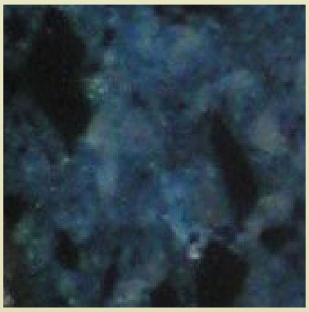
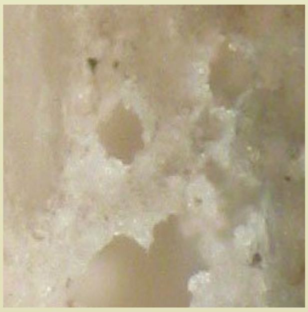
# THE VISIBLE IMAGE IS NOT ALWAYS CORRECT

## *The differentiation of layers by optical microscopy in samples' cross sections*

*by Carolina Barata, António João Cruz and Marta Ferro*

*The stratigraphic characterization of polychrome surfaces in works of art is frequently done through the observation of cross-section samples by optical microscopy (OM). Three examples that show some limits to this method are presented here. In samples where at first only one layer was visible, several strata were detected through electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS). These and other examples ought to be taken into consideration by conservators, who should be aware of similar possibilities in other cases.*

At least since the middle of the 19th century, minute samples collected from works of art were mounted in resins, cut, polished and observed using optical microscopes [1]. However, only during the first half of the 20th century has this method been developed and employed more frequently [2,3]. Through the optical microscopy (OM) of cross sections from works such as paintings and polychrome sculptures, usually under a magnification of 100x to 300x, it is possible to determine the number and sequence of layers and to characterize each one of them with respect to matrix heterogeneity, particle size, color, shape and transparency, among other aspects.

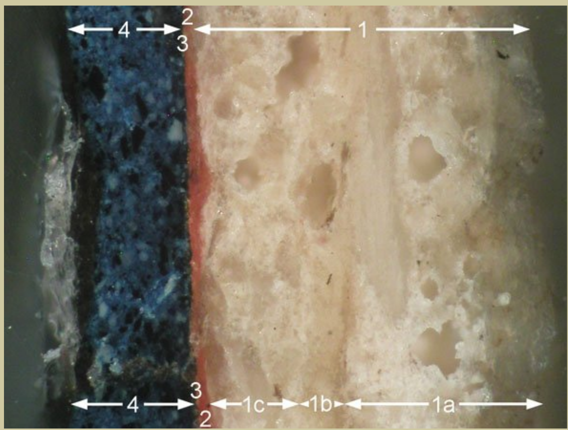
The information obtained is useful, for instance, for the characterization of artist techniques, the distinction between original materials and overpaintings, and the diagnosis of the conservation condition. As it is known by any conservator, many examples are found in countless publications concerning the materials of paintings and sculptures.

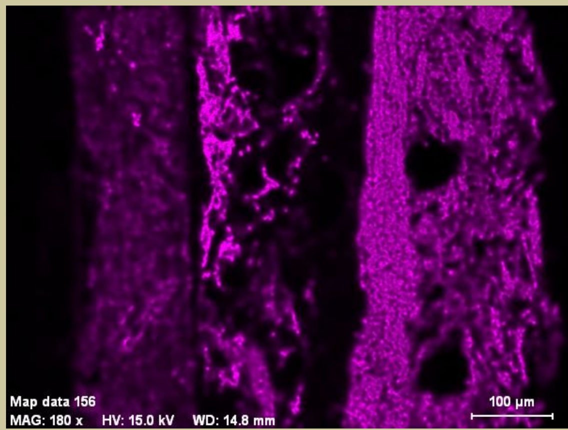
Today, with the same goal in mind, the embedded cross sections are also analyzed through some advanced analytical methods, namely scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR) and mass spectrometry [3]. However, despite the extra information provided by these methods, particularly in what concerns the chemical composition, the examination of cross sections by conservators is usually limited to OM. The situation can be explained by a number of reasons such as the relative low cost of the equipment that is required, and to the fact that the information obtained in many cases is both easily interpreted and sufficient.

