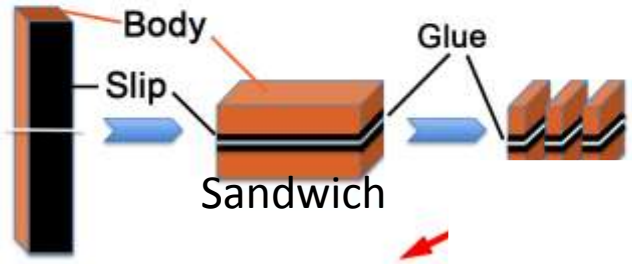
TXM samples preparation
- Thin cross sections -

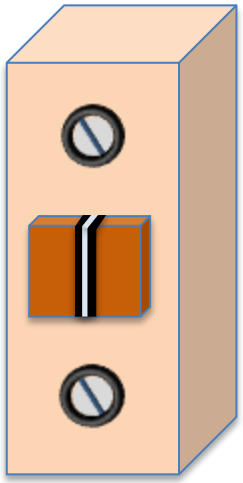
Protocole developped by the team of
Philippe Sciau

Full-field XANES analysis of Roman ceramics to estimate firing conditions - A novel probe to study hierarchical heterogeneous materials. F. Meirer, Y. Liu, E. Pouyet, B. Fayard, M. Cotte, C. Sanchez, J.C. Andrews, A. Mehtab and P. Sciau. J. Anal. At. Spectrom., 2013, 28, 1870–1883. DOI: 10.1039/C3JA50226K

Thin cross-sections of the samples are prepared using a method derived for study of materials in a transmission electron microscopy.

Sections of sherds are cut and glued (Gatan G-1 epoxy) together facing each other to form a sandwich, and thus protecting the two surfaces with black gloss slip from damage. If necessary; it is possible to add glue (Gatan G-1 epoxy) to strengthen the sandwich.

