Investigation into the nature of metal threads in a Renaissance tapestry and the cleaning of tarnished silver by UV/Ozone (UVO) treatment

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Precious metal threads from a XVIth century tapestry were visually classified using optical microscopy and scanning electron microscopy (SEM). The nature of the surface metals and the nature of the corrosion products were studied by Energy dispersive X-ray analysis (EDX) and dynamic secondary ion mass spectrometry (SIMS).

The cleaning of metal artefacts by UV/Ozone treatment was investigated and the surface properties before and after UVO treatment were determined by SIMS.

1. Introduction

During the first half of the XVIth century King Henry VIII commissioned a Flemish Gobelin tapestry cycle for the Great Hall in Hampton Court Palace (HCP). The ten tapestries depicting the Story of Abraham's Life were designed and woven in the most prominent workshops of Brussels, which was at the time the centre of this lucrative industry. Each one of these tapestries is estimated to have cost as much as building a fully equipped battleship and today the tapestries are important assets in Britain's valuable national heritage [1–5]. The materials used are wool for the warp threads and wool, silk and metal threads for the weft. Large areas of the design, especially those depicting clothing, contain the precious metal threads [5].

Several studies on the history and provenance [6–10], structural and elemental composition [8, 11–15] and manufacturing methods [16–19] have described five categories for the incorporation of metal in textiles (Table I).

The metal strips, predominantly gold and silver, were made by cutting strips from a sheet, or flattening a drawn wire by hammering or passing it through rollers [20]. The fibrous core is usually silk but metal threads with a linen, cotton or animal hair core have also been reported [6, 21, 22]. The organic base, categories IV and V, could be either cellulosic (paper) or proteinaceous (animal gut or leather). The nature of the adhesive has not yet been accurately identified [14].

Over the centuries the metal threads have corroded and within a textile environment in particular, they present a problem to the textile conservator. The removal of corrosion can be technically difficult and is ethically questionable in terms of restoring the metal to its original appearance as opposed to conserving in its present state. There are various cleaning techniques but none offers protection from re-corrosion and all have the disadvantage of being harmful to both metal and textile. Therefore the metal threads are often not cleaned [20, 23–25].

In this study light microscopy and scanning electron microscopy (SEM) have been employed to characterise 110 metal thread samples removed from one of the 'Story of Abraham's Life' tapestries during cleaning. The surface nature of some metal threads together with a method for potentially cleaning silver artefacts with UV/Ozone treatment has been investigated using SEM coupled with Energy dispersive X-ray analysis (EDX) and dynamic secondary ion mass spectrometry (SIMS).

The UV/Ozone cleaner irradiates a sample with short-wavelength UV radiation, specifically at 184.9 nm and 253.7 nm. Atmospheric oxygen absorbs radiation at 184.9 nm generating atomic oxygen and ozone, both of which are strong oxidising agents. Ozone, in turn, is dissociated by the 253.7 nm radiation, leading to the generation of further atomic oxygen [26, 27].

While SEM coupled with EDX is a routine analytical tool for textiles, dynamic SIMS is novel. In dynamic SIMS the surface of a specimen, kept under ultra-high vacuum, is bombarded with energetic primary particles, causing sputtering, i.e., the emission of secondary particles, which may be single atoms or molecules and molecular fragments [28]. The primary ion beam can be raster scanned across the surface and by collecting secondary ions at each point of the area, a chemical

TABLE I Categories for inclusion of metal in textiles

I.	Metal applied (with adhesive) to already woven fabric
II.	Metal wire or flattened strips
III.	Metal wire or strips wound around a fibre core
IV.	Metallic surface applied (with adhesive) to organic wrapping wound around fibre core
V.	Metallic surface applied (with adhesive) to organic strips

image of varying elemental or molecular intensities can be generated. If the primary ion beam is repeatedly scanned across an area the surface will erode over time, changes of elemental composition with depth can thus be monitored and represented as depth profile graphs of Time versus Intensity for certain elemental or molecular isotopes. Conversion to Depth scale versus Intensity is possible by direct measurement of the etched surfaces formed during bombardment [29, 30].

2. Experimental

2.1. Materials

The Textile Conservation Studio of Hampton Court Palace provided 110 samples of metal threads taken from the tapestry ‘Abraham meets Melchizedek’. The Victoria and Albert Museum kindly donated silver foil.

2.2. Classification of metal threads

The metal thread samples were viewed under an Olympus model optical microscope with a maximum magnification of 40× and were classified according to their appearance.

2.3. SEM and EDX

A Philips SEM 525 system coupled to an Edax DX4 Energy dispersive X-ray analysis system and software was operated at 20 kV with a collection time of 100 s.

2.4. Dynamic SIMS

The metal thread samples were analysed using a CAMECA IMS 4f Magnetic Sector SIMS instrument, and mass versus intensity spectra, depth profiles and SIMS images obtained.

