ALS Beamline 12.2.2

2017 COMPRES Annual Report

November 2016 – October 2017

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Overview

In this last year, the staff on beamline 12.2.2 have successfully navigated potentially catastrophic beamline issues, implemented new user facilities and worked as a cohesive team to provide scientific support to a wide range of users. The ALS staff, Martin Kunz and Andrew Doran, and the COMPRES staff, Christine Beavers and Jinyuan Yan, have supported 31 distinct user groups over 70 visits, and their efforts were rewarded with the beamline having 42 peer reviewed publications in 2017. During this past year, COMPRES users were allocated 52.3% of shifts at 12.2.2 (if HPSTAR users are included as COMPRES users, this percentage rises to above 62%), which is well in excess of the 35% mandated by the COMPRES Approved Program. There continues to be a strong synergy between the COMPRES funded HP IR program (the bridging redeployment of the NSLS IR effort at the ALS) and 12.2.2, with numerous users continuing to schedule their beamtimes for the infrared beamline 1.4.3 and 12.2.2 in parallel. The high pressure lab, dedicated to sample preparation, has been in high demand- the gas loader has been used over 200 times since Jan 2016. The Nd:YAG laser at the heart of the laser miller, which has been a staple of users since its purchase by COMPRES from ARRA funds in 2009, is receiving a factory refurbishing, paid for by the ALS. The beamline itself had a strenuous year, with a monochromator issue to begin the 2017 user schedule. This was fortunately quickly resolved by Doran; the incident highlighted the age of the beamline, and the need for optical refurbishing, which ALS management has decided is a funding priority. The ALS also invested in a large format fast CMOS detector ($240k), which is currently installed on the laser heating endstation 2 (ES2) and is undergoing commissioning. In this report, we will detail the current status of the newest user facilities, radial diffraction during double sided laser heating, and the tungsten external heater, as well as the nearly fully commissioned single crystal diffractometer. We will also discuss the ambient pressure high temperature system, which has also become quite an attractive user facility. Naturally, we continue to regularly serve both “standard” polycrystalline x-ray diffraction high-pressure users, as well as users who utilize less routine techniques, including laser-heated and externally-heated diffraction at high pressures, radial diffraction users, and single crystal users.

Beamline Personnel

The core personnel responsible for beamline 12.2.2 has not changed in the last year, nor has our organizational chart. Martin Kunz (with occasional consultation from Alastair MacDowell) is the ALS-employed beamline manager, and Andrew Doran provides half-time technical support to 12.2.2. Each of these folks is ALS funded, and leveraged by the COMPRES investment in the 12.2.2 facility. On the COMPRES side, Christine Beavers and Jinyuan Yan continue to develop their respective projects and support the COMPRES community, as detailed in their individual annual reports. Other ongoing staffing included an ALS doctoral fellow (O'Bannon during the reporting period, who helped with the single-crystal set-up and shake-downs), and an HPSTAR post-doc, who is specifically dedicated to 12.2.2.

Scientific Highlights

Kapustin, E.A., S. Lee, A.S. Alshammari, and O.M. Yaghi, "Molecular Retrofitting Adapts a Metal-Organic Framework to Extreme Pressure," *ACS Cent Sci* **3**(6), 662-667 (2017). (doi:10.1021/acscentsci.7b00169)

The first highlight, from Kapustin et al, presents a high pressure single crystal experiment on a metal organic framework (MOF). MOFs are porous coordination polymers with inflexible organic ligands that interconnect metal nodes. They are primarily attractive for the available internal surface area, which has applications in gas separations, transport and storage as well as catalysis and structural characterization. For MOFs to find applications in industry, they need to be fairly rugged, and be able to resist some degradation due to environmental factors, i.e., heat and pressure. In this case, Kapustin and co-workers were aiming to produce a MOF that was more resistant to pressure. MOF-520, the focus of this study, is composed of aluminum-formate rings, ligated together with tripodal 1,3,5- benzenetribenzoate (btb). Single crystals of MOF-520 were loaded in a DAC with 4:1 methanol: ethanol as the pressure transmitting medium (PTM), and diffraction data were collected on beamline 12.2.2, from ambient pressure up to 2.82 GPa, at which pressure the crystal became amorphous. This amorphization is not unusual, in that the high pressure pushes the PTM into the pores, which degrades the long-range order of the crystal. Similar phenomena have been observed in cyclosilicates. This invasion of PTM was quantified by examining the unit cell dimensions for an increase in volume, as well as the unassigned electron count being determined using the program SQUEEZE. To stabilize the MOF crystal against the high-pressure incursions of the PTM, single crystals of MOF-520 were soaked in a saturated solution of 4,4'-biphenyldicarboxylic acid(H_2bpdc), which replaced the formate molecules, and cross-linked across the pores. This new ligand was chosen for its length, which closely matches the pores of MOF-520. A single crystal of this new compound, known as MOF-520-BPDC, was then subjected to high pressure.

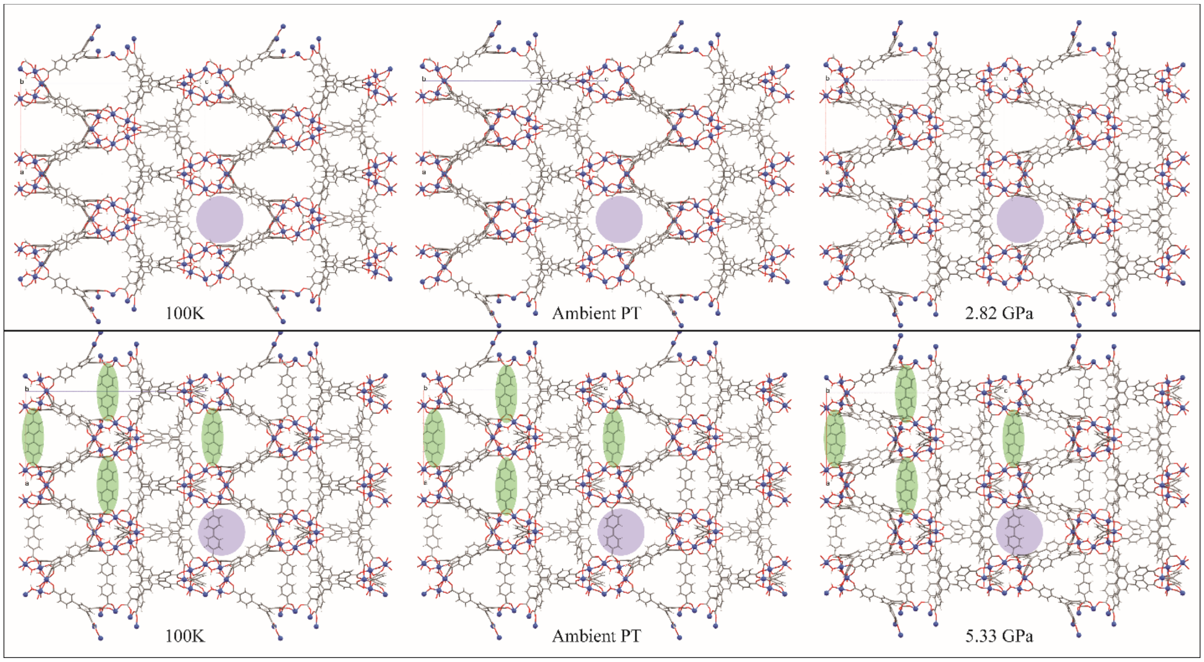

Figure 1-MOF-520 and MOF-520-BPDC, looking down the b-axis, at 100K, ambient PT and the maximum pressure reached. Aluminum atoms are shown as blue spheres, with all other atoms represented as capped sticks. Green ovals guide the eye to some of the BPDC ligands, and the purple circle serves to emphasize the pore size. Figures made in Mercury.

The structures of both MOF-520 and MOF-520-BPDC are shown above. The 100 K structures are very similar, with the BPDC inobtrusively spanning the voids (green ovals). Thermal expansion from 100K to ambient temperature leaves both structures again looking very similar, with the pore size similarity shown using purple circles. The images on the far right are the structures at the maximum pressure where the crystal was still able to diffract. MOF-520 has, by 2.82 GPa, swelled with PTM, and is now showing volume decreases, along with decreasing diffraction intensity. The bottom right shows a similar looking MOF-520-BPDC structure, but the pressure is 5.33 GPa. The distortion of the BPDC ligand seems to indicate that the crystal is near failure, but this modification has doubled the high-pressure lifetime range of this material. Using single crystal diffraction, Kapustin et al., were able to observe that a specific modification to a known MOF vastly improved its mechanical strength and stability.

This highlight is particularly chosen not for its geologic importance, but rather because it emphasizes the capabilities of the ES2 single crystal system, the construction of which has been a primary focus of the COMPRES effort at 12.2.2. The asymmetric unit of the reinforced compound, MOF-520-BPDC, contains ~45 non-hydrogen atoms. MOFs are generally known to be weakly diffracting. This successful study, along with others in press and submitted, are demonstrating that the 12.2.2

single crystal system on ES2 is mature and able to tackle both highly complex and weakly diffracting crystallographic systems under pressure.

