**Annual Report and 2016-2017 Request**

**COMPRES Multi-anvil Cell Assembly Project**

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**Overview and History**

The COMPRES Multi-Anvil Cell Assembly project at Arizona State University (ASU) began in 2002 as a project to develop and calibrate a series of cell assemblies for the multi-anvil community. At first, small batches of assemblies were provided to COMPRES members free of charge on request. As cell assemblies were developed and characterized, and as the use of some of them became routine, COMPRES advised that the routine use of assemblies by COMPRES-affiliated laboratories should become self-sustaining and the assemblies should be provided at cost to participating laboratories, while maintaining a separate project for new developments still funded by COMPRES. This proved to be very prophetic, because although at first the COMPRES funds could provide for free all the assemblies that were desired by the community, the demand grew steadily and in 2010 the budget for standard assemblies provided to the community at cost surpassed the entire budget of the development project. Last year the budget for the standard cell assemblies was approximately 3x the budget for development.

Administrative support for the standard assembly project was handled by the Chemistry Department at ASU for several years, and then was transferred to a new ASU entity called RTS, Research Technical Services. This has proved to be very useful for our community, because although Chemistry would not allow any negative balance in our account, RTS is able to go “into hock” by large amounts (for example. recently by more than $200K when a large order for carbide cubes was placed) in order to procure supplies for the community, as long as the money is recovered at the end of each Fiscal year (June 30). This allows inventory to be held and shortens the waiting time for assemblies. This year though, we are trying to keep the balance near zero most of the time in order to avoid the stress of the occasional wild fluctuations in balance. RTS provides a well-functioning and sometimes even helpful administrative umbrella for this project.

In parallel with this standard assembly project, we still wish to try new things, and that is the focus of the Multi-Anvil Cell Assembly Development Project. This project was long part of the Infrastructure Development part of COMPRES, but in 2014 it was converted to a Facility. This makes sense now because the project is very long-lived, and also because it is dovetailing with several beam line facilities for cell assembly developments.

**Activities in 2014-2015**

1. **Beginning of a carbide project**

It had been suggested by someone – probably Dave Walker – that COMPRES should be involved in developing and testing carbide for the community. At the same time, KL had a separate project with Sandvik Hyperion (which now operates the diamond-making plant in Worthington, Ohio where GE made the first synthetic diamonds) on pressure/temperature measurements in industrial settings. It has been a matter of some interest since Ivan Getting’s paper on carbide was published that several grades of Sandvik carbide did very well in Getting’s carbide strength testing, so there has been wide interest in their possible use in multi-anvil experiments. For that reason, late in 2014 KL used COMPRES funds to purchase a test batch of Sandvik Grade 6UF carbide cubes 25.4 mm truncated to 5 mm for performance testing with the COMPRES 10/5 assembly. The carbide arrived in September 2015 and though the finish was excellent, the truncations were made at a slightly wrong angle, so a correction is being made in the ASU machine shop. This project is in process and the main testing activity will occur in fall 2015. We will also confer with Sandvik on getting the truncations angles correct.

The carbide project will be continued in the next year and will target harder grades of carbide such as the Fujiloy grades also being tested in Japan and at Bayerishes Geoinstitut, and Sandvik grades of similar hardness. In the search for economical alternatives, we will also seek out and test inexpensive grades of carbide that are brought to our attnetion (for example that described recently by Gabrial Gwanmesia, personal communication).

1. **Beam line contributions.**

The Cell Assembly Development Project works closely with several beam lines in developing and providing cell assemblies for beamline scientists and users. In chronological order of involvement, firstly, the GSECARS Large-Volume Press beamline uses several cell assemblies from the project, ordering batches of them for each beam time and providing them for some users. Some users also order batches of their own to prepare at their home institutions to bring to beam lines. These are similar to the offline assemblies but have special windows cut into them for x-rays or use x-ray translucent materials in place of the normal materials used offline. This has been ongoing since 2006 with Yanbin Wang and more recently Tony Yu.

