

Micro scanning X-ray diffraction study of Roman Terra Sigillata ceramics

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Terra Sigillata is the most famous fine ware of the Roman period. This class of pottery appeared in the mid-first century BC in Italy. From the Augustan period (27 BC – 14 AD), it has been widely spread within and outside the Italian Peninsula and branches were established in Pisa and in the south of Gaul. Among the southern Gaul workshops, *La Graufesenque* was the most important. During the first century AD, this workshop produced vast quantities of the sigillata pottery, which were sold all over the Roman Empire, mainly in Gaul, Great Britain, Germany and Spain [1].

The success of this pottery was due in great part to the brightness and colour of the gloss. However, up to now few studies were devoted to this topic. Also in order to better understand the origins and elaboration process of these ceramics, a study of the microstructure of the Gaul *sigillata* slip has been carried out by μ -beam X-ray diffraction.

The XRD experiments were carried out at the beam line 7.3.3 of the Advanced Light Source (Berkeley, USA) by successively scanning a white and monochromatic X-ray beam over the sample surface with a submicron spot size [2]. Simultaneously, the main chemical species Fe were located by their X-ray fluorescence emission. The diffraction diagrams were recorded with a 2D CCD detector. The phase, the size range and the orientation distribution of the grains are determined for the Fe host phase and for the other main crystalline constituents.

The different populations of crystallites were also imaged by TEM and their chemical composition estimated by EELS.

The gloss is mainly formed of μm -sized quartz, with smaller hematite and corundum crystallites embedded in a glassy matrix. The matrix does not contain any colouring agent. The colour of hematite crystallites usually varies from black to red, depending both on the Al/Fe ratio and on the crystallite size. In the sigillata, hematite is red, due to the presence of Al and to the submicronic grain size. In the case of corundum, we observe a light yellow colour to these crystals in relation with the presence of iron. Both the hematite and corundum crystal populations are homogeneously dispersed in the matrix and they give, together, the red-orange colour to the sigillata. The diffraction 2D diagrams show that corundum is composed of nanocrystallites. This phase is formed during the firing and its structural and chemical features can be imparted to the elaboration process of the ceramic.

References

[1] - P. Sciau et al., poster at the Material Research Society Symposium OO “*Materials Issues in Art and Archaeology VII*”, Nov. 29 – Dec. 12, 2004, Boston (USA), MRS proceedings 852 (2004).

[2] - N. Tamura, R. S. Celestre, A. A. MacDowell, H. A. Padmore, R. Spolenak, B. C. Valek, N. Meier Chang, A. Manceau, J. R. Patel, *Submicron x-ray diffraction and its applications to problems in materials and environmental science*, Review of Scientific Instruments 73 (3) (2002) 1369-1372.