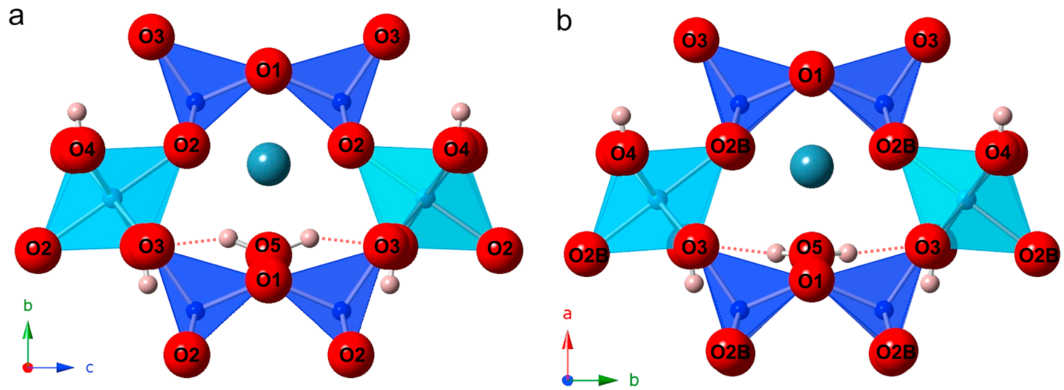

Figure 2- a view of the ambient (left) and high pressure (right) structures of lawsonite. The changes are extremely subtle, and would be impossible to determine without single crystal diffraction (the water molecule's orientation shifts, the Si-O1-Si angle changes, and the Ca ion shifts in the big site). Image from O'Bannon, E.F., C.M. Beavers, M. Kunz, and Q. Williams, "The high-pressure phase of lawsonite: A single crystal study of a key mantle hydrous phase," *J. Geophys. Res. Solid Earth* **122**(8), 6294-6305 (2017). (doi:10.1002/2017JB014344)

There are many other excellent papers from 12.2.2 to choose highlights from, but it seems fitting to highlight another single crystal paper, with a high degree of earth science relevance, but also associated with considerable crystallographic complexity. Lawsonite was the first mineral to be explored on the ES2 system- its journey to publication was mirrored by the single crystal program's journey to becoming a user facility. The high pressure phase behaviour of lawsonite had been speculated on for nearly 20 years, but had not received the clarity that single crystal structures can provide. The transition to monoclinic symmetry near 9 GPa had been observed using both spectroscopy and powder diffraction, but the mechanism of the transition and the resulting atomic rearrangements were still unclear. Due to lawsonite's status as a potential water bearer to the deep earth, there were also lingering questions about the hydroxide units and water molecules in the structure and their hydrogen bonding configurations. The high pressure study of lawsonite, with its determination of the structure of the high-pressure phase, has removed some of this uncertainty, and states that monoclinic lawsonite is "likely to be a major water carrier in colder, deep subducted slabs." This stability is tied to the high pressure phase's larger entropy, which is inferred from the increase in symmetry independent atoms, and the continued dynamic disorder in the hydroxide and water molecules. This level of detailed insight into the high pressure structure is only attainable through single crystal diffraction.

Overview of developments, completed and in progress (1): Single Crystal Program Status

The high pressure single crystal program on beamline 12.2.2 can be split into two flavors- the single axis system on endstation 2 (ES2), which has been in operation since late 2013, and the new Stoe Stadi-vari Eulerian cradle diffractometer, which is positioned on endstation 1 (ES1). The ES2 system, which currently uses the Perkin-Elmer amorphous silicon detector, has been used by a growing number of groups, and is considered a mature user facility. Single crystal users in the last year include Jackson (CIT), Soghomonian (VPI), Parsons (U. Edinburgh), Brown (UW), Yaghi (UCB) and Williams (UCSC) (Jackson, Brown and Williams are COMPRES affiliates). This system is currently undergoing an upgrade, with the new RDI CMOS8 detector. This change should be transparent to the user- the user interface and the output files will be nearly identical, but the spatial resolution and the residual noise will be obviously improved.

Last year, the status of the single crystal diffractometer in ES1 was reported as follows:

In terms of our hardware, the Stoe Stadi-Vari 4-circle diffractometer equipped with an RDI CMOS fast detector was purchased by the ALS in fall 2015 (the diffractometer was $181 K from ALS/DOE funding, and the CMOS detector was also ALS funded at $75 K: these were, of course, partially leveraged by COMPRES' commitment to this beamline). The diffractometer was installed in spring 2016 on a temporary three-point mount, to allow for rough alignment and system optimization. COMPRES awarded funds from its EOID program ($85 K) to purchase robust stages (and a custom collimator) to accurately position the diffractometer on the beam focus point in spring 2016; these stages have now arrived, and will be installed during beam down-time in December 2016.

Unfortunately, there was a fabrication error in the diffractometer bracket, which was not noticed until the diffractometer was reassembled on the mount, on the beamline. This limited the travel of the diffractometer on the new Aerotech stages, due to the mount being 3 mm too tall. This issue was remedied by the time user beam resumed in March, but valuable commissioning time in December was lost.

The ALS was shutdown from January until March to allow for a major upgrade to the storage ring RF system. The most fraught part of the upgrade was the replacement of analog components with new digital systems, which required a steep learning curve for the accelerator physicists. The impact of this upgrade was felt far into the calendar year, and by the end of April, many of the planned commissioning days had been lost to a major RF issue that required cancelling user beam for 3 days (April 26-28th). The next ES1 commissioning session was planned for May 10th & 11th, but on the evening of the 9th, the entire LBL site was plunged into darkness for six hours by an external power outage. Synchrotron light sources have difficulties recovering from power outages, and this was the case for the ALS- the RF system was damaged in the outage, and useful user beam did not return until the 12^{th}. By this time, we were hesitant to schedule any further ES1 commissioning time, lest it summon an earthquake or tsunami, but the reality was there was little time left on the 2017 schedule.

Effective commissioning requires multiple consecutive days, due to the endstation switch being non-trivial. To this end, a block of two weeks has been put aside for commissioning in January 2018. This final commissioning has three main goals: to test the automated sample alignment routines that have been adapted from the ES2 system, to determine the appropriate data collection defaults, and to streamline the data analysis process. Barring some exotic accelerator crisis, the single crystal diffractometer should be (finally) fully commissioned for the spring.

In incidental news, the 12.2.2 beamline also obtained a spare MAR-345 detector in April that was being surplused out of a lab in San Diego. That involved a trip to pack it and bring it back up to LBNL, and was enabled by Beavers' connections in the single-crystal community.

Overview of developments (2): Sample Heating Modalities

Achieving deep-earth relevant, aka, elevated temperatures has been a constant struggle for non-axial geometries or mantle-relevant, but lower-than-radiant-laser-heating temperatures. The axial laser heating system is stable and dependable, but not every experiment is in axial geometry, and not every experiment requires temperatures above 1000°C. In this last year, strides forward have been made for both of these situations.

The compact laser heating system currently in use on 12.2.2 was designed to enable imaging and laser heating on the upstream and downstream sides in axial geometry, and, with the addition of an arm of optics, at 90° and 270° in radial geometry. At this time last year, it was becoming obvious that the 270° arm for dual sided laser heating in radial geometry was not functional. The sample could be imaged at room temperature, but the image would blur as soon as the IR lasers were used. Some component along the laser path was heating, causing either lengthening or distortion from the optical axis. The solution was to build a mini laser table at the sample height, and affix the necessary optics on this isolated platform. After some initial adjustment, this platform has remained nearly aligned through multiple cycles of placing and removing onto ES2. This system is now available to users, and has been used by the Wenk, Williams and Miyagi groups.

Heating samples to temperatures around 1000-1500°C in a DAC is difficult on multiple levels. Laser heating is not ideal, not only because coupling can be poor at the low power levels required, but also because any temperature quantification will have large errors, because of the weak and largely infrared pyrometry signal. Historically, what has typically been done in these temperature regimes has been external heating with a band heater, but that requires scarily large power supplies, good grounding, surplus courage, and a renewable supply of DACs as they get heated and oxidized. Jinyuan Yan, one of the COMPRES staff members has been working on an internal (to the DAC) and external(to the sample chamber) heater. It has been tested up to 1500°C, and has been consistently used at 1000°C. This heater has been used successfully by Yan in support of HPSTAR, and numerous other groups are interested.

Variable temperature, ambient pressure powder experiments have become accessible in the last year on beamline 12.2.2, due to the 12.2.2-designed lamp furnace, which was described earlier this year ("Compact low power infrared tube furnace for in situ X-ray powder diffraction," Rev. Sci. Instrum. 88(1), 013903 (2017). (doi:10.1063/1.4973561)). This facility has enabled many studies of materials undergoing phase transitions and chemical reactions, some with time resolution that allowed for kinetic understanding. It is distinguished by its compactness and temperature control, and has been deployed to temperatures of 1200°C. This facility has been primarily used by materials scientists and chemists, but it may be attractive to earth and planetary scientists: in particular, this system can readily allow characterization of devolatilization reactions at ambient pressures.

Obviously, a primary development direction moving forward is a marriage between two of our main development foci, high-pressure single-crystal x-ray diffraction and external heating. Such measurements have, of course, previously been made, but our goal is to make them a routine capability, rather than an occasional, high degree of difficulty one-off. We have had some initial successes in this arena that have also served to illustrate some of the difficulties: these include stable sample positioning, and ensuring that heating is highly localized (and, for example, does not extend into the load-bearing portions of the diffractometer). Because of the importance of pursuing accurate single crystal structures of materials at simultaneous high-pressure and high-temperature conditions, we anticipate that this will be an area of emphasis in the coming year.

