Kraków 2009

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TELL EL-FARKHA 2007
MINERALOGICAL AND PETROGRAPHICAL INVESTIGATION OF GRAVE No. 55*

Excavations at the Tell el-Farkha, Kom E, tomb No. 55 in the Nile Delta, brought to light a variety of substances and artifacts. Mineralogical and petrographical examination of objects discovered has been carried out, in order to identify and establish the primary sources of materials unearthed. The results are presented below.

Methods of investigation

The samples collected were examined using digital microscope (DM), polarized light microscope (PLM), scanning electron microscope (SEM), as well as X-ray diffraction (XRD) techniques. PLM of Carl Zeiss brand was used, with the phenomena observed documented in series of microphotos. SEM Jeol 540, coupled with the EDS counter, was used for determination of morphology and chemistry of tested samples. XRD analysis focused on identifying the products of copper corrosion. Philips diffractometer and Cu Kα radiation were used, while the interpretation of results was conducted using X-ray software.

1. Fragment of a melting pot (Figs. 1 A-D; 2 A-B, 3 A-C)

Preliminary observation using DM confirmed granular structure of fragment of vessel and the presence of pores and inclusions of metal. Metal inclu-

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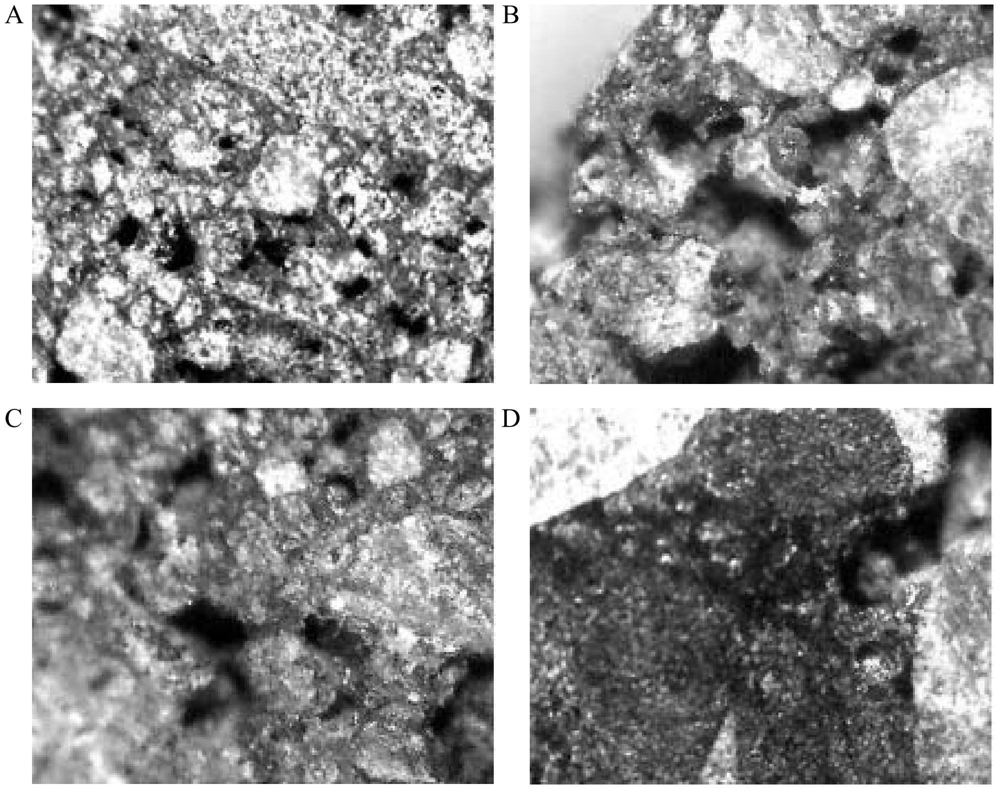


