Staffordshire Hoard
Research Report 23

Analysis of Weathered Green Inlays in the Staffordshire Hoard

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2016

This report forms part of
The Staffordshire Hoard: an Anglo-Saxon Treasure
edited by C. Fern, T. Dickinson and L. Webster
and published by the Society of Antiquaries of London

Information about this report

This report was produced in 2016 as part of Stage 2 of the project. At that time the catalogue had not been finalised. The concordance of the K numbers given in the report to the catalogue numbers as they appear in the final publication is as given below. The list also includes the names of the objects as used in the final publication.

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The owners, Historic England and Barbican Research Associates are grateful to the Esmée Fairbairn Collections Fund for the resources which enabled the work to be carried out.

K number	Catalogue number	Name in publication
349	39	Pommel in gold, of cocked-hat form, with filigree decoration and on one side a cloisonné panel.
572	44	Pommel in gold, of cocked-hat form, with cloisonné decoration.
355	54	Pommel in gold, of cocked-hat form, with cloisonné decoration.
451	578	Pyramid-fitting in gold with garnet and glass cloisonné decoration.
660	166	Hilt-collar in gold, of high form, with cloisonné decoration.
680	40	Pommel in gold, of cocked-hat form, with filigree and on one side a cloisonné panel.
682	171	Hilt-collar in gold, of narrow form, with cloisonné decoration.
967	165	Hilt-collar in gold, of high form, with cloisonné decoration.
1166	579	Pyramid-fitting in gold with garnet and glass cloisonné decoration.

Analysis of weathered green inlays in the Staffordshire Hoard

Marcos Martinón-Torres

Abstract

The cloisonné cells of a small number of artefacts in the Staffordshire Hoard appear filled with a green powdery substance. These objects were examined under a microscope, and some samples were removed for further analyses by optical microscopy and SEM-EDS, both as polished cross-sections and as unprepared specimens. The results indicate that the material is now dominated by heavily weathered copper carbonates, in addition to some accessory minerals, with very low levels of silica, alkali or alkali earth oxides. The green material is shown to constitute the weathered remains of an original inlay, but its current composition and microstructure are not consistent with those typical of corroded glass or enamel. It is hypothesised that the original inlay may have been a copper-rich mineral such as cuprite, but this point cannot be confirmed at present.

Introduction

A small number of artefacts with cloisonné decoration in the Staffordshire Hoard appear to have been inlaid with a material different from the much more common garnet, and also visually different from the glass or niello identified in a few objects. The material is heavily weathered in most cases, now appearing predominantly as a powdery green substance. This report presents the results of the microanalytical examination of a few samples that could be removed from cloisonné cells for investigation. The work aimed to verify that the material filling the cells is indeed a deliberate inlay, as opposed to a post-depositional fill from the soil or the result of corrosion of the cloisonné backing material. In addition, it was hoped that the analyses could allow inferences as to the nature of this material before it corroded to its current condition.

Samples and methods

All the objects reported to include this unusual inlay material were first examined visually and microscopically using a Keyence VHX-S50F microscope at the laboratories of the Birmingham Museum and Art Gallery (Appendix 1). Additional analytical work was carried out at the laboratories of the UCL Institute of Archaeology. Sampling was restricted to cloisonné cells where the material appeared loose, so that it could be removed without applying undue pressure or affecting the integrity of better preserved examples. All the samples were smaller than 1mm in diameter, often much less, particularly as the material crumbled as it was removed. Two samples were removed by Pieta Greaves from two different cells in K355 during a first visit in March 2015. These were embedded in epoxy resin and mounted as polished cross-sections by Harriet White. The polished blocks were subsequently examined using a Leica DM4500 P LED polarisation optical microscope equipped with a Leica DFC 290 HD camera. After carbon-coating, they were further analysed with a Philips XL30 scanning electron microscope equipped with an Oxford Instruments X-sight energy dispersive spectrometer (SEM-EDS). SEM analyses were conducted at 20kV, with EDS acquisition times of 100s. The EDS was standardised with metallic cobalt. Given the high porosity of the material, it was decided to examine EDS spectra visually rather than quantifying the results, and hence no reference materials were analysed.

In January 2016, four additional samples were removed during conservation from objects K572, K680, K682 and K967. These samples were examined directly without further preparation, using a Dino-Lite AM4815ZT digital microscope and an Hitachi S-3400 SEM with an Oxford Instruments X-sight EDS. Given that these samples were not prepared for chemical analyses, no attempt was made at quantifying the results. All SEM images shown in this report are from the backscattered electron detector (BSE).

The terms 'green inlay' or 'green material' are used in this report to refer to the material filling cloisonné cells investigated here. It should be noted that the term refers to its current appearance, since we cannot be certain of its original colour before weathering. As such, this should not be confused with the inlay identified in object K744 as artificial green glass (Meek 2013b).

Results

Typologically, the objects bearing this material are broadly comparable to others in the hoard. In particular, three hilt collars (K660, K682, K967) and three pommels (K355, K572, K680) with cloisonné decoration are quite similar to others in the assemblage, with the main peculiarity that no garnet inlay is present, and the cells appear filled with this green material instead (Appendix 1; Fig. 1). Pommel K349, decorated with beaded wire, is peculiar in that the inlay is placed in a separate square panel, which was subsequently attached to the body of the pommel proper (Fig. 2). It is therefore possible – though by no means confirmed – that this panel could have been re-used. In at least one case (K660), this green material appeared associated to niello (La Niece 2013). Finally, pyramid-shaped objects K451 and K1166 are special in that they are the only objects were this green material has been found in combination with garnet inlays (Fig. 3). In these two cases, particularly severe corrosion and material loss make it impossible to ascertain if the green inlay would have been the same as in the other objects, but they are also included in this report for the sake of completeness.

Figure 1. K572 showing typical appearance of the green material mixed with soil in the cloisonné cells.

Figure 2. K349, showing the green inlay material in a separate panel.

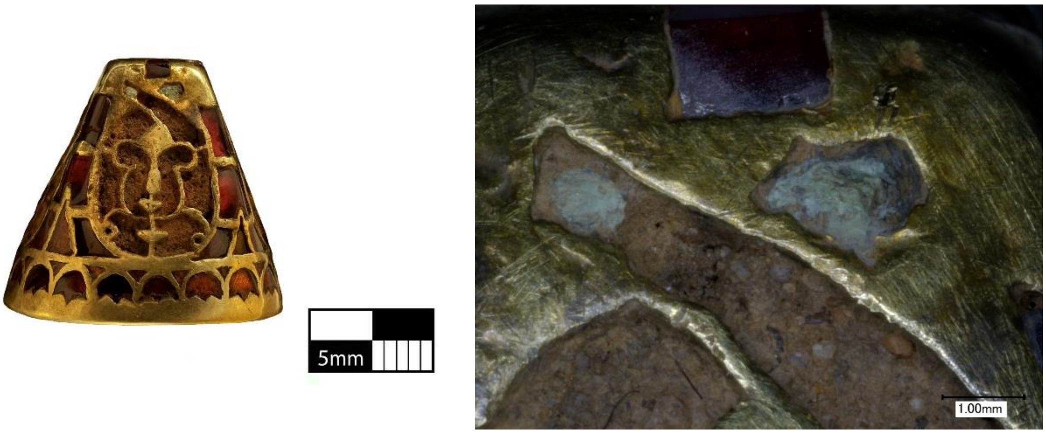


Figure 3. K451 (left) and detail of the cloisonné cells mostly filled with soil but preserving some green material.

