

Dajust/sgaid example

Powder diffraction data at high pressure
inorganic material

Structural Phase Transitions on AgCuS Stromeayerite Mineral under Compression

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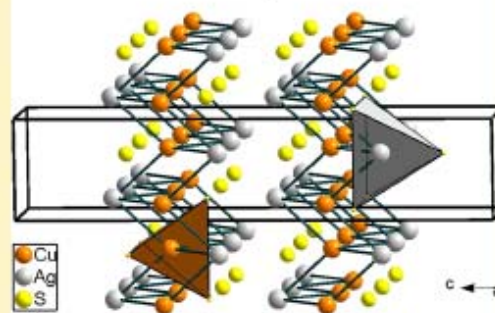
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ABSTRACT: The structural behavior of mineral Stromeayerite, AgCuS, has been studied by means of angle-dispersive X-ray diffraction measurements up to 13 GPa and ab initio total-energy calculations. Two high-pressure phase transitions are found at 1.4 and 5.7 GPa, from the initial distorted Ni₂In-type phase (AuRbS-type, RP, space group *Cmc*2₁) 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.

Structure of AgCuS at 3 GPa



First publication with Alba data

[dx.doi.org/10.1021/ic302116b](https://doi.org/10.1021/ic302116b) | *Inorg. Chem.* 2013, 52, 355–361

