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EVIDENCE OF AU – HG GILDING PROCESS IN POST BYZANTINE ECCLESIASTICAL SILVERWARES (CHALICES) OF EASTERN THESSALY BY PXRF

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ABSTRACT

Silver and gold alloys have been widely applied in the making of precious objects during the byzantine and post byzantine eras. A major category is the "ecclesiastical silver", which was used in the celebration of the liturgy (chalices, disks, asterisks, blessing and benediction crosses, processional crosses, liturgical fans, wedding crowns etc.). This study focuses on post-byzantine ecclesiastical silver chalices, kept in parish churches and monasteries of Eastern Thessaly. They have been studied with non-destructive analysis technique (XRF) for the clarification of the role of gold (Au) in the Silver – Copper – Gold alloy. The main question answered in this work is whether gold (Au) was part of the compositional alloy or decorative and moreover if it was applied with amalgamation procedure (Au - Hg alloy). By using X-Ray fluorescence spectroscopy, mathematic procedures for data processing it is proved that in all cases gold was decorative and applied with fire gilding amalgamation process. Thus, in this work, it is proved that "fire mercury process" was the primary procedure for gilding ecclesiastical silver in Eastern Thessaly in the post-byzantine period.

KEYWORDS: Post – Byzantine period, ecclesiastical silver, Thessaly, XRF spectroscopy, amalgamation, Mercury, peak deconvolution

1. INTRODUCTION

From the establishment of Orthodox Christian until our days the rite involves metallic vessels in the celebration of the liturgy (chalices, disks, asterisks, blades, spoons, crosses etc). All this church plate is usually called "ecclesiastical silver" or "church silver" due to the main use of sliver, even in some cases less valuable materials were used. Moreover, these sacred objects are usually made of Silver – Gold – Copper alloy and decorated with distinctive techniques and semi-precious stones (Ikonomaki-Papadopoulou, 1980). They are often of exceptional art and of particular historical significance, which increases if they bear inscriptions with names of devout donors, of craftsmen who created them and of the date of their manufacture. That is why they constitute precious witnesses of both the aesthetics and the high level of the casting and treating techniques of metal alloys, which have been inherited from Byzantium and evolved along with the craftsmanship of the Ottoman period.

Ecclesiastical silversmithing, especially of the post-Byzantine period, is among the less studied fields. Specifically, for Thessaly, but other churches elsewehere too, research devoted to church silver has so far been minimal (Ballian 2015; Kousouni and Panagopoulou, 2018). However, monasteries and churches in the regions of Larissa and Magnesia preserve unexpected treasures of ecclesiastical art, which offer valuable evidence for workshops and craftsmen (Sdrolia et al., 2015; Varalis et al., 2020 under preparation).

Until today archaeological research has revealed six (6) ecclesiastical silver workshops in Thessaly, that of Trikala, Domeniko, Agrafa, Retsiani, Larisa and Rapsani, active from 16th up to 19th century and with more than one well-known craftsmen (Ballian 1992; Sdrolia et al., 2015; Varalis et al., 2019).

During the Byzantine period gold was the primary material for crafting ecclesiastical sacred objects but gradually and especially in the post-byzantine period was replaced by Silver. But even then, gold was remained as decoration either for aesthetic reasons or in the case of chalices and spoons that it was necessitated. The application of gold for decoration in ecclesiastic silverwares until mid-19th c. was based on the amalgamation process (Ikonomaki-Papadopoulou, 1980).

Because gold is rare metal due to its special properties and value, the development of application procedures of gilding capable of producing a golden appearance by using as small as possible quantities of gold has always been a challenge for all cultures (Ynsa et al., 2008).

As earlier works mention there are two main methods of gilding a metal object involving mercury (Hg), the "cold mercury gilding" and the "fire gilding" processes (Ynsa 2008; Vittory 1979; Oddy, 1981; Vlachou et al., 2002; Bayley and Russel 2008; Borges et al., 2008; Ingo et al., 2016; 2018; Liritzis et al., 2020).

As Vittori (1979) mention, Pliny in "Natural History" distinguish the two techniques and therefore suggests that Pliny was referring on the "cold mercury gilding" process in which mercury is used only as an adhesive for the gold leaf. In the same works (Vittori 1970, Ingo et all 2016, Borges et al 2008) it is mentioned that the "cold gilding process" is mainly used for gilding copper and bronze and therefore the purity of the substrate metal is crucial.