In the artefacts as recovered, the green inlay appears covered by the reddish soil from which the hoard was excavated (Fig. 4). After cleaning and microscopic investigation, however, it becomes obvious that the bulk of the material proper is a powdery green substance, sometimes mixed white brown, reddish or yellow tints. Embedded within this paste or in cavities in the same cells, translucent minerals can be found occasionally – but these are likely to derive from soil contamination (Fig. 5-6). It is important to note that the top sides of the gold cell walls are typically rubbed over quite intensively. This is a common feature in cloisonné work generally, to hold the inlay in place, but where the green material is present the burnishing is often unusually intense, suggesting that the inlay could have been polished or cleaned *in situ* (Fig. 7-8). However, this green material now appears raised, sometimes quite considerably, above the height of the walls (Fig. 9-10). This is a strong indication that the material has grown post-depositionally, i.e. after it was placed in the cells. This post-depositional growth during the alteration process would have facilitated the incorporation of soil material.

Figure 4.Two views of K967 showing details of the green material mixed with soil.

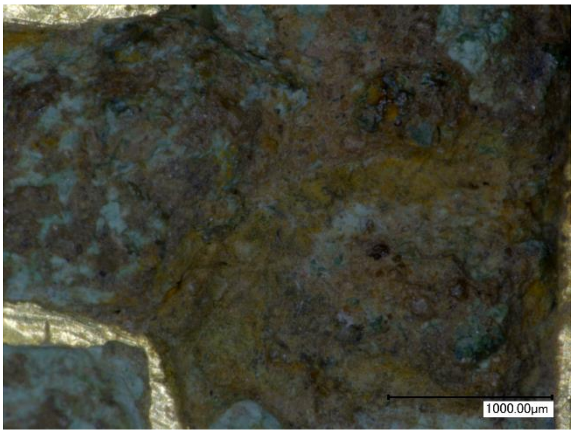


Figure 5. Detail of K349 showing the predominantly green, powdery nature of the material, as well as some translucent inclusions.

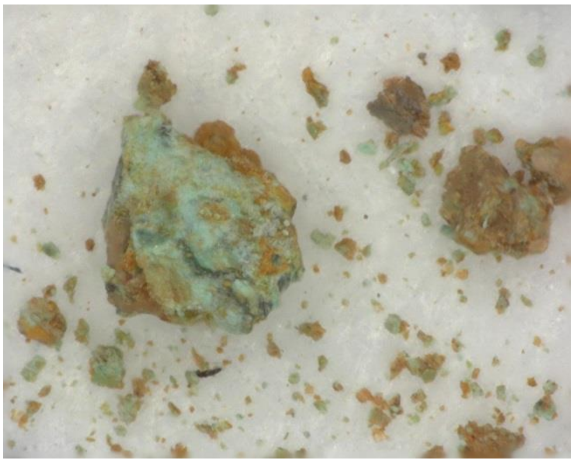


Figure 6. Sample removed from K967 showing its crumbly nature.

Figure 7. Detail of K349 (left) compared to a more typical garnet cloisonné. Note the more intense rubbing on the top of the cell walls of K349.

Figure 8. Detail of K355 showing the green material and the intense rubbing on the top of the cell walls.

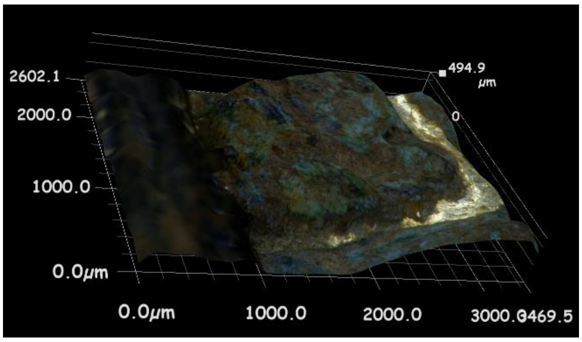


Figure 9. Detail of a cell in K349 showing the post-depositional growth of the green material above the height of the cell walls.

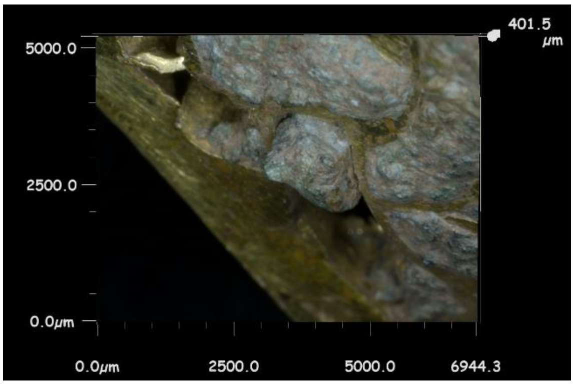
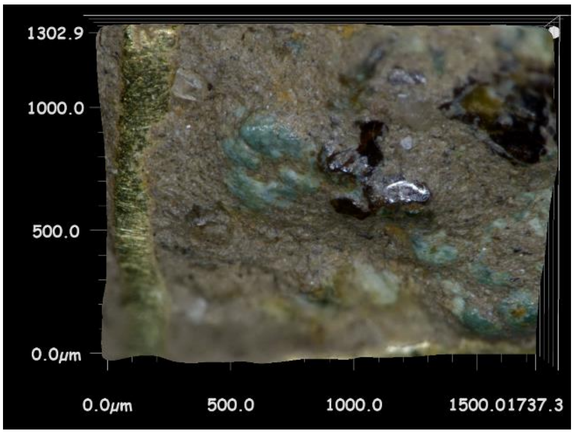


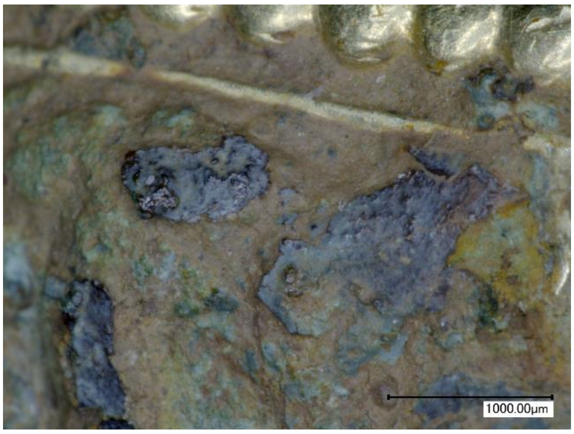
Figure 10. Detail of K355 showing the post-depositional growth of the green material. The small particle in the middle of the photograph was one of the samples prepared as a polished cross-section.

