Optical Cryostat Realizations at Absolut System

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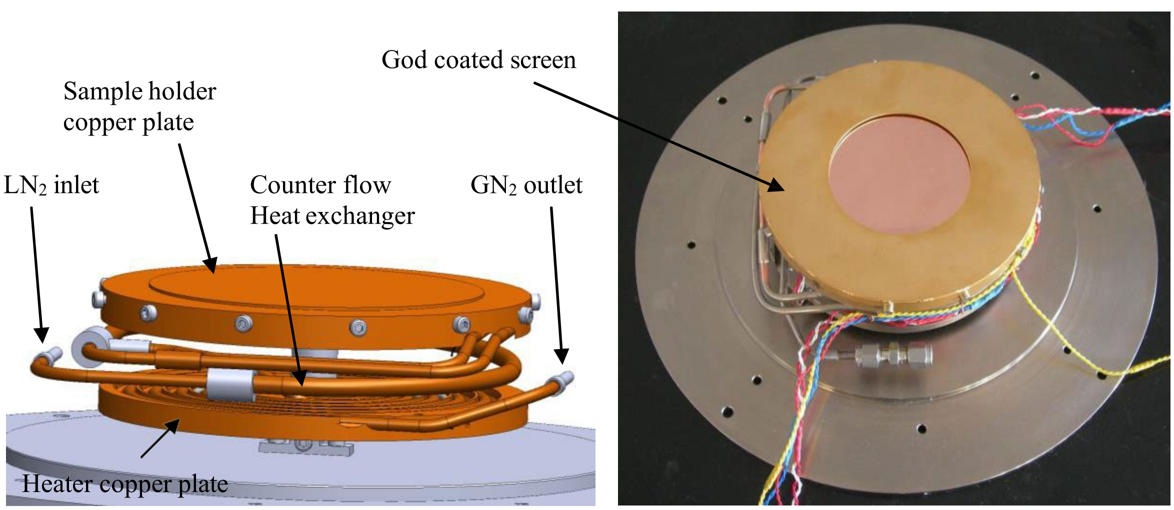
Abstract. This paper describes two kinds of optical cryostats designed and manufactured at Absolut System. The first one makes use of pressurized LN_2 for temperature control of a sample holder in the 80 K - 470 K temperature range. An optical window is implemented above the sample holder to allow for rugosity and 3D distortion of heterogeneous semicon sample assemblies on a wafer. The second one makes use of CRYOMECH remote motor type pulse tube cryocoolers for temperature control of the sample holder in the 3 K - 300 K temperature range. In this type of cryostats, particular attention has been paid to reduce the vibrations exported by the cooler. These 4 K ultra low vibration cryostats are used for characterization of samples via optical windows. Both designs will be presented and the performance reported.

Keywords: Cryostat, cryocooler, Pulse Tube, optical windows.
PACS: 07.20.Mc (Cryogenics; refrigerators, low-temperature detectors, and other low-temperature equipment).

COMPACT PRESSURIZED NITROGEN COOLED OPTICAL CRYOSTAT

An 80-470 K has been designed and manufactured by ABSOLUT SYSTEM for rugosity and 3D distortion of heterogeneous semicon sample assemblies on a wafer. The design of this cryostat consists in two copper plates and one counter flow tube-in-tube heat exchanger as shown on the Fig. 1a.
Pressurized (600 mbar) LN_2 flows from a self-pressurized LN_2 tank through a vacuum insulated transfer line to the counter-flow heat exchanger, then flows to the upper copper plate (sample holder), cross the counter flow heat exchanger and then flows downstream to the lower copper plate. Both copper plates are equipped with a heater element (thermocoax type) and are separately temperature regulated via PID temperature control loops provided by a LAKESHORE 336 temperature controller. The temperature set point of the bottom copper plate is fixed to room temperature. This plate ever warms up the nitrogen flow before exhausting the cryostat whatever the set point is applied at the sample holder between 80 K and 470 K. This suppresses the need of external heater element. As shown on the Fig. 1b, the sample holder is covered with a gold coated copper screen with an aperture of 72 mm.