One the other hand the "fire gilding process" was to apply a gold-mercury (Au-Hg) amalgam to the surface of the object, then by heating mercury was evaporated and a thin layer of gold remained. This method was in use since Hellenistic times (Merrianos 2017), extensively use was in the Roman period (Oddy, 1981, Vlachou et al., 2002; Bayley and Russel 2008), post-byzantine period and up to mid-19th c. (Ikonomaki-Papadopoulou, 1980).

The surface elemental composition for each of these gilding techniques is distinctive (Ynsa 2008). A high amount of residual mercury is often present in "fire gilding" process. This is because, even though mercury boils at 357 ^{o}C, gold and mercury form an intermetallic compound Au_3Hg which is stable up to 420 ^{o}C. Moreover, mercury still forms a solid solution with gold above this temperature and up to 1000 ^{o}C. This is the main reason why is almost impossible to drive off all of the mercury in the "fire gilding" process (Meeks 1993). Also, other works mention that the existence of residual mercury is evidence of "fire gilding" process and therefore it's easy to identify the manufacturing technique (Lins and Oddy 1975, Ingo et al 2013, 2016, 2018).

This paper presents the first-ever made study on determining the gilding procedure applied from Thessalian silver workshops and craftsmen through the use non-destructive analysis (pXRF) of post-byzantine ecclesiastical silver chalices kept in parish monasteries and churches in the regions of Larissa and Magnesia.

2. RESEARCH AIMS

This research aimed to:

1. Identify either the gold, found on compositional analyses of ecclesiastical silver chalices of the post-Byzantine period in Eastern Thessaly, was part of the Ag – Cu – Au alloy or it was gild applied.

2. Identify the gilding procedure applied by craftsmen in post byzantine Eastern Thessaly
3. Investigate the potentiality of developing a procedure to identify gilded surfaces with portable XRF spectrometers.

3. MATERIALS AND METHODS

Archaeological material

Through a chronological frame from March of 2018 until July of 2019 the authors made several trips to Eastern Thessaly to find, identify, record, study and analyze with non-destructive analytical techniques the remaining post-byzantine ecclesiastical silvers in parish monasteries and churches. Among other liturgical objects, seventy – six chalices were in – situ studded for the composition of main parts and decorations (Varalis et al., 2020 under preparation). From these seventy-six chalices, the twenty-five (33% of total) seems to had goldish decoration. Table 1 and 2 presents the details of these twenty – five chalices that have been studied in this work for their gilding procedure identification.

These twenty – five (25) chalices are separated into two main categories. The first category (table 1) are those belonging to the post – Byzantine period (14^{th} – 18^{th} c. The second category (Table 2) are those belonging in the 19^{th} and 20^{th} c. The dating of both sets of chalices was based either to inscriptions or in comparison of typology with well-known chalice types (Ikonomaki-Papadopoulou, 1980; Sdrolia 2018; Ballian 2001; Varalis et al 2019; Varalis et al., 2020).

At this point, it is necessary to clarify the use of the term "post-byzantine" in this work and therefore why both objects of tables 1 and 2 are equally important. As already indicated in previous works (Ballian, 2015) the term "post-byzantine" in ecclesiastical art covers a period from 1453 onwards. Alongside, this term is referred to geographical regions under Venetian rule and the ottoman – ruled areas of mainland Greece (Gratziou, 2005). As Ballian (2015) quotes, the term "post-byzantine" as a chronological and geographical frame "encompasses a vast geographical and chronological span (up to 1669 for Venetian Crete and 1821 for southern Greece)". As a result, for the geographical area of Thessaly the term "post-byzantine" it refers to a period from 1453 to the Convention of Constantinople in 1881 and the cession of the Region of Thessaly to Greece. It is worthy to mention that in case of metalwork during this extended post-byzantine period in Thessaly (mid 15^{th} c - Late 19^{th} c) the Byzantine tradition is mixed and affected by Ottoman and Balkan art (Ballian 2001, 2015).

Therefore, taking in account the time frame of post – byzantine period for Eastern Thessaly (mid 15^{th} c – late 19^{th} c) and the earlier reports for the use of gold as the primary material for decoration in ecclesiastical silver until mid-19^{th} c (Ikonomaki-Papadopoulou, 1980) it turns out that both sets of chalices (Tables 1 and 2) are equally important to evaluate and determine the gilding procedure in Eastern Thessaly during this period.

