



# Cryogenics and Water Migration in ETpathfinder

GWADW 2021, May 18 H.J. Bulten, for the ETpathfinder collaboration







Research facility to test and develop cryogenic techniques for future GW detectors

- Being constructed right now in Maastricht, the Netherlands
- 2 arms ~ 10m long: each arm can host 2 Fabry-Perot cavities for small optics (150mm diameter, 3.5 kg Si mirrors) or 1 FP cavity with large optics (ET-size, up to 250 kg mirrors)
- Beampipe diameter 800 mm rather large cryogenic shields may be tested
- Aim is to to develop and test cryogenic techniques and interferometer operations at room temperature, at 123K, at 10-20 K; mirror ASD < 10<sup>-18</sup> m/sqrt(Hz) above 10 Hz (sufficient to be limited by thermal noise)







- Cryogenics in future GW detectors (including KAGRA):
  - Cryolinks to trap water to protect the beam: large cryolinks at temperatures around 100 K are needed.
  - Silicon mirrors operated with high-power beams (Voyager, Cosmic Explorer, possibly ET?) - around 123 K (where the thermal expansion coefficient for Si equals 0) – thermal control around 123K needed
  - Thermal noise (ET, KAGRA) mirrors at 10-20K needed, or if feasible even lower? - requires sets of thermal shields
- Lots of power needed at  $\sim 100 \text{K}$ 
  - ~450 W/m<sup>2</sup> thermal radiation hits the thermal shields in steady operation - (>10kW cooling power needed in ET)
- Little Power needed in equilibrium
  - the laser beam will dump < 0.1W in the mirror even with ETsized mirrors; the thermal radiation and conductive heat losses at the mirror will be below that!
  - Total thermal conductivity of monolithic suspension of the mirror will be limiting the available cooling power to <1W anyways; unless one can produce flexible monolithic links with a large cross-sectional area (single crystal with flextures or something). A high-conductive link will be too stiff.
- Lots of power needed during cool-down
  - About 1GJ of latent heat to cool down a few tons of (Aluminum) payload from room temperature to 1-100 K.
  - Radiative cooling might be sufficient above 120K, but is not sufficient to reach final temperature for ET/KAGRA (>1 year of cool-down required when mirror is cooled only radiatively)



ET last stage: Aluminum marionette (~400 kg) hanging from a Ti wire for angular and large movement control. From that, the mirror (silicon) and the reaction mass (both ~210 kg) are suspended. The mirror is monolithically suspended by 4 silicon crystalline wires (Loss angle <10<sup>-8</sup>)

Thermal shields around the 850-kg payload should limit thermal radiation; input on the mirror in equilibrium <100 mW.





- A monolayer of water binds strongly to metals (aluminum, stainless steel) – 0.7-1 eV binding energy
- Typical residence time of water molecules

$$\tau_{soi} \approx 10^{-13} e^{E_{bind}/k_b T}$$

 Sticking times from milliseconds to days, leads to roughly ~ 1/t\_pump outgassing behavior: the amount of water vapor that comes off the walls of a vacuum chamber can be described by



- Vacuum tower: contains a few hundred square meters of surface area. Thousands of wall bounces before a water molecule reaches a pump.
- Virgo, LIGO, KAGRA : ultimate pressure in mirror towers after a few weeks of pumping around 10<sup>-5</sup> Pa, dominated by water vapor. Very difficult to get much better than that.
- Gas kinematics: impingement rate (wall bounces) as a function of pressure/temperature:

$$J_{A} = 1/4 \rho \langle v \rangle = \sqrt{\frac{\pi k_{b} T}{2 m}} ; J_{A} = p \sqrt{\frac{\pi}{2 m T}} = 3.57 \times 10^{22} \frac{p/[1 Pa]}{\sqrt{T/[300 K]}} [Hz m^{-2}]$$

- At 10-7 mbar pressure, a monolayer of water builds up in about 30 seconds – change of optical properties of the mirror and of mechanical properties of the suspension! Limits the sensitivity severely.
- At KAGRA [43 nm of ice grew on the mirror per day, even though the mirror was protected by the cryogenic shields around it; the partial water vapor pressure at the mirror was around 5x10<sup>-9</sup> mbar.



