Recent developments in the manufacture of cryogenic deuterium targets

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Introduction

Due to the low density of solid, cryogenic hydrogen/deuterium there is significant current interest in producing targets made from the materials for laser experiments. CLF have been researching and developing a system to produce thin films of hydrogen and deuterium ice to deliver such targets.

After first being installed in TAP for experimental beamtime in July 2014 there were issues with the survival time of the deuterium/hydrogen ice grown on the target mount on the base of the pulse tube. Survival times of deuterium ice targets were on the order of ~30 seconds for thick (3mm) layers. This was caused by two effects and limiting them has been the key focus of recent research into the system:

1) In a low pressure environment solid H/D will only survive at temperatures lower than its sublimation threshold. The rate of sublimation is inversely proportional to the chamber pressure and proportional to the operating temperature of the target.
2) Upon target exposure to the outer vacuum sublimed gases will heat up via collisions with warmer surfaces. Any further collisions with the coldhead will cause warming of the surfaces consequently increasing the sublimation rate and exacerbating the effect.

System Overview

During TAP beamtime in July 2014 the fully thermally insulated coldhead, with 2-stage shielding, was able to reach a minimum temperature of 5.8K. However, having apertures open in the shielding and superinsulation for diagnostics and with the motor drive in place, the minimum obtainable temperature was ~9.5K. The system has a theoretical operating minimum temperature of 4.2K and thus limiting the thermal radiation impinging on the coldhead was a main focus for improving the capability of the system.

Thermal profiling research

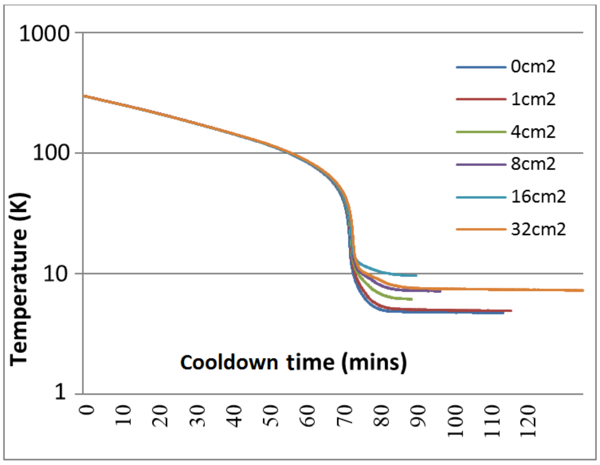

__Figure 1.__ Effects of varying the aperture size in the thermal radiation shielding surrounding the target mount.

Research into the effect of thermal radiation on the minimum system temperature was carried out by directly exposing the coldhead to thermal radiation over a range of aperture sizes.

Figure 1 confirms the expectation that the minimum obtainable temperature is increased by increasing the area exposed to black-body radiation.

Following the results from the thermal radiation studies a tertiary radiation shield was designed keeping the same solid angle to the target centre available for the diagnostics but limiting the total incoming ambient thermal radiation. The previous 2-stage aluminium-alloy shielding was also remachined out of high purity aluminium to improve the thermal conduction.

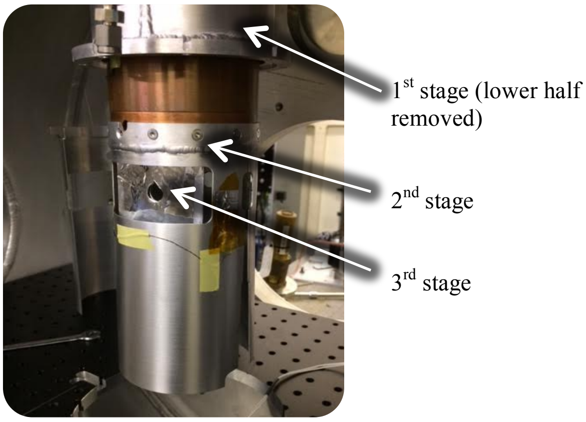

__Figure 2.__ A view of the three stage shielding for mitigating heat loading from thermal radiation.

Design modifications

Coldhead redesign

Following an in depth study into the effects of thermal radiation the copper coldhead target mount was modified in order to minimize its surface area. A further benefit of this was achieving a temperature readout located closer to the actual target foil.

Integration of highly calibrated Cernox TM sensors also allowed for more accurate temperature diagnostics.

Growth chamber redesign

Repeated cooldown cycles with the previous design of the growth chamber eventually produced cracking of the optical windows. This was due to shear stress on the window from the difference in thermal expansion between the stainless steel of the gas cell, the quartz window and the resin bonding the two surfaces.

