**COMPTECH Annual Report,** *November 1, 2015- October 31, 2016*

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**Overview**

The COMPRES-supported COMPTECH project at Argonne National Laboratory, managed by the University of Hawaii has been active since June 1, 2014, but will discontinue after May 30, 2017. In December 2015, the principal project employee, COMPTECH Technology Officer, Dr. Jin Zhang, accepted a faculty appointment at the University of New Mexico, and consequently, COMPRES Executive Committee decided to phase out the project after her departure, which took place on July 15, 2016. Because this happened in the middle of fiscal year, and one of the instrument development projects has not been concluded, we continued limited activities, as described below, after July 15, 2016, to bring this project to completion.

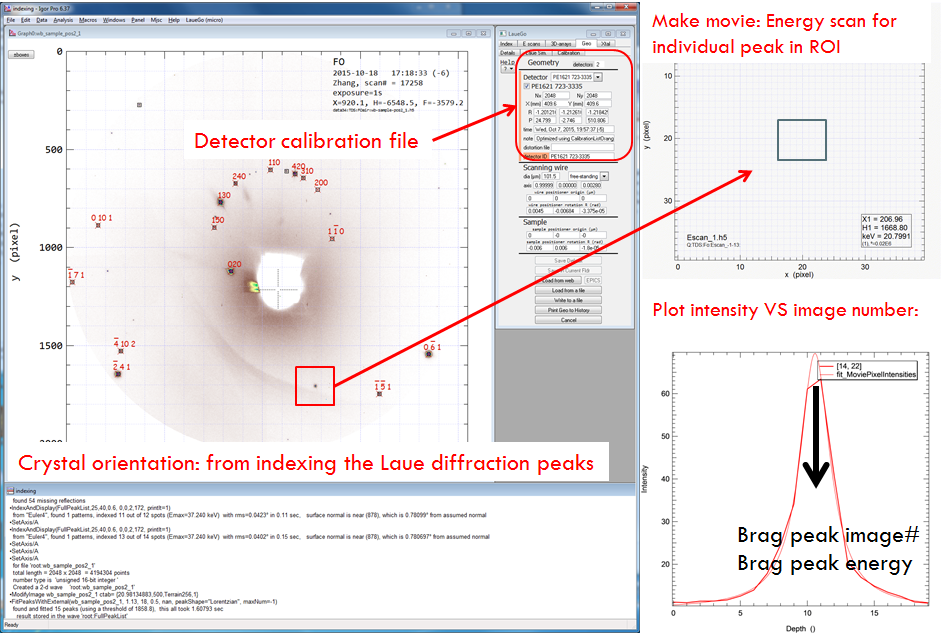
**Technology Advisory Board meetings**

COMPTECH has been advised by Technology Advisory Board (TAB) that met using teleconferencing services approximately 3 times a year, and in person at the COMPRES Annual Meeting. The last meeting of the TAB took place on January 22, 2016. During that meeting Dr. Zhang presented a summary of her activities since the beginning of the project, and a plan of work until the end of her employment with COMPTECH. Because the project was to be discontinued, the planned activities focused on wrapping up ongoing project.

**Thermal Diffuse Scattering**

X-ray thermal diffuse scattering (TDS) has been used to determine elastic properties of solids for several decades, however, its applications in high-pressure mineral physics have not been widely explored. Recent interest in this direction, mainly at Spring-8 in Japan, and at ESRF, have demonstrated very encouraging results. Sound velocities of Earth materials at relevant high pressure (P) and temperature (T) conditions are essential for interpreting seismic data, which provides the most accurate image of the Earth interior. Compared to other techniques used for measuring sound velocities in the high-pressure mineral physics community, for example, Brillouin scattering, stimulated light scattering, inelastic X-ray scattering (IXS), nuclear resonance inelastic X-ray scattering (NRIXS) or MHz/GHz ultrasonics, TDS has several significant advantages and but also some disadvantages. TDS is not limited to transparent samples or nuclear resonant isotopes, single-crystal elastic properties, not only aggregate elastic properties, can be obtained through TDS, and finally, experimental setup for TDS measurement is generally compatible with that for single crystal diffraction. However, TDS is much less straightforward in data interpretation, which usually involves micro force constant modeling (Born-von Karman model) to account for interactions between the neighboring atoms.