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FIG. 9. Reflectance oscillations of the ITM (top), ETM (middle), and the arm FP cavity (bottom) of KAGRA due to the molecular adlayer by using the parameters in Table II. In this calculation, the formations of a molecular adlayer for the ETM and ITM start at the same time. Because the original reflectance of the ETM is higher than that of the ITM, the amplitude of reflectance oscillation of the ITM is larger than that of the ETM. The reflectance of the ETM decreases over time due to the optical absorption inside the adlayer. On the other hand, the reflectance of the ITM does not decrease. This is because the ITM reflectance is lower than the ETM reflectance, and the absorption of the adlayer is not large enough to significantly reduce the ITM reflectance of the ITM decreases because the reflectance of the ETM is not large than that of the ITM.

Hasegawa et. al. PRD99, 022003 (2019)



#### Water, vacuum, cryogenics



- Cryotraps:
  - Partial water vapor pressure in towers is relatively high
  - Arm is baked to 250 deg; the monolayer of water is fully depleted
  - Cryotrap separates tower from arm: no water should enter the arm
  - First pump station is 250 m downstream: water will stick to the wall of the beam pipe and bounce >10,000 times before reaching pump
  - Sojourn time hours; outgassing of water in arm will build uo slowly
  - Need to know: how much water can pass the cryotrap? How much can still evaporate?
    - Model and measure!
- Mirrors:
  - Partial water vapor pressure in towers is relatively high
  - Some water may pass holes in the thermal shields and freeze on the mirror or suspension
  - Mirror surface gets a (very thin) layer of ice on it
    - Changes optical properties: how much?
    - Structural damping: outside resonance, noise is proportional to loss angle phi.
    - Ice layer will have a low mechanical loss angle: ice frozen on the monolithic suspension or on the mirror coating might introduce excess broadband thermal noise
    - Model! Measure! Investigate!



Figure 6.24: A liquid nitrogen cryotrap.



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- Vibrational and acoustic noise. Pressure fluctuations of ~  $1MPa@1-2Hz \rightarrow >1N/mm^2$  force applied to coldhead/walls
  - For KAGRA, a low-vibration PT cooler has been designed and developed

Class. Quantum Grav. 31 (2014) 224001

Still many vibration filter stages needed for thermal links to reach ET/KAGRA sensitivity









Cryogenics and water migration in ETpathfinder - GWADW 2021

2 m

Pulse tube

Cold head

Ch2

1st VR stage

2nd VR stag

Commercial Pulse-Tube Cryocooler



From Ushiba(KAGRA), GW workshop Taiwan(2015)

## Liquid Nitrogen cooling



 KAGRA and ET baseline: pulse tube coolers to cool the intermediate shields

**ETpathfinder** 

- Relatively noisy. Many coolers needed.
- Virgo cryolinks: liquid Nitrogen
  - Enthalpy of evaporation: 199 kJ/kg → HUGE cooling power
  - Introduces some noise, nucleate boiling – is this less than PT coolers?
- ETpathfinder: Our cooling scheme uses LN2 for powerfull cooling. We will investigate noises introduced by such a set-up and aim to demonstrate that this is the preferable option for vibration-free cooling of intermediate shields and cryotraps.





# LN2 cooling – vessel support and connections

ETpathfinder: the LN2 vessel and the thermal shields are mounted on a support ring connected to ground – minimal movement is given by seismic activity. However, shields could be suspended when reduction of noise requires it (more easily than suspending a cryocooler



Bottom plate vessel is mounted to a base ring with a 240-mm long, thinwalled, stainless-steel steel support; shrinking is accommodated by 2 flextures that can bend towards the center of the base plate. Conductivity of such a flexture is around 0.002W/K (completely negligible).

Bottom plate of inner shield is mounted on top of the LN2 shield with a Vespel support for larger thermal resistivity (at the inner shield we require a smaller heat load).



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Connection of nitrogen through the tower flange. a thin stainless-steel bellows forms a heat resistance between cold inlet tube and warm flange.



#### Cooling of mirrors, ETpathfinder concept



- Steady-state: about 300W of cooling power at 80K, 2.5W at 30K, and 0.1W at 8K needed.
- Liquid nitrogen @80K, sorption coolers at 30K, 8K, switchable Helium cooler @30K for initial cool-down.
- Conductive cooling of mirror limited by heat resistance of monolithic suspension: max ~30 mW of conductive power. Therefore, a small sorption cooler suffices for the 8K temperature.
  - Sorption coolers: thermal compressor, negligible pressure ripple. See talk Marcel ter Brake, Arvi Xhahi.
- Heat links of sorption cooler to marionette, reaction mass: ultra-pure Al (KAGRA design), 32 wires with 0.15 mm diameter.
- 3 sets of thermal shields: passive (red), LN2-cooled (green), inner shields (blue) at ~30K.
- Pipes around the beam to shield the mirror from thermal radiation: contain conical baffles at the end (not shown), about 50 mm opening diameter.



