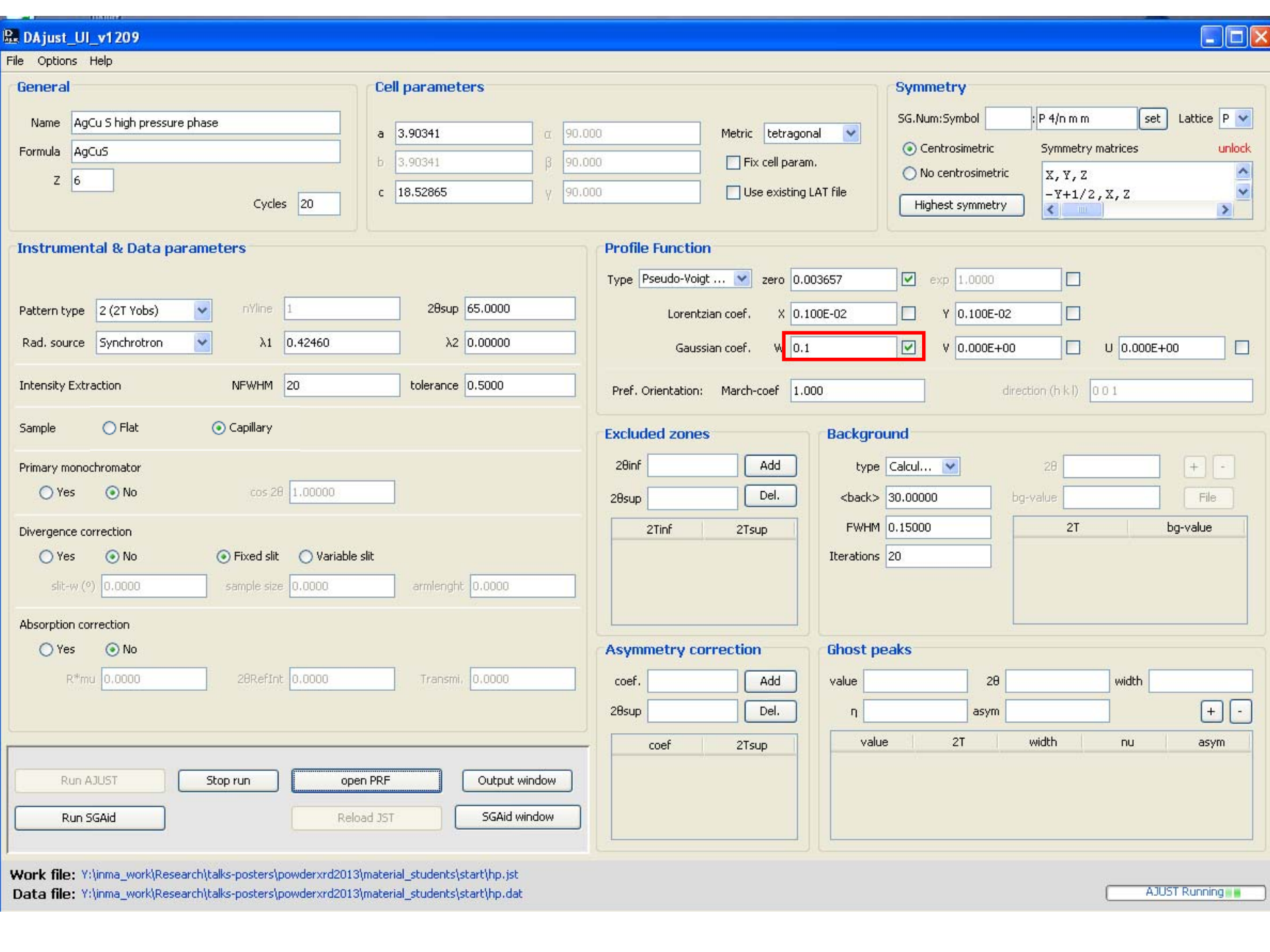

DAjust_UI_v1209
File Options Help
General
Name AgCu S high pressure phase
Formula AgCuS
Z 6
Cycles 20
Cell parameters
a 3.90312
b 3.90312
c 18.5268
α 90.000
β 90.000
γ 90.000
Metric tetragonal
Fix cell param.
Use existing LAT file
Symmetry
SG.Num:Symbol 129 : P 4/n m m
set
Lattice P
Centrosimetric
No centrosimetric
Highest symmetry
Symmetry matrices
unlock
Y,X,Z
X,-Y+1/2,Z
-Y+1/2,-X+1/2,Z
Instrumental & Data parameters
Pattern type 2 (2T Yobs)
nYline 1
Rad. source Synchrotron
λ1 0.4246
Intensity Extraction
NFWHM 20
Sample Flat Capillary
Primary monochromator
Yes No
cos 2θ 1.000
Divergence correction
Yes No Fixed slit
slit-w (°) 0.000
sample size 0.000
Absorption correction
Yes No
R*mu 0.000
2θRefInt 0.000
Save
Save in: start
hp.dat
My Recent Documents
Desktop
My Documents
My Computer
My Network Places
File name: hp.dat
Files of type: All Files
Save
Cancel
1.000
0.001
0.000
U 0.000
direction (h k l) 0 0 1
2θ
bg-value
File
2T
bg-value
2θsup
Del.
coef
2Tsup
η
asym
2θ
width
value
2T
width
nu
asym
Run AJUST
Stop run
open PRF
Output window
Run SGAid
Update fields
SGAid window
Work file: no file
Data file: no file

WinPLOTR [CDIFX UMR6226 Rennes / ILL Grenoble]

File Plot Options Points Selection X space Calculations Rietveld plot options Text External applications Tools Help