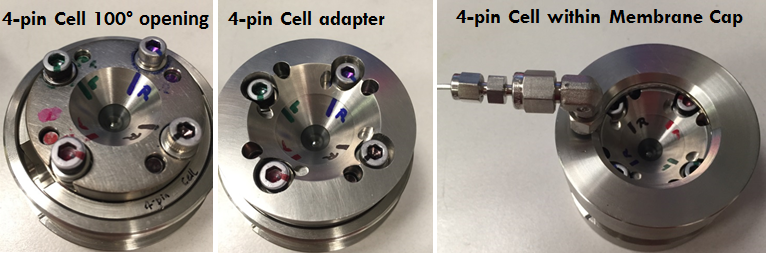
We developed several alternative experimental strategies of performing TDS experiments at different beamlines (including 13BMC, 13IDC and 34IDE) using different experimental setups (Mar CCD detector, Pilatus detector, flight path, energy scan). We successfully measured TDS signal up to 40 GPa for foresterite and 1.4 GPa for Si. J. Zhang also developed Python fitting code, based on continuum elastic wave model, and demonstrated that the final fitted single crystal elasticity tensor of Si is identical to what is obtained through the traditional approach using Born-von Karman model. We also demonstrated the TDS signal to be very sensitive to single crystal elastic anisotropy. The software, along with written operating instruction are available to interested users, and PX^2 Beamline Scientist has been trained in the procedures for conducting the measurements. Two manuscripts, one describing our preliminary results on silicon, and details of measurement procedure, and the second one, on the pressure-evolution of elastic tensor of forsterite, are in preparation.



*Fig. 1. Data analysis for high-pressure TDS measurements at 13IDE experimental station (energy-scan approach).*

**Universal dac membrane cap system**

Membrane caps can turn traditional screw-driven DACs into membrane-driven DACs, allowing remote precise pressure control during an experiment. Membrane caps are especially useful for synchrotron experiments. However, current membrane cap designs limit DAC opening to less than 60°. Large DAC opening is essential for high-pressure single-crystal studies, e.g. single-crystal X-ray diffraction, single-crystal Brillouin spectroscopy, etc. Jin Zhang designed a new type of membrane cap which addresses the above problems. The cap is designed to fit as many different types of DACs as possible, and ensures no loss of diamond anvil cell opening. The new universal membrane cap system has been completed tested, and made available to PX^2 users. Approximately ¾ of the PX^2 user utilize the membrane system, and thus far all of them have been happy with the new device. The device significantly increases the efficiency of beam time utilization, and improves the precision of pressure control. All designs drawings have been uploaded to the COMPTECH website.



*Fig. 2. COMPTECH DAC cap in action during user experiments at PX^2.*

**Inexpensive standardized DAC heaters**

Traditional heaters made of Pt wires are very expensive (>$200/pc), and in general are not useable after 2-3 experiments. The power curve of Pt heater is also not reproducible, varies with pieces. Therefore, cost-effective DAC heaters with standard and stable characteristics are in great demand in high-pressure mineral physics field. Jin Zhang worked with a vendor of W-Al2O3 metal ceramics heaters and developed a modified design of the device, with dimensions that fit DACs. Stacking of two heaters in opposite directions solved the non-uniformity heating of individual heaters. The heaters have also been calibrated both in air and inside DACs. The highest temperature reached in DAC sample chamber when using single piece heater is about 650 K. Higher temperature (700-800K) is expected with the optimized design of double-heaters. A number of COMPRES community members including Drs. Leinenweber and Shim at Arizona State University and Z. Liu at BNL expressed interest and received COMPTECH heaters for tests. The heater have also been extensively used by PX^2 users in 2016.