Beamline Operations

(1) Number of beamtime proposals received

Cycle 1-2017: 46 (+2 Approved Programs), 17 of which are COMPRES, 11 from HP*

Cycle 2-2017: 64 (+1 AP), 15 COMPRES, 29 HP*

(2) Number of beamtime proposals granted beamtime

Cycle 1-2017: 22, 9 COMPRES, 1 HP*

Cycle 2-2017: 25, 11 COMPRES, 4 HP*

(3) Total number of shifts requested

Cycle 1-2017: 391 (+ 36 for APs), 142 COMPRES, 96 HP*

Cycle 2-2017: 529 (+14 for Gurlo AP), 116 COMPRES, 257 HP*

(4) Total number of shifts granted

Cycle 1-2017: 187 (incl. APs), 66 COMPRES, 30 HP* (24 AP shifts + 6 GU shifts)

Cycle 2-2017: 212 (incl. AP), 96 COMPRES, 30 HP*

(5) Total number of shifts available

Cycle 1-2017: as (4): 187

Cycle 2-2017: as (4): 212

(6) Oversubscription rate (= shifts requested / shifts available)

Cycle 1-2017: 2.59 (2.15 COMPRES)

Cycle 2-2017: 2.67 (1.21 COMPRES)

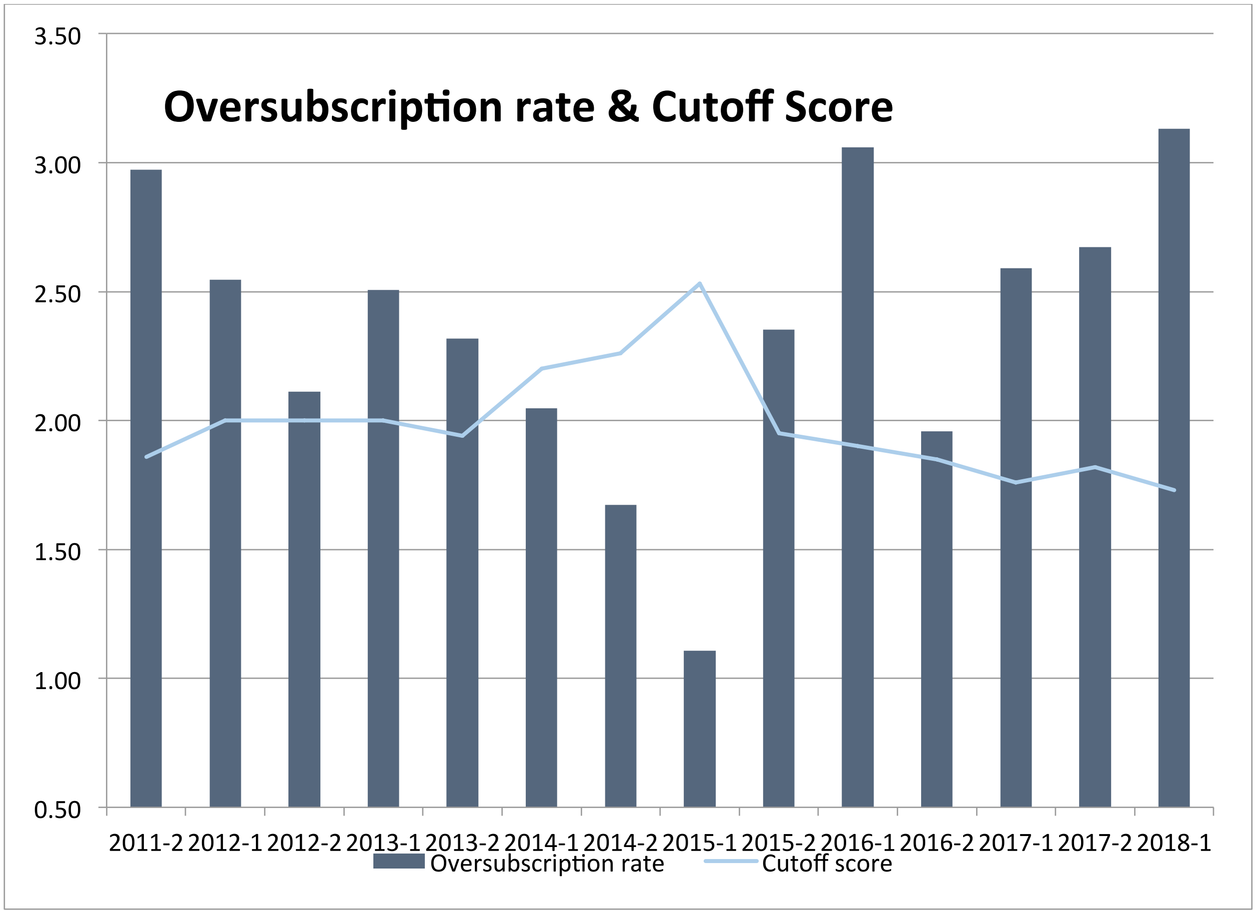

Figure 3. Oversubscription and Cutoff scores for 12.2.2. Proposals are rated on a 1-5 scale, with 1 being the highest and 5 being the lowest. For reference, 2015-1 was the cycle in which the ALS User office shifted their PI notification policies in stealth mode: proposal PIs, who had always been sent personalized email reminders to resubmit their proposals, only received one generic "call for proposals" email. This change, which was not recognized until after the proposal deadline caused a dramatic dip in proposals around-the-ring.

Performance Metrics

In the Nov 2016- present span, we had 29 distinct research groups visit the ALS to use 12.2.2 in 70 different visits. The funding sources and days allocated for these groups are listed in the performance metrics section. Our user office reports that we had visits from 87 distinct badged users, comprised of 14 faculty, 39 GSRA, 14 post-docs, 13 scientists/technical staff, and 7 undergrads. Person visits, as we understand them, are less straightforward to track (especially for local groups, a shifting cast of characters shows up at different times), but our estimate is that we had about 160 person visits.

COMPRES users made up more than 62% of allocated shifts over the course of Nov 2016-Nov 2017 if HPSTAR usage is included in the COMPRES count (some HPSTAR beamtime is clearly COMPRES-related: for example, collaborative work with H.K. Mao can be viewed as COMPRES time, since he is associated with a COMPRES institution: we lack, however, the knowledge of which (or how much) HPSTAR beamtime is used for which projects to pro-rate the HPSTAR beamtime according to its COMPRES relevance); and 52.3% without HPSTAR. In either case, the COMPRES allocated time far exceeds the mandated 35% dictated by the COMPRES AP.

The table below shows the different groups, their time allotments, their country, and their funding sources over the last year. These are subdivided into the three beamtime cycles that are spanned by the report period. Director's Discretionary Time is not included in this table: during the past year, one COMPRES user received DD time (J.M. Brown, U. Washington).

