Maintaining Clean Surfaces in Cryogenic Measurement Environments

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ABSTRACT

Surfaces of nanoscale materials of all types must be kept clean to properly characterize their behavior. Materials that are characterized at cryogenic temperatures and in vacuum present additional challenges to maintaining clean surfaces. Any residual contaminants will cryo-pump onto the cold surface of the system, including the sample. We describe a method of independent cooling of the cryostat and the sample. In this method the sample is kept at an elevated temperature, driving off condensates on the surface, while the cryostat is cooled to base temperature. All contaminants in the vacuum are cryo-pumped onto the cold surfaces. These surfaces are maintained at the lowest temperature possible. At this point the sample is cooled to base temperature. Analysis, including residual gas analysis, of the vacuum and surface shows that the condensations on the surface of the sample are reduced to a minimal level.

Keywords: probe station, cryogenic measurements, surface preparation, split flow cryostat

1 INTRODUCTION

In a cryogenic probe station the sample is often in vacuum and the system, including the sample, is cooled to cryogenic temperatures using a flow cryostat. Of course the vacuum is never ideal. As the system cools, the contaminants in the vacuum will cryopump onto the cold surfaces in the system [1]. The most common contaminants are water vapor, nitrogen and oxygen.

As the temperature of a surface cools below the freezing point of these gases, they can form a layer of ice on the surface of the sample. This layer of contamination will, at best, greatly affect any measurements of the sample, and at worst, destroy the sample. The measurement of the transport properties of many materials and devices of current research interest must be done at variable temperatures and with the sample in vacuum [2].

This paper is a description of using a split flow cryostat to control the rate of cryo-pumping on the sample. The sample is a simple piece of Kapton® film. The hydrophilic nature of the Kapton® film makes it an ideal model system. The Lake Shore Model HVTTP6 cryogenic probe station was used.

2 METHOD AND DISCUSSION

2.1 Description of Split Flow Cryostat

A split flow cryostat is a continuous flow cryostat with the ability to independently cool the radiation shields and the sample stage. The radiation shields are arranged as two stage radiation shields. This allows a cold inner shield and a warmer outer shield, and it also provides a radiation barrier between the room temperature vacuum chamber and the inner cold shield. With the split flow design, the sample stage can be warmer or colder than the coldest radiation shield. Keeping the sample warmer than the cold radiation shield makes the radiation shield the cryo-pump in the system. This comes with the small penalty of some radiation heating of the cold radiation shield by the sample. However, the solid angle of the sample seen by the radiation shield is small.

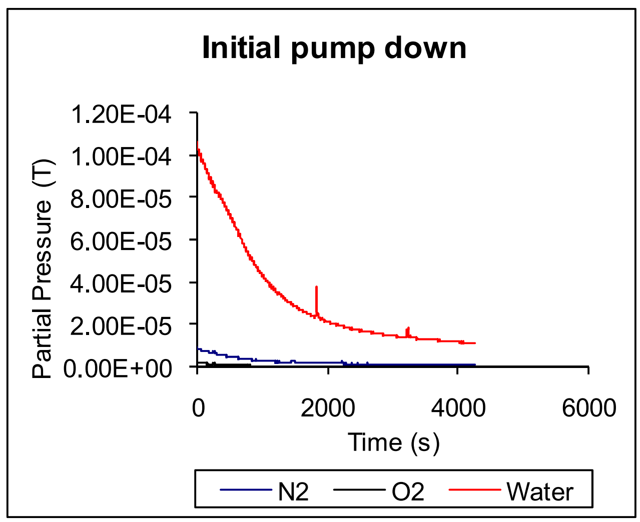

Figure 1: Initial pump down curve

2.2 Initial Sample Preparation

A Kapton® sample was used as the sample in this procedure. It was placed in the HVTTP6 probe station and the system was evacuated. A residual gas analyzer (RGA) was also connected to the cryostat chamber.

The initial pump down curve, with the system at room temperature, is shown in figure 1. Within about 1 hour of starting the pump down, the partial pressure for water is 20 µTorr

2.3 Raise the Sample Temperature for Bake-Out

Initially, the sample was heated to 400 K. Figure 2 is the RGA output vs. time. Notice the increase in water vapor as the sample heats up. This is the water vapor on the Kapton® sample driven off the surface of the Kapton®.

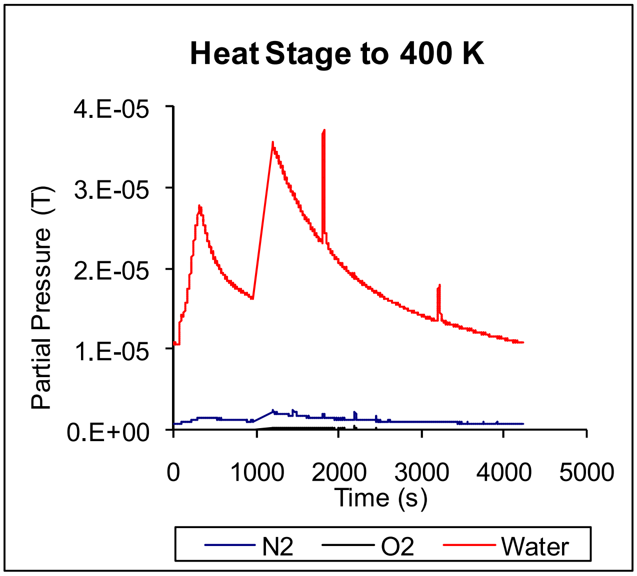

Figure 2: RGA partial pressure as the sample is warmed to 400 K

2.4 Using Split Flow Cryostat to Cool the System

Now the sample and sample stage are at 400 K. They are the warmest surfaces in the vacuum space. The HVTTP6 probe station uses a split flow cryostat. This design allows the radiation shields in the system to be cooled while keeping the sample stage at an elevated temperature. Nearly all of the contaminants will cryo-pump onto the radiation shields. Figure 3 shows the partial pressure recorded by the RGA during the cool down of the system.