The Cs^+ primary ion beam was operated at 10 keV, with a beam current of 1 nA and for depth profiles 2–5 nA and 15 nA The areas analysed were $100 \times 100\ \mu$m and $150 \times 150\ \mu$m, respectively. Typical acquisition times were 300 s for spectra and 3000 s for depth profiles. A reference crater was measured using a Dektak Profilometer; subsequent depth calibrations were made based on the reference crater measurement, using a sputter rate factor. The approximate depth of analysis into the subsurface was 250–550 nm.

2.5. UV/Ozone cleaning of a silver foil

The silver foil, 0.75×1 cm, was subjected to the Oddy-test, which involves incubating silver foil with wool in a sealed test tube, to simulate tarnishing during display and storage [31, 32]. A control sample underwent similar treatment but without the wool present in the test tube. The tarnished foil was divided and half was treated in the UV/Ozone Cleaner Model 42-220 from the Jelight Company, USA. The treated (cleaned) and the tarnished samples were analysed using SIMS, with the conditions as described, however the analysed area was larger, $300 \times 300\ \mu$m and the depth of analysis down to 8 nm.

3. Results and discussion

3.1. Classification of metal threads

All 110 metal threads examined in this study belong to category III, Table I, with the core material being exclusively silk. Within category III five different groups of metal threads were identified, Table II, with one group comprising individual threads with special features, group 5. It was assumed that the samples within groups 1 to 4 belong to a minimum of four different manufacturing batches, respectively. The thread diameter differs between 0.25 mm and 1 mm, the tightness of wrapping varied significantly and the colour of the silk core of silver threads was a lighter yellow than that of the gilt silver threads. All samples of group 3 are less corroded than any of the other metal threads, suggesting a thicker or purer gold layer. Representative photographs of the groups 1–4 are illustrated in Fig. 1.

Seven of the group 5 samples have two layers of metal wrapping, with each type being distinct, Fig. 2.

TABLE II Classification of metal threads

Criteria for classification	Groups				
	1	2	3	4	5
Colour of silk core	Yellow	Yellow	Yellow	Light yellow	Light yellow and yellow
Diameter	Medium $\sim$0.5 mm	Large $\sim$0.75–1 mm	Small $\sim$0.25 mm	Medium $\sim$0.5–0.75 mm	Variable
Nature of metal strip	Gilt silver	Gilt silver	Gilt silver	Silver	Gilt silver and silver
Width of metal strip	Medium $\sim$0.4 mm	Medium $\sim$0.4 mm	Narrow $\sim$0.25 mm	Narrow-medium $\sim$0.25–0.5 mm	Variable
Direction of metal strip twist	S	S	S	S	S and Z
Tightness of wrapping	Medium	Loose	Tight	Medium	Variable
Degree of corrosion	Differing	Medium	Low	Heavy	Variable
Special features			Very small gaps between coils		Looped, twisted threads, double wrappings, possibly underlined coils, unidentifiable samples
Quantity of samples	39	15	13	23	20

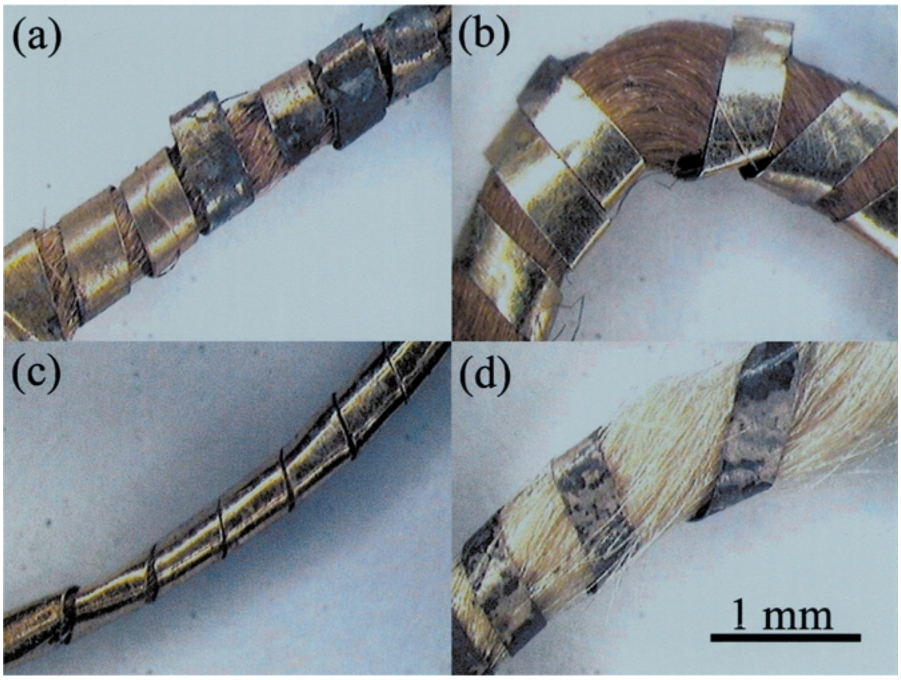

Figure 1 Samples of groups 1–4: (a) sample 55 F3 of group 1, (b) sample 47 F6 of group 2, (c) sample 159 B6 of group 3, and (d) sample 52 F5 of group 4.