Table 1. List of the examined 16^{th} – 18^{th} c. chalices from Eastern Thessaly kept in parish churches and monasteries (Varalis et al 2020 under Preparation). Sampling points are: b=base of the chalice and ch=main bowl of the chalice

No	Sampling Point	Place of origin	Dimensions	date	Photo
1	b	Keimeliophylakion of Aghia	19.5×13.4 cm	16^{th} c. (inscription date: 1561)	

2	b, ch	Village of Ma-kriraxi, Church of Timios Prodromos	22.8x12.6 cm	16th c.	
3	ch	Zagora Village, Church of Ag. Georgios	21x12 cm.	17th c.	
4	b, ch	Zagora Village, Church of St. Par-askeui	20.3x14 cm	17th c. (inscription date: 1673)	
5	b, ch	Village of Ma-kriraxi, Church of Timios Prodromos	21,5x12,4 εκ.	Early 18th c. (inscription date: 1717)	

6	ch	Zagora Village, Church of St. Paraskeui	26.4x15.5 cm	18th c. (inscription date: 1731)	
7	ch	Zagora Village, Church of St. Kyriaki	26x15.2 cm	18th c.	
8	b, ch	Trikeri Village, Church of Holy Trinity	28.5x7.5 cm	Mid. 18th c. (inscription date: 1749)	
9	b, ch	Kissos Village, Church of St. Marina	22.7x12 cm	Mid 18th c. (inscription date: 1767)	

10	b	Keimeliophylakion of Kato Moni Kseni-as	25.5x18 cm	Late 18th c. (inscription date: 1795)	
11	ch	Keimeliophylakion of Kato Moni Kseni-as	23x16.5 cm	Late 18th c.	
12	ch	Kissos Village, Church of St. Mari-na	21.3x13.3 cm	Late 18th c.	
13	b, ch	Zagora Village, Church of Ag. Georgios	31x19.5 cm	Late 18th c. (inscription date: 1794)	
14	ch	Zagora Village, Church of Ag. Georgios	18.5x16.4 cm	Late 18th c.	

15	b	Zagora Village, Church of Meta-morfoseos	26.8x15 cm	Late 18th c.	
16	b, ch	Zagora Village, Church of St. Kyria-ki	30x19.2 εκ.	Late 18th c.	

Table 2. List of 19th – 20th c ecclesiastical silver chalices found also in parish churches and monasteries during this work that also has been analyzed for comparison to table 1 results. Sampling points are: b=base of the chalice and ch=main bowl of the chalice

No	Sampling Point	Place of origin	Dimensions	date	Photo
1	ch	Drakeia Village, Church of St. Nico-las	22.7x14.8 cm	19th c. (inscription date: 1814)	

2	b, ch	Drakeia Village, Church of St. Nicolas	23.7x16.4 cm	19th c.	
3	b	Trikeri Village, Church of Holy Trinity	29.5x17.8 cm	19th c. (inscription date: 1822)	
4	ch	Village of St. Lavrentios, Church of St. Apostoles tou Neou	21.5x15.7 cm	19th c. (inscription date: 1824)	
5	ch	Village of St. Lavrentios, Church of St. Apostoles tou Neou	20.4x15 cm	19th c. (inscription date: 1824)	

6	ch	Zagora Village, Church of Ag. Georgios	22.4x14.3 cm	Early 19th c.	
7	ch	Zagora Village, Church of St. Paraskeui	26.2x15 cm	Late 19th – early 20th c.	
8	ch	Tsangarada Village, Church of Taxiarches	25.5x14.8 cm	20th c. (inscription date: 1901)	
9	ch	Village of St. Lavrentios, Church of St. Apostoles tou Neou	28x17.2 cm	20th c. (inscription date: 1902)	

XRF spectroscopy

For the qualitative and quantitative determination of metal alloys compose the gilding surface of the studied chalices a portable hand-held X-ray Fluorescence Spectrometer (ThermoScientific Niton XLp XRF Analyzer) was used with an Am-241 source, solid-state detector and calibrated for metal alloys.

For data acquisition from XRF handheld instrument, a build-in software was used for the data point set of each measurement get saved as Unicode ASCII file format to be compatible for further analysis with specialized software. All XRF spectrum analysis and process of all measurements were made with scientific software Spectragryph (F. Menges "Spectragryph - optical spectroscopy software", Version 1.2.13, 2019).