Secondly, in 2012 we were approached by Matt Whitaker to provide DIA assemblies for the NSLS beamline, which has now transitioned to Sector 6 at APS and with the added involvement of Haiyan Chen. We now provide a steady stream of Deformation DIA, ultrasonic DIA, and standard DIA assemblies to the beam line and to some of the users who want to prepare in their home labs. This also involves some back-and-forth and tweaking of assemblies. This year we worked on the recipe for firing pyrophyllite, and we have ordered a new mullite pressure medium for the ultrasonic DIA assembly that is expected to arrive soon from Ceramco.

Thirdly, we have provided cell assemblies to the ESRF beamline using trial D-DIA designs from Nadege Hilairet and Paul Raterron. The designs have progressed and we have been involved in each step of the redesign as cell assemblies for ESRF have been fine-tuned from the original concept design.

Next year, we wish to add boron-epoxy windows to the GSECARS assemblies. This will open up lower-energy parts of the spectrum to diffraction.

We are working on sourcing for sintered diamond anvils to help with the push toward megabar pressures. This has already been a focus of research at GSECARS. Our contribution this year 2015-2016 has been to undertake the machining of conical slits for the first-stage anvils (Figure) and to seek quotations for a second source of sintered diamond for testing (Sandvik). Next year in 2016-2017 we will undertake a program of testing the sintered diamond anvils from Sandvik and comparing them to those from Sumitomo. Sandvik provides a new source for more versatility of methods and also a company with which we could communicate about the basic formula and construction of the anvils.

1. **Offline assembly maintenance and improvements**

The maintenance of the standard assemblies involves making sure that the assemblies to not go “off” in any way when manufacturers change the formulation for the materials we use, as happens not infrequently. Although not glamorous, this work is very very important. Currently we are in the middle of a change in the basic formula of the injection-molded octahedra that form the pressure media for many of the assemblies. One of the two formulas of MgO used in the magnesia-alumina starting mix for the octahedra has been discontinued. We are evaluating the new formula with a replacement MgO to check for changes in shrink factors, densities, or pressure calibrations. This work has straddled the fiscal year change and is currently causing delays in delivery of the 10/5 assemblies (the most popular and the first size to run out of original formula octahedra).

Improvements in the offline “standard” assemblies must be made with care when the assemblies are already standardized (in fact, standardization implies a fixed assembly with no changes allowed). But, with newly developing assemblies we can still proceed with development until they are fully calibrated and standardized. This is true with the 25/15 assembly – the largest in the COMPRES series and still not fully developed.

With the 25/15 assembly, sample diameters as large as 7.5 mm will be achievable. The pressures can be pushed to fairly high values if enough tonnage is available, but on typical 1000-T presses the expected pressure limit is 8 GPa. Still, with such large sample volumes, this is a significant improvement in yield.

An obstacle in the development of the 25/15 assembly was the high cost of some of the larger parts. In particular, the cost of the zirconia sleeve for thermal insulation was eclipsing that of most other parts because it was cored from an expensive block of zirconia and yields were low. This challenge could ultimately be solved by extruding zirconia (another Dave Walker suggestion) but for now an interim solution is to use porous mullite octahedra. This material is very thermally insulating, but suffers the limitation that there is a breakdown of mullite to corundum+coesite at pressures around 6 GPa. As long as we work below this pressure, we can use a mullite octahedron in direct contact with a graphite furnace. This approach opens up a new window in pressure-temperature-volume space with the 25/15.

In 2014-2015 COMPRES funds were used to purchase a mold for making 25 mm porous mullite octahedra (using the proprietary MUL-6 mixture that was first used by our project) and a batch of two hundred 25 mm porous mullite octahedra with holes and slots for multi-anvil assemblies was fabricated. This year we will test and calibrate this assembly and add it to the list of standard assemblies. We also will need to prepare various sample capsules with the large size – between our project and Depths, Inc., the largest precious metal tubes used so far have been 5 millimeter outer diameter.

