on metallic artifacts with simple structures unlike those of paintings. Our ongoing investigation is devoted to optimizing GIXRD analytical routines to obtain the best quality diffractograms in complex, small and uneven painting samples [6].

Historical objects comprise a wide variety of composite materials made up of inorganic and organic components. To attain a full material characterization, other analytical techniques are essential in addition to XRD methods, such as microscopic techniques that provide key information on artwork microtexture, structure and composition via the use of mineral maps obtained from SEM-EDX analyses. Also application of Raman microscopy is crucial since it non-destructively identifies amorphous, poorly ordered and crystalline phases of small size (1µm). We have demonstrated the benefits of combining XRD methods together with Raman microscopy and SEM-EDX analyses in painting samples and patinas [5], [7].

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Keywords: XRD methods, cultural heritage, crystallography

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Synthetic or manufactured ancient pigments studied by means of synchrotron radiation–based methods

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Although recent use of laboratory X-ray powder microdiffractometers showed promising results on very small or multi-layer samples [1], synchrotron facilities produce X-ray beams (both tunable in energy and size) which allows a lot of combinations, well adapted to the heterogeneity of ancient materials. X-ray powder diffraction is thus combined with fluorescence and absorption to identify pigments in paint layers [2] and to understand manufacturing or alteration processes [3]. We shall illustrate through three examples the different possibilities offered by synchrotron radiation–based methods to study synthetic or manufactured ancient pigments. These pigments are often ill-ordered (because of their synthesis, their processing or their ageing) and remain difficult to be studied by using traditional diffraction methods. We use modern reproductions as references in order to consolidate data treatment and to interpret the results obtained on Cultural Heritage materials. The first example concerns Maya Blue, an artificial pigment manufactured in pre-Columbian Mesoamerica [4], one of the best examples of organic-inorganic hybrid materials. Its durability is due to a unique association after heating together indigo and a particular clay. Combining thermogravimetric analysis and synchrotron X-ray powder diffraction data with molecular modelling, we are able to propose a new explanation of the chemical stability and the durability of Maya Blue [5]. The second example will deal with Prussian Blue. This artificial pigment, accidentally discovered in Berlin in 1704 and very popular in the 18th and 19th centuries, shows a tendency to fade under light [6]. The degradation process seems related to the crystalline quality of the powders, depending of the method of preparation. In the case of ill-ordered powders, we have recorded the total scattering signal at 100 keV and we are currently carrying out Pair Distribution Function (PDF) analysis in order to provide suitable data for structural investigations of Prussian blue. The third pigment which will be discussed, galena (PbS), was extensively studied several years ago as main ingredient of ancient Egyptian cosmetics [7]. Actually, thermal treatments are commonly used nowadays to prepare eye make-up of different colors based on galena in northern Africa, but heating processing of galena in Ancient Egypt remains an opened question. We have recently performed Laue micro diffraction experiments on both artificially heated galena crystals and archaeological powders, in order to compare the thickness of the oxidized layers and the formed phases.

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Quantitative characterization of japanese ancient swords through time of flight neutron diffraction and energy resolved neutron imaging

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A total of nine broken Japanese swords pertaining to a period ranging from 14th until 19th century have been analysed through neutron diffraction and neutron imaging techniques [1]. The samples are the lower part of ancient swords broken approximatively at 50-60 mm from the beginning of the blade. They are signed and the authorship and attribution can be accurately identified. The samples have been made available by the Stibbert Museum staff as test samples for non destructive characterization through innovative methods.

Neutron diffraction has been applied on all the selected samples by using the INES diffractometer at the ISIS pulsed neutron source in UK [2]. The measurements have been performed on the average gauge volume both in the tang and in the blade in order to determine the quantitative distribution of the metal and non metal phases. The cementite to ferrite ratio has been used in order to quantify the carbon content. The comparative analysis of the phase distribution among the samples permitted to identify peculiar characteristics related to the forging traditions and periods of the Japanese history. I.e. the carbon content, the fayalite amount, the presence of wuestite and troilite has been comparatively checked. On few selected samples