SEM and microanalysis in the study of historical technology, materials and conservation (SEM 2010)

Thursday 9 – Friday 10 September 2010
BP Lecture Theatre, British Museum

FIRST CIRCULAR: CALL FOR PAPERS

The deadline for submission of abstract is 19 March 2010

Title of paper:

Studying the cracking system in 17-18th century Portuguese Azulejos with SEM

The presentation is for an oral ☐ or poster ☒ presentation

Summary of the paper (in no more than 500 words):

Portuguese glazed ceramic tiles - azulejos – are a distinctive mark of Portuguese culture. The conservation of the Baroque churches, palaces and even gardens calls for the conservation of the tile panels that decorate them. However they suffer from several pathologies that result in the loss of the glaze and, with it, of the pictorial layer.
A review of azulejo pathologies throughout Portugal revealed that the most worrying forms of decay are related to defects that typically result from the manufacturing process and thus lie in wait in seemingly sound tiles until the proper conditions lead to the setting of decay. Defects include crazing (or craquelé), which, microscopically, resolves into two different morphologies of crack propagation, and shivering, which results in glaze delamination leading to an empty space between glaze and biscuit, prone to both soluble salt crystallization and to biological growth. Salt crystallization calls for an evaporation zone inside the tile and thus is linked with defects that provide a path to the exterior. In a crazed tile these paths are obvious. A shivered tile can decay from the borders, where evaporation is easy, but for it to decay from the centre there must also be a second defect - a pore bypassing the glaze and through which evaporation may be straightforward. Further observations lead to the conclusion that when challenged by aggressive conditions, azulejos without defects may remain macroscopically impervious, while contiguous tiles with even minor defects suffer extensive glaze loss. Hence the importance of a clear understanding of the cracking patterns and other defects, particularly when these offer an evaporative path.
Cracks and other defects may be studied by optical microscopy but very thin cracking is not always apparent. A further problem results from the fact that the biscuits of early Portuguese tiles are usually of a very light colour, so as to avoid impairing the whiteness of the majolica. This means that the exact boundary between glaze and biscuit may be difficult to pinpoint by optical microscopy. SEM imaging can complement optical microscopy to map and help to understand the cracking paths through the glaze and other defects.
Fragile cracked specimens are vacuum impregnated with a resin and then sectioned and polished for the SEM. Backscattered imaging is preferred because the lead-rich glaze is enhanced, ensuring clear boundaries. The samples are coated with Au/Pd (80/20) so that carbon is only present in the filling resin. The resin-filled voids show as very dark areas in

the image and the use of EDS (X-ray mapping of C) confirms that these are indeed filled with organic resin and are thus cracks accessible from the surface of the glaze or from the side of the glaze-biscuit interface.

Our communication contains the SEM/EDS results of a study of cracking paths through the glaze and into the biscuit, and delamination patterns in the glaze-biscuit interface. The purpose is a better understanding of the fissural systems with a view to develop solutions that may prevent the onset of decay in fragile glazed tiles.

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