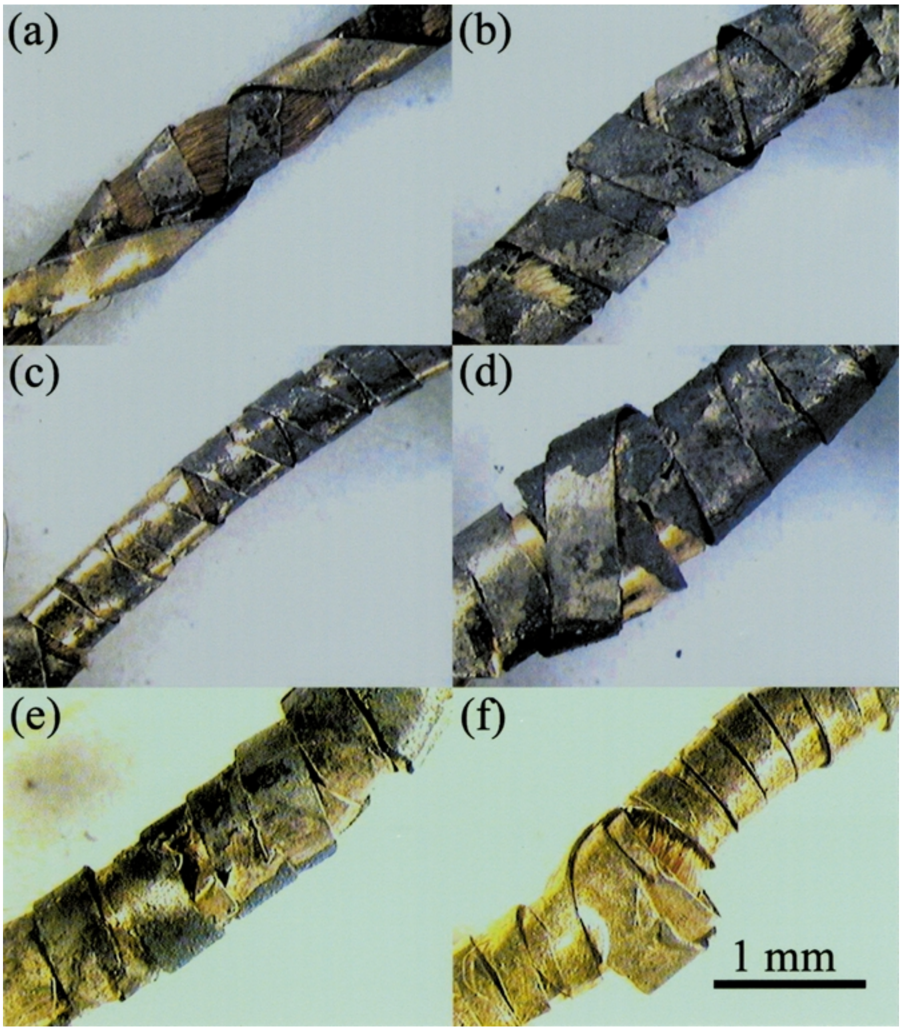

Figure 2 Samples of group 5: (a) sample 43 F6: Z over S, gilt Ag over Ag, yellow core, ↓ ~0.5 mm, strip width ~0.3 mm, wide gaps ~1 mm between Z layer coils, (b) sample 94 D6: Z over S, Ag, light yellow core, ↓ ~0.8 mm, strip width ~0.4 mm, (c) sample 64 E6: Z over S, gilt Ag, yellow core, ↓ ~0.5 mm, strip width ~0.3 mm, possibly same batch as sample 28 G6 (not shown), (d) sample 67 E6: S over S, possibly Ag over gilt Ag, yellow core, ↓ ~1 mm, strip width ~0.4 mm, (e) sample 136 C6: S over Z, gilt Ag, yellow core, ↓ ~0.8 mm, strip width ~0.4 mm, and (f) sample 73 E4: S over Z, gilt Ag, yellow core, ↓ ~0.6 mm, strip width ~0.25–0.3 mm but shows irregularity, very shiny gold strips similar to those of group 3 singular wrapped threads.

Little information is available about the nature of these extremely rare double wrapped threads. Being noted only once previously [13], they appear to be rare examples of overt wealth. They are situated in the bottom border or lower part of the design where a close viewer could appreciate the abundance of precious metal and the intricacy of individual threads.