Fig. 1. A – Structure of the small black spots of metal disseminated at thermally altered slag. DM, magnification 40 x; B – Dark pores present at melted slag. DM, magnification 40 x; C – Dark pore present at slag; D – Dark concentration of metal

sions are disseminated irregularly in form of small grains. They are located mostly at places where are present small pores. Observation of structures of sample shoved the presence of secondary phases formed at high temperature (Phot. 2A) as well as inclusions of metal containing secondary phases formed due to high temperature and cooling of material (Phot. 2B). Mentioned phenomenon documents heating of vessel at high temperature. Data obtained with using DM as well as PLM informed about heating of vessel at high temperature. Because of this next examination was focused on determination of metal inclusions present at the vessel. The examination performed using SEM and EDS methods confirmed the presence of various metal inclusion (Phot. 3A-C). Mentioned inclusions are small and of size mostly smaller than 20-50 μm.

Chemical investigation of metal inclusions (Fig. 3 I-III) performed using EDS method showed that they contain Cu, Fe, Pb, Mn, traces of Ni, Cr, Sn. Together with metals are present non metallic phases including Al – silicates containing Ca and traces of Mg.

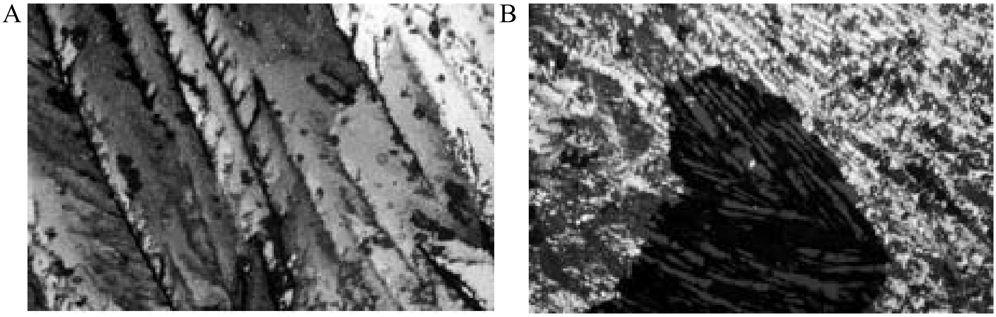


Fig. 2. A – Structure of secondary formed transparent phases. PLM, N part X, magnification 80 x; B – Inclusion of metal with isotropic phases. PLM, N part X, magnification 80 x