ALS 12.2.2 2016-2 11/1/16-12/31/16

PI Name	# days	Beam time	COMPRES User ©	Funding Source	Country	NSF-EAR	NSF-DMR	NSF-Chem	DOE	DOD	NSF-China	NNSA	Foreign
Gurlo(AP)	1	11/1;		Foreign (NSF)	Germany								1
HP*	4	12/7-12/11		Foreign (NSF)	China						1		1
Jackson	4	11/16-11/20	C	NSF-EAR	USA	1							
Jacob	2	11/30-12/2		Foreign (NSF)	Australia								1
Kavner	3	11/11-11/14	C	NSF-EAR	USA	1							
Mattox	1	12/14;		DOE	USA				1				
Miyagi	3.66	11/3-11/6	C	NSF-EAR	USA	1							
Montiero	1.67	12/20-12/21		DOE	USA				1				
Shim	2	11/9-11/11	C	NSF-EAR	USA	1							
Stavrou	1.67	11/22-11/23		NNSA/DOE	USA				1			1	
Turner	2	12/17-12/19	C	NSF-EAR	USA	1							
Wenk	2	11/2; 12/6	C	NSF-EAR	USA	1							
Willams	4	11/15;12/2-12/5	C	NSF-EAR	USA	1							
	32	14	64.56%										
	2017-1	1/1/17-7/31/17											
PI Name	# days	Beam Time											
Ciezak-Jenkins	4	5/3-5/5;6/2-6/4		DOD	USA					1			
Godwal	2	4/28;5/25	C	DOE	USA				1				
Gurlo(AP)	4	6/7;6/13;7/13;7/18		NSF-EAR	USA	1							
HP*(AP)	8	4/13-4/17;6/8-6/12		Foreign (NSF)	China								
Jackson	2	5/5-5/7	C	NSF-EAR	USA	1							
Karundasa	3.33	4/1-4/2;5/2;6/1;	C	NSF-Chem	USA			1					
Long	2.67	4/30;5/21;7/30		NSF-Chem	USA			1					
Miyagi	3	7/20-7/23	C	NSF-EAR	USA	1							
Montiero	2	6/14-6/16		DOE	USA				1				
Parsons	1	4/19;		Foreign (NSF)	UK								1
Reagan	3	4/6-4/9	C	NSF-EAR	USA	1							
Santamaria-Perez	3	4/21-4/23		Foreign (NSF)	Spain								1
Shim	2	5/17-5/19	C	NSF-EAR	USA	1							
Soghomonian	2	4/4-4/6		NSF-DMR	USA		1						
Stavrou	5	6/16-6/18;7/15-7/17		NNSA/DOE	USA				1			1	
Tolbert	3	5/12-5/15	C	NSF-DMR	USA		1						
Wenk	2.67	3/24-3/26	C	NSF-EAR	USA	1							
Williams	5.33	3/24;3/31;4/20;5/19-5/21;5/26;6/16;7/14	C	NSF-EAR	USA	1							
Yaghi	2	4/2;4/29;		DOE	USA				1				
Yang	2	5/27-5/28		NSF-DMR	USA		1						
Zeng	2	7/28-7/30	C	Foreign (NSF)	China						1		1
	64	39											
	2017-2	8/1/2017-11/1/2017	44.27%										
PI Name	# days	Beam Time											
Alivisatos	1	8/15;		DOE	USA				1				
Beavers	4.67	**	C	NSF-DMR	USA								
Burtch	3	10/12-10/15		DOE	USA				1				
Dionne	1	8/16;		NSF-Chem	USA			1					
Godwal	1.67	8/5-8/6	C	DOE	USA				1				
HP*(AP)	9	8/17-8/20;9/8-9/11; 9/28; 10/5; 10/18		Foreign (NSF)	China						1		1
Long	1	9/23;		NSF-Chem	USA			1					
Miyagi	4	10/26-10/30	C	NSF-EAR	USA	1							
Parsons	2.67	4/19;		Foreign (NSF)	UK								
Soghomonian	2	9/21-9/23		NSF-DMR	USA		1						
Tolbert	3	10/6-10/9	C	NSF-DMR	USA		1						
W.Mao	3	8/2-8/5	C	NSF-EAR	USA	1							
Wenk	3	8/10-8/13	C	NSF-EAR	USA	1							
Williams	5	7/1-7/2; 7/18-7/19	C	NSF-EAR	USA								
	44.01	17	55.31%	Totals	36	11	5	4	7	1	2	1	7
						22.45%	10.20%	8.16%	14.29%	2.04%	4.08%	2.04%	19.44%
						NSF-EAR	NSF-DMR	NSF-Chem	DOE	DOD	NSF-China	NNSA*	Foreign

**Beavers' time is 0.5 of the BL scientist time, and her dates were not differentiated from MK's
All time was allocated through the GU panel, with the exception of the HPSTAR Approved program which was allocated 15% of the time in 2016-2 and 2017-1. HPSTAR now operates under the COMPRES model, and has to compete for time.

List of Users (Group Leaders) with Affiliations 11/1/15 to present:

P. Alivisatos (UCB Chemistry/LBNL)
J.M. Brown (U. Washington: Director's Discretionary Time)
N. Burtch (Sandia National Lab)
J. Ciezak-Jenkins (Aberdeen Proving Ground)
J. Dionne (Stanford)
R. Ewing /K. Turner (Stanford)
B. Godwal (UC Berkeley, R. Jeanloz collaboration)
A. Gurlo (AP)((Ceramics, TU Berlin)
HP* (Rotating cast of ~15 people)
J. Jackson(Caltech)
D. Jacob (S. Clark collaboration, Macquarie U.)
H. Karunadasa (W. Mao collaboration, Stanford)
A. Kavner (UCLA)(including Santamaria-Perez time)
M. Kunz (ALS/LBNL)
J. Long (UC Berkeley Chemistry)
T. Mattox (LBNL)
W.Mao, including Reagan and Zeng time (Stanford)
L. Miyagi (Utah)
P. Monteiro (UCB Materials Science)
S. Parsons (U. Edinburgh)
S.-H. Shim (ASU)
V. Soghomonian (VPI)
E. Stavrou (LLNL)
S. Tolbert (UCLA)
H.R. Wenk (UC Berkeley)
Q. Williams including O'Bannon and Yan time (UCSC)
O. Yaghi (UC Berkeley Chemistry)
S. Yang (Southern U.)

Beamline Community Activities

The professional activities of the beamline scientists are described in detail in their personal statements. This includes active engagement in a range of scientific meetings (at which an explicit goal is to promote the beamline, and its capabilities), and Beavers has done service in a range of capacities, including as a member of the High Pressure Proposal Review Panel at the APS. Moving forward, we are in communication with the PX^3 group about conducting a high-pressure single-crystal workshop (in addition to ongoing sharing of expertise between COMPRES groups in the single-crystal domain). The general consensus was to conduct the workshop in conjunction with the

2018 ALS users meeting, and Beavers is preparing an EOID proposal to partially fund that workshop.

Budget Justification for Next Cycle:

The budget that we request for the coming year is identical to that requested within the COMPRES-IV renewal. In passing, we note that our requests for supplies and travel are substantially reduced relative to prior years by the annual fiscal targets given to us in conjunction with that renewal.

Staff: We request funding for two beamline scientists deployed for user support and instrumentation development from June 1, 2018 to May 31, 2019. This complement is viewed by ALS management as a match to the ALS staff that are deployed at 12.2.2, thus leveraging a substantial development and user support community. Christine Beavers is presently at the Assistant Researcher V level (the promotion process to Associate Researcher has been initiated), and Jinyuan Yan is presently at the Associate Project Scientist III level. Dr. Beavers has designed and developed the single-crystal capabilities at 12.2.2, and is the primary user support person for single-crystal diffraction experiments (she also assists other users, as well). Dr. Yan provides software developmental work and day-to-day user support, and has worked on the development of external heating systems. The beamline scientists' achievements over the last year are described in more detail in their annual statements/inventory of work for COMPRES. Critically, both employees routinely do a range of user-critical tasks at the beamline, such as maintenance and retuning of the off-line ruby fluorescence system, supporting users in the operation of the laser-miller and the gas-loader (or, more frequently, loading gasses for users themselves, which ultimately usually saves time and cost), and a range of miscellaneous trouble-shooting associated with both our sample preparation lab and the beamline. Hence, this request represents *status quo* staffing of the facility moving forward.

Supplies and Expendables: A modest budget is requested for expendables utilized by COMPRES users at the beamline. This includes items such as replacement parts for equipment utilized by users (such as the gas-loading apparatus, the laser miller, sample preparation equipment, pressure measurement apparatus, gasket materials, and locally-made external heaters).

Travel:

A budget for travel is requested. This will be utilized primarily for the beamline scientists to attend meetings (including the COMPRES meeting: these are important for their professional development, their knowledge of new techniques, and for acquiring new potential users of the beamline), and to occasionally visit other major facilities to share best practices and to exchange expertise. We have enhanced (as per recommendations of the COMPRES Facilities Committee) our level of communication and coordination with complementary enterprises at the Advanced Photon Source, and we expect that a portion of the travel budget (if it is sufficient) will fund coordinating trips to that facility.

NSF Budget Year 2

A. Senior Personnel			NSF Funded Person Months CAL	ACAD	SUM	Funds
1.	Christine Beavers Assoc. Researcher II		12	0	0	$99,088
2.	Jinyuan Yan Assoc. Project Scientist		12	0	0	$83,387
3.			0	0	0	$0
4.			0	0	0	$0
5.			0	0	0	$0
6.	0	Others (list individually on budget explanation page)	0	0	0	$0
7.	2	Total Senior Personnel (1 through 6)	0	0	0	$182,475
B. Other Personnel						
1.	0	Post Doctoral Associates	0	0	0	$0
2.	0	Other Professional (Technician, Programmer, etc)	0	0	0	$0
3.		Graduate Students				$0
4.		Undergraduate Students				$0
5.	0	Secretarial-Clerical (if charged directly)				$0
6.	0	Other				$0
					Total Salaries and Wages (A+B)	$182,475
C. Fringe Benefits (If charged as direct costs)						$88,314
					Total Salaries, Wages and Fringe Benefits (A+B+C)	$270,789
D. Equipment (list item and dollar amount for each item exceeding $5,000)						
					Total Equipment	$0
E. Travel						
1. Domestic (Including U.S. Possessions)						$3,129
2. Foreign						$1,387
F. Participant Support						
1. Stipends		$0				
2. Travel		$0				
3. Subsistence		$0				
4. Other		$0				
		Total Participant Support				$0
G. Other Direct Costs						
1. Materials and Supplies						$3,301
2. Publication Costs/Documentation/Dissemination						$0
3. Consultant Services						$0
4. Computer Services						$0
5. Subawards						$0
6. Other						$0
					Total Other Direct Costs (G1 through G6)	$3,301
H. Total Direct Costs (A through G)						$278,606
I. Indirect Costs (F&A) (Specify Rate and Base)						
		26.0%	x	$278,606	Base	$72,437
J. Total Direct and Indirect Costs (H+I)						$351,043
K. Small Business Fee						
L. Amount of this request (J or J-K)						$351,043

Appendix 1. Publications for 12.2.2 for 2016 & 2017

2016 Publications and Theses (33)

Refereed Journal Articles (29)