The optical windows were upgraded from 1mm thick borosilicate glass to 2.3mm sapphire glass due to the latter's ability to withstand more stress caused by thermal contraction.

The cryogenic resin seal bonding the window to the gas cell was replaced with an indium seal design which allows more flex than hardened resin to mitigate the effects of shear forces.

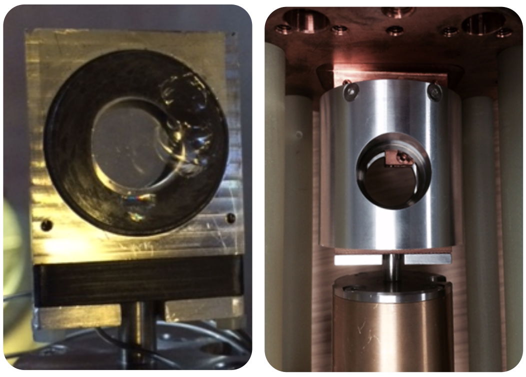

Figure 3. *Left: Previous growth chamber design, with 1mm borosilicate windows bonded with resin. Right: Current design: 2.3mm sapphire glass windows bonded using an indium seal. The optical window of the previous chamber has been damaged by shear stress.*

Target foil studies

The warm gas heat loading effect on exposing the deuterium ice target to the outer vacuum was studied and it was observed that temperature spiking increased with volume of gas inlet into the growth chamber as shown in Figure 4.

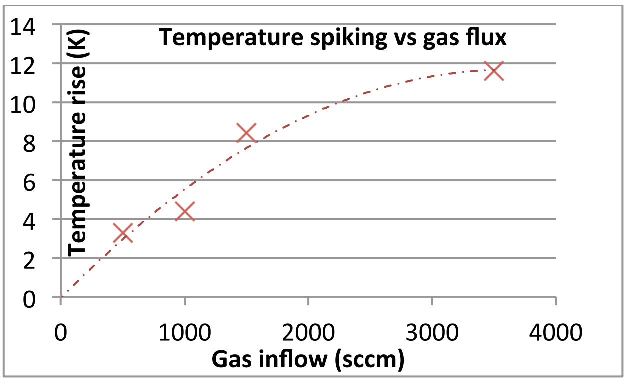

Figure 4. *Temperature spiking of the coldhead when exposing ice target to outer vacuum over different total deuterium gas volumes.*

From the study it was clear that limiting the total volume of gas in the chamber was necessary for prolonging ice survival time after exposure to the outer vacuum.

Prior to the modifications made to the shielding and coldhead of the system a 3mm thick target mount was required to ensure ice survival times of over 30seconds. Using such a target mount liquid deuterium would only flow over the target aperture after completely saturating the rest of the coldhead leading to the high gas volumes.

Target ice survival testing was repeated on the 3mm thick target stalk after installation of the improved shielding and although a large temperature spike began subliming the ice target after exposure a layer of ice remained under vacuum for a period in excess of 2 hours.

Liquid flow tests were then undertaken over much thinner (100µm thick) target mount foils with a 3mm diameter aperture. The direction of the flow in the growth chamber was adjusted to ensure the target material covered the hole in the mount.

The modification enabled target mount saturation with much lower gas volumes inside the growth chamber, in turn limiting the temperature spiking due to the warm gas heat loading after target exposure.

Various foil designs were researched to aid guiding the liquid flow over the target aperture. Figure 5 compares the original 3mm thick mount to the current 100µm thick target foil. The deuterium ice on the older mount fully saturated the copper whereas the recent foil limits the saturation to be primarily over the target aperture.

Figure 5. *Left: Original 3mm thick, 1mm diameter target mount. Right: Current 100µm thick, 3mm diameter target foil.*

Characterisation

Due to the confines of the setup having a front and rear mounted confocal sensor system was found to be impractical for the working distances necessary for experimental beamtime.

A method was developed using a HeNe laser side probe which projected onto a Stingray camera. The system was used after the growth chamber had been withdrawn and the target foil consequently exposed. Before cooldown, a calibration image was taken by focusing on a 100µm wire placed at the centre of the target aperture.