3.2. SEM and EDX

SEM analyses of some metal strips indicate the edges are round and smooth suggesting the metal strips were made from cast gilt rods drawn into wire and flattened by rollers. However, micrographs of other metal strips show edges with lines and sharp angles, which could be tool marks left from an earlier manufacturing route whereby a top-gilt block was beaten into a thin sheet and subsequently cut into strips, Fig. 3. This latter manufacturing route was rarely used by the beginning of the sixteenth century [8, 9] and little is known about the gilding methods, the tools used for cutting strips and spinning metal threads [6, 9]. The easier method of cast, drawn and rolled wire was already known in early mediaeval times [19] and from the 14th century onwards found its use in textile metal threads [11]. However, the change-over from single-sided to double-sided gilding, i.e., from 'beaten and cut' to 'cast, drawn and rolled' metal threads took place in the late 15th to 16th century [33, 34]. The presence of both kinds of metal threads in the "Abraham meets Melchizedek" tapestry gives evidence for this transitional period of manufacturing methods.

Visual and X-ray analysis indicate the presence of silver and gold. Copper is also detected as part of the

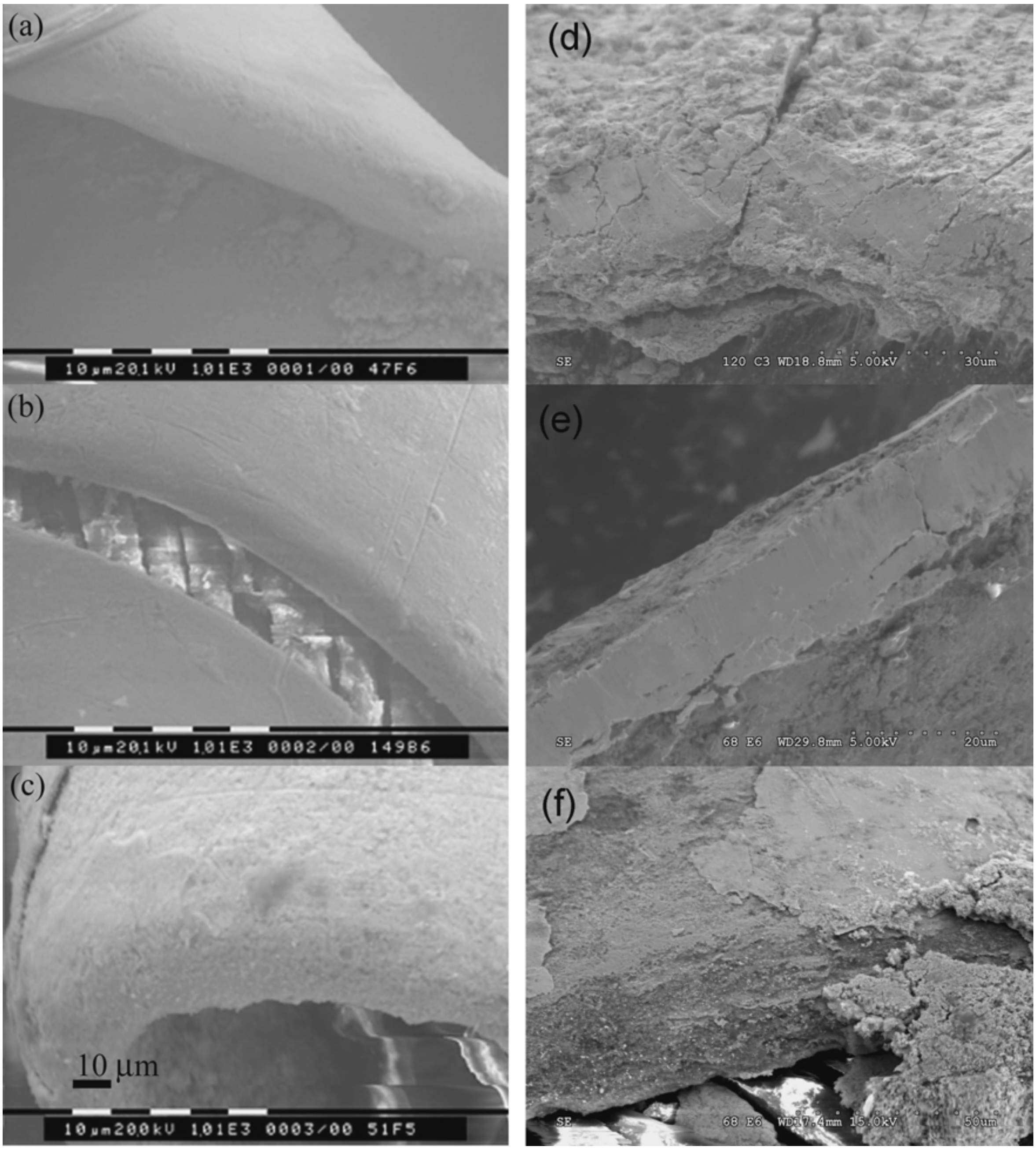

Figure 3 Edges of metal strips: (a) sample 47 F6 of group 1, (b) sample 51 F5 of group 4, (c) sample 149 B6 of group 3 (d) sample 120 C3 of group 1 (e) & (f) sample 68 E6 of group 3.

