

Annual Report of COMPRES Facilities Committee

February 2, 2018

The Facilities Committee met twice at the 2017 AGU meeting in New Orleans to review the annual reports submitted by the PIs of each COMPRES facility (with the exception of the Gas Loading Facility at the APS which was reviewed by the EOID committee because of Rivers' conflict of interest). There was a teleconference on December 11 at which the following were present: Mark Rivers (Facilities Committee Chair), Bin Chen, Arianna Gleason, Anne Pommier (Committee Members), Abby Kavner (Chair of Executive Committee), and Carl Agee (President of COMPRES). There was also an in-person meeting on December 13 at which the following were present: Mark Rivers, Dan Shim (Committee Member), Abby Kavner, and Carl Agee.

Prior to these meetings each committee member prepared written comments on each proposal. Summarized below are the issues that were discussed by the committee in these comments and at the meeting.

General points

It was agreed that in future years we should solicit information from each facility on user satisfaction. We were supposed to do so in 2016 and 2017, but this was overlooked! The synchrotron User Office should be collecting this information from end-of-run forms that each user group is asked to complete. This is definitely done at the APS and the ALS. We do not know if NSLS-II is currently collecting this information.

ALS 12.2.2

The report emphasized three main areas of new developments: single-crystal XRD system, external heating at HP, and two-sided laser-heated radial diffraction. It also discussed the recent development of a high-temperature program at ambient pressure on this beamline.

The management team is functioning efficiently. The laser heating system is reported to be working well. COMPRES continues to do an excellent job leveraging considerable funding from the ALS. COMPRES-related users are reported to have large share of the overall beamtime (50-62%). The collaboration between beamline 12.2.2 with compressed-supported IR setup led by Zhenxian Liu is good use of resources, and is a unique capability. The number of publications (42) is very good, though we estimate that less than half are relevant to the COMPRES earth-science mission.

The committee was pleased to see that Dr. Yan's report is more detailed and now reflects his efforts and slightly increased productivity (4 pubs and 5 submitted, against 4 and 4 last year).

Concerns

The single crystal program continues to develop rather slowly. It has been a significant part of each annual report since at least 2014, but there are still very few single crystal publications in earth science. It is unfortunate that there were a number of technical problems which has led to further delays in commissioning the ES1 single crystal diffraction endstation. This was supposed to have been commissioned over 9 months ago.

Has the availability of the ambient pressure high temperature capability which is mostly used by material scientists adversely affected COMPRES users getting high-pressure time? Is this a worry for the future?

There was some concern about the user-support from the beamline staff. It appears that Dr. Beavers is only assisting the single-crystal users, but they are only a small fraction of the total. There was also concern that some of the groups that Dr. Yan said he assisted were in fact only assisted by Martin Kunz. The committee would expect that whenever a COMPRES-relevant project is being run on 12.2.2 that at least one of the COMPRES supported beamline scientists should be actively participating.

Recommendations

The facility report appears to always list all of the publications from 12.2.2, not just the COMPRES relevant publications. While the Committee is OK with this, we would like to see the following in future years:

- Identification of the publications that the committee views as COMPRES relevant, i.e. that might have reasonably been funded by NSF-EAR, even if they were not.
- Identification of the publications that used the external heating system.
- Identification of the publications that used single-crystal diffraction at 12.2.2.

For the last 3 years the COMPRES report said: “The Committee recommends that 12.2.2 staff keep better records of gas loading and laser mill users ...” The ALS 12.2.2 report this year again did not include this information and we strongly encourage the authors of the report to provide this information.

Last year the Committee expressed concern that the external heating system appeared to be restricted to BX-90 diamond anvil cells. This report did not address that concern, therefore the Committee reiterates its request to get information about it.

There is a brief mention of the HPSTAR post-doctoral researcher on this beamline. The Committee would like to see more information on this. In particular, details are needed about what this scientist does, how he/she is mentored and integrated into the planning for beamline operations and upgrades.

Finally, the report says that the PIs plan to conduct a workshop at the ALS user meeting. We continue to encourage them to include the PX² group in this workshop, or perhaps another one at the COMPRES annual meeting, as well.

IXS at APS Beamline 3-ID and 30-ID

As it was the case last year, this facility continues to produce high-impact Earth science papers. There were 7 published papers, and 7 submitted, including 1 PhD thesis in 2017. The fraction of users with NSF-EAR support is high, which is excellent for COMPRES. There is a high acceptance rate for COMPRES proposals compared to general acceptance rate (55 and 43% vs. 30%). 22 grad students and 1 undergrads were involved in experiments. ~49% of the available time goes to COMPRES users, which is much larger than COMPRES share of the total beamline support costs. 18 COMPRES user groups have been allocated beamtime in the past year. The report included excellent highlights on 3 selected Earth-related studies (sound velocity and density of magnesiowüstites, stability of ferrous-iron-rich bridgmanite under reducing mid-mantle conditions, iron isotopic fractionation between silicate mantle and metallic core at high pressure). The Committee appreciates that the report is very well written, and that it presents statistics for the offline Mossbauer system Raman systems, which is something we asked the PIs to do last year.