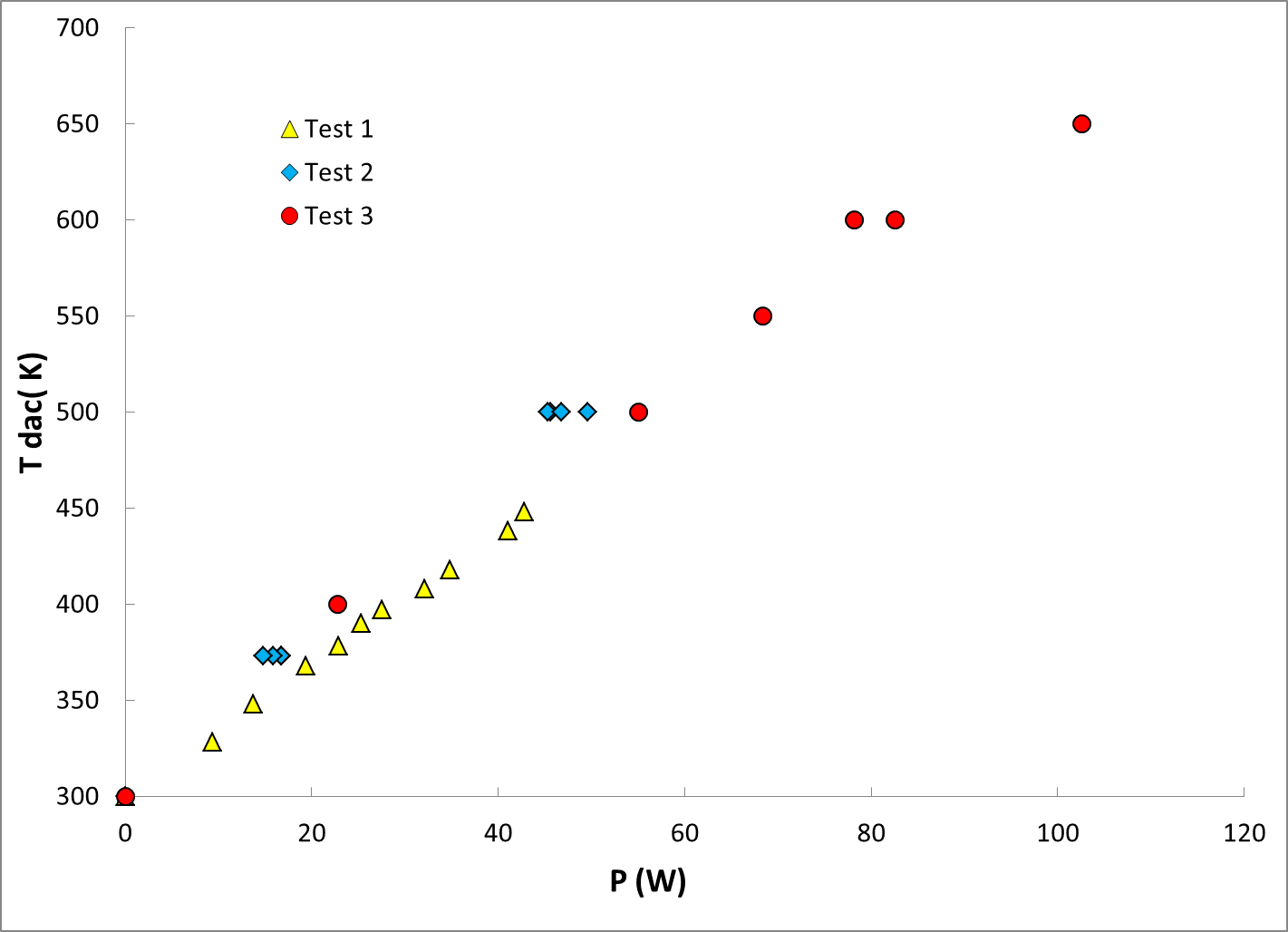


Fig. 3. Power curve for COMPTECH standard heater mounted in a BX90 DAC. Maximum temperature reached inside the DAC was 650 K.