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

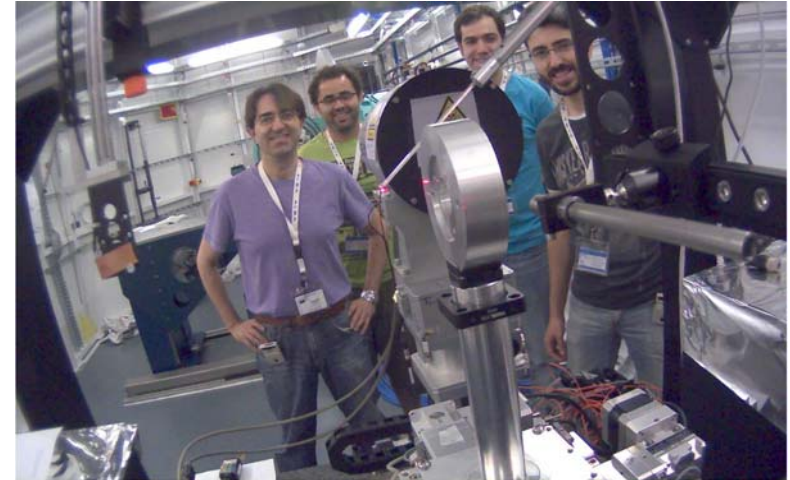
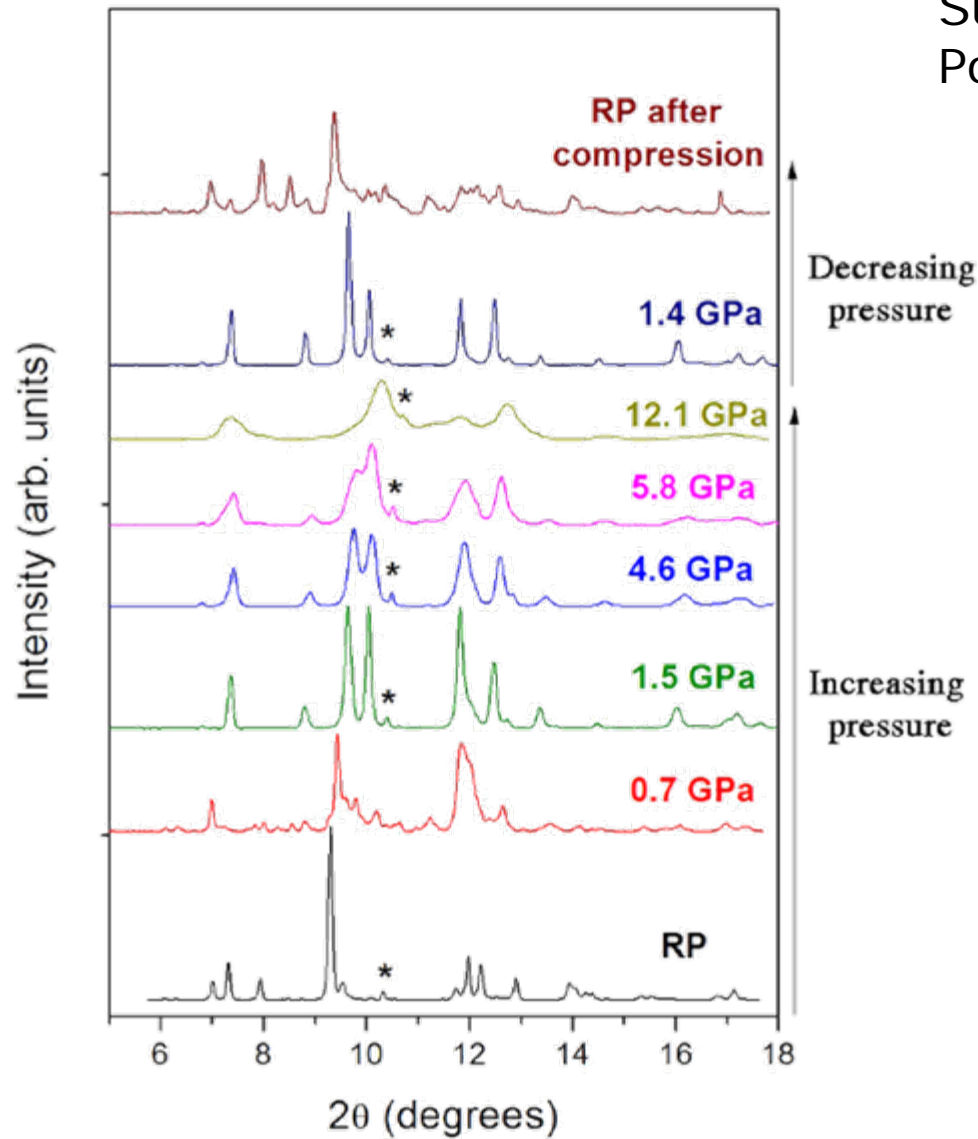
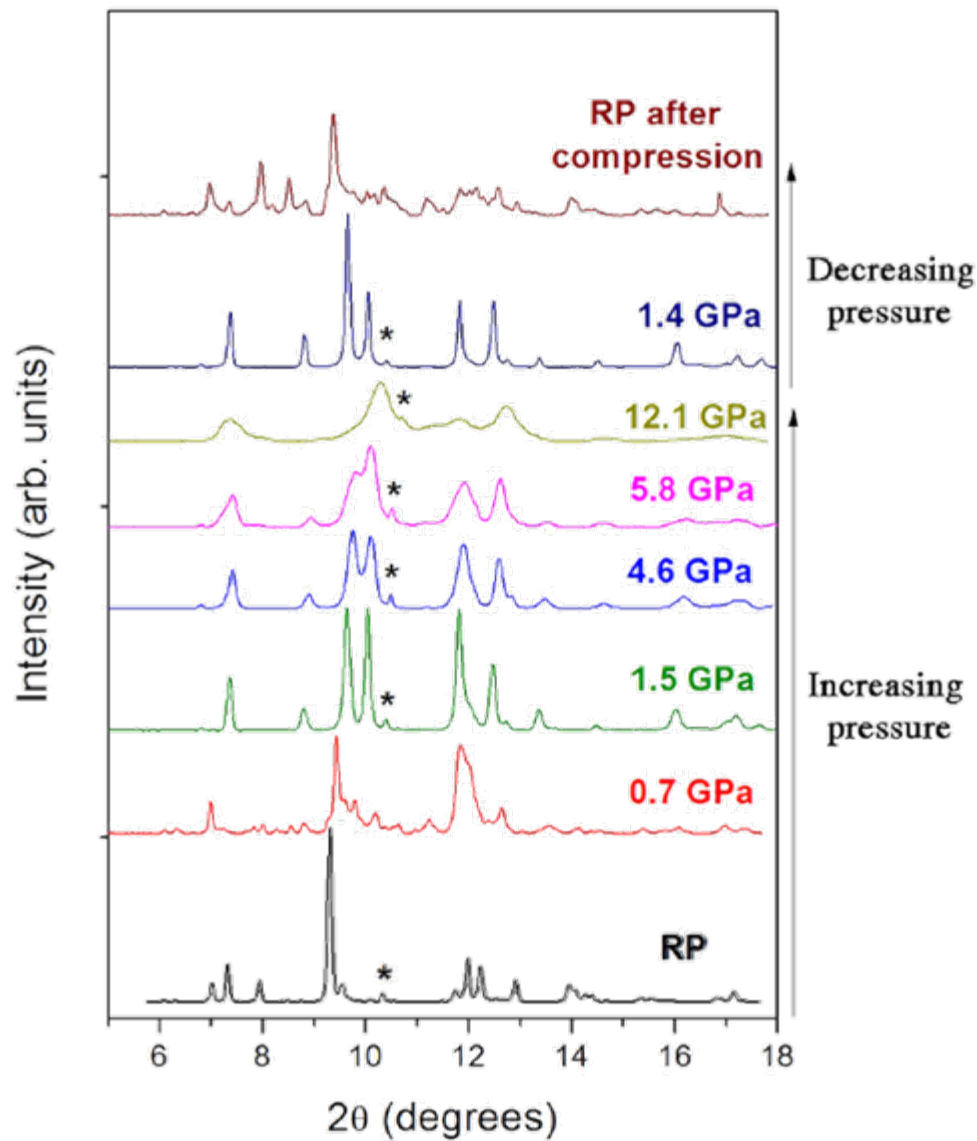


