Non-destructive Analysis of Byzantine Gold-Leaf Glass Tesserae Using Ion Beam Analysis

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Abstract. During the byzantine period gold-leaf glass tesserae were used in abundance for the creation of wall mosaics for the adornment of Byzantine churches. Metal-leaf glass tessera is a sandwich structure of two layers of glass (the support and the top glass) enclosing a metal leaf and its characterisation necessitates a more sophisticated approach compared to other glass tesserae. Studies of gold-leaf glass tesserae have been based on destructive analysis (cross-section analysis), however cultural heritage objects address for a different approach. The technical progress and accessibility to more elaborate techniques could facilitate a non-destructive research. The aim of this work is to present the potentials of a non-destructive and non-invasive methodology for the analysis of byzantine gold-leaf glass tesserae using ion beam techniques. The study of gold-leaf glass tesserae was carried out at the Byzantine Monastery of Daphni (11th century) Greece. The research combined systematic examination of tesserae and analysis of selected samples. Tesserae were analysed as received, using mainly Scanning Electron microscopy coupled with Energy Dispersive x-ray Spectroscopy (SEM/EDS) and Ion beam techniques. In the context of this contribution the results will be presented to demonstrate the efficiency of the selected methodology. μ-PIXE/PIGE analysis provided elemental analysis for both the glass layers and via elemental maps combined with photographic recording of analysed areas, direct information for technological features.

Keywords: Gold-leaf glass tessera · Ion beam analysis · Non-destructive

1 Introduction

The use of metal-leaf (gold/silver or alloy) glass tesserae, begun in the roman times and was established during the byzantine period. Gold-leaf tessera is a composite object of glass and metal, as a gold leaf is enclosed between two layers of normally transparent glass and it can be defined in terms of the physical characteristics of the two glass layers (cartellina/top and the support) and the gold leaf. Tessera colour is given by the shade of the support glass as the layer of cartellina is usually very thin (less than 1 mm).

Limited published data is available for gold-leaf glass tesserae and particularly their nature and decay [13, 14] thus, the research focused on technological issues and the corrosion of the tesserae [6–9].

The tesserae studied derived from the wall mosaics of the Byzantine Monastery of Daphni (dated to the 11th century) Athens, Greece, a monument included in the UNESCO World Heritage List. Tesserae were classified according to technological features such as glass colour, shape and defects [as well as to condition and alteration phenomena. The glass appeared either slightly coloured in natural hues or with a stronger shade, while no true colourless gold-leaf glass tesserae were detected. The tesserae according to the hue of the support glass were classified as yellowish, aqua-green and roughly purple [6, 9]. Optical examination could not easily record the colour of the top/cartellina mainly due to the small thickness of the top glass (average thickness 0.2 to 0.3 mm). Cartellina was usually more transparent and colourless compared to the support and appeared to be made of glass with a light aqua or purple hue [6, 7, 9].

Furthermore, tesserae were classified from unaltered or slightly altered to heavily altered while tesserae where only the support glass was preserved, due to the loss of cartellina and the metal leaf, were classified in a separate group [6, 7]. Macroscopically, the surface of the altered tesserae exhibited according to condition, a dull or iridescent surface, a whitish surface (milky) with opalescence, as well as dark discolouration in the form of brown-black areas or layers. The most distinctive phenomenon was the alteration of the gold tesserae surface into a greyish-silvery colour resembling that of a silver-leaf tessera. Microscopic examination of these tesserae revealed that the top glass exhibited opaque layers with strong shine (greyish layers) near the interface and in contact with the gold leaf a dark layer (beneath and occasionally on top of the leaf) was present [6–8].

Analysis of glass tesserae is typically carried out destructively (on cross-section), while the less optically invasive option of cutting a small piece from the base of the tessera (usually embedded in the mosaic's mortal), cannot be applied for metal-leaf tessera when analysis of both glass layers and the metal leaf is aimed. However, cultural heritage objects preservation necessitates a different approach and currently the need for less invasive research methodologies is often discussed.

In recent years, an increased number of publications present the use of nuclear techniques for the analysis of cultural heritage. Accessibility to ion beam analysis and technical progress facilitate non-destructive and non-invasive research. Techniques like PIXE/PIGE, RBS and NRA, earlier inaccessible, are now often applied for the analysis of artefacts and works of art. The ability to scan a very small area combined with the facility of a digital camera provides compositional information on microscopic scale, without sampling. PIXE and PIGE techniques that offer rapid and accurate elemental analysis have already been applied without vacuum using an external beam setup [2].

