HPCAT SOP for Preparation of Beryllium (Be) Gaskets in Laboratory E030, Bldg 434 and cleaning of diamond anvil cells contaminated by broken Be gaskets.

Version 1.8, 09/08/2017 (H. P. Liermann, E. Rod, G. Shen, Revised by C. Kenney-Benson and S. Sinogeikin)

1. Introduction

This is a Standard Operating Procedure (SOP) to prepare Be gaskets for diamond anvil cell (DAC) experiments and load DAC's with Be gaskets in room 434E-030, sector 16 (HPCAT). The dimensions of typical Be gasket are 0.5 mm in thickness and 5 mm or 3 mm in diameter. After pre-indentation, the thickness of the pre-indented part of the gasket is typically less than 50 micrometers.

The main work conducted under this SOP is related to drilling/laser machining of a hole (sample chamber) with the diameter of 10 – 100 microns at the center of the indented area. Drilling/machining involves removing Be of the order of 1µg. Most commonly the drilling/machining is performed using an HPCAT laser micro-machining system and gas-tight Be enclosure dedicated to laser machining of Be parts (See Appendix 1); alternatively the hole can be machined using a dedicated micro-EDM machine with the process conducted under a turpenoid liquid (See Section 3.3). Finally, the gasket needs to be positioned in a diamond anvil cell and loaded with the sample. When outside the dedicated Be area, either the whole cell, or the area directly around the Be gasket must have Kapton protective containment to avoid contamination of the lab and experimental stations in case of gasket breakage.

2. Hazards

Beryllium is highly toxic; inhalation of the dust can result in berylliosis.

3. Controls

3.1. Personal Protective Equipment and Tools

Since all operations related to the handling of Be gaskets have the potential to contaminate the designated fume hood located in the chemical (curtained) area of room 434E-030, personnel working in the fume hood area are required to wear a lab coat, nitrile gloves and protective eye wear. Note that when the user needs to use another piece of equipment during the procedures described below, they are required to dispose of the potentially contaminated gloves and use a new pair of gloves upon reentering the fume hood area so that cross contaminations can be avoided.

Also, tools that have been used in the fume hood have to be cleaned by qualified staff/user before they can be taken out of the fume hood. However, all tools used to load the Be gasket are marked with purple nail polish and should remain in the fume hood. Any gloves and tissue paper used during the loading process has to be disposed as hazardous waste and should be temporarily stored in the clearly marked plastic container (provided). The containers must be turned over to chemical waste management by the chemical safety officer annually. Please follow these rules for the safety of yourself and the others around you. A sign of "Be work area" is posted in front of the fume hood.

3.2. Indentation of Be gaskets

Because there is a chance that the gasket will break during the indentation process, the user is required to cover all openings of the diamond anvil cell with Kapton not thinner than 0.001" (25 μm). If the gasket breaks during indentation, the cell has to be cleaned of any Be gasket fragments and dust as described in Section 3.6 "Cleaning of broken Be gaskets".

3.3. Drilling/machining of Be gaskets

The majority of Be gasket drilling/micro-machining at HPCAT is performed using HPCAT's IR laser micromachining system located outside Be handling area. **When the gasket is transported to the laser-drill, it must be sealed inside the gas-tight dedicated aluminum canister and the canister must remain closed at all times. Under no circumstances may a beryllium gasket be laser-machined in open air.** The detailed procedures are described in Appendix 1.

If laser micro-machining is not available or impractical, drilling of the Be gaskets can be performed using a dedicated micro-EDM machine located in the Be fume hood in 434E-030.

The drill bits used for drilling Be are clearly marked "FOR BERYLLIUM", and can be only used for drilling Be gaskets. A clearly marked brass container that can be inserted into the EDM drilling machine must be used to hold the Be gaskets and the turpenoid liquid. The Be gasket will be drilled under a turpenoid liquid inside the brass container. The turpenoid is used to prevent any beryllium from becoming airborne.

