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A versatile setup in reflection mode for *in situ* diffraction patterns acquisition from flat polycrystalline specimens bathed in a liquid medium

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A cell to perform *in situ* diffraction measurements of specimens regularly immersed in a liquid medium has been developed. The assembly (see Fig. 1) has been adapted to work in beamline XRD2 in reflection mode. Basically consists of a polypropylene box with two windows made of polyimide to allow the passage of X-rays (incident and diffracted) at lower angles. The flat sample is placed in the middle of the box that is on top of the goniometer head. For safety reasons the central box is positioned over a tray to avoid spillage of liquid. On the bottom of the cell there is a connection to the pipe linked to the syringe. The syringe is driven by a beamline stepping motor. This way it is easy to synchronise the filling and evacuation of liquid from the cell with the actual data acquisition of diffraction patterns. The idea is to soak the flat specimen with the liquid and then remove it to acquire the diffraction data. This can be done in a sequential way for several hours to monitor the crystalline phases present on the surface (or rather a few microns deep) of the flat specimen. Various reactions can be investigated this way, provided that the sample is flat. Ceramics, metals, and polymers in contact with liquids could be studied. Preliminary tests have been performed in two theta – theta geometry on metal plates in contact with acid solutions to simulate accelerated corrosion. However a large variety of experiments with other geometries, (grazing incidence) could be carried out and of course this assembly could be used on SIRIUS with better time resolution.

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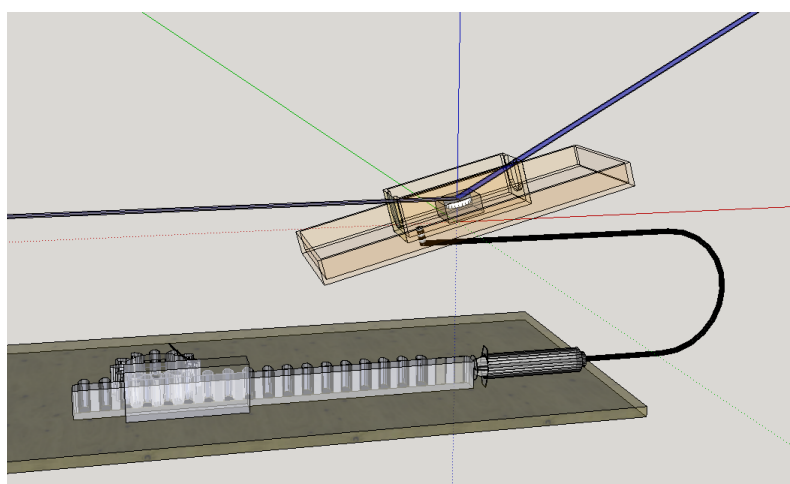


Figure 1. Schematic setup of the assembly showing the cell placed on the goniometer head (omitted for clarity) irradiated by the beam and linked to the automated syringe.