(a) (b)

FIGURE 1. (a) CAD view of the copper plates and counter flow tube-in-tube heat exchanger. (b) Manufactured copper plates, heat exchanger and screen assembled on the bottom plate of the cryostat.

The optical window consists in a 4 inches diameter Spectrosil® synthetic fused silica. As shown in the Fig. 2, due to this ultra-high purity, the Spectrosil® has excellent optical transmission in the deep UV with a useful range from 180 nm in the deep UV through 2000 nm in the infrared, thus it obviously cuts off the IR from ambient 300 K (about 10 µm).

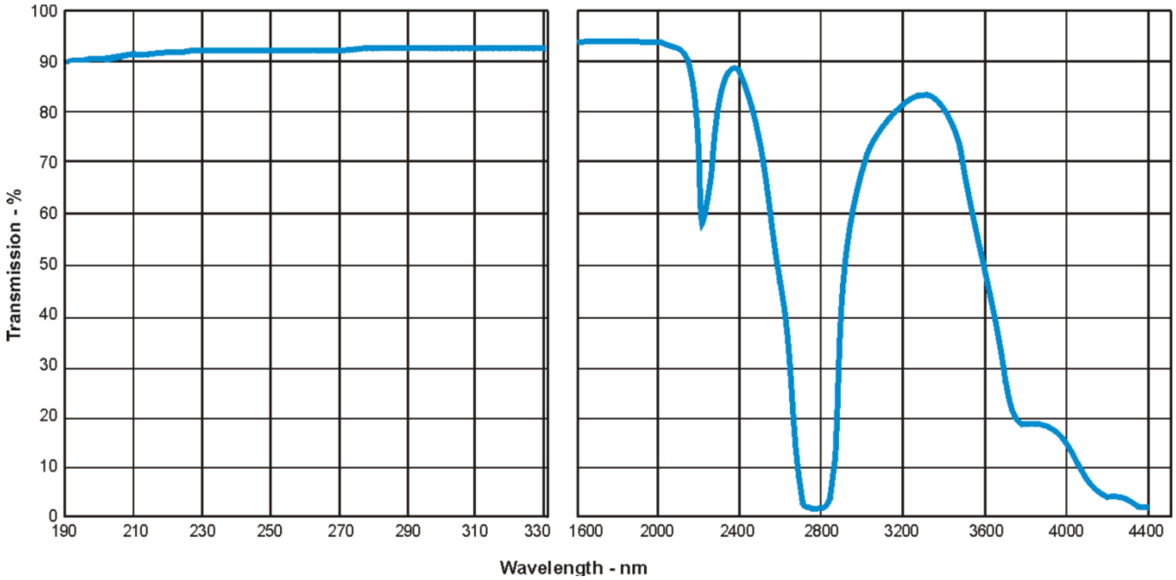


FIGURE 2. Typical transmission of Spectrosil®.

Figure 3 shows the complete cryostat. The overall diameter is 250 mm and the height is 90 mm. The clear access diameter is 60 mm considering a 90° angle of admittance to the surface of the sample.