1. Bae, Y.e., E.n. Cho, F. Qiu, D.T. Sun, T.E. Williams, J.J. Urban, and W.L. Queen, "Transparent Metal,-Organic Framework/Polymer Mixed Matrix Membranes as Water Vapor Barriers," *ACS Applied Materials* **8**(16), 10098-1010 (2016). (doi:10.1021/acsami.6b01299) 12.2.2

2. Barreda-Argüeso, J.o., F. Aguado, J. González, R. Valiente, L. Nataf, M.N. Sanz-Ortiz, and F. Rodríguez, "Crystal-Field Theory Validity Through Local (and Bulk) Compressibilities in CoF_2 and $KCoF_3$," *Journal of Physical Chemistry C* **120**(33), 18788-1879 (2016). (doi:10.1021/acs.jpcc.6b06132) 12.2.2

3. Borstad, G.M., I.G. Batyrev, and J. Ciezak-Jenkins, "Cyanoacetohydrazide under Pressure: Chemical Changes in a Hydrogen-Bonded Material," *Journal of Physical Chemistry A* **120**(17), 2712-2719 (2016). (doi:10.1021/acs.jpca.5b11954) 12.2.2

4. Chen, Y., S. Zhang, W. Gao, F. Ke, J. Yan, B. Saha, C. Ko, B. Chen, J.W. Ager III, W. Walukiewicz, R. Jeanloz, and J. Wu, "Pressure-induced structural transition of CdxZn1-xO alloys," *Applied Physics Letters* **108**(15), 152105 (2016). (doi:10.1063/1.4947022) 12.2.2

5. Ciezak-Jenkins, J., "High-pressure polymorphism of the electrochemically active organic molecule tetrahydroxy-p-benzoquinone," *J. Mol. Struct.* **1119**, 71-77 (2016). (doi:10.1016/j.molstruc.2016.04.062) 12.2.2

6. Du, W., S.a. Clark, and D. Walker, "Excess mixing volume, microstrain, and stability of pyrope-grossular garnets," *Am. Mineral.* **101**(1), 193-204 (2016). (doi:10.2138/am-2016-5128) 12.2.2

7. Gleissner, J., D. Errandonea, A. Segura, J. Pellicer-Porres, M.A. Hakeem, J.E. Proctor, S.V. Raju, R.S. Kumar, P. Rodríguez-Hernández, A. Muñoz, S. Lopez-Moreno, and M. Bettinelli, "Monazite-type $SrCrO_4$ under compression," *Physical Review B* **94**(13), 134108 (2016). (doi:10.1103/PhysRevB.94.134108) 12.2.2

8. Hong, F., B. Yue, Z. Chen, M. Kunz, B. Chen, and H.-K. Mao, "High pressure polymorphs and amorphization of upconversion host material $NaY(WO_4)_2$," *Applied Physics Letters* **109**, 041907 (2016). (doi:10.1063/1.4960104) 12.2.2

9. Jaffe, A., Y. Lin, C.M. Beavers, J. Voss, W.L. Mao, and H.I. Karunadasa, "High-Pressure Single-Crystal Structures of 3D Lead-Halide Hybrid Perovskites and Pressure Effects on their Electronic and Optical Properties," *ACS Cent Sci* **2**(4), 201-209 (2016). (doi:10.1021/acscentsci.6b00055) 11.3.1, 12.2.2

10. Lech, A.T., C. Turner, J. Lei, R. Mohammadi, S.H. Tolbert, and R.B. Kaner, "Superhard Rhenium/Tungsten Diboride Solid Solutions," *Journal of the American Chemical Society* **138**(43), 14398-1440 (2016). (doi:10.1021/jacs.6b08616) 12.2.2

11. Liu, G., L. Kong, J. Yan, Z. Liu, H. Zhang, P. Lei, T. Xu, H.-k. Mao, and B. Chen, "Nanocrystals in compression: unexpected structural phase transition and amorphization due to surface impurities," *Nanoscale* **8**(23), 11803-11809 (2016). (doi:10.1039/C5NR09027J) 12.2.2

12. Luz, I., A. Loiudice, D.T. Sun, W.L. Queen, and R. Buonsanti, "Understanding the Formation Mechanism of Metal Nanocrystal@MOF-74 Hybrids," *Chem. Mater.* **28**(11), 3839-3849 (2016). (doi:10.1021/acs.chemmater.6b00880) 12.2.2

13. Miyagi, L., and H.-R. Wenk, "Texture development and slip systems in bridgmanite and bridgmanite + ferropericlase aggregates," *Physics and Chemistry of Minerals* **43**(8), 597-613 (2016). (doi:10.1007/s00269-016-0820-y) 12.2.2

14. O'Bannon, E.F., and Q. Williams, "Beryl-II, a high-pressure phase of beryl: Raman and luminescence spectroscopy to 16.4 GPa," *Physics and Chemistry of Minerals* **43**(9), 671-687 (2016). (doi:10.1007/s00269-016-0837-2) 12.2.2

15. Raju, S.V., B.K. Godwal, J. Yan, R. Jeanloz, and S.K. Saxena, "Yield strength of Ni,-Al,-Cr superalloy under pressure," *J. Alloys Cmpd.* **657**, 889-892 (2016). (doi:10.1016/j.jallcom.2015.10.092) 12.2.2

16. Rasmussen, A.M., E. Mafi, W. Zhu, Y.i. Gu, and M.D. McCluskey, "High pressure Œ≥-to-β phase transition in bulk and nanocrystalline In_2Se_3," *High Pressure Research* **36**(4), 549-556 (2016). (doi:10.1080/08957959.2016.1214729) 12.2.2

17. Reagan, M.M., A.E. Gleason, L. Daemen, Y. Xiao, and W.L. Mao, "High-pressure behavior of the polymorphs of FeOOH," *Am. Mineral.* **101**(6), 1483-1488 (2016). (doi:10.2138/am-2016-5449) 12.2.2

18. Santamaria-Perez, D., C. McGuire, A. Makhluf, A. Kavner, R. Chuliá-Jordan, J.L. Jorda, F. Rey, J. Pellicer-Porres, D. Martinez-García, P. Rodriguez-Hernández, and A. Muñoz, "Correspondence: Strongly-driven Re+CO_2 redox reaction at high-pressure and high-temperature," *Nature Communications* **7**, 13647 (2016). (doi:10.1038/ncomms13647) 12.2.2

19. Santamaría-Pérez, D., C. McGuire, A. Makhluf, A. Kavner, R. Chuliá-Jordán, J. Pellicer-Porres, D. Martinez-García, A. Doran, M. Kunz, P. Rodríguez-Hernández, and A. Muñoz, "Exploring the Chemical Reactivity between Carbon Dioxide and Three Transition Metals (Au, Pt, and Re) at High-Pressure, High-Temperature Conditions," *Inorganic Chemistry* **55**(20), 10793-1079 (2016). (doi:10.1021/acs.inorgchem.6b01858) 12.2.2

20. Solomatova, N.V., J.M. Jackson, W. Sturhahn, J.K. Wicks, J. Zhao, T.S. Toellner, B. Kalkan, and W.M. Steinhardt, "Equation of state and spin crossover of (Mg,Fe)O at high pressure, with implications for explaining topographic relief at the core-mantle boundary," *Am. Mineral.* **101**(5), 1084-1093 (2016). (doi:10.2138/am-2016-5510) 12.2.2

21. Stavrou, E., Y. Yao, J.M. Zaug, S. Bastea, B. Kalkan, Z. Konopkova, and M. Kunz, "High-pressure X-ray diffraction, Raman, and computational studies of $MgCl_2$ up to 1 Mbar: Extensive pressure stability of the β-$MgCl_2$ layered structure," *Scientific Reports* **6**, 30631 (2016). (doi:10.1038/srep30631) 12.2.2

22. Steiner, M.H., E.M. Hausrath, M.E. Elwood Madden, O. Tschauner, B.L. Ehlmann, A.A. Olsen, S.R. Gainey, and J.S. Smith, "Dissolution of nontronite in chloride brines and implications for the aqueous history of Mars," *Geochim. Cosmochim. Acta* **195**, 259-276 (2016). (doi:10.1016/j.gca.2016.08.035) 12.2.2

23. Su, N.C., D.T. Sun, C.M. Beavers, D.K. Britt, W.L. Queen, and J... Urban, “Enhanced permeation arising from dual transport pathways in hybrid polymer,-MOF membranes,” *Energy Environ. Sci.* **9**, 922-931 (2016). (doi:10.1039/C5EE02660A) 12.2.2

24. Tschauner, O., S.V. Ushakov, A. Navrotsky, and L. Boatner, “Phase transformations and indications for acoustic mode softening in Tb-Gd orthophosphate,” *Journal of Physics: Condensed Matter* **28**(3), 035403 (2016). (doi:10.1088/0953-8984/28/3/035403) 12.2.2

25. Umeyama, D., Y.u. Lin, and H.I. Karunadasa, “Red-to-Black Piezochromism in a Compressible Pb,-I,-SCN Layered Perovskite,” *Chem. Mater.* **28**(10), 3241-3244 (2016). (doi:10.1021/acs.chemmater.6b01147) 12.2.2

26. Wang, Y., L. Wang, H. Zheng, K. Li, M. Andrzejewski, T. Hattori, A. Sano-Furukawa, A. Katrusiak, Y. Meng, F. Liao, F. Hong, and H.-k. Mao, “Phase Transitions and Polymerization of C_6H_6-C_6F_6 Cocrystal under Extreme Conditions,” *Journal of Physical Chemistry C* **120**(51), 29510-29519 (2016). (doi:10.1021/acs.jpcc.6b11245) 12.2.2

