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Application Note

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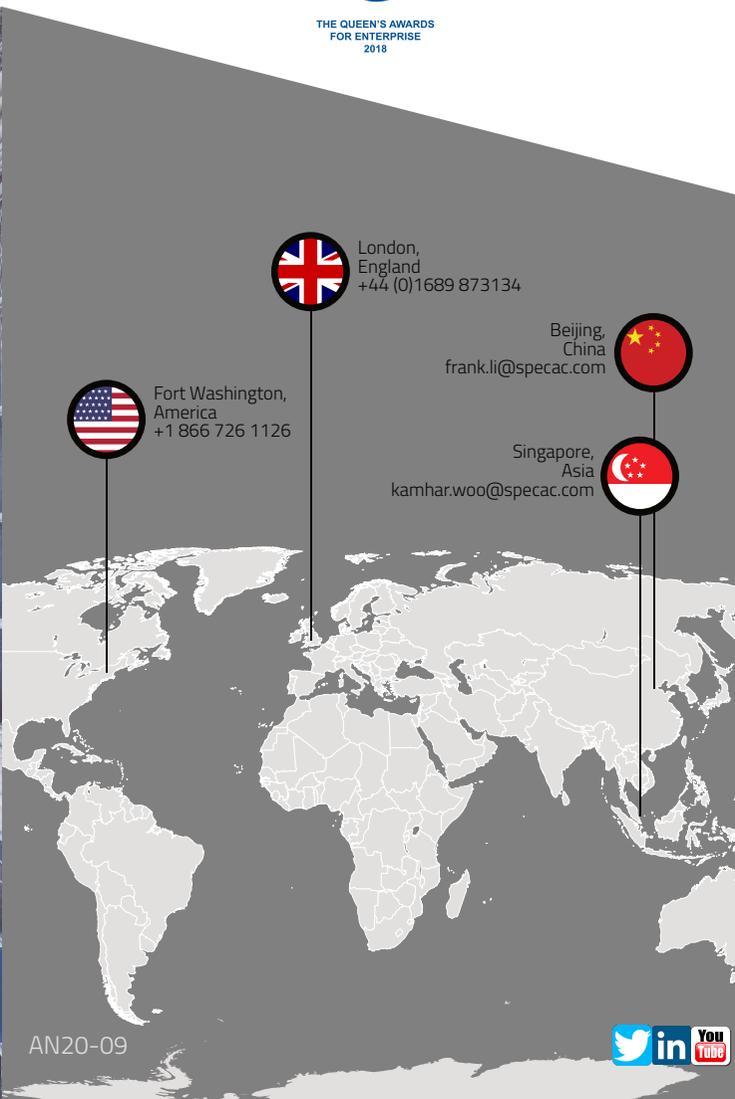
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SurveyIR and Cultural Heritage. Non-invasive insight of gilded paint samples using the Infrared Microscopy Accessory



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Inside: learn how the SurveyIR can be used to investigate gilding technique on a paint stratigraphy from the ceiling of a church

Introduction

Gildings are commonly found highlighting details of ornamental or iconographic value such as halos. They are documented on a variety of artefacts including statues, stuccos, wall paintings as well as paintings on canvas, wood panels, and illuminated manuscripts [1]. Gildings consist of micrometer thick leaves of different metals (e.g., gold, silver, metal alloys) applied to the substrate by a thin adhesive ground layer. Depending on the gilding technique, the composition of the ground may vary from mainly inorganic to mainly organic, and may be coloured with pigments (as e.g., lead pigments, ochres) [2].

Over time, such materials undergo unavoidable decay due to oxidation of metal leaves and loss of adhesive properties of the ground, hence conservation treatments can be applied to restore the mechanical-optical properties of the original materials, or to protect the gilding from further interaction with the environment. This note will investigate the gilding technique.

The SurveyIR™ is an in-compartment FTIR microspectroscopy accessory designed for rapid and non-destructive analysis of materials at high spatial resolution. It is ideal for cultural heritage studies requiring spectral acquisition of microscopic features and layers within a sample.

- ▶ Simple, repeatable measurement procedure
- ▶ Large depth of field and field of view
- ▶ Transmission, Reflection and ATR analysis modes
- ▶ No separate detector or detector cooling required

Author Acknowledgement

The present work was authored by Elena Possenti¹, Cai Li Song², and Sergei Kazarian² and adapted as an application note by Specac Ltd.

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When defining the composition of the ground and of possible materials present on the metal leaves, one of the most critical issues is the sample itself. In cultural heritage, a common limitation is the need to characterize the materials with minimum possible alteration of the precious and unique samples. Thus, an FTIR investigation with a conventional sampling technique (analysis in transmission or attenuated total reflection (ATR) on micro-particles sampled with a needle from the fragment) is not feasible, as brittle samples may break during the procedure. At the same time the typical preparation of polished cross section is discouraged in some cases, as the polymers used for embedding show strong infrared signals that interfere in the identification of the analytes in the FTIR spectra.

Experimental

The composition of the ground and substances applied over the gold leaves is characterized without any contact by micro-FTIR spectra collected in reflectance mode. Thanks to the optical and infrared microscope of the SurveyIR, it was possible to select a region of interest (ROI) and, based on the surface morphology, set the aperture size to suit. This allowed the investigation of micro-metric layers with a spatially resolved outcome and a completely non-destructive approach. The diameter of the ROI was 2000 μm . The two white circles in Fig 1. show the investigated ROIs. The SurveyIR was inserted in an instrument mounting a DTGS detector. The measurements were collected at 8 cm^{-1} spectral resolution and co-adding 64 scans.

