**Partnership for eXtreme Xtallography (PX^2) and COMPRES Technology Center (COMPTECH)** 2014 COMPRES Annual Report

November 2013 – October 2014

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**Overview**

*PX^2*

The goal of the PX^2 project is to establish a new COMPRES-supported experimental facility for high-pressure mineral physics research at the Argonne National Lab that will be operated in partnership with GSECARS and the University of Hawaii. The primary target user base for this new facility are researchers that have been conducting Earth science focused research at the NSLS X17-DAC facility, whose access to beam time will be significantly limited after the closure of NSLS in September 2014. The new instrument at the existing 13BMC experimental station will offer excellent capabilities for conducting powder diffraction in diamond anvil cell, which are in the highest demand at the current NSLS X17-DAC facility. Because of the unique instrumentation available in the new station (6-circle Newport diffractometer) some of the experimental characteristics for these experiments, including resolution/peak width and angular coverage will be dramatically improved, compared to the current NSLS setup. Besides the focus on powder diffraction, the new station will offer currently unmatched performance for single-crystal experiments in diamond anvil cell, including laser heating of single crystals.

The PX^2 project received initial approval of the COMPRES Executive Committee in early 2013. In early August 2013 the PI, in coordination with the COMPRES President and GSECARS staff prepared an NSF supplement request to fund capital equipment purchases necessary for the project commissioning. The request was submitted in mid-August 2013. NSF approved the supplement request on September 27, 2013. After the COMPRES budgetary discussions, the PX^2 subaward documents were submitted to the University of Illinois on March 17, 2014, with a project start date scheduled for June 1, 2014. University of Illinois sent an award approval notification on May 21, 2014, prior to the project start date which enabled the PI to set up an advance account at the University of Hawaii, granting access to approximately 30% of the project funds, and enabling initiation of the personnel recruitment process. However, the subaward transfer from the University of Illinois has not been completed until October 15, 2014.

*COMPTECH*

COMPRES Technology Center (COMPTECH) represents a new model of COMPRES’ presence at the Advanced Photon Source (APS). The main mission of COMPTECH is to initiate, facilitate and coordinate new Partner User Proposals (PUPs) to create new capabilities and preferential access to state-of-the-art facilities at existing beamlines at the APS for the COMPRES community. The project started on March 1, 2013. COMPTECH was initially operated from March until December 2013, with funding (including employment of the COMPRES Technology Officer) handled directly by COMPRES Central, and project managerial oversight provided by P. Dera, then at GSECARS. In early 2014 COMPRES Executive Committee approved transfer of the project to the University of Hawaii (Dera moved to the University of Hawaii in August 2013). The project was scheduled to re-start on June 1, 2014 (start of new COMPRES budget year), with operating budget covered by the same subaward as PX^2.

There are several significant advantages for the presence of COMPTECH at APS: (1) the unique technical capabilities enabled by the most advanced hard X-ray synchrotron source can push forward the novel, forefront experimental studies for the community; (2) COMPRES has several ongoing projects hosted by ANL and there are hopes to establish more permanent COMPRES facilities through PUP proposals; (3) A number of resident experts on experimental technology, software and methods development, are interested in collaborating with COMPTECH; (4) APS is a central research facility frequently visited by the majority of active COMPRES members, which fosters collaborations and communication. Furthermore, APS is currently in the initial stage of a major midterm upgrade, which will not only extend its current capability, but also create collaboration opportunities for COMPTECH with existing and future APS beamlines.

In contrast to the General User Proposals (GUPs) that are focused on experiments utilizing techniques already established and available at beamlines, PUPs are more focused on development of new capabilities or methodologies at existing beamlines in addition to science goals. These proposals usually span over longer period of time (typically 1-3 years) and require users' substantial contribution in terms of personnel, instrumentation and/or software development. PUPs, if granted, have guaranteed access to some portion (typically up to 15%) of the beam time available at the facility for the duration of the project. COMPTECH plays an important role in streamlining and coordinating the PUP projects through creating centralized technology-oriented resource that can be utilized and shared for supporting several PUP projects at once.

**Scientific Highlights**

Neither of the two projects has started a regular operation through the UH subcontract yet, therefore scientific highlights from recent efforts are not yet available.

From the late 2013 COMPTECH activities the following scientific outcomes are noteworthy:

**The sound velocities and spin transitions of Fe7C3 under high pressures (PI: Bin Chen, COMPTECH and the University of Hawaii at Manoa, conducted at Sectors 3-ID-B and 16-ID-D of APS)**

The nuclear resonant inelastic X-ray scattering (NRIXS) measurements of an iron carbide Fe7C3 up to 154 GPa were carried out at 3-ID-B from July 31 to August 5, 2013 (18 shifts), continuing efforts to determine the sound velocities of this candidate component of the inner core. The new set of data from the NRIXS measurements shows that sound velocities of Fe7C3 increase with density at an exceptionally low rate after going through a pressure-induced magnetic transition near 50 GPa. Extrapolating to the inner core pressure and accounting for the temperature effect, we found that an Fe7C3-dominant mixture with iron can reproduce the vS of the inner core, with no need to invoke partial melting or the postulated large temperature effect. Such a carbon-rich inner core would make the central sphere the largest reservoir of carbon on our planet.

To confirm the pressure-induced spin transition in Fe7C3, X-ray Emission Spectroscopy (XES) measurements of Fe7C3 were also performed at Sector 16-ID-D from July 13 to July 16, 2013 (9 shifts). The experiments revealed a high-spin to low-spin transition of iron in Fe7C3 occurring at the pressure range consistent with that predicted from the previous X-ray diffraction experiments and theoretical calculations and provided essential knowledge on the nature of the exceptionally low shear-wave velocity of Fe7C3 after the magnetic transitions determined by the NRIXS measurements.

A manuscript has been submitted after revision to Proc. Nat. Acad. Sci. (PNAS) and is currently under review. Dr. Chen was also invited to submit a book chapter on “Carbon in the core” in AGU monograph on “Chemistry and Physics of Earth’s interior” and the book chapter is currently under review.

**Performance Metrics**

Neither of the two projects has started a regular operation through the UH subcontract yet, therefore recent performance metrics are not yet available.