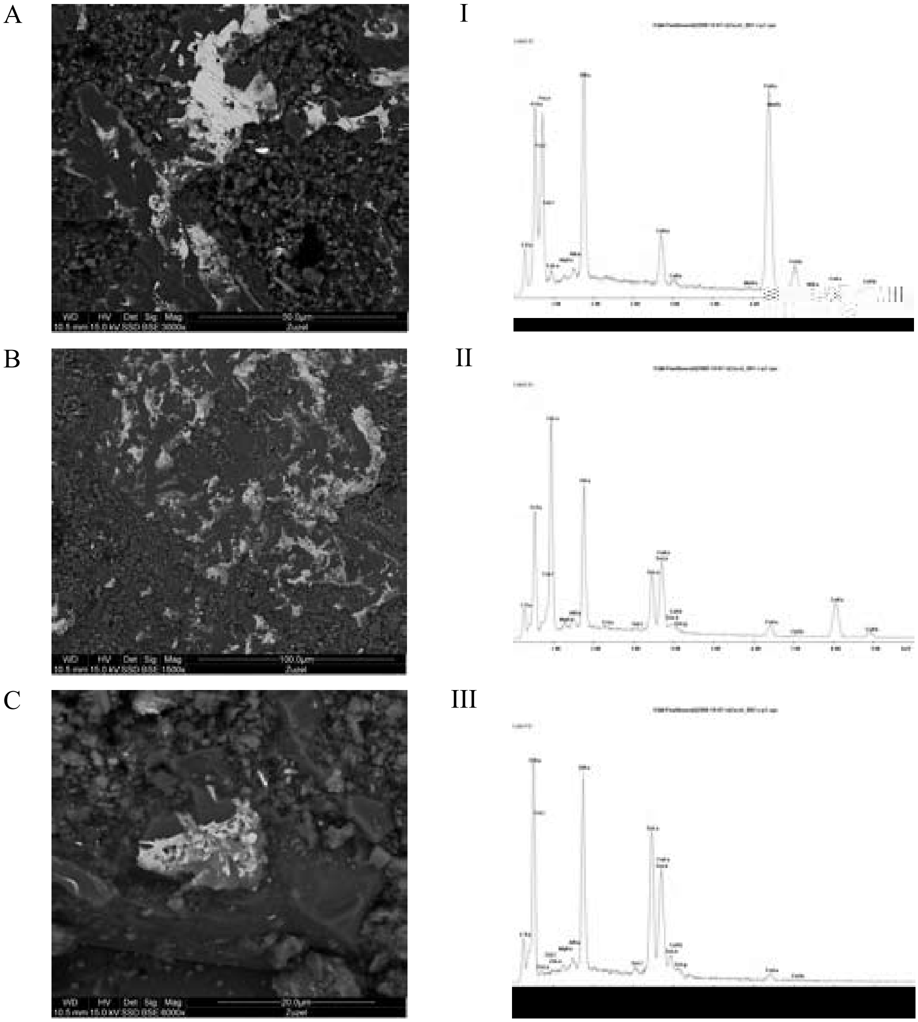


Fig. 3. A-C – A-C Light concentration of metals at the wall of the melting pot. SEM; I-III – EDS diagrams of light metals showed at photos 3A-C

It is necessary to stress that during exploration of tomb No. 55 two copper harpoons were discovered (see below, sample No. 13). Examination of not weathered metal present inside of objects showed not homogeneous structure and chemical composition. Observation of structure of metal performed using SEM method documents the presence in pure Cu inclusions containing admixtures of As, Ni in form of dark inclusions. Together with the mentioned forms one can see in pure copper small crystals of sulphides.

Together with copper harpoons small fragments of slag containing traces of metal were discovered (see below, sample No. 8). Microscopic examination of slag conform it is composed of glass, not crystalline substance containing various inclusions of gas as well as solid phases. Not transparent inclusions present suggest the slag is the result of metal production – melting.

Investigation confirmed that the fragment of vessel is fragment of a melting-pot used for copper production. This discovery is also confirmed by the presence of copper harpoons and grains of slag at tomb No. 55. The origin of copper is up to now not known. But according to literature, deposits of Sinai or Jordan may be taken into account[1].

2. Yellow pigment (Fig. 4)

Microscopic observation showed a fine-grained carbonate substance, displaying optical features typical of siderite. Together with carbonate, disseminated opaque limonite grains were present. Powdered material is of the yellow-reddish colour.

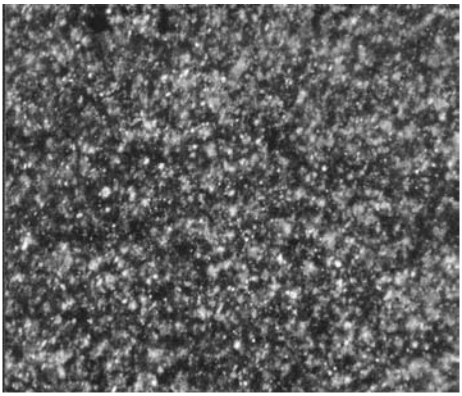


Fig. 4. Yellow pigment – siderite mixed with limonite.
PLM, polarization X, magnification 120 x

[1] S. Klaein, A. Hauptmann, *Iron Age Leaded Tin Bronzes from Khirbet edh-Dharih, Jordan,* Journal of Archaeological Science 26, 1999, p. 1075-1082; A. Hauptmann, *Zur frühen Metallurgie des Kupfers in Fena/Jordanien*, Deutsches Bergbau Museum, Freiburg 2000, p.238; Mikoś T., *Górnicze skarby przeszłości. Od kruszców do wyrobu i zabytkowej kopalni*, Kraków 2008, p. 231.

3. White pigment (Fig. 5)

Characteristic of this sample is a crystalline – porous inner structure with white-beige crystals of calcite, that makes the pigment soft and easy to use.