1. **Pressure and temperature calibration standards**

A relatively new initiative in the project is to create inter-laboratory pressure and temperature standards so COMPRES members can calibrate their own cell assemblies easily. Independently, a pressure standard for 0-10 GPa has been developed under the sponsorship of Sandvik Hyperion (formerly Diamond Innovations) with Technology Program Manager Abds-Sami Malik and Development Engineer Emil Stoyanov. This new reference material has been calibrated and the results recently published (Gullikson et al., 2015; Leinenweber et al., 2015) It is now available for use by COMPRES members; however, in order to supply samples of the starting material (SiO2-GeO2 glass) to interested persons, we need to have a new batch of this material made at Semcon, a glass manufacturer. Sandvik is willing to release the recipe for this glass and we will have it made at Semcon at cost. Following that, the material will be made available to COMPRES members at no cost, to precisely calibrate their lower-pressure COMPRES assemblies up to 10 GPa.

In parallel with this, a small facility is being set up at ASU to produce high-quality samples of other pressure standards. The goal is to provide these standards, also free of charge, to any COMPRES member needing to calibrate their assemblies. These will include Mg2SiO4, MgSiO3, CaSiO3, CaGeO3, and FeSiO3 standards. Although these are fairly standard syntheses, the availability of well-characterized and uniform starting materials will be useful to any laboratory that does not wish to expend the effort to make such starting materials, and will also provide inter-laboratory consistency.

**Budget Summary and Justification for 2015-2016**

The budget no longer includes a salary line for a COMPRES supported technical support person (lab assistant). In practice, in the more recent years, we found that this person was pulled more and more into working to fulfill orders for standard assemblies. Thus it makes more sense in the future to use the cost center side of the project (Research Technical Services) to support this position. The development of new materials and methods is so specialized that in practice most of that work is done by the project PI’s.

One month of salary for PI Kurt Leinenweber is requested to allow the pursuit of the technical improvements outlined in this proposal. $6147.

Materials and Supplies will cover a wide range of things needed for this project: new formula carbide anvils for testing, sintered diamond anvils for testing. Ceramic formulas, extrusions, and parts for trial assemblies are also included. The production of materials for pressure and temperature calibrants for the COMPRES community is also included in this category. 40 K.

Domestic travel is for trips to beam lines (GSECARS, APS Sector 6, others) for testing and development. This includes trial use of new beam line assemblies, as well as development of pressure and temperature calibration materials. Travel to the annual COMPRES meeting is also included. 4 K.

An international travel line is included for a trip to either Japan or Germany to research the large-volume press, ultrahigh-pressure, and large D-DIA and cubic press installations, for helping to guide the anticipated large-volume press initiative in COMPRES III. A similar trip is planned in the current year – so both places will be covered by the two trips. 6 K.

Facility use fees are for multi-anvil experiments performed in the ASU laboratory on behalf of COMPRES. They also cover x-ray diffraction, electron probe microanalysis, and other techniques required for this project. 5 K.

Equipment is for Capital items needed for furthering the objectives of the COMPRES multi-anvil project. An automatic mortar and pestle for production of large samples of pressure and temperature calibrants for the project is requested for this year. 10 K.

Indirect costs: $34,685

TOTAL: $108,328

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**Three primary publications**

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Table 1. Participating laboratories 2014-2015.