Current research used micro-Proton Induced X-ray and γ-ray Emission Spectrometry (μ-PIXE/PIGE) for the analysis of the tesserae as received, combined with a systematic microscopic examination prior to analysis. The study provided promising results for a non-destructive and non-invasive analysis of metal-leaf glass tesserae.

According to the authors best knowledge, this was the first attempt to analyse in this way gold-glass tesserae using ion beam analysis.

2   Experimental

The research of the gold-leaf tesserae incorporated the macroscopic examination of tesserae detached and in situ, a systematic microscopic examination and the analysis of thirty-six selected tesserae using Scanning Electron Microscopy coupled with energy dispersive x-ray spectroscopy and supplementary micro-Proton Induced X-ray and γ-ray Emission Spectrometry (μ-PIXE/PIGE). The selected tesserae comprised well preserved and decayed ones, with different state of preservation. Some well-preserved tesserae derived from known mosaic depiction, while all the decayed were detached tesserae of unknown provenance. Decayed tesserae exhibited different corrosion phenomena along with variations in surface condition, facilitating the study of the altered areas from deeper levels, without sample preparation.

The opportunity to have access to μ-PIXE/PIGE facility was obtained by the European Commission, through the Consortium CHARISMA-Fixlab and was performed at the Scanning Nuclear Microprobe installed at the 5 MV Van de Graaff electrostatic accelerator of the Institute of Nuclear Research of the Hungarian Academy of Sciences (ATOMKI-HAS) in Debrecen, Hungary. In the available beam time obtained by CHARISMA-Fixlab (5 days), seven well preserved and three decayed tesserae were analysed at proton energies of 2 and 3.5 MeV.

The main goal of μ-PIXE/PIGE analysis was to determine the composition of both glass layers from selected gold-leaf tesserae (well-preserved and mainly of known origin) and assist the corrosion study with accurate analysis of the altered areas.

The micro-PIXE experimental arrangement and the data acquisition system have been described in publications [3, 4, 11]. As a first step, the major elements were measured with H+ beam of 2.0 MeV energy supplied by the 5 MV Van de Graaff electrostatic accelerator of ATOMKI. The second step was the measurement of the trace elements with increased sensitivity applying H+ beam of 3.5 MeV. In both cases the beam was focused to a spot size of approximately 2×2 μm^2 by the Scanning Nuclear Microprobe. The accumulated charges at the samples were typically ~ 0.1 μC and ~ 0.2 μC for the analysis of the major and trace elements, respectively, at typical beam currents of ~ 150 pA. Measurements were carried out on each sample scanning the beam across $\sim 100 \times 100 - 1000 \times 1000$ μm^2 areas.

The experimental setup consisted of a Gresham-type Si(Li) detector with an area of 30 mm2 and a Be-window of 12.5 mm thickness having a resolution of 136 eV (at 5.9 keV). It was placed at 135º with respect to the direction of the beam. The backscattered protons from the samples were stopped using a 50 mm Be-foil and 250 mm kapton foil at 2 and 3.5 MeV energies, respectively. The X-ray spectra were analysed with the PIXEKLM-TPI program [12] normalizing the oxide composition of the sample to 100%. The PIGE experimental setup (used at 3.5 MeV proton beam energy) consisted of a Canberra HPGe gamma-ray detector with 40% relative efficiency which was placed at 45° with respect to the direction of the beam and at a distance of 11 cm from the sample, in contact with the scattering chamber.

The tesserae were analysed as received, folded in aluminium foil prior to be fixed on the sample holder.

μ-PIXE/PIGE analysis apart from compositional data, provided element distribution maps and photographic recording of the analysed areas, that facilitated immediate recognition of technological and decay features.

3   Results and Discussion

3.1   Glass Composition

The chemical composition of the tesserae as obtained by PIXE/PIGE is shown in Table 1. Major and minor elements are expressed as concentration percentages of oxides or elements. Most of the tesserae were well preserved, so the composition of the top/cartellina and the support glass are given separately (identified as top and sup respectively), along with the analysis of both layers marked as map.

The basic composition is a soda-lime-silicate glass of the so called, mixed natron-plant ash glass type, as both sodium and potassium are detected. This type of glass is considered to be the result of a transitional technological phase, when the use of plant ash and natron glass was carried out simultaneously [1].

