EUCALL

The European Cluster of Advanced Laser Light Sources

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Work Package 6 – HIREP

Deliverable D6.5
Sample holder with heating and cooling capacity

Lead Beneficiary: European XFEL

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Deliverable Type	
R = Report DEM = Demonstrator, pilot, prototype, plan designs DEC = Websites, patents filing, press & media actions, videos, etc. OTHER = Software, technical diagram, etc.	DEM
Dissemination Level	
PU = Public, fully open, e.g. web CO = Confidential, restricted under conditions set out in Model Grant Agreement CI = Classified, information as referred to in Commission Decision 2001/844/EC	PU

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1. Abstract

In this Deliverable we present prototypes and define concepts for the implementation of the standard for the large sample holder system to enable an efficient heating and cooling of the sample. The document will not deal with details of the small sample holder, since its design is from the beginning optimized for cryogenic cooling and a heating is technically neither possible, nor scientifically demanded.

For the technical realisation of the heating and cooling one has to distinguish between two types of experiments.

Firstly, experiments with high energy lasers (10 J - 1 kJ) where the cooling of the sample holder will mainly ensure the prevention of the radiation induced heating-up caused by absorption of photons or secondary radiation generated from laser-target interaction. External heating of the sample holder is in most cases not demanded. If a sample needs to be heated, a radiation source like an additional laser is the tool of choice.

Secondly, experiments where the temperature of the sample has to be controlled as an important experimental parameter and no high energy or high power laser is involved. Here the heating will be realised by heaters located on the individual sample frame. For the cooling of the sample to temperatures below 273 K an external cooling source with the sample frame as an integral part of the scanner will be foreseen.

For both cases the mechanical components used for the inner and outer part of the sample holder system have to consist of highly thermal conducting materials. Furthermore, the interfaces between the components have to be designed in a way to reduce the thermal

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2. Introduction

The HIREP work package of EUCALL unifies fixed target and solid sample handling for the participating collaborators. Common software will simplify the access of users to the different x-ray and high energy laser sources. Sample and target pre-characterization procedures will allow analyzing sample quality and localizing points of interest. Common approaches in sample positioning will save development time and allow user support groups at the facilities to focus on the specialties of their facility.

HIREP will make sample characterization and positioning transparent for users of very different facilities. A large variety of samples to be studied, targets for energy conversion or screens and detectors for beam characterization need reliable placement into the beam. The X-ray and laser facilities, on the other hand, have specific demands on their positioning hardware: Vacuum requirements, necessity to screen hardware from electromagnetic pulses or debris, geometry of incoming beams and scattered radiation. To ensure compatibility of any kind of sample or target with any suitable radiation source, common interfaces between facility and sample/target holder need to be defined.

Investigations of the samples of interest for the user communities (Milestone 6.2) and of the demands on sample holders (Milestone 6.1) showed that there are contradictory demands on sample positioning in two subsets of the user community. Biological samples are often available in small quantities only and have to be kept either under conditions of controlled humidity or at cryogenic temperatures in order to prevent them from drying out and thereby changing their structural properties. Therefore these samples ideally require relatively **small sample holders**. Samples under ambient temperatures, on the other hand, profit a lot from **large sample holders**. When the samples are prepared or investigated with high energy lasers, long distances between target positions are unavoidable and a small sample holder is impractical. The project is therefore split in two standards for sample frames serving the different user communities.

The small sample holders allow ultra-precise positioning in combination with maintaining a controlled constant humidity level or cryo-cooling. Heating of the samples is neither foreseen nor demanded by the community.

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3. Cooling of samples in high energy laser experiments

Thermal energy can be transported by the three following ways:

- heat conductance through solids
- heat radiation
- heat convection through liquid or gaseous media

Since almost all experiments with such lasers will be performed in good vacuum conditions (10^{-5} - 10^{-7} mbar), a cooling by heat convection does not occur.