Notice in figure 3 that the partial pressure of water has decreased from 10 μTorr at the end of the data in figure 2 to 3 μTorr in figure 3. This decrease in the partial pressure is due to the cryo-pumping effects of the cold radiation shields.

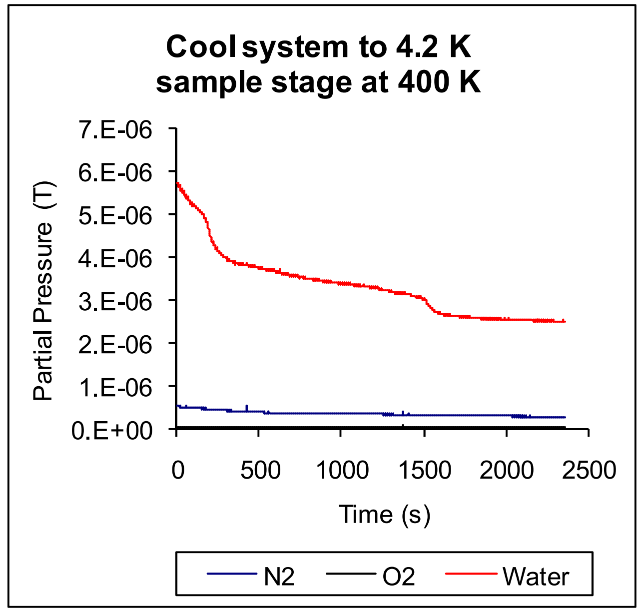

Figure 3: Partial pressure as the system is cooled to 4.2 K. Sample is at 400 K.

2.5 Cooling the Sample to 4.2 K

Now that everything in the system, except the sample stage and sample, are at 4.2 K, the split flow cryostat can be used to cool the sample. During this cooling of the sample the cold radiation shields are kept at 4.2 K. Figure 4 show the partial pressure during this cooling of the sample. Notice water partial pressure is nearly constant during the cool down. This is an indication that water was not cryo-pumped onto the sample during the cool down.

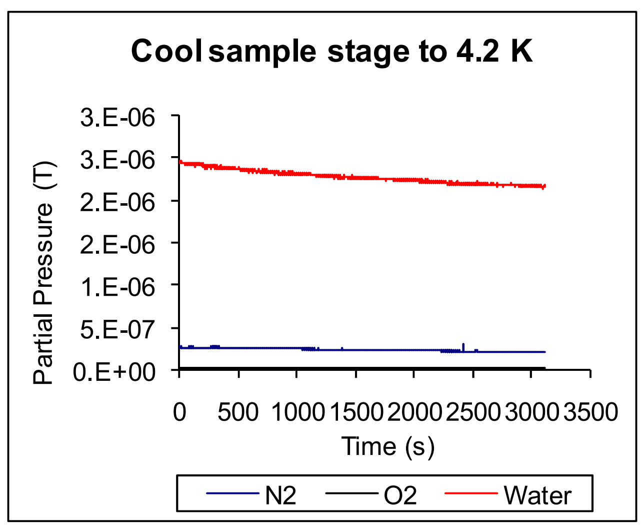

Figure 4: Partial pressure as the sample cools to 4.2 K

2.6 Warming the Sample

At this point the sample can be raised to any desired temperature for measurement. Using the split flow cryostat, it is possible to keep the radiation shields as the coldest surface in the system. Figure 5 is the RGA data from the sample. The sample is back to 400 K, the radiation shields are at 4.2 K. Note that the partial pressure of water remains at 2 µTorr. Compare this plot of the sample at 400 K to the data in figure 2.

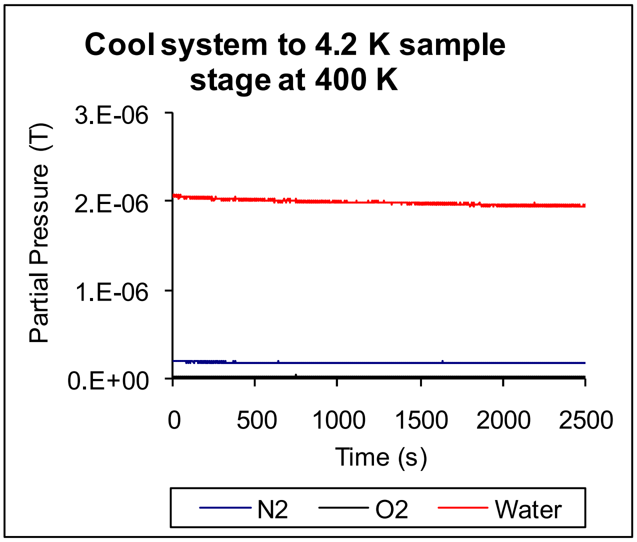

Figure 5: Partial pressure as the sample warms back to 400 K

3 CONCLUSIONS

By using a split flow cryostat in a cryogenic probing station, contaminates from the vacuum reaching the surface of a sample can be greatly reduced. The split flow cryostat allows the sample to be the warmest surface in the vacuum, while eliminating any cryo-pumping onto the sample.

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