**Portable on-line sample observation and ruby fluorescence system**

The last project in which COMPTECH engaged was development of portable on-line sample observation and ruby fluorescence system for synchrotron instrument that were not designed for high-pressure DAC studies (e.g. 13ID-C, 34ID-E) and lack this essential component. Another portable ruby system was developed few years ago at the APS by the HPSYNC group, but COMPRES users complained that it was difficult to use and prone to easy misalignment, and in the response to the 2014 COMPTECH Annual Report, it was suggested that COMPTECH undertakes such development. In 2016 J. Zhang designed the new system emphasizing stability and ease of alignment, and utilizing modular Navitar microscope components. Dr. Zhang was not able to complete this last project prior to her leaving COMPTECH, because not all of the parts of the custom Navitar zoom microscope arrived on time, before the end of her employment. In the response to the 2015 COMPTECH Annual Report this development was recognized as important and worthwhile, therefore we enlisted services of resident graduate student Yi Hu (UH), currently on a year-long internship at Argonne, to complete this task. In June and July Yi Hu was trained by Dr. Zhang in the optical design and alignment procedures. As of the time of this report, the optical part of the system has been successfully assembled and aligned by Yi Hu. Spectrometer, motorized zoom system and video camera software have been set up and are operational. Successful tests of imaging of samples in diamond anvil cells and acquiring ruby florescence spectra have been conducted. The last part of work on this setup involves commissioning of motorized motion control system XYZ translation stages, motion controller, EPICS software setup). We plan to complete the work by the end of the current funding cycle and make the system available to COMPRES users at the APS in 2017-2 run.