**PUP and GUP beamtime at APS for COMPTECH operation in late 2013**

Table 1. PUP and GUP beam times awarded to COMPRES and our collaborators during this term

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Proposal No. | PI Name | Beamline | # days | Beam time |
| PUP 28037 | Jay Bass, Ahmet Alatas | 3-ID-C | 5 | 11/6(08:00)-11/11(08:00) |
| GUP 33053 | Bin Chen, James Van Orman | 13-ID-D | 3 | 11/18(08:00)-11/22(08:00) |
| GUP 36108 | Bin Chen, Przemyslaw Dera | 16-ID-B | 2 | 12/4(08:00)-12/6(08:00) |

In addition to the above online experiments, Bin Chen supported off line LVP experiments: PI J. Bass (UIUC), Sector 13-ID-D. Synthesis of single-crystal hydrous wadsleyite and ringwoodite using the 1000 ton large volume press (LVP) at the GSECARS (16-ID-D) in September 2013. Dr. Chen provided training to Prof. Bass' graduate students, Jin Zhang and Stephen Picek, on the preparation of the cell assemblies for the LVP experiments and helped them conduct the offline synthesis experiments.

**Community/Broader Impacts**

**EarthCube Domain End-user workshop**

Dera co-chaired NSF EarthCube Domain End-user workshop for Rock Deformation and Mineral Physics, which was held in Alexandria, VA in November 2013. COMPTECH officer Chen was also actively involved in the workshop as beakout session chair.

**On line survey and Collaborative NSF Geoinformatics proposal**

An element of collaborative activity that was identified in the COMPTECH proposal as one of the COMPTECH mission targets was stimulating and coordinating new fundraising activities to address important mineral physics community needs. After the EarthCube Domain End-user workshop a collaborative team was formed, including Dera, Chen, L. Ehm (Stony Brook), A. Wolf (Univ. Michigan) and R. Downs (Univ. Arizona). The goal of the collaborative team was to assess the major software needs of the mineral physics community utilizing synchrotron facilities and formulate a plan on how to address these needs.

In late February 2014 our group conducted an anonymous online survey to analyze the level of interest and support for federally funded open source software development in mineral and environmental science, with particular emphasis on X-ray scattering research at National User Facilities. The survey was advertised though COMPRES, GSECARS, HPCAT and Mineralogical Society of America mailing lists. The survey was answered by 110 participants who ranged from graduate students to senior faculty and researchers. 74% the respondents represented mineral and environmental sciences, with research interests within the main focus area of the COMPRES (remaining 26% were materials scientists and chemists). 38% of the respondents were in Mineral Physics and Rock Deformation fields. Great majority (72%) of the survey participants believe that there are still many significant and transformative discoveries that can result from X-ray scattering experiments. 98% of the participants expressed opinion that currently available software needed to fully analyze the experimental data and enable these future discoveries is insufficient. 73% of the respondents were interested in contributing to development of open source code either in person, or by encouraging their students and postdocs. 80% of the community believes that the most efficient model for addressing the data analysis software challenge is through coordinated collaboration between academic researchers and user facility scientists. A detailed survey report is available through EarthCube SIG:

<http://workspace.earthcube.org/software-mineral-sciences>.

Based on the results of the survey, our group prepared a collaborative software development proposal “ATREX: Advanced Tools for Research in Extreme Xtallography”, which was submitted to the NSF EAR Geoinformatics progam. The proposal, which to a large extent grew out of the COMPTECH activities, was approved for funding for 2 years ($597K total budget) and the ATREX project began on October 1, 2015.

**APS Annual User Meeting talk and poster**

Dera presented an invited keynote talk at the APS Annual User Meeting at Argonne, IL, during which the PX^2 project status and goals were summarized. A poster presentation with outline of the PX^2 design concept and details of preliminary performance tests has also been presented.

**COMPRES Annual Meeting presentation**

Dera presented a status report on both COMPTECH and PX^2 projects. A poster presentation with outline of the PX^2 design concept and details of preliminary performance tests has also been presented.

**HPCAT Time-resolved high-pressure phenomena workshop**

Dera presented an invited talk and co-organized a software demonstration. The ATREX and PX^2 project status and goals were described.

**Mineral Physics Long Range Planning Workshop and Report**

Dera organized and co-chaired the Mineral Physics Long Range Planning Workshop held at Argonne in October 2014, and attended by ~60 COMPRES community members. The newly selected COMPTECH Officer, Jin Zhang was actively involved in this meeting as Discussion Group Leader for Equations of State topic. At the workshop the key new developments and new initiatives for the next decade of mineral physics research have been discussed.

**Personnel**

*COMPTECH Technology Officer involvement in the PX^2 project*

PX^2, though formally not a PUP, represents an example of a large, collaborative partner project with significant matching financial and other in-kind contributions from APS and GSECARS, resulting in broad and long-term benefits for the COMPRES community. The project fits within the broad definition of the COMPTECH target activities, and the COMPTECH Officer has been involved in planning, design and commissioning activities related to PX^2 from the earliest stages. The two projects are currently combined within one subaward, and we plan that the synergy and collaboration between COMPTECH and PX^2 will continue.

First activities related to the PX^2 project started in the fall of 2013, after the NSF approval of the capital equipment supplement to COMPRES, however, the project has not had any formal personnel until now (the newly recruited PX^2 Beamline Scientist is scheduled to start on January 1, 2015, and the new COMPTECH Technology Officer will start on November 16, 2014. During the period September 1 – December 31, the COMPTECH Technology Officer Bin Chen was involved in the early design and procurement work (prepared Solid Works drawings for the cleanup slit assembly) for PX^2.

*PX^2 Beamline Scientist recruitment*

The PX^2 project includes one Beamline Scientist position located at the Argonne National Lab. Recruitment for this position was conducted through the Research Corporation of the University of Hawaii (RCUH). A five member Search Committee was appointed to oversee the recruitment, including P. Dera (UH), J. Bass (COMPRES), T. Duffy (Princeton), M. Rivers (GSECARS) and B. Chen (UH). The Committee met through videoconferencing service. The closing date for the applications was set for August 8, 2014. The position was advertised at the RCUH website, through the COMPRES and MSA mailing lists, and was posted at IUCr and AIRAPT websites. 10 qualified applications were received. The Search Committee selected 4 candidates for interviews, which were conducted in late August, also via teleconferencing. After completion of the interviews, the Committee ranked the interviewees and selected Dongzou Zhang, currently a Senior Graduate Student at Caltech (expected graduation date December 3, 2014) and Resident Research Assistant at APS Sector 3, as the top candidate. Mr. Zhang accepted the offer of employment and the starting date for his new appointment has been set for January 1, 2015.