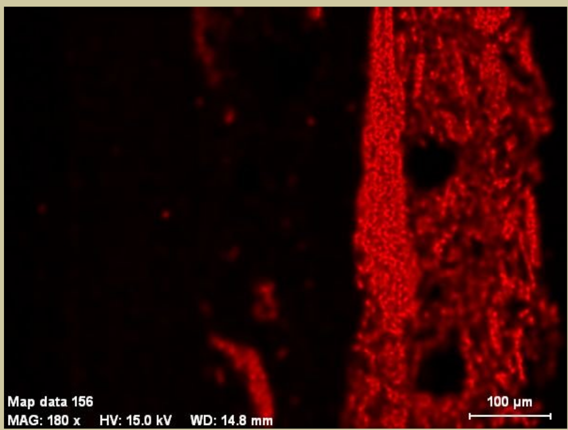
In some situations, however, the images acquired by OM can lead to false conclusions. These situations are probably not that frequent, but it is important for conservators to be aware of this possibility, especially when false conclusions may have significant consequences.

The observation of only one stratum by OM when several strata are in fact present, is probably the most common situation.
In the context of a Masters dissertation that aimed to contribute to the characterization of the Portuguese polychrome wooden sculpture from the Baroque period [4], the observation of cross sections, both by OM and SEM with energy dispersive X-ray spectroscopy (SEM-EDS), led to the detection of some interesting cases. For OM, an Olympus binocular microscope, model BX41, equipped with an Olympus Digital C-4040 Zoom camera with infinity corrected optical system, was used. The analysis by SEM-EDS was done in Hitachi SU-70 UHR Schottky FE-SEM with samples coated with carbon. In some samples, according to OM, the ground layer was composed of one stratum. However, through SEM-EDS, particularly through the maps of elements obtained, it was determined that the ground was actually composed of several strata, as showed by the chemical differences or the limits that were detected between them. In that study, the conclusions about the real number of strata were important, since one of the aspects under research was the relation between the information found in art treatises and working contracts, according to which the ground should be composed of several strata, and the workshops' practice.

One example is the cross section taken from the blue vestment of a sculpture representing Saint Andrew, dating from the second half of the 18th century, and part of the collection of the museum of Santa Maria de Lamas (Figure 1). Although a certain heterogeneity in the ground layer was visible, it was not possible to subdivide it. However, as revealed by SEM-EDS it was composed of a first stratum, enriched in calcium and lead



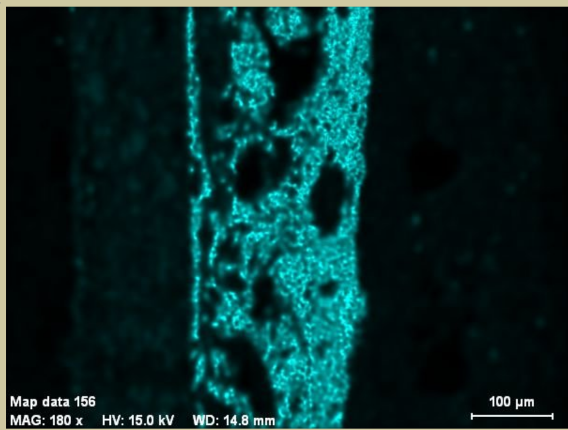
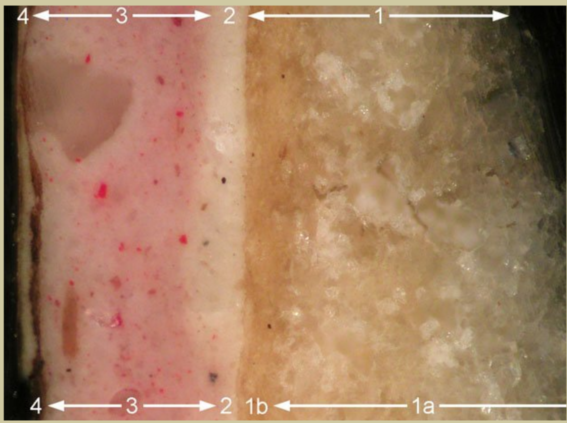