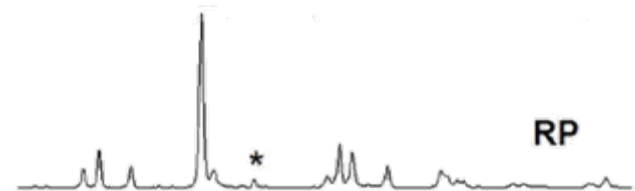
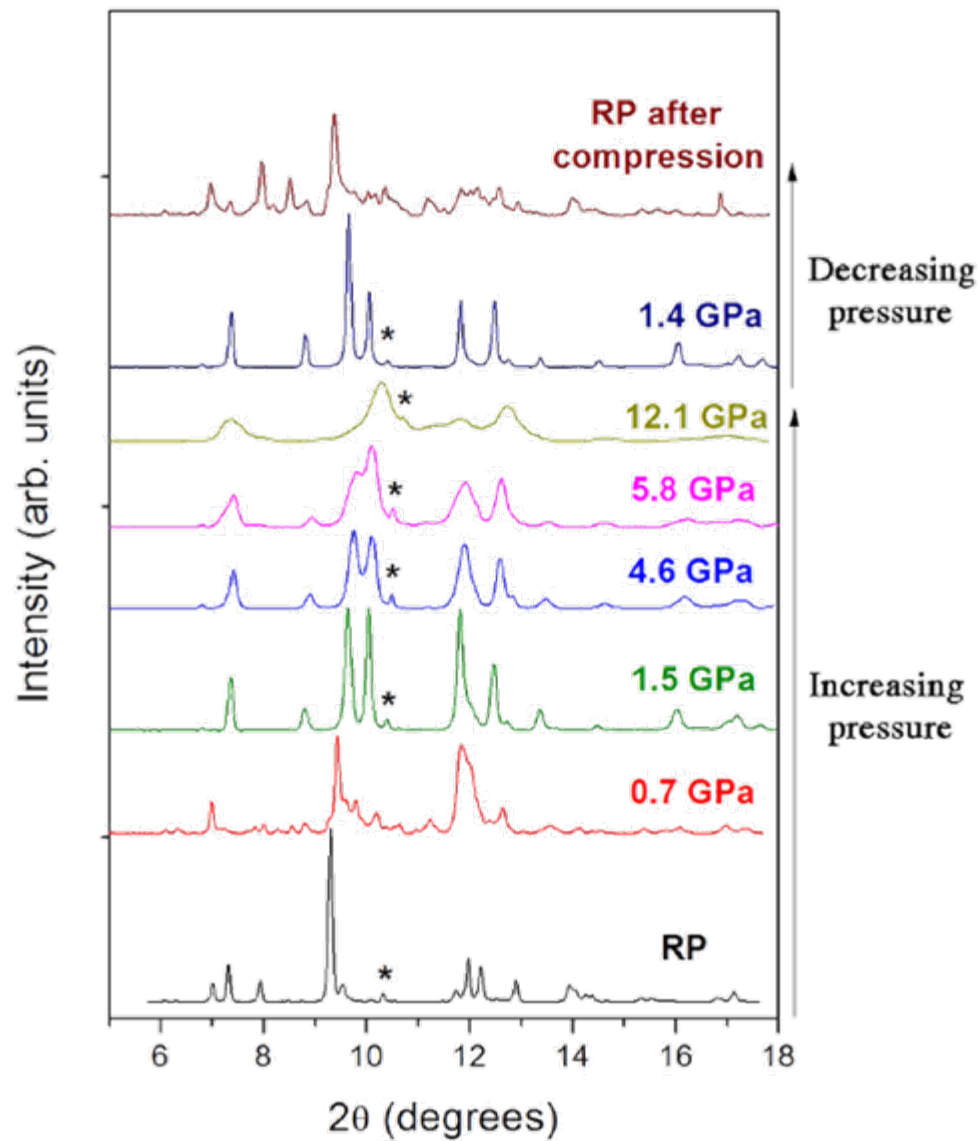
Figure 2. Powder XRD patterns of AgCuS at different pressure conditions. Upstroke and downstroke processes are indicated on the right-hand side. Asterisks denote the diffraction maxima of silver.