*New COMPTECH Technology Officer recruitment*

The COMPTECH project includes one Technology Officer/Researcher position located at the Argonne National Lab. Recruitment for this position was also conducted through RCUH. The same five member Search Committee was appointed to oversee the recruitment. The Committee met through videoconferencing service. The closing date for the applications was also set for August 8. The position was advertised at the RCUH website, through the COMPRES and MSA mailing lists, and was posted at IUCr and AIRAPT websites. 6 qualified applications were received. The Search Committee selected 5 candidates for interviews, which were conducted in late August, also via teleconferencing. After completion of the interviews, the Committee ranked the interviewees and selected Jin Zhang, currently a Postdoctoral Researcher at the University of Illinois Urbana Champaign as the top candidate. Dr. Zhang accepted the offer of employment and the starting date for her new appointment has been set for November 16, 2014.

**Operations and Planned Activities**

**COMPTECH**

Of the current reporting period, the COMPTECH project was active only during the three month period between September 1, 2013 and December 31, 2014 due to departure of the previous Technology Officer, Chen and transfer of the project to the University of Hawaii. From January 1, 2014 until November 16, 2014, when the new COMPTECH Officer starts her appointment, the project has been in suspension.

Three on-going or planned PUP projects in which COMPTECH was involved in late 2013 were:

* PUP project "High-Energy Resolution Inelastic X-ray Scattering for Studying Properties of Planetary Materials (PUP28037)" at Sector 3,
* Exploratory project “Multigrain diffraction on laser heated samples at ultrahigh pressures”, which may lead to a PUP submission in the near future,
* Collaboration project between COMPRES, GSECARS and University of Hawaii on “PX^2 Partnership for eXtreme Xtallography” development of novel DAC capabilities at the experimental station 13BMC.

Besides these activities, in 2013 the COMPTECH Officer was pursuing development of the COMPTECH website, online content and tools for the COMPRES community:

1) COMPTECH website (http://comptech.compres.us)

2) Database of JCPDS cards for use in high pressure research

(http://comptech.compres.us/tools/jcpds)

3) Pressure scale online calculation software: Ruby fluorescence gauge:

(http://comptech.compres.us/tools/ruby) and Diamond anvil Raman pressure gauge:

(http://comptech.compres.us/tools/diamond)

4) Java program "Focus", for rotating and scan to move sample to the X-ray focus position, written in Jruby

5) IDL program to convert American Mineralogist Crystal Structure Database files (XPOW format) to JCPDS format

Bin Chen initiated one infrastructure development project - development of new graphite/LaCrO3 resistive heater design for high-temperature DAC experiments, which still continues today at UH.

During the period 9/1/13-12/31/13 Bin Chen offered assistance and training to APS users for off line experiments with LVP at GSECARS.

Chen was also actively involved as co-PI in preparation and submission of the ATREX collaborative software development proposal to the NSF EAR GeoInformatics program.

**PX^2**

Commissioning activities and timelines

Considering the timing constraints of the funding transfer to Hawaii and recruitment of the new PX^2 Beamline Scientist we heavily relied on generous help from the GSECARS staff in the early stages of the new program development. The current project timeline includes 5 Stages of instrument development:

*Stage 1 (June-July 2014)*

* Monochromator upgrade (installation of new Si 311 asymmetrically cut crystal)
* Installation and testing of secondary horizontal focusing mirror
* Installation of preliminary DAC specific components, including cleanup slit assembly, retractable photodiode, and retractable high magnification visual observation system.
* Preliminary system performance tests

*Stage 2 (November-December 2014)*

* Installation of final optical platform with sample observation optics and horizontal mirror enclosure with temporary 200 mm KB mirror
* Intermediate system performance tests 1

*Stage 3 (January-March 2015)*

* Installation of new 320 mm horizontal KB mirror inside the new enclosure and installation of Raman spectroscopy system.
* Intermediate system performance tests 2

*Sage 4 (May-July 2015)*

* Start of limited GUP user operation (without laser heating)
* Installation of laser heating system
* Final system performance tests

*Stage 5 (October 2015)*

* Start of full user operation (full system capabilities)

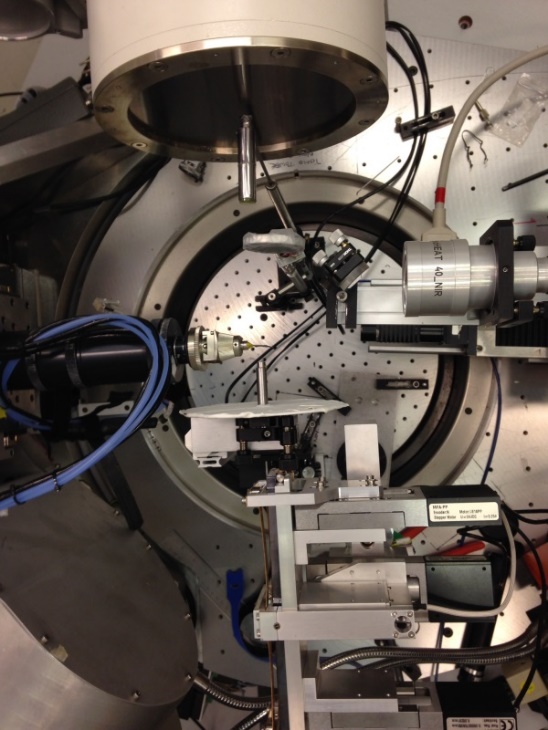
**New beam characteristics after the X-ray optics upgrade**

The main two reasons why the experimental station 13BMC was not used for DAC experiments before the PX^2 project started were the fixed energy of 15 keV (too low for DAC) and large size of the focused beam of 300 x 500 m. To address both of these issues, our team came up with a plan to upgrade the monochromator (which was designed to hold up to 3 different crystals, while only one crystal was initially installed) and add to the existing Si 111, 15 keV crystal, a second Si 311 crystal that would produce a 30 keV beam, optimal for DAC experiments. In order to achieve more than an order of magnitude of improvement in focusing ability, the monochromator crystal bender has been re-designed (horizontal focusing in the pre-PX^2 13BMC was done by dynamically bending the asymmetrically cut monochromator crystal, but the crystal bender flaw was causing underperformance of the focusing system) and a second stage focusing with short KB mirror has been introduced. To take full advantage of this concept, a new custom 320 mm horizontal KB mirror has been ordered. We negotiated with the APS Management an arrangement, according to which APS paid for the manufacturing of the new Si 311 crystal. The new Si 311 crystal was installed in the spring of 2014 and fist Stage 1 performance tests were carried out in June-July 2014. Since the new KB mirror manufacturing takes several months, GSECARS made available an old 200 mm mirror for initial tests of the two-stage focusing.