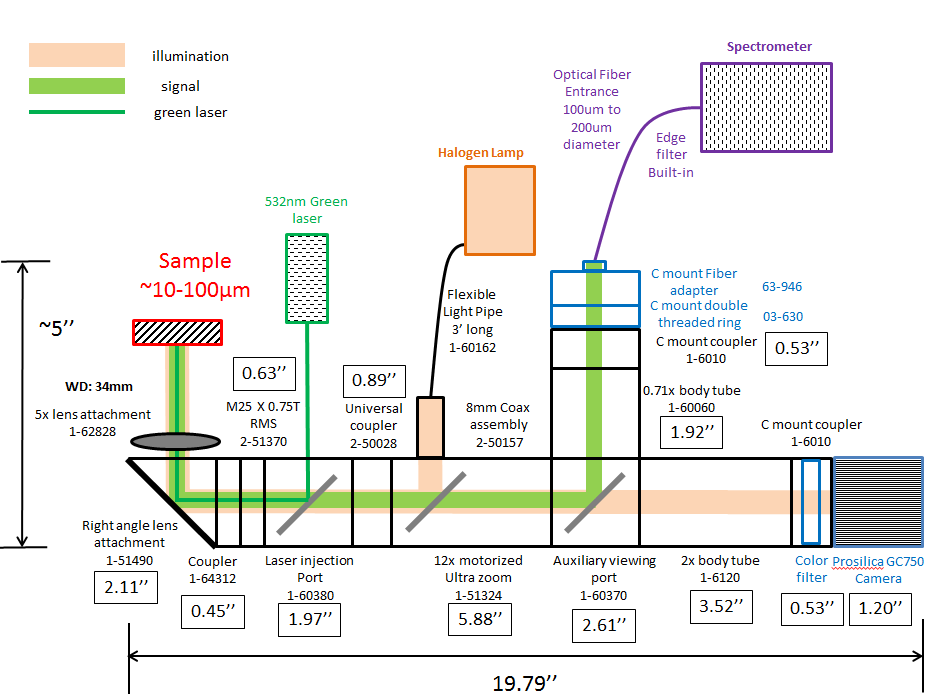
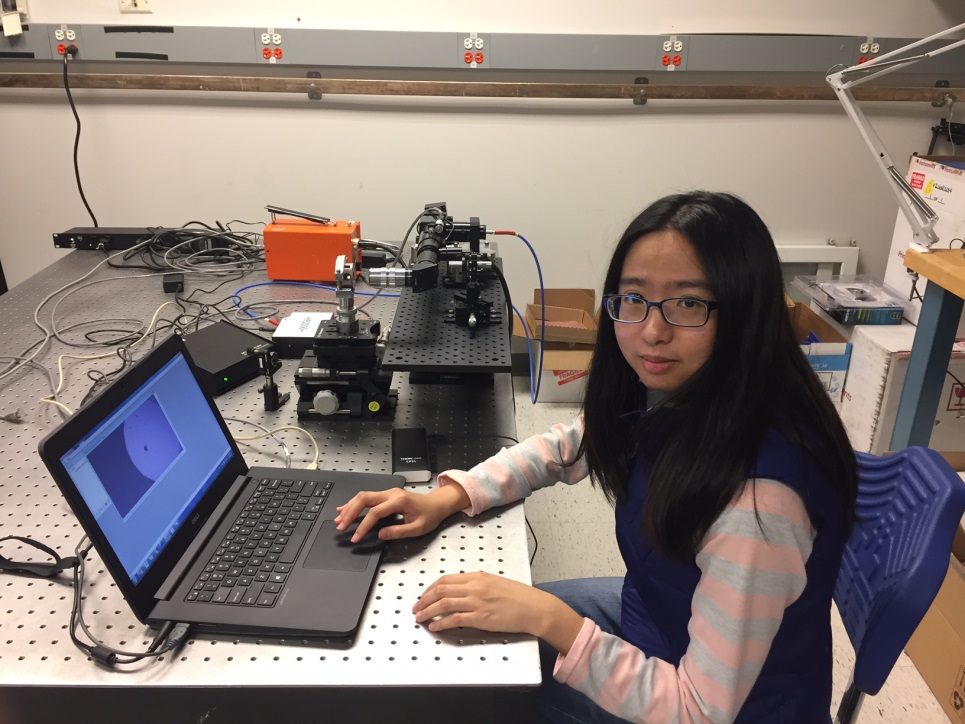