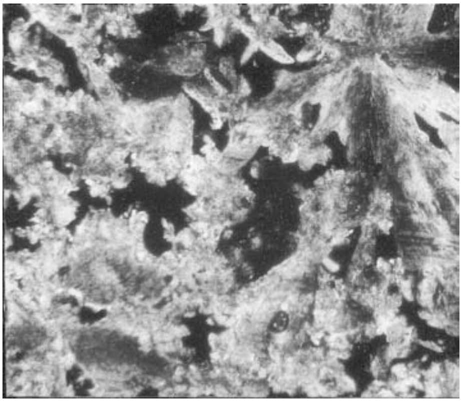


Fig. 5. White pigment- light crystalline calcite with dark, empty spaces between crystals creating a soft structure. PLM, polarization X, magnification 120 x

4. Reddish pigment (Fig. 6A-B)

Investigation by means of a DM showed the presence of small, rounded grains of hematite with inclusions of small quartz grains. Further study, carried out with a PLM, confirmed the presence of hematite cemented with a silty mass.

A B

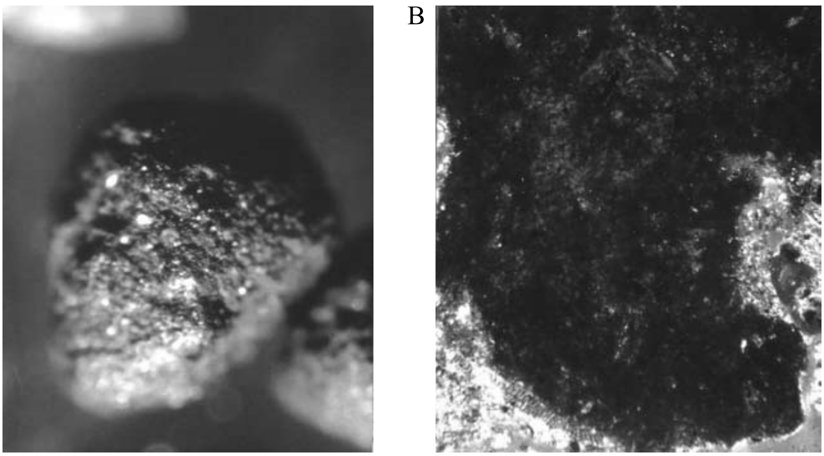


Fig. 6. A – grain of hematite separated from the reddish pigment. DM, magnification 60 x; B – grain showed at photo 3A under PM. Red hematite with inclusions of small quartz grains, surrounded with silty material represented by clay minerals and fine quartz. PLM, polarization X, magnification 120 x

5. Black pigment (Fig. 7)

Inner structure of fine-grained sandstone, cemented with black manganese minerals. Grains of sandstone are represented by quartz, rounded fragments of flints and, occasionally, altered feldspars. The exact function of the black pigment in this particular case remains unknown. It is valid however to speculate that it was imported from the Eastern Desert as the cretaceous sandstones of the Western Desert do not contain this kind of manganese cement.

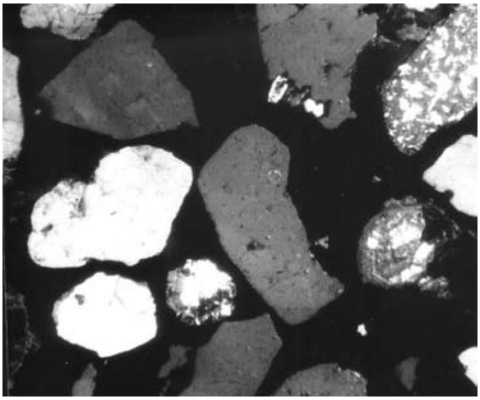


Fig. 7. Black pigment – fine-grained sandstone, cemented with black manganese oxide. PLM, polarization X, magnification 120 x

6. Burned clay coating the internal walls of a vessel used as a gasket (Fig 8 A-C)

Study of the sample's structure, carried out by means of a DM, revealed the presence of small cracks (Fig. 8A) and remains of burned organic matter, seen