The cooling effect by radiation depends on the temperature difference between the sample and the visible environment. The sample emits energy by thermal radiation but also absorbs the thermal radiation emitted by the surrounding equipment and chamber walls. Since the cooling should prevent the sample from heating up, the temperature of the different radiation sources differ only by a small factor. Hence the net energy release of the sample is negligible.

As a result, the only effective way of cooling the sample is by heat conductance through the sample frame system. Therefore it is crucial to use materials with high thermal conductivity and – because of the activation of high Z elements by laser generated neutrons – a small atomic number.

In this case the material of choice for the construction of inner and outer frame is aluminium. To lower the thermal contact resistance a thin indium foil must be used to ensure a real two-dimensional contact between the components of the sample frame. The outer frame must be an integral part of the sample manipulation scanner (Figure 1). Hence the sample exchange cannot be performed without breaking the vacuum conditions and manual work has to be performed to replace the inner frame with the samples.

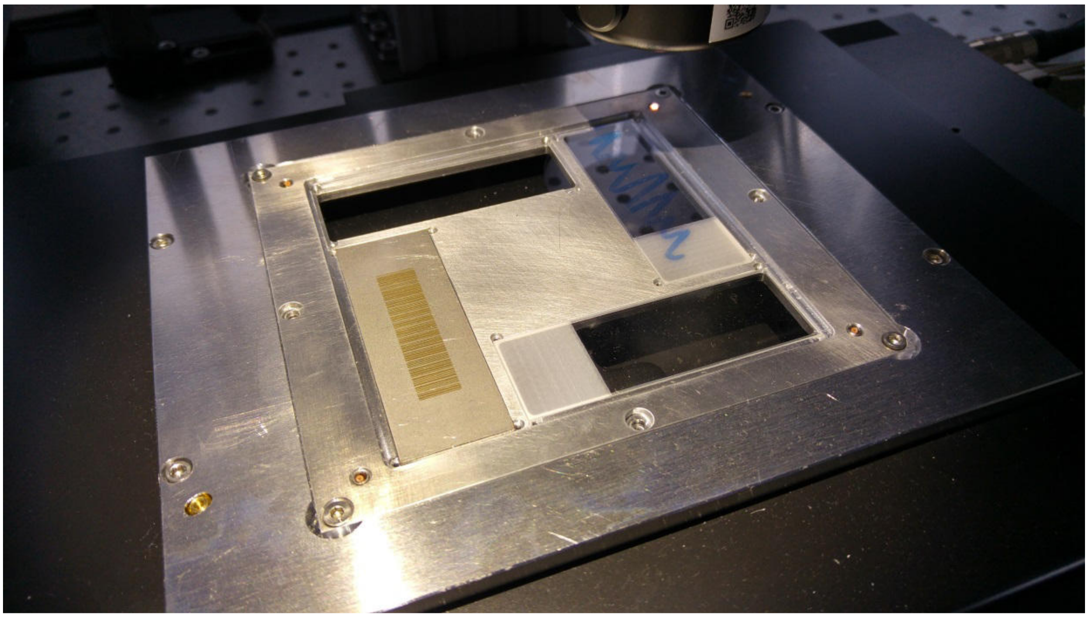

Figure 1: Sample holder system with the outer frame as an integral part of the scanner.

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4. Heating of samples in high energy and high power laser experiments

Laser heating is an established and very effective method of heating small areas of samples with a minimum of impact on the surrounding sample areas and on the experimental environment. Since the heated area corresponds to the focal size of the experiment, the amount of the deposited energy is very small and an influence on neighbouring samples and the sample frame system can be neglected. The outer frame of the system can be designed as a transferable sample holder, so that the chamber can stay under vacuum conditions during the exchange of the sample (Figure 2). No constructional modifications in respect to the standard shown in Deliverable 6.1 have to be taken into account. Additionally, the high thermal contact resistance of the clamping interface between transferable frame and scanner prevents the sample manipulation system from heating.