The calibration for metal alloys was set as the factory default operation and the build-in shutter calibration was performed. Also, before each study of an object of tables 1 and 2, two reference materials were measured, a certified authentic gold sovereign of 1909 purchased from the Bank of Greece (91,66% gold) (Fig. 1) and a custom-made reference material of Lead-Tin alloy of Pb-60% and Sn-40% which on the one side was coated with a gold leaf of 22K applied with organic resins (Fig. 2).

Figure 1. A certified authentic gold sovereign from the Bank of Greece used as reference material for gold (91,66% gold).

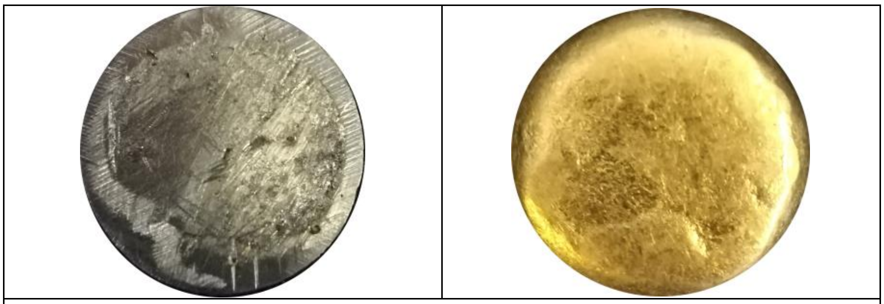

Figure 2. Custom-made reference material of Lead-Tin alloy of Pb-60% and Sn-40% which on the one side was coated with a gold leaf of 22K applied with organic resins

4. RESULTS AND DISCUSSION

The first step of this study was to analyse the XRF spectra of the repeated measurement of the certified gold sovereign. After that, the XRF spectra of a pure gold leaf of 22K (~91,66% Au) was analyzed along with the reference material of Pb60Sn40 gilded with a layer of that same gold leaf. Finally, the XRF spectra of thirty-five measurements of the table's 1 twenty-five chalices were also analyzed.

Figure 3 presents all XRF spectra of gold sovereign and gold leaf and Fig. 4 presents the XRF spectra of goldish decorations in studied objects, both in the energy (KeV) region of gold. All spectral data are normalized with the rule of max value equal to one.

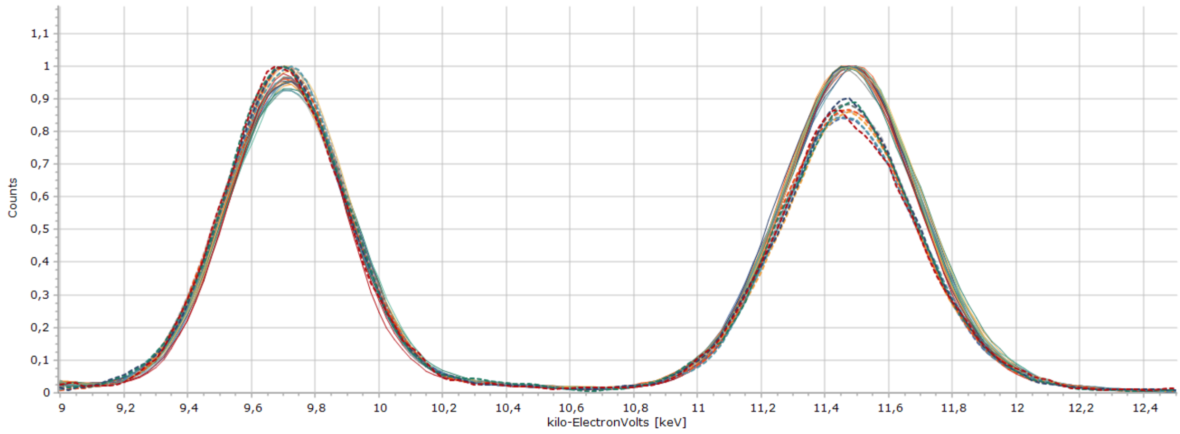

Figure 3. XRF spectra of the pure certified gold sovereign (91,66% Au) (solid lines) and 22K gold leaf (dash lines)

