

2010年12月17日(金) ~ 12月22日(月)

共同利用実験 2009G131

「軌道放射光粉末回折による多結晶体の組織評価」

"Texture Analysis of Polycrystalline Materials
Using SR Powder Diffraction Method

Takashi: IDA (Nagoya Inst. Tech.)

Davor BALZAR (Univ. Denver)

2010年12月17日(金)

○ Check of beam-line optics

Coolant for monochromator

Upstream: 24.2°C. Downstream 24.5°C

Optics

> getpara

Lambda: 1.2000

Mono. TEM: 10.9062

Mono. Z2: 12.7299

Mono. DTH2: 0.2650

Mirr. MPV: 29.4036

Mirr. MRV: 31.5334

Mirr. MBFH: 7.3590

Mirr. MRH: 10.1550

Mirr. BNT: 0.0000

○ Initialization

Control / measure most system once Powered Off and ON again.

Reset the main controller.

All-axis initialized.

- Slit-base adjustment (slit $W 2.5 \times H 0.05$)
Default value is changed to (Att. (n 5 (2))
-8.6 mm from -8.35 mm

★ Trouble in network system (!)

4B2 local network is disconnected?

- Gonio-base adjustment
(center-slit inserted)

Default value is changed to

-10.932 mm from -10.837 mm

(Amount of change is different from slit-base?)

④ (Ω)-scan with center slit.

assumed correction angle: 0.6950°

maximum at -1.1°

→ default value for ④

changed to -0.405° from 0.6950°

Gonio-base ~~adjust~~ scan again

Maximum position: -10.930 mm

Default value unchanged

⇒ MAS20101217-1.pxp

○ Auto-centering of analyzers

No.	$\Theta_A(^{\circ})$	$\Delta 2\Theta_G(^{\circ})$	$\Delta 2\Theta_E(^{\circ})$
1	10.436	124.8231	124.8256
2	10.579	99.7826	99.7830
3	10.504	74.7849	74.7841
4	10.647	49.8017	49.7992
5	10.566	24.7963	24.7929
6	10.578	0.0335 -0.0332	-0.0335

⇒ MAS20101217-2.pxp

○ Auto-measurement of correction angles
(analyzer gap : 6 mm)

No.	$\Delta 2\Theta_G(^{\circ})$	$\Delta 2\Theta_E(^{\circ})$
1	124.8203	124.8227
2	99.7846	99.7846
3	74.7772	74.7762
4	49.8026	49.7998
5	24.7975	24.7937
6	-0.0324	-0.0330

⇒ MAS20101217-3.pxp

○ Preparation for $\Theta(\Omega)$ -adjustment

On 2 Θ -scan of No. 1 - arms.

More than 0.015° -difference ~~in~~ in peak position.

The base-plate has more tightly ^{been} fastened ~~the~~ the rotation axis for analyzer No. 1.

○ Auto-centering of analyzer No. 1 & 2

No.	$\Theta_A (^\circ)$	$\Delta 2\Theta_G (^\circ)$	$\Delta 2\Theta_E (^\circ)$
1	10.498	124.8214	124.8237
2	10.578	99.7810	99.7812

\Rightarrow MAS20101217-4.pxp

○ Auto-correction measurements ~~No. 1 & 2~~

(analyzer gap: 6 mm)

No.	$\Delta 2\Theta_G (^\circ)$	$\Delta 2\Theta_E (^\circ)$
1	124.8198	124.8221
2	99.7829	99.7830
3	74.7848	74.7836
4	49.8027	49.7999
5	24.7966	24.7928
6	-0.0336	-0.0336

○ 2θ -scan, direct beam, No. 1

Peak position (with correct angle) : 0.0000°

○ ~~Flat~~ Flat-specimen Z -adjustment

& Θ (Ω) correct angle

$$\Delta\Theta = -0.330^\circ$$

\Rightarrow MAS20101217-5.pxp

○ NIST SRM 640c, asymmetric reflection, segmented

slit $W 10\text{mm} \times H 0.75\text{mm}$

$$\Omega = 8^\circ$$

\Rightarrow MAS20101217-6.pxp

○ LaB₆ (NIST SRM 660) all-arm meas.

~~#~~ 2θ : $0^\circ \sim 29.98^\circ$ (0.005° step)

$$FT = 0.5\text{s}$$

start : 20:11'16"

finished : 23:10'24"

\Rightarrow MAS20101217-7.pxp

○ Indialite (Cordierite (1200°C , 10 hr) #1
all-arm meas

$$\Omega = 6^\circ$$

start : 23:25'07", stop : 08:02'13" 25.7°C

\Rightarrow MAS20101217-8.pxp (8 hr 37 min)

December 18, 2010 (Saturday)

○ Indialite/Cordierite (1300°C, 10hr) #2
all-arm measurement

08:30 Injection to PF-AR

08:54'18" scan start

17:31'16" scan finished (8hr 37min)

⇒ MAS20101218-1.pxp

○ NIST SRM640c, segmented meas.
asym. reflect. mode $\Omega = 6^\circ$

18:32'38" #1 scan start

20:39'09" #23 scan finish

⇒ MAS20101218-2.pxp

○ Indialite/Cordierite (1400°C, 10hr) #3
all-arm measurement

21:07'49" scan start

05:45'02" scan finished ↘ 8hr 37min

⇒ MAS20101218-3.pxp

December 19, 2010 (Sunday)