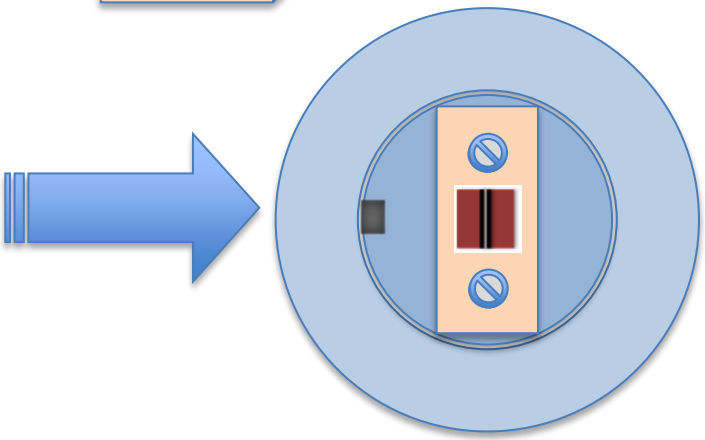




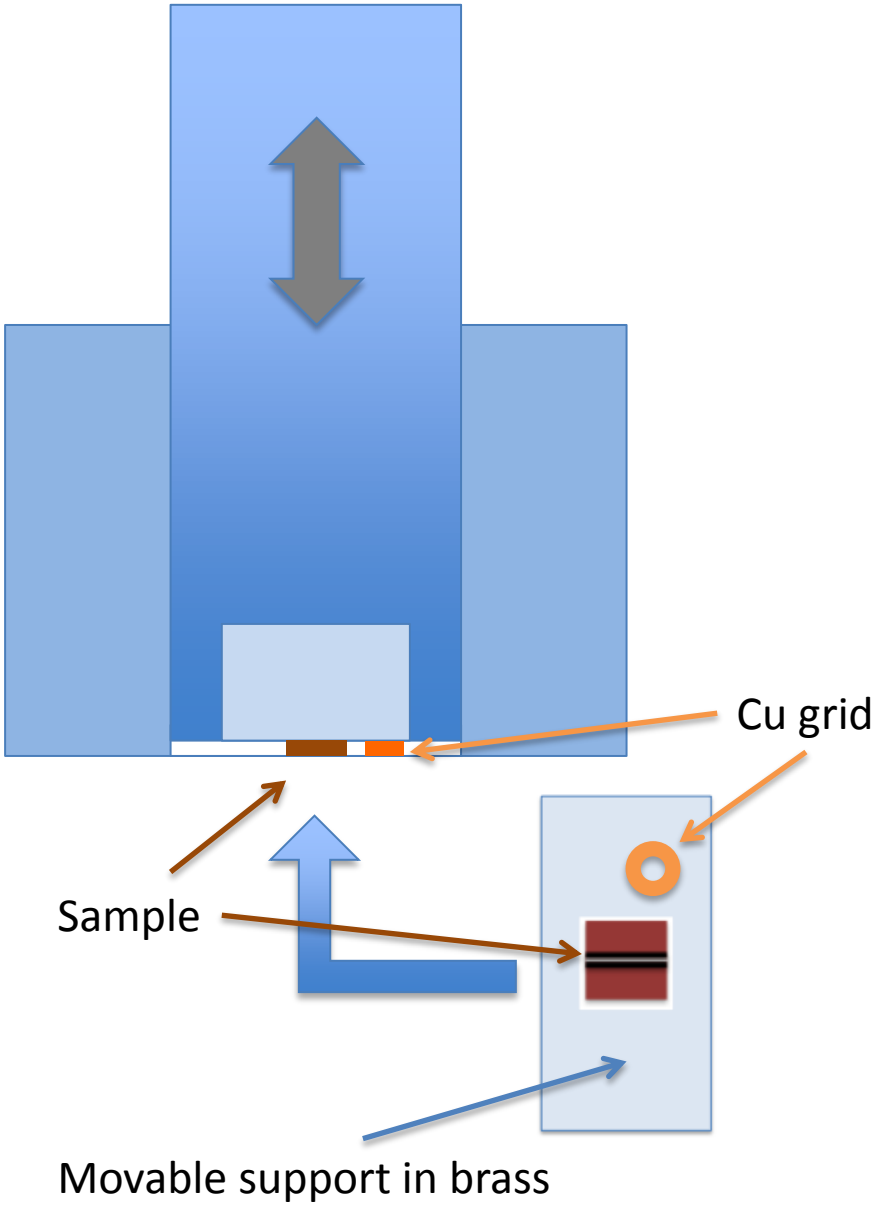
The sandwich is then cut in 300 µm slices. To strengthen the porous samples glue can be used to embed them. Afterward, the prepared slices are mounted on a glass slide (protected with tape) and glue can be deposited on top.

Movable support in brass

The slice is bonded with a dental wax on a movable support in brass.