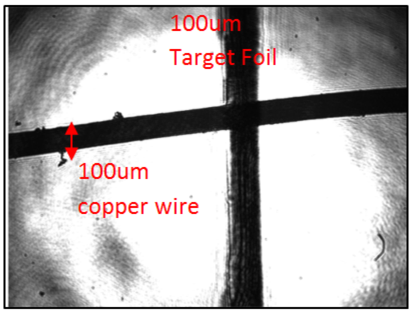

Figure 6. *Calibration image of target foil.*

Thin-film survival rates

After achieving successful growth of a thin film of deuterium over a 3mm diameter hole on a 100um thick foil, and developing a method for characterizing its thickness, the next step was to prove that such targets could be exposed to the outer vacuum without subliming too rapidly.

A liquid film of deuterium was frozen over the target foil and subsequently exposed to the vacuum and monitored at regular intervals to assess its survivability. Over a period of 14 hours exposed to a vacuum of 1×10^{-6}mbar the ice had thinned by 6% proving that both the system modifications and target foil/liquid flow studies successfully prolonged the ice survivability over the original system by several orders of magnitude.

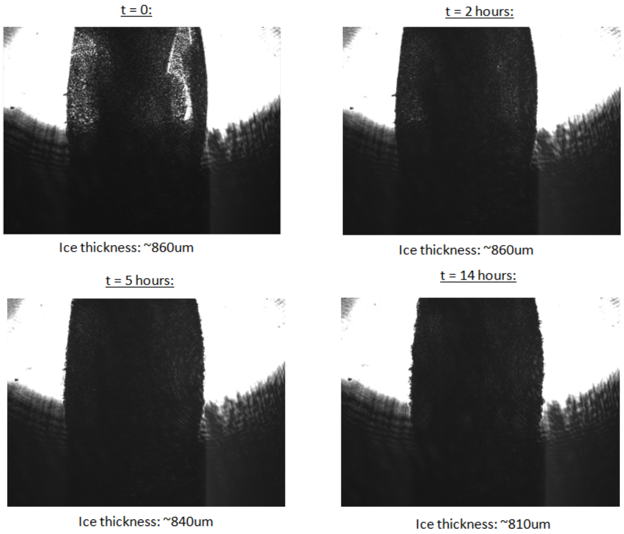

Figure 7. *Time-lapse sequence of deuterium ice survivability over a period of 14 hours. After prolonged exposure the ice surface becomes visibly rougher.*

The thin films could then be thinned further using a Lakeshore 336 PID temperature controller.

Future work

Quartz Crystal Microbalance (QCM) thickness monitor

A quartz crystal microbalance sensor has been designed which is coupled to the base of the coldhead to measure deposited cryogen thicknesses. The quartz crystal is connected to an oscillator supplying a voltage across it which causes it to resonate at a well defined frequency. Changes in the frequency are dependent on the mass of material deposited on the crystal and using the Sauerbrey equation (Eqn.1) the thickness of a given deposit can be measured.

$$\Delta f = -\frac{2f_0^2}{A\sqrt{\rho_q \mu_q}}\Delta m$$

Equation 1. *f_0 – Resonant frequency (Hz), Δf – Frequency change (Hz), Δm – Mass change (g), A – Piezoelectrically active crystal area, ρ_q – Density of quartz, μ_q – Shear modulus of quartz for AT-cut crystal.*

Commonly used QCM sensor heads are much too large to allow for crystal cooling and a custom prototype design has been manufactured to allow the crystal to cool sufficiently for deuterium/hydrogen deposition to occur (figure 8).

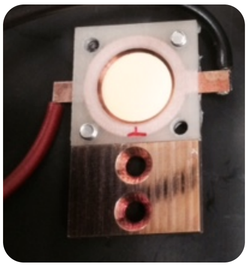

Figure 8. *Prototype QCM cooled crystal holder.*

Improving repetition rate:

Because the frequency of target growth is limited by the need to warmup, vent the target chamber and replace target foils methods to improve the repetition rate of the system are being examined:

- Allowing multiple shots per target foil by having multiple apertures on each foil.
- Increasing the target foil aperture to negate/sufficiently reduce foil damage might enable relayering of the same foil.
- Mechanical foil reloading mechanism.
- EMP shielding to prevent sensor damage following each laser pulse.

Conclusions

A reduction in the heat loading on the cryogenic targetry system as well as research into new target foils has resulted in the ability to grow deuterium ice films of the order of several hundred microns thick with survivability in excess of 14 hours. The advances will allow for much more reliable target manufacture for TAP beamtime in November 2015 and January 2016.

The system can be modified to mount different target foils (currently 100µm thick copper foils are being used) but foils as thin as 12.5µm have been etched to attempt to grow thinner deuterium/hydrogen films during the next few months. A 100µm gold foil will be used for the January 2016 beamtime and there is capability to grow multiple layers of different cryogens.