A small percentage of manganese and iron in the glass give the colour variations observed. The greenish colour due to iron impurities is partially neutralised by the addition of manganese acting as a decolourant. The intentional addition of manganese aimed to obtain a colourless glass, while a higher percentage was responsible for the purple hue on tesserae B5 and C12 (MnO 2.67% and 1.79% respectively). Likewise, the colour of the aqua and greenish tesserae (B2 και AH2) could be attributed to a slightly higher content of iron in conjunction with a lower content in manganese. A higher content of manganese was detected also at the yellowish top glass of sample B3 (MnO 2.2%) indicating that possibly the colour optically observed was misleading due to the small thickness of the glass layer (0.13 mm).

Small variations in the content of manganese and iron were detected for glass layers with dissimilar appearance i.e. the top glass (light purple) of AH2 tessera exhibit a manganese content of around MnO 2.0% and iron (FeO 0.76%), while the support glass of B4 tessera (yellowish with light purple strikes) MnO 1.87% and FeO 0.7%, and the purple support of C12 tessera MnO 1.79% and FeO 0.64%. Colour variations in glasses (with a stronger shade) of similar manganese and iron concentrations is a feature recently indicated by other workers [10] and attributed to the fact that the glass colour is mainly determined by the production's conditions (atmosphere and time).

Previous study of the glass tesserae from the Monastery of Daphni and Osios Loukas [1] characterised them as typical medieval glasses. The tesserae analysed were mainly of coloured opaque glass, however a limited number of gold-leaf glass tesserae (four samples) were also included. The results of PIXE/PIGE analysis will be further discussed in connection to the above-mentioned publication, as it is the most systematic study of 10–11th century glass tesserae from Greece and the Daphni Monastery in particular. Arletti *et al.* have used destructive analytical techniques, Inductively Coupled Plasma Optical Emission Spectroscopy and Electron Microprobe [1].

Arletti *et al.* presented an average composition of the samples from the Daphni after the exclusion of the 'non-original' tesserae, the ones not belonging to the medieval

glass production. The average composition of the main oxides as detected by PIXE/PIGE present a small deviation for the content of silicon, aluminium, magnesium and sodium from the published data [1] (Table 2). However, the average composition of gold-leaf glass tesserae as estimated by their results [1] (samples D11, D52_2, D54_2 and D57_2) presents a better correlation with the PIXE/PIGE analysis results. The main deviation between the two studies is the content of magnesium (MgO 2.57% for 1.88%), that still stays in the standard deviation values defined by the Italian researchers (2.02% with standard deviation 0.46%) (Table 2).

The most irregular composition detected was of the top glass of B3 tessera, for this reason was excluded from the calculations of the average composition. The composition of the top glass shown deviated values for most of the elements (aluminium, calcium, phosphorous, potassium, manganese, iron and chloride) and presented a high gold content. However, as this was only observed for the top glass the tessera was considered to be an original one. PIXE results due to the way of analysis (scanning of a selected area) in some cases collected also data from the gold leaf, for this reason the analysis B2-map2 that gave a higher content in Au (2.05%) was also excluded (Fig. 1).
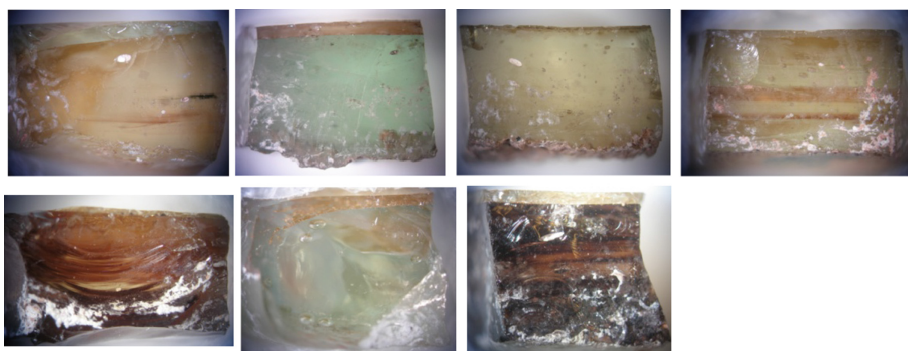
Fig. 1. Side view of the analysed well preserved tesserae

Arletti et al. categorised the tesserae as lead free, when the percentage of lead in the glass was less than (PbO 0.2%). It is interesting to note that both greenish tesserae (B2 και AH2) exhibit a percentage of lead (Pb 0.2%), coupled with the exact percentage of copper (Cu 0.03%) that was the higher detected in the analysed tesserae.