03-14-2013 16:41


DAjust_UI_v1209
File Options Help
General
Name AgCu S high pressure phase
Formula AgCuS
Z 6
Cycles 20
Cell parameters
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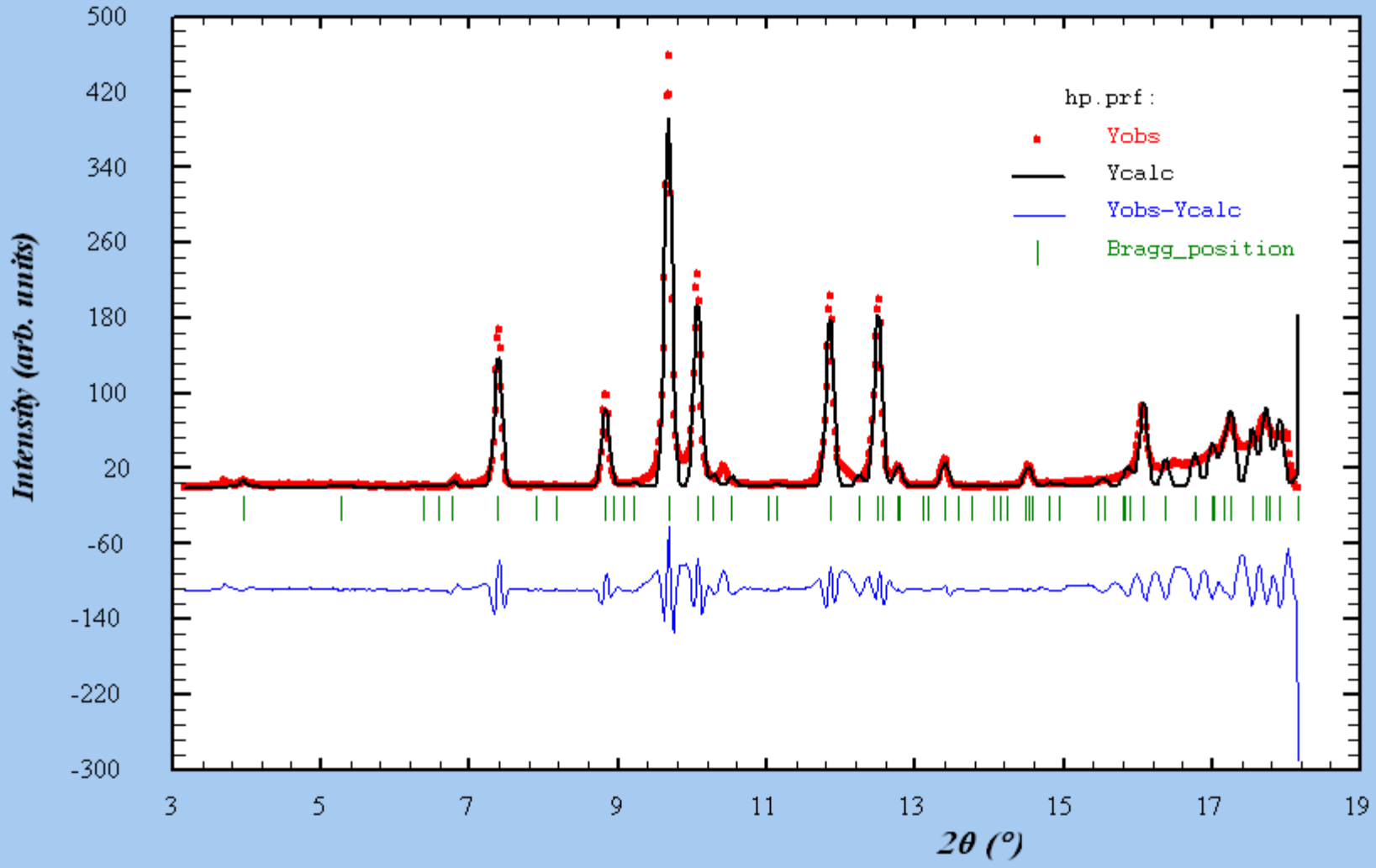
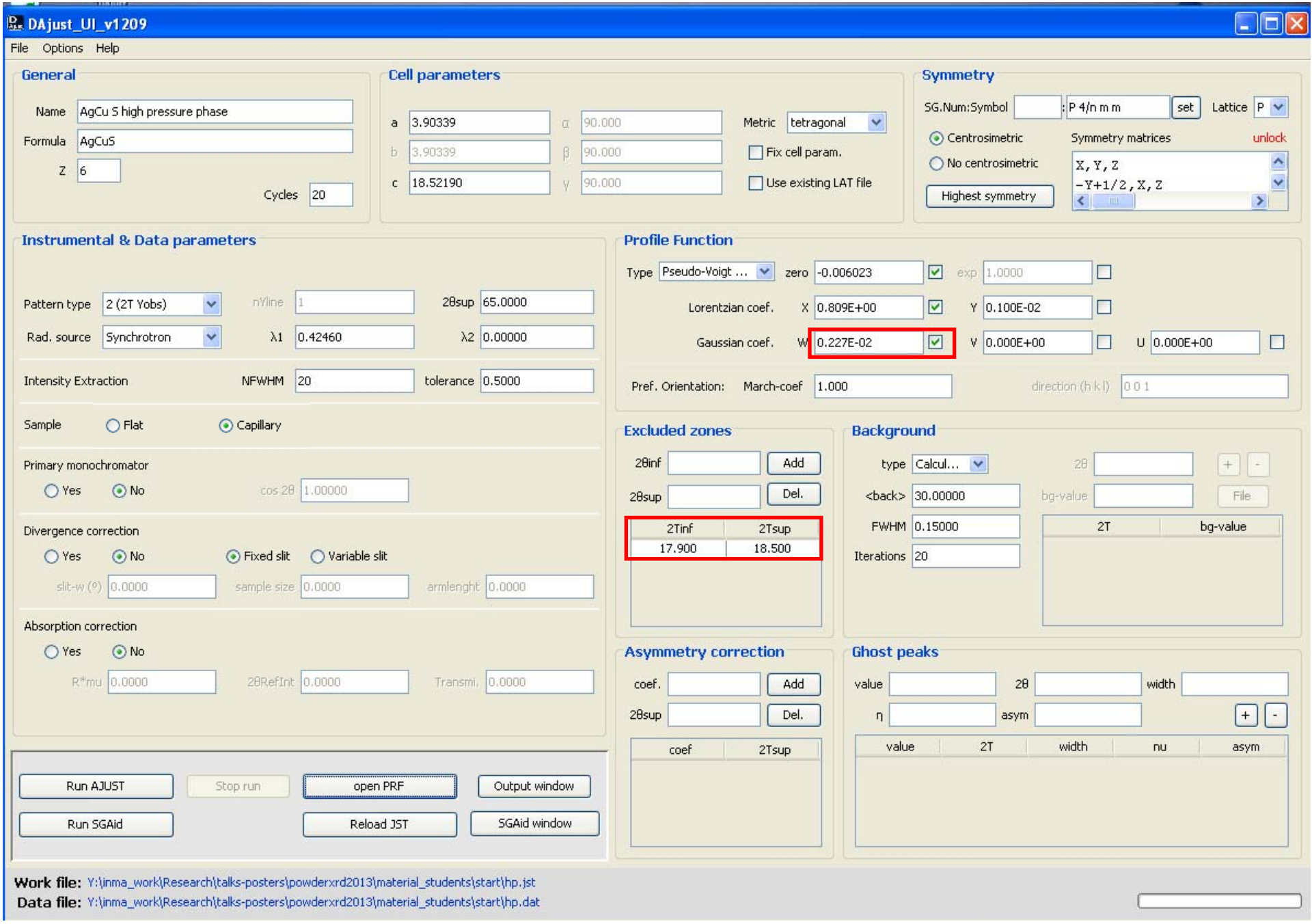
b 3.90341 β 90.000
c 18.52865 γ 90.000
Metric tetragonal
Fix cell param.
Use existing LAT file
Symmetry
SG.Num:Symbol : P 4/n m m set Lattice P
Centrosimetric
No centrosimetric
Highest symmetry
Symmetry matrices unlock
X, Y, Z
-Y+1/2, X, Z
Instrumental & Data parameters
Pattern type 2 (2T Yobs) nYline 1 2θsup 65.0000
Rad. source Synchrotron λ1 0.42460 λ2 0.00000
Intensity Extraction NFWHM 20 tolerance 0.5000
Sample Flat Capillary
Primary monochromator
Yes No cos 2θ 1.00000
Divergence correction
Yes No Fixed slit Variable slit
slit-w (°) 0.0000 sample size 0.0000 armlenght 0.0000
Absorption correction
Yes No
R*mu 0.0000 2θRefInt 0.0000 Transmi. 0.0000
Profile Function
Type Pseudo-Voigt ... zero 0.003657 exp 1.0000
Lorentzian coef. X 0.100E-02 Y 0.100E-02
Gaussian coef. W 0.1 V 0.000E+00 U 0.000E+00
Pref. Orientation: March-coef 1.000 direction (h k l) 0 0 1
Excluded zones
2θinf Add
2θsup Del.
2Tinf 2Tsup
Background
type Calcul... 2θ
<back> 30.00000 bg-value File
FWHM 0.15000
Iterations 20
2T bg-value
Asymmetry correction
coef. Add
2θsup Del.
coef 2Tsup
Ghost peaks
value 2θ width
η asym
value 2T width nu asym
Run AJUST Stop run open PRF Output window
Run SGAid Reload JST SGAid window
Work file: Y:\inma_work\Research\talks-posters\powderxrd2013\material_students\start\hp.jst
Data file: Y:\inma_work\Research\talks-posters\powderxrd2013\material_students\start\hp.dat
AJUST Running