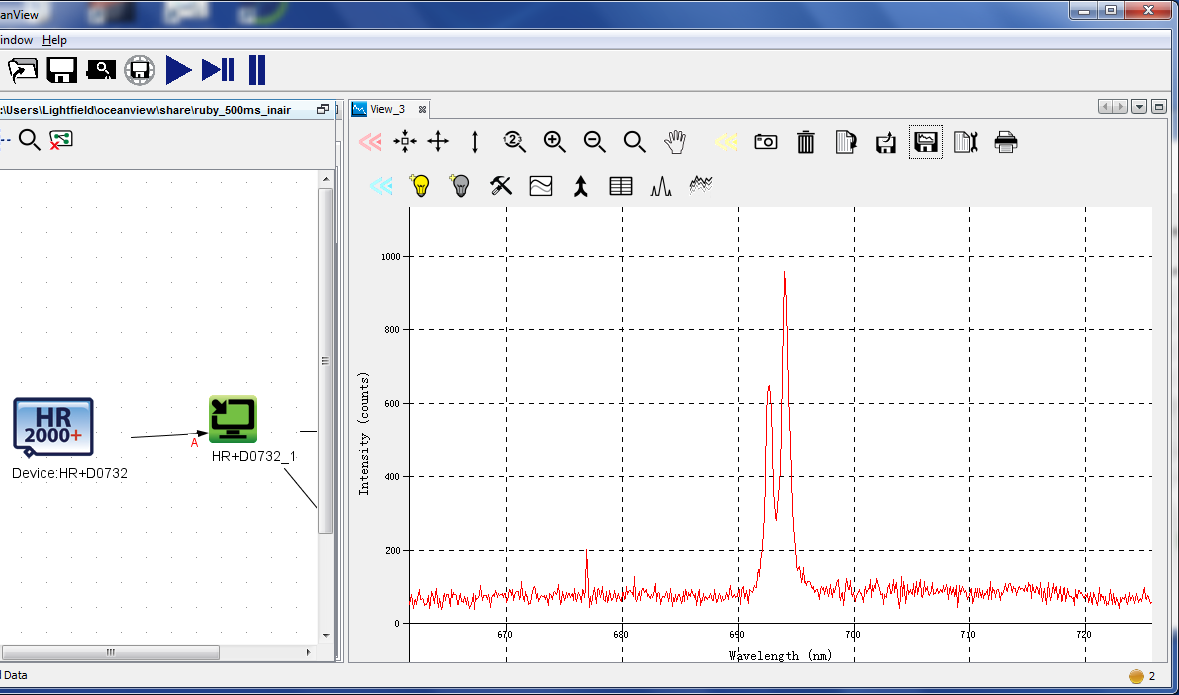
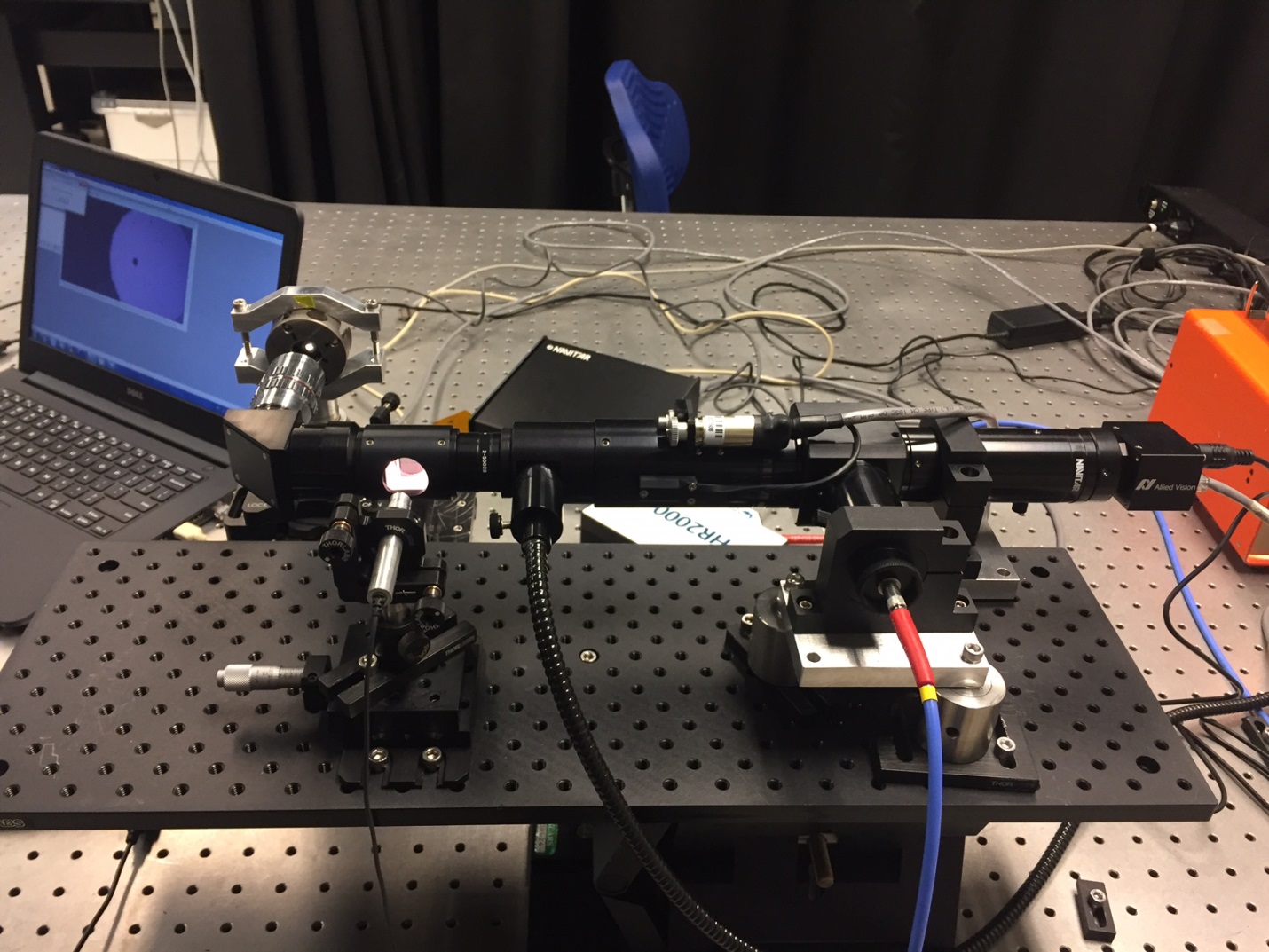


