Analytical Research Section
{Private}
Report No. AR 06/16

Investigation of two objects from Tarbat, which may be associated with gold working

(C&AR 13145)

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Object Nos.: D2180 and D3502
Requested by: Dr Andrew Heald (Archaeology Department)

Summary

Two objects found in Tarbat site (7th to 9th century) and though to be associated with gold working were investigated with X-Ray Fluorescence Spectroscopy (XRF) and Scanning Electron Microscopy (SEM-EDX). No traces of gold could be found, however, some traces of brass alloy (copper/zinc) were identified in the surface of the touchstone.

Keywords: Gold working, Tarbat, touchstone, assay tray, 8th century AD

Introduction

Two objects found in Tarbat site and though to be associated with gold working were investigated with X-Ray Fluorescence Spectroscopy (XRF) and Scanning Electron Microscopy coupled with Energy Dispersive X-ray (SEM-EDX). The objects are a possible assay tray of quite important size and a dark touchstone made of a soft dark material (refer to pictures 1 and 2).

Method

The objects were first examined under a low power optical microscope to allow more detailed examination of their appearance. The fragments were then examined and analysed on different areas by X-ray fluorescence spectroscopy (XRF). They were not cleaned and no surface preparation was undertaken. The analysis was qualitative and semi-quantitative.

The touchstone was also observed with scanning electron microscope in Backscattered mode (SEM-BSE) and the composition of some particles observed on the surface was qualitatively investigated using EDX analysis. Due to the too important size of the assay tray, it has not been possible to do SEM observation.

Results

1- Assay tray – D3502

The assay tray was investigated with X-ray Fluorescence. The aim of the analysis was to characterise possible gold trace inside the cavity. However due to the quite important size of the object, the analysis of the cavity was quite complicated to carried out (problem of focalisation of the X-ray beam).

The results didn't show the presence of gold inside the cavity, only the composition of the stone has been characterised: mainly based on iron element.

Figure 1: Possible assay tray, D3502
With XRF analysis localisation

2- Touchstone – D2180

The touchstone is made of a dark soft material. Under low optical observation few gold coloured particles as well as bright white particles, red and white traces of pigments were observed on the surface (refer to pictures 3 and 5).

The analysis of the touchstone using X-Ray fluorescence allowed the characterisation of the stone material: mainly based on manganese, iron and calcium element. The different types of particles observed on the surface were then observed and investigated using Scanning Electron microscopy (backscattered mode) with EDX system.

The result showed that the gold in colour particles are in fact made of brass alloy. The presence of these particles on the surface of the touchstone could possibly be characteristic of metal working use (copper alloy).

The traces of red pigment observed on the surface are made of ochre and iron oxide as the white particles are based on lead pigment or zinc pigment. The presence of this last white pigment corresponds most probably to a pollution of the surface with a modern material[1].

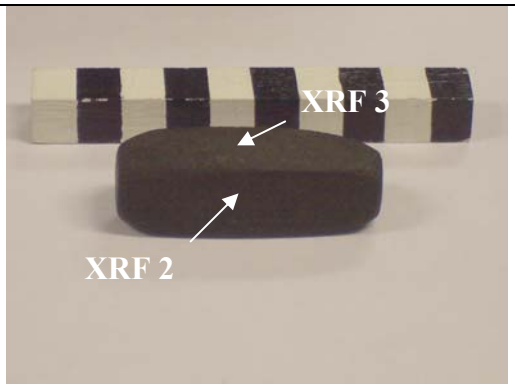

Figure 2: Touchstone D2180
With XRF analysis localisation

Figure 3: Touchstone D2180
Detail of the surface under binocular microscope, with SEM analysis localisation (Area 1)

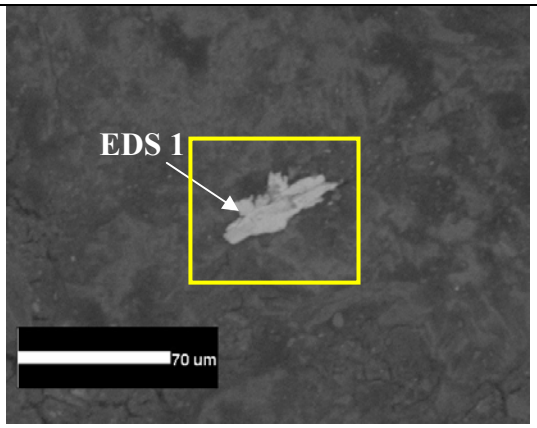

Figure 4: Touchstone D2180
Microphotograph in backscattered electron at x500 magnification, showing Area 1