File Plot Options Points Selection X space Calculations Rietveld plot options Text External applications Tools Help

AgCu S high pressure phase, P 4/n m m, Z=6, AgCuS, Synchrotron



03-14-2013 16:42 X = Y =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File Options Help
General
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Formula AgCuS
Z 6
Cycles 20
Cell parameters
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b 3.90339 β 90.000
c 18.52190 γ 90.000
Metric tetragonal
Fix cell param.
Use existing LAT file
Symmetry
SG.Num:Symbol P 4/n m m set Lattice P
Centrosimetric
No centrosimetric
Highest symmetry
Symmetry matrices unlock
X, Y, Z
-Y+1/2, X, Z
Instrumental & Data parameters
Pattern type 2 (2T Yobs) nYline 1 2θsup 65.0000
Rad. source Synchrotron λ1 0.42460 λ2 0.00000
Intensity Extraction NFWHM 20 tolerance 0.5000
Sample Flat Capillary
Primary monochromator
Yes No cos 2θ 1.00000
Divergence correction
Yes No Fixed slit Variable slit
slit-w (°) 0.0000 sample size 0.0000 armlenght 0.0000
Absorption correction
Yes No
R*mu 0.0000 2θRefInt 0.0000 Transmi. 0.0000
Profile Function
Type Pseudo-Voigt ... zero -0.006023 exp 1.0000
Lorentzian coef. X 0.809E+00 Y 0.100E-02
Gaussian coef. W 0.227E-02 V 0.000E+00 U 0.000E+00
Pref. Orientation: March-coef 1.000 direction (h k l) 0 0 1
Excluded zones
2θinf Add
2θsup Del.
2Tinf 2Tsup
17.900 18.500
Background
type Calcul... 2θ
<back> 30.00000 bg-value File
FWHM 0.15000
Iterations 20
2T bg-value
Asymmetry correction
coef. Add
2θsup Del.
coef 2Tsup
Ghost peaks
value 2θ width
η asym
value 2T width nu asym
Run AJUST Stop run open PRF Output window
Run SGAid Reload JST SGAid window
Work file: Y:\inma_work\Research\talks-posters\powderxrd2013\material_students\start\hp.jst
Data file: Y:\inma_work\Research\talks-posters\powderxrd2013\material_students\start\hp.dat

DAjust_UI_v1209

File Options Help

General

Cell parameters

Symmetry

Name AgCu S

Formula AgCuS

Z 6

unlock

Instrumental

Pattern type

Rad. source

Intensity Extraction

Sample

Primary monochrom

Yes

Divergence correct

Yes

slit-w (°)