Starting point: room temperature phase
known structure

Room Pressure Phase of Stromeayerite, AgCuS
Cmc21, $Z=4$, $a=4.06\text{\AA}$, $b=6.64\text{\AA}$ and $c=7.97\text{\AA}$

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.

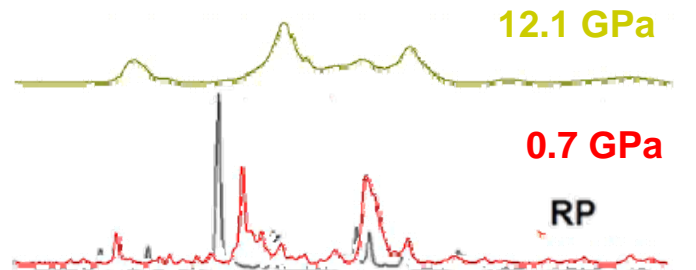
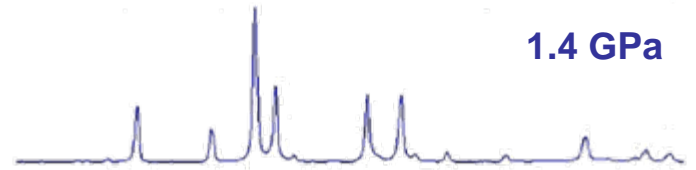


Room Pressure Phase of Stromeyerite, AgCuS
 $Cmc21$, $Z=4$, $a=4.06\text{\AA}$, $b=6.64\text{\AA}$ and $c=7.97\text{\AA}$

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.

P=1.4 GPa
(when decreasing the pressure)

The peaks can be indexed with
Space group P4/nmm $a=3.90\text{\AA}$, $c=18.52\text{\AA}$



Room Pressure Phase of Stromeierite, AgCuS
Cmc21, $Z=4$, $a=4.06\text{\AA}$, $b=6.64\text{\AA}$ and $c=7.97\text{\AA}$

Strategies to index the new phase

- Group subgroup relationships (*Bilbao crystallographic server*)
- Ab initio calculations
- Already known phases of the same family of compounds

General

Name

Formula

Z

Cycles

Cell parameters

a α 90.000 Metric

b β 90.000 ☐ Fix cell param.

c γ 90.000 ☐ Use existing LAT file

Symmetry

SG.Num:Symbol : set Lattice

☐ Centrosymmetric Symmetry matrices unlock

☐ No centrosymmetric

Highest symmetry

Instrumental & Data parameters

Pattern type nYline 2 θ sup

Rad. source λ 1 λ 2

Intensity Extraction NFWHM tolerance

Sample ☐ Flat ☒ Capillary

Primary monochromator

☐ Yes ☒ No

cos 2 θ

Divergence correction

☐ Yes ☒ No

☐ Fixed slit ☐ Variable slit

slit-w (°) sample size armlenght

Absorption correction

☐ Yes ☒ No

R* μ 2 θ RefInt Transm.

Profile Function

Type zero ☐ exp ☐

Lorentzian coef. X ☐ Y ☐

Gaussian coef. W ☐ V ☐ U ☐

Pref. Orientation: March-coef direction (h k l)

Excluded zones

2 θ inf Add

2 θ sup Del.

