Work package 7 / Sample Environment

Work package leader: E. Lelièvre-Berna

Venue: Coimbra University, Portugal (room E10B, 2nd floor)

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Agenda and group photo: See annexed pages

List of participants

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[A] Advisors, [I] Invited high-pressure expert

ELB welcomes the participants and appreciates the presence of so many attendees.

The slides presented at the meeting are available in the shared cloud and should remain confidential.

Task 7.1: FLOSS software standards for sample environment

Klaus Kiefer presents the motivations for establishing a standard of communication between instrument control workstations and sample environment equipment: reduce recurrent workloads; simplify implementation of new equipment and ease exchange of equipment. This protocol, called SECoP (Sample Environment Communication standard Protocol), must be easy to implement at future and existing facilities.

The core team met several times and prepared a draft document that was refined after the HZB meeting held in April. This document will be presented and discussed at the forthcoming SE@SF international workshop in 10 days before being circulated among all facilities.

EB enquires whether companies will be involved. KK and ELB answer that one clear aim is to convince industrials to adopt it and supply SECoP compliant equipment. Preliminary discussions started about 2 years ago and the SE@SF workshop will be an opportunity to discuss further with them.

[**ACTION**] KK et al. to circulate draft SECoP documentation to facilities

[**ACTION**] KK et al. to present the SECoP protocol to industrials with the aim to determine what would convince them to adopt it.

Task 7.2: towards efficient sample environment for neutron research

- 7.2.1: Signal to background ratio improvements

Victor Laliena presents the methods identified to simulate the presence of microstructures and to calculate the inelastic scattering of polycrystals (incoherent approximation). He started comparing his calculations with Laue images acquired from aluminium alloy calorimeters on Cyclops (ILL).

[**ACTION**] VL to code the inelastic case in the coming months.

[**ACTION**] ILL team to supply data collected with bodies of pressure cells on IN6.

[**ACTION**] ISIS team to supply data collected with dummy cells (task 7.3).

ELB presents results obtained by reducing the thickness of the IN5 cryostat calorimeter following ISIS advices and help. The thin calorimeter machined at ISIS and mounted in the ILL/IN5 cryostat reduced the background production but slowed down temperature changes by 3x. That was fixed by connecting thermally the bottom of the calorimeter with the heat exchanger.

ELB also presents successful results obtained on D4 with a thinner vanadium calorimeter brazed by a company working for ISIS. The total background is divided by 1.5 and flatter, easing very much data reduction and analysis.

- 7.2.2a: Non-magnetic goniometer for dilution refrigerators

  Marek Bartkowiak announces that Ruchika Yadav started working on this task the 1st of July. He compiled a list of geometrical parameters of dilution refrigerators used at European facilities with the aim to build a goniometer that can be used everywhere.

  During the summer, MB and RY worked on the thermal isolation of the sample with the goniometer (Attocube ANG 101). They simulated and 3D-printed spacers from Acryl, Nylon and VespelSP22. PEEK is also envisaged in a near future.

  EB enquires about rigidity and specs. MB tells that the goniometer will allow tilting samples by ±3.5°. A discussion follows on techniques to isolate and ensure rigidity. KK proposes to look at available porous materials and experienced issues with destructive torques encountered with ferromagnetic samples.

  [**ACTION**] PSI team to summarise the survey and share conclusions with partners.

  [**ACTION**] PSI team to prepare a prototype by April 2017.

- 7.2.2b: Fast cooling furnace

  Chris Goodway presents a comparison of cooling methods from 1000°C to room temperature in ILL-type furnaces. Flowing the gas from the top or bottom of the vacuum vessel give similar results. The gas will therefore be injected from the top (no tube in beam).

  CG et al. also measured argon and helium exchange gas evacuation times. With a standard pump used at ISIS, pumping helium takes approximately 5 min more than pumping argon. That is negligible compared to the few hours gained during cool down and helium is adopted as the most appropriate coolant.

  ELB enquires about long-term lifetime of heating elements. CG is confident with vanadium furnaces (a few units are in operation for a few months already). However, Nb furnaces are less used at ISIS and it is difficult to provide an answer for the moment. During the tests, the flow-rate was of 10 L/min. A helium recovery line might be used but pollution would quickly lead to the destruction of the heating elements.

  [**ACTION**] ISIS team to automate the flowing technique and determine the optimum flow rate.

Task 7.3: next generation pressure cells for neutron and muon research

- 7.3.1: Improved muon spectroscopy piston cell

  Rustem Khasanov announces that they published a review of µSR experiments performed at high pressure (High Pressure Research 36:2 (2016) p140). They build a new CuBe double-wall cell and added a Teflon ring to proper seal the sample volume.