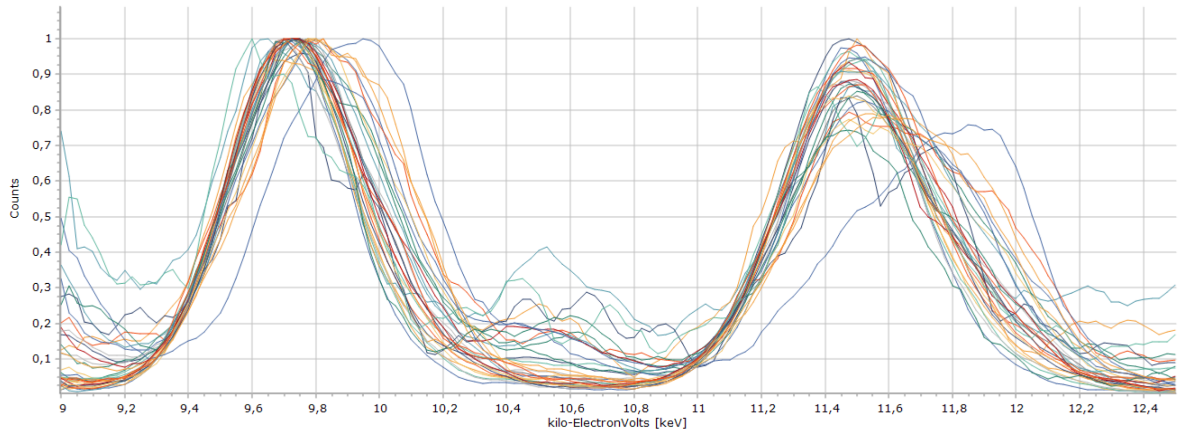

Figure 4. XRF Spectra of goldish decorations in studied objects (tables 1 and 2). Energy band of gold (9 - 12.5 KeV)

Fig. 5 presents, in contrast, an average spectrum of gold sovereign/gold leaf and an average spectrum of a gilded object. Fig. 6 presents a few of the gold sovereign/gold leaf and gilded object's XRF spectra all normalized with the rule: the max counts at energy point of 9.71 KeV equal to 1. The value of 9.71KeV was selected as it is the principal La emission line of Au (Grieken and Markowicz 2002)

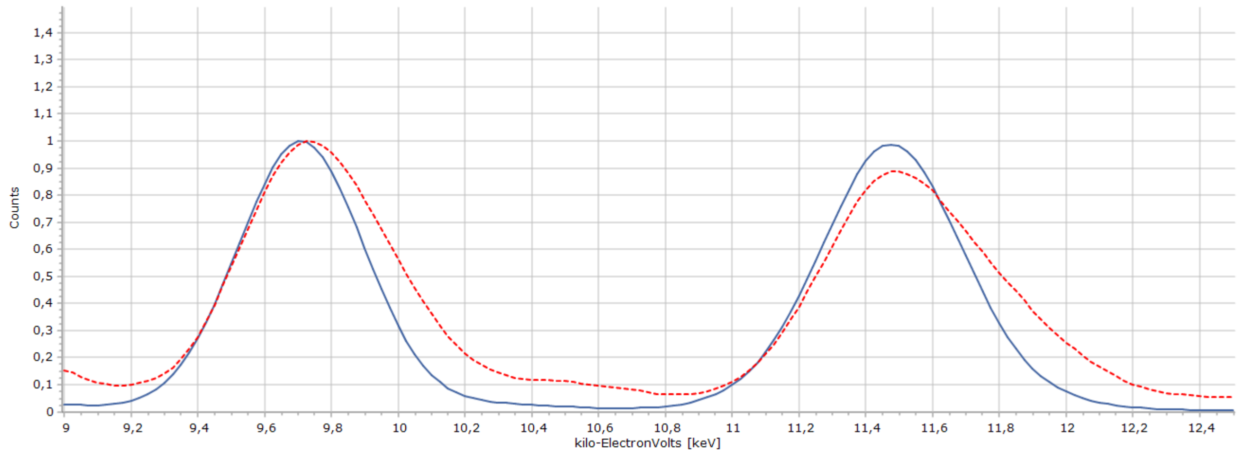

Figure 5. Average XRF spectra. Blue solid line for pure gold and red dashed line for goldish decorations