Mirrors: 2x3.5 kg Si – emissivity ~ 0.5. Marionette, reaction mass, safety structure ~2x21 kg Al-6063; emissivity 0.9 for radiative cool-down, Inner shield: double-walled Al-6063; total mass 130 kg. Options with inside black (emissivity 0.9) or shiny (0.1). Outer shield: mass 180 kg. Emissivity 0.1, option inside 0.9.

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### Shield design, holes and openings



- Section around beam pipe: ETpatfinder: beam spot ~ 2.5mm radius – 1 m of pipe around beam with 25mm radius baffle enough to reduce opening angle to <1 msrad and thermal radiation load to <0.01%</li>
- Einstein telescope: much larger beam radius: about 40 m of cryogenic shield around beam needed to shield mirror from environment.
- Holes:
  - Track movement of water molecules to determine
    - 1) if water can be pumped off initially (monolayer of water)
    - 2) when shields are cold, water vapor from outside should freeze on the shield and protect the mirror.
  - Holes allow outside thermal radiation to enter cold area: track also thermal radiation:
    - Wall bounces
    - Where is it absorbed/emitted.
  - Ray-tracing simulation to track thermal radiation and water molecules from all surfaces; these determine the view angles between surfaces and the migration of water in the system.



Simulation code (raytrace): randomly start particles at the surface ; in case of heat flows: generate photons with weighed emissivity; keep scattering until it is absorbed at another surface. The simulation gives the effective view angles and radiative transfers between all surfaces. Heat flows and temperatures of all volumes can be calculated as a function of time by integrating the system of coupled differential equations.

In case of vacuum properties: generate molecules at the surfaces, and keep rescattering until they reach a pump. Store traveled distance and wall bounces. These results determine local impingement rates, pump speeds, adsorption rates, ice build-up, and the evolution of partial pressures at different parts in the system.

The numerical evaluation of these stiff coupled differential equations is complicated: the time constants span 10 orders of magnitude (milliseconds to weeks) so very small steps in time are necessary.



#### Thermal calculations





- The thermal simulation code has been used to design the shields and determine the required cooling power.
   Radiation is tracked between all surfaces, and conductive flows are calculated through all connected bodies. Heat currents and temperatures of several bodies are shown.
- In this calculation: Shields from AI-6063, liquid Nitrogen flow adapted to keep the bottom of the LN2 shield to 77K. Helium-loop switchable cooling provides 30W of cooling power; sorption coolers 2.5W at 30K and 0.1W at 8K.
  - Currently we are re-designing the sorption cooler scheme, we intend to apply an extra thermal shield at 15K.
- The LN2 shields can be cooled to 80K in about 1 day of time. The inner shield can be kept at 300K during that time if a 120-W heater is applied. The temperature gradient over the shield (top to bottom) is about 10 K in this simulation, the gradient over the inner shield is <0.1K.</li>
- For operations at 123 K, the inner shield and payload can be cooled to the working temperature in about a week. The ambient mirror temperature can then be kept stable at 123K by applying moderate heating to the inner shield (about 5W is required)
- For operations at 10K, about 2-3 weeks of cool-down time is needed. At equilibrium, the heat load on the cold finger at 8K is only about 50 mW; the load on the inner shields equals about 3W, and the load on the liquid nitrogen is around 200W.
  - Liquid Nitrogen consumption: about 14 l/h for the LN2 shields in 1 arm (including 4 l for sub-cooling and loss in the feed lines) and about 30l/h for the sorption cooler (see presentation of Arvi Xhahi).



#### Water Outgassing, model calculations

• The same shield configuration is used to model water outgassing. We assume a Temkin-like isotherm; average binding energies of the monolayer varies from 0.7 eV (fully occupied , eta=1) to 1.05 eV (fully depleted, all sites are available):  $E_{bind} = E_{min} + (1 - \eta)(E_{max} - E_{min})$ 



• However, also adsorption takes place. For water we use the coupled equations:

$$\eta'(t) = -\eta(t)/t_{soj}(\eta) + \frac{J_{impingement}}{n_{mono}} stick(1-\eta); \ t_{soj} = e^{a+b(1-\eta)}$$
$$J'_{impingement} = \sum_{surfaces} \frac{-A_{surf}}{A_{tot}} n_{mono} \eta' \langle \frac{n_{bounces}}{t_{av}} \rangle + \sum_{volumes} (J-J_{vol}) \frac{A_{cond,i}}{\sum_{volumes} A_{cond,i}} \frac{1}{t_{av}}$$

n\_mono is the number of molecules per unit area for a full monolayer

To solve the coupled equations for all surfaces, temperatures, times, pressures, the coupled differential equations are integrated numerically in time. This is tedious because of the stiff conditions (sojourn times of days versus flow towards other volume regions in milliseconds); both Comsol and Molflow could not provide this.