During the commissioning experiments conducted in 2014-2 we were able to achieve a 25 x25 m focusing of the 30 keV beam. A preliminary cleanup slit system was installed and allowed to collect clean diffraction patterns from a 20 m single crystal sample inside an 80 m diameter gasket hole inside a diamond anvil cell. Despite the temporary focusing mirror, with non-optimal length and surface quality, the beam flux was surprisingly high. For single crystal data collection we estimated that the diffraction signal strength was only 5-10 times lower than in the 13IDD insertion device station. After the new focusing mirror is installed we expect even further improvement in the beam characteristics.

**Preliminary optical system for stage 1 tests**

As a part of Stage 1 Commissioning of the PX^2 instrument at Sector 13, BM-C, we installed and tested preliminary high resolution optical imaging system. Such system must be capable of achieving resolution up to few microns, be retractable (removed from the X-ray beam path during data collection), stable, reproducible, and easy to operate. The preliminary system we assembled consisted of infinity-corrected 5x special objective GEO-HEAT, designed by Dr. Laskin from AdlOptica company, motorized XY stage for the motion and alignment of camera to the X-ray focus point, and Z-stage for retracting the whole optical system during X-ray data collection. Imaging objective was connected to the 12x Navitar manual Zoom with stable focal point and variable magnification 0.6X—7.5X, and ProSilica digital camera, which collected visual image that could be viewed remotely on the beamline control computer. This imaging system allows one to observe samples only few microns in size and position them with micron precision. The system used during Stage 1 Commissioning could be only partially controlled remotely (zoom control was manual). The details of the experimental setup layout are presented in Figs. 1 and 2.



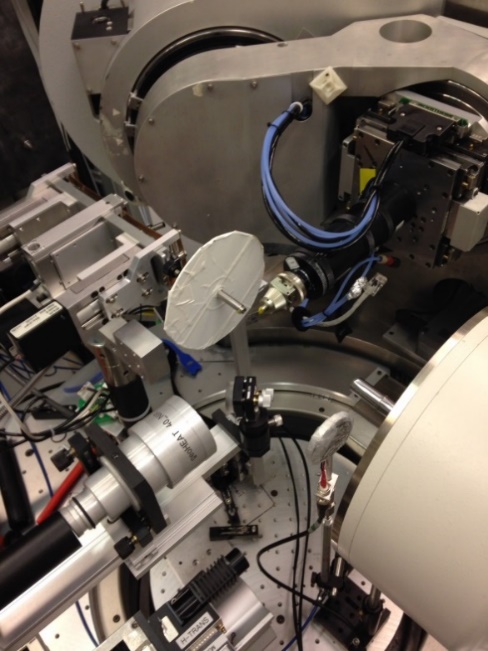
5x infinity-corrected objective

K-B mirror

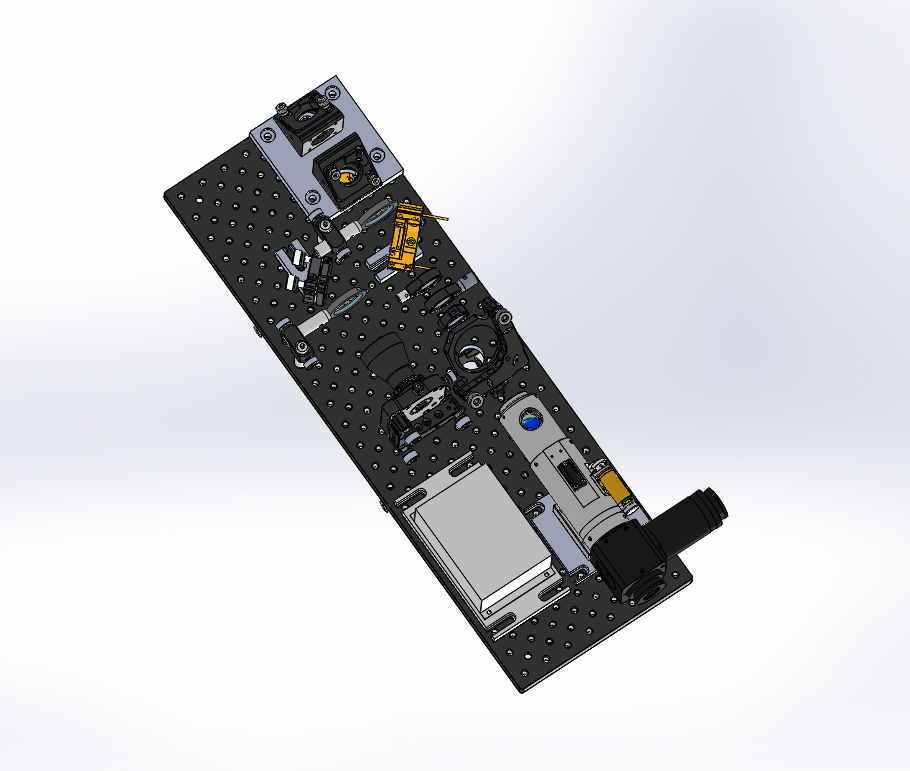
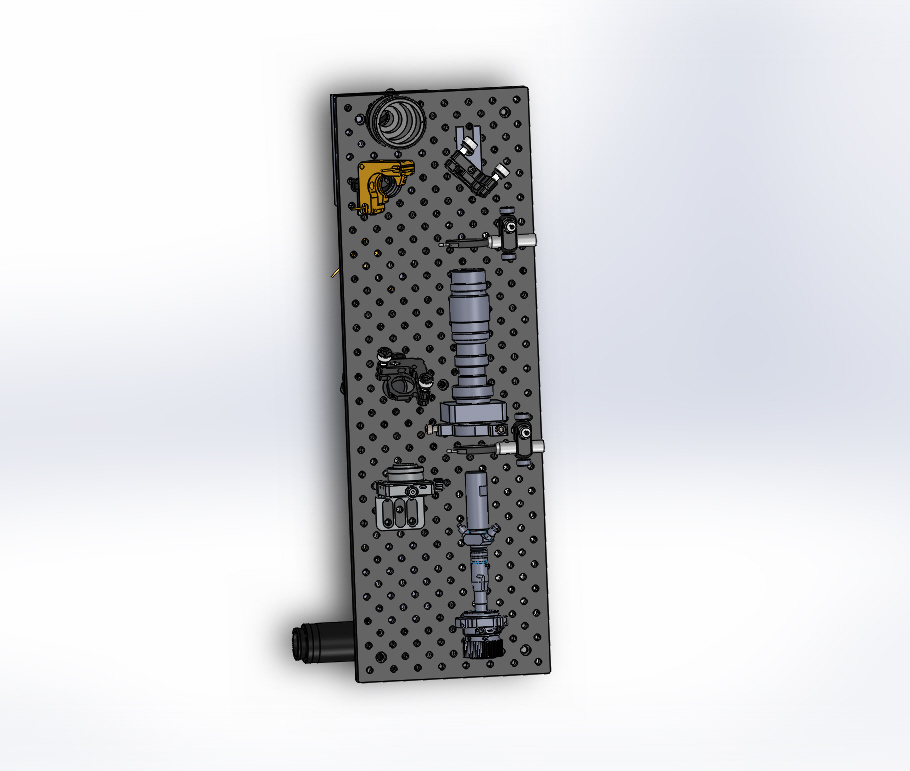
Sample

