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Note: Simple and portable setup for loading high purity liquids in diamond anvil cell

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Here we explain a simple and inexpensive procedure to preserve the original purity of the liquid samples during the loading process in a diamond anvil cell. The idea is to keep the sample in frozen form during the loading process while preventing the condensation of the water or other introduction of contaminants. The system can be quickly and easily assembled in a basic laboratory setup. This process can be used for loading some of the common pressure media in a diamond anvil cell. © 2016 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4943258]

Diamond anvil cell (DAC) is one of the major tools in generation of high pressure conditions. While the operating principle of DAC is very simple, the experiments in a DAC are often challenging due to the small sample size and uncontrollable conditions present during the preparation and experiments. One of the common problems in the DAC technique is preserving the sample quality during the loading. The sample is prone to contaminations depending on many factors including the reactivity of the sample and its exposure to the environment and potential contaminants during the initial loading process. The process of sample loading is least susceptible to contamination when a non-reactive solid sample and pressure medium are used. However, in many cases sample or pressure media or both are in liquid or gas form at ambient conditions. Loading samples or pressure transmitting media which are in gas form at ambient conditions is done by using pressure-bomb technique or cryogenic loading (e.g., Refs. 2–9). Both techniques involve submerging the whole or large portions of the DAC outside of the pressure chamber in liquefied (cryogenic loading) or highly pressurized fluid with density comparable to the liquid (pressure bomb) during the loading. Therefore, for preserving the purity of the sample during these procedures, great care in cleaning the gasket and diamond cell and removing any possible contaminant prior to loading is required. For loading materials that are liquid at ambient conditions and have low vapor pressure (which would not evaporate quickly) typically a droplet of the liquid is directly placed on the gasket culet and the cell is pressurized. For loading a liquid sample with high vapor pressure, the process above requires sealing the pressure cell very quickly. Alternatively similar to cryogenic loading, such samples can be loaded into the diamond cell by pressurizing the DAC while submerged in bath of the liquid. In either methods of loading liquids into the pressure chamber, it is likely for the liquid sample to wash or dissolve the contaminations from the surrounding into the pressure chamber.

Here we designed a simple and portable setup in which a controlled size of a solid sample (liquid under ambient conditions) is directly placed into the gasket hole under a stereo microscope at low temperature while preventing water condensation problem. Figure 1 shows the schematic of the setup. A DAC is prepared to the point prior of the sample loading in regular manner, making sure the gasket is securely placed on one diamond and will not move easily. We placed thermal insulation on parts of all the equipment including the DAC and cutting tools for easily handling them at low temperature. A glove-bag with volume of ~17 000 in.3, which is commercially available at low price, is used in the remaining of the procedure.

The bag is modified by cutting two large holes (~30 cm × 30 cm) which will be later patched by layers of heavy duty aluminum foil and sealed on the sides with good adhesive tape such as duct tape or electrical tape. These aluminum surfaces will be cooled from outside of the bag through thermal contact with coolant. We made a small tray from plastic with good machinability and thermal insulation properties to fit under the microscope objective (Figure 1). This tray was filled with coolant and used during the sample preparation under microscope (when using dry-ice as coolant, though holes on the sides of the tray must be made for ventilation).

For loading samples with melting temperature higher than the sublimation point of CO2 (194.65 K), such as benzene (Tm ≈ 278 K), silicon oil (Tm ≈ 223 K), mineral oil (Tm ≈ 264 K), and Daphne oil (Tm ≈ 226 K), the process is very simple. All the required equipment including the DAC and the sample vial are transferred inside the glove-bag. A stereo microscope is placed inside the glove-bag such that one of the holes that we cut earlier will be placed right around its base. From the main opening of the glove-bag we place an aluminum foil above the base of the microscope and taped and sealed it to the sides of the hole. Similarly another piece of aluminum foil will be taped on the other hole. These aluminum foil surfaces will be cooled from underneath for cooling the tools, the DAC, and preparing the sample and loading it into the DAC under the microscope. The main glove-bag port is then sealed around the microscope eyepieces and the bag is pumped out and filled with argon or other moisture-free inert gas.

The plastic tray is filled with dry ice (solid CO2), topped with the copper plate, and is placed on top of the microscope base and under the aluminum foil. A Styrofoam tray filled...
FIG. 1. (a) Schematic of the setup used for freeze-loading of liquids inside the glove-bag. (1) Microscope head is taped and sealed to the insert port of the glove-bag while the main body is inside the bag. (2) Aluminum foil base is taped to a hole made in the bag. This foil is cooled by dry-ice placed underneath and will be used to load the sample. (3) Aluminum foil is taped to the hole that is made in the glove-bag and is placed on top of a dry-ice container. The DAC, the sample, and the tools will be placed on this sheet and are cooled down. (4) Vacuum pump port. (5) Moisture-free-gas inlet. (b) Picture of the setup used for loading frozen benzene in the DAC. The cooled surfaces (aluminum foil) are placed on top of the containers of dry-ice and are cooled from outside. The inset on the lower left corner shows the plastic tray filled with dry ice. The copper plate will fit on the red square.

with dry ice is placed underneath the glove-bag and directly beneath the other aluminum foil surface. The sample and all the tools such as needle to pick up the sample and the whole body of the DAC will be placed on the aluminum foil and cooled to temperatures below freezing point of the sample. For ease of sample handling we replaced the gloves of the glove-bag with regular laboratory gloves. The remaining of the process for cutting, placing the sample inside the pressure chamber, and sealing the DAC is similar to typical loading under microscope. Thermal insulations that are placed on the handles of the tools and the sides of the DAC prevent transfer of heat from the hand to the cold-end which will come in contact to the sample. It is essential for the dry ice to be placed outside of the glove-bag since it usually contains a large amount of condensed water that will be gradually outgassed and condensed on the sample and the DAC. Figure 2(a) shows formation of ice crystals on the diamond when in presence of dry ice inside the glove-bag (despite the fact that glove-bag was evacuated thoroughly and was filled with argon gas, there was sufficient moisture released from the dry-ice).

Some specialized high pressure laboratories use dedicated complex systems designed for loading their samples such as inert-gas gloveboxes with built-in microscopes equipped with low temperature freezers. Variations of the above process can be done in such facilities. However, these equipment are not typically portable for loading samples in off-site experimental facilities such as beamlines and high magnetic field sites and in many cases there are constrains on introducing liquid samples to high purity dry glovebox. Since assembling this setup only takes less than an hour, it can be permanently or temporally built and re-built as needed.

The process above can be easily modified to freeze samples with melting temperature lower than the sublimation point of CO$_2$ by using alternative cooling baths such as frozen 2-methyl butane (2-methyl butane has a melting point of 160 K and can be frozen in a liquid nitrogen bath prior to placing it under the aluminum foils). In this case the plastic tray has to be made without venting holes to contain the liquefied 2-methyl butane for reusing. This process can be used to load pressure media such as ethanol-methanol mixtures which are used in many quasi-hydrostatic experiments. Care has to be taken to avoid frostbite.

The use of flexible glove-bag highly simplifies the cooling process under microscope, easily allows the adjustment of the microscope’s focus without requiring special modifications, and prevents condensation of water on the equipment. The system described here can be easily modified for a variety of loading procedures inside the DAC and can be quickly adapted to be used in any basic laboratory setting.

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