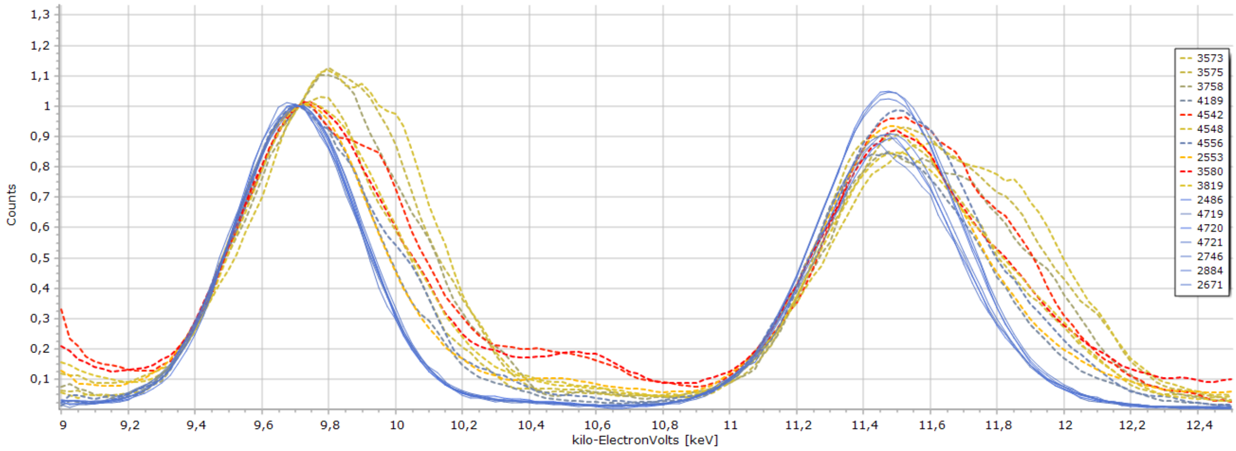

Figure 6. representative XRF spectra normalized as the 9.71KeV energy equal to 1. Solid lines for pure gold and dashed lines for goldish decorations in studied chalices

As it is observed in Figs. 3 to 6 the spectra lines of gold in goldish decorations of studied objects has a broader peak in 9.71 KeV and 11.43 KeV in contrast to pure gold of sovereign or leaf. Also, in some cases, a displacement in the KeV axis of peak value for gilded objects was observed. To evaluate this disturbance and difference, the FWHM of each peak in both La and Lb emission lines of Au was calculated for all aforementioned spectra (gold sovereign, gold leaf, gold leaf on Pb60Sn40 sample and of gilded objects).

Fig. 7 presents the bi-plot of FWHM vs KeV and the difference of gilded objects in both KeV of emission lines and FWHM in comparison to pure gold is clear. Even the FWHM of Au peak in Pb60Sn40 covered with gold leaf is same as those of gold sovereign and pure leaf suggesting that a) the gilding with organic resins does not affect the Au FWHM and b) the underlying surface also does not affect the Au spectrum and FWHM.