Fig. 1. X-ray diffraction setup, top view.

This preliminary system was found to be easy to operate, reliable, and reproducible within +-10 micron accuracy. Such reproducibility is already sufficient for conducting high-pressure experiments involving multiple single crystal specimens loaded in one sample chamber.

Fig. 2. X-ray diffraction setup, side view.

Future setup will involve more complex imaging system, incorporating motorized, remotely controlled 12x Navitar zoom. The imaging system will also be integrated with NIR laser heating and compact Raman and ruby fluorescence spectrometer. All three systems will be mounted on a single breadboard and put onto rotation-translation stages system for simultaneous rotation of the sample and optics, along with the capability of retracting the whole system. Fig. 3 illustrates the full layout of the system, which will be installed in the 2015. The use of breadboard will improve system stability and increase reproducibility, which will, in turn, allow users to perform more challenging experiments, including single crystal X-ray diffraction experiments in the laser heated diamond-anvil cells.



IR beam shaper

Green laser

NIR fiber laser coupling

12x motorized Zoom

NIR 5x-objective

Fig. 3. Full layout of compact laser heating, Raman, and imaging system for 13-BM-C.

**Current budget status and expenditure projections until the end of the current budget year (June 1, 2014-May 31, 2015)**

1. **PERSONNEL**

COMPRES personnel

Because of the delayed start of both appointments, only respective 5.0 and 6.5 months of salary will be charged in this budget year for the PX^2 Beamline Scientist and COMPTECH Officer. The starting annual salary levels for the two new appointees have been set at $62K and $68K, respectively.

Engineering support

Commissioning stage of development of advanced user facility requires extensive machining of custom parts. We were able to negotiate with SOEST an agreement to have 40% time of experienced mechanical engineer available for the PX^2 and COMPTECH project needs. The SOEST machine shop is equipped with a modern advanced CNC machine and a range of other high quality machining tools. The cost of this support is calculated based on a discounted (about 50%, compared to standard recharge fees) monthly 1 FTE salary of $5,000 and a total of 2.4 FTE months that will be contributed over the remaining 6 months of this budget year. The engineer services will be used to design and machine any necessary adapters, custom holders and parts needed for the PX^2 and COMPTECH projects.

Consultant Services

To conduct the commissioning experiments in June-July 2014 it was necessary to have a full time on-site support at APS. Since the PX^2 Beamline Scientist position was not filled yet, we hired Dr. Kirill Zhuravlev, former Postdoctoral Researcher at GSECARS (until May 31, 2014) as a Project Consultant for the period June 11-29, 2014. The main consulting services rendered by Dr. Zhuravlev included:

1. Assembly and alignment of high-resolution sample observation system (GeoHeat special lens and video camera) to allow obtaining high-quality digital images of transparent 20 micrometer sized samples in diamond anvil cell.

2. Assembly and alignment of cleanup slit system with a 30 micrometer pinhole, with the focused X-ray beam.

3. Assistance, as needed in commissioning instrument performance test experiments.

For these services Dr. Zhuravlev was paid a consulting fee of $4,000. Similar arrangement has been made for commissioning activities scheduled for October 20 - November 20, 2014, with another commissioning fee of $5,000.

1. **Fringe benefits**

Fringe benefits are calculated based on the University of Hawaii at Manoa rate of 45.46%. Fringe benefits were not applied to consulting fees.

1. **TRAVEL EXPENSES**

Since no project personnel was available during the Stage 1 PX^2 commissioning, it was necessary for the PI to be present in person at the APS to conduct the instrument performance tests and meet with the GSECARS staff and the Consultant Dr. Zhuravlev to discuss the project and work on finalizing the instrument design. During the 2014-2 run, two trips of PI Dera (7/17-8/18 and 8/29-9/7) between Hawaii and Argonne were supported from the PX^2 and COMPRES Operation budget, totaling $3,204.60. It should be emphasized that:

* The PI travel did not include hotel accommodation cost
* For the 7/17-8/18 trip the airfare was not charged to the COMPRES subaward
* The PI covered the cost of 3 more trips devoted exclusively to the PX^2 project from other sources (UH startup).

The PI anticipates requiring travel expense support for the PX^2-related activities at Argonne 3 times a year (once during each APS run), with estimated cost of each trip at $2,000. Two such trips are anticipated until the end of the first budget year.

For the reminder of the first budget year we anticipate that each of the two new employees will travel to Hawaii once to receive training in crystallographic techniques and software from the PI, to meet with the SOEST mechanical engineer and discuss any specific engineering project needs, as well as to meet with the ATREX project personnel to discuss future software needs and requirements. Estimated cost of each of these trips is $2,000.

The COMPTECH Technology Officer plans to participate in the 2014 AGU Annual meeting. Estimated cost of this trip is $1,500.

An additional travel budget of $7,300 is allocated to cover the relocation expenses of the two new personnel.

1. **CAPITAL EQUIPMENT**

**PX^2 Capital equipment**

After the approval of the capital equipment supplement, but before the PX^2 subaward documents have been submitted to the University of Illinois, a portion of the supplement funds was made available for first hardware component purchases (order was processed directly by the University of Illinois). This way we acquired the following equipment:

* Newport XPS Q8 motion controller with 8 DRV-01 driver cards
* Newport UTS100CC motorized linear stage with 100mm travel range
* 2 Newport MFA-CC miniature motorized linear stages with 25mm travel range for the cleanup slit assembly

The total for this purchase was $18,964.98. The above equipment allowed us to assemble a preliminary Stage 1 setup to test the initial instrument performance during the commissioning beamtime in June-July 20014. The amount of this purchase was not included in the UH subaward budget.