Element Distribution Maps

Element maps acquisition from the side of the tesserae provided the difference in composition between the two glass layers, along with information about the gold leaf. The gold used to produce the leaf, was either pure metal or gold alloy with silver, while a twin line for gold map indicated perhaps the multiple use of the metal leaf [5] (a feature verified with SEM analysis) [8]. Regrettably, the way of analysis carried out by μ-PIXE, did not permit the detection of the exact composition for the metal leaf (elements from the glass were also collected). However, the presence of silver in the gold leaf was verified at least at four tesserae.

Table 1. Compositional analysis of well-preserved tesserae

| Number	Colour	Na₂O	MgO	Al₂O₃	SiO₂	P₂O₅	S	Cl	K₂O	CaO	TiO₂	MnO	FeO	Cu	Sr	Ag	Ba	Au	Pb
B1-sup	Yellowish	12.60	1.50	2.12	70.15	0.17	0.07	1.12	2.60	7.01	0.14	1.67	0.60	0.002	0.07	—	0.06	0.0002	0.0004
B1-sup		14.00	1.50	2.09	70.01	0.22	0.08	1.14	2.57	7.11	0.14	1.63	0.68	0.002	0.09	0.0006	0.042	—	0.0008
B1-top	Greenish	12.81	1.44	2.40	70.26	0.38	0.21	1.16	2.7	6.39	0.20	1.26	0.74	0.003	0.035	0	0	0	0.0005
B1-map		12.80	1.73	2.32	69.62	0.55	0.24	1.15	2.47	6.29	0.14	1.40	0.61	0.0025	0.067	0.0036	0.040	0.625	0.0003
B2-sup	Aqua	14.3	2.15	1.91	65.46	0.25	0.14	0.83	2.72	8.936	0.12	1.32	0.78	0.03	0.066	0.003	0.061	—	0.21
B2-sup		14.4	1.86	2.10	66.69	0.20	0.14	0.79	2.55	8.25	0.11	1.22	0.71	0.03	0.062	0.005	0.049	0.0005	0.20
B2-top	Yellowish	15.1	2.16	1.90	67.46	0.48	0.20	0.89	2.49	7.32	0.11	1.31	0.51	0.0016	0.051	0.009	0.045	0.001	0.0007
B2-map		14.60	2.06	1.83	66.96	0.46	0.20	0.89	2.60	7.33	0.11	1.13	0.59	0.040	0.056	0.043	0.048	0.52	0.11
B2-map2		14.00	2.04	1.79	65.73	0.47	0.22	0.88	2.37	7.34	0.13	1.03	0.59	0.016	0.040	0.11	0.034	2.05	0.10
B3-sup	Yellow/greenish	13.10	2.23	1.93	69.69	0.53	0.22	0.99	2.61	6.64	0.12	0.85	0.53	0.0015	0.049	—	0.036	0.0002	0.0008
B3-sup		14.00	2.23	1.93	69.58	0.53	0.22	0.98	2.64	6.61	0.11	0.91	0.59	0.0021	0.065	0.0046	0.04	—	—
B3-sup		14.80	2.23	1.93	69.69	0.53	0.22	0.99	2.58	6.63	0.11	0.87	0.54	0.002	0.05	0.0007	0.04	0.0008	0.0008
B3-top	Yellowish	13.70	1.02	4.10	57.76	1.22	0.67	1.36	3.79	12.96	0.21	2.22	0.94	0.003	0.081	0.02	0.09	0.0014	0.0006
B3-top		13.20	0.98	3.97	55.80	1.16	0.66	1.35	3.99	12.91	0.20	2.19	1.02	0.005	0.08	0.03	0.088	2.025	—
B3-map		13.60	2.21	1.92	69.06	0.52	0.22	0.99	2.31	6.62	0.10	0.98	0.50	0.0016	0.040	0.023	0.053	0.87	—
B4-sup	Yellowish with purple strikes	14.40	1.01	2.02	69.23	0.29	0.09	1.08	2.66	7.16	0.14	1.87	0.71	0.003	0.097	0.0055	0.048	—	0.0004
B4-top		14.02	1.03	1.92	69.21	0.45	0.14	1.06	2.69	7.26	0.13	1.35	0.67	0.0026	0.107	0.0006	0.05	0	0
B4-map		14.40	1.54	1.97	68.09	0.55	0.19	0.94	2.51	6.81	0.13	1.37	0.66	0.003	0.108	0.013	0.028	1.176	0.0013
B5-sup	Purple	12.54	1.62	2.09	68.75	0.51	0.14	1.01	2.69	7.09	0.13	2.67	0.67	0.0028	0.099	0	0.074	0	0.0009
B5-top	Yellowish	12.10	1.68	2.13	68.35	0.59	0.21	0.99	2.77	8.50	0.14	1.36	0.66	0.0024	0.095	0.009	0.057	0.0005	0.0011
AH2top	Light purple	13.80	2.02	1.85	66.55	0.47	0.18	0.93	2.61	7.67	0.12	1.90	0.76	0.009	0.15	0.036	0.06	1.32	0.02
AH2top		14.00	1.97	1.81	67.34	0.50	0.19	0.91	2.73	7.97	0.12	2.05	0.79	0.005	0.18	0.020	0.08	—	0.003
AH2-sup	Aqua green	13.00	2.09	1.96	68.52	0.50	0.18	0.85	2.41	7.77	0.10	1.18	0.70	0.03	0.06	—	0.04	0.0003	0.20
AH2map		14.10	2.14	1.94	68.21	0.47	0.16	0.96	2.38	7.47	0.10	1.30	0.67	0.025	0.067	0.015	0.036	0.050	0.14
G12-top	Yellowish	13.40	2.80	2.13	69.11	0.21	0.26	1.09	2.93	7.45	0.13	0.89	0.55	0.002	0.045	—	0.04	0.0002	—
G12-sup	Purple	11.90	1.77	2.04	69.47	0.23	0.13	1.01	2.59	7.72	0.12	1.79	0.64	0.019	0.05	0.0006	0.05	0.001	0.02
G12map		12.70	2.07	1.97	69.49	0.21	0.13	1.11	2.50	6.83	0.11	1.17	0.54	0.008	0.04	0.07	0.05	1.34	0.005