27. Yeung, M.T., J. Lei, R. Mohammadi, C. Turner, Y. Wang, S.H. Tolbert, and R.B. Kaner, “Superhard Monoborides: Hardness Enhancement through Alloying in $W_{1,\text{âíx}}Ta_xB$,” *Advanced Materials* **28**(32), 6993-6998 (2016). (doi:10.1002/adma.201601187) 12.2.2

28. Yue, B., F. Hong, S. Merkel, D. Tan, J. Yan, B. Chen, and H.-K. Mao, “Deformation Behavior across the Zircon-Scheelite Phase Transition,” *Physical Review Letters* **117**(13), 135701 (2016). (doi:10.1103/PhysRevLett.117.135701) 12.2.2

29. Zhao, Z., H. Wei, and W.L. Mao, “Pressure tuning the lattice and optical response of silver sulfide,” *Applied Physics Letters* **108**(26), 261902 (2016). (doi:10.1063/1.4954801) 12.2.2

Refereed Conference Proceedings (0)

Theses (M.S., Ph.D., etc.) (4)

1. Palaich, S.E., “Carbon in the Deep Earth: A Mineral Physics Perspective,” Doctoral Dissertation, Univerisity of California, Los Angeles, Los Angeles, CA, USA, 2016, advisor Abby Kavner. 12.2.2

2. Rasmussen, A.M., “Pressure-induced Phase Transitions of Indium Selenide,” Doctoral Dissertation, Washington State University, Pullman, WA, 2016, advisor Matthew D. McCluskey. 12.2.2

3. Rodenbough, P.P., “Crystallite Size Dependency of the Pressure and Temperature Response in Nanoparticles of Ceria and Other Oxides,” Doctoral Dissertation, Columbia University, New York, NY, 2016, advisor Siu-Wai Chan. 12.2.2

4. Zhang, D., “Applications of nuclear resonant scattering to further our understanding of Earth’s interior,” Doctoral Dissertation, California Institute of Technology, Pasadena, CA, 2016, advisor Jennifer Jackson. 12.2.2

Non-refereed Publications (magazine article, book review, etc.)(0)

2017 Publications and Theses (47: Posted through 11/12/17)

Refereed Journal Articles (42)

1. Abramson, E.H., O. Bollengier, and J.M. Brown, "Water-carbon dioxide solid phase equilibria at pressures above 4 GPa," *Scientific Reports* **7**(1), 821 (2017). (doi:10.1038/s41598-017-00915-0) 12.2.2

2. Adcock, C.T., O. Tschauner, E.M. Hausrath, A. Udry, S.N. Luo, Y. Cai, M. Ren, A. Lanzirotti, M. Newville, M. Kunz, and C. Lin, "Shock-transformation of whitlockite to merrillite and the implications for meteoritic phosphate," *Nature Communications* **8**, 14667 (2017). (doi:10.1038/ncomms14667) 12.2.2

3. Bae, S., R. Taylor, D. Kilcoyne, J. Moon, and P. Monteiro, "Effects of Incorporating High-Volume Fly Ash into Tricalcium Silicate on the Degree of Silicate Polymerization and Aluminum Substitution for Silicon in Calcium Silicate Hydrate," *Materials* **10**(2), 131 (2017). (doi:10.3390/ma10020131) 5.3.2.1, 5.3.2.2, 12.2.2

4. Borstad, G.M., and J.A. Ciezak-Jenkins, "Hydrogen-Bonding Modification in Biuret Under Pressure," *Journal of Physical Chemistry A* **121**(4), 762-770 (2017). (doi:10.1021/acs.jpca.6b09670) 12.2.2

5. Cai, W., M. Dunuwille, J. He, T.V. Taylor, J.K. Hinton, M.C. MacLean, J.J. Molaison, A.M. Dos Santos, S. Sinogeikin, and S. Deemyad, "Deuterium Isotope Effects in Polymerization of Benzene under Pressure," *The Journal of Physical Chemistry Letters* **8**(8), 1856-1864 (2017). (doi:10.1021/acs.jpclett.7b00536) 12.2.2

6. Cai, W., R. Zhang, Y. Yao, and S. Deemyad, "Piezochromism and structural and electronic properties of benz[a]anthracene under pressure," *Phys. Chem. Chem. Phys.* **19**(8), 6216-6223 (2017). (doi:10.1039/C6CP08171A) 12.2.2

7. Chen, Y., F. Ke, P. Ci, C. Ko, T. Park, S. Saremi, H. Liu, Y. Lee, J. Suh, L.W. Martin, J.W. Ager, B. Chen, and J. Wu, "Pressurizing Field-Effect Transistors of Few-Layer MoS_2 in a Diamond Anvil Cell," *Nano Letters* **17**(1), 194-199 (2017). (doi:10.1021/acs.nanolett.6b03785) 12.2.2

8. Chen, Y., S. Zhang, F. Ke, C. Ko, S. Lee, K. Liu, B. Chen, J.W. Ager, R. Jeanloz, V. Eyert, and J. Wu, "Pressure-Temperature Phase Diagram of Vanadium Dioxide," *Nano Letters* **17**(4), 2512-2516 (2017). (doi:10.1021/acs.nanolett.7b00233) 12.2.2

9. Ci, P., Y. Chen, J. Kang, R. Suzuki, H.S. Choe, J. Suh, C. Ko, T. Park, K. Shen, Y. Iwasa, S. Tongay, J.W. Ager, L.-W. Wang, and J. Wu, "Quantifying van der Waals Interactions in Layered Transition Metal Dichalcogenides from Pressure-Enhanced Valence Band Splitting," *Nano Letters* **17**(8), 4982-4988 (2017). (doi:10.1021/acs.nanolett.7b02159) 12.2.2

10. Ciezak-Jenkins, J.A., and T.A. Jenkins, "Shear induced weakening of the hydrogen bonding lattice of the energetic material 5,5,Ä≤-Hydrazinebistetrazole at high-pressure," *J. Mol. Struct.* **1129**, 313-318 (2017). (doi:10.1016/j.molstruc.2016.09.084) 12.2.2

11. Ciezak-Jenkins, J.A., B.A. Steele, G.M. Borstad, and I.I. Oleynik, "Structural and spectroscopic studies of nitrogen-carbon monoxide mixtures: Photochemical response and observation of a novel phase," *The Journal of Chemical Physics* **146**(18), 184309 (2017). (doi:10.1063/1.4983040) 12.2.2

12. Ciezak-Jenkins, J.A., G.M. Borstad, and I.G. Batyrev, “Characterization of the Isothermal Compression Behavior of LLM-172,” *Journal of Physical Chemistry A* **121**(22), 4263-4271 (2017). (doi:10.1021/acs.jpca.7b03300) 12.2.2

13. Doran, A., L. Schlicker, C.M. Beavers, S. Bhat, M.F. Bekheet, and A. Gurlo, “Compact low power infrared tube furnace for in situ X-ray powder diffraction,” *Rev. Sci. Instrum.* **88**(1), 013903 (2017). (doi:10.1063/1.4973561) 12.2.2

14. E. Vennari, C., E.F. O’Bannon, and Q. Williams, “The ammonium ion in a silicate under compression: infrared spectroscopy and powder X-ray diffraction of $NH_4AlSi_3O_8$--buddingtonite to 30 GPa,” *Physics and Chemistry of Minerals* **44**(2), 149-161 (2017). (doi:10.1007/s00269-016-0844-3) 12.2.2

15. Geng, G., R.J. Myers, J. Li, R. Maboudian, C. Carraro, D.A. Shapiro, and P.M. Monteiro, “Aluminum-induced dreierketten chain cross-links increase the mechanical properties of nanocrystalline calcium aluminosilicate hydrate,” *Scientific Reports* **7**, 44032 (2017). (doi:10.1038/srep44032) 5.3.2.1, 12.2.2

16. Geng, G., R.J. Myers, M.J. Qomi, and P. Monteiro, “Densification of the interlayer spacing governs the bynanomechanical properties of calcium-silicate-hydrate,” *Scientific Reports* **7**(1), 10986 (2017). (doi:10.1038/s41598-017-11146-8) 12.2.2

17. Gomis, O., B. Lavina, P. Rodríguez-Hernández, A. Muñoz, R. Errandonea, D. Errandonea, and M. Bettinelli, “High-pressure structural, elastic, and thermodynamic properties of zircon-type $HoPO_4$ and $TmPO_4$,” *Journal of Physics: Condensed Matter* **29**(9), 095401 (2017). (doi:10.1088/1361-648X/aa516a) 12.2.2

18. Groome, C., I. Roh, T.M. Mattox, and J.J. Urban, “Effects of Size and Structural Defects on the Vibrational Properties of Lanthanum Hexaboride Nanocrystals,” *ACS Omega* **2**(5), 2248-2254 (2017). (doi:10.1021/acsomega.7b00263) 12.2.2

19. Hong, F., B. Yue, Z. Liu, B. Chen, and H.-K. Mao, “Pressure-driven semiconductor-semiconductor transition and its structural origin in oxygen vacancy ordered $SrCoO_{2.5}$,” *Physical Review B* **95**(2), 024115 (2017). (doi:10.1103/PhysRevB.95.024115) 12.2.2

