EUCALL - WP6 – HIREP

Milestone 6.4: M15 / 31.12.2016

„Specification of cooling and heating demands for samples“

In WP6 (HIREP) there exist two kinds of user communities in different scientific fields and there are contradictive demands regarding the sample holders and sample stages. Thus the decision was made in milestone 6.1 to split the specifications for these components into two standards which will satisfy the needs of each community relating to their specific samples and experiments.

The two specifications meet the following experimental demands:

1. **Small sample holder and ultra-high precision cryo sample stage**

Biological compounds such macromolecular crystals in a vacuum environment will lose their solvent and as a consequence will undergo structural changes. In case of macromolecular crystals this loss of solvent is accompanied with an unwanted loss of their diffraction properties. So for diffraction experiments from biological samples it is inevitable to either keep them in a humid environment such as a wet cell, or to vitrify the solvent water by flash freezing of the samples and keeping them at cryogenic temperatures below their glass transition temperature at about 160 K. The latter approach has the advantage that the samples can be kept under vacuum conditions for several hours. In comparison, wet cells require sealing of the compartments e.g. with Mylar foil which leads to an increased background scattering level and bear the risk of a vacuum leakage.

Therefore we intend to implement a cooling option for the ‘small sample holder’ to temperatures below 100 K. Cooling will be realized by flexible copper braids connecting the sample holder to a liquid nitrogen cold reservoir attached to the sample chamber. The copper braids will be designed such that they exert minimal forces on the sample holder in order to still allow for high precision raster scanning of the samples at high speed. We will design a sample mount with extremely low thermal conductivity between the sample holder and the scanning stages. Due to the small size and the good thermal conductivity of the sample holder we expect the required cooling capacity to be in the few watt range. The motors of the scanning stages will be further passively cooled by copper braiding.

2. **Large sample holder and high precision sample stage**

The majority of samples to be mounted on the large sample holder do not demand high temperature stability. For dry solid samples, the main cooling objective is keeping the target holder temperature below the melting temperature of the target. Here it is mainly important to provide a heat sink for absorbed laser energy. Only for exceptional samples, were the phase transition at a given temperature is of interest, temperature stabilization might be an issue. For wet samples like biological substances on a solid substrate, heating is not the problem but drying out in vacuum or dry atmosphere. Here, the entire sample frame should be kept in a suitable environment.

Heat sources can be the motorized stages or the high energy or high intensity lasers.

This project has received funding from the *European Union's Horizon 2020 research and innovation programme* under grant agreement No 654220

To avoid heating of the targets by motorized stages, the designs will ensure good thermal contact of the motorized stages to the outside and isolation to the target frame.

High energy lasers, as used in the HED instrument at European XFEL and at the HZDR, will provide ~100J pulse energy operating at 10Hz. This results in 1 kW optical energy, of which about 50% could heat the target. The rest is either reflected or transferred to radiation, particles, or shrapnel. For high intensity femtosecond lasers, the heat load is typically two orders of magnitude lower.

For high energy laser research, cooling of the target frame of the order of 500W might be required. The standard target frame, as defined in delivery 6.1 of the HIREP work package, is fabricated from aluminium ensuring good heat conductance. The carrier frame, on the other hand, is facility specific. Passive cooling can be provided by fabricating the facility specific parts of the frame from heat conducting material at the facilities where cooling is necessary. For better heat transfer, a thin sheet of indium foil can be used between the target frame and the carrier frame.

The concept of the HIREP work package for the large sample holder will rely on passive cooling for the start. No additional means of heating or cooling will be foreseen at the target frame. The carrier frame and the contact between target and carrier frame will be designed for optimal heat conductance where high energy lasers are used. If at a later stage active cooling turns out being necessary, it will be implemented on a facility level at the carrier frame.

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