Table 2. Average composition of the samples from the Daphni and the gold-leaf glass tesserae extruded from Arletti *et al.* [1] presented in conjunction with PIXE/PIGE average for gold-leaf glass tesserae.

	Average Arletti *et al.* 2010	St. dev Arletti *et al. 2010*	Average for gold-leaf estimated from Arletti *et al. 2010*	Average PIXE/PIGE Analysis of gold-leaf tesserae current study[a]
SiO_2	69.81	3.67	69.34	68.70
TiO_2	0.10	0.03	0.13	0.12
Al_2O_3	1.90	0.32	1.99	2.02
MgO	2.02	0.46	2.57	1.88
CaO	7.52	1.01	7.60	7.31
Na_2O	11.19	2.06	13.39	13.60
K_2O	2.30	0.38	2.48	2.60
P_2O_5	0.27	0.08	0.34	0.41
S	0.09	0.04	0.12	0.17
Cl	0.68	0.17	0.88	0.99

[a] Excluded the analyses of top glass from B3 tessera and the B2-map2.

Element distribution maps are presented as relative colour maps ranging from low concentration (dark blue) to high concentration (bright red). Selected examples of element distribution maps would be presented and discussed.

C12 Tessera. The tessera was categorised as purple due to the shade of the support glass, while the top glass appeared less colourless with a yellowish hue. The colour of the support was not homogeneous due to strikes of deeper purple hue. Element distribution maps of the support glass revealed zones with higher concentration of manganese along with iron, corresponding perhaps to the strikes optically observed. Maps collected from the side of the tesserae at the interface of both glass layers, showed for the support glass a higher concentration in manganese combined with higher content in copper and iron (verified by elemental analysis) (Fig. 2).
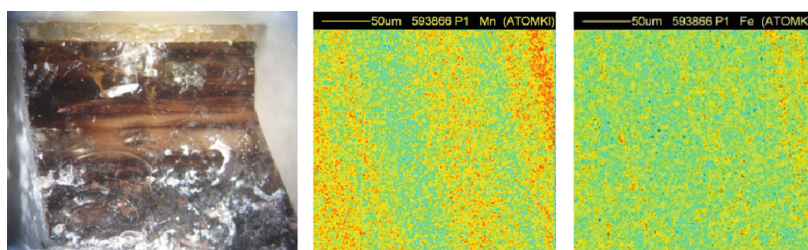