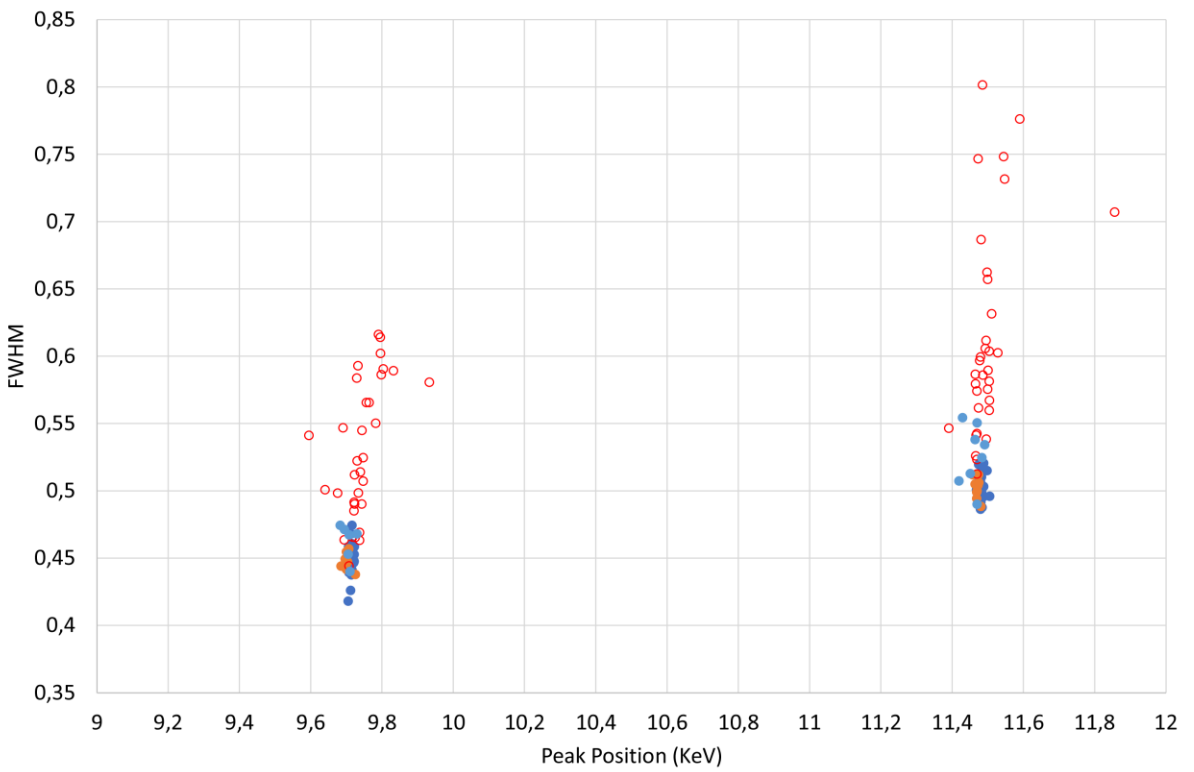

Figure 7. FWHM vs Peak position. Deep blue points refer to pure gold from the certified sovereign, orange for the gold leaf, light blue for the gold leaf applied on Pb60Sn40 sample and red circles for goldish decorations.

Gold (Au) and mercury (Hg) are adjacent elements with atomic numbers of 79 for gold and 80 for mercury and therefore the XRF emission lines are also coherent with gold to have La of 9.71 KeV and Lb of 11.43 KeV and mercury to have La of 9.98 KeV and Lb of 11.82 KeV. Taking in account this, the broader FWHM of the gold peak in XRF spectra of gilded objects seems to be the convolution of gold's with residual's mercury's emission lines and hens the first evidence of the use of gold – mercury amalgam with "fire gilding process" on these objects.

To evaluate the above assumption of the convolution of gold emission lines with those of residual mercury a deconvolution process of XRF spectra of gilded objects was performed. Deconvolution procedure was performed with the proposed peak fitting algorithm by O' Haver (2019) for Matlab or Octave. The peak fitting parameters used in this work were: center = 0, window = 0, number of peaks 2, peak shape Lorentzian, no extra parameters and repeated trials of 20. With these parameters, every convoluted peak was deconvoluted into two Lorentzian peaks after 20 trials with slightly different start values and finally, the algorithm selects the best one (O' Haver, 2019).

As it is well known the spectral line of characteristic radiation of a single x-ray line has a Lorentz distribution (Espen 2002). Also, when the spectrum is observed with semiconductor detector the observed peak is a convolution of this Lorentz distribution and the Gaussian detector response function (Espen 2002). Therefore, an observed peak shape lies between a Lorentzian when spectral effects dominate and Gaussian when instrumental effects are stronger (Huang and Lim 1986; Espen, 2002).

The detector operation is constant for all measurements and therefore if the broadening of observed peaks (Figs. 3-6) was produced by a Gaussian – Lorentz blended distribution (Voigt profile) the response had to be the same for pure gold sovereign and gold leaf as for the spectra lines of gilded objects. But as it is observed in Fig. 6 spectra of pure gold is deferent that of gilded gold. Therefore, it is clear that the observed broader peaks are the convolution of Lorentz distribution emission lines of gold and mercury. Taking all these in account the deconvolution has to be made back in Lorentz distribution of separated emission lines. Thus, in the aforementioned deconvolution algorithm by O Haver (2019) the peak shape set to Lorentz distribution.

Fig. 8 presents a result for such a deconvolution procedure and Fig. 9 the peak position of energy KeV of deconvoluted peaks. As it is proved and clearly observed every peak is the convolution of gold emission lines and residual mercury emission lines.

