

ISIS Preparation

Data analysis

Gudrun: load settings from txt file
 set directory with raw data
 add raw data files to analyse (several will be averaged) if you don't chose "run file separately"
 Results are shown in cmd.

DCS = differential cross section should be close to 100%
 → adjust thickness estimate to do that

python:

give program the list of files that have to be run
 and a list of densities (for different temperatures)
 run program → it will run Gudrun
 99.7% to 100.3% DCS is good enough

NIMROD scripts:

fraser 8, 9, 10 should work
 run the heating and scans during heating automatically

fitting: first Fumier-Porod to have a better idea of peak position (periodic spacing)

Gudrun needs Java for installation

20.05.2015

Gudrun:

1. Load settings

Samples_iceforming.txt (tells also which backgrounds to use)
 file → open → don't save current → choose .txt file

2. Instrument fas → set datafile directory

Logarithmic step size determines how many data points you get out. (0.075 should be fine)

3. Choose Sample tab: (e.g. ASW forming)
choose file(s) to analyse (usually one at a time, unless you want averaging)
click → run → check files exist to check get proper density from densities file

→ Run → run BudrunN

26.05.2015

Budrun results that can be further analysed have the extension molcs01

27.05.2015

Python analysis: 1. periodic-spacings.py
2. gp.py
3. porod-constant.py

BudrunN: Pathnames must not be too long!

Otherwise BudrunN will not be able to find existing files.

Python: Gathered python scripts require the following files to be in the folder %

code.txt : saying something like d052
(i.e. deposition at 52 K)

density.txt : stating the ^{initial} temperature and density for as many times as there are files to plot

temp-M10.txt (for example): stating the temperature steps (for heating from 100 to 110 K)

ISIS Preparation

Tighten leak valve a bit more

Get equipment for freezing D₂O: Lab jack, glasses, gloves, Dewars → Catherine
Diagram + Tape (saying what to do)

Do Risk assessment before Monday

Get new lab book and put all information into the front (2 books) put important phone numbers (LN₂ etc.)

Take old lab books and papers and summary (put in folder to leave in the lab)

Post-its, scissors, Tape

Dinner Bettendecker

Email Address to Helen

Vanadium-window on cold head (transparent to neutrons)
capillary to carry the gas has heating wire to keep it at 5°C
→ goes around window and has lots of holes → Sprays D₂O at window

base pressure 10⁻⁶ mbar, plate base temperature 12K

cold finger warms up as well when warming up sample

system has two cryo shields 10K and 80K, they now have holes in front of the vanadium, so they don't stick D₂O
which won't interact with the neutrons

check that temperature controller controls temperature of plate
not of cryostat

deposition for 20 hours at 50 K (first run) (1)

broad peak at 3\AA tells that ice is amorphous
crystalline ice has narrow lines at $> 3\text{\AA}$

slope below 1\AA : granular material

bump at 0.1\AA : pores (how far apart are they (factor 2π))

17 K deposition (18 hours) → bump much smaller and shifted

material stays granular when you heat it

heating rate 0.5 K/min

heating steps: 10 K ; then isothermal

periodic spacings: how far apart are the pores?
do they move / cluster?

check whether bump position analysis works by fitting
slope + gaussian to data instead of scaling it
with q^3

repeat 17 K experiment 24-36 hours → spectrum heat
up stepwise → until desorption

↪ 3-4 days

if that goes well, get comprehensive dataset: $30, 50, 77\text{ K}$
 $1/2$ hour scans should be good enough

deposition times of 8-12 hours should be good enough

at the end maybe use another molecule: N_2 or CO (or methanol, NH_3)

Sabrina.Gaertner

From: Helen.Fraser <helen.fraser@open.ac.uk>
Sent: 11 May 2015 10:50
To: Sabrina.Gaertner; Sergio Ioppolo; Catherine.Hill; tristan.youngs@stfc.ac.uk; chris.goodway@stfc.ac.uk
Subject: Re: ISIS preparation

Hi Sabrina - thank you :)

I also cc'd catherine / sergio / tristan and Chris in.

On June 2nd the Nv setup will anyway need leak and pressure testing again by chris before it goes on the line - to expedite this

(a) try and sue the same NV and also the same swagelock connectors

(b) you may need to check with catherine / chris (at STFC not HALL) re exact connections

NV should have a leak rate of 10^{-9} / min or so with atmopsheric P on one side and UHV on the other - I think the range might need adjusting as it was a pretty clear on / off last time...

however also the current base T of the bin is only 10^{-4} Torr (mBARS).. we want to control the flow to 2×10^{-4} or 3×10^{-4} - its a bit of a black art.... note this is NOT the leak rate... but the flow rate :)

hope this helps

Helen

p.s. i think the NV assembly that was laying around in the PF lab might indeed have been the ISIS Nv - so getting that back is a good idea...