Fig. 4. Design diagram of the portable on-line sample observation and ruby fluorescence system

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d)

c)

(b)

(a)

Fig. 5. Current status of the portable on-line sample observation and ruby fluorescence system: (a) Yi Hu aligning the assembled system in the APS laser spectroscopy lab (b) image of a 0.250mm diamond anvil culet with a ruby sphere on top acquired with the new system (c) close-up of the optical platform of the system and (d) an example ruby spectrum (inside DAC) acquired with the new system at 1mW laser power and 0.5sec exposure time.

**Scientific publications and presentations**

During 2016 Jin Zhang participated in the COMPRES Annual Meeting and presented a talk with update on COPTECH activities. She also first-authored four scientific papers [1-4] in peer-reviewed journals.

1. Zhang, J. S., Bass, J.D. and G. Zhu (2015),“Single-crystal Brillouin spectroscopy with laser-heating and variable” Rev. Sci. Instrum. 86, 063905.
2. Zhang, J. S. and J. D. Bass (2016),“High-pressure single crystal elasticity of  natural Fe-bearing orthoenstatite across a high-pressure phase transition” Geophysical Research Letters DOI 10.1002/2016GL069963
3. Zhang, J. S. and J. D. Bass (2016), Sound Velocities of Olivine at High Pressures and Temperatures and the Composition of Earth's upper mantle Geophysical Research Letters DOI 10.1002/2016GL069949
4. Zhang J.S., Hu Y., Shelton H., Kung J. and Dera P. “Single crystal X-ray diffraction study of Fe2SiO~~4~~ fayalite up to 31 GPa and its implications to subduction zone dynamics” Phys. Chem. Minerals DOI 10.1007/s00269-016-0846-1

**Lessons and conclusions from the COMPTECH project**

I have been involved in formulating the concept, bringing to life, and managing the COMPRES Technology Center since approximately 2010, before the 2011 COMPRES renewal. Because the project has now come to an end of its current form, I would like to offer below few concluding comments and suggestions, which I hope can help COMPRES governance in future decisions regarding centralized technology-oriented personnel resources.

COMPRES is a grass-roots organization, and as such, it relies heavily on community initiative. However, limited R&D financial resources, and lack of centralized personnel that could contribute advanced know-how to new projects, and could be deployed to variety of technology-oriented tasks is a significant shortcoming. Most of other large community organizations on COMPRES-scale I am familiar with, particularly the NSF-funded community consortia, employ such staff.