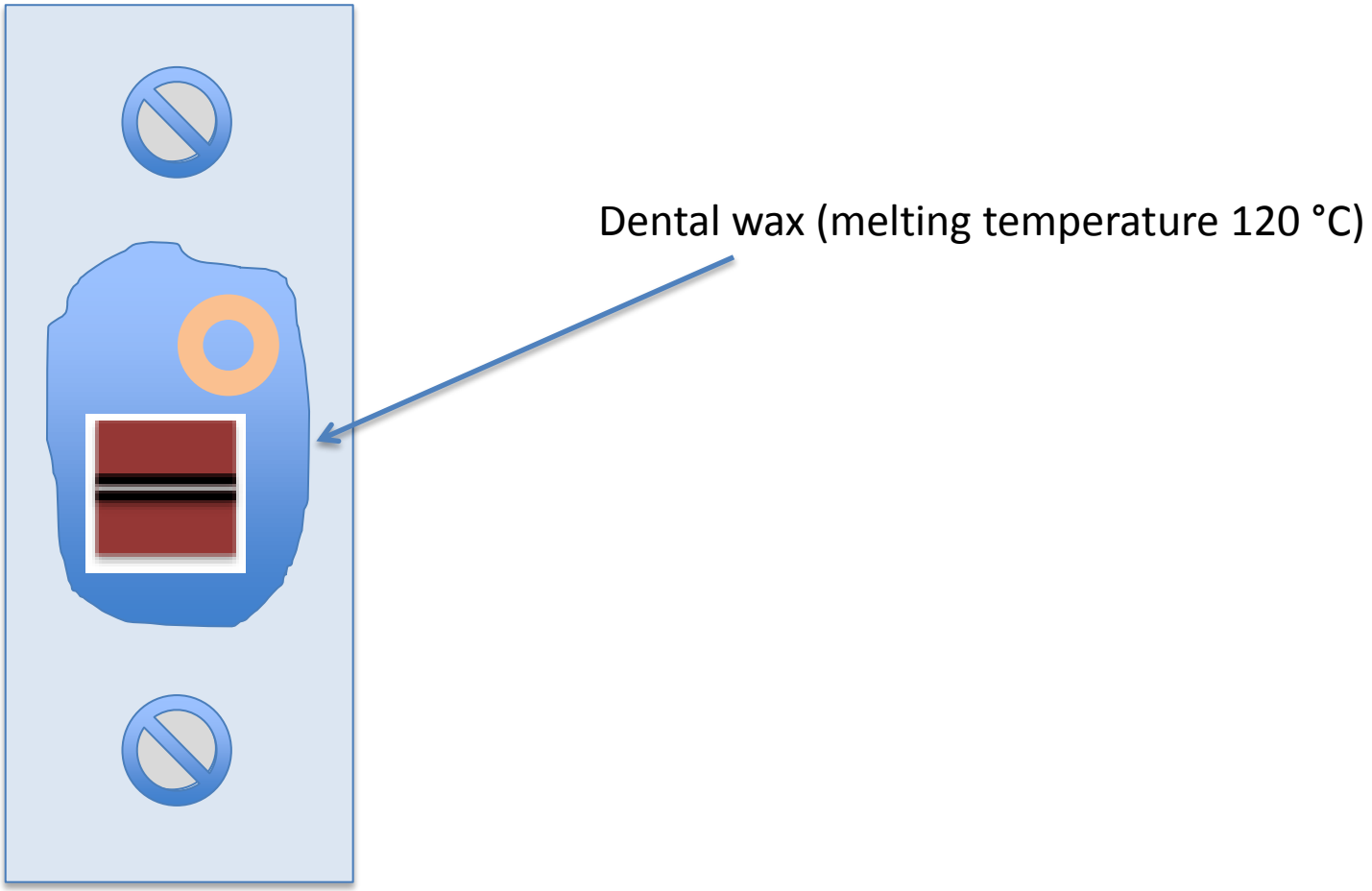


Polishing holder with the movable support

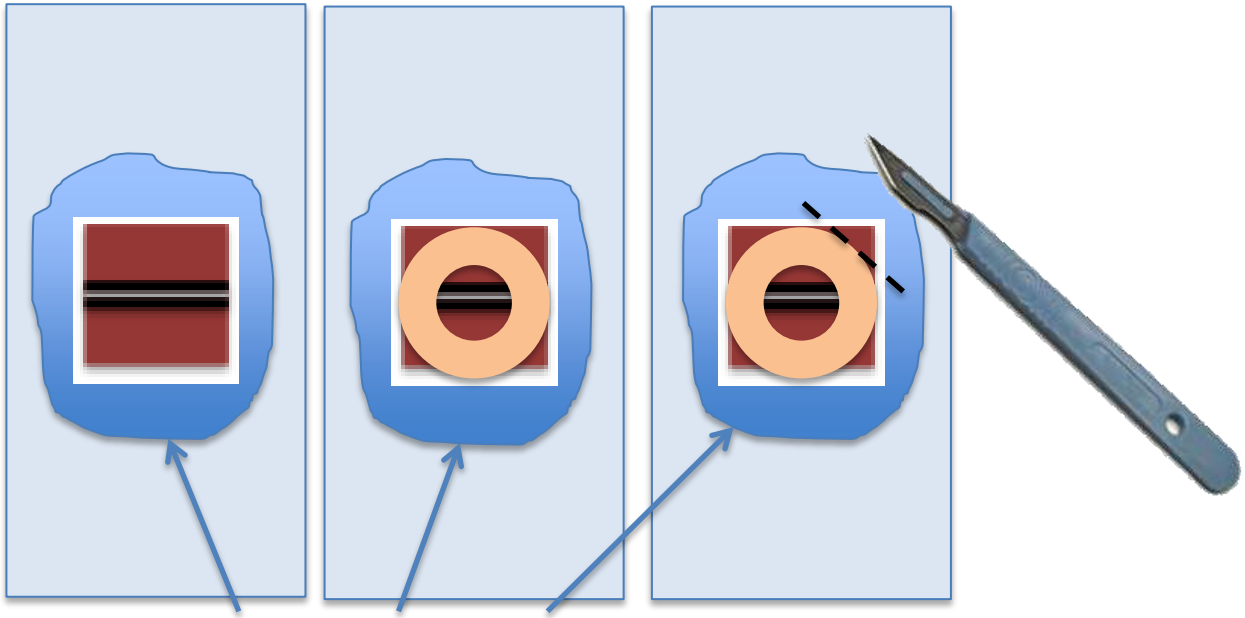


Polishing holder without the movable support in brass

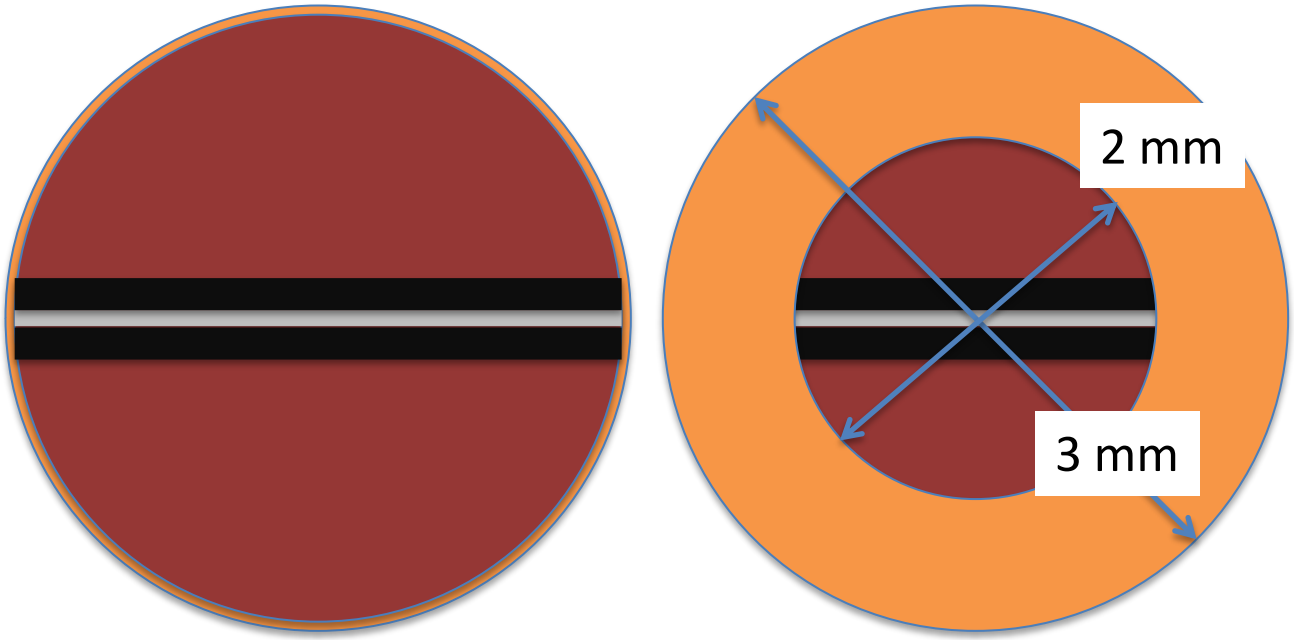
After polishing the first face, the movable support is heated in order to melt the wax. The sample is flipped and glued with wax. A copper grid (used as reference for the thickness) is added beside the sample and glued with wax as well.



