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INVESTIGATION OF GOLD TISSUES SURFACE FROM THE CATHEDRAL OF ASSUMPTION (KYIV) SARCOPHAGUS

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The objects of the cultural heritage discovered by archaeologists on the area of ancient Kyiv (Old Russia) were examined by non-destructive techniques such as SEM with EDXRS, XPS and AFM. The topographical examination shows that the fragments of gold tissues (XI–XVI AD) content two kinds of materials: golden spiral threads and light colour flakes. In the composition of the threads, Au is the basic element but the flakes contain C, O, Mg, Al, Si, P, S, Ag, Ca, and Fe. The origin of these flakes today is unknown (the traces of cloth or varnishes possible). The presence of Rb in golden artefacts testifies the existence of own gold output and technology on the area of ancient Kyiv. The absence of Cu, which presents in oldest gold tissue, in the samples manufactured in XV–XVI AD can be connected with an improvement of melting technology. The difference in the composition in the golden threads for all samples examined shows them to be manufactured in various regions of Old Russia.

INTRODUCTION

Archaeology tries to reconstruct the culture and history of past societies, especially of those on which no or poor written sources exist and for which information on every day life is in scare. Archaeology today uses a variety of methods and tools: survey and excavation, environmental analysis or glacial records, scientific and historical dating methods, historic and iconographic sources, archaeological experiments and, last but not least, material analysis of found artefacts. The material analysis has two main topics: the characterization of the material of which objects consist and the characterization of technical treatment or manufacturing of the objects. The first can, for instance, give information about the provenance of the material while the latter helps us to reconstruct ancient techniques.

The excavations and investigations of archaeologists often lead to discovery of golden artefacts. Among the metals used by man in past, gold has a privileged place. Mainly obtained by the exploitation of quartz veins (primary deposits) and placers (second deposits), found free in nature, gold was one of the first metals used in fabrication of objects [1]. The analysis of gold objects contributes to the knowledge of many aspects of their fabrication [2]. Various information can be obtained on the manufacturing technologies of the objects and on the provenance of the metal as a result of the analytical methods use. The aim of the first studies is an accurate description of the objects using information given by their analysis. The aim of the second point is the identification of the extraction, refining and alloying techniques.

A variety of instrumental analytical technique can be applied to the physical and chemical examination of the works of art and archaeology but mainly without any sampling. The most examined from the gold finds are different jewellery, coins, metal alloy and shards discovered in America and Europe [2–16]. The use of modern methods of non-destructive analysis permits us to determine the provenance of gold or its origin difference [2, 9–13, 17, 18] and to propose a technique of the objects manufacture or treatment [2, 8, 13–15, 17, 18]. In the same time, it is necessary to note that the objects of the cultural heritage found in the East Europe, on the area of Old Russia particularly, belong to the number of less studied.

The fragments of gold tissues, somewhat exotic materials, found by archaeologists in different tombs of the Cathedral of Assumption (Kyiv) were the objects of this investigation. SEM with

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EDXRS (Scanning Electron Microscopy with Energy Dispersion X-ray Spectrometry), XPS (X-ray photoelectron spectroscopy) and AFM (atomic force microscopy) methods were used for non-destructive chemical composition and topographical analysis of these archaeological finds. It is necessary to note that two last methods were not used for the study of gold artefacts in contrast to the first technique [17, 18].

EXPERIMENTAL

The samples of the gold tissues studied in this work were found in different sarcophagi of the Cathedral of Assumption (Kyiv). The characteristics of the objects studied are shown in Table 1.

Table 1. Description of tissue samples

Sample	Characteristic of sample
T1	Fragment of gold tissue 2.4x1.5 m from sarcophagus N 2; the tomb a man (probably the member of family of Knight S.Olel'kovich); XV–XVI AD
T2	Fragment of gold tissue of a head-rest coverlet from sarcophagus N 4; the tomb a woman (probable the wife of Knight M.Olel'kovich); XV–XVI AD
T3	Fragment of gold tissue from sarcophagus N 3; the tomb of unknown man (probably the church man); XI–XIII AD

The images of the gold tissues fragments and their element composition on the subsurface region (probing depth up to 2 μm) for all samples, without their cover by carbon, were obtained by the method SEM with Jeol JSM 5400 and EDXRS LINK ISIS Seria 300 instruments. The composition analysis was realized by an electron microbeam in several places of each sample (see Figs).

The surface composition up to 2 nm was obtained from XPS spectra recorded by a VG Scientific ESCA-3 photoelectron spectrometer using AlKa radiation (1486.6 eV). Binding energies of electrons of analyzed elements were referenced to the C 1s peak at 284.8 eV. The spectrometer was interfaced with IBM PC AT microcomputer for acquisition and processing. The relative content of elements in the samples was determined by a method analogous to that described in [19].