Absorption correcti

Yes

R*mu

Run AJUST

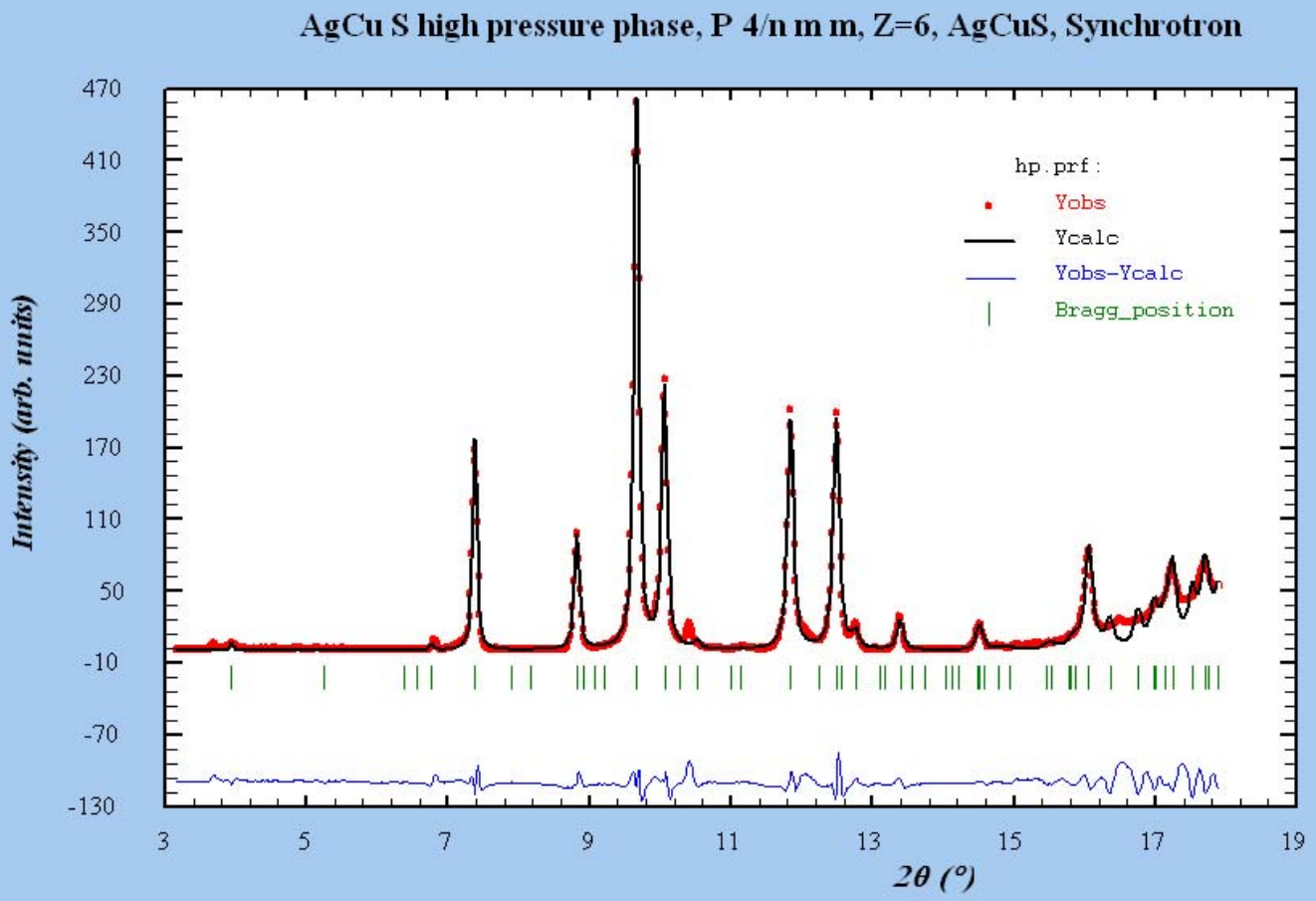
Run SGAid

Work file: Y:\inma

Data file: Y:\inma

WinPLOTR [CDIFX UMR6226 Rennes / ILL Grenoble]

File Plot Options Points Selection X space Calculations Rietveld plot options Text External applications Tools Help