Fig. 2. C12 tessera -Side view of tessera, area of analysis on support glass and elemental map for manganese (Analysed area 250×250 μm^2 on 3.5 MeV).
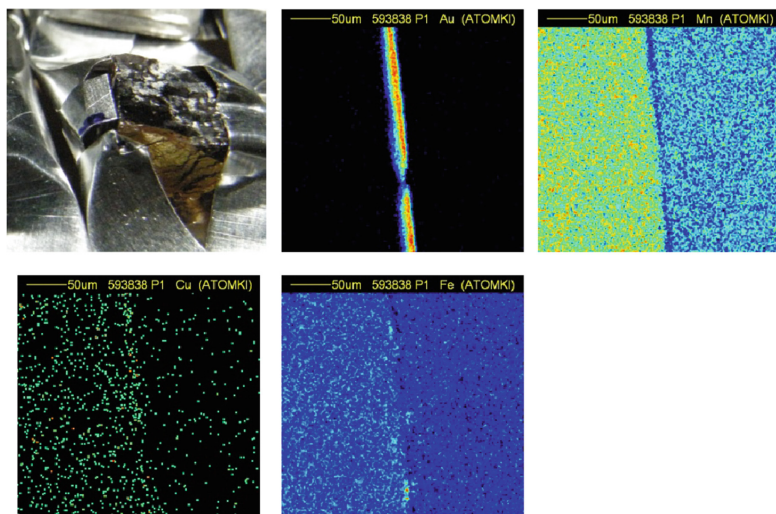
Fig. 3. Element map acquisition from the side of C12 tessera. The tessera on the sample holder and elemental maps for gold, manganese, copper and iron at the interface (Analysed area $300 \times 300 \ \mu m^2$ on 3.5 MeV)

AH2 Tessera. The colour of the support glass was light aqua greenish and of the top glass light purple. Maps collected from the side of the tesserae indicated a higher content in manganese in the top glass (Fig. 3).
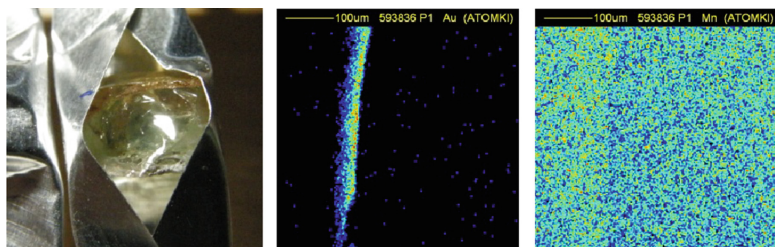
Fig. 4. AH2 tessera on the sample holder and element maps for gold and manganese (Analysed area $500 \times 500 \ \mu m^2$ at 3.5 MeV).

B2 Tessera. The colour of the support glass was aqua while the top glass appeared more colourless. Maps collected from the side of the tesserae indicated for the support glass a higher content in iron and calcium, along with the presence of lead. Furthermore, the occurrence of silver was revealed in the gold leaf.
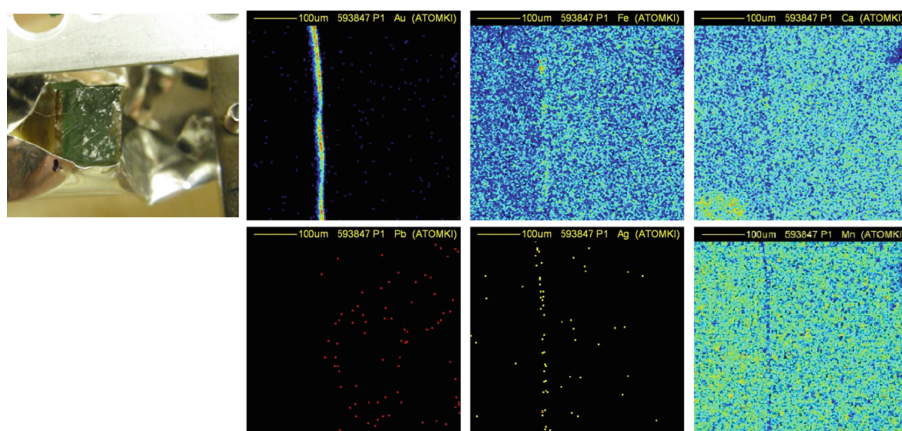
Fig. 5. B2 tessera on the sample holder and elemental maps for gold, iron, calcium, lead and silver (3.5 MeV).

3.2   Decay

Scanning of selected areas revealed interesting data via element distribution maps of the altered glass. Analysis was focused on areas of the top surface and at the interface with the gold leaf (sides of tesserae) exhibiting the silvering effect or a dark discoloration. In addition, elemental analysis of the exposed surface of the top glass and the altered areas in contact with the gold leaf was carried out (Fig. 4).