[1] Zinc oxide pigment was first synthesised at the beginning of the 20th century

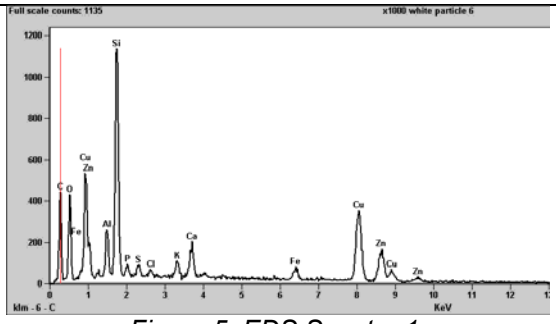

Figure 5: EDS Spectra 1,
showing the presence of brass particle (Cu/Zn)

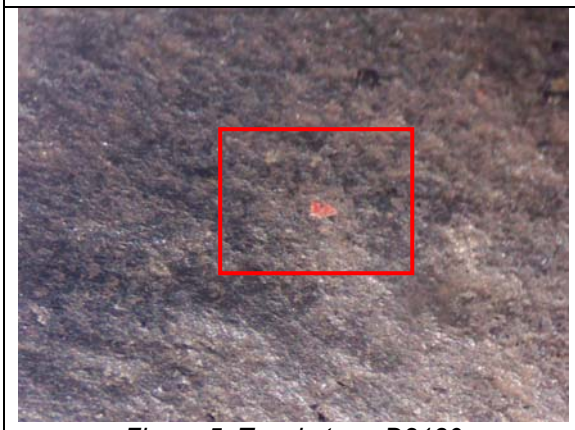

Figure 5: Touchstone D2180
Detail of the surface under binocular microscope, with SEM analysis localisation (Area 1)

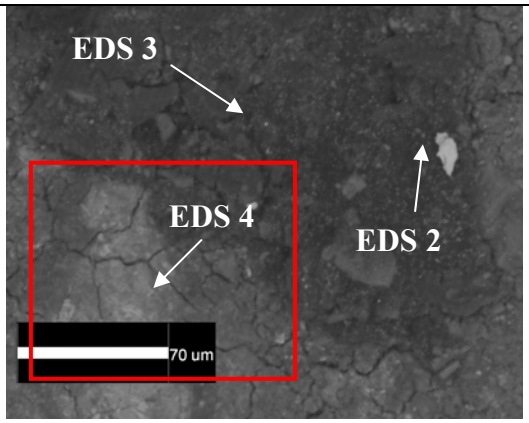

Figure 6: Touchstone D2180
Microphotograph in backscattered electron at x500 magnification, showing Area 1

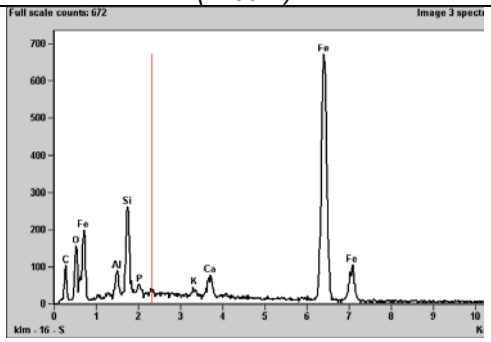

Figure 7: EDS Spectra 2, showing the presence of iron oxide particle

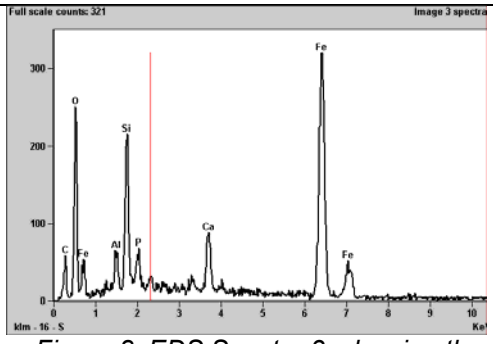

Figure 8: EDS Spectra 3, showing the presence of an earth/ochre material rich in iron