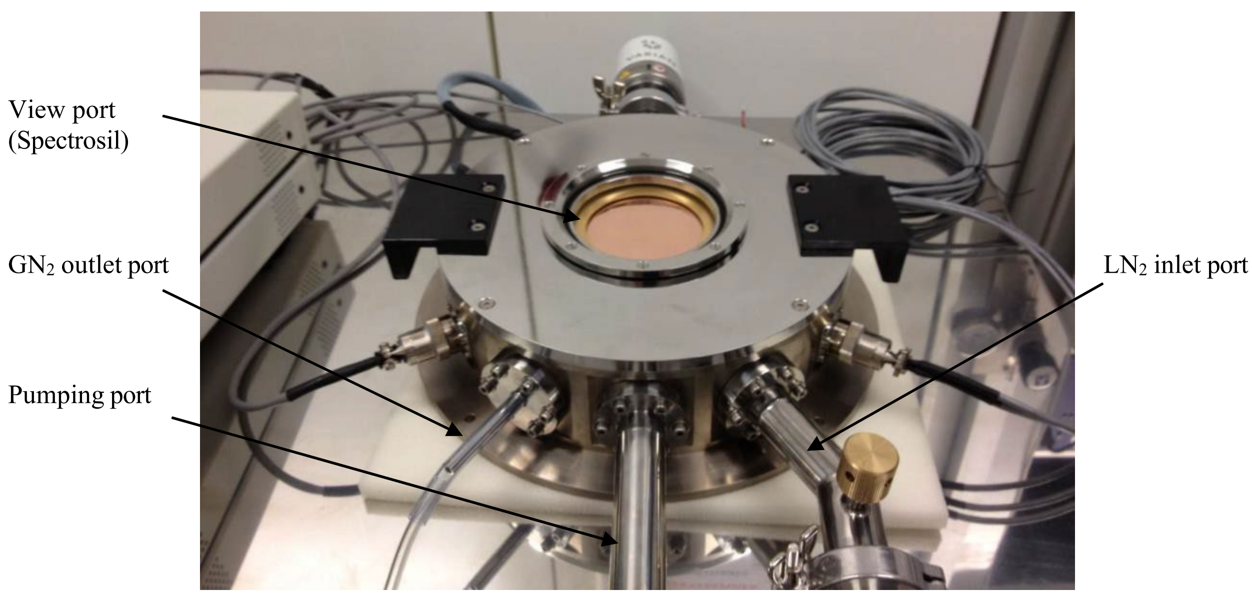


FIGURE 3. Complete cryostat assembly during test phase.

The test result of the temperature regulation loop of the sample holder is reported in the Fig. 4 for several set points in a complete day. As shown, the cool down time to the minimum temperature (81.6 K) is about 3 hours due to the mass of the system and the low mass flow rate (about 1 liter/hour).

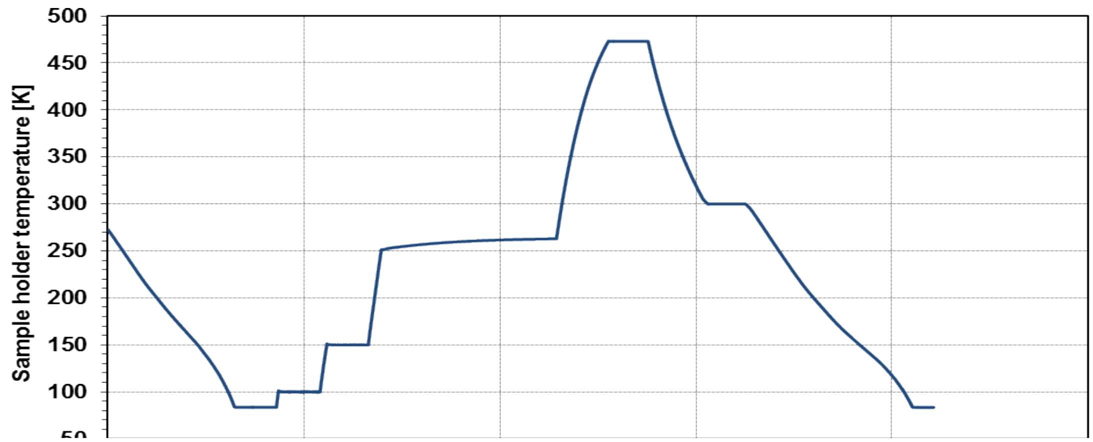


FIGURE 4. Temperature regulation test of the sample holder.

LOW VIBRATION CRYOGEN FREE 4K OPTICAL CRYOSTATS

ABSOLUT SYSTEM designs and manufactures cryogen free cryostats based on two stage Pulse Tube type PT4xx from CRYOMECH Inc. and especially the remote motor (RM) versions.