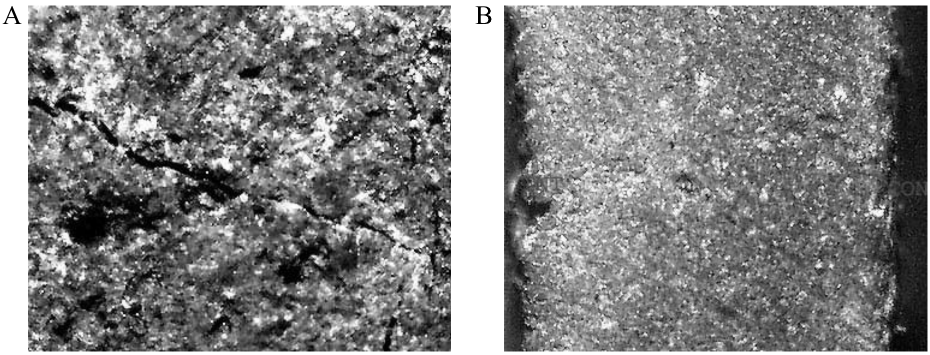


Fig. 8. Thin layer of fired clay from the internal wall of the vessel used as a gasket.
A – surface of the gasket. One can see small black cracks and dark spots of organic matter;
B – cross section of the gasket. Reddish, internal part (INT) – oxidized.
Grey part at the point of contact with pottery (CON) – not oxidized. DM, magnification 40 x

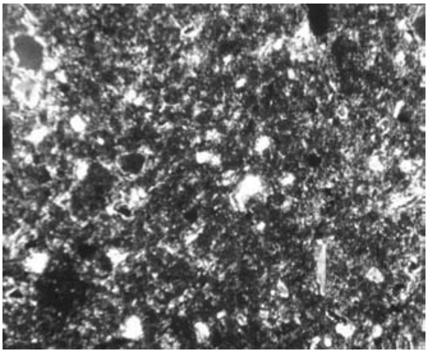


Fig 8. C – The structure of clay used as a seal on the internal wall of a vessel for storing liquids. PLM, polarization X, magnification 120 x

below as dark spots. Analysis of the cross section confirmed, that the clay used as a gasket was fired together with the vessel as indicated by the reddish colour of the internal clay walls (Fig. 8B). This, in turn, suggests that the artifact was used for storing liquids (beer jug). Remains of the organic substance, seen as dark spots on the surface of the sample, are unfortunately too scarce to allow for an identification. Microscopic observation of the clay's thin section provided a picture of very fine silts, seemingly specially prepared for sealed vessels (Fig. 8C).

7. Fragments of a vessel of "Egyptian alabaster" (Fig. 9A-C)

Investigation into the surface of the vessel shows the difference in morphology between the external and internal walls, which are coated with traces of various organic substances (Fig. 9A, B). Under the polarizing light, "Egyptian alabaster" shows porous structure where its crystals are separated from one another by empty, elongated fissures (Fig. 9C). Polarizing colours of crys-

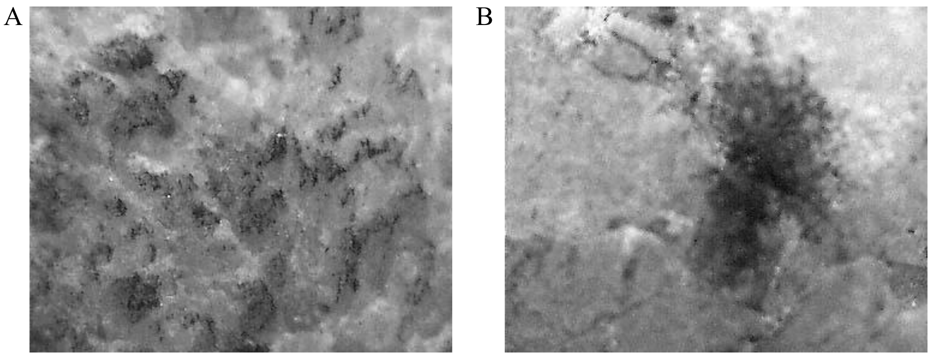


Fig 9. Surface of the vessel coated with traces of an organic substance. A – external surface of the vessel coated with soot; B – internal surface with preserved traces of the organic matter stored in the vessel. DM, magnification 85 x

