MS43 Crystallography for cultural heritage materials

MS43-05

Scanning electron diffraction microstructural studies to differentiate between ancient black glazed pottery and its contemporary replica

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Abstract

In the present work, Scanning Electron Diffraction in Transmission Electron Micrsocope (TEM-ASTAR) (1,2) was applied to characterize phases present in the black glazed surfaces of Hellenistic pottery down to nm scale resolution and to investigate the potential to differentiate between archaeological pottery with contemporary replicas (3). Two samples were analyzed: one archaeological sample (AL49) originating from Aliartos, Boeotia, Greece dated to the Hellenistic period; and one authentic replica (TH3) produced by Thetis Ltd, using the same raw materials and manufacturing processes as the original pottery. Additionally, Thetis products undergo a process of artificial weathering, to ensure the maximum similarity with the archaeological samples. Both samples contain a high-quality glaze, as observed by their black and shiny surface. Previous crystallographic analyses of the ceramic body and the glaze have suggested the presence of various phases. Most commonly, quartz, feldspar, hercynite, maghemite and hematite are identified on the body and hematite, hercynite, magnetite, magnetite, titanomagnetite on the glaze (4); however EDS analysis has revealed similar chemical composition for both samples (original & its replica) with systematic differentiations on only EDS glaze profiling studies (3). In order to reveal possible differences between two samples, thin lamellas (from AL49 and TH3) were prepared by FIB technique and studied in a Jeol 2100F TEM equipped with ASTAR (2). Scanning Precession (precession angle 1.2 deg) ED data was collected using ASTAR from an area of 300x300 pixels with step size 2 nm. The examination of several areas shows that the two samples show significant difference in magnetite Fe_3O_4 content (46% in TH3 vs 70% in AL49, 20% amorphous area in TH3 vs < 5% in AL49, where the hematite Fe_2O_3 phase is equally present in both samples. As an exmaple; ASTAR phase (colour) and relibility map (grey scale) from one area for TH3 contemporary sample is shown in Figure 1: 46% magnetite Fe_3O_4 (blue), 2% FeO Wüstite (red), 32% Fe_2O_3 hematite (green) and 20% amorphous phase (black) and in Figure 2 one area of AL49 archaeological is shown: 70 % magnetite (blue), 3% Wüstite (red), 27% hematite (green). Although our results are “preliminary”, they highlight the potential of scanning electron diffraction based crystallographic techniques to reveal differences between various archaeological artifacts, having similar chemical composition.

References

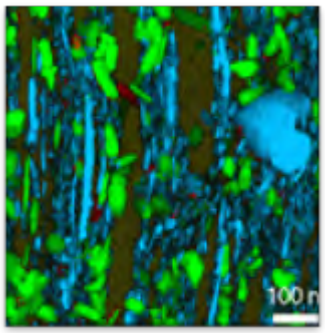
1. P. H. Sciau, “Chapter two - transmission electron microscopy: emerging investigations for cultural heritage materials,” Advances in Imaging and Electron Physics, vol. 198, pp. 43–67, 2016.

2. S. Nicolopoulos, P. P. Das, A. G. Pérez et al., “Novel TEM Microscopy and Electron Diffraction Techniques to Characterize Cultural Heritage Materials: From Ancient Greek Artefacts to Maya Mural Paintings,” Scanning, 2019.

3. N. Zacharias, G. Mastrotheodoros, M. Katsiotis, N. Laskaris, Glazed Pottery Surfaces Under the Microscope: Which Criteria to Decide About Archaeological and Modern Items, ISA 2012, Tampa Florida May 18-22, Book of Abstracts p.85, 2012.

4. Y. Maniatis, E. Aloupi, A.D. Stalios, New evidence for the nature of the Attic black gloss. Archaeometry 35, 1: 23-34, 1993.

ASTAR Phase and Reliability Map for TH3 sample



ASTAR Phase and Reliability Map for AL49 sample