Tessera C9. A purple tessera characterised as heavily altered exhibiting a silvering surface of top glass. Microscopic examination of the sides of the tessera facilitated the study of the glass/gold interface and revealed broad altered zones of the glass with dark discoloration. Element maps acquisition from the side of the tesserae, revealed next to the gold leaf an altered zone on both glass layers. The altered glass zone was depleted in sodium and was enriched in manganese and iron [8]. At the same time, areas enriched in calcium and titanium where detected near the gold, while generally potassium was not depleted from the altered zone. Furthermore, the occurrence of silver was revealed in the gold leaf (Fig. 5).

Element maps acquisition from the top surface of the same tesserae (near the edge), where decayed glass (whitish with silvering shine), gold leaf and black layer beneath the gold were exposed due to damage, revealed also a strong enrichment in manganese and iron in both the altered top glass and the black areas. Manganese and iron exhibited a correlate strong enrichment in specific areas (indicated with orange-red coloration), while the combined enrichment in titanium is also revealed along with the presence of silver in the gold leaf (Fig. 6).

Tessera D6. A heavily altered aqua tessera with partly exposed silvering layers of the top glass, the gold leaf and dark layer beneath the gold (support glass). The tessera was scanned both at 3.5 MeV and 2 MeV. At 3.5 MeV scan a strong enrichment in manganese and iron was detected both in the altered silvering layers of the top glass
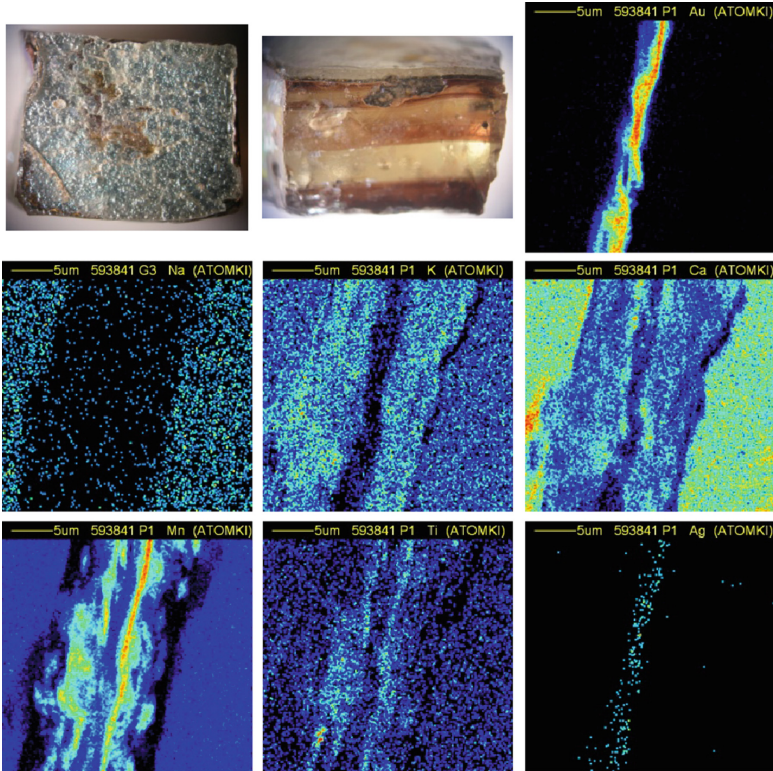
Fig. 6. C9 tessera – Top and side general view and element maps for gold, sodium, potassium, calcium, manganese, titanium and silver.
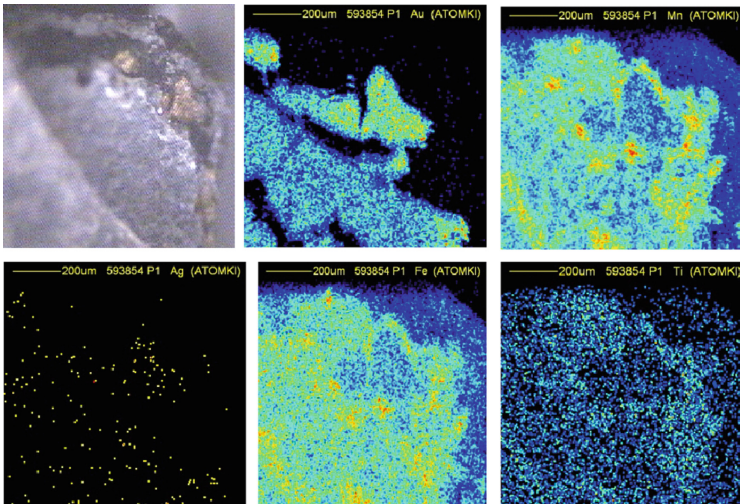
Fig. 7. C9 tessera – General view of analysed area and element maps for gold, manganese, silver, iron and titanium.