The surface morphology of the samples and its roughness in nanometric region was studied by atomic force microscopy (AFM) using a Nanoscope E, Digital Instruments Scanning Probe Microscope Controller.

RESULTS AND DISCUSSION

The microphotographs of two fragments of T1 sample presented in Fig. 1 show that the topography of this object is complicated. The amorphous compounds of light colour, looking like flakes (points B1 and B2), present in the surface of golden colour spiral threads (point A, A1, and A2). It can be seen that these golden threads represent the twisted foil with the width equal to 150–200 μm and the thickness near 2–5 μm. The flakes have the greater thickness but the lesser other dimensions. The habitus of these objects permit the use of microbeam technique for the reliable quantitative analysis of their chemical composition.

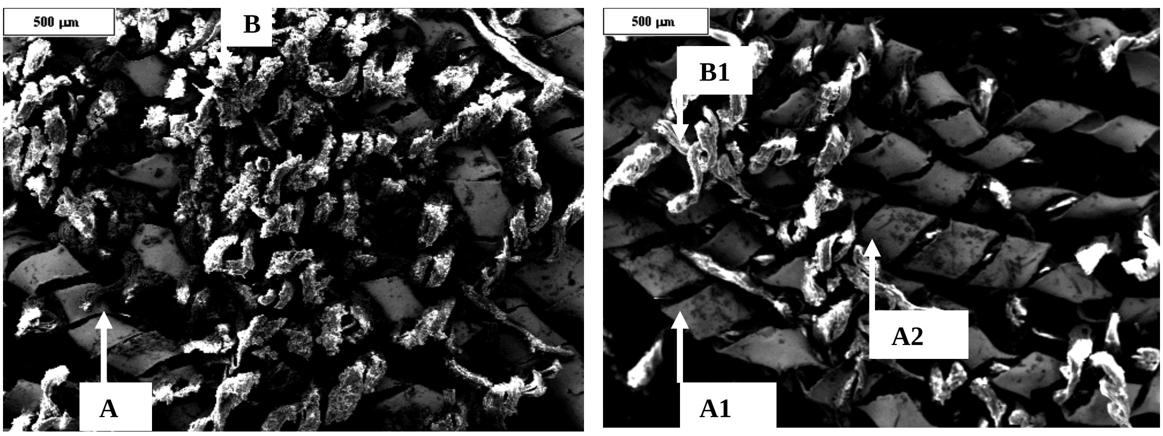
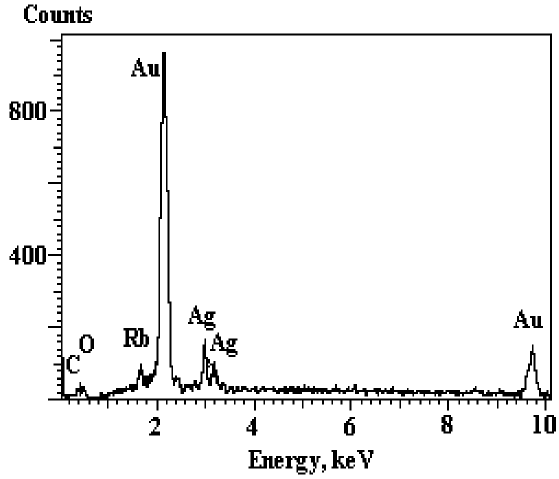


Fig. 1. The microphotographs of gold tissue T1



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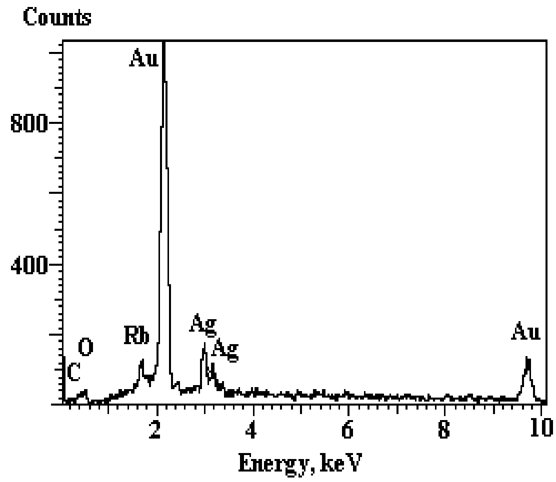

