Cross-section characterization by SAED/HRTEM of ground layers of "The equestrian portrait of the Duke of Lerma" by Rubens

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The intention of this work is to undertake a comprehensive analysis of part of the pictorial work of P.P. Rubens belonging to the collection of the Prado National Museum. This study should help to clear up some doubts as to the origin of several of the painter's works. The ultimate objective is to complete a nanostructural characterization using high-resolution transmission electron microscopy (HRTEM). The painting currently under analysis is entitled *"The equestrian portrait of the Duke of Lerma"* (Fig. 1a); it was painted at Valladolid during Rubens' second sojourn in Spain. As in many of his paintings, the artist joined two pieces of canvas, each prepared in a different way. On the basis of a previous examination by LM and SEM [1], the object here was to conduct a point-to-point examination of the materials comprising the ground layers in the two pieces and to identify them by means of electron diffraction (SAED). This characterization will enable us to determine the nature of the crystalline species in the layers and undertake a comparative study of the materials used. In the present case, the research team followed the same method as was used for analysis of other works by Rubens.

For HRTEM examination, microsamples were prepared, and then ultra-thin sections (50-100 nm) were cut in such a way as to retain all the particles in the original layers (Fig. 1b, 1c). This is a laborious process but has been found to be highly effective for these studies [2]. By means of EDS microanalyses using point-to-point HRTEM, we have been able to locate the elements contained in each particle; this information is being supplemented by identification of the crystalline species by SAED. These results complete the characterization, confirming the data gathered previously by SEM.

In this way, we have confirmed the presence (in the main panel) of abundant calcite, along with an umber earth pigment and various types of minerals, all typical of earths of this kind (feldspars and micas). We have also confirmed a high concentration of quartz crystals and of amorphously-structured lead. In the preparation added at a later date we have identified a very pure iron oxide (hematite) and large quantities of white lead pigment. A complete characterization of these particles was possible thanks to the data from electron diffraction analysis of their crystalline structures. Appropriate indices of the resulting diffractions were assigned on the basis of parameters and reticular distances taken from the Joint Committee for Powder Diffraction Studies (JCPDS) specifications. Following are representative examples of the two preparations, one calcite (main panel) (Fig. 2a) and one hematite (upper strip) (Fig. 2b). Table I shows the reticular parameters. Finally, the electron diffraction data and assignment of indices for the crystals are shown in Table Ia and Ib.

Thanks to the high precision of this technique, it has been possible to identify each of the pigments and filler that Rubens used in the painting on a point-to-point basis and clearly establish the differences between the two pieces. This preliminary information will allow us to determine where several works by Rubens were painted [3].

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1. Also presented at this Conference.
2. M. San Andrés et al., Journal of Microscopy **188**, 42-50 (1997).
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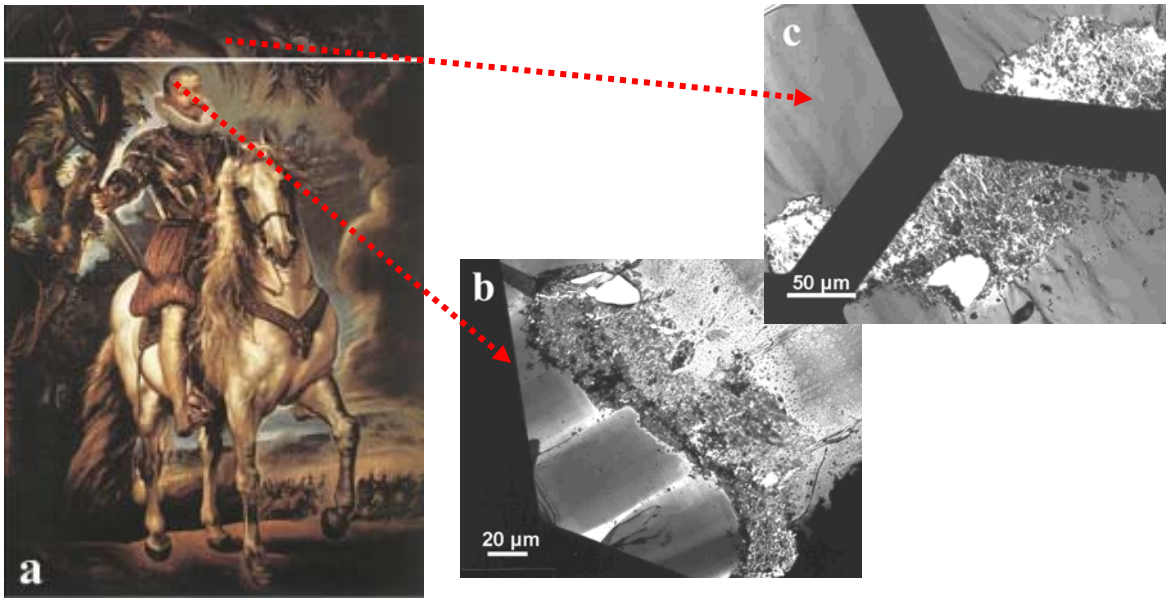

Figure 1. a) "The Duke of Lerma on Horseback" (1603). Oil on canvas. The line indicates the size of the two pieces of canvas making up the painting. b) and c) General stratigraphic view of an ultrathin section from the sample (TEM): main panel (b) and added strip (c).

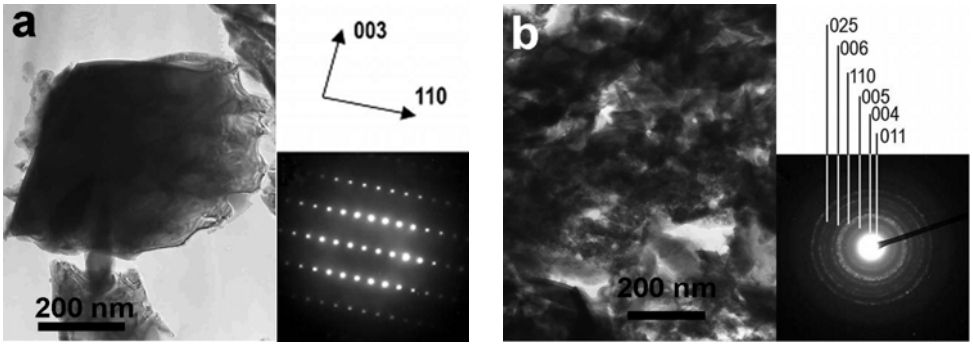

Figure 2. TEM. Image and electron diffraction patterns: a) Calcite (main panel) Zone axis [1 -1 0]. b) Hematite polycrystal (added strip).

a	hkl	$R_{(mm)}$	d_{sample}	d_{JCPDS}	angle (θ)	θ_{sample}	θ_{JCPDS}
CALCITE	110	8.00	2.50	2.49	110∧003	90	90
	003	3.50	5.71	5.68	110∧113	24	23.68
	113	8.75	2.29	2.28	003∧113	66	66.32

b	d_{sample}	d_{JCPDS}	hkl
HEMATITE	4.10	4.15	011
	3.46	3.43	004
	2.76	2.75	005
	2.50	2.51	110
	2.29	2.29	006
	1.70	1.71	025

Table I. Electron diffraction data. Assignment of indices. a) Calcium carbonate. b) Hematite.

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