

## Poster Presentation

MS55.P03

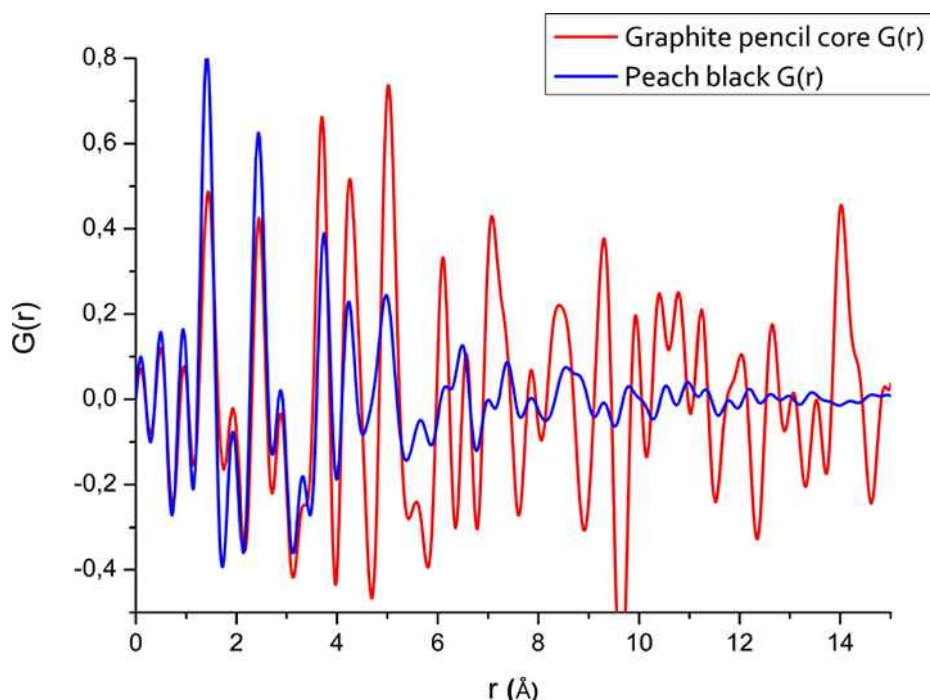
### Analysis of archaeological samples using XRPD-PDF and Raman spectroscopy

S. Cer soy<sup>1,2</sup>, P. Martinetto<sup>1,2</sup>, P. Bordet<sup>2,1</sup>, J. Hodeau<sup>2,1</sup>, P. Walter<sup>3,4</sup>, E. Van Elslande<sup>3,4</sup>

<sup>1</sup>Univ Grenoble Alpes, Institut Néel, Grenoble, France, <sup>2</sup>CNRS, Institut Néel, Grenoble, France, <sup>3</sup>Sorbonne Universités, UPMC Univ Paris 06, UMR 8220, Laboratoire d'archéologie moléculaire et structurale, LAMS, Paris, France, <sup>4</sup>CNRS, UMR 8220, LAMS, Paris, France

Carbon black materials have been frequently used from prehistory as pigments for drawings and paintings and also as dyes, inks and cosmetics. If these material are easy to make by burning organic matter from animal or vegetal origin (e.g. peach black), they form carbonaceous phases, often ill-ordered, that can hardly be characterized [1]. This project is part of studies on archaeological cosmetics, shedding light on ancient manufacturing in physical-chemistry. Six black Roman micro samples found in vessels in Pompeii Houses were studied. To understand the composition of these complex, heterogeneous (mixture of organic/mineral, crystallized/amorphous phases) and precious black powders, a new methodology had to be developed. X-ray powder diffraction tomography enabled to locate the various phases (either crystallized or not) in virtual slices among which ill-ordered materials were analyzed using the Pair Distribution Function (PDF) [2]. X-ray diffraction data were recorded on D2AM beamline at ESRF on the samples and on reference modern carbon black powders purchased from pigments suppliers to help the interpretation of the diffuse signal. High Q data were acquired by scanning the 2D detector and using 25 keV X-rays. This enabled us to obtain well-resolved PDFs to study the amorphous carbonaceous phases contained in our samples (see figure). A comparison between archaeological samples and pure carbon black references was carried out after identification and quantitative analysis of the crystallized phases using Rietveld refinement. The proportion of crystallized phases versus ill-ordered carbonaceous ones was estimated on the PDFs using the maximization of Pearson product-moment correlation coefficients [3]. Results were confronted with those from Raman spectroscopy, known to account for the degree of disorder of non-graphitic carbons. The combination of both methods also provided specific information about the origin (plant, animal or mineral) of carbon black pigments.

[1] M., Alvarez-Murga, P., Bleuet, L., Marques, et al., *Journal of Applied Crystallography*, 2011, 44, 163., [2] T., Egami, S. J. L., Billinge, "Underneath the Bragg Peaks: Structural analysis of complex materials", 2003, Pergamon, Oxford, England., [3] T., Davis, M., Johnson, S.J.L., Billinge, *Crystal Growth & Design*, 2013, 13(10), 4239.



**Keywords:** X-Ray Powder Diffraction Pair Distribution Function (XRPD-PDF), Raman spectroscopy, Cultural Heritage