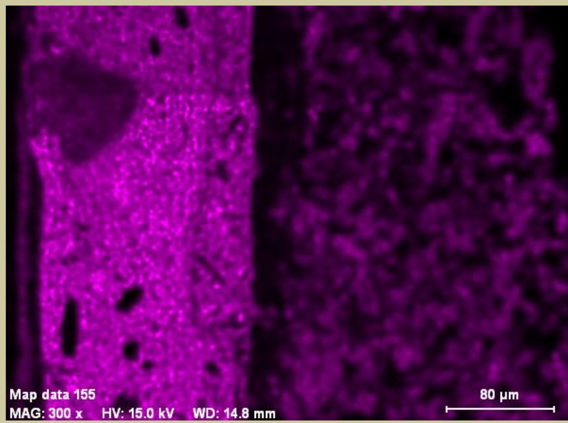

*Figure 1. OM - cross section taken from the blue vestment of Saint Andrew (100x).*
*At the top, layers identified by OM: 1 – ground; 2 – bole; 3 – gold leaf; 4 – paint layer. At the bottom, 1a, 1b and 1c correspond to three different strata with different composition identified by SEM-EDS in the ground layer.*

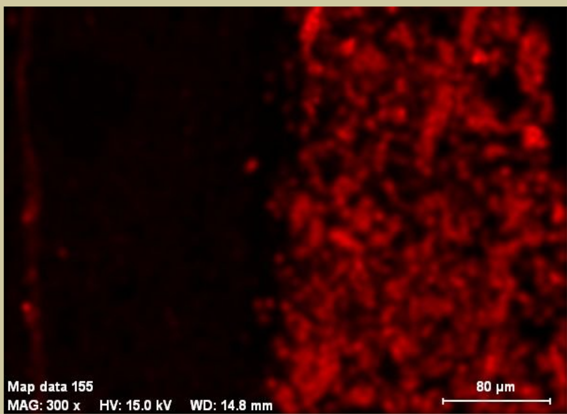
*Figure 2. SEM-EDS – map of Ca of the cross section observed in figure 1.*
*Figure 3. SEM-EDS – map of Pb of the cross section observed in figure 1.*
*Figure 4. SEM-EDS – map of Al of the cross section observed in figure 1.*

(Figures 2-3) (probably a mixture of gypsum and white lead), a second stratum, enriched in aluminum (Figure 4) and silicon (probably a mixture of clay minerals and other silicon compounds), and a third stratum, also enriched in aluminum and silicon but with a high content of lead (probably a mixture of the same compounds present in the second stratum with white lead).

Another example is provided by the sample taken from the flesh area of a sculpture representing Saint Francis Xavier (Figure 5), which dates from the last quarter of the 17$^{th}$ century and belongs to the same collection. The brownish color on the top of the ground layer was first interpreted as being a result of the impregnation of that layer by glue. This interpretation was also supported by the fact that no significant differences in the particles' size and shape were detected inside the ground. The maps of elements obtained by SEM-EDS, however, showed that this was not correct. Instead, they revealed that the ground is composed of a stratum enriched in calcium and lead (probably white lead mixed with a calcium filler) at the base (Figures 6-7), followed by a stratum mainly composed of clay minerals, revealed by its high contents of aluminum (Figure 8) and silicon.