2Tinf	2Tsup

Background

type 2 θ + -

<back> bg-value File

FWHM

Iterations

2T	bg-value

Asymmetry correction

coef. Add

2 θ sup Del.

coef	2Tsup

Ghost peaks

value 2 θ 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

FileOptionsHelp

General

Name

AgCu S high pressure phase

Formula

AgCuS

Z

6

Cycles

Cell parameters

a

3.90312

α

90.000

b

3.90312

β

90.000

c

18.5268

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

Centrosymmetric

No centrosymmetric

Highest symmetry

Symmetry matrices

Y, X, Z

X, -Y+1/2, Z

-Y+1/2, -X+1/2, Z

Instrumental & Data parameters

Pattern type

2 (2T Yobs)

Rad. source

Synchrotron

Intensity Extraction

Sample

Flat

Capillary

Primary monochromator

Yes

No

Divergence correction

Yes

No

Fixed slit

Variable slit

Absorption correction

Yes

No

nYline

1

2 θ sup

65.000

λ 1

0.42460

λ 2

0.0

NFWHM

20

tolerance

0.5

cos 2 θ

1.000

slit-w (°)

0.000

sample size

0.000

armlenght

0.000

R*mu

0.000

2 θ RefInt

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

2 θ inf

Add

2 θ sup

Del.

2Tinf

2Tsup

Background

type

Calcul...

2 θ

<back>

30.0

bg-value

File

FWHM

0.150

2T

bg-value

Iterations

20

Asymmetry correction

coef.

Add

2 θ sup

Del.

coef

2Tsup

Ghost peaks

value

2 θ

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

FileOptionsHelp

General

Name

AgCu 5 high pressure phase

Formula

AgCu5

Z

6

Cycles

20

Cell parameters

a

3.90312

α

90.000

b

3.90312

β

90.000

c

18.5268

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

Centrosymmetric

☒

No centrosymmetric

☐

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

λ

0.4246

Intensity Extraction

NFWHM

20

Sample

☐ Flat

☒ Capillary

Primary monochromator

☐ Yes

☒ No

cos 2 θ

1.000

Divergence correction

☐ Yes

☒ No

☐ Fixed slit

☐ V

slit-w (°)

0.000

sample size

0.000

Absorption correction

☐ Yes

☒ No

R* μ

0.000

2 θ RefInt

0.000

Save

Save in:

start

hp.dat

File name:

hp.dat

Files of type:

All Files

Save

Cancel

Run AJUST

Stop run

open PRF

Output window

Run SGAid

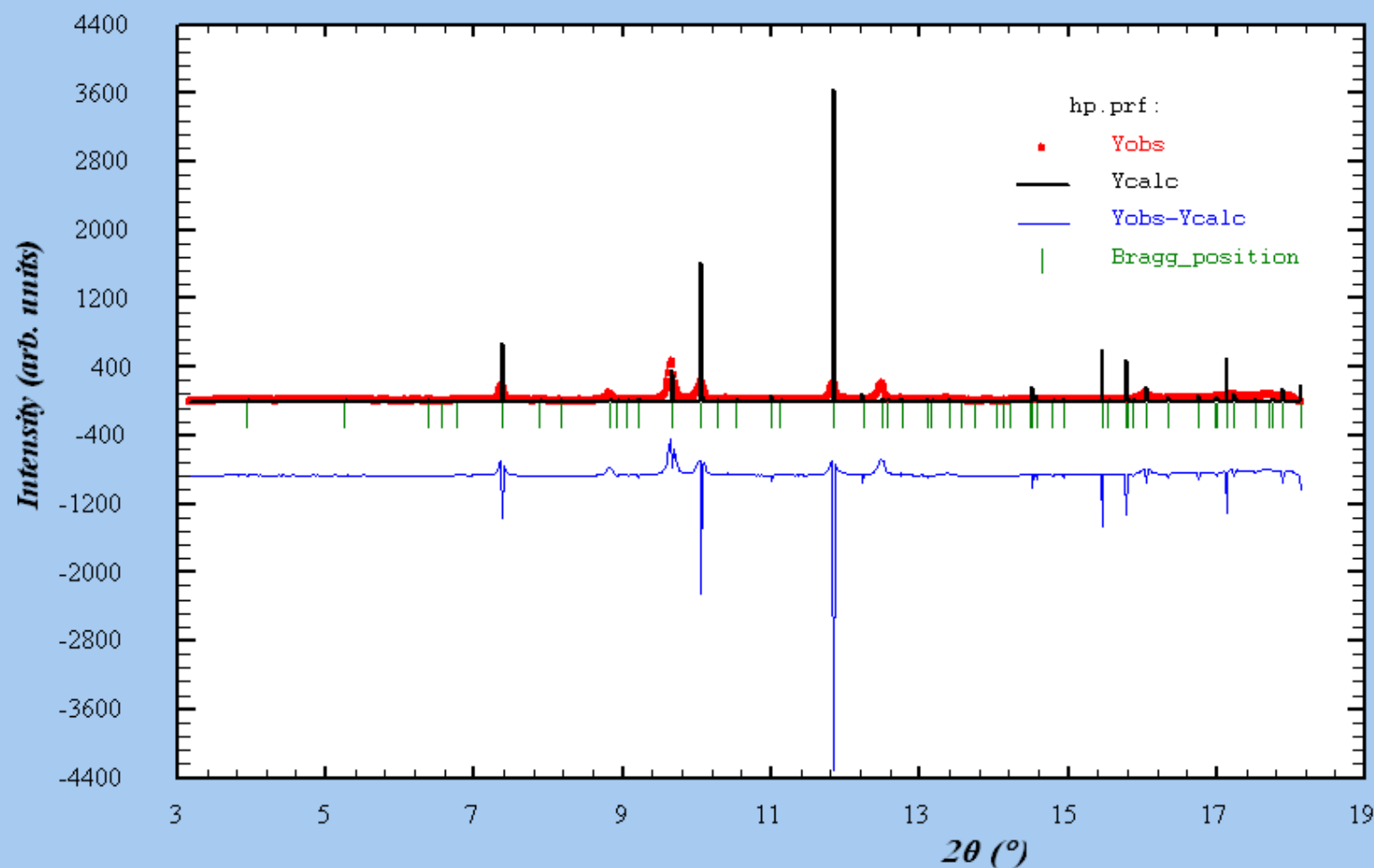
Update fields

SGAid window

Work file: no file

Data file: no file

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



General

Name AgCu 5 high pressure phase

Formula AgCu5

Z 6

Cycles 20

Cell parameters

a 3.90341 α 90.000b 3.90341 β 90.000c 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

☒ Centrosymmetric☐ No centrosymmetric

Highest symmetry

Symmetry matrices

X, Y, Z
-Y+1/2, X, Z

unlock

Instrumental & Data parameters

Pattern type 2 (2T Yobs) $n\lambda$ line 1 2 θ sup 65.0000Rad. source Synchrotron λ 1 0.42460 λ 2 0.00000

Intensity Extraction NFWHM 20 tolerance 0.5000

Sample ☐ Flat ☒ Capillary

Primary monochromator

☐ Yes ☒ Nocos 2 θ 1.00000

Divergence correction

☐ Yes ☒ No☒ Fixed slit ☐ Variable slit

slit-w (°) 0.0000

sample size 0.0000

armlength 0.0000

Absorption correction

☐ Yes ☒ NoR* μ 0.00002 θ RefInt 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

2 θ inf Add2 θ sup Del.

2Tinf 2Tsup

Background

type Calcul... 2 θ + -

<back> 30.00000 bg-value File

FWHM 0.15000

Iterations 20

2T

bg-value

Asymmetry correction

coef. Add

2 θ sup Del.