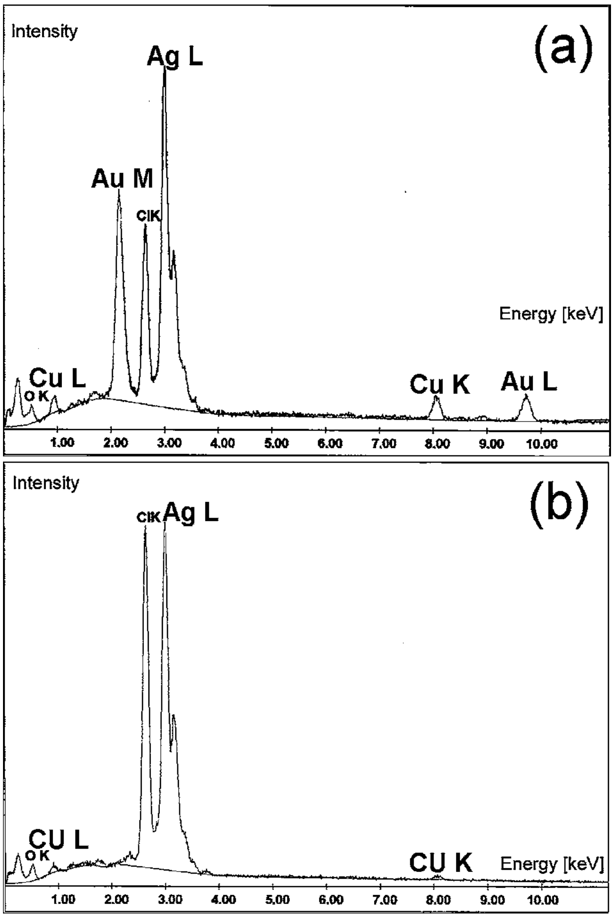

Figure 4 EDX micrographs of sample 46 F6 of group 1 (electron energy [keV] versus intensity): (a) top of coil, and (b) underside of coil.

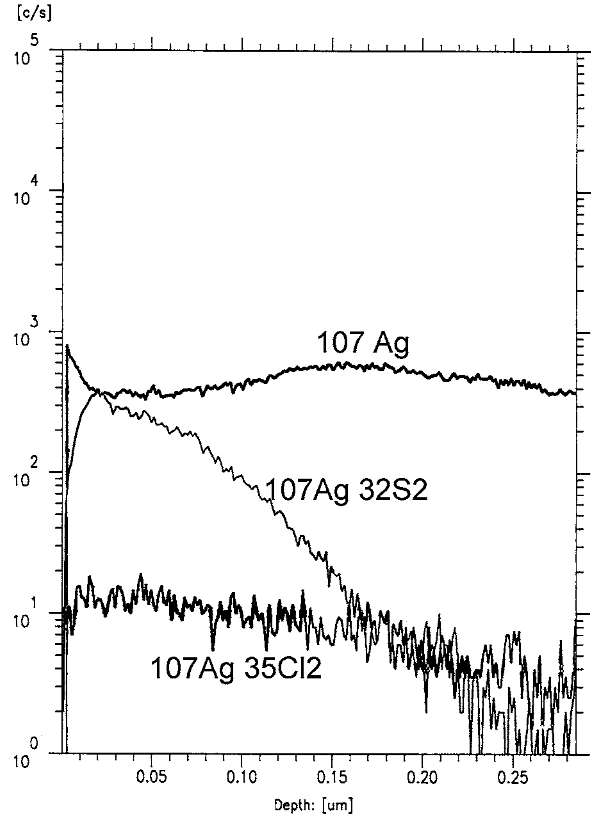

Figure 5 SIMS negative ion depth profile of sample 150 B3 of group 4.

original alloy. EDX micrographs of the unexposed underside and exposed top of a group I sample indicate higher levels of chlorine on the underside and the absence of gold, Fig. 4.

The intensities for chlorine and sulphur coincide with the degree of corrosion and the silver corrosion products are attributed to mainly sulphur and chlorine containing compounds [33].

3.3. SIMS

For all the samples studied the mass spectra and the depth profiles indicate silver chloride and silver sulphide are the major sources of corrosion tarnishing on the metal surface, with the chloride species appearing to be predominant. The mass spectra also revealed high intensities for chlorine species containing sodium and potassium. These could originate from handling or other contamination sources and may possibly act as a chlorine source for the formation of silver tarnish. Sulphur sources may include atmospheric pollution and probably the breakdown of cystine amino acids in the adjacent wool fibres due to long term photodegradation and thermal degradation [34, 35].