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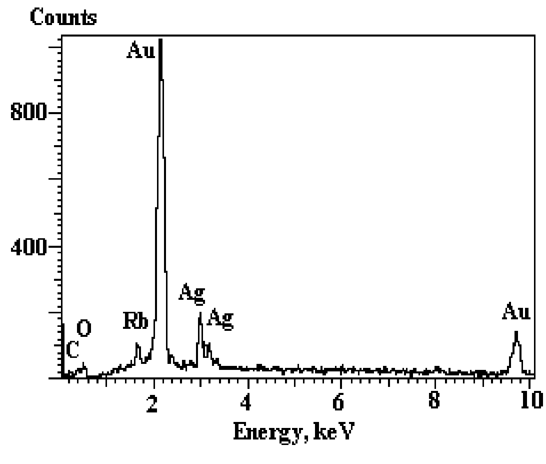

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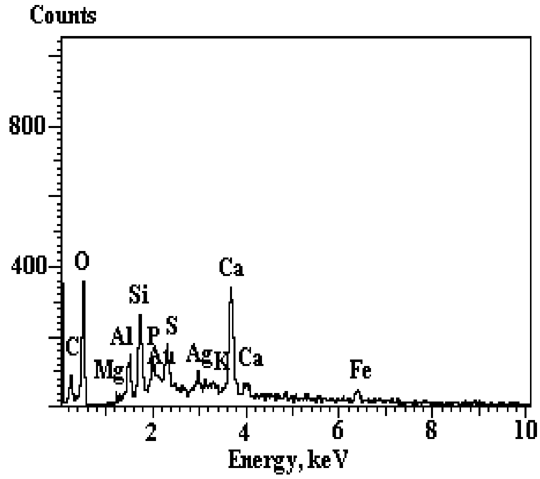
Fig. 2. EDXR analysis of the gold tissue T1 chemical composition in points A (*a*), A1(*b*) and A2 (*c*)



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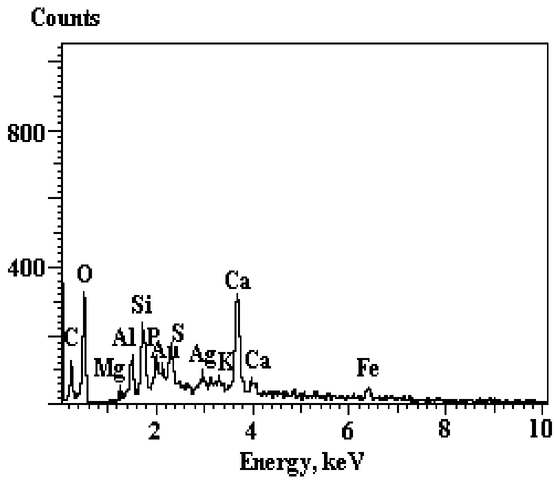

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Fig. 3. EDXR analysis of the gold tissue T1 chemical composition in points B (*a*) and B1 (*b*)

The chemical analysis of the T1 sample by an electron microbeam was realized in five points of golden spiral threads (three points shown in Fig. 1) and in four points of light flakes (two points shown in Fig. 1). The analysis data presented in Fig. 2 show that the basic element in the composition of spiral threads (points A) is Au. Simultaneously they content a small quantity of Ag and Rb. The composition of the threads determined in other points is practically equal to the data presented in Fig. 2. The analysis of light flakes is presented in Fig. 3. The data of the analysis demonstrate that these objects content the following elements: C, O, Mg, Al, Si, P, S, Ag, Ca, and Fe. The traces of Au were also observed. Analogous results were obtained for other points B of T1 sample fragments.

The calculation results of the objects composition found in golden tissue T1 are presented in Table 2 and Table 3.

Table 2. Composition of gold threads in tissues determined by EDXRS

Tissue	Point	Component, wt. %			
		Au	Ag	Rb	Cu
T1	A	96.2	2.7	1.1	-
	A1	95.8	3.0	1.2	-
	A2	96.4	2.6	1.0	-
	A3	96.8	2.3	0.9	-
	A4	97.1	2.2	0.7	-
T2	A	96.6	1.9	1.5	-
	A1	96.6	2.0	1.4	-
	A2	96.5	2.1	1.4	-
	A3	96.4	2.3	1.3	-
	A4	97.1	1.8	1.1	-
T3	A	94.5	2.0	2.4	1.1
	A1	94.3	1.9	2.6	1.2
	A2	95.0	1.5	2.7	0.8
	A3	95.4	1.4	2.5	0.7
	A4	94.7	1.9	2.5	0.9
	Sum	94.9	1.8	2.3	1.0

Table 3. Composition of light flakes in tissue T1 determined by EDXRS

Point	Component, wt. %									
	C	O	Ca	Si	Al	P	S	Mg	K	Fe
B	25	16	17	14	9	6	8	1	1	3
B1	27	18	14	12	8	5	7	2	2	5
B2	20	16	19	17	11	4	5	1	3	4
B3	32	21	11	6	7	8	3	3	2	7