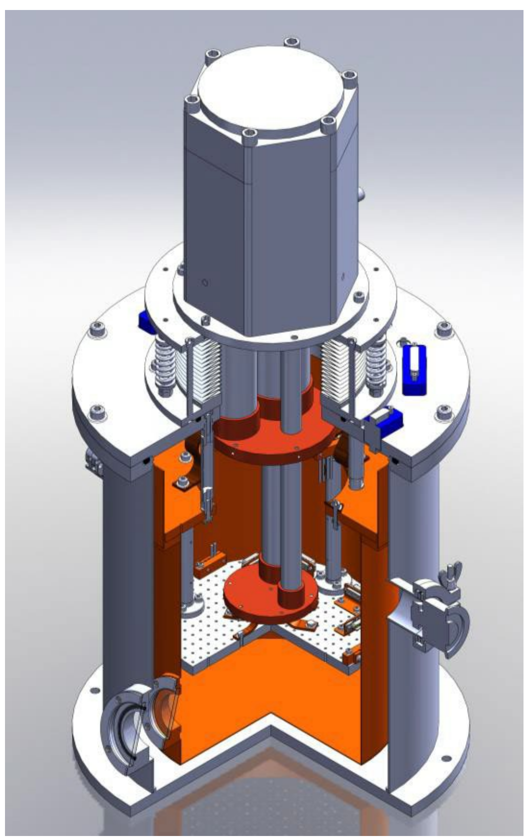


FIGURE 5. Design principle of low vibration 4 K pulse tube cooler cryostat.

Particular attention is paid to reduce the vibrations exported by the cryorefrigerator. To do so, the cold head room temperature flange is mechanically disconnected from the cryostat with an adjustable vacuum tight bellows assembly as shown in the Fig. 5.
An intermediate copper plate is suspended to the cryostat upper flange with some low conductive rods. A copper screen is attached to this plate which is thermally connected to the PT4xx first stage (60 – 80 K) via some conductive flexible links.
For those flexible thermal links, we generally make use of copper foils stacked and press-welded at both extremities as shown of the Fig. 6.
The sample holder is suspended to the intermediate copper plate also with some low conductive rods (either stainless steel or fiberglass depending of the mass of the sample holder) and thermally connected to the second stage (4 K) of the PT4xx. The flexible thermal link is also made with some flexible foils previously described. With these design principles, the PT4xx is completely disconnected and moves freely with very low transmission to the sample holder and the cryostat.
Optical window's materials, dimensions, arrangement and numbers are specifically accommodated upon the customer requirements, on the cryostat vessel and on the copper cold shield.
The sample holder instrumentation can also be customized upon specific requests and comprises, as minimum, a temperature sensor (Silicon diode) and a heater compatible with common temperature controller resistance specifications. The intermediate cold plate is equipped also with a thermal sensor and a heater to control the first stage of the PT4xx cryorefrigerator.

FIGURE 6. Press-welded copper foils flexible thermal links.

The first example of realization given in the present paper concerns a low vibration optical cryostat designed and manufactured for Raman electronic spectroscopy experiment on superconductive mono-crystals in the 3 K-300 K temperature range and in 0.1 MPa-20 GPa pressure range. A photo of the cryostat hardware is shown in the Fig. 7. The cryostat uses a PT407 RM from CRYOMECH. A dedicated capillary pipe is routed from 300 K down to the 4 K plate in order to supply the customer pressure cell with high pressure helium gas. Thus the pressure can be changed in situ during experimentation at low temperature. An optical fiber has been also implemented for optical access. Two 1-1/2" and one 3" optical windows have been implemented both on cryostat envelope and copper cold screen. They are all made of Spectrosil® synthetic fused silica.

The cryostat has been characterized prior to delivery. A no load temperature of 2.9 K at the second stage is reached in 2 hours, while the first stage of the cryorefrigerator stabilized at about 41 K. This leads to about 600 mW available cooling power at 4.2 K for the experiment. With the 50 µm diameter laser beam illuminating the sample, the temperature raised to 3K. Regulation tests of the sample holder from 3.7 K up to 270K have been performed successfully.