The COMPRES supported staff, Wenli Bi, again organized a workshop Nuclear Resonance Scattering. Dr. Bi also successfully conducted her own research and is actively developing high-pressure techniques for the COMPRES users.

Concerns
None.

Recommendations

The Committee supports the request to purchase an on-line Raman system. The report did an excellent job of justifying this purchase, and it is not increasing the requested budget significantly since they have decreased the requested fraction of Dr. Bi's salary for this year.

Multi-anvil project at ASU

This facility continues to provide standard assemblies to many laboratories and to do new development for the community. They provided a large number of cell assemblies (2085) to the community in the last year, which is very impressive and demonstrates how useful this project is.

The committee was pleased to see that the report includes 21 publications for 2016 and 14 for 2017 to date. This is nearly twice the previous number reported last year. The facility is working hard to ensure that users acknowledge the COMPRES support in publications that use the cell assemblies.

The committee was also pleased to see the effort that has been made on evaluation of carbide to increase the maximum pressure available. This is partly done in support of the COMPRES DELVE project led by Dr. Yanbin Wang. It continues the systematic work that needs to be done to improve the multi-anvil technology, but which individual labs typically do not have the time and resources to do.

The efforts on crushable forsterite seem like a very good project to pursue.

The committee liked the inclusion of the 1-pagers from the 3 different facilities. The facility should continue this, and highlight different facilities each year.

Concerns

None.

Recommendations

The committee recommends that in the future the report should break down the publications by category: those resulting from the development projects, and the science papers resulting from the cell assemblies they provide to users.

GWU IR

Significant progress has been made in the commissioning of the IR beamline at NSLS-II. The experimental stations are complete, and the IR extraction port is under construction. The project appears to be on track to have IR beam and science commissioning in summer 2018, 4 years after the shutdown of NSLS.

Concerns

There were 16 publications in 2016-2017. Of these well under 50% are earth science related. The Committee hopes that once the NSLS-II system comes online the participation from the COMPRES community will increase.

The Committee is concerned about the fate of the equipment that is now at the ALS. That program has been quite successful, and since COMPRES has invested in new equipment for NSLS-II. We thus hope that the equipment can be left at the ALS even after Hemley and Liu move their operations to NSLS-II. The ALS IR system works OK but appears to be installed temporarily. The system is installed on a thin aluminum breadboard which is very unstable. It seems that with small investments the system could be much more stable and user friendly.

The report does not include the user statistics we requested in our template file provided to them. This should really be provided for 2017, and certainly this template should be used for the 2018 report when NSLS-II will be operating. The chair of the Committee sent an e-mail to Dr. Liu on Jan. 16, 2018 requesting this information. We received this information on January 27, 2018. For 2017 there were four outside groups with NSF-EAR support who used the IR lab: Quentin Williams (3 days), Dan Shim (4 days), Elizabeth Thompson (7 days), and Anne Davis (4 days).

The budget request includes funds for a second beamline scientist. However, there is no justification provided in the report for this position, though this is vital when requesting a new hire. The Committee needs to know what will this person do, and how will his/her efforts complement those of Dr. Liu.

Recommendations

The Committee does not recommend funding the second beamline scientist at this time, given the significant lack of justification in the report. We encourage the PIs to build a stronger case for a new hire as part of a future report or as response to the comments from COMPRES.

The Committee feels the workshop would be very worthwhile if it is widely advertised to the COMPRES community. We recommend that the PIs to work on the organization of the workshop. This would presumably be an EOID proposal.

Chen DAC at NSLS-II

-Science: This proposal would provide both standard and cutting-edge techniques. The new techniques (XPCS, nanoprobe) have the potential to open up new scientific areas. The match from HPSTAR is very positive aspect of the proposal. The ability to do XRD and IR in the same diamond anvil cell at the same facility is an attractive part of the proposal. However, given the limited time that DAC XRD is likely to be available, it is not clear how feasible this will be.

The Committee raised some important questions about how the PIs will actually implement some of the things they propose.

Multigrain. This is a technically challenging problem. HP-CAT has been working on this for a number of years, and as far as we know it is still not available to the general users. Who will develop this at NSLS-II? This uses single-crystal technology but with multiple grains. This means they need an accurate rotation stage, and software for single crystal data collection. There are no details on this.