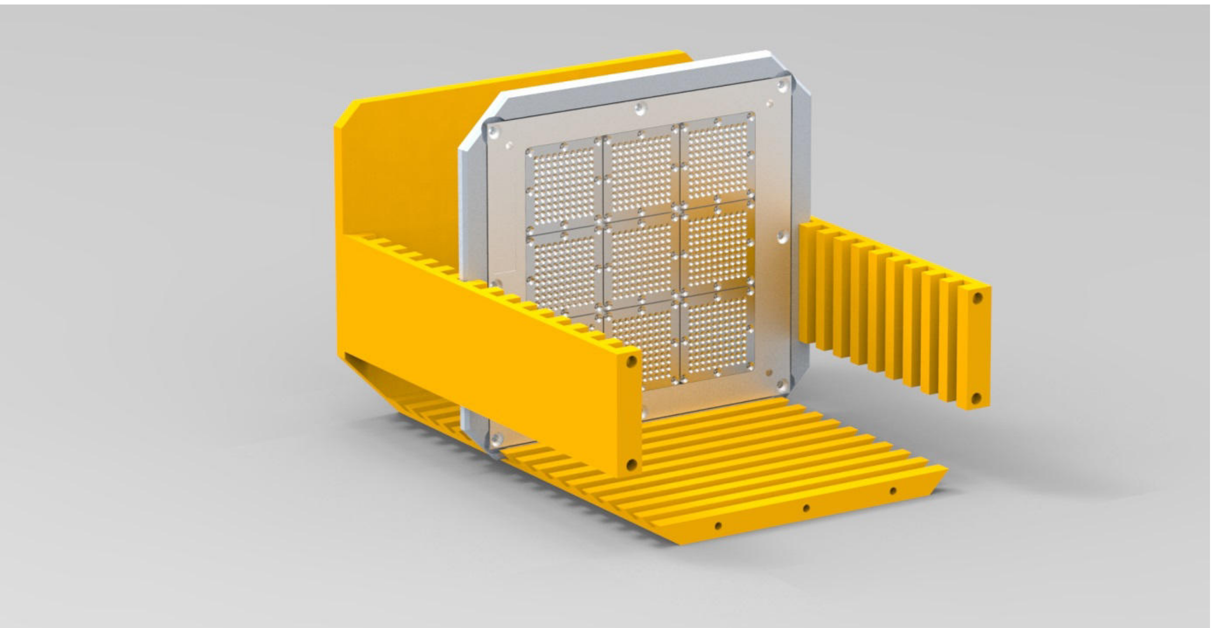

Figure 2: Transferable sample holder for experiments with high power lasers.

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5. Cooling the sample as an experimental parameter – no high energy or high power lasers

For the implementation of an effective cooling of a large (100 mm x 100 mm) sample to temperatures well below 273 K the following two measures have to be taken:

- Heating of the sample by the absorption of radiation has to be eliminated by shielding (gold plated steel) the whole sample leaving only the actual experimental area exposed to the necessary equipment (excitation sources, sensors, detectors, …)
- The coupling between the sample and the cold finger of the cryostat has to be designed in a way that the heat transfer is maximized. Therefore materials with high heat conductivity have to be chosen. Since the risk of activation due to laser induced neutrons can be excluded, copper is the material of choice and can be used if its mass does not restrict the dynamics of the sample manipulation scanner too much. If the mass of the frame plays a role for the scanning aluminium can be used, instead (Figure 1). All mechanical linkages have to be form-fit and realized by screw connections. The interfaces have to be enhanced by the usage of indium foil to ensure a real two-dimensional contact with high cross section and therefore low thermal contact resistance.

6. Heating the sample as an experimental parameter – no high energy or high power lasers

To achieve temperatures above for example 800 K on a sample area of 100 mm x 100 mm a heating power above 800 W is necessary. This power cannot be transported from an external heating source to the specimen without damaging the manipulating scanner or sensitive equipment in the vicinity of the sample. The thermal radiation emitted by the sample will heat up the whole interior and the walls of the vacuum chamber which have to be cooled.