FIGURE 7. 2 stages pulse tube optical cryostat for Raman e electronic spectroscopy. (Laboratoire Matériaux et Phénomènes Quantiques, University Paris Diderot – Paris VII)

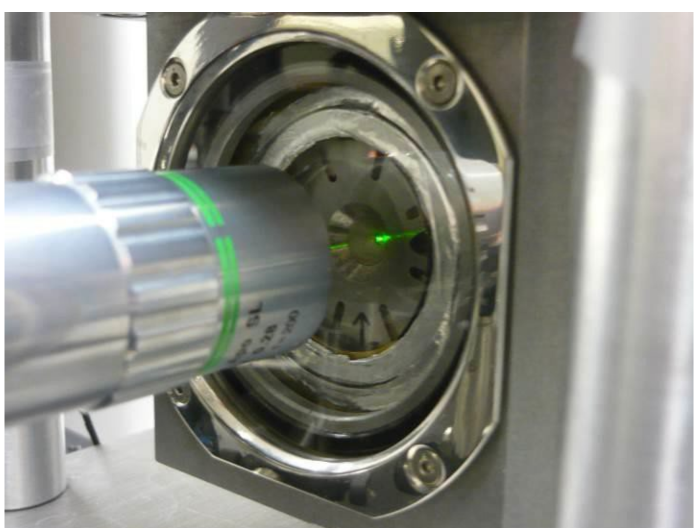


FIGURE 8. Detailed view of the pressure cell behind optical window. (Laboratoire Matériaux et Phénomènes Quantiques, University Paris Diderot – Paris VII)

The second example of realization concerns a cryostat designed for heterodyne measurements of mixers in the THz frequency domain. The required temperature range is 4 K – 20 K. The design of the cryostat is presented in the previous Fig. 5.
The cryostat uses a PT405 RM from CRYOMECH. The 4 K sample holder is a 200 mm diameter gold coated aluminum plate. Four 25 pins Dsub hermetic connectors are used for the electrical cables. Four low losses cryogenic UT85 coaxial cable are routed down to the 4 K plate.
A 2-1/2" diameter window on the cryostat envelope and a 2" diameter window on the cold copper screen have been implemented. The windows are made of high-density polyethylene (HDPE) which cuts off IR radiation from room temperature as shown in the Fig. 9, while providing a transmittance of about 90% at frequency up to 700 µm (THz region not shown on X abscise). A 15° inclination angle has been specifically implemented to suppress reflection and stationary waves.
This cryostat is currently under manufacture and will be tested in July 2013.

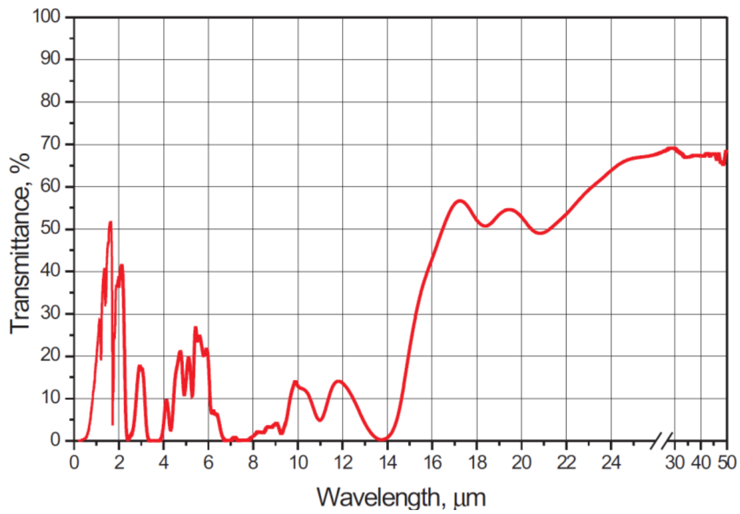


FIGURE 9. Typical transmission of high-density polyethylene (HDPE) in the NIR & MIR regions.

CONCLUSIONS

ABSOLUT SYSTEM develops cryostats specifically tuned to the customer requirements. Pressurized liquid nitrogen flow cryostat or liquid bathes cryostats (nitrogen/helium) have been designed, manufactured and delivered. The cryostat business activity is mainly oriented towards cryogen free low vibrations 4 K cryostats which involve expertise in both cryocoolers and thermal/mechanical management.

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