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### Validating the simulation code



- Both Comsol and molflow are not capable of tracking the outgassing rate as a function of history of the surfaces. Nevertheless, we can compare the simulation model with Comsol and molflow for steady-state, and in the case of Comsol for surfaces with outgassing rates that are linear wrt monolayer occupancy.
- Since the simulation for the full ETpathfinder setup with 6 shield walls around the payload was too time-consuming, the model has been tested against a simpler set-up, the Nikhef outgas chamber (Vera Erends, minor project Mechanical Engineering). Results are reported in NeVac Oct 2020 (the Dutch journal on vacuum technology).



Pressure as a function of time. Comparison of measurement (red), this simulation (green) and Comsol simulation (blue) for 2 days of pumping, where the temperature was increased from 300K to 340K for 3 hours after 1 day. In this simulation code and Comsol, a distribution of binding energies between 0.75 and 1.1 eV was chosen for different parts of the surface. The discrepancy between 100,000 and 130,000 seconds for between Comsol and this simulation is due to a different assumption for the temperature. The excess pressure in the measurement is due to hydrogen gas (as was apparent from the RGA).



Outgas chamber at Nikhef. Samples can be brought in at the bottom and raised to the top chamber. Both chambers are pumped with a small turbo pump (56 l/s), the top chamber contains a stabil-iongauge and a Prisma-Pro 200 RGA. The chamber can be heated to 120 deg. by air.



#### Hydrogen outgassing



- The development of the pressure as a function of time could initially not be matched, since the partial pressure was dominated by hydrogen gas already at 10<sup>-7</sup> mbar, which we did not expect. Before we injected water, the pressure was in the 10<sup>-10</sup> mbar region.
- Hydrogen outgassing: typically described as two-step process: diffusion of hydrogen through the stainlesssteel followed by recombination at the surface. This leads to an outgassing rate proportional to 1/sqrt(t\_pump).
- Jousten, "Thermal Outgassing" (1999) https://cds.cern.ch/record/455558/files/open-2000-274.pdf

$$\begin{split} j_{diff} &= \frac{2D}{d} c_0 \sum_{i=0}^{\infty} e^{-(2i-1)^2 t/t_c} \\ c_0 &= 10^{25} atoms/m^3; \ D &= D_0 e^{-E_{diff}/k_{bT}} \ ; \ t_c = \frac{4d^2}{\pi^2 D} \\ d &= 1 mm, \ D_0 \approx 1.2 \, 10^{-6} m^2/s, \ E_{diff} = 0.56 \, eV \\ t_c(300 \, K) &= 1.8 \times 10^9 s \ ; \ t_c(530 \, K) = 1.1 \times 10^5 s; \ t_c(1200 \, K) = 92 s \\ j_{diff}(t \ll t_c) \approx 2 \frac{D}{d} c_0 \sqrt{\frac{\pi t_c}{16} t} \ ; \ j(t > 0.5 \, t_c) \approx 2 \frac{D}{d} c_0 e^{-t/t_c} \end{split}$$

- Characteristic diffusion time: centuries at room temperature, seconds at 1000 deg.C.
- At room temperature, diffusive flow to surface leads to outgas rate around 10<sup>-10</sup> mbar l/s/cm<sup>2</sup> (for a month of pumping). After baking 10 days at 250 deg (or half an hour at 950 deg. C, vacuum firing), this is reduced by 2 orders of magnitude.



Pre-baking: diffusion and recombination at the surface; leads to a concentration profile that is "frozen in". At room temperature, the lower diffusion rate in the metal leads to a low dissorption rate.

Negligible adsorption of hydrogen at room temperature (desorbed gas is pumped away), however, impinging water molecules may dissociate and bind (OH group to the surface), hydrogen atom dissolving in the metal lattice.



## Vacuum and water migration, model parameters





 Measurements made from Feb. 19,2021 till now. Vented with very moist air at Feb. 19 and March 4. Temperatures were changed at regular times. Instantaneous outgassing rates were measured by opening/closing valve between turbo pump and chamber.