After drilling, the brass holder has to be removed from the EDM machine and the waste turpenoid has to be poured into a clearly marked waste container. The brass insert must be rinsed with iso-propanol. The beryllium gasket may be cleaned in a beaker with isopropanol (not acetone), in the ultrasonic bath located in the fume hood (**It is absolutely forbidden to use the ultrasonic bath in the general lab area!**). After cleaning, the isopropanol needs to be poured into the clearly marked waste container. Used Be gaskets have to be disposed in a Be-dedicated waste container located in the fume hood.

3.4 Loading of Be gaskets and handling of Be loaded DAC in experiment stations.

Aligning of the Be gasket in the diamond anvil cell has to be conducted in the designated fume hood located in room 434E-030. The fume hood contains an extra microscope that has been designated for indenting and loading of Be gaskets.

Because occasionally a Be gasket can break during the experiment and multiple Be fragments as well as Be dust can contaminate the experimental station, all openings in the Be loaded diamond anvil cell need to be covered with Kapton film/tape not thinner than 0.001" (25 μm). This includes the axial holes at each end of the DAC since breakage of the gasket often leads to severe fracture of one of the diamonds, opening a path for contamination through the aperture in the diamond seat. If it is impractical to make whole cell containment (e.g. when parts of x-ray optics such as cleanup pinhole or polycapillary lens has to be close to the sample) it is acceptable to make only local containment of Be gasket (i.e. using thin-walled kapton tube glued on both sided to diamond seats to avoid gaps and openings, see Fig. 1). The enclosure of the DAC/gasket with kapton will limit any possible contamination to the inside of the DAC. The containment should be preserved and **cannot be removed or modified while the DAC is outside of the Be handling area**.

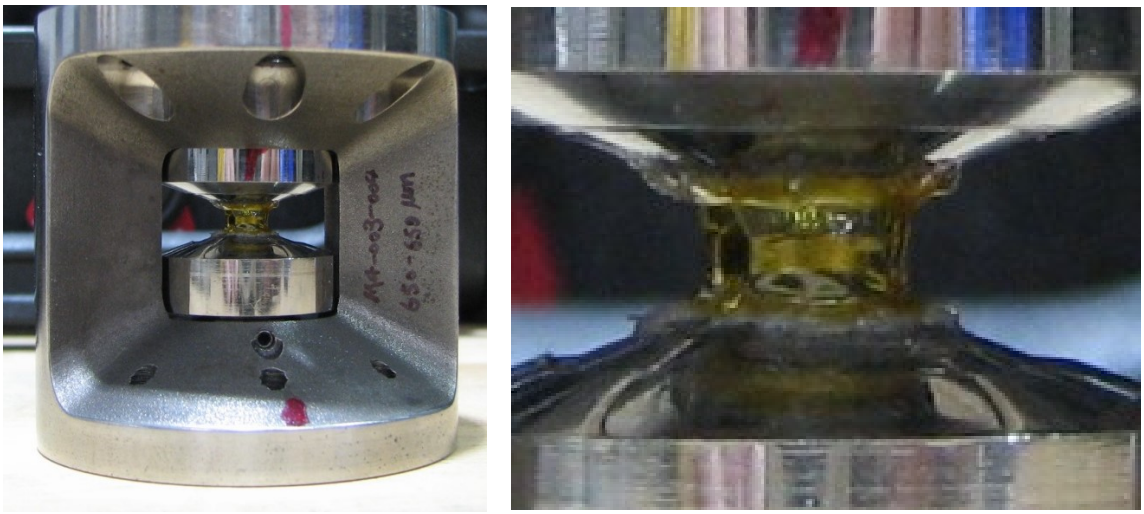

Figure 1. An example of how a Be gasket can be locally contained.

If a Be gasket breaks and the Kapton tape fails, contact the on-duty Floor Coordinator (2-0101) for the proper cleanup procedure.