The fragments of golden tissues T2 and T3 are presented in Fig. 4. A small number of light flakes is present on surface of golden spiral threads in sample T2 but the surface of tissue T3 is practically free from these objects. The results of chemical analysis by an electron microbeam of these flakes (points B and B1) presented in Fig. 5 show that their composition differs from that determined on tissue T1 (Fig. 3). The presence of Ca and Si lines (the most intensive in Fig. 3) and a high intensity of C and O lines against background of elements spectrum characteristic of golden threads can be only observed. The absence of other elements characteristic of the flakes (Fig. 3) can be connected with small dimensions and friable structure of these objects on sample T2. As a result, the microbeam analysis shows the base composition of gold threads disposed under or near the flakes. The chemical analysis of gold threads (points A and A1 on sample T2) demonstrates the presence of Au (basic elements) and little quantity of Rb and Ag (Fig. 6). These data have no qualitative difference with the results presented in Fig. 2 for tissue T1. The quantitative data of the analysis of gold threads composition are present in Table 2. The absence of all components of light flakes (the major elements, Ca and Si are only present in the spectrum,) did not permit us to realize their composition quantitative determination.

The microbeam analysis of golden threads (points A and A1) in T3 sample permits the presence of Cu additive side by side with basic elements Au, Ag, and Rb found (Fig. 7). Since of absence of the flakes on tissue T3, an EDXR spectrum of the whole fragment presented in Fig. 4 was obtained. The results show (Fig. 7, spectrum Sum) the presence of Ca traces and increased content of C and O in comparison with the data for points A and A1 which are characteristic for the composition of the flakes.

The composition of the flakes presented in Table 3 shows that they content a large quantity of carbon and oxygen and this fact permits us to assume the organic origin of these objects. From the one point of view, these flakes could present the traces of cloth but, from the other hand, these objects content enough number of inorganic elements. The analysis of published data shows that these objects can belong to remains of the varnishes. It is known that varnishes are mixtures of natural products in which the main components are organic substances but, beside these major components, some inorganic substances (as pigments, siccatives, etc) were added to control the properties of the historical varnishes [6].

The analysis of small flakes of varnishes detected more than 20 elements in their composition [6] among which silicon, phosphorus, sulphur, potassium, aluminium, calcium, and iron, also determined by us in this study. The published data shown the impossibility to calculate the mass fractions of the detected elements in relation to the total sample mass and the relative content of element with respect to the sum of all detected elements was only determined.

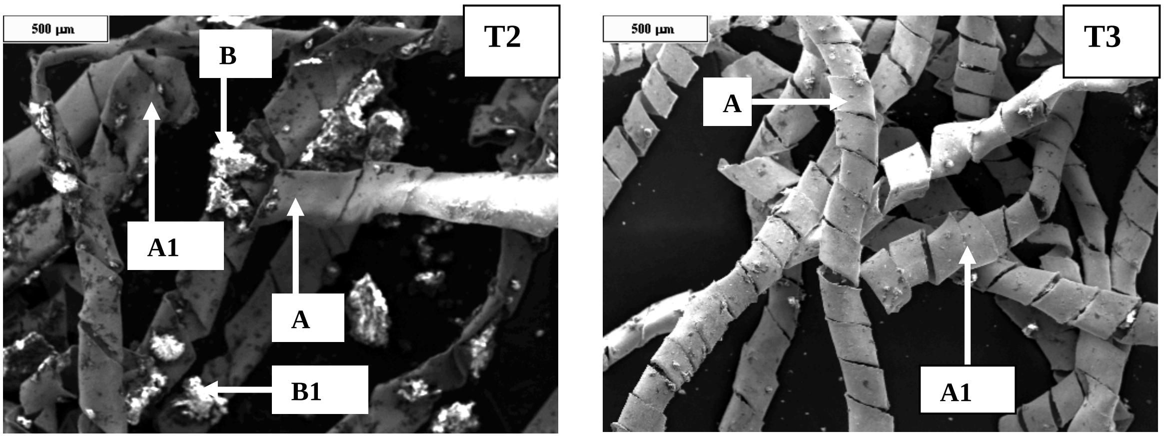
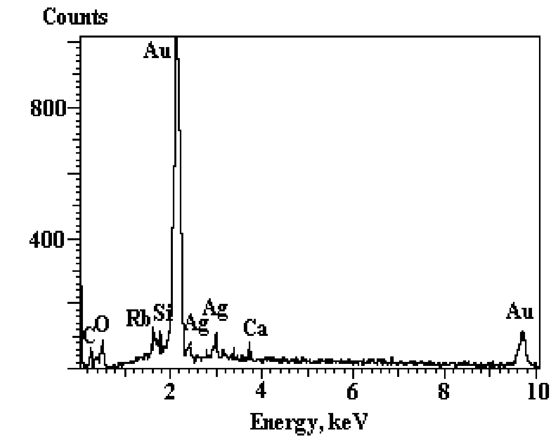