Notwithstanding the predominantly weathered and powdery nature of the material, in a few cases it is possible to identify small parts of the inlay where the material appears glassy and translucent. These phases, generally less than 500 µm in diameter, are typically dark read in colour, though depending on their thickness and lighting conditions they can range in colour from green to dark ruby or black (Fig. 11).



K572

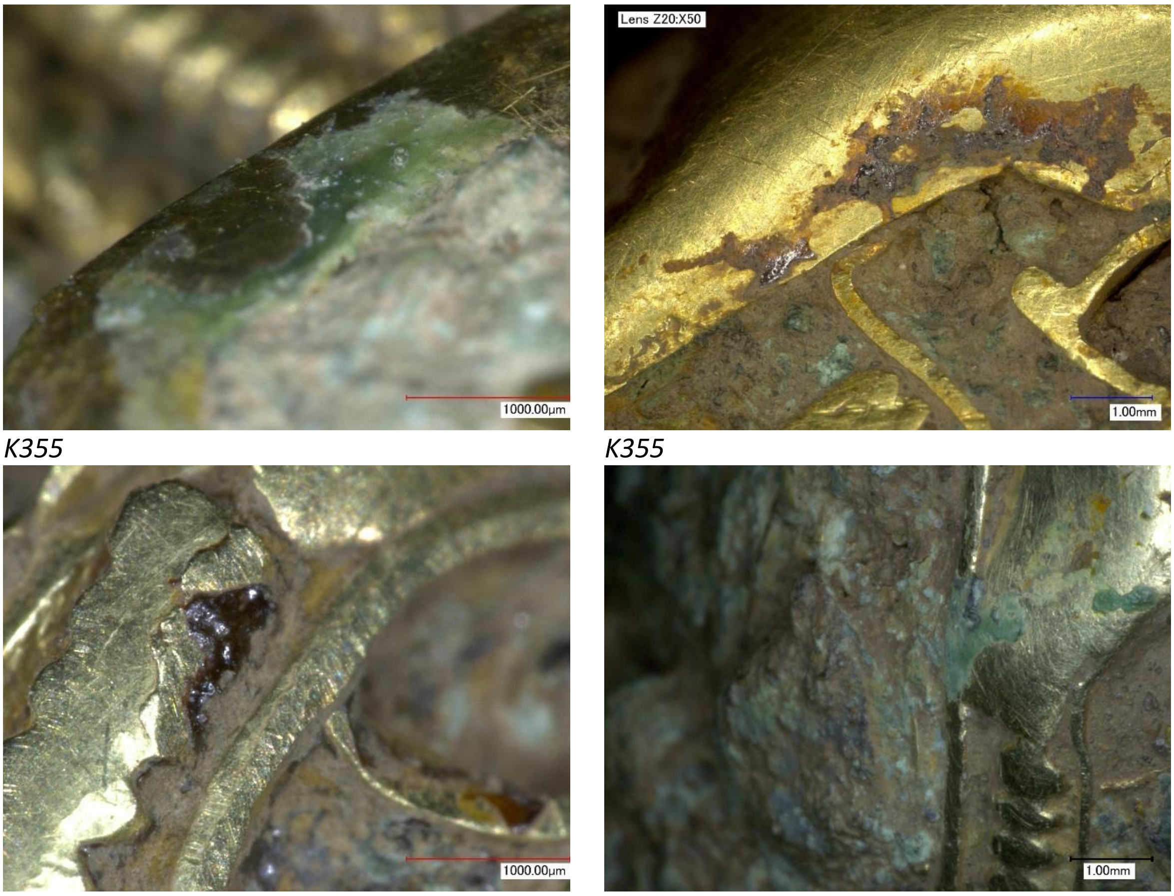
K572



K360

Figure 11. Details of three cloisonné cells where some translucent particles are preserved within the weathered green material.

Another feature worth noting are the small patches of dark red or green, seemingly vitreous material that appear adhering to the top sides of cloisonné walls in a few cases, adjacent to the cells proper (Fig. 12). These would seem coherent with the idea that the material inlaid in the artefacts was perhaps fused within the cells themselves, with these patches representing spillage or overflow that was not properly removed during polishing and cleaning. However, it is also possible that these patches resulted from post-depositional alteration.



K355 *K355*

K572 *K360*

Figure 12. Four views of vitreous material adhering to the top of the cell walls.

Where the inlay material has been lost or removed, examination of the partially empty cells suggests that the inlay material was placed directly inside the metal cells, with no clear evidence of additional backing sheets as documented for the garnet inlays, or of any adhesive or other backing material. The only exceptions to this pattern are objects K451 and K1166, which are also special in showing a combination of garnet and green material. Here, especially in K451, a matt black layer can be noticed adhering to the inside of the cells (Fig. 13). FTIR analysis of one sample revealed the presence of a proteinaceous glue (McElhinney 2015).

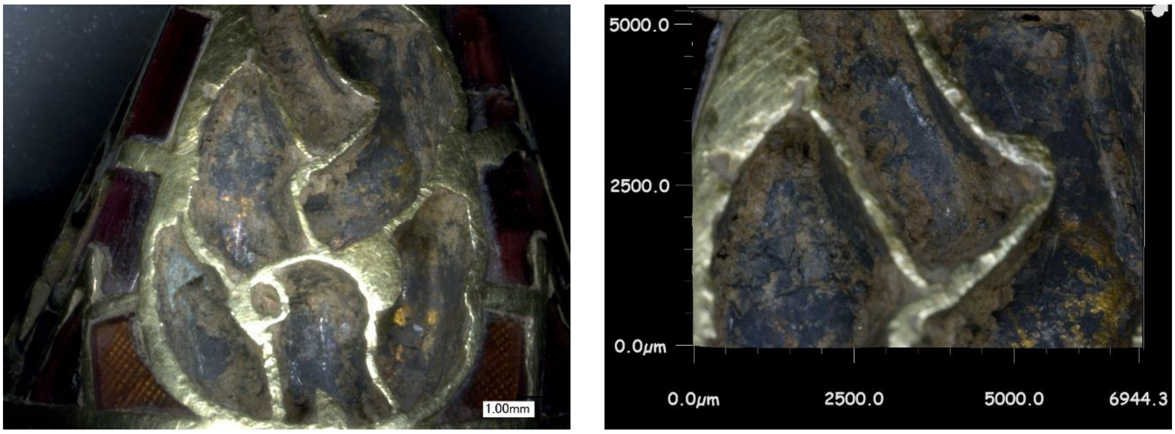


Figure 13. Two views of K451 showing traces of a matt black layer inside the cells, possibly some glue or backing material for the inlay.