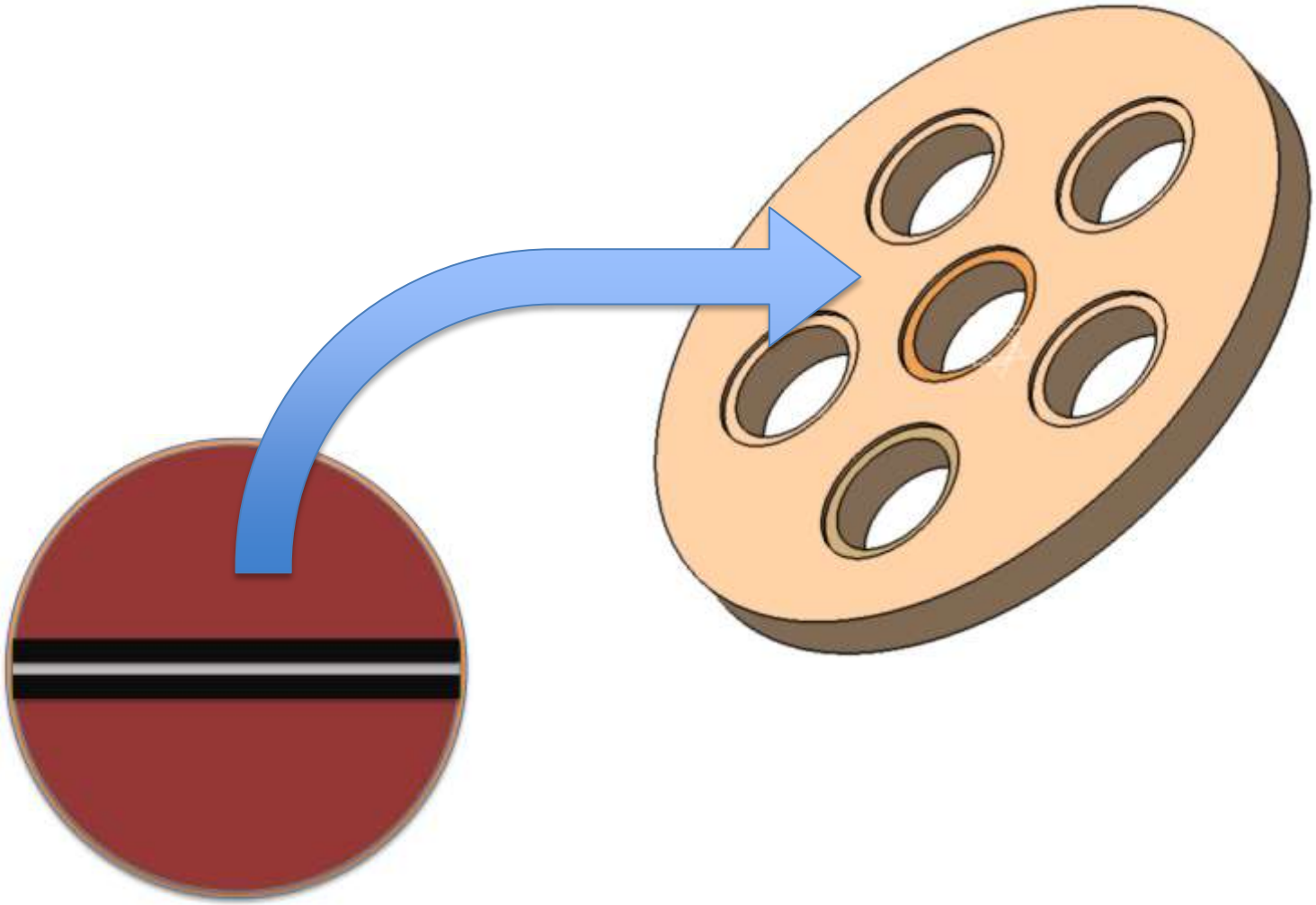
The second face is polished up to the copper grid.



Dental wax (melting temperature 120 °C)



When the required thickness of the sample is obtained (30-50 microns), a new Cu grid is glued on the sample surface. It is necessary to use a glue resistant to temperatures above 120°C and acetone. The dental wax around the sample is not taken off (yet). It is very important to prevent the glue to fix the copper grid on the movable support.
The parts of the samples which exceed the grid are then cut with a scalpel and the set is taken off (heated at 120°C then cleaned with acetone).



The is now is ready and can be mounted on a TXM sample holder adapted for this type of sample