Fig. 4. The microphotographs of gold tissue T2 and T3



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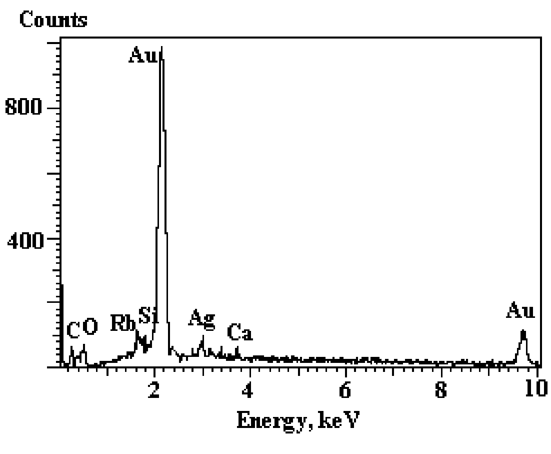

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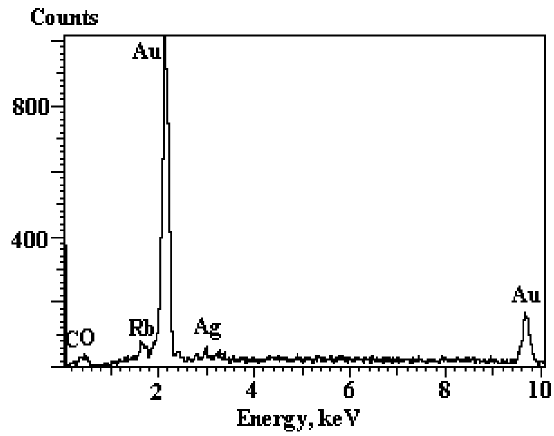
Fig. 5. EDXR analysis of the gold tissue T2 chemical composition in points B (*a*) and B1 (*b*)



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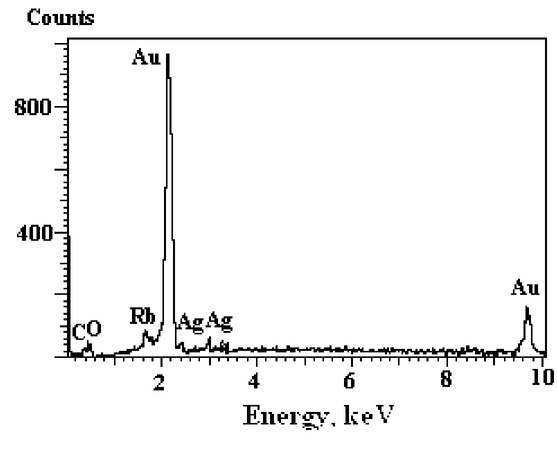

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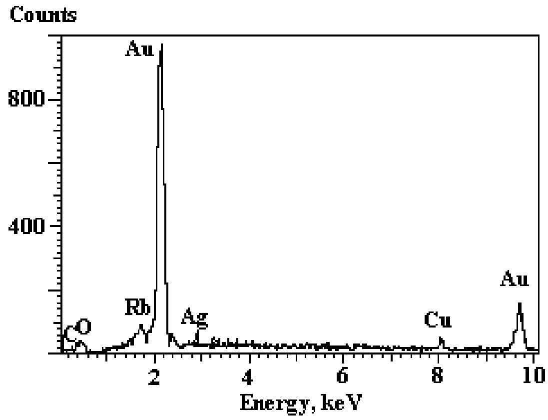
Fig. 6. EDXR analysis of the gold tissue T2 chemical composition in points A (*a*) and A1 (*b*)

Presented in literature data demonstrate a large change of the component ratio in different samples of these artefacts. This operation was also realized in our case and obtained data are presented in Table 3. It is necessary to note that quantity of the light flakes decreases for the sample T3 which is older for 2–3 centuries than T1 and T2.This fact can be connected with destruction of these objects (cloth or varnishes) and dissolution of inorganic elements or with the absence of this technique early and lower social situation of the burial place object. Thus, the origin of the flakes detected by us today is unknown and only further investigations would provide a definitive answer about the nature of light flakes. In order to determine the origin of gold artefact not only the concentration of the base component (gold) but the fingerprinting of gold or the content of specific trace and ultra-trace other elements has a big importance. The results of the EDXR analysis of golden threads of the tissues T1-T3 presented in Figs. 2, 6 and 7 are summarized in Table 2 for all places of the composition determination.