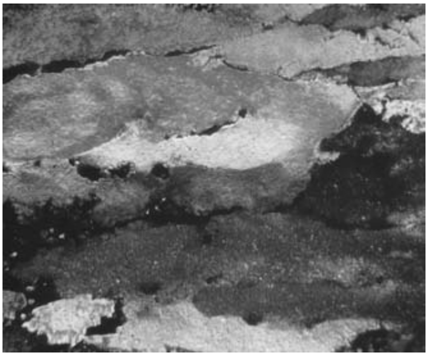


Fig 9C. The structure of Egyptian alabaster of the vessel tested. One can see elongated crystals of gypsum altered partially into anhydrite. Crystals are oriented perpendicularly to internal and external surfaces of the vessel. The dark pores seen between crystals signify a relatively high porosity of the vessel wall, which in turn suggests that it wasn't used for storing liquids. PLM, polarization X, magnification 120 x

tals confirm that a part of gypsum crystals was altered into an anhydrite. This phenomenon is probably the result of the reaction (dehydration) between the gypsum crystals ("Egyptian alabaster") and a substance present in the vessel, as there are no traces of high temperature alternation of the vessel.

8. Slag (Fig. 10A-B)

Microscopic examination of the sample confirmed that it is composed of glossy, not crystalline substance containing various inclusions of gas, as well as solid phases. The investigation does not specify the origins of the slag – whether it is a result of the metal (copper?) melting or of firing at a very high temperature. Though opaque inclusions present suggest the former situation, further investigation by means of electron microprobe method is needed.

A B

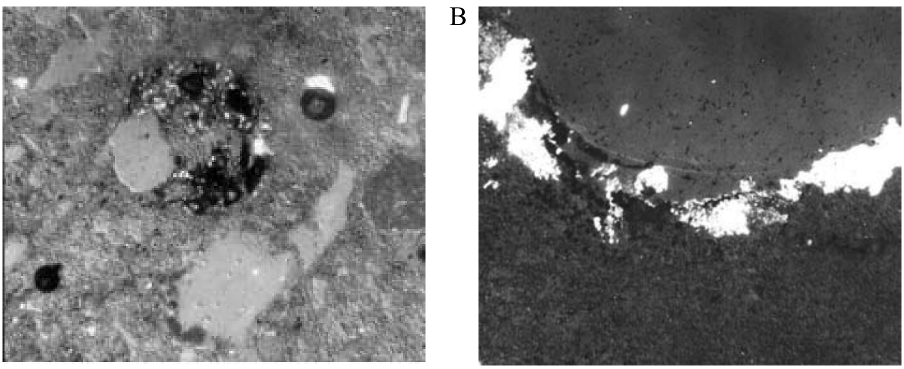


Fig. 10. Microscopic pictures of the slag tested. A – structure of slag containing transparent and metal inclusions; B – quartz and calcite grains (light) on the internal surface of a small gas inclusion present in the slag. PLM, polarization in part X, magnification 120 x

9. White clay (Fig. 11)

Clay is a composition of kaolinite, illite/sericite and small, rounded grains of quartz. There is a possibility, that it was used as white pigment. Because of the admixture of kaolinite, stable at high temperatures, this substance was much more useful for decorating e.g. heated pottery, than thermally unstable powdered calcite or gypsum. Though the origins of clay are unknown, it is definitely imported as all the local clays and silts are of gray colour.

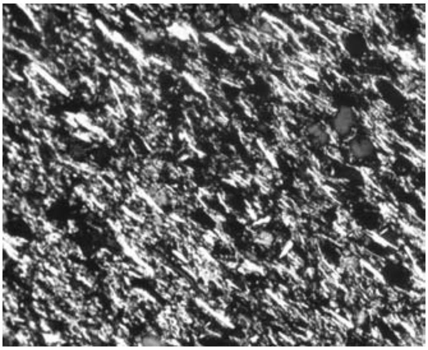
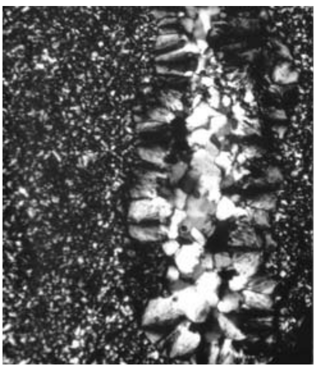