Examination of the polished cross-sections from two cells in K355 by optical microscopy and SEM revealed them as a mixture of copper minerals, mostly carbonates, likely resulting from the weathering of a copper-rich material (Fig. 14-18). In some areas these appear as subhedral, very porous grains made up of filaments and separated by an amorphous phase; in other areas they appear as hydrated layers progressively filling pores. Some red copper- and iron-rich material was found filling some of these cavities too, as well as occasional grains of quartz, feldspar and clay, mostly towards the surfaces, and hence confirming their post-depositional nature. Multiple SEM-EDS analyses of the green matrix material consistently showed copper, oxygen and carbon as the main elements present, in addition to relatively small amounts of lead and, occasionally, zinc (Fig. 19).

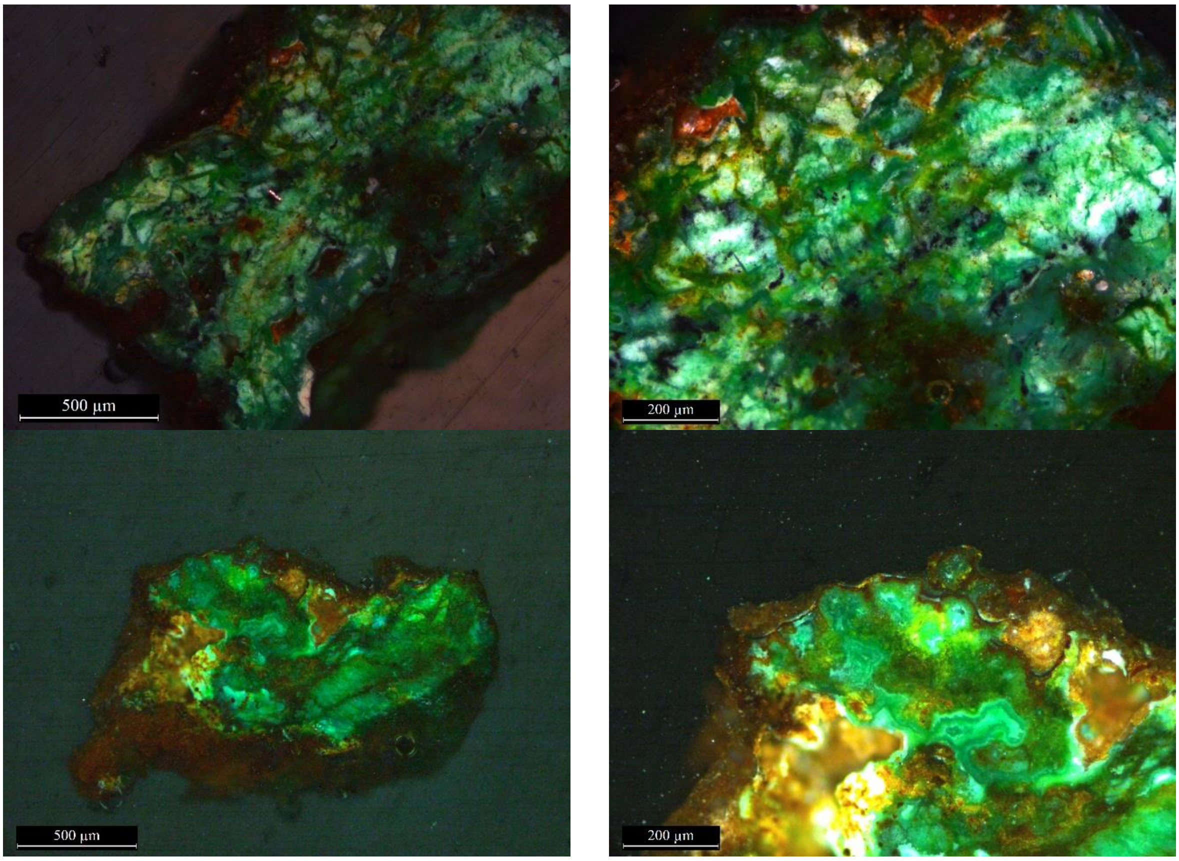


Figure 14. Four views of polished cross-sections of green material from K355, showing the predominance of green copper carbonates, together with some iron- and copper-rich red phases.

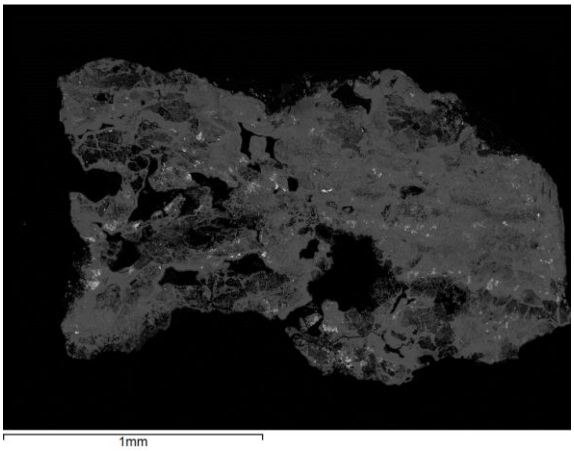


Figure 15. SEM image of a cross-section sample from K355 showing the very porous nature of the material.

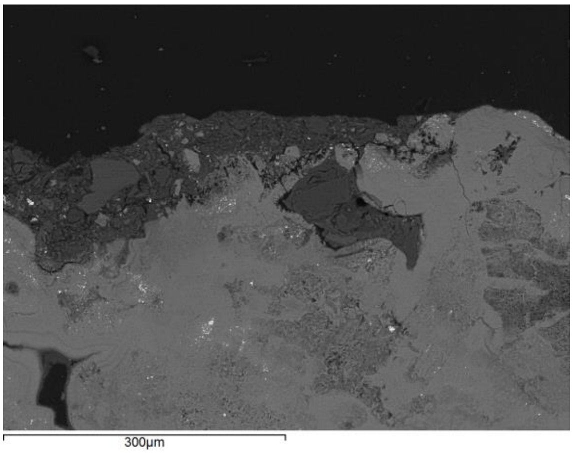


Figure 16. SEM image of a cross-section sample from K355 showing an enrichment in soil contamination (darker grey) towards the surface.

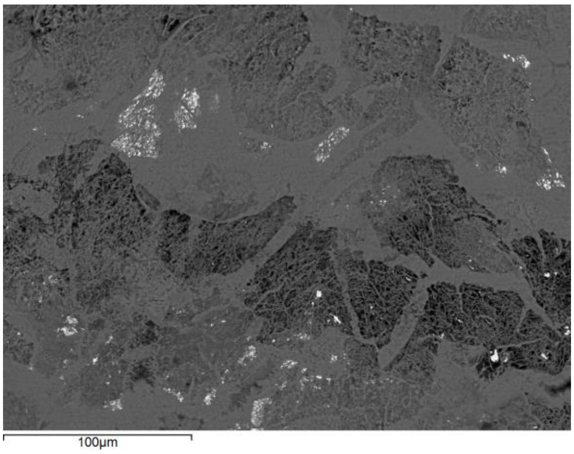


Figure 17. SEM image of a cross-section sample from K355 showing the porous crystals of copper carbonate, with some cavities filled with bright silver sulphides. See Figure 19 for an EDS spectrum of the dominant phase.