PDF on melts. This is very challenging for several reasons. The amorphous scattering is weak and must be measured to high precision. This requires long acquisition times, but maintaining a stable melt in the DAC for those times is difficult. Because the scattering is weak it can be swamped by the scattering from the diamonds which are orders of magnitude thicker than the sample. For both of these reasons the melt scattering at the APS is being done in a Paris Edinburg cell, not a DAC. The PE cell allows using a Soller slit to remove the non-sample scattering, but this does not have the resolution for the smaller DAC samples. It is also much easier to maintain stable liquid with resistive rather than laser heating.

Sound velocities. What is the advantage of the IXS beamline for these sound velocity measurements compared to 3-ID and 30-ID at the APS, which COMPRES is already supporting?

-User community: It is clear that the capacity of the high-performance beamlines at the APS is insufficient for the community demand. 13-ID-D and HP-CAT undulator beamlines are heavily oversubscribed. While the performance of the XPD station will not be at the same level as these stations because of the significantly lower brightness, there are many projects that could be done well at XPD. The problem with XPD is that there is also unlikely to be a large amount of time for DAC experiments there either because they will compete for COMPRES time with the multi-anvil program, and with a very large community of non-COMPRES users who do powder diffraction.

-Management team: The team has good experience and is qualified to conduct the proposed work. There will be 1 or 2 beamline scientists on site, no other management from the team. The success of the project will depend heavily on the beamline scientists.

-Facility:

The PIs propose to use SRX for emission spectroscopy and for sub-micron imaging. SRX does not currently have a high-resolution analyzer for emission spectroscopy. It also does not yet

have sub-micron focusing, and the mirrors they purchased for that purpose have been shown to be unable to achieve sub-micron focus.

They propose in the table to use HEX for full-field imaging. This beamline will not have the optics or brightness to do this in the DAC. It can be done in the multi-anvil press.

They propose to use the side-branches of HEX for DAC work. However, the side branches are much poorer sources, because they are looking at the extended wiggler source from a non-zero angle. This makes the source size much larger than when looking on axis in the central end station, and means the source cannot be well focused in the DAC. This is exactly the problem that X-17-C had at the old NSLS.

The list of existing equipment is interesting, but not too useful without information on what items are actually in-hand and available for use at NSLS-II.

The section on the KB mirrors in the budget justification indicates a misunderstanding. They imply that the reason the mirrors that XPD plans to buy can only reach 25x10 microns is because of the mirrors themselves, while if they use mirrors from HP-CAT they can get a 5x7 micron spot size. This is completely incorrect. The reason for the larger spot size is the source, upstream optics, and beamline layout, not the mirrors themselves. Using a different set of mirrors will not help.

The PIs say they need to purchase another laser heating system because the one that COMPRES previously purchased is in use at Stony Brook. The Committee finds this argument very questionable and it cannot be accepted. COMPRES purchased this for use on the COMPRES beamlines at NSLS. It should move to NSLS-II and not be lost to Stony Brook.

There is no description of how the system will be mounted in XPD. We heard at the workshop that another PUP has been approved with their own table in the upstream part of this station. Where will the DAC system be mounted? Does it need to purchase its own table, etc?

The cost of the GSECARS/COMPRES gas loading system is \$300K from Univ. of Chicago, not \$170K as they propose. It is unlikely anyone else can build it for \$170K.

There is no explanation or justification provide for the \$30K/year consultants in the budget.

-Recommendations:

The Committee recognizes that NSLS-II management would very much like to see a DAC program at NSLS-II. There are beamlines with new opportunities for DAC research, such as high-resolution inelastic scattering, x-ray photon correlation spectroscopy, and nanoprobe. COMPRES should consider what investments might be made to make these techniques accessible to the COMPRES DAC user community. The Committee does not recommend a dedicated DAC setup at XPD without a stronger science case. There is not a strong argument based solely on capacity, since the fraction of DAC beamtime available at XPD is likely to be quite small.

PX²

PX² continues to progress quite well. Beamline developments and planned work are in excellent agreement with the user community needs. The main development in 2017 was on the laser heating system. A radiography camera was added for locating mineral inclusions in diamonds. A broad user base has been developed in the COMPRES community, and COMPRES members received ~65% of total beamtime granted under this program. The PX² program was originally planned to use 50% of the beam time on this station, but is actually using over 70%.

The Committee greatly appreciated that this was the only report that addressed point by point (in a convincing way) the recommendations from the Committee's last year report.

15 publications are reported, 11 from 2017 compared to 5 publications last year. Nearly all are high-pressure single-crystal diffraction, and approximately 65% are in earth science.

61 beamtime requests were received, compared to 45 in 2016, which shows continued growth in demand.

Przemek Dera is investing in the facility outside of COMPRES by stationing a student on a year-long stay at the APS doing single-crystal studies.

The facility is requesting increase in staff from 1 FTE to 1.5 in order to reduce the load on Dr. Zhang because it is difficult for one person to run the beamline 70% of the time. They have proposed that this be done by increasing Sergey Tkachev's support from 50% (gas loading only) to 100% (gas loading plus PX²).