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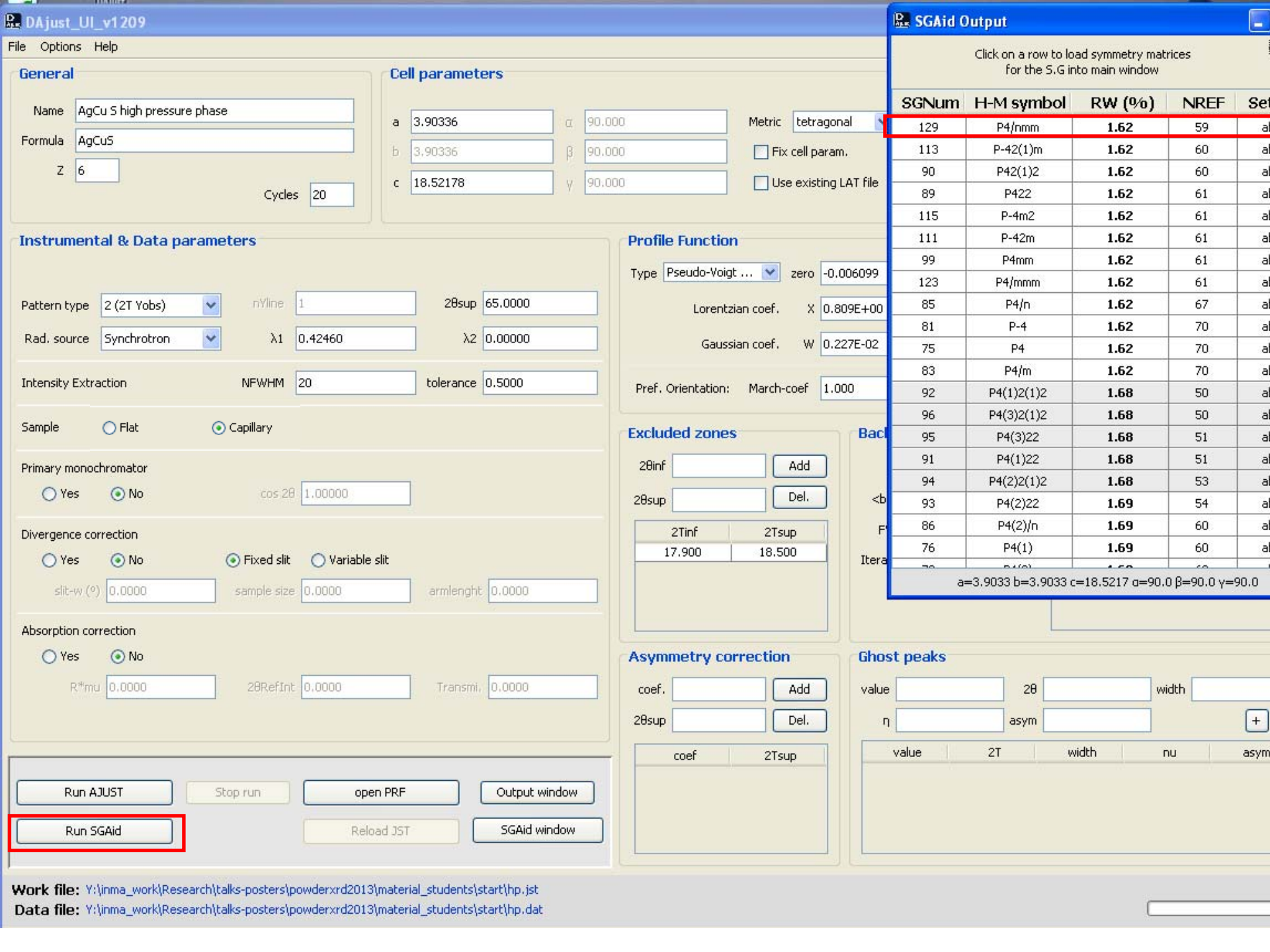
DAjust_UI_v1209
File Options Help
General
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Formula AgCuS
Z 6
Cycles 20
Cell parameters
a 3.90336 α 90.000
b 3.90336 β 90.000
c 18.52178 γ 90.000
Metric tetragonal
Fix cell param.
Use existing LAT file
Instrumental & Data parameters
Pattern type 2 (2T Yobs)
nYline 1
2θsup 65.0000
Rad. source Synchrotron
λ1 0.42460
λ2 0.00000
Intensity Extraction
NFWHM 20
tolerance 0.5000
Sample Flat Capillary
Primary monochromator
Yes No
cos 2θ 1.00000
Divergence correction
Yes No Fixed slit Variable slit
slit-w (º) 0.0000
sample size 0.0000
armlenght 0.0000
Absorption correction
Yes No
R*mu 0.0000
2θRefInt 0.0000
Transmi. 0.0000
Profile Function
Type Pseudo-Voigt ...
zero -0.006099
Lorentzian coef. X 0.809E+00
Gaussian coef. W 0.227E-02
Pref. Orientation: March-coef 1.000
Excluded zones
2θinf
2θsup
Add
Del.
2Tinf 2Tsup
17.900 18.500
Asymmetry correction
coef.
2θsup
Add
Del.
coef 2Tsup
Ghost peaks
value 2θ width
η asym
+
value 2T width nu asym