COMPTECH was an experiment in which COMPRES engaged twice. First, from January 1, 2013 until the end of December 2013, the project was administered by COMPRES Central, with me still at GSECARS as Project Manager, and Bin Chen as COMPTECH Technology Officer (employed by UIUC). After Dr. Chen’s move to a faculty position at the University of Hawaii in January 2014, the project was suspended for about a year, until November 15, 2014, for transition of subaward to Hawaii and hiring process of the new Technology Officer, Jin Zhnag, and restarted in June 2014.

COMPTECH model proved to be very successful from career development perspective. Both job searches attracted excellent applications from domestic, as well as international candidates. The applicants were from the whole spectrum of career stages, from recent PhDs to very seasoned research scientists. The Technology Officers that were hired served for 1.0 and 1.5 years, both successfully transferred to tenure-track faculty positions at very good universities, and remain actively involved in COMPRES activities until now. The COMPTECH setting helped both of them to become better connected, visible, and recognized within the COMPRES community and establish new collaborations. The relative stability of the position tied to the COMPRES long-term grant offered longevity of employment, though uncertainty of annual renewals was a significant drawback, and one of the factors why both incumbents eventually decided to move on to regular faculty appointments. I made efforts to shape these positions in a way which was as close to regular research faculty positions as possible, including affiliate faculty appointments, proposal PI eligibility and access to student co-mentoring, and these features were certainly appreciated by the Technology Officers. If the COMPTECH model were to be revisited in the future, my suggestion would be to tie it to directly to COMPRES Central with renewal and performance evaluation process similar to the COMPRES president and administrative staff (as opposed to annual subcontract renewals), thus providing a better sense of security and stability of employment.

Argonne National Lab as location of the position was also a strong positive. Multitude of synchrotron instruments, sample preparation and off-line spectroscopy facilities opened numerous opportunities to engage in state of the art research and development projects. The COMPTECH Officers stayed research active and continued to publish (e.g. J. Zhang’s 4 first-authored papers in 2014). Steady flux of COMPRES users at the APS offered great opportunities for the Technology Officers to interact and establish new collaborations. Interaction with APS staff scientists provided great training possibilities. This position was meant to be quite independent, and in my assessment this aspect also worked out well, but some kind of on-site oversight and senior advisor was necessary (it was, and can be in the future provided by the PI of the host facility).

The instrument-independent setting of the position proved to be a little challenging when it came to performance evaluation. COMPRES Beamline Scientists are evaluated based on the user statistics, user satisfaction, and publication output of the facility, whereas with the COMPTECH position the performance review criteria were a little more elusive. If the COMPTECH model were to be revisited, perhaps tying fraction of this position to a COMPRES instrument support would be preferred. For the last incarnation of COMPTECH this connection had been provided by PX^2, however it was never formalized (i.e. following COMPRES advice, both projects were kept separate).

The combination of short duration of employment of both COMPTECH Technology Officers, and switch of the operation model (from part of Central Office to a subaward) were a significant drawback for the continuity of operation of COMPTECH. Hiring best young talent that is likely to advance their careers within few years had both the positive of bringing really bright people, but also the negative that they did not stay on for very long. This is not a critical fault, as having a rotation of exceptional people with varied principal expertise may help to facilitate a variety of projects in different areas, but good coordination of rehiring and long term perspective/stability of the project and employment are of fundamental importance. An option for possible future continuation of COMPTECH-style activities without necessity to provide long-term employment for Technology Officers could be to convert the project to a faculty sabbatical support program in which interested sabbatical-eligible applicants with relevant expertise could be matched with high priority COMPRES projects for one year or shorter duration.

COMPTECH has been a very interesting exercise and a big learning experience for me. I am very grateful to COMPRES for facilitating and funding this project, and I hope it will have some lasting positive impact on the COMPRES community.