and the dark layer beneath the gold. In addition, enrichment in calcium was revealed at the support glass and the presence of strontium in the gold. Elemental distribution map at 2 MeV, that comprised silicon, revealed a higher content of silicon in the silvering top glass (Fig. 7).

The elemental analysis of the altered glass verified a strong enrichment in manganese and iron, both for areas exhibiting a dark discoloration and silvering layers, characterised them as mn-rich areas [8]. The altered areas of glass exhibited also enrichment in potassium or/and calcium, although is expected to be depleted due to the corrosion mechanism. A feature generally observed for the rest of the decayed tesserae analysed only with SEM/EDS. On the other hand, the relative enrichment in titanium in these areas is a feature clearly revealed by PIXE/PIGE analysis. Furthermore, an interesting difference between the altered areas of tessera D6 (silvering layers and the dark layer) was detected. Although both elements were enriched, the content of potassium (K_2O 10.44%) is higher in the silvering layers than in the dark layer (K_2O 5.5%) and the opposite is happening for calcium (CaO 10.06% and 4.6% respectively) (Fig. 8).
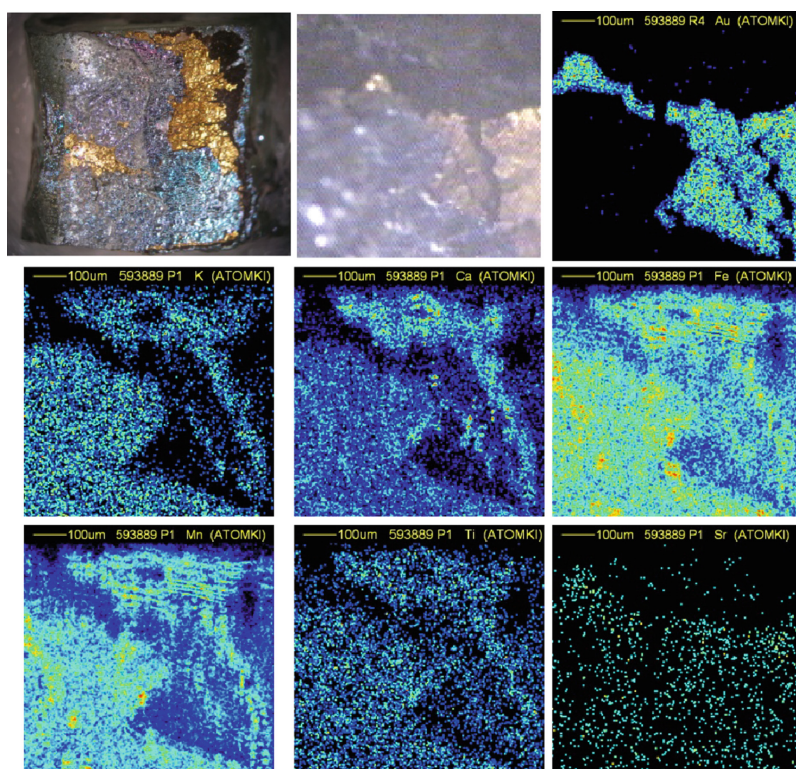
Fig. 8. D6 tessera – Top view of tessera and the analysed area and elemental map for gold, potassium, calcium, iron, manganese, titanium and strontium.

4   Conclusions

The study demonstrated the potential use of μ-PIXE and PIGE techniques as an analytical tool for the analysis of gold-leaf tesserae technology and decay, without prior sample preparation. Selected microscopic areas were analysed with the aid of a proton beam of very small size (2×2 μm^2) monitored with a digital camera.

The compositional data of glass on well preserved tesserae provided was in good agreement with previous results based on destructive analysis. Element distribution maps provided data for compositional difference between the two glass layers of the tesserae and the gold leaf, along with an initial evidence of the stratigraphy of altered areas prior to SEM/EDS analysis. In addition, μ-PIXE and PIGE revealed the presence of high manganese and iron content in the decayed areas studied and assisted in further differentiation among them.

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