coef 2Tsup

Ghost peaks

value 2 θ width + - η asym

value 2T width nu asym

Run AJUST

Stop run

open PRF

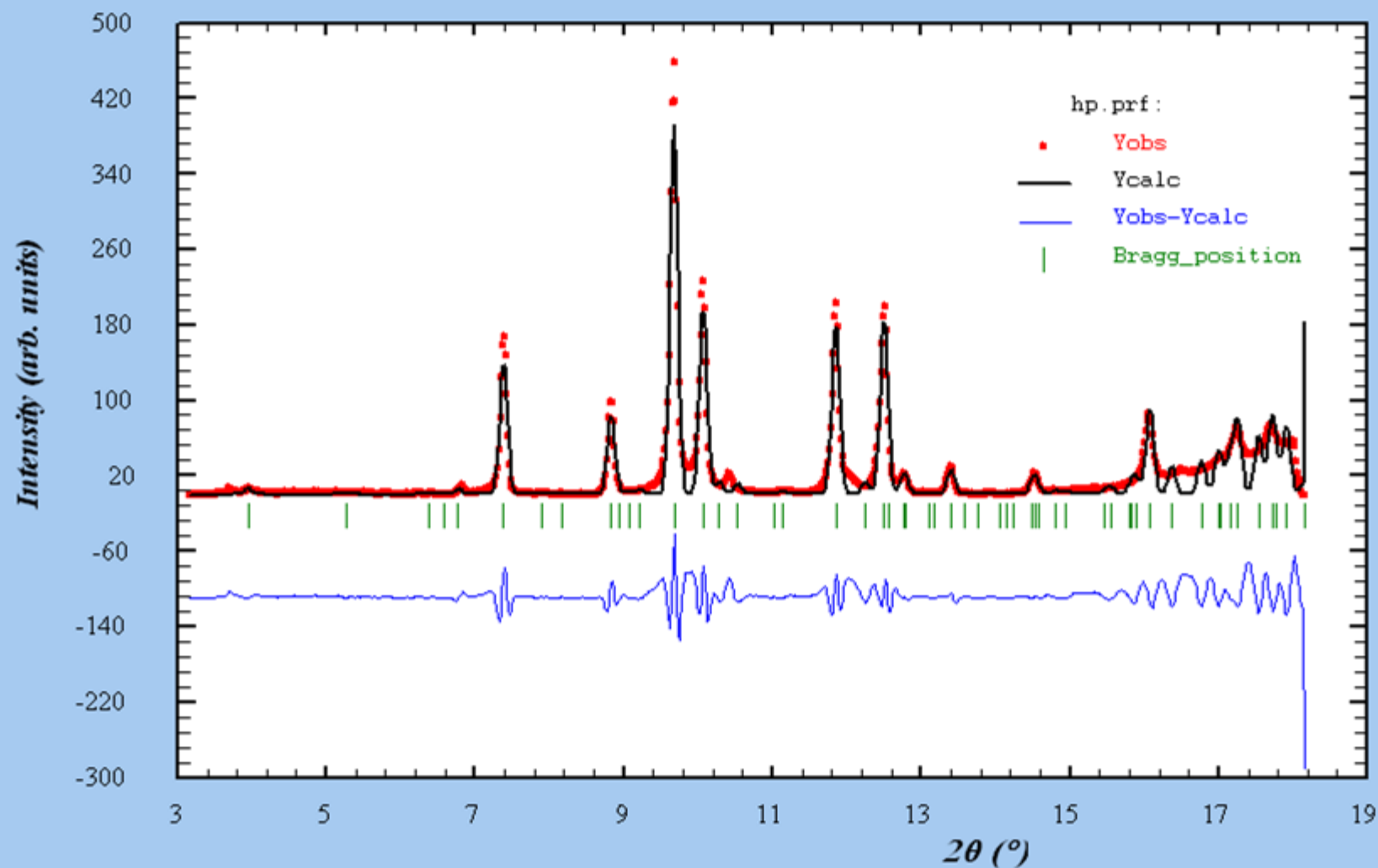
Output window

Run SGAid

Reload JST

SGAid window

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



DAjust_UI_v1209

FileOptionsHelp

General

Name

AgCu S high pressure phase

Formula

AgCuS

Z

6

Cycles

20

Cell parameters

a

3.90339

α

90.000

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

☒ Centrosymmetric

Symmetry matrices

unlock

☐ No centrosymmetric

X, Y, Z

- Y+1/2, X, Z

Highest symmetry

Instrumental & Data parameters

Pattern type

2 (2T Yobs)

nYline

1

2 θ sup

65.0000

Rad. source

Synchrotron

λ 1

0.42460

λ 2

0.00000

Intensity Extraction

NFWHM

20

tolerance

0.5000

Sample

☐ Flat

☒ Capillary

Primary monochromator

☐ Yes

☒ No

cos 2 θ

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

2 θ RefInt

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

2 θ inf

Add

2 θ sup

Del.

2Tinf

2Tsup

17.900

18.500

Background

type

Calcul...

2 θ

+

-

<back>

30.00000

bg-value

File

FWHM

0.15000

Iterations

20

2T

bg-value

Asymmetry correction

coef.

Add

2 θ sup

Del.

coef

2Tsup

Ghost peaks

value

2 θ

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

General

Cell parameters

Symmetry

Name AgCu S

Formula AgCuS

Z 6

Instrumental

Pattern type 2 (

Rad. source Syn

Intensity Extrac

Sample

Primary monochrom

Yes

Divergence correct

Yes

slit-w (°) 0.

Absorption correct

Yes

R*mu 0.