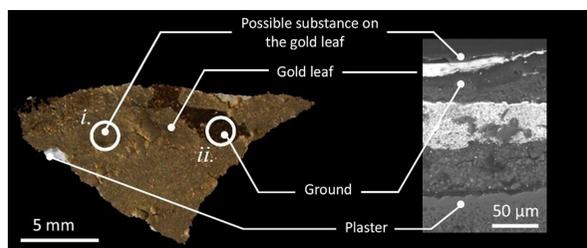


Figure 1: Optical (left) and SEM (right) showing gold leaves on the top and a lacuna revealing the underlying brownish ground. i and ii are the ROI of this study.

Results and Discussion

Starting from the composition of the ground layer (pattern i., Fig.2), the strong band at 1738 cm^{-1} (C=O), as well as the bands at 1456, 1416 cm^{-1} (C-H) and 1166 cm^{-1} (C-O) unambiguously identify a lipid, most likely a linseed oil. The two bands at 3188 and 612 cm^{-1} can be attributed to

the O-H and Fe-O of the pigment goethite ($\text{FeO}(\text{OH})$), added to the oil to give a warm yellowish colour to the final gilding. The combination of a lipid medium mixed with pigments allows us to define this ground as part of the typical missione technique. Nitrocellulose is identified in the FTIR spectrum by the marker absorption at 1280 cm^{-1} (NO_2), and by the other characteristic bands at 1660 cm^{-1} (NO_2), 1067 cm^{-1} (COC), 846 cm^{-1} (N-O). It is not possible to determine if nitrocellulose is used in mixture with the oil or it belongs to the inner layers of the stratigraphy. In any case, the use of nitrocellulose identifies this gilding as a modern one.

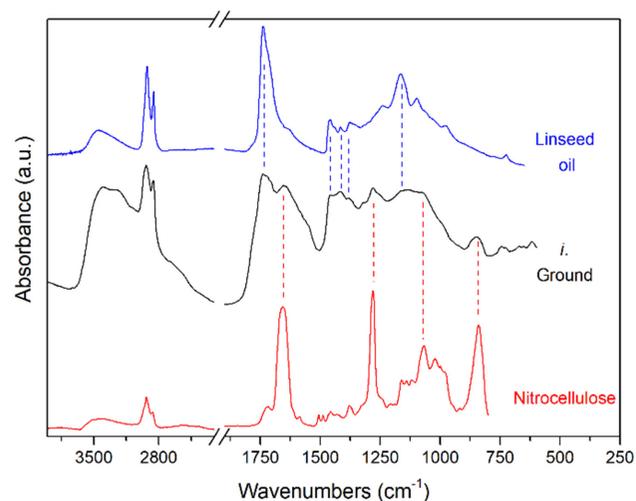


Figure 2: FTIR spectrum of the ground (i.) in comparison with the FTIR reference patterns of linseed oil and nitrocellulose.

The materials found on the gold leaves, calcite, gypsum, silicates and whewellite are detected in the corresponding FTIR spectrum (ii. of Fig. 3). It is conceivable that these phases (especially calcite, gypsum, and silicates) are more likely due to the formation of deposit and not intentionally applied on gold leaves. However, the past application of a finishing treatment cannot be completely excluded, as suggested by the presence of two weak peaks at 2920 and 2850 cm^{-1} (C-H) ascribable to traces of an organic substance (possibly wax-paraffin) and by whewellite, a calcium oxalate commonly detected in correspondence of decayed natural organic substances (e.g. Proteins).

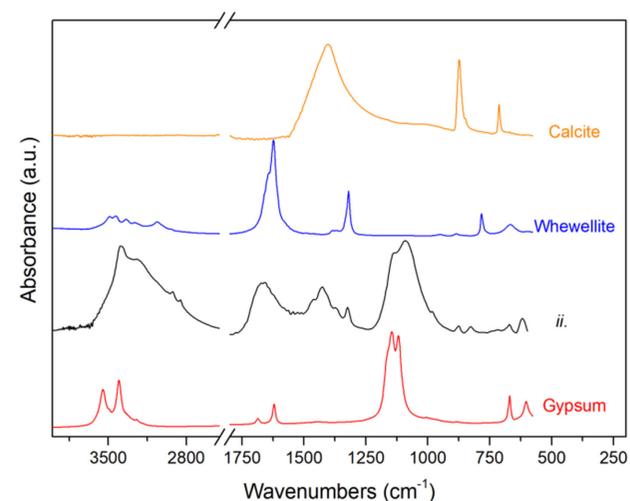


Figure 3: FTIR spectrum of the materials on the gold leaves (ii.) in comparison with the FTIR spectral reference patterns of calcite, whewellite and gypsum.

Conclusions

The SurveyIR was used to collect spectral information in reflection of a wide range of organic and inorganic materials in a completely non-destructive and non-contact mode. This allowed us to measure fragile samples of gildings from cultural heritage and to preserve the sample for other techniques to be used in conjunction. Furthermore, by mounting the SurveyIR in instruments with DTGS detectors, the ROI investigated can be selected without resorting to IR microscopes having the typical low-wavenumber spectral region cut-off of MCT and FPA detectors. With this instrumental setup it is possible to explore the full Mid-IR range, which can be particularly important in the investigation of cultural heritage materials because many pigments, fillers, binders, and decay products have their marker bands in this fingerprint region.

References

[1] Emeline Pouyet. New methods for the preparation and analyses of paint samples from Cultural Heritage artifacts with combined hyperspectral techniques. Materials Science [cond-mat.mtrl-sci].

[2] Elena Possenti, Chiara Colombo, Marco Realini, Cai Li Song, Sergei G. Kazarian, Insight into the effects of moisture and layer build-up on the formation of lead soaps using micro-ATR-FTIR spectroscopic imaging of complex painted stratigraphies, Analytical Bioanalytical Chemistry (2020) <https://doi.org/10.1007/s00216-020-03016-6>