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- RGA currents and total pressure do not fully scale; the RGA reading is dependent on the pressure, the ion gauge and RGA degas and pump a little bit, the SEM gain may vary slightly, etc. But the total pressure in Pa (black) is quite close to the total RGA current from mass 2 (red) and mass 18 (blue) measured in microAmps.
- Vented with water vapor on day 1 and day 10.
   Periodically, the valve to the turbo pump was closed to derive the instantaneous outgassing rate.
- The outgassing rate for H2 doubles roughly every 15 deg.
   C. This is consistent with the expected increase of diffusion in the metal.
- The outgassing rate for water doubles roughly every 7 degrees; the partial water pressure falls off much more quickly than the partial hydrogen pressure.
- After a week of pumping, at room temperature, the pressure is almost constant (it decreases <1% per day). But raising the temperature to 90 deg. helps in the hydrogen outgassing.
- The water vapor partial pressure can be reasonably modeled by assuming 5x1019 molecules/m2 for a full monolayer (5 times the geometric value) and a Temkin outgassing isotherm (binding energies in the range 0.7-1.1 eV).



#### Valve closing



- Valve closed for 600 seconds (T=90 deg. C). Pressure increases factor 50. Ion currents for H2, water, and CO increase a factor 200, 4, 1200, respectively. When opening the valve, all partial pressures decrease about a factor of 6 in 1 second, as expected from the modeled pumping speed.
- Since the volume is pumped 2.5 times per second, a linear rise of the pressure of a factor of about 1500 is expected. This is only observed for mass 28 (Carbon Monoxide). The hydrogen pressure increases almost linearly. From this we conclude that hydrogen adsorption is not very large (literature gives an absorption probability in the order of 0.01% per wall bounce).
- The RGA current increases more than the ion gauge. Maybe there is some non-linear effect in the RGA. Also, the increase in total RGA current flattens off; maybe the SEM gain is automatically brought down at pressures around 10-6 mbar.
- The increase in water vapor pressure is tiny; we believe that that is due to the large sticking probability, the large adsorption probability. The wall
  acts as a pump for water with a pumping speed of roughly 100,000 l/s/m2. The initial rise in pressure in the first seconds after closing the valve
  may be due to different temperature/coverage of the valve surface.



These measurements are used to finetune our model. For instance, from these measurements we conclude that our ion gauge has a small pumping speed for H2, in the order of 0.05 l/s (consistent with literature).

We get close agreement between simulated and measured pressure response, both for total pressure and partial water vapor pressure, apart for our hydrogen outgassing rate. The distribution of hydrogen in the metal after venting needs more modeling, as well as the dissociation of water into hydrogen and OH at the surface.



# Vacuum and water migration, model parameters Nik hef



- The simulated water currents seem to follow the RGA currents reasonably well, with the caveat that at low temperatures and low partial pressures, the base level of the RGA seems to be above the model. This can be due to extra water load from recombination (the hydrogen in the steel recombines with the oxygen or OH groups bonded at the surface), stronger-bound water trapped behind the surface layer of the metal (not included in the simulation), or imprecision of the RGA (the currents with SEM for water are below  $10^{-13}$  A)
  - The simulated H2 outgassing is still quite off. In this simulation, I assumed that the newly introduced H2 would occupy a layer of 100 micrometer thick, and that 10<sup>21</sup> atoms/m2 were brought into the surface. The initial outgassing rate in this model is a bit too high and the fall-off too steep; after 10 days the pressure does not decrease in the measurement but in the model it still falls off.

Model calculations for the first evacuation

between the top and bottom chamber was

opened and an extra load of water was

run. The partial water and hydrogen

pressure at the ion gauge position is

simulated and compared to the RGA currents for mass 2 and mass 18 in

microamps. On day 4, the valve in

introduced to the chamber.





#### Conclusions



- Vibrations from cooling and ice buildup on the mirror are important noise sources
- ETpathfinder aims to investigate the water migration through the vacuum system and measure how much water may pass thermal shields and cryotraps
  - Relevant for Einstein Telescope as well
- Simulation model is being tested; turns out that small details have large effects:
  - Re-introduction of hydrogen by dissociative processes during venting with moist air
  - Small but measureable pumping speed from RGAs and ion gauges
  - Small parts such as valve bodies that are either not pumped out or not heated homogeneously
- Hydrogen diffusion in stainless steel is currently not modeled well.
- ETpathfinder aims to use subcooled liquid nitrogen to remove noise from bubbling in the feed lines
  - We expect large reduction in noise from liquid nitrogen cooling, but this needs to be measured!





#### How much does this vibrate?