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| --- | --- | --- |
| **Institution** | **Location** | **Contact(s)/Advisors** |
| Aarhus University | Aarhus, Denmark | Martin Bremholm |
| Argonne National Laboratories, APS Sector 6 | Argonne, IL | Matthew Whitaker, Haiyen Chen |
| Arizona State University | Tempe, AZ | Kurt Leinenweber |
| Australian National University | Canberra, Australia | Robert Rapp |
| Bayerishches Geoinstitut | Bayreuth, Germany | Dan Frost |
| Brown University | Providence, RI | Stephen Parman, Geertje Ganskow |
| Carnegie Institution of Washington, Geophysical Laboratory | Washington, D.C. | Haidong Zhang, Valerie Hillgren, Venkata Srinu Bhadram |
| Case Western Reserve University | Cleveland, OH | James Van Orman, Audrey M. Martin |
| China University of Geosciences | Wuhan, China | Zhenmin Jin |
| Chinese Academy of Sciences, Physics Institute | Beijing, China | Changqing Jin, L. Sun |
| Delaware State University | Dover, DE | Gabriel Gwanmesia |
| Florida International University | Miami, FL | Jiuhua Chen |
| IPGP | Paris, France | James Badro, Julien Seibert |
| Jacobs, located at NASA JSC | Houston, TX | Lisa Danielson |
| Jilin University Superhard Materials | Changchun, China |  |
| Lehigh University | Bethlehem, PA | Kai Landskron |
| Los Alamos National Laboratories | Los Alamos, NM | Jianzhong Zhang |
| Macquarie University | Sydney, Australia | George Amulele |
| Massachusetts Institute of Technology | Cambridge, MA | Bill Durham |
| NASA Johnson Space Center | Houston, TX | Lisa Danielson |
| National Cheng Kung University | Tainan, Taiwan | Jennifer Kung |
| Renmin University | Beijing, China |  |
| Rice University | Houston, TX | Kyusei Tsuno |
| Seoul National University | Seoul, Korea | Eun Jung Kim |
| Stony Brook University | Stony Brook, NY | Baosheng Li, Xuebing Wang |
| Texas A&M University | College Station, TX | Caleb Holyoke, Zhicheng Jing |
| Universite Lille (and ESRF) | Villeneuve d'Ascq, France | Paul Raterron, Nadège Hilairet |
| University of Amsterdam | Amsterdam, The Netherlands | Wim van Westrenan |
| University of California at Davis | Davis, CA | Charles Lesher |
| University of California San Diego, Scripps Institute of Oceanography | San Diego, CA | Anne Pommier |
| University of Chicago, GSECARS | Argonne, IL | Yanbin Wang, Tony Yu |
| University of Hawaii | Honolulu, HI | Bin Chen |
| University of Michigan | Ann Arbor, MI | Jie (Jackie) Li |
| University of Minnesota | Minneapolis, MN | Jed Mosenfelder |
| University of Nevada at Las Vegas, Dept of Geoscience | Las Vegas, NV | Pamela Burnley |
| University of Nevada at Las Vegas, HiPSEC /Dept. of Physics & Astronomy | Las Vegas, NV | Liping Wang |
| University of Western Ontario | London, Ontario, Canada | Richard A. Secco, Wenjun Yong |
| Vrije University | Amsterdam | Edgar Steenstra |
| Yanshan University, State Key Lab of Metastable Materials | Qinhuangdao, China | Dongli Yu |

Table 2: Multi-anvil assemblies developed and standardized by the COMPRES project

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| --- | --- | --- | --- |
| Assembly name | Peak pressure | Peak temperature | Design |
| 8/3 | 25 GPa | 2319 °C | Rhenium furnace |
| 10/5 | 20 GPa | 2000 °C | Rhenium furnace |
| 14/8 “G2” | 13 GPa | 1200 °C | Graphite box furnace |
| 14/8 step heater | 15 GPa | 1400 °C | Graphite step furnace |
| 14/8 “HT” | 15 GPa | 2200 °C | Rhenium furnace |
| 18/12 | 9 GPa | 1500 °C | Graphite box furnace |
| 8/3 window assembly in-situ | 25 GPa | 2200 °C | LaCrO3 sleeve and rhenium furnace with windows |
| 10/5 window assembly *in-situ* | 20 GPa | 2200 °C | LaCrO3 sleeve and rhenium furnace with windows |
| 10/5 equatorial assembly  *in-situ* | 20 GPa | 1700 °C | TiB2+BN straight furnace, MgO equatorial window, mullite octahedron. |
| 14/8 “G2” *in-situ* | 13 GPa | 1200 °C | Graphite box furnace, forsterite sleeve |
| 14/8 equatorial assembly *in-situ* | 15 GPa | 1500 °C | TiB2+BN step furnace, MgO equatorial window, mullite octahedron |
| Spherical D-DIA assembly | 6 GPa | 1700 °C | Based on Durham sphere and seat |
| D-DIA (Deformation DIA) | 8 GPa | 1400 °C | Stony Brook design made at ASU |
| U-DIA (Ultrasonic DIA) | 8 GPa | 1400 °C | Stony Brook design made at ASU |
| DIA (regular DIA for diffraction) | 8 GPa | 1400 °C | Stony Brook design made at ASU |