No gold was present in the mass spectrum of the surface of a group 4 sample. The negative ion depth profile of the dynamic SIMS analysis of a heavily corroded area on this sample suggests the absence of elemental silver, 107Ag, on the immediate surface; but in the first few nanometers of the subsurface the concentration of silver chloride, 107Ag $35Cl_2$, and silver sulphide, 107Ag $32S_2$, decrease while the elemental silver apparently increases, Fig. 5.

Mass spectra and positive ion depth profiles of the top and underside of group 1 and group 3 metal strips indicate a thin (~40 nm) gold layer, 133Cs 197Au, over silver, 133Cs 107Ag, alloyed with copper, 133Cs 63Cu, and possibly gold 133Cs 197Au, Figs 6 and 7. Silver and copper are also detected on the surface, either due to a damaged gold layer or the presence of alloys or due to a diffusive migration process of metal ions as identified previously by Howell and Jaro [34, 36]. The intensities of contaminations such as potassium, sodium and chlorine, possibly originating from handling of the silver before the gilding process, reach a second maximum at the interface between the silver and gold layers, Fig. 6.

To achieve comparative semi-quantitative data, the intensities for gold and copper were normalized by dividing with the silver bulk intensity, Table III. The substantial decrease in the normalized intensities of Au at various approximate depths gives evidence for a surface gilding of the topside but not the underside and is a possible indication of Au migration into the silver bulk. Variations in the Cu intensities are less obvious and most likely due to the formation of corrosion compounds.

Based on the natural elemental isotopic abundances and assuming the same ionization probabilities for $CsAu^+$, $CsAg^+$ and $CsCu^+$ the elemental concentrations for gold and copper in the silver bulk at 250 to 500 nm depth were calculated as 0.01–0.5% Au and 9–30% Cu.

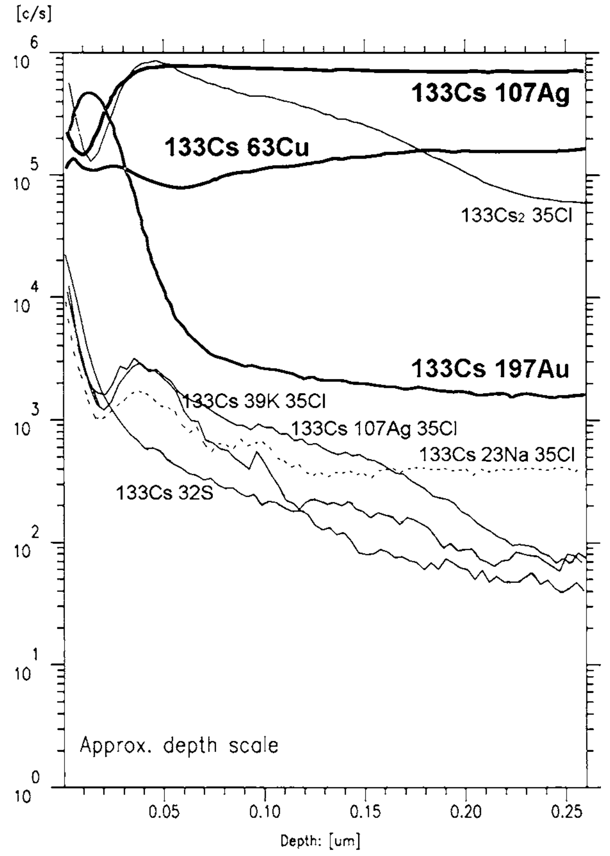

Figure 6 SIMS positive ion depth profile of the topside of sample 68 E6 of group 3.

The presence of alloyed or migratory gold in the silver bulk was also confirmed in negative ion mode, which typically offers higher relative sensitivity for gold [37]. The absence of a gold layer on the underside of the metal strips suggests the use of the 'beaten and cut' manufacturing method. Further work on the nature of Renaissance metal threads is currently ongoing.

3.4. UVO Cleaning of tarnished silver foil

Extended UV/Ozone irradiation of the tarnished silver foil, approximately 15 min, apparently removed the visible corrosion on the exposed surface. The unexposed side remained tarnished but subsequent UVO treatment similarly cleaned the other surface. However, after ceasing the UVO exposure a light brown tarnish reappeared but was easily removed with tissue paper and gentle rubbing. The sample was subsequently left for six weeks and no further tarnishing reappeared.

