

Poster

Optimizing *in Situ* and *Operando* Experiments: Customized Sample Chamber with Heavy Load GoniometerT. Weigel^{1,2}, M. Zschornak^{2,3}, H. Stöcker^{1,2}, L. Folkers⁴, T. Pippinger⁴, J. Richter⁴, D. C. Meyer^{1,2}

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X-ray radiation offers unparalleled advantages in material characterization due to its remarkable ability to penetrate materials without destroying them. This unique capability enables researchers to investigate the physical properties of materials *in situ* and *operando*, revealing structural transitions that are affected by external factors such as temperature and electric fields. Customized sample environments, tailored to specific experimental requirements, play a crucial role in facilitating such investigations. Although cooling or heating chambers are widely available commercially, the simultaneous manipulation of samples with an electric field is often not possible. To comprehensively address these requirements, we have developed a specialized sample chamber [1]. Constructed from standard vacuum components, this chamber features a versatile heating/cooling system capable of maintaining temperatures from 100 K to 1250 K. Additionally, an xyz manipulator enables precise electrical measurements using a contact needle. The chamber design includes an interchangeable hemispherical dome, providing unhindered access for structural characterization across a full solid angle. Several X-ray transparent domes made from different materials are available. However, due to its size of 24 cm (maximum diameter) and weight of 7 kg, the sample chamber is primarily suitable for use with heavy-load goniometers, which are typically found in synchrotron sources. Since the allocation of synchrotron measurement time is limited and dependent upon various factors like meticulous experiment planning, conducting experiments with fixed wavelengths in the laboratory is beneficial. To increase the versatility of the sample chamber and reduce the need for allocation, it is accompanied by a unique heavy-load goniometer designed for laboratory use. This device is based on the standard setup of the STADIVARI diffractometer from STOE & Cie GmbH (see Figure 1) and features a specially designed configuration capable of accommodating sample environments weighing up to 10 kg. The integration of the customized sample environment with a compatible diffractometer significantly enhances the capabilities for *in situ* and *operando* experiments in the laboratory. This allows for greater autonomy, addressing questions that were previously limited to synchrotron experiments.

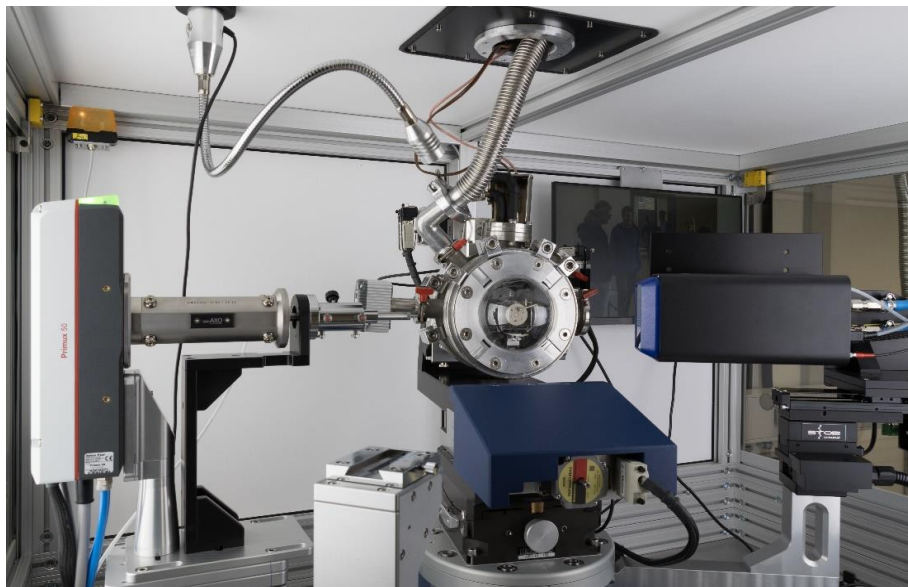


Figure 1. Heavy-load goniometer for a STADIVARI with a customized sample chamber: an optimized setup for *in situ* and *operando* experiments.

[1] Nentwich, M., Weigel, T., Richter, C., Stöcker, H., Mehner, E., Jachalke, S., Novikov, D. V., Zschornak, M. & Meyer, D. C. (2021) *J. Synchrotron Radiat.* **28**, 158