Run AJUST

Run SGAd

Work file: Y:\inm

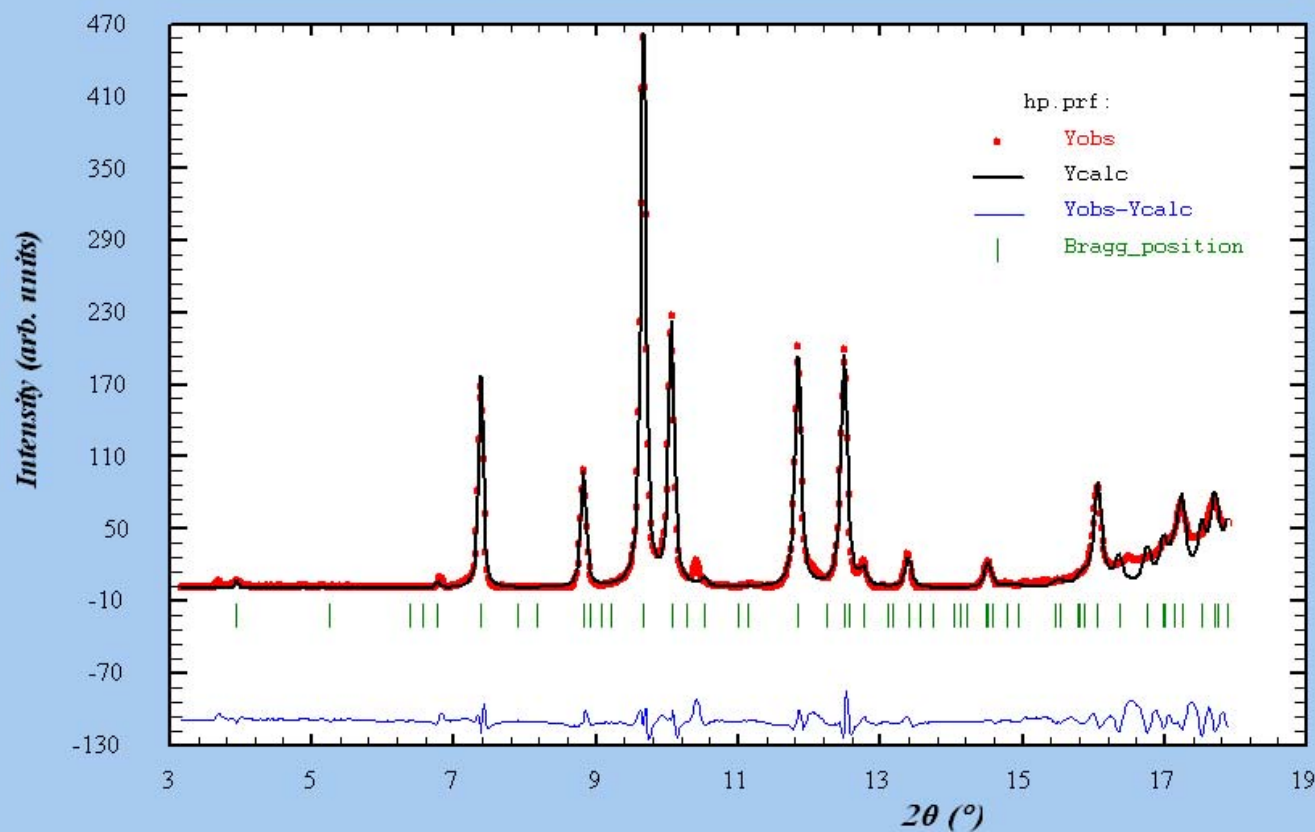
Data file: Y:\inm

WinPLOTR [CDIFX UMR6226 Rennes / ILL Grenoble]

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

Pcr Ed Pcr Bond Str FP PRF Stu dio VIEW CIF FP CVC SEQ SEQ hkl SEQ dse SEQ MIC DiC Vol DOS RESET X

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



03-14-2013

16:44

X =

Y =

General

Name

Formula

Z

Cycles

Cell parameters

a α Metric

b β ☐ Fix cell param.

c γ ☐ Use existing LAT file

Instrumental & Data parameters

Pattern type nYline 2 θ sup

Rad. source λ 1 λ 2

Intensity Extraction NFWHM tolerance

Sample ☐ Flat ☒ Capillary

Primary monochromator

☐ Yes ☒ No

cos 2 θ

Divergence correction

☐ Yes ☒ No

☒ Fixed slit ☐ Variable slit

slit-w ($^\circ$)

sample size

armlenght

Absorption correction

☐ Yes ☒ No

R* μ

2 θ RefInt

Transmi.

Profile Function

Type zero

Lorentzian coef. X

Gaussian coef. W

Pref. Orientation: March-coef

Excluded zones

2 θ inf

2 θ sup

2Tinf	2Tsup
17.900	18.500

Asymmetry correction

coef.

2 θ sup

coef	2Tsup
------	-------

Ghost peaks

value 2 θ width

η asym

value	2T	width	nu	asym
-------	----	-------	----	------

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

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

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)