These data show a high content of gold in all studied artefacts. The other elements determined in these objects were Rb, Ag, and Cu in the sample T3.Comparison with published data of the gold artefacts analysis show that two last elements present practically in all goods but Rb was determined by different methods in any ancient objects found in Belgium [13], Macedonia [14], Spain [8, 10, 21], Siberia and Mongolia [2, 11,16], South America and Mexico [17], Portugal [11, 18], France [18], Italy [4, 9, 20], Greece [9] and other places. The presence of this element in gold objects from sarcophagus of the Cathedral of Assumption can testify about the availability of own gold output on Old Russia. On the other hand, the oldest tissue T3 contents a Cu additive in the composition which is absent in another samples. This fact can be connected with the difference in gold sources or in places of these tissues manufacture in ancient Kyiv (Old Russia). The change of gold production technology which could take place at 2–3 centuries can be taking in consideration also. It is necessary to note that the concentration of base element gold in threads of T3 (mean value is equal to 94.8% wt.) is lower than in T1 and T2 (96.5 and 96.6% wt., respectively). The content of copper in artefact T3 is also low (mean quantity is equal to 1.0% wt., Table 2) and expected for native gold.



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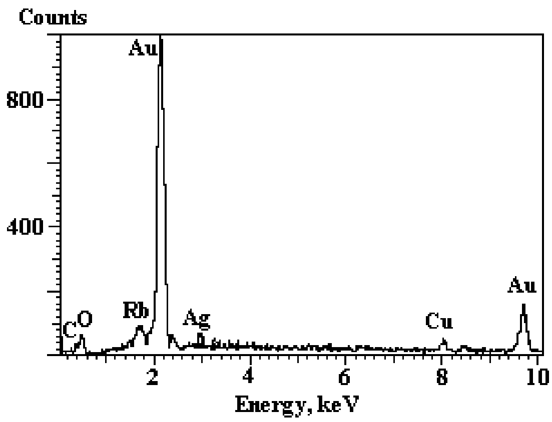

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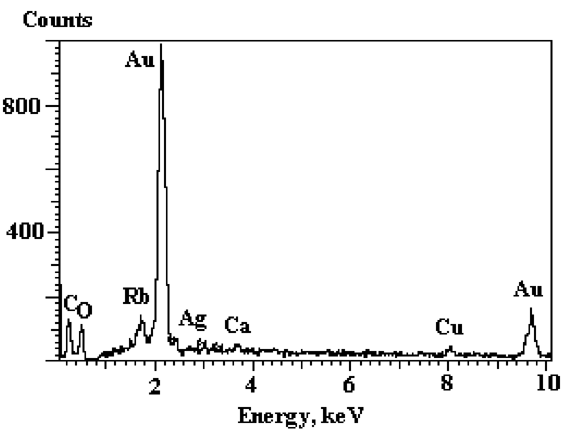

c

Fig. 7. EDXR analysis of the gold tissue T3 chemical composition in points A (*a*), A1(*b*) and over fragment presented in Fig. 4 (Sum) (*c*)

The samples T1 and T2 have the same content of gold (see above) but the concentration of additives (Ag and Rb) differs. If the amount of silver and rubidium in threads of T1 is equal to 2.5 and 1.0% wt., their content in T2 is of a 2.0 and 1.4% wt., respectively. The observed difference between T1 and T2 in amount of impurities can be connected in our opinion with the modification of purification method of the same provenance gold.

It is known that for the gold rich objects problems of patina or corrosion are absent. In connection with this the data of surface analysis by XPS method and their comparison with the results obtained by EDXRS permit us to determine the homogeneity of the samples or shed light on the method of their manufacture. The determined values of the electron binding energies (BE) of the base elements of golden threads are presented in Table 4. These data show that in samples T1 and T2 gold has one peak with BE value near 84.0 eV which is characteristic of metallic Au (0). In the case of sample T3, two components in spectrum of Au 4 $f_{7/2}$-electrons are observed: the first has BE = 83.9 eV (Au^0) and the second with BE = 85.6 eV which characterize the presence of oxidized gold Au^{3+}. The quantity of this last gold form is equal to 10%. It is necessary to note that practically the same BE of gold (84.0 and 85.5 eV) and amount of Au^{3+} (14%) values were determined for a gold artefact in [21]. Analogous results were observed for BE of Ag $3d_{5/2}$-electrons: in the case of T1 and T2 samples the spectrum was well fitted by one peak with value of BE characteristic of Ag^0 but two peaks with BE of 367.8 and 368.6 eV were obtained for T3 sample which characterize the presence of Ag^0 and Ag^+ simultaneously with amount of Ag^+ near 12%. All three samples demonstrate one peak of Rb $3d_{5/2}$-electrons with value of BE near 110.0 eV. The low number of publications connected with the XPS spectra of Rb did not permit us to determine the valence state of this element. In accordance with published data, this value of BE was observed for metallic rubidium and its compounds [22–24]. Copper was observed in the XPS spectrum of T3 sample only and this spectrum of Cu $2p_{3/2}$-electrons had one peak with BE = 932.3 eV corresponded to metallic element, and the second peak with BE = 933.6 eV which could be connected with oxidized copper. The presence of the satellite peaks permits us to indicate the existence of Cu^{2+} in the sample (Cu^+ does not a satellite). The ratio of these copper forms Cu^0/Cu^{2+} can be estimated as 70/30. The results obtained permit us to suppose that presence of copper in gold alloy initiates and catalyses the surface oxidation of other elements or the formation of their carbonate surface layer.