On 11/05/2015 10:40, Sabrina.Gaertner wrote:

Hi Helen,

Catherine suggested to test the needle valve(s) again before going to ISIS, so Sergio and me set up a small test chamber.

What is the pressure range we are aiming at with the needle valve?

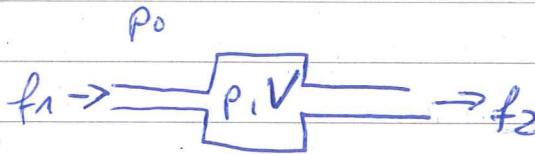
Sabrina

--
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no flow: $p \cdot V = \text{const}$

flow:



○ $f = \frac{dV}{dt} = f_1 - f_2$ at a certain pressure

actually it is number of molecules per second

$$\rightarrow N = \frac{p \cdot V}{kT}$$

$$\rightarrow \frac{dN}{dt} = 0 \text{ wenn } \frac{p_0 \cdot f_1}{f_0} = \frac{f_2 \cdot p}{f_1}$$

$$\rightarrow p = p_0 \cdot \frac{f_{0 \cdot 1}}{f_{0 \cdot 2}}, \quad f_2 = \left(\frac{p_0}{p}\right)^{-1} \cdot f_1$$

$$f_1 = \left(\frac{p_0}{p}\right)^{-1} f_2$$

$$p = 3 \cdot 10^{-8} \text{ mbar}, \quad p_0 = 10^3 \text{ mbar}, \quad f_2 = \frac{1}{4} \cdot 10^3 \frac{\text{L}}{\text{s}}$$

$$\Rightarrow f_1 = \left(\frac{10^3}{3 \cdot 10^{-8}}\right)^{-1} \cdot \frac{1}{4} \cdot 10^3 \frac{\text{L}}{\text{s}} = \frac{3}{4} \cdot 10^{-8} \frac{\text{L}}{\text{s}}$$

→ leak rate of chamber

leak valve probably starts around $\frac{0.4}{4} \cdot 10^{-8} \frac{\text{L}}{\text{s}}$

$$= 1 \cdot 10^{-9} \frac{\text{L}}{\text{s}}$$

$$k = 1,4 \cdot 10^{-23} \text{ J/K}$$

$$1g = 1 \text{ Pa m}^3$$

$$= 10^{-2} \text{ mbar} \cdot 10^3 \text{ l}$$

$$= 10 \text{ mbar l}$$

$$\text{At } T \approx 300 \text{ K}$$

$$p \approx 10^3 \text{ mbar}$$

$$f = 10^{-9} \text{ l/s}$$

$$\frac{dN}{dt} = \frac{f}{RT} \cdot f = \frac{10^3 \text{ mbar} \cdot 10^{-9} \text{ l/s}}{1,4 \cdot 10^{-23} \cdot 10 \text{ mbar l/K} \cdot 300 \text{ K}}$$

$$\approx \frac{10^{-6} \text{ l/s}}{4 \cdot 10^{-23} \cdot 10^3 \text{ l}}$$

$$= \frac{1}{4} \cdot 10^{14} \frac{1}{\text{s}} = \frac{60}{4} \cdot 10^{14} \frac{1}{\text{min}}$$

$$= 15 \cdot 10^{15} \frac{1}{\text{min}}$$

maybe change heating rate (to better understand

why specific surface area of 77 K data increased
with heating before decreasing)

↳ least important thing

maybe investigate pore collapse closer

beamtime from 9 am 3rd June

to 9 am 10th June packing \approx 1 hour

maybe grow crystalline ice at 180 K deposition temperature

set flow when everything is warm and leave leak valve
at this position. To start / stop deposition use
the open/close valves on the deposition gas line

away times:

Helen

4th 7³⁰ - 18⁰⁰

Sergio

7³⁰ - 18⁰⁰

Sabrina

8th 8⁰⁰ - 18⁰⁰

(no night before that)

10th

check travel plans
no night shift before

gatwick or heathrow are closest airports

26.02.2015

PDRA Meeting

Sabrina.Gaertner

From: Sabrina.Gaertner
Sent: 26 August 2015 16:29
To: Helen Fraser
Subject: subjects for PDRA meeting

Hi Helen,

Here is a list of things that I would like to talk about. It's too much for tomorrow's meeting, so I've marked the most important ones red.