The total budget of the PX^2 capital equipment supplement subaward is $195,236.

Once the subaward advance account at UH has been set up, procurement of the critical long lead time components has begun. To date the following orders have been placed:

10/23 Thorlabs $3,901

Miscellaneous optical components

10/23 Newport $10,190

UTS50PP, UTS100PP, UTS150PP motorized linear stages

9/9 Coastline Optics $6,700

Silicon crystal for the horizontal KB focusing mirror

8/8 IDT $38,829

Horizontal KB mirror bender system

6/23 Milan Machining $8,079

Optical platform and KB mirror cage assembly

Total committed to date: $67,699

Since the full capital equipment budget became available only on October 15, 2014, most of the procurement activities to acquire the expensive spectroscopic equipment for PX^2 are planned for November – December, 2014.

Of the big ticket items that remain to be ordered during the current budget year, and account for the remainder of the year 1 capital equipment budget, the most important are:

*200W Yb:YAG fiber laser* $28,000

*Pulse generator for the fiber laser* $5,000

*GEO-HEAT long working distance objective* $5,000

*Navitar motorized zoom system* $5,000

*Princeton Instruments triple-grating spectrograph* $15,000

*Princeton Instruments LightView software* $5,000

*Princeton Instruments back-illuminated deeply depleted PIXIS CCD detector* $35,000

*Raman 1W 532nm solid state laser* $15,000

*Misc. optics (notch and dichroic filters, beam expanders, manual and motorized mounts and mirror-flippers, miniature motorized translation and tilt stages, fiber optics)* $15,000

Total expected expenditures $128,000

The rationale behind the above components of the PX^2 system has been explained in the PX^2 budget request submitted to COMPRES in early 2014 and the component list has not changed significantly.

**COMPTECH capital equipment**

Because of the delayed hires of the two personnel, some surplus funds will be available in the current year budget for much needed equipment for the COMPTECH project. In its pre-UH operation COMPTECH did not have any capital equipment funds available, which created challenges with seeking partnerships with existing beamline facilities at the APS and being able to substantially contribute to these collaborations. From the current leftover funds we are planning to acquire a number of devices that emphasize portability, have universal applications in high pressure experiments, and will be usable at both PX^2, as well as for any other partner user collaborative projects:

6 Single crystal megabar DACs @ $5000 each $30,000

Ocean Optics QE spectrometer $12,000

2 GE Druck electronic gas membrane controllers $24,000

2 double membrane DAC boxes $12,000

BX-90 gear box for gas loader $5,000

Optical table for new off-line laser lab at Argonne $10,000

Total expected expenditures $93,000

One of the main focus directions of the PX^2 is single crystal diffraction, which requires specialized diamond anvil cell with wide angular access, compatible with the conical anvils. DACs of this type, capable of reaching Mbar pressures, e.g. BX-90 design, are gaining popularity, however, currently most members of the COMPRES community do not yet have access to this kind of equipment. We would like to acquire 6 new DACs of this type which could be used by both PX^2 and COMPTECH users, as well as personnel. DACs of this kind will also be beneficial to other PUP projects in which angular access or resistive heating are important (e.g. the Inelastic X-ray Scattering project at Sector 3).

Ocean Optics miniature spectrometers are an inexpensive, but very attractive solution for spectroradiometry (temperature measurement) as well as fluorescence based pressure callibration. The very small size and fiber optic signal transport will allow easy and convenient integration with the PX^2 instrument, while providing high sensitivity, sufficient spectral range as well as quantum efficiency for temperature measurement. The Ocean Optics QE series UV-VIS spectrometer is equipped with back-thinned, thermoelectrically cooled CCD detector to assure low dark current and increased sensitivity.

For long-duration single crystal and powder laser heating experiments, which will be conducted at PX^2 (or any other PUP partner facilities) the ability to remotely and accurately control pressure inside the DAC will be critical. The novel gas membrane DAC solutions developed at HPCAT (double-membrane universal box) in combination with GE Druck electronic gas controllers are an excellent solution to address this need. The device not only allows accurately changing pressure in very small intervals, but also provides possibilities of feedback stabilization of pressure during long exposures, as well as allows to control the compression rate. GSECARS currently owns one Druck controller, but it is in use at 13IDD for most of the time. We would like to acquire two new Druck controllers for the double-membrane device (one controller needed for each membrane) that could be dedicated to the PX^2 facility, or used at other PUP project locations, when not needed at PX^2. Together with the controller, we would like to purchase two of the double-membrane boxes.

The GSECARS gas loading system has greatly benefited hundreds of COMPRES users and enabled many high quality and more advanced experiments since its construction. Two types of DAC-closing devices are currently used for this system: a gearbox that can accommodate symmetric and standard 4-pin DACs, and a clamping device, which is used for the less standard DACs, including BX-90. The clamping device is a little less convenient to operate and makes it difficult to perform controllable loading to low pressures (<4 GPa), which is often quite desirable in cases when lower pressure phase transitions are studied. We would like to acquire a new gear box designed specifically for BX-90 DACs, which would significantly improve the reliability of gas loading for most experiments performed with these cells.

APS has allocated a laser spectroscopy lab in the APS 400 building for the off-line experiments and instrument developments for COMPTECH and PX^2. This lab is not equipped with an optical table, and in order to be able to efficiently work on new system development and optimization, we will have to acquire such table, and equip it with proper laser safety enclosure and interlock system.

1. **Materials and Supplies**

Diamond anvils and support plates for single crystal experiments $20,000

Chemicals, heating wires, gaskets, materials for custom machining $10,000

Office and computer equipment and supplies for the two new personnel $5,000

Software license fees $15,000

We are at the initial stage of extensive instrument development efforts involving advanced and custom technologies and laser heating. For commissioning activities, staff research as well as for collaborating users, we will need to acquire an initial stock of conical anvils and backing plates.

Any experiments utilizing the new PX^2 instrument will require supplies such as commercially available chemicals as samples or substrates for sample synthesis, solvents, rhenium gaskets for diamond anvil cells, raw materials for machining of custom parts and adapters, etc.

With the new staff members, we will have to equip their offices at both GSECARS as well as in the APS 400 building with the necessary computer and office equipment.