Table 4. Electron binding energies of the elements of golden threads

Sample	Binding energy of electrons, eV			
	Au $4f_{7/2}$	**Ag $3d_{5/2}$**	**Rb $3d_{5/2}$**	**Cu $2p_{3/2}$**
T1	83.9	367.8	109.8	-
T2	84.0	367.9	109.7	-
T3	83.9; 85.6	367.8; 368.6	110.0	932.3; 933.6

It is known that the determination of absolute concentration of the elements by XPS method is a very difficult problem but the atomic ratio of the elements within surface layer can be determined sufficiently exactly. These data are presented in Table 5 as compared with the atomic ratio of the elements calculated from the results obtained by EDXRS. From Table 5 it is possible to see that the atomic ratio of the components in samples T1 and T2 within surface and sub-surface layers is practically the same. But an excess of copper within surface layer of the sample T3 and a simultaneous decrease in silver content is observed. This fact can be a reason of some corrosion of the sample T3 surface layer. The increased content of copper on surface in comparison to bulk of the gold threads can be connected with diffusion of this element in the times. On the other hand, these results confirm the opinion connected with difference of technology of the manufacture of tissues T1, T2, and T3.

Table 5. The atomic ratio of the elements of golden threads

Sample	Atomic ratio of the elements					
	XPS analysis			EDXRS analysis		
	Au/Ag	Au/Rb	Au/Cu	Au/Ag	Au/Rb	Au/Cu
T1	22.6	42.3	-	21.3	41.9	-
T2	25.7	30.1	-	26.2	29.6	-
T3	26.8	15.3	41.2	30.0	16.6	30.6

The results of study of golden threads of sample T1–T3 surface by AFM method are presented in Fig. 8. The measurement of roughness of the threads showed that its value for the T3 sample