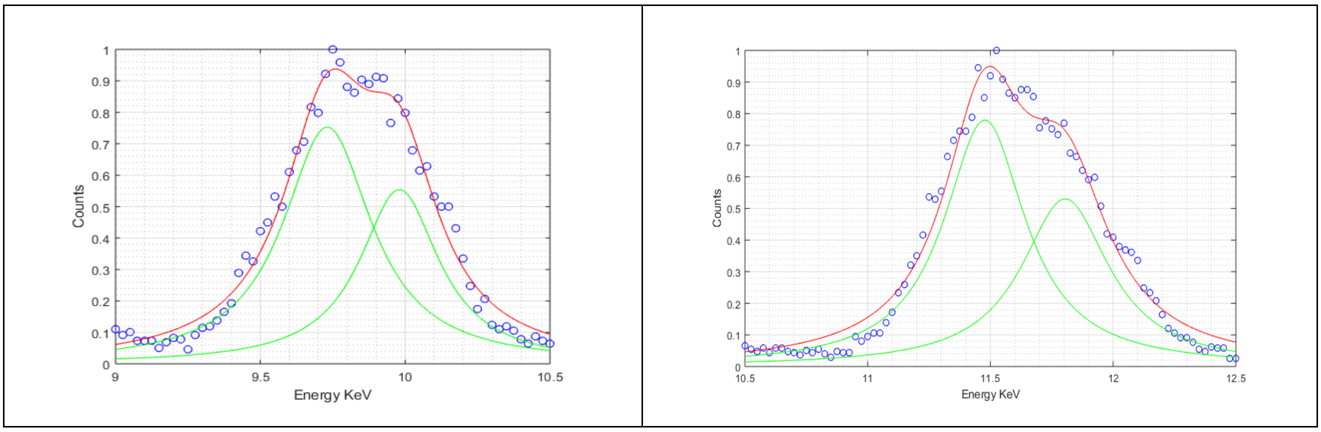

Figure 8. Deconvolution of goldish peaks in the Au-Hg region of 9 - 12.5 KeV. Blue circles refer to real XRF data of goldish decoration, green lines the deconvoluted peaks and red line the interpolation of green lines.

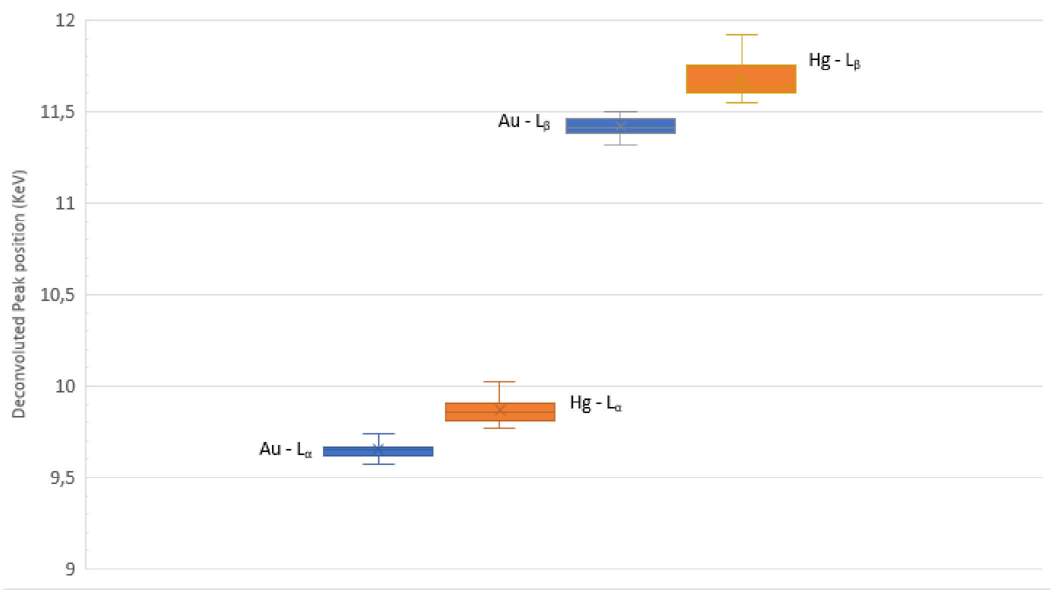

Figure 9. Deconvoluted Peak positions (KeV) for all goldish decorations.

5. CONCLUSION

Through the present study, we were given the opportunity to deal with remarkable ecclesiastical silverwares of eastern Thessaly and to apply novel approaches for the determination of crafting procedures.

Summarizing the results: thirty-five measurements on goldish decorated parts of twenty-five chalices of the post-byzantine period (mid 15^{th} c – late 19^{th} c) were analysed in order to determine the gilding procedure. In all cases, the gold x-ray fluorescence peak line was convoluted with a second emission line of residual mercury from the amalgamation process. Hence it is proved and well documented that all studied objects were partially gilded for decoration by using the "fire mercury gilding" process.

All results leading to the strong hypothesis that during the post – byzantine period, the dominant procedure for gilding silver in Eastern Thessaly was the procedure of fire gilding.

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