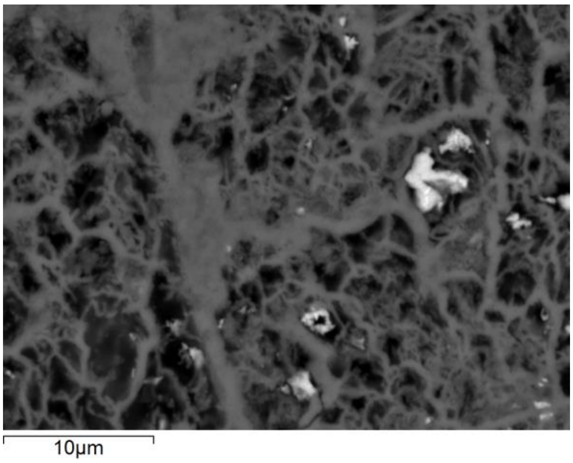


Figure 18. Detail of the silver sulphides filling the pores of the copper-rich matrix in K355, under the SEM. See Figure 20 for a relevant EDS spectrum.

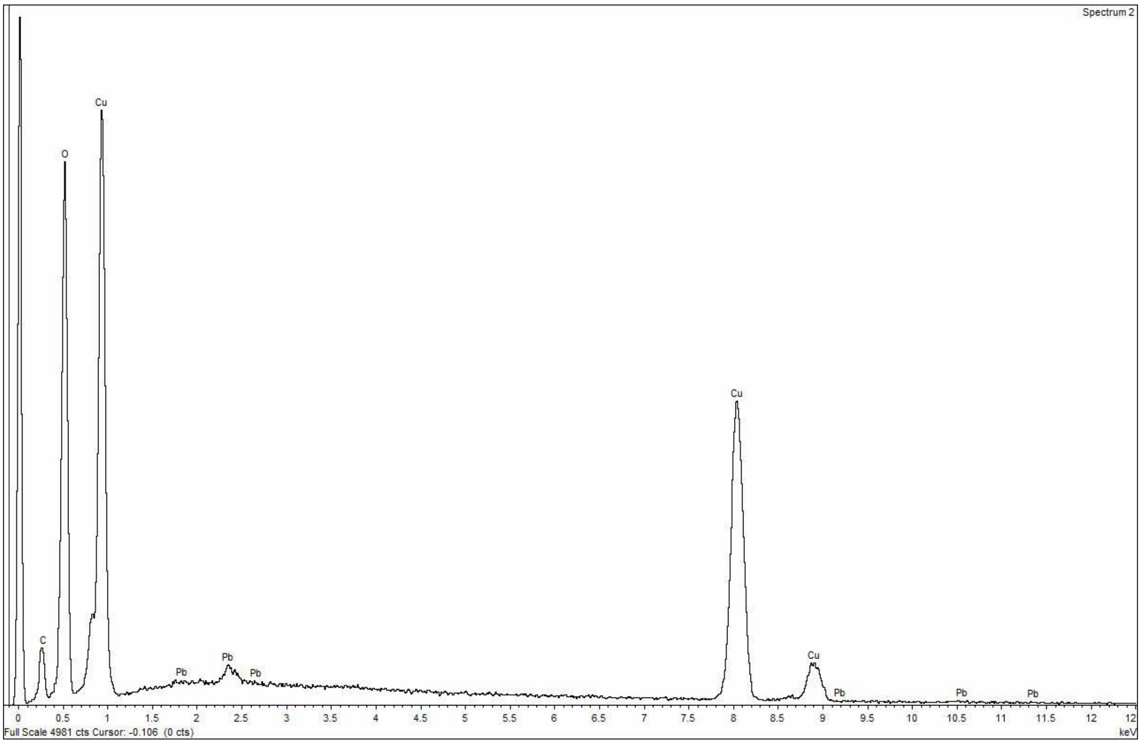


Figure 19. Typical SEM-EDS spectrum obtained in the copper carbonate grains in a polished cross-section of the green material from K355.

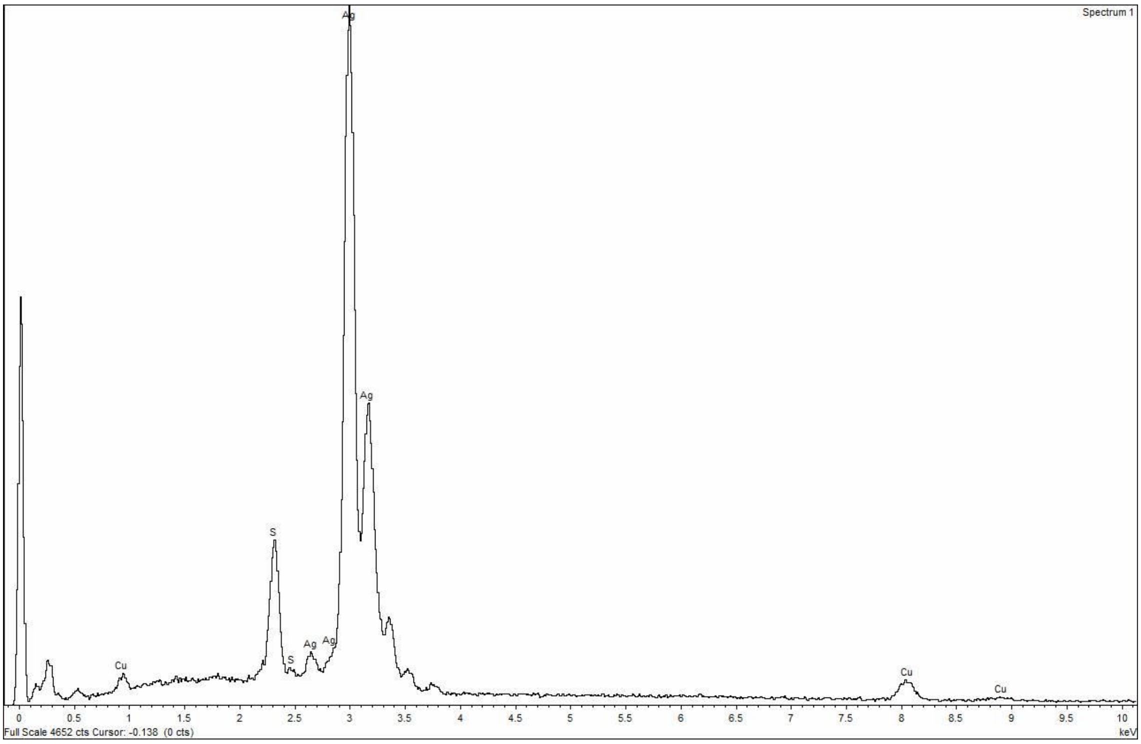


Figure 20. Typical SEM-EDS spectrum of the silver sulphide inclusions within the copper carbonate grains in sample from K355.