- ✓ DPS abstract (I mixed up character and word counts earlier, so the one I sent you can be roughly twice as long as it currently is)
- ✓ Parabolic flight proposal redo page 1.1 picture of set-ups in plane
- ✓ Support rack design up-to-date pictures of racks
- ✓ ISIS: Skype doodle touch pads instead of mouse
- Lab:
 - Leak
 - Temperature readout - connector? USB 1 or 2 - Helen might have cable
 - Fire pistons manually (I have borrowed a power supply to generate +/-5V from Fraser Robertson, which I need to return tomorrow afternoon. Once we are sure whether we need + or - 5V, he will build the required thing for us.)
- Collision Analysis:
 - ✓ ○ Velocities determined by different people vary a lot due to pixel-mm conversion (measuring the piston diameter). Do better statistics for my calibration
 - ✓ ○ Rotation analysis qualitatively or quantitatively? (one value for each video → average)
 - Watch more PF videos (13 left)
 - ✓ ○ Analysis of particle-target collisions? Wait
- Time plan for parabolic flight preparations, papers etc.
 - ✓ Ok

see u tomorrow,
Sabrina

✓ When is next ISIS run?

16th Nov - 20th Nov

→ Move flight 1 day

30th Nov - 4th Dec

21.09.2015

Topics for next PDRA Meeting:

- watch more collision videos
- ✓ - Support Rack design → comments in latest dropbox docx
- locate (monitor mounting, toolbox)
- ✓ - status of EPOS unit
 - USB A to USB adaptor (temperature readout)
- ✓ - decide on touchpad for support rack
 - money for DPS conference?
 - room for DPS conference → single/double? general rule?

Support Rack

- keep Toolbox
- move monitors to the front of rack
 - if need be, we can increase frame size of rack to cover all of monitor-height

EPOS:

- give files Helen found to E-workshop

Touchpad:

- ergo, medium size, without buttons

07.10.2015

Topics for next PDRA meeting:

watch more collision videos

- USB A to USB adaptor (temperature readout)

- money for DPS conference? ✓

- room for DPS conference → single/double? general rule?

EPOS files

- ✓ support rack → aren't monitors too close to eye at front of rack? no, fine

- ✓ video analysis: *how about using change in centre-of-mass velocity / COR as uncertainty indicator?

- * discuss results from airplane tilt calculations

- ✓ mass calculations in Salter 2009 (I get $1.6 \cdot 10^{-6}$ to $4.3 \cdot 10^{-2}$ g instead of 0.01-3 g) | later papers more reliable

ISIS ASW (April 2016) preparation

Samples done in June 2015:

deposition	temperature	time	temperature steps (K)
1	18 K	24 h	20, 30, 40, 50, 60, 70, 80, 90, 100, 110, 120, 130, 140, 150, 160, ¹⁷⁰ 180 → 250
4	30 K	12 h	30, 40 (12 h), 50, 60, 70, 80, 90, 100, 110, 120, 130, 140, 150 → ended on heating ramp to 160
2	50 K	12 h	50, 60, 70, 80, 90, 100, 110, 120, 130, 140, 150, 160, 170, 180 → 250
3	80 K	12 h	80, 90, 100, 110, 120, 130, 140, 150, 160, 170, 180 → 250

Setup at ISIS:

- write down pump number
- ✓ - clean and refill D₂O bottle
- ✓ - write down how much D₂O we put into bottle
- check all temperature control and readout connections before starting experiments
- Tom, Tristan & Daniel will be away on Tuesday
- pump 38
- ✓ Sunday: set up gas panel & leak valve, leak test
clean & fill bottle with D₂O

Before going to ISIS:

- ✓ - print risk assessment
- ✓ - print deposition procedures
- ✓ - find setting of leak valve (page 6)
- ✓ - find out what procedures we used (fraser... - scripts)
- ✓ - tell Tristan pump number → 38
- ✓ - pack: leak valve ✓, dewarsh gloves ✓, spgflst ✓, las jacks ✓
las books ✓, hard drive ✓
- ✓ - login and check all safety boxes are ticked

Experiment plan:

- temperatures: 40, 60, 100, 120 K
- page 15: info what went wrong with heating last time
→ sort out with Tristan on Monday

Arrival:

12:00 (sunday) meet at reception
get access cards from main control room
Lunch 12:00 - 13:30

Away times

Helen:

- Tuesday afternoon 2-4 Helen teaching meeting (telecon)
- Friday morning Catherine's graduation ceremony
Helen might leave Thursday evening
- Fri evening - monday morning, Helen at home
Skype at some point to decide on plans for spare time
- Skype with Catherine & Andrew about 60s
(Helen + Sabrina)

Sergio:

- some day-time at home (Tue/Wed/Thu)