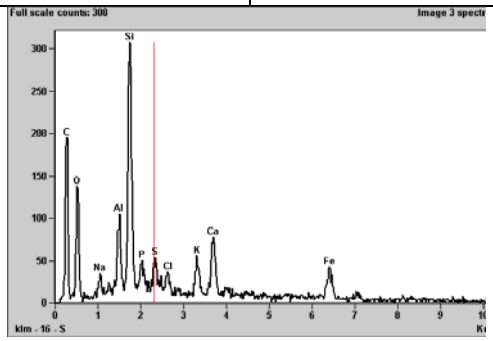

Figure 9: EDS Spectra 4,
showing the presence of an ochre material

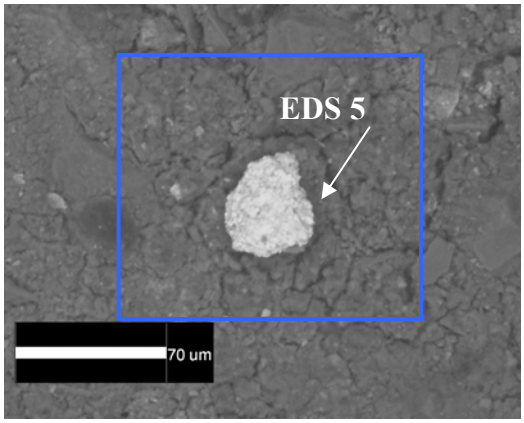

Figure 10: Touchstone D2180 Microphotograph in backscattered electron at x500 magnification, showing Area 3

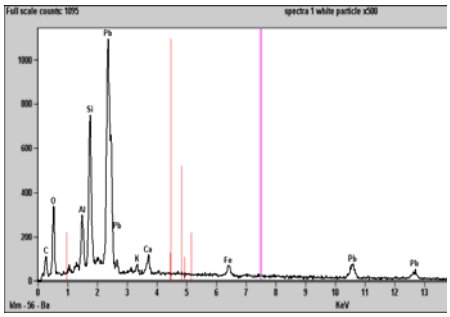

Figure 11: EDS Spectra 5, showing the presence of a lead particle

Conclusions

Only traces of brass alloy were characterised on the surface of the touchstone. These particles could show an association with metal working (copper alloy).

No traces of gold were found on the surface of the touchstone or in the cavity of the assay tray.

Appendix 1

X-ray Fluorescence Method

The XRF system used was an Oxford Instruments ED 2000 with Oxford Instruments software ED 2000SW version 1.31. The analysed area was irradiated with a primary X-ray beam produced by a Rhodium target X-ray tube. The primary beam was collimated to give an analysed area of about 4mm x 2mm. Secondary X-rays were detected with a silicon (lithium) solid state detector. The detection limit varies depending on the elements, matrix and analytical conditions, but is typically in the range of 0.05%-0.2%. As the analytical technique has a limited penetration depth, the reported compositions may not be representative of the bulk of the alloy if there is a chemically distinct surface layer. Spectra were collected under the conditions "Old XRF". This uses an operating voltage of 46kV and a current of up to 1000μA (set automatically for a 45% dead time) without a primary beam filter to ensure detection of all elements of atomic number 19 or above of interest in copper and lead alloys.

SEM –EDX Method

The sample was placed directly onto an aluminium sample holder examined using the CamScan MX2000 SEM in Envac (controlled pressure) mode using various pressures between 15 and 25 Pa. The analysis and imaging were done at a focal distance of 35mm. The selected area was analysed using the Noran Vantage system at x100 magnification in the spot mode (1/16 area). All analyses and photographs at 20kV in Envac mode spot 2 with the aperture fully open.

Spectra

D3502
QT132228 – XRF 1, old XRF conditions

D 2180
QT132225 – XRF 2, old XRF conditions
QT132226 – XRF 3, old XRF conditions