An additional phase was identified, in small clusters scattered across both samples. This appears black under cross-polarised reflected light and bright under BSE imaging (Fig 17-18). SEM-EDS of this phase showed it to be silver sulphide, often with some copper (though it is possible that the copper signal may derive from the surrounding Cu-rich volume) and, in one case, gold (Fig. 20). The arrangement of this material, always filling cavities within the copper carbonate grains, leaves little doubt that it is the result of post-depositional migration. Given that a variety of decorative silver sulphides have been identified in objects from the hoard (La Niece 2013; Blakelock 2016), it seems reasonable that some sulphides could have migrated from adjacent objects before redepositing in the locations where we find them now. The sample from K680, examined unprepared under the SEM, also showed the presence of a few silver sulphide grains, albeit more rare (Fig. 21). One of the cells from K355 additionally contained some iron oxide and sulphide particles, predominantly towards the surface.

Since mounted specimens did not reveal any seemingly pristine material surviving in the cross-sections exposed, it was decided that subsequent samples would be examined without any prior preparation. It was hoped that any better-preserved material potentially surviving could be analysed directly, rather than risking to lose it during sample preparation. Only one of the samples examined, from pommel K572, was found to contain a small fragment of dark red, lustrous, translucent material, surrounded by green and brown powdery products (Fig. 22-24). Both area and spot analyses of this particle by SEM-EDS showed iron and oxygen as the main elements. Light elements found in much lower concentrations include silicon and aluminium, followed by magnesium, phosphorus, potassium and calcium – the latter found in some analyses only. Heavier elements include copper, which was invariably detected, and lead. In addition, tin, sulphur and chlorine were detected in minor amounts in a few areas (Fig. 25). While this phase appears homogeneous, a few micrometric bright particles were identified on its surface under the BSE detector. These were found to be richer in copper and sulphur as well as, occasionally, zinc and cobalt.

When the composition of the red phase is compared to that of the surrounding soil-rich weathered material, the latter appears as richer in all the light elements up to calcium as well as copper, and correspondingly poorer in iron (Fig. 26).

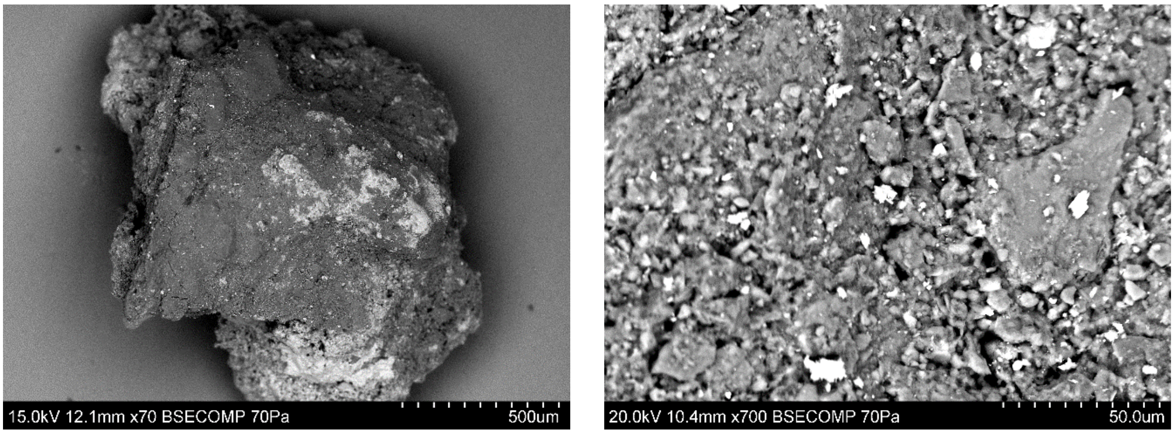


Figure 21. Overall view of sample from K680 under the SEM (left), and detail of its surface, showing clay minerals and some bright silver sulphides.

Figure 22. Sample from K572 under the digital microscope, showing a red particle preserved in the porous mass of green and brown minerals. Figure 22.

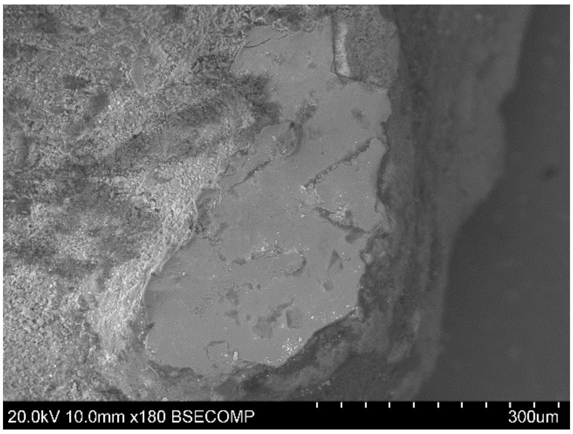


Figure 23. Detail of the red particle shown in Figure 22, as seen under the SEM. For an EDS spectrum of this area, see Figure 25.

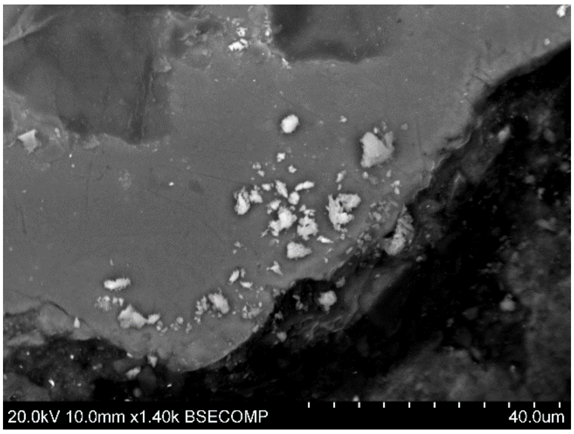


Figure 24. Detail of small bright cristals on the red particle in K572, as seen under the SEM.

