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Surface preparation of cross sections from traditional and modern paint using the Argon ion milling polishing CP system.

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Paint cross sections (XS) are commonly examined by microscopy after embedding and mechanical polishing or microtoming of minute paint samples. Disadvantages of this methodology are imbibing of resin material, dissolution of varnish, mechanical abrasion of pigment particles, scouring of weaker parts especially organic rich layers, loss of particles in the case of thin sections and smearing of paint substances over the surface. Some of these problems can be avoided but others become painfully evident when the surface of a cross section is examined with surface sensitive chemical microscopes such as imaging FTIR and SIMS. The AMOLF painting research group has tried to solve this problem by embedding in the light curing resin Technovit 2000L, paint XS handling in a sample holder and dry polishing with Micromesh polishing cloths [1]. As a result, the reflection FTIR spectra have less noise and microscopy FTIR image maps are more homogenous. The mass spectral resolution obtained with imaging SIMS is greatly improved due to a smoother and flatter surface, which leads to more defined SIMS image maps. Despite these improvements, pigments like lead white in 17th century paint cross sections still show scratches visible on the larger lumpy particles and some rounding off of the finer pigment particles (Fig 1). A softer acrylic paint XS (Fig 2) shows the scouring away of the organic binding medium (note the harder particles of Titaniumoxide protruding above the surface).

We are now exploring Argon ion polishing using the JEOL Cross section Polisher (CP®). XS of traditional 19th C lead white oil paint (Fig 3) as well a multistack buildup of modern acrylic paints (Fig 4) have been successfully ion milled. Samples were glue mounted on silicon wafers for "polishing" in vacuo using the argon ion beam and examined without coating using a JEOL 7000-F SEM. Earlier studies on avian eggshells have demonstrated that the ion beam cuts evenly through organic and inorganic components on a nanoscopic level [2]. The paint samples were ion milled twice thus preparing a smooth surface. The paint surfaces obtained demonstrate crystal particles with a very sharp edges and much more easily identifiable well defined layer margins. These results open new vistas for microstructural analysis of paints, improved mapping of elementary and phase composition. The layer buildup of a brush stroke from black and white paint boundary in Fig 4 taken from a mid 19th panel painting of Bosboom is remarkably complex. The lead white in layer C resembles the traditional Dutch stack process pigment with larger aggregated lumps in a finer matrix, while lower layers (D and E) are more reminiscent of the lower quality paint made with lead white with thin flat rectangular crystals from an industrial precipitation production process. Layer D also shows a microstructural orientation along the longitudinal of the flat lead white crystals.

References

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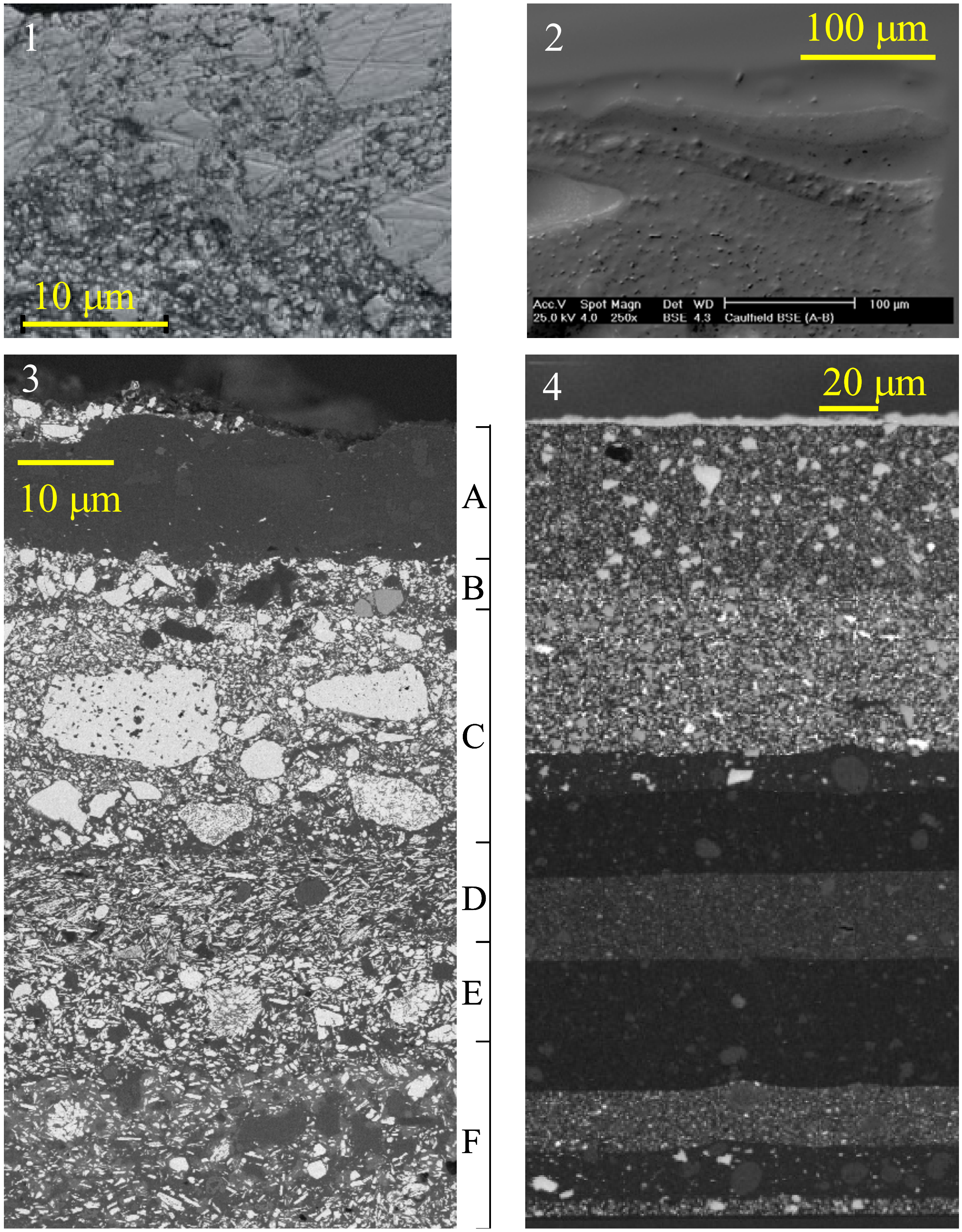

Figure 1-4. (1) Mechanically ultra-polished section of an embedded paint cross section of 17th C lead white paint taken from Rembrandt's *Anatomy lesson of Dr. Nicolaes Tulp* (MH 146), (2) CP argon ion polished cross section of paint from a 19th C panel by Bosboom, (3) Mechanically ultra-polished section of an embedded paint cross section from a painting by P.Caulfield (Tate nr T07112), (4) CP argon ion polished multilayer stack of nine acrylic paints on Melinex.

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