○ Spinner-scan measurements of #3 sample

• $2\Theta = 8.12^\circ$, $\Omega = 6^\circ$, $FT = 0.5s$ (scan #2)

$\Phi: 0^\circ \sim 360^\circ$ (0.8° step)

06:35'04" scan start

06:46'15" scan finished \downarrow 11 min 11s

• $2\Theta = 8.12^\circ$, $\Omega = 4.06^\circ$ (scan #4)

$\Phi: 0^\circ \sim 360^\circ$ (0.8° step)

• $2\Theta = 16.83^\circ$, $\Omega = 8.415^\circ$ (scan #6)

• $2\Theta \sim 20.37^\circ$, symmetric, auto-measurement
(scan #7~8)

08:30 ~~AR~~ Continuous injection stopped
for AR injection

08:55 $I = 450 \text{ mA}$

○ Spinner-scan auto measurement (#3 sample)

$2\Theta = 8.12^\circ, 16.83^\circ, 20.38^\circ, 22.01^\circ, 22.75^\circ$

symmetric mode, $FT = 1s$

09:41'42" #13 scan (2Θ) start

09:45'02" " finished \downarrow 3 min 20s

09:45'09" #14 scan (Φ) start

09:56'45" " finished \downarrow 11 min 36s

10:56'22" #22 scan (Φ) finished

1 hr 14 min 40s for 5 scans

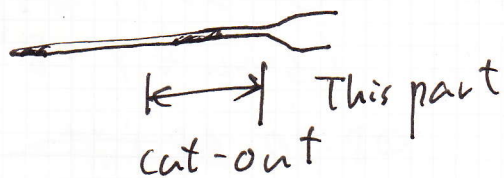
\rightarrow 14 min 56s per scan

\Rightarrow MFS20101219-1.pxp

- Indialite/Cordierite #4 (1200°C, 50hr)
 all-arm measurement, $2\theta: 0^\circ \sim 29.98^\circ (0.005^\circ \lambda \tilde{u}, \tilde{v})$
 11:18'17" scan start (FT = 4s)
 19:55'12" scan finished

⇒ MAS20101219-2.pxp

- Ag, small particle, rod-like
 synthesized by Mr. Iwamoto
 capillary ($\phi 0.1 \text{ mm}$) specimen



- transparency meas.

Center position: -10.911 mm

Diameter: 0.162 mm

$\mu: 62.6(3) \text{ cm}^{-1}$

(cf. goniocenter -10.932 mm)

- all-arm meas

slit $W 10 \text{ mm} \times H 0.5 \text{ mm}$

analyzer edge: 1 mm

$2\theta: -5^\circ \sim 29.98^\circ (0.01^\circ \lambda \tilde{u}, \tilde{v})$

FT = 8s

20:57'53" scan start
 @ 5:50'06" scan finished

December 20, 2010 (Monday)

○ Ag, small particle, rod-like

- Fine scan (2 θ step 0.005°) for 111, 200, 220 reflections

@ 6:44'13" 111-reflection, scan (#3) start

@ 7:44'10" " finished

@ 7:46'46" 200-reflection scan (#4) start

@ 8:46'45" " finished

(continuous injection stopped @ 8:30 ~ 8:50)

@ 8:50'26" 220-reflection scan (#5) start

@ 9:50'27" " finished

⇒ MAS20(01219-2.pxp)

○ Ag, small particle, shape unknown
 synthesized by Mr. Iwamoto
 capillary (ϕ 0.1 mm specimen)

- transparency meas.

center : -10.902 mm

diam : 0.201 mm

μ : 4.15(12) cm⁻¹

- Preparatory meas.

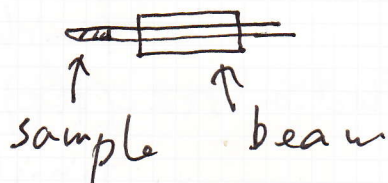
slit: $W 10 \times H 0.5$

$2\theta: 29.07 \sim 29.67$, ~~0.01~~ 0.01° step

FT = 4s

No peak detected.

Because the horizontal position of
Probably capillary was wrong



Someone should have changed the
setting of the microscope.....

⇒ MAS20101220-1.pxp

- Set the same sample, at the position ~
capillary
~ 2 mm shorter.

center: -10.936 mm

diameter: 0.160 mm

$\mu: 12.4(2)$ cm^{-1}

- 111 & 200 peak profile measurement
 2θ : $28.37^\circ \sim 30.37^\circ$ (0.005° step) (scan #2)
 $33.03^\circ \sim 35.03^\circ$ (") (scan #3)

Too weak!

⇒ MAS 20 (91220-2.pxp)

○ Attach flat-specimen spinner

○ Auto-evaluation of correct angles

15:57'03" start

16:14'42" finished

No.	$\Delta 2\theta_G (^\circ)$	$\Delta 2\theta_E (^\circ)$
1	124.8470	124.8493
2	99.7823	99.7825
3	74.7884	74.7873
4	49.8024	49.7997
5	24.7966	24.7927
6	-0.0339	-0.0339

○ Find "half intensity position & correction

$$\Delta 2\theta_{\text{correction}} = -0.237^\circ$$

⇒ MAS 20 (91220-3.pxp)