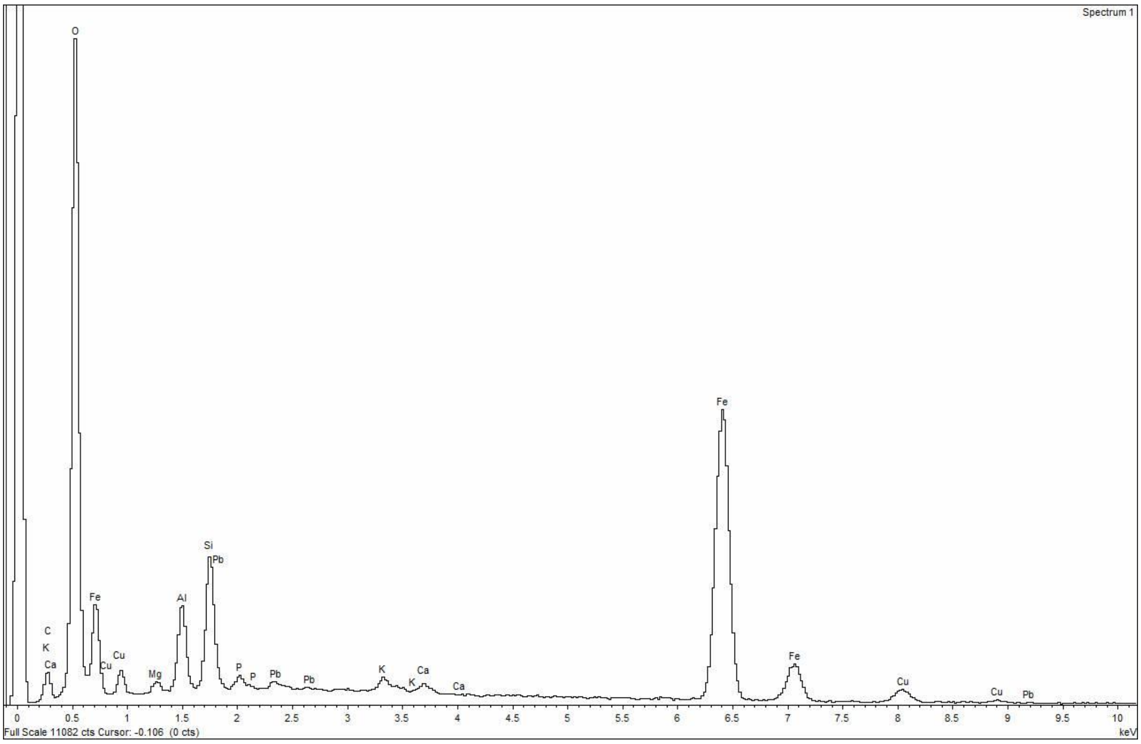


Figure 25. Typical SEM-EDS spectrum for the red area identified within the sample from K572, obtained on an unprepared surface.

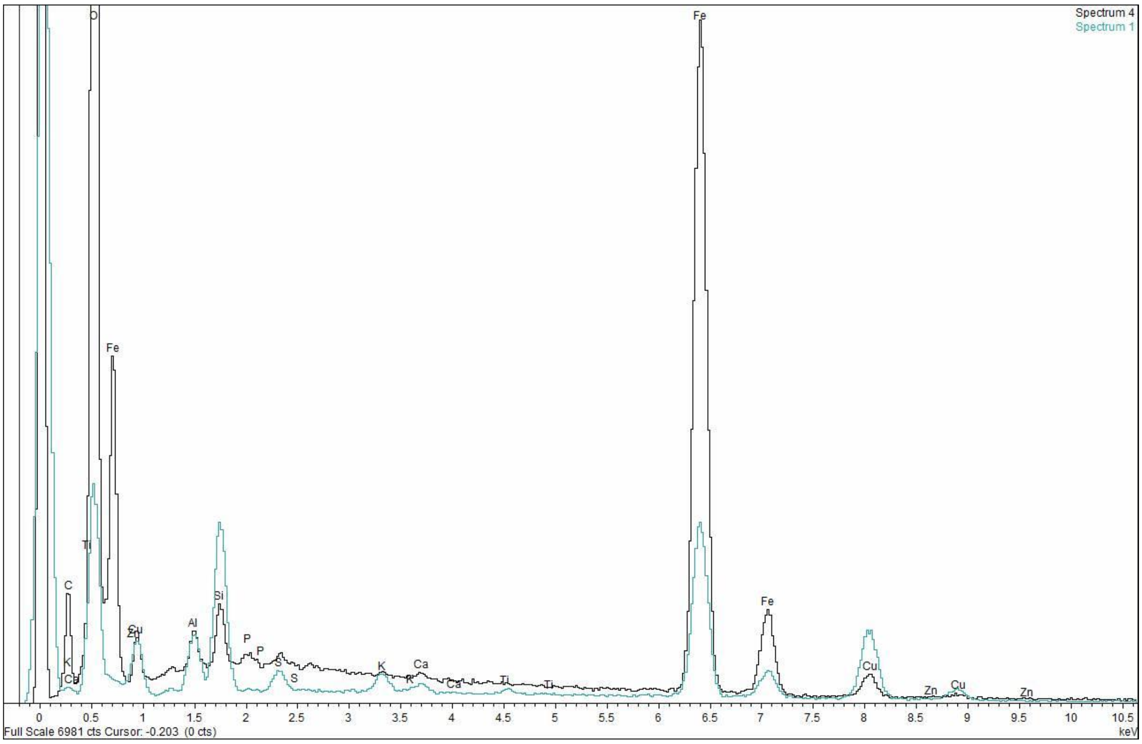


Figure 26. Comparison of SEM-EDS spectra for the red particle (black line) and the adjacent matrix (blue line), as shown in Figures 22 and 23.

Discussion

Susan La Niece conducted an initial assessment of this green, copper-rich material, which she encountered in hilt collar K660 during her investigation of niello inlays. She raised the hypotheses that it could constitute decayed inlay material, backing paste or metal corrosion – although discounting the latter as unlikely, given the absence of any other copper components in the object (La Niece 2013). The findings of the current investigation are in agreement with her suspicion that this material, although rich in copper, does not derive from the corrosion of this metal. In fact, metallic copper is infrequent in the Staffordshire Hoard, and no metallic copper was identified in any of the objects covered in this report (Blakelock 2015). In addition, the microstructure of the material is not consistent with those typical of corroded metals, which tend to display a layered texture, and often preserve the ghost structure of the parent metal in some areas at least. Similarly, it is unlikely that this green material constitutes the remains of a backing paste: not only it would seem too bulky, but also a very unusual choice. An extensive survey of the backing pastes in a wide range of artefacts from the hoard identified a variety of both inorganic and organic materials, but none is comparable to those recorded here (McElhinney 2015).

Hence, we can be reasonably confident in inferring that the material in question is the result of the weathering of some inlay that was deliberately placed in these objects during manufacture, with the expectation that it would be seen. This practice might have aimed at obtaining visual effects different from the more common red garnets, and/or it may simply reflect the use of a different material where garnet was not available or desirable.

A reasonable assumption would be that this material could have been glass or enamel, now severely corroded. The patches of seemingly molten material identified on the cell walls, as well as the translucent coloured particles remaining within some of the green mass, would seem to support this inference. There are, however, some problems that prevent confirmation of this point. The first and most important is the composition, as detected by SEM-EDS in the samples analysed. As described, the bulk of the material in the more weathered, predominant part of the samples is now a copper carbonate. Glass corrosion often involves the loss of silica; alkali and alkali earth elements can be lost too through weathering (Freestone 2001). As a result, corroded glass may appear richer in heavy elements than their pristine counterparts. On this basis, it would be tempting to think that the material under examination might have been copper-coloured, red glass, perhaps used instead of garnet. The presence of lead would be consistent with the lead-rich nature of some Anglo-Saxon glass and enamels analysed previously (e.g. Stapleton et al. 1999; Peake and Freestone 2014). However, the levels of silica and other light oxides would seem too low here, even for a corroded glass. In the samples analysed as cross-sections and therefore cleaner of soil, virtually no silica, alkali or alkali earth oxides were detected. Their microstructure is different from the typical layered arrangement of devitrified glass, and it preserves no particles that could be interpreted as remainders of colourants or opacifiers added to glass.