In crystallographic research some of the software used for data analysis and interpretation is commercial. Examples of such software include the Endeavour program by Crystal Impact (structure solution using simulated annealing), JADE program by MDI (powder XRD analysis), and Igor Pro (peak fitting, image analysis), all of which have been used extensively by the PI. In addition to the data analysis software, commercial crystallographic databases, such as the Inorganic Crystal Structure Database, and Powder Diffraction File database, and commercial programming environments such as IDL, Microsoft Visual Studio and Embarcadero Delphi are indispensable components of mineral physics research, that will be required at PX^2.

1. **Indirect costs**

Indirect costs are calculated based on the University of Hawaii approved off-campus rate of 24%.

1. **COST SHARING and in kind-contributions**

**APS**

APS contributed to the PX^2 project by covering the cost of Si 311 monochromator crystal manufacturing. Estimated cost of this expense was $50K. Additionally, APS agreed to allocate a dedicated laser spectroscopy lab for the needs of the COMPTECH and PX^2 projects without a cost to COMPRES. APS will also provide two office spaces for the PX^2 Beamline Scientist and COMPTECH Officer, also without any cost to COMPRES.

**GSECARS**

GSECARS equipment contributions to the project include access to the experimental station, the 6-circle Newport diffractometer with motion control infrastructure and access to GSECARS area detectors (MAR165 CCD, MAR345 IP, Perkin Elmer flat panel detector, PILATUS 1K).

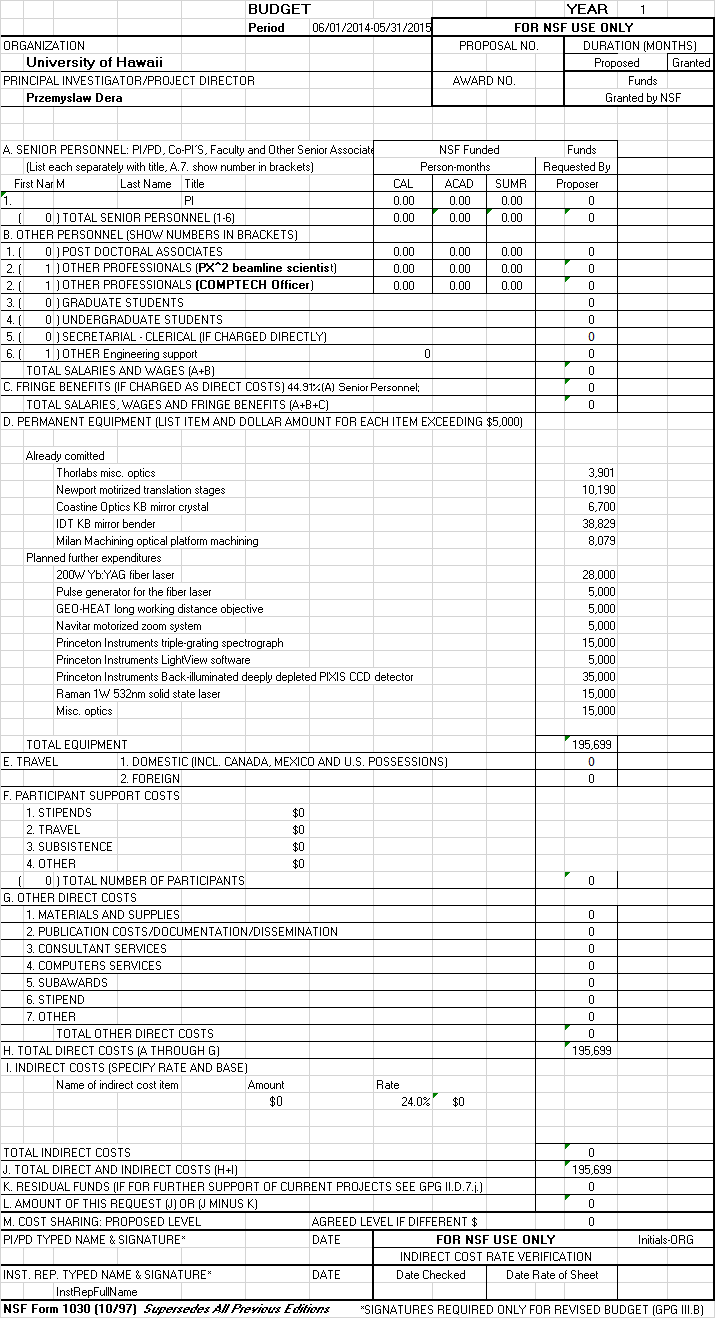
Since the PX^2 project does not yet have on site personnel, majority of the PX^2 design, construction and commissioning activities to date have been carried out by GSECASR staff Peter Eng, Joanne Stubbs Patrick Dell and Mike Proskey. Since September 2013 GSECARS has contributed towards PX^2 significant fraction of effort of these staff members, without any cost to COMPRES:

1. An estimated two months of effort of design drafter, Patrick Dell, who prepared a 3 dimensional model of the 13BMC instrument and integrated the DAC specific solutions described in the attached Technical Design Plan. Another one month effort is expected in 2014 to finalize the design.
2. An estimated one month efforts of Senior Scientist Dr. Peter Eng and Beamline Scientist Joanne Stubbs have been contributed towards upgrade, modification, optimization and testing of the monochromator and focusing optics to adapt them to the diamond anvil cell research requirements.
3. An estimated one week effort of GSECARS Director, Dr. Mark Rivers has been contributed towards setting up instrument control software for the new PX^2 XPS motion controller.
4. An estimated two weeks of effort of Mechanical Support Technician Mike Proskey has been contributed towards assembling the new optical platform system for PX^2.