Fig. 11. Structure of the white clay (pigment?). One can see small light flakes of sericite, fine gray mass composed of kaolinite and fine grains of quartz. PLM, polarization X, magnification 120 x

10. Beads of agate (Fig. 12A,B)

They are of red-brownish, red and orange colour, with the majority of them unearthed already damaged. Under the microscope they present a variety of hues and quite often do not possess a zonality typical for this mineral – the bead tested represents just this sort of agate. It is transparent and of a red colour. Its

A



B

Fig. 12. A – a zone built of fine quartz (light bigger crystals) and chalcedone (fine light points); B – a zone of agate built of coarser quartz and chalcedone. PLM, polarization X, magnification 120 x

external surface is polished and the hole shows morphology characteristic for preparation with the use of a crescent drill, most probably made of flint. Internal structure of the agate is also typical – under the PLM one can see zones constructed with quartz of various crystallinity, with fine and coarse crystals of chalcedony and quartz.

Agates, like the one tested, occur plentifully in Egypt, their primary sources being magmatic, ingenous rocks. They are especially easy to collect in the desert, where they often occur in the secondary position on top of Qena sand and also in younger sediments. Those discovered at Tell el-Farkha however, are not of local origin but were transported from areas located outside of the Delta, mostly from Upper Egypt where they appear frequently in the top layers of Qena Sands[2].

11. Faience with white glossy coat (Fig. 13)

Microscopic observation of the sample's internal structure shows fine, sharp grains of quartz (light, gray), cemented with isotropic glossy substance. The relation between the quantities of those two elements implies the extra strength of the glossy cement. One can also see small, irregular concentrations of brownish substance – another component of the faience mass.

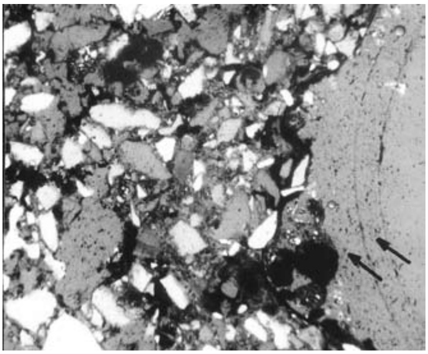


Fig. 13. Microscopic picture of the faience bead cross section, near the internal hole. One can see mixed quartz-glossy mass and a part of the internal hole coated with two layers of glass (arrows). PLM, polarization in part X, magnification 60 x

[2] Pawlikowski M., *Climatic changes during Holocene in the region of Armant* [in:] B. Ginter, J.K. Kozłowski, *Predynastic settlements near Armant*, Heidelberg 1994, pp. 125-132; Pawlikowski M., *Reasons for the Predynastic – Early Dynastic Transition in Egypt. Geological and Climatic Evidence,* [in:] Hendrickx S., Friedman R.F., Ciałowicz K., Chłodnicki M. (eds.), *Egypt at its Origin*, Leuven 2004, pp. 919-923; Pawlikowski M., *Sedimentary Structures and Mineralogy of Tell el-Farkha Archaeological Site (Nile Delta – Egypt), International Symposium Prehistory of Northeastern Africa. New ideas and discoveries*. Poznań 2007, pp. 16-17.

The presence of two layers of glass coating the external walls of the object, as well as the internal hole of the faience bead suggests at least two stages of the coat's preparation. Observation suggests, that the bead was prepared first as a "ceramic mass", coated with glossy light substance, before being additionally covered with a second layer of a very thin glossy substance, containing small admixture of blue pigment. The aforementioned technology is the reason for the good condition of the older light glass cover and the almost complete corrosion of the thin blue coloured external layer of glass. This is why we find light (not blue) faience beads at Tell el-Farkha.

12. Organic substance coating the external walls of a vessel of "Egyptian