If a Be gasket breaks during the experiment, but the Kapton tape remains intact, the user needs to stop the experiment and transfer the DAC into the fume hood located in room 434E-030. A qualified staff/user then has to clean the contaminated diamond anvil cell as described in Section 3.6. "Cleaning of broken Be gaskets".

3.5. Taking a Be gasket loaded DAC out of the fume hood.

Before removing a Be gasket loaded DAC from the fume hood, all DAC surfaces must be cleaned to prevent cross contamination. To clean the surfaces, use a Kim-wipe and wet it with isopropanol. Prepare a clean area with a fresh paper, wipe all surfaces, take the cell out of the fume hood, and dispose of the nitrile gloves and the Kim-wipes. Also, when you leave the lab area, leave the lab coat behind by putting it on the coat hanger next to the fume hood.

The Kapton tape containment can be placed on DAC either before or after cleaning. Nevertheless the DAC with installed Be gasket cannot leave Be fume hood / chemical area without properly installed Kapton tape containment. **Only DACs with whole-cell or local containment of the gasket can leave the Be handling area and be taken to outside the curtained area in the lab or to an experimental hutch. The users are not allowed to remove or modify containment while the DAC is outside the Be handling area!**

3.6. Cleaning of broken Be gaskets.

When a diamond anvil cell is contaminated during indentation of the Be gasket or the experiment itself, due to the breaking of the Be gasket, the kapton enclosed diamond anvil cell needs to be transferred into the fume hood in room 434E-030 and has to be decontaminated by a qualified staff/user. In order to clean the contaminated diamond anvil cell the authorized staff/user needs to follow the procedure outlined below:

a) Staff/user needs to wear nitrile gloves, safety glasses, and lab coat.

b) The area where the diamond anvil cell will be decontaminated needs to be laid out with a large sheet of tissue paper that can be removed after decontamination. This is to minimize further contamination of the fume hood.

c) Remove all Kapton tape and watch out for Be fragment. All Be fragments should be immediately transferred in the provided container.

d) Rinse the diamond anvil cell and the diamonds with iso-propanol over to remove any loose Be particles and dust.

e) Swipe all surfaces of the diamond anvil cell and the diamonds with a tissue paper that was soaked in iso-propanol.

f) Examine the diamond culets under the microscope for any remaining Be gasket fragments and remove them with a fine grit sand paper (which needs to be disposed into Be waste container). If the diamonds are broken beyond repair, remove the diamond from the seat and dispose of it in the plastic container.

g) Dispose of all tissue paper, gloves and dust mask in the provided plastic container labeled Be waste.

In case the Kapton enclosure is breached outside of the Be fume hood area, the experimenters should follow procedures outlined in APS ICMS document APS_1191124, which can be found

online at: (*https://www1.aps.anl.gov/icms_files/centraldocs/policy_procedures/user/docs/APS_1191124.pdf*)

4. Authorized Personnel

4.1. Personnel qualified for indenting, drilling, and loading of Be gaskets.

Personnel authorized to indent, drill and load Be gaskets need to take the Beryllium Hazard Awareness Class ESH211 and have training by the HPCAT chemical safety officer, which includes signing a statement that the user understands and agrees to abide by this SOP document.

The last pages of this document contain a list of personnel authorized to indent, drill and load Be gaskets. An updated list of authorized personnel will be posted beside the fume hood in room 434E-030.

4.2. Personnel qualified to clean Be contaminated cells

Personnel authorized to clean Be contaminated diamond anvil cells need to meet the requirements stated under 4.1. and, in addition, take class ESH246 "Safe Handling of Carcinogens". In addition the personnel need to be trained by the chemical safety officer or his/her alternate in decontaminating of diamond anvil cell.

Below is a list of personnel authorized to clean diamond anvil cell that are contaminated due to the breaking of a Be gasket in the diamond anvil cell. An updated list of authorized personnel will be posted beside the fume hood in room 434E-030.