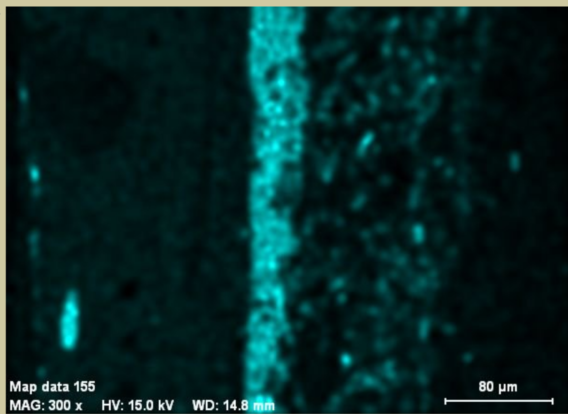



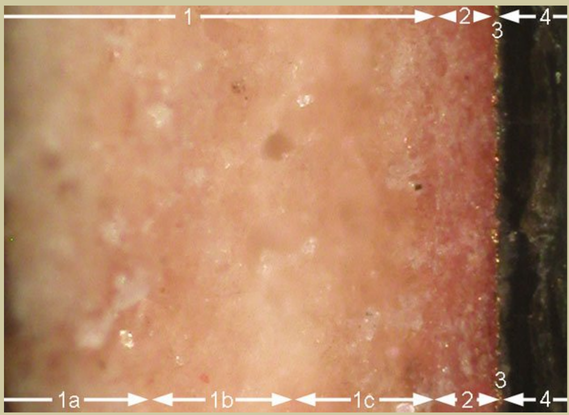

*Figure 5. OM - cross section taken from the flesh tone of Saint Francis Xavier (100x). At the top, layers identified by OM: 1 – ground; 2 – lead white; 3 – paint layer; 4 - overpainting. At the bottom, two different strata identified by SEM-EDS in the ground layer: 1a – mixture of calcium filler and white lead; 1b – clay minerals, first interpreted as animal glue.*

*Figure 6. SEM-EDS – map of Ca of the cross section observed in figure 5 (300x).*
*Figure 7. SEM-EDS – map of Pb of the cross section observed in figure 5 (300x).*
*Figure 8. SEM-EDS – map of Al of the cross section observed in figure 5 (300x).*

The last example comes from a sculpture from the second half of the 18th century representing Saint Dominic, also belonging to the collection of the museum of Santa Maria de Lamas [5]. In the sample taken from the black vestment, a reddish layer of bole seems to be present between the layer of gold leaf and the ground, although the separation between the bole and the ground was not clear (Figure 9). The ground layer seems to be uniform, but the combined map of several elements, obtained by SEM-EDS, suggests that it may be composed of at least three strata, as some lines are observed that probably corresponds to theirs limits (Figure 10). Additionally, the extension of the bole layer in this map is clearly delimited.

We must point out that, in principle, cases like these can also be detected by other methods. Ultraviolet microscopy, which involves the observation of a sample exposed to ultraviolet radiation through an optical microscope, is such an example [3, 6]. Its usefulness is particularly expected when a highly fluorescent material shows different concentrations in different layers. Stain tests directly applied on cross sections are also an accessible alternative to SEM-EDS [6]. In this case different materials should react in different ways to a specific reagent.

As a conclusion, the main point that we would like to emphasize is that one should bear in mind that



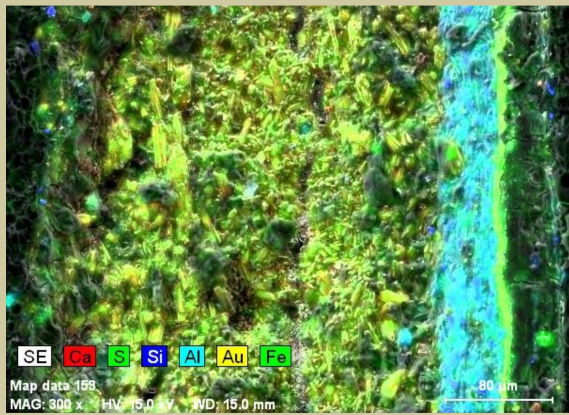




*Figure 9. OM - cross section taken from the black vestment of Saint Dominic (100x). At the top, layers identified by OM: 1 – ground; 2 – bole; 3 – gold leaf; 4 – paint layer. At the bottom, 1a, 1b and 1c correspond to the three different strata identified by SEM-EDS in the ground layer.*
*Figure 10. SEM-EDS - map of elements of the cross section observed in figure 9.*

when only one layer is detected by OM, in some cases several other layers might be present and be detectable by other methods. Although several examples of this have already been published, we none the less think that it is important to draw attention to this fact in a clear and explicit way.

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## CAROLINA BARATA

*Contact: cbarata@porto.ucp.pt*

Carolina Barata, sculpture conservator, owns a bachelor's degree in Conservation and Restoration and a master's degree in Chemistry Applied to Cultural Heritage. Presently she is a lecturer at the School of Arts of the Portuguese Catholic University.

## ANTÓNIO JOÃO CRUZ

*Contact: ajcruz@netvisao.pt*

António João Cruz, conservation scientist, received his Ph.D. in Analytical Chemistry from the Faculty of Sciences of the University of Lisbon. At the moment he teaches at the Polytechnic Institute of Tomar.

## MARTA FERRO

*Contact: marta.ferro@ua.pt*

Marta Ferro, specialist in electron microscopy, is currently working at the University of Aveiro and at the Centre for Research in Ceramics and Composite Materials (CICECO).