The new cryogenic facility at LMA

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Abstract. To support the research effort for the third generation of gravitational wave interferometers, the Laboratoire des Matériaux Avancés (LMA) at Lyon, France has developed a new cryogenic facility to characterize optics at low temperature. The new cryostat is installed in a clean room and allows samples to be cooled down to 10 Kelvin in around 12 hours. Currently, two independent experiments have been installed in the cryostat: the measure of the optical absorption of silicon and the measurement of the coating mechanical loss. After a short presentation of the cryogenic and optical setup, preliminary results from the optical absorption experiment will be presented.

1. Motivation

While the second generation of Gravitational Wave (GW) laser interferometer is being installed, a major research effort is underway to shape the third generation of detectors. Whereas the two first generations share similar technologies, the instrument of the next generation will be likely based on different light wavelength, mirror substrate made of new material and different working temperature. For example, the interferometer of the Einstein Telescope [1] dedicated to low frequency will use a laser with a 1550 nm wavelength in conjunction with silicon test masses cooled down to a temperature of 20 K.

To design the Einstein Telescope (ET) and to help predicting its performances, knowledge of the physical properties of the material used for the mirror coating and substrate are required. Unfortunately, properties for thin layers or bulk material at low temperature are sometimes only interpolation or simply unexistent. For example, the optical absorption of silicon at 1550 nm which directly constrains the size of the suspension fiber used to evacuate the heat generated in the mirror, is still unknown (albeit supposed to be very low). An exhaustive list of missing physical data relevant to the thermal noise calculation for ET can be found in Franc et al. [2].

An important research effort focuses now on finding the most suitable materials regarding the substrate and coating for cryogenic gravitational wave detectors [3]. Mechanical loss of uncoated and coated silicon cantilevers were measured at the university of Glasgow [4, 5] as a function of the temperature whereas Q factor of bulk material were measured at the University of Jena [6, 7]. As part of the research toward LCGT [8], the optical absorption of sapphire has also been characterised at low temperature [9].
Our confidence to design successful third generation GW interferometer will be greatly improved by the characterisation of substrate and coating at low temperature. In this perspective the LMA has commissioned a new cryogenic facility in order to extend our knowledge about material at low temperature. This article aims to introduce this facility to the GW community. In a first part, the design and performances of the cryostat will be described. Then in a second part, the two first experiments at low temperature will be presented: the measurement of the coating loss angle and the optical absorption measurement.

2. The cryostat
In this section, the design and the functioning of the cryostat is described. The cryostat was custom built by Air Liquide and was installed in a clean room at LMA in September 2010.

2.1. Requirements and design
One of the main purposes of the cryostat is the measurement of the coating quality factor at low temperature. A rapid turnover of samples in the cryostat is required to test different coating formulas (with various thicknesses or dopant concentrations for example). In parallel of the coating quality factor measurement, a second independent experiment can take place: the measurement of the optical properties of silicon at low temperature. Running two experiments in parallel allows important saving on the cooling cost.

In a nutshell, while looking for a cryostat, three main requirements were derived:

• it must be able to reach a temperature of 10 K
• the low temperature state must be rapidly reached (in the order of a night time)
• the cryostat must be big enough to accommodate two experiments.

The above specifications were met with a continuous flow cryostat. The idea of using a pulse tube cryostat was disregarded since it could generate additional vibration noise. The cryostat is connected to a 100 l liquid Helium dewar and by varying the circulating flow of the liquid helium, the cooling rate can be adjusted. A copper breadboard of diameter 200 mm is mounted on top of the cryostat to install the experiments. The cryostat itself is integrated in the center of a large optical table (2 m × 1.25 m) with the copper breadboard sticking out 50 mm above the table. All the cryostat plumbing and the vacuum equipment are installed below the optical table.