Diamond Anvil Cleaning Authorization:

Name/Affiliation

C. Kenney-Benson, HPCAT

P. Chow, HPCAT

Y. Xiao, HPCAT

Appendix 1. Procedure for Laser drilling of Beryllium at HPCAT

This procedure is an addendum to the existing HPCAT SOPs for Be handling and drilling in the fume hood in building 434E-030. All Be specific work will fall under that SOP while the operation of the laser drilling apparatus will be conducted according to our existing laser drilling SOP.

Laser drilling of diamond anvil cell (DAC) gaskets has clear advantages over electrical discharge drilling in terms of speed, ease of use for the non-specialist, and clean-up of the drilled part. The drawback to laser drilling is the cost of the drilling apparatus and the resulting inability to dedicate one laser drill entirely for use on beryllium. Since the drill must be shared with users of non-hazardous gasket materials such as steel and rhenium, this procedure lays out the necessary steps to prevent contamination of the drill with Be dust.

Procedure

Users will prepare gaskets for drilling in the fume hood of 434E-030. Pre-indentation of the gaskets must be done there, or with Kapton tape covering all the holes in the DAC or encapsulating the gasket in case of cracking of the Be as pressure is increased. Once the gasket is ready to be drilled/machined, it must be mounted in a dedicated air-tight canister (see Fig. 1) which is then sealed. At this point the exterior of the canister has to be cleaned by a thorough rinse of isopropanol and wiped with a Kim-wipe to prevent transport of Be dust to outside of Be area. Then the canister has to be transported from the Be fume hood to the laser drill and bolted onto the drilling stage (see Fig. 2). Laser drilling/micro-machining should be performed according to the laser drilling SOP, with the caveat that the canister never be opened while at the laser drill and all alignment and drilling is done through the sapphire window permanently mounted in the top of the canister.

Figure 1. Gasket mounting plate and assembled canister.

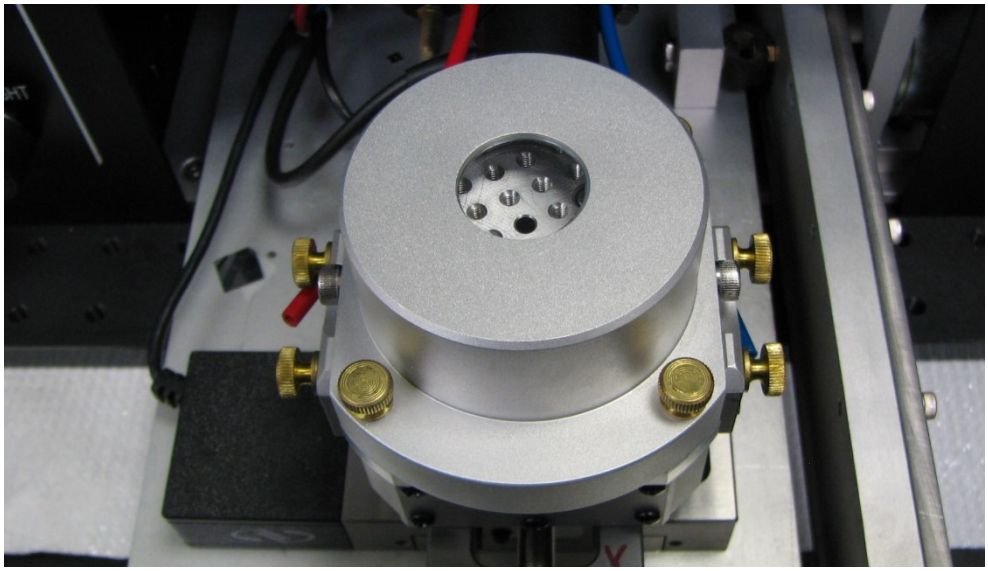

Figure 2. Canister mounted on laser drilling stage.

Once drilling is complete, the canister has to be moved back into the fume hood. The sample may then be removed from the canister and the interior parts of the canister has to be wiped with isopropanol soaked Kim-wipes. Further loading work on the gasket/DAC can be continued according to the existing Be SOP and the canister has to remain in the Be fume hood to await the next laser drilling user.