In the red particle within the K572 sample reported above, the levels of iron seem too high to be comparable to those of any glass known for the period (cf. Freestone et al. 2010). If iron-rich copper slag had been used to colour the glass, as recorded in medieval red enamel (Stapleton et al. 1999; Peake and Freestone 2012), one would expect remainders of slag particles to have survived corrosion and thus be identifiable under microscopic examination. It should also be noted that glass of various compositions has been identified as an inlay in other objects of the hoard, and hence exposed to the same post-depositional environment, but it was reasonably well preserved (Meek 2012; 2013a). Thus the identification of this material as glass or enamel remains problematic.

As an alternative explanation, it may be hypothesised that the material was already inlaid in these objects in a mineral form. The green copper carbonates currently dominating the samples are likely to constitute malachite [$Cu_2CO_3(OH)_2$, or $Cu(OH)_2 \cdot (CuCO_3)$]. It is unlikely that natural malachite would have been the original inlay employed since, as discussed above, it seems that the material has expanded considerably after it was placed in the cells, probably by incorporating additional oxygen and hydrogen during weathering. One possible candidate is azurite [$Cu_3(CO_3)_2(OH)_2$, or $Cu(OH)_2 \cdot 2(CuCO_3)$]. The ratio of $Cu(OH)_2$ to $CuCO_3$ is 1:2 in azurite and 1:1 in malachite. Given that $Cu(OH)_2$ is more highly oxidised than $CuCO_3$, malachite can easily result form the oxidation of azurite as per the following equation:

$$2[Cu(OH)_2 \cdot 2(CuCO_3)] + H_2O \rightarrow 3\,[Cu(OH)_2 \cdot (CuCO_3)] + CO_2$$

An even better candidate might be cuprite (Cu_2O), which can weather to form malachite in a variety of redox and pH environments (Pourbaix 1973: 302-306), for example:

$$Cu_2O + H_2CO_3 + H_2O \rightarrow Cu(OH)_2 \cdot CuCO_3 + 2H^+ + 2e^-$$

Minor amounts of heavy elements such as iron, lead, zinc and cobalt can occur in accessory minerals associated to the above copper minerals, and thus the fact that they are detected in the green inlay would not argue against its origins as a natural mineral. Residual grains such as the more iron-rich red particle characterised above for sample K572 could also constitute remainders of accessory minerals, perhaps haematite (Fe_2O_3) – more resistant to weathering and not representative of the bulk of the original inlay. The main objection to this interpretation would be the presence of the seemingly molten patches adhering to the cell walls, as well as the apparent absence of adhesives or other backing material preserved inside most cells: these features would seem more consistent with an inlay that was melted *in situ*, rather than soft stones cut and carved before placing them in the cloisonné.

Conclusions and further work

Examination of the crumbly green material preserved inside cloisonné cells of some Staffordshire Hoard has verified that this constitutes a remnant of an original and intentional inlay, as opposed to the corrosion products of a metal or other backing material. This inlay is now composed predominantly of copper carbonates, with minor amounts of lead and occasional presence of zinc and cobalt. Lighter compounds such as silica, alumina, alkali and alkali earth oxides are detected, but typically in low concentrations and towards the soil-contaminated surfaces. A more lustrous, red grain preserved in one of the cells was found to be particularly rich in iron. Although we cannot rule out the possibility of this green inlay deriving from glass or enamel, its composition as recorded in the few samples analysed is not consistent with any known glasses, even if we allow for the loss of silicon and alkali elements through severe corrosion. As an alternative explanation, it is hypothesised that the original inlay could also have been a natural copper-bearing mineral, perhaps cuprite, which subsequently weathered to its current condition – but this point cannot be confirmed conclusively. As a red mineral, cuprite could have served as an alternative to garnet where this mineral was not available.

The present work was constrained by logistical difficulties, as complete artefacts could not be taken to the laboratory for direct examination under the SEM. As such, only loose samples could be removed for analyses, and these were not always the best preserved. The characterisation presented here could be improved if full objects could be analysed under an SEM, perhaps with selected polishing of some cell portions to facilitate more reliable SEM-EDS analysis. This would allow analyses of multiple cells, and a more adaptive selection of the optimum areas for analysis, including the apparent patches of translucent material preserved on top of some cloisonné wall cells. It would also be useful to conduct a survey of medieval goldwork seeking possible examples of the use of soft stones for inlays, particularly copper minerals, ideally showing different extents of weathering. This may help falsify the hypotheses presented here.

Acknowledgements

I am grateful to Pieta Greaves for her help in the initial examination of the artefacts and her dexterity removing samples. I am also grateful to Harriet White for her meticulous sample preparation, and to Ian Freestone for many valuable comments. All errors and deficiencies remain my own.

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London, 2016

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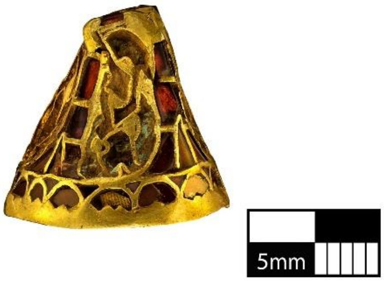
Appendix 1. Artefacts in the Staffordshire Hoard with weathered green inlays.

K number	Description	Image
349	Pommel. All detail obscured by dirt. No garnets	
355	Pommel: green deposits. No garnets	
572	Pommel. Particularly delicate cell construction. No garnets	
660	Collar: Gold and garnet cloisonné in zoomorphic intertwined patterns. No garnets.	 

680	Pommel. Particularly delicate cell construction. No garnets	
682	Narrow collar. No garnets.	
967	Collar: Gold and garnet cloisonné in zoomorphic intertwined patterns. No garnets.	
451	Pyramid fitting. Cloisonné garnets and glass.	 
1166(s)	Pyramid fitting. Cloisonné garnets and glass.	 

Staffordshire Hoard Research Reports

Staffordshire Hoard Research Reports were produced by the project

Contextualising Metal-Detected Discoveries: Staffordshire Anglo-Saxon Hoard

Historic England Project 5892

The Staffordshire Hoard is owned by the Birmingham City Council and the Stoke-on-Trent City Council and cared for on their behalf by Birmingham Museums Trust and The Potteries Museum & Art Gallery.

The Staffordshire Hoard research project was conducted by Barbican Research Associates Ltd and funded by Historic England and the owners.

The work in this report was funded by a grant from the Esmée Fairbairn Collections Fund.

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City of Stoke-on-Trent

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The Potteries museum & art gallery