Dynamic SIMS of the tarnished foil surface indicated again the presence of silver sulphide and also to a lesser extent silver chloride. This was in contrast to the historical metal threads where their relative intensities were reversed and essentially reflects the sulphur rich atmosphere in the Oddy test. The corrosion was present to a depth of three to five nanometers. After the UVO treatment both the impurity levels obviously decreased but with the sulphide species decreasing to a far greater extent, Figs 8,9, Table IV. The high intensity of AgCl on the cleaned surface is probably due to manual handling.

The cleaning mechanism of UVO treatment is based partly on the formation of ozone and atomic oxygen.

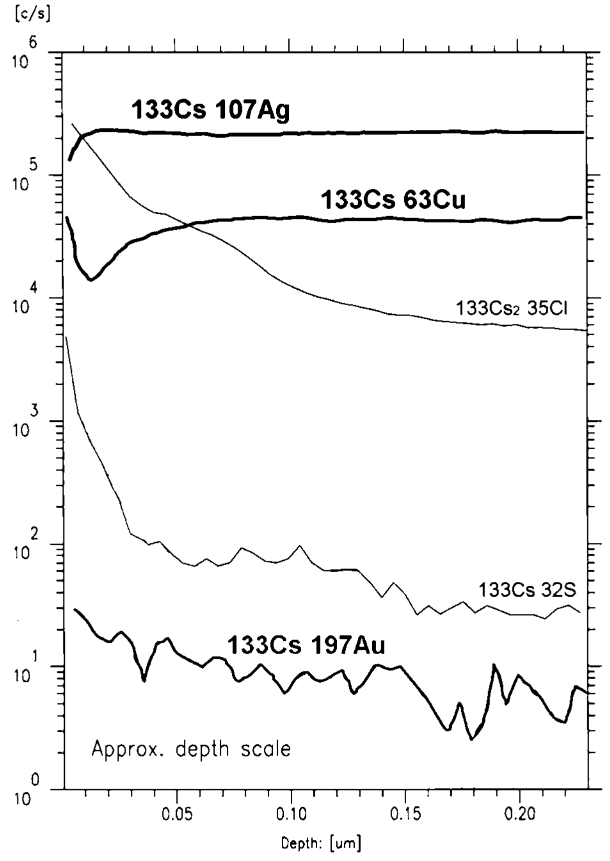

Figure 7 SIMS positive ion depth profile of the underside of sample 68 E6 of group 3.

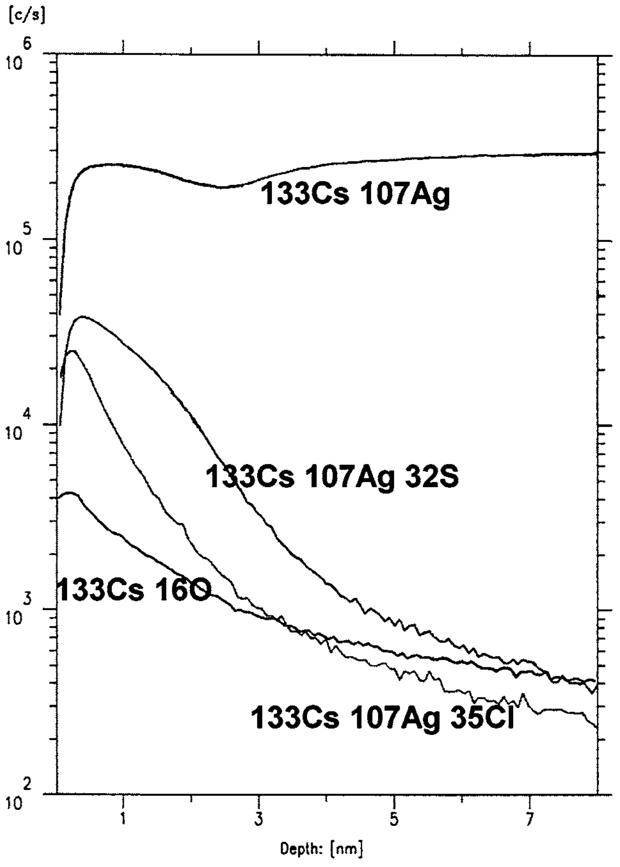

Figure 8 SIMS positive ion depth profile of tarnished silver foil.

TABLE III Normalized gold and copper intensities at different approximate depths

	Depth (nm)	Group 3, sample 68 E6			Group 1, 149 B6	Group 1, sample 120 C3	
		Topside 1	Topside 2	Underside	Topside	Topside	Underside
Au/Ag	20	0.686	0.5	0.00009	0.059	0.17	0.062
	250	0.002	0.0006	0.00003	0.0036	0.013	0.011
	500[a]	–	0.0002	–	–	0.003	–
Cu/Ag	20	0.143	0.26	0.071	0.1	0.097	0.35
	250	0.243	0.25	0.214	0.143	0.097	0.5
	500[a]	–	0.305	–	–	0.187	–

[a]Samples with '–' were only analysed to a depth of 250 nm.