20. Jaffe, A., Y. Lin, W.L. Mao, and H.I. Karunadasa, “Pressure-Induced Metallization of the Halide Perovskite $(CH_3NH_3)PbI_3$,” *Journal of the American Chemical Society* **139**(12), 4330-4333 (2017). (doi:10.1021/jacs.7b01162) 12.2.2

21. Kapustin, E.A., S. Lee, A.S. Alshammari, and O.M. Yaghi, “Molecular Retrofitting Adapts a Metal-Organic Framework to Extreme Pressure,” *ACS Cent Sci* **3**(6), 662-667 (2017). (doi:10.1021/acscentsci.7b00169) 12.2.2

22. Köck, E.-M., M. Kogler, T. Götsch, L. Schlicker, M.F. Bekheet, A. Doran, A. Gurlo, B. Klötzer, B. Petermüller, D. Schildhammer, N. Yigit, and S. Penner, “Surface chemistry of pure tetragonal ZrO_2 and gas-phase dependence of the tetragonal-to-monoclinic ZrO_2 transformation,” *Dalton Trans.* **46**(14), 4554-4570 (2017). (doi:10.1039/C6DT04847A) 12.2.2

23. Köck, E.-M., M. Kogler, C. Zhuo, L. Schlicker, M.F. Bekheet, A. Doran, A. Gurlo, and S. Penner, “Surface Chemistry and Stability of Metastable Corundum-Type In_2O_3,” *Phys. Chem. Chem. Phys.* **19**(29), 19407-19419 (2017). (doi:10.1039/C7CP03632A) 12.2.2

24. Mattox, T.M., C. Groome, A. Doran, C.M. Beavers, and J.J. Urban, “Anion-mediated negative thermal expansion in lanthanum hexaboride,” *Solid State Commun.* **265**, 47-51 (2017). (doi:10.1016/j.ssc.2017.07.012) 12.2.2

25. Nisr, C., Y. Meng, A.A. MacDowell, J. Yan, V. Prakapenka, and S.-H. Shim, “Thermal expansion of SiC at high pressure-temperature and implications for thermal convection in the deep interiors of carbide exoplanets,” *Journal of Geophysical Research: Planets* **122**(1), 124-133 (2017). (doi:10.1002/2016JE005158) 12.2.2

26. O'Bannon, E.F., C.M. Beavers, M. Kunz, and Q. Williams, “The high-pressure phase of lawsonite: A single crystal study of a key mantle hydrous phase,” **122**(8), 6294-6305 (2017). (doi:10.1002/2017JB014344) 11.3.1, 12.2.2

27. Raju, S.V., R. Hrubiak, V. Drozd, and S. Saxena, “Laser-assisted processing of Ni-Al-Co-Ti under high pressure,” *Mater. Manuf. Processes* **32**(14), 1606-1611 (2017). (doi:10.1080/10426914.2016.1269913) 12.2.2

28. Rittman, D.R., S. Park, C.L. Tracy, L. Zhang, R.I. Palomares, M. Lang, A. Navrotsky, W.L. Mao, and R.C. Ewing, “Structure and bulk modulus of Ln-doped UO_2 (Ln = La, Nd) at high pressure,” *Journal of Nuclear Materials* **490**, 28-33 (2017). (doi:10.1016/j.jnucmat.2017.04.007) 12.2.2

29. Rittman, D.R., K.M. Turner, S. Park, A.F. Fuentes, J. Yan, R.C. Ewing, and W.L. Mao, “High-pressure behavior of $A_2B_2O_7$ pyrochlore (A=Eu, Dy; B=Ti, Zr),” *J. Appl. Phys.* **121**(4), 045902 (2017). (doi:10.1063/1.4974871) 12.2.2

30. Rittman, D.R., K.M. Turner, S. Park, A.F. Fuentes, C. Park, R.C. Ewing, and W.L. Mao, “Strain engineered pyrochlore at high pressure,” *Scientific Reports* **7**(1), 2236 (2017). (doi:10.1038/s41598-017-02637-9) 12.2.2

31. Rodenbough, P.P., and S.-W. Chan, “Crystallite-size dependency of the pressure and temperature response in nanoparticles of magnesia,” *Journal of Nanoparticle Research* **19**(7), 241 (2017). (doi:10.1007/s11051-017-3922-7) 12.2.2

32. Ryu, Y.-J., C.-S. Yoo, M. Kim, X. Yong, J. Tse, S.K. Lee, and E.J. Kim, “Hydrogen-Doped Polymeric Carbon Monoxide at High Pressure,” *Journal of Physical Chemistry C* **121**(18), 10078-1008 (2017). (doi:10.1021/acs.jpcc.7b01506) 12.2.2

33. Santamaría-Pérez, D., T. Marqueño, S. MacLeod, J. Ruiz-Fuertes, D. Daisenberger, R. Chuliá-Jordan, D. Errandonea, J.L. Jordá, F. Rey, C. McGuire, A. Mahkluf, A. Kavner, and C. Popescu, “Structural Evolution of CO_2-Filled Pure Silica LTA Zeolite under High-Pressure High-Temperature Conditions,” *Chem. Mater.* **29**(10), 4502-4510 (2017). (doi:10.1021/acs.chemmater.7b01158) 12.2.2

34. Schlicker, L., M.F. Bekheet, and A. Gurlo, “Scaled-up solvothermal synthesis of nanosized metastable indium oxyhydroxide (InOOH) and corundum-type rhombohedral indium oxide (rh-In_2O_3),” *Z. Kristallogr.* **232**(1-3), 129-140 (2017). (doi:10.1515/zkri-2016-1967) 12.2.2

35. Slavney, A.H., R.W. Smaha, I.C. Smith, A. Jaffe, D. Umeyama, and H.I. Karunadasa, “Chemical Approaches to Addressing the Instability and Toxicity of Lead-Halide Perovskite Absorbers,” *Inorganic Chemistry* **56**(1), 46-55 (2017). (doi:10.1021/acs.inorgchem.6b01336) 11.3.1, 12.2.2

36. Stavrou, E., J.M. Zaug, S. Bastea, and M. Kunz, “A study of tantalum pentoxide Ta_2O_5 structures up to 28 GPa,” *J. Appl. Phys.* **121**(17), 175901 (2017). (doi:10.1063/1.4982708) 12.2.2

37. Steele, B.A., E. Stavrou, J.C. Crowhurst, J.M. Zaug, V.B. Prakapenka, and I.I. Oleynik, “High-Pressure Synthesis of a Pentazolate Salt,” *Chem. Mater.* **29**(2), 735-741 (2017). (doi:10.1021/acs.chemmater.6b04538) 12.2.2

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Refereed Conference Proceedings (2)

1. Götsch, T., C. Praty, M. Grünbacher, L. Schlicker, M.F. Bekheet, A. Doran, A. Gurlo, M. Tada, H. Matsui, N. Ishiguro, B. Kloetzer, and S. Penner, "Iron Exsolution Phenomena in Lanthanum Strontium Ferrite SOFC Anodes," *ECS Transactions* **78**(1), 1327-1341 (2017). [Proceedings of SOFC-XV: 15th International Symposium on Solid Oxide Fuel Cells, (Hollywood, FL, 2017)]. (doi:10.1149/07801.1327ecst) 12.2.2

2. Steele, B.A., E. Stavrous, V.B. Prakapenka, H. Radousky, J. Zaug, J.C. Crowhurst, and I.I. Oleynik, "Cesium pentazolate: A new nitrogen-rich energetic material," *AIP Conference Proceedings* **1793**, 040016 (2017). [Proceedings of American Physical Society Topical Group on Shock Compression of Condensed Matter, (Tampa Bay, Florida, 2015)]. (doi:10.1063/1.4971510) 12.2.2

Theses (M.S., Ph.D., etc.) (3)