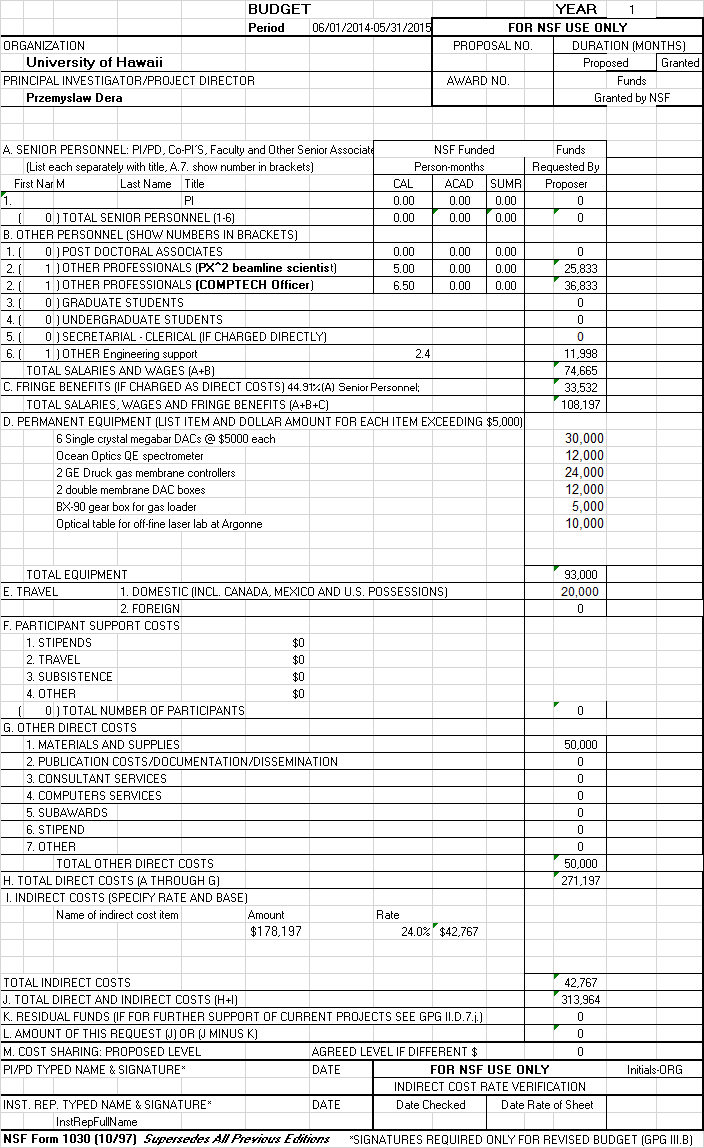
**UH**

University of Hawaii contributes the project management services of the PI without any summer salary support. During 2014 three trips of the PI to the APS to work specifically on the PX^2 project were covered from the PI UH startup account. UH also contributes to the project by providing SOEST mechanical engineering support at the 50% discounted rate. Through the efforts of the PI and the previous COMPTECH Officer, Bin Chen, UH currently holds the ATREX grant from NSF Geoinformatics program. Services of the ATREX Software Engineer to work on PX^2 specific software needs will be provided at no cost to COMPRES.

**Capital equipment budget for 6/1/2014-5/31/2015 (total allocated $195,236)**



**Operation budget for 6/1/2014-5/31/2015 (total allocated $313,440)**



**Budget request for 6/1/2015-5/31/2016**

1. **PERSONNEL**

COMPRES personnel

We anticipate a customary inflation-based 3% increase of all base salaries, including engineering support costs, compared to the current budget year. The projected base annual salaries for the PX^2 Beamline Scientist and COMPTECH Officer are $63.8K and $70.0K, respectively.

Engineering support

During the period of the second budget year of this project significant commissioning activities will continue, including implementation and optimization of the complex laser heating system. COMPTECH will also start actively working on new projects, which will require custom machining of optical and mechanical components. We would like to continue the arrangement with the discounted services of SOEST mechanical engineer. The cost of this support is for the second budget year is calculated based on a discounted monthly 1 FTE salary of $5,150 (customary 3% annual increase) and a total of 3.6 FTE months (reduced from 40% effort in year one to 30% effort in year two) for the whole second year.

1. **Fringe benefits**

Fringe benefits are calculated based on the University of Hawaii at Manoa rate of 45.46%.

1. **TRAVEL EXPENSES**

The PI anticipates requiring travel expense support for the PX^2-related activities at Argonne 3 times a year (once during each APS run), with estimated cost of each trip at $2,000.

For both the COMPTECH Technology Officer and the PX^2 beamline scientist we request funds to travel to the COMPRES Annual Meeting and the AGU Annual meeting. Estimated cost of each trip is $1,500.

A single annual trip to Hawaii is requested for each of the two project personnel to continue training in crystallographic techniques and software with the PI, to meet with the SOEST mechanical engineer and discuss any specific engineering project needs, as well as to meet with the ATREX project personnel to discuss future software needs and requirements. Estimated cost of each of these trips is $2000.

1. **CAPITAL EQUIPMENT**

**PX^2 Capital equipment**

The only capital equipment item requested for both the COMPTECH and PX^2 projects is Ocean Optics NIRQuest near-Infrared InGaAs detector for spectroradiometric temperature measurements below 1000 K. Ocean Optics miniature spectrometers are an inexpensive, but very attractive solution for spectroradiometry. The very small size and fiber optic signal transport will allow easy and convenient integration with the PX^2 instrument, while providing high sensitivity, sufficient spectral range as well as quantum efficiency. The new series of thermoelectrically cooled InGaAs detector-based NIRQuest spectrometers, provides access to spectral range 1000-1700 nm, necessary for constraining the Planck blackbody radiation curve below 1000 K. The quantum efficiency of standard CCD detectos falls to zero above 1000 nm.

1. **Materials and Supplies**

Diamond anvils and support plates for high pressure experiments $5,000

Chemicals, heating wires, gaskets, materials for custom parts machining $10,000

Software license fees $5,000

One of the major expenses in high-pressure experiments is the cost of diamond anvils (~$1K/piece). A budget of $5K is requested for repair or replacement of anvils that will be damaged during commissioning experiments.

Any experiments utilizing the new PX^2 instrument will require supplies such as commercially available chemicals as samples or substrates for sample synthesis, solvents, rhenium gaskets for diamond anvil cells, raw materials for machining of custom parts and adapters, etc.

In crystallographic research some of the software used for data analysis and interpretation is commercial. Examples of such software include the Endeavour program by Crystal Impact (structure solution using simulated annealing), JADE program by MDI (powder XRD analysis), and Igor Pro (peak fitting, image analysis), all of which have been used extensively by the PI. In addition to the data analysis software, commercial crystallographic databases, such as the Inorganic Crystal Structure Database, and Powder Diffraction File database, and commercial programming environments such as IDL, Microsoft Visual Studio and Embarcadero Delphi are indispensable components of mineral physics research, that will be required at PX^2. Some of these software licenses (e.g. Jade, ICSD database) carry annual fees.

1. **Indirect costs**

Indirect costs are calculated based on the University of Hawaii approved off-campus rate of 24%.

**Operating budget request for 6/1/2015-5/31/2016**