Therefore, the necessary high temperatures can only be realized for smaller experimental areas which separately have to be heated by resistive heaters located inside the inner frame of the sample frame system. The necessary electrical power has to be delivered from the scanner via a clamp mechanism with individual contacts (Figure 3). As a material for the inner and the outer frame titanium or stainless steel can be used. Both substances have a low thermal conductivity to prevent the scanner from heating.

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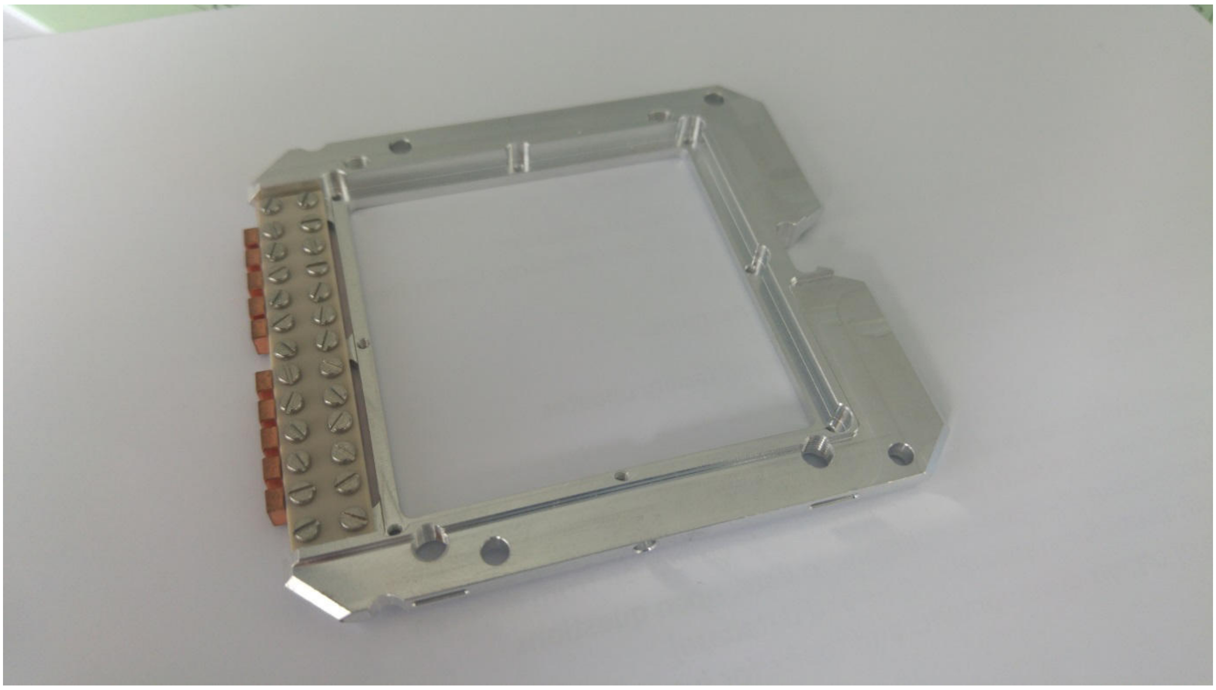

Figure 3: Transferable sample holder system width electrical contacts for resistive heating.

7. Summary

We present two prototypes of sample holder systems which are specialised for either cooling or heating of the sample.

The outer frame presented in Figure 1 is an integral part of the scanners stage and therefore in tests provided a very high heat transfer capacity to prevent the heating up of the sample due to the high energy laser pulses. As frame material aluminium was chosen because of the risk of neutron activation and the interfaces are enhanced in terms of heat transfer by the usage of 50 µm thin indium foils.

For the heating of samples to elevated temperatures in the range of >800 K we build a prototype (Figure 3) of a transferable outer frame with ten electrical contacts to supply ten different resistive heaters distributed on the inner frame, each carrying one individual sample. The heaters are commercial products, which are in the process of being tested. For the material of the contacts copper was chosen and the frame consists of aluminium to ensure a very dynamic scanning scheme.

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