They visited K. Kamenev at the Univ. of Edinburgh after the ISIS meeting in May to learn the autofrettage technique. They then improved the technique by employing a flattened ball or plunger in combination with pistons. They also replaced one of the Ni-WC pistons with a non-magnetic WC piston. Results are extremely good: 2.1 GPa safety limit and already 3 experiments carried out at PSI.

Simulations show that the GPD instrument detector configuration might be improved to enhance the fraction of muons stopped (46 → 55%) and reduce the background (5 → 3%).

[**ACTION**] PSI team to produce CuBe double-wall cells.

[**ACTION**] PSI team to modify GPD detector configuration

[**ACTION**] PSI team to study 3-wall MP35N cell

Pabitra Biswas explains that ISIS beamlines provide larger beams and presents the design of the 4 CuBe dummy cells for RIKEN-RAL. The cells manufactured should be complete in November and the tests on beam will follow. They will optimise the muon beam momentum, number of muons stopping in the sample and the signal to background ratio. If results show that pressure experiments are possible, PB et al. will build a prototype CuBe cell with appropriate dimensions from PSI design.

[**ACTION**] ISIS team to test dummy cells on RIKEN-RAL.

- <u>7.3.2.a Anvil pressure cells for µSR</u>

In the absence of Steve Cottrell (broken clavicle), Rustem Khasanov summarises the situation.

KK and ELB did not find solutions allowing to build anvil pressure cells that are compact enough to fit in existing instruments and provide better specs than new CuBe cells. ELB also envisaged to rebuild Goncharenko's cells but BA explains that these cells used Sapphire anvils able to sustain 6 GPa. The technique employed to release the strains accumulated during the crystal growth was lost with the Russian company and today Sapphire crystals can only sustain 4 GPa.

In the past, PSI team did also some tests showing that the use of anvil cells on a muon beamline is very challenging. Only 20% of the muons were stopped at the sample.

So clearly, from discussions held with 3 high-pressure experts and µSR experts, only the construction of a dedicated instrument would allow exploiting anvil pressure cells on a muon beamline.

[**ACTION**] SC to produce a report (deliverable D7.8) summarising discussions and explaining why D7.16 cannot be delivered.

- 7.3.2b: Novel anvils for Paris-Edinburgh cells

  In the absence of Stefan Klotz (conference on high-pressure research), ELB presents the results obtained on the powder diffractometer D20 at ILL. A standard Paris-Edinburgh configuration with cBN anvils compressing Fe_2O_3 sample in Fluorinert was compared with SiC-sintered diamond anvils compressing the same sample in argon transmitting media.

  The first interesting result is that SiC-SD anvils produce only a few Bragg peaks. It is therefore possible to refine sample structures without too much difficulty. The second result is that difficulties were encountered to eliminate the background. A significant background reduction was obtained by adding hBN+B_4C slits in front of the cell. However, the intensity of the sample Bragg peaks is lowered. Further tests will be performed next week on the same instrument.

- 7.3.3: Novel non-magnetic, neutron, clamp cell

  Mark Kibble contacted facilities to determine the outer diameters of high-pressure cells that would be compatible with most cryostats and cryomagnets. To summarise, the most common bore size for magnets and cryostats is Ø48 mm to Ø50 mm diameter. So obviously, a Ø47 mm diameter would be a good starting dimension. For working under higher magnetic fields, the cell will have to be less than Ø30 mm diameter, which will reduce the maximum pressure.

  The manufacture of the standard material test pieces is complete. They are either Ø18 mm x 5 mm thick (disc) or Ø18 mm x 75 mm (bar). S. Klotz and K. Kamenev provided Ni/Cr alloy samples. The background and transmission measurements will be performed at the end of this year and at the beginning of 2017. There is however some uncertainty as buffer beamtime might have to be used for backlogged experiments. The measurements will be performed at ISIS and at ILL.

  The collected data will be sent to VL with the aim to check the new McStas component prepared within task 7.2.1.

  [**ACTION**] ISIS and ILL teams to organise and perform transmission and background measurements of test pieces.

  [**ACTION**] ISIS and ILL teams to send collected data to VL with complete description of the instruments configurations.

- 7.3.4: 700 bar H_2 container for studying storage equipment

  KP informs participants that the science case will finally not require access to the imaging technique. Efforts are concentrated in the design of a sample cell that is compatible with SANS instruments. The gas handling system, already used on x-ray beamlines, should be suitable for carrying out experiments at neutron facilities. KP presents a photo of a prototype sample cell made from Sapphire. The facility for leak testing is now available and pressure tests are going to start.

ELB suggests KP to contact safety engineers of neutron facilities well in advance to ensure that the 700 bar H_2 equipment will be approved.

[**ACTION**] HZG team to check safety requirements of several neutron facilities.

[**ACTION**] HZG team to determine max pressure accessible with the prototype.