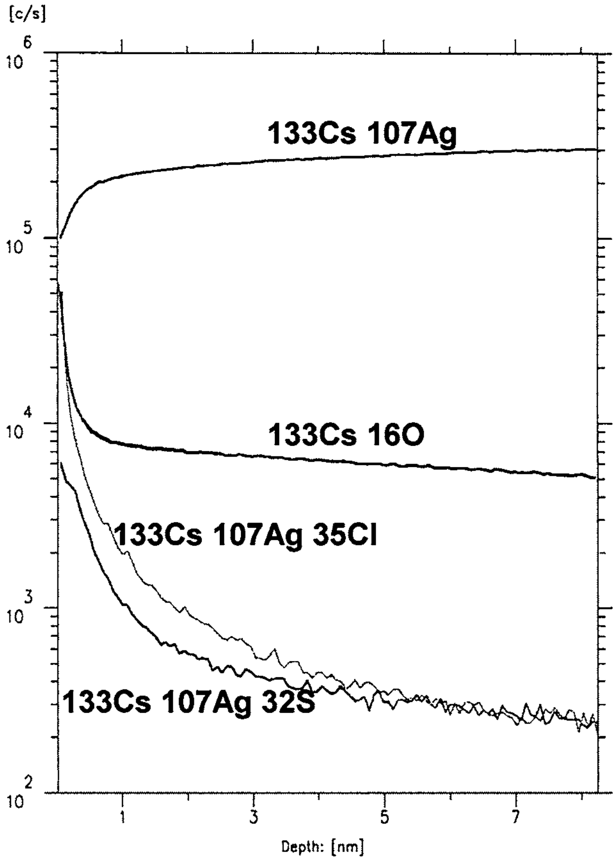

Figure 9 SIMS positive ion depth profile of cleaned silver foil.

TABLE IV Normalized intensities of silver sulphide, silver chloride and oxygen at different approximate depths

	Depth [nm]	Tarnished silver foil	Cleaned silver foil
AgS/Ag	0	0.06	0.02
	1	0.127	0.003
	3	0.011	0.001
	8	0.001	0.0008
AgCl/Ag	0	0.033	0.193
	1	0.08	0.007
	3	0.003	0.001
	8	0.0008	0.0008
O/Ag	0	0.01	0.17
	1	0.011	0.025
	3	0.003	0.023
	8	0.001	0.017

The latter will react quickly with contaminant sulphide and chloride to form potentially volatile oxidised species, SO$_2$ and ClO$_2$, while ozone will react with silver to form a black silver oxide [38]. However this is not long lived due to conversion to silver under the influence of light [39]. According to a study by Czanderna [40] silver does not form a significant amount of oxide and at most one monolayer of oxygen atoms adhere to the silver surface.Jaro proposed that a silver oxide layer on the silver surface acts corrosion protective by completing the structure of the metal lattice and leaving few unsaturated valencies free for attack from sulphur or chlorine ions [24]. The presence of silver oxide or a layer of oxygen containing species on the immediate surface of the cleaned silver foil is indicated by the high initial intensity of oxygen, 133Cs 16O, Fig. 9, Table IV.

Previous studies on the effect of UV/Ozone irradiation on wool and silk have reported the loss of surface lipids, fibre oxidation and obvious yellowing [27, 41]. In addition several studies have also demonstrated that the short wavelength UV light has a degradative effect on textile fibres and dyes [42, 43]. It is therefore questionable whether the UV/Ozone cleaning method, using the currently available exposure system, could be used on metal threads in textiles in isolation, but rather may be applicable to discrete metal artefacts.

4. Conclusions

Metal threads from a priceless Renaissance tapestry were found to typically consist of metal strips wound around a silk core. However variations in material content and dimensional structure indicate that a range of distinct metal threads had been incorporated into the tapestry and derived from different manufacturing routes. Highly unusual samples with two layers of metal wrappings were also identified, with their presence and location in the tapestry indicating the designers' desire for enhancing impression. SEM and SIMS analyses suggested the two general manufacturing methods 'beaten and cut' and 'cast, drawn and rolled' for metal threads, again indicating some variability in the processing. EDX and SIMS analyses verified that most metal strips were gilt silver, although some consisted of only silver with low levels of copper. The main surface corrosion products originate from reactions of sulphur and chlorine compounds with silver.

The potential for UV/Ozone treatments to clean silver has been investigated and after two months no re-tarnishing had occurred. SIMS analyses of the cleaned silver indicated that the concentration of the contamination products was significantly lower than that on the tarnished silver foil. This technique could provide an alternative cleaning method for discrete ancient metal artefacts, which are often left in their corroded or tarnished state because of the detrimental effect of conventional mechanical and chemical cleaning methods. However, metal threads in textiles will still present a problem because the irradiation with UV light and the exposure to ozone will have significant degradative effects on wool and silk fibres.

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