Concerns

Both the ALS 12.2.2 and PX² have proposed workshops which will involve personnel from the other facility to increase coordination. COMPRES should ensure that these actually take place, even if not as originally proposed. Perhaps the COMPRES annual meeting would be the best venue.

Recommendations

The Committee recommends continuing the expansion of the COMPRES user base.

The committee recommends increasing the staffing on PX² to 1.5 FTE providing it can be demonstrated that this is supporting the COMPRES community's use of the beamline.

The committee had questions about whether the best way to provide such support is to increase Sergey Tkachev's support from COMPRES to handle these additional duties. There is concern that he appears to have a heavy load with the gas loading system. He is also currently supporting the Brillouin system on 13-BM-D, and has more expertise in that than in the single crystal diffraction. The committee wonders whether this is actually something Sergey is interested in pursuing.

NSLS XPD Multi-anvil program

The XPD beamline will provide access to high intensity monochromatic beams for the 1000-ton press. The scientific plans include ultrasonic and deformation experiments, which have been developed by this group for many years.

Concerns

Unfortunately, the progress on XPD has continued to be very slow. It is now nearly 3 years behind what was forecast in the 2014 annual report, i.e. high-pressure commissioning with beam in June 2015. Other Partner User beamlines at the NSLS-II (e.g. XFM) have been commissioned much more quickly.

In the 2016 annual report the PIs said that the following would be accomplished during the 2017-2 run (summer, 2017):

- First Science Commissioning Beamtime: 2017-2 Cycle
- Experiment Performed TBD
- Tasks to be Accomplished in Preparation for Beamtime
- Final load testing of hydraulic systems – main ram and differential pumps
- Testing heater power supply and electrical insulation of pressure toolings
- Temperature measurement system designed and installed
- Allen Bradley logic controller deployed
- Development of data collection and experiment control protocols
- Testing and optimization of anvils and sample cell assembly(ies)
- Development of on the fly preliminary data analysis tools
- Installation of any necessary shielding to minimize background scatter
- Installation and integration of acoustic velocity measurement system
- We will continue anvil development during the coming year.

To the Committee's knowledge very few if any of these tasks have been completed, and it is now the beginning of the 2018-1 run. In the current report the PIs said the following:

- 12/02-04/2017 – FIRST SCIENCE COMMISSIONING EXPERIMENT

It seems that this did not take place, and this activity has been delayed 3 months, until March 2018.

The issue of how a DT25 to work with monochromatic beam will be built is an important one, and the Committee regrets that only 2 sentences were dedicated to it. The cost for 25mm sintered diamond cubes is estimated at about 4 times the cost of tungsten carbide. What will the estimated annual costs be?

Recommendations

The following 2 recommendations from last year are still applicable:

- The project needs to produce a clear project timeline which is agreed to by NSLS-II. The Committee is very concerned that if this is not done immediately the project milestones will continue to slip. COMPRES management needs to closely monitor the project progress, perhaps with monthly status reports and phone calls.
- The Committee recommends that COMPRES and Stony Brook negotiate a final Partner User Agreement with NSLS-II as soon as possible. If the fraction of beam time for the multi-anvil program is less than the currently agreed 20% then COMPRES needs to know this for future planning.

APS 6-BM

The 6BM beamline served 36 user groups in 2017, with ~30% oversubscription. The scientific thrusts are rheology of the deep Earth and elasticity measurements with ultrasonics, addressing fundamental earth science questions. A proposal to continue operations at 6-BM-B for another year was reviewed by this committee in later 2017, and approved by the Executive Committee.

Concerns

The number of publications is still rather low. There are 6 published papers for 2017, 4 published and 2 in press (the first Kaboli paper has now been published). One of the published papers (Farla et al.) is an erratum to a 2015 paper, and was not done at 6-BM-B (or XPD). 6-BM-B has only been running for about 2 years, so hopefully more papers will begin to appear from research already conducted there.

Recommendations

The Committee recommends that the facility improve its outreach to solicit users to ensure full utilization of beamtime by as broad a COMPRES community as possible.

The future of 6BM should be left open until XPD begins to operate and we can assess the relative strengths of the two programs.

The Committee feels COMPRES needs to consider the multi-anvil programs going forward. NSF-EAR is currently funding 4 multi-anvil beamlines (13-ID-D, 13-BM-D, 6-BM-A, XPD). Could they be consolidated to use the best source and maximize beam time? The new module being developed for XPD would work better at 13-ID-D in that 1000 ton press, since it has a monochromatic undulator beam, not a wiggler. At the APS it could get more than 10% of beam time. 6-BM-B and 13-BM-D could be combined to take advantage of good mirror focusing and perhaps a 100% dedicated station.