Task 7.4: Complementary in-situ measurements for neutron and muon experiments

- 7.4.1: In-situ muonium studies

  Pabitra Biswas informs that the rig for deoxygenating/handling liquid samples has been refurbished and is being recommissioned. A new ceramic sample cell and a centred stick, compatible with both instruments, have been developed and shall be commissioned on beam before the end of the year. They will determine the loading/unloading capabilities of the pump, the deoxygenation efficiency and the stability of samples.

  The design of a new variable temperature insert able to hold large ceramic cells and with in-situ liquid loading capability is considered. New RF coils compatible with this system shall also be studied.

  [**ACTION**] ISIS team to launch the second recruitment as soon as possible.

  [**ACTION**] ISIS team to commission the liquid sample handling system.

  [**ACTION**] ISIS team to design variable temperature insert and RF coils.

- 7.4.2: In-situ NMR setup

  ELB presents the slides provided by Patrick Judeinstein. The low-field NMR technique is operational at the lab and efforts focus on the construction of a sample cell compatible with SANS and imaging techniques. The system will allow to measure proton density profiles, proton relaxation times (T_1 and T_2) and self-diffusion coefficients without prior model with the field-gradient technique.

  Despite some issues encountered during the vacations, Rodrigo de Oliveira Souza (Brazilian) started working as post-doc on the 1st of September. ELB contacted ILL colleagues working on the horizontal reflectometer. The conclusion of the discussions is that there seems to be no scientific case for using NMR on a horizontal reflectometer as the spatial resolution of 15 µm is far too large.

  [**ACTION**] LLB team to determine the specs of the sample cell from SANS/Imaging instruments scientists working at facilities of partners.

A.O.B.

ELB thanks very much the speakers and all participants for their active contribution.

As discussed in January, ELB contacted partners with the aim to organise a common purchase of TiZr ingots. BA provided documents explaining why we should reduce the admissible amount of oxygen as well as other elements. ELB will update the specs accordingly and shall contact companies to get quotations. ESS, ILL and LLB requested ingots and PSI will tell as soon as possible whether they need some.

ELB remind partners that a list of deliverables must be provided by March 2017. They should be delivered on time, except those related with task 7.4. It is proposed and agreed to postpone them by 12 months.

ELB informs that 3 reports will have to be produced in 2017: report to EC in March, report to external experts appointed by EC in September and a SINE2020 monitoring report in December. He thanks in advance partners to help him produce them on time.

ELB also presents the budget situation known in July 2016. There is no staff cost shown for CSEC and IMPMC as we were waiting for an answer of the EC project officer. TUM and HZB did not charge any staff cost until now. HZB and ESS have agreed to spend the money over the whole period of the contract to ensure a smooth evolution of task 7.1.

[**ACTION**] PSI team to determine TiZr ingots needs before end of September.

[**ACTION**] Partners to provide list of TiZr suppliers before end of September.

[**ACTION**] Participants to provide early October 2016 the number of person.months charged to the project and costs spent.

Participants

Coimbra University

WP7 — 5th Meeting

7 September 2016 — Agenda

Each progress report will be followed by discussions with partners on forthcoming actions.

Task 7.1 — FLOSS software standards for sample environment	
09:00	Klaus Kiefer (HZB) — SECoP and SE standard metadata

Task 7.2 — Towards efficient sample environment for neutron research	
09:30	Victor Laliena (CSIC) — Signal to background ratio improvements (7.2.1)
10:00	Eddy Lelièvre-Berna (ILL) — Signal to background ratio improvements (7.2.1)
10:30	Coffee break
11:00	Marek Bartkowiak (PSI) — Non-magnetic goniometer for dilution refrigerators (7.2.2.a)
11:30	Chris Goodway (ISIS) — Fast cooling furnaces (7.2.2.b)

Task 7.3 — Next generation pressure cells for neutron and muon research	
12:00	Rustem Khasanov (PSI) — Improved muon spectroscopy piston cell (7.3.1)
12:20	Rustem Khasanov (PSI) — Anvil cell suitable for neutron and μSR (7.3.2a)
12:40	Buffet lunch
13:40	Pabitra Biswas (ISIS) — High pressure cell for μSR (7.3.2.a)
13:50	Eddy Lelièvre-Berna (ILL) — Novel anvils for Paris-Edinburgh cells (7.3.2.b)
14:00	Mark Kibble (ISIS) — Novel non-magnetic, neutron, clamp cell (7.3.3)
14:15	Klaus Pranzas (HZG) — 700 bar H_2 container for studying storage equipment (7.3.4)

Task 7.4 — Complementary in-situ measurements for neutron and muon experiments	
14:30	Pabitra Biswas (ISIS) — In-situ muonium studies (7.4.1)
14:45	Eddy Lelièvre-Berna (ILL) — In-situ NMR setup (7.4.2)
15:00	A.O.B.
16:00	Coffee break

Coimbra University

WP7 — 5th Meeting

7 September 2016 — Agenda

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