Run AJUST
Stop run
open PRF
Output window
Run SGAid
Reload JST
SGAid window
Work file: Y:\inma_work\Research\talks-posters\powderxrd2013\material_students\start\hp.jst
Data file: Y:\inma_work\Research\talks-posters\powderxrd2013\material_students\start\hp.dat
SGAid Output
Click on a row to load symmetry matrices for the S.G into main window
SGNum H-M symbol RW (%) NREF Set
129 P4/nmm 1.62 59 al
113 P-42(1)m 1.62 60 al
90 P42(1)2 1.62 60 al
89 P422 1.62 61 al
115 P-4m2 1.62 61 al
111 P-42m 1.62 61 al
99 P4mm 1.62 61 al
123 P4/mmm 1.62 61 al
85 P4/n 1.62 67 al
81 P-4 1.62 70 al
75 P4 1.62 70 al
83 P4/m 1.62 70 al
92 P4(1)2(1)2 1.68 50 al
96 P4(3)2(1)2 1.68 50 al
95 P4(3)22 1.68 51 al
91 P4(1)22 1.68 51 al
94 P4(2)2(1)2 1.68 53 al
93 P4(2)22 1.69 54 al
86 P4(2)/n 1.69 60 al
76 P4(1) 1.69 60 al
a=3.9033 b=3.9033 c=18.5217 α=90.0 β=90.0 γ=90.0

From this point on …

- Solve the structure -> ab-initio structure solution.
    - file.pow & xlens_pd6 (tomorrow's talk).

-Deduce the structure, from space group relationships or/and with the aid of ab-initio calculations (energetic)