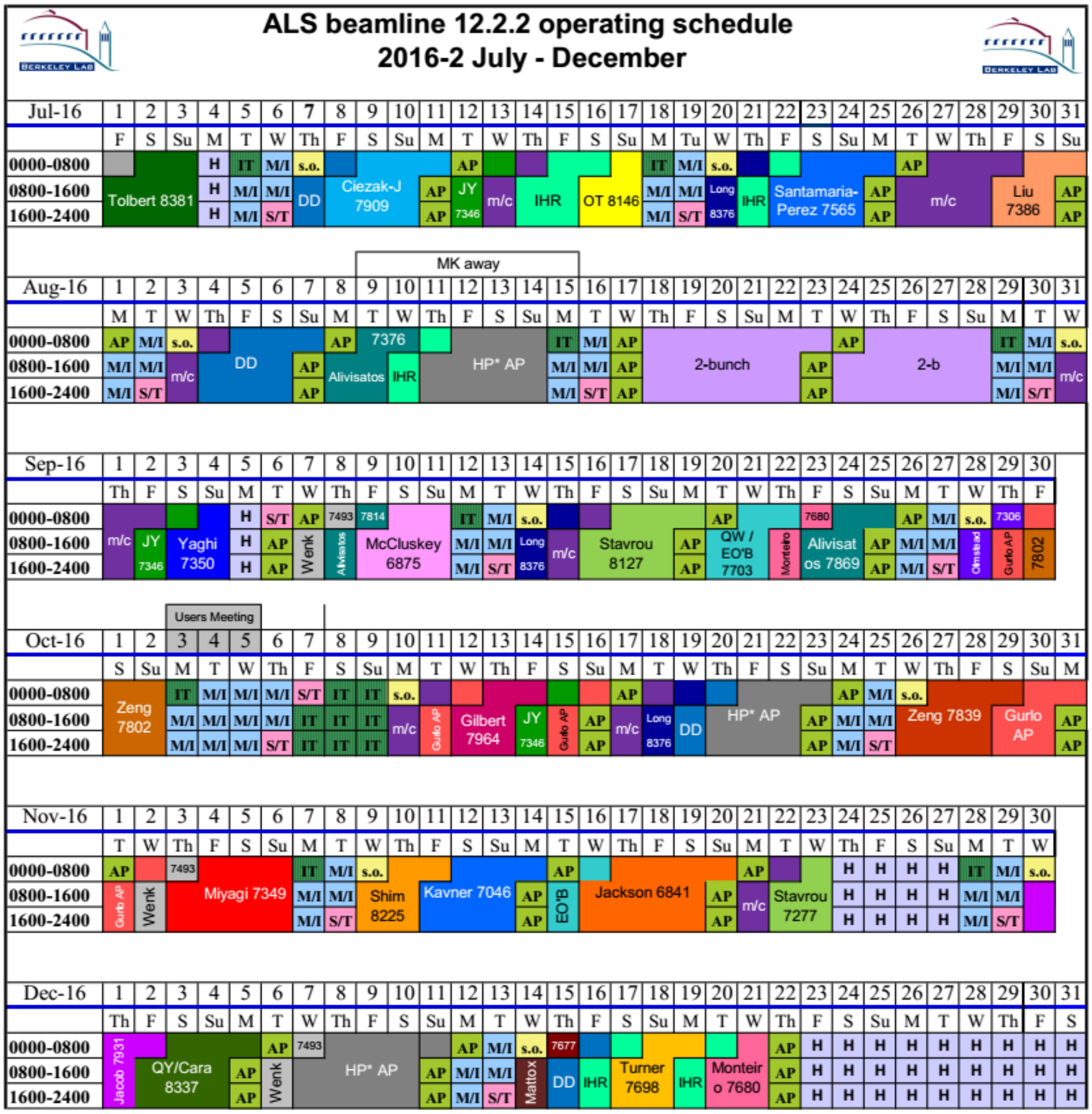
1. O'Bannon, E.F., "High-pressure studies of subduction zone related mineral phases," Doctoral Dissertation, University of California Santa Cruz, Santa Cruz, CA, 2017, advisor Quentin Williams. 12.2.2, 11.3.1, 1.4

2. Zepeda-Alarcon, E., "Texture Development and Polycrystal Plasticity of Two-Phase Aggregates," Doctoral Dissertation, University of California, Berkeley, Berkeley, California, USA, 2017, advisor Hans-Rudolf Wenk. 12.2.2

3. Zhou, X., "Study on the plastic deformation and elastic properties of nano metal," Doctoral Dissertation, Center for High Pressure Science and Technology Advanced Research (HPSTAR), Shanghai, China, 2017, advisor Bin Chen. 12.2.2, 12.3.2

Non-refereed Publications (magazine article, book review, etc.)(0)

Appendix 2: The beamtime schedules for the last year's cycles



ADVANCED LIGHT SOURCE

COMPRES

Beamline 12.2.2 Operating Schedule Jan-July 2017-1

Jan-17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T
0000-0800	H	H	I	I	I	I	I	I	I	I	I	I	I	I	I	H	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I
0800-1600	H	H	I	I	I	I	I	I	I	I	I	I	I	I	I	H	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I
1600-2400	H	H	I	I	I	I	I	I	I	I	I	I	I	I	I	H	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I

Feb-17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T
0000-0800	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	H	I	I	I	I	I	I	I	I
0800-1600	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	H	I	I	I	I	I	I	I	I
1600-2400	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	H	I	I	I	I	I	I	I	I

Mar-17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F
0000-0800	I	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	BLC	BLC	BLC	BLC	BLC							IT	M	s.o.		DD
0800-1600	I	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	BLC	BLC	BLC	BLC	BLC			m/c		JY	Wenk 7493		M	M	m/c	DD	JY
1600-2400	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	S/T	BLC	BLC	BLC	BLC	BLC								M	S/T			

Apr-17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su
0000-0800	Hema			AP	Sogho					AP	M	s.o.						AP	DD					IT	M	s.o.				
0800-1600		Ya	AP	monian			Reagan		AP	M	M	m/c			HP-*		AP	DD	SP			Santamaria-Perez 7565		M	M	m/c	IHR	BG		JL
1600-2400		ghi	AP	8668			8732		AP	M	S/T						AP			JY				M	S/T				OY	

May-17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
																										ACA					
	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W
0000-0800		AP						AP	M	s.o.						AP	DD					IT	M	s.o.					H	S/T	AP
0800-1600	AP	HK	Ciezak-J		Jackson		AP	M	M	m/c	IHR	Tolbert 8381			AP	DD	Shim		William		Lo	M	M	m/c	BG	JY	Yang 8261		H	AP	m/c
1600-2400	AP		8531		8826		AP	M	S/T						AP		8227		s 7703		ng	M	S/T						H	AP	

Jun-17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F
0000-0800					AP	M	s.o.						AP						IT	M							AP			
0800-1600	HK	Ciezak-J		AP	M	M	AP Gu rlo			HP-*		AP	AP Gu rlo	Monteiro		JY	Stavrou 8127		M	M			2-bunch			AP			2-bunch	
1600-2400		7909		AP	M	S/T						AP		7680					M	S/T						AP				

Jul-17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
									COMPRES 2017																						
	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M
0000-0800	2-bunch		AP	H	S/T					IT	M	s.o.						AP						AP	M	s.o.		DD			
0800-1600			AP	H			2-bunch			M	M	m/c	AP Gu rlo	JY	Stavrou 7277		AP	AP Gu rlo	IHR		Miyagi 7349		AP	M	M	m/c	DD	Zeng 7839		Lo ng	AP
1600-2400			AP	H						M	S/T						AP						AP	M	S/T						AP

ALS beamline 12.2.2 operating schedule

August - December 2017

Aug-17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th
0000-0800	AP	8968	s.o.				IT	M/I	s.o.					8847	AP	7814	9020				AP	s.o.							AP	M/I	S/T
0800-1600	QW	Mao (Yu Lin) 8702			Godwal 8743		M/I	M/I	m/c	Wenk 8954			SP	AP	PA	JD	Chen HP* 9027			AP	M/I	Ciezak-J 8531		m/c	DD	m/c	IHR	AP	M/I	M/I	S/T
1600-2400							M/I	S/T						AP						AP	S/T							AP	M/I	S/T	S/T

Sep-17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S
0000-0800	S/T	S/T	S/T	H	M/I	S/T	S/T					AP		8968	8847		IT	AP	M/I	s.o.				8376		AP			9071	
0800-1600	S/T	S/T	S/T	H	M/I	S/T	m/c	Zengh HP* 8980			AP	DD	QW	SP	Shim 8225		AP	M/I	M/I	m/c	Sohom. 8668		JL	IHR	AP	m/c	IHR	Li	QW - EFO 7703	
1600-2400	S/T	S/T	S/T	H	S/T	S/T					AP						AP	M/I	S/T						AP			HP*		

		Users Meeting																													
Oct - 17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T
0000-0800	7703	IT	M/I	s.o.		9247				AP						AP	M/I	s.o.	9247		7703	IT	IT	AP							M/I
0800-1600	SP	M/I	M/I	m/c	Yue	Tolbert 8381			AP	IHR	DD	Burtch 9054			AP	M/I	M/I	Yue	IHR	QW / EFO	IT	IT	IT	m/c	DD		Miyagi 7349			M/I	M/I
1600-2400	8847	M/I	S/T		HP*				AP						AP	M/I	S/T	HP*			IT	IT	AP							M/I	S/T

Nov-17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th
0000-0800	s.o.	9247	8847				AP						AP	Sfty	M/I	s.o.		8732		8376	8968		H	H	H	H	S/T	AP		
0800-1600	Yue	SP	QW / Vennaro 8337			AP	m/c	Jackson 8826				AP	Sfty	M/I	M/I	m/c	Mao (Reagan)		JL	QW	AP	IHR	H	H	H	H	AP	m/c	AP	IHR
1600-2400	HP*	8847				AP						AP	Sfty	M/I	S/T						Gurlc	IT	H	H	H	H	AP		Gurlc	

Dec-17	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su	M	T	W	Th	F	S	Su
0000-0800					M/I	AP	s.o.				8376	AP									AP		H	H	H	H	H	H	H	H	H
0800-1600	Monteiro 7680			M/I	M/I	AP	AP	Stavrou 8127		JL	AP	IHR	AP	IHR	DD	IHR	Zeng HP* 7839			AP	AP	DD	H	H	H	H	H	H	H	H	H
1600-2400				M/I	S/T	AP	Gurlc				AP		Gurlc							AP	Gurlc		H	H	H	H	H	H	H	H	H