Two temperature probes inside the cryostat are available: the first one is mobile and can be positioned on the sample to test, the second one is incorporated into the copper breadboard and is used for the temperature regulation. The stabilization of the temperature is achieved through a heater (maximum power of 50 W) which is also in contact with the breadboard. The heater and the temperature probes are connected to a controller where the target temperature can be entered. With the controller, we can stabilize the temperature ± 0.2 K around the desired temperature. During the experiments, the state of the cryostat (temperature and pressure) is continually monitored using Labview.

To allow the injection and extraction of probe laser beams, the vacuum chamber and each of the two internal heat shields have three two inches diameter viewports made of borosilicate glass. All the mounts of the cryostat windows were specifically designed to allow a quick and easy removal and exchange of the windows. So the cryostat could later be used with different laser wavelengths than the ones presented in this article.
Figure 1. Evolution of pressure (upper plot) and temperature (lower plot) during the cooling of the cryostat. The pumping started at the time $t = 0$

2.2. Performances
The evolution of the pressure and temperature over time in the cryostat is presented in figure 1. The values of the two temperature probes are shown. One probe is at the level of the copper breadboard whereas the second probe is attached to a large (2 inches diameter) silicon substrate. At the beginning of the measurement the cryostat is at room pressure and temperature. Then at time $t = 0$ we started the primary scroll pump to evacuate the air in the cryostat and 20 minutes later the secondary high vacuum turbo pump was also turned on. 50 minutes after the start of the pump down, when a pressure of less than $5 \times 10^{-4}$ mbar had been reached, we opened the valve to let the liquid helium circulate inside the cryostat bowels.

The temperature of the breadboard inside the cryostat started to cool immediately as shown by the blue curve in the lower plot of figure 1. The situation was different for the sample, which took longer to cool since it is not in full contact with the breadboard (as we will see in section 4). The sample took also longer to cool as it must reach the radiative equilibrium with the inner thermal shield.

By adjusting the flow rate of gaseous helium at the output of the cryostat (via an aspiration pump), the consumption of helium can be controlled. A digital flowmeter was installed to ensure an accurate monitoring and control of the helium consumption. To reach the cryogenic temperature overnight, we found the optimal flow of gaseous helium to be around 6 liter/minute.

In the cooling example presented in figure 1, in less than 6 hours after we close the lid of the cryostat, the breadboard is at 12 K. That is also the temperature of the cantilever used for coating loss measurement as its mount was designed to maximise the thermal link between the cantilever and the breadboard. The cooling in this example was relatively fast requiring a large
flow of liquid helium. In normal conditions, the helium circulating rate is two times smaller and we cool our samples overnight.

3. Coating loss measurement
To measure the coating mechanical loss, a small layer of material is deposited on a silicon cantilever. This small layer has a thickness of the order of the micrometer which is enough to reduce the quality factor of the mechanical modes the cantilever. The dimensions of the cantilever are chosen for the frequency of the first mechanical mode to be around 70 Hz.

A picture of the cantilever in its mount inside the cryostat is shown in figure 2. The mount is made of copper and is bolted to the cryostat breadboard. A vacuum grease is used to maximize the thermal contact between the mount and the cryostat. The bending modes of the cantilever are excited using a contactless electrostatic drive. A 633 nm laser beam reflected by the cantilever is send to a 2 dimension position sensing device located outside the cryostat. The Q factor is directly determined by doing a fit in the time domain of the decreasing amplitude of the resonant motion of the cantilever [10].

During the measurement, the Q factor of the first three bending modes of the cantilever are measured sequentially. Since we only want to excite one mode at a time, one mode is excited only after the previous mode is no longer resonating with a large amplitude. So for each temperature, the measurements of the coating loss angle of the first three modes are repeated automatically and can take up to 2 hours per measurement (but only 5 minutes if we skip the measurement of the first mode).

The first test measurement was done on an uncoated silicon cantilever to serve as a reference. At the time of writing, we are repeating this measurement with a new cantilever and results will be presented soon.
Figure 3. Evolution of the silicon sample temperature when the laser is switched on (left plot) and the measured temperature rise 50 s after the laser is switched on for different input power (right plot).