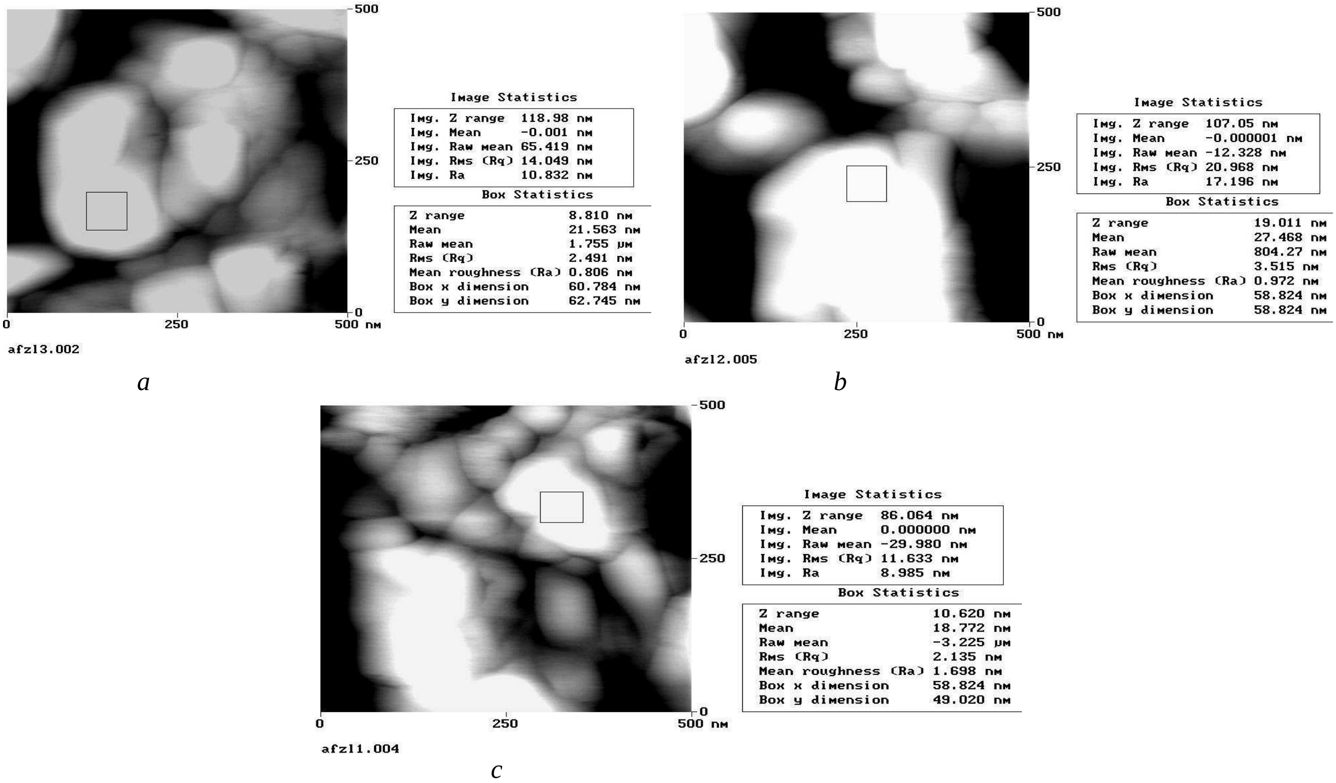

Fig. 8. Surface morphology of the golden threads samples T1 (*a*), T2 (*b*), T3 (*c*) (AFM)

(1.70 nm) is practically two times more than that the in samples T1 (0.81 nm) and T2 (0.97 nm). This fact can be connected with partial surface oxidation of gold threads T3 or with the difference in manufacture technique of artefacts studied. The difference in roughness of the golden threads of samples T1 and T2 is less significant.

Thus, the investigation of the surface of gold tissues found in sarcophagus of the Cathedral of Assumption on the area of ancient Kyiv permits us to determine the presence of own original gold output on the territory of Old Russia. The composition of the theses gold artefacts differs from that of golden objects found in other countries of Europe and America by the presence of Rb. It was found that oldest tissue (XI–XIII AD) contents along with base elements (Au, Ag, Rb) the addition of copper which is absent in tissues manufactured in XV–XVI AD. The low amount of copper in tissue is a characteristic of native gold. The presence of this addition leads to light corrosion of surface layer of golden threads of the oldest tissue which is absent in other artefacts. The process of surface oxidation leads to formation of Au^{3+}, Ag^{+}, and Cu^{2+} ions and increases the roughness of golden threads. It can suppose the improvement of smelting technology during 2–3 centuries which led to a decrease of impurities in gold. A definite difference in the composition of golden threads for all three tissues can testify that these goods were manufactured in different regions of ancient Kyiv or Old Russia. On the surface of gold threads of tissues produced in XV–XVI AD the traces of cloth or varnishes were found. Their absence in tissue manufactured early can testify the difference of tissues technique preparation or the destruction of these objects in time.

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Дослідження поверхні золотих тканин зі саркофагів Успенського собору (Київ)

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Об'єкти культурної спадщини, знайдені археологами на території старовинного Києва (Київська Русь), було досліджено за допомогою неруйнівних методів СЕМ разом з ЕДРС, РЕС і АСМ. Топологічне дослідження показало, що фрагменти золотих тканин (XI–XVI ст. н.е.) містять два типи об'єктів: золотисті спіральні нитки і пластівці ясного кольору. В складі ниток основним елементом є золото, в той час як пластівці містять C, O, Mg, Al, Si, P, S, Ag, Ca та Fe. Природа цих пластівців на цей час не з'ясована (можливо це залишки одягу чи лаку). Наявність Rb в золотих нитках свідчить про існування власного видобутку золота і технології його обробки на території стародавнього Києва. Відсутність Cu, яка є в складі виробленої в XI ст. тканини, в об'єктах, що походять з XV–XVI ст. н.е., може бути пов'язано з покращенням технології виділення золота. Відмінності у вмісті елементів в усіх досліджених нитках свідчать, що вони були вироблені в різних регіонах стародавньої Русі.

Исследование поверхности золотых тканей из саркофагов Успенского собора (Киев)

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Объекты культурного наследия, найденные археологами на территории древнего Киева (Киевская Русь) были исследованы неразрушающими методами СЭМ совместно с ЭДРС, РЭС и АСМ. Топологическое исследование показало, что фрагменты золотых тканей (XI–XVI в. н.э.) содержат два типа объектов: золотистые спиральные нити и хлопья светлого цвета. В составе нитей основным элементом является золото, в то время как хлопья содержат C, O, Mg, Al, Si, P, S, Ag, Ca и Fe. Природа этих хлопьев в настоящее время не выяснена (возможно, остатки одежды или лака). Наличие Rb в золотых нитях свидетельствует о существование собственной добычи золота и технологии его обработки на территории древнего Киева. Отсутствие Cu, которая присутствует в наиболее старой ткани, в объектах, произведенных в XV–XVI веках н.э., может быть связано с улучшением технологии выделения золота. Различие в содержании элементов во всех исследованных нитях показывает, что они были произведены в различных регионах древней Руси.