4. Optical absorption measurement
In parallel to the measurement of the silicon absorption at low temperature described thereafter, we are also doing measurement at room temperature. In that case, we use the photo-deflection technique [11]. A 30 W pump laser at 1550 nm laser is focused on the sample to create a gradient of temperature inside, while a second low power laser at 1310 nm is used to monitor the gradient of temperature created by the first laser. This technique is already intensively used at LMA on other benches with a 1064 nm pump laser to measure 2D and 3D absorption maps on large fused silica mirrors [12].

We aim also to measure the optical absorption of silicon substrate at low temperature with the LMA cryostat. At cryogenic temperature, where materials can exhibit very low specific heat, a small amount of absorbed power results in a noticeable increase of temperature. This technique of laser calorimetry has already been successfully applied to measure the optical absorption of sapphire at low temperature [9].

The silicon sample tested was manufactured by Silicon Materials [13] using the float zone growth method. This silicon has an orientation < 100 >, dopant type n, a purity superior to 99.9999% and a resistivity of 10 kOhm.cm. We choose one of the highest resistivity available on the market since it is linked to low optical absorption [14]. The two uncoated flat surfaces were diamond dust-dry polished giving a surface roughness of less than 0.5 nm RMS.

Inside the cryostat, the silicon sample is resting on two thin holders made of copper. The holders were designed to allow enough contact for the substrate to cool while the thermal link between the substrate and cryostat breadboard must be small enough to not dissipate immediately the heat generated from the absorbed optical power. A probe is directly attached to the sample to monitor its temperature.

The silicon sample is heated with a 30 W laser with a wavelength of 1550 nm. However, in order to avoid impacting the heat equilibrium in the cryostat, we limit the laser output power for this experiment to 5 W (power measured with an external powermeter). An example of the temperature rise when the laser is switched on is shown figure 3.

During the experiment, we keep the temperature rise of silicon sample below 3 K in order to neglect the heat lost by conduction. The rise of temperature $\Delta T$ during the time $\Delta t$ is directly proportional to the amount of heat absorbed $P_{abs}$:
$$P_{\text{abs}} = \frac{\Delta T}{\Delta t} C m$$ (1)

with $C$ the average specific heat of the sample during the measurement and $m$ the mass of the sample. Since the laser power incident on the cryostat is known and we can estimate the amount of light in the silicon sample (keeping in mind that each side of the uncoated silicon substrate reflects 30% of the light), we can derive the absorption per unit of length.

Using the method described above, we found the silicon absorption for a temperature between 15 K and 20 K, to be around 200 ppm/cm (+/- 15%). The power absorbed is proportional to the incident power on the sample (right plot in figure 2) indicating no nonlinear effect.

With the laser calorimetry technique, only the total absorbed power is measured, surface or substrate absorption can not be distinguished. To have an idea of the contribution of the surface absorption to the total absorbed power, samples with different lengths of the same silicon (same physical properties and made by the same manufacturer) have also been bought. A new campaign of measurement will test the effect of the sample length to the absorbed power.

The results presented in this article are preliminary and a very simplistic model has been used to derive the absorption. A more complex model using ANSYS and including the temperature dependence of the material properties and realistic thermal contact has already been developed. This model will be used to get a more accurate measurement of the absorption in the future.

5. Conclusions and perspectives
In this article, the new cryogenic facility recently installed at LMA has been presented. This cryostat is used to test coating and bulk materials at low temperature, as part of the research toward the third generation of gravitational wave detectors.

We spent the last year to learn how to master the cryostat and get familiar with low temperature technology. We are now ready to start new campaigns of measurement as using the cryostat becomes routine work. The two research directions described in this article (coating loss measurement and optical absorption measurement) will be pursued in 2012 with a more rigorous analysis than the preliminary results presented here. It is planned that new coating formulas will be tested to minimise the coating mechanical loss at low temperature. On the other front, in parallel, different samples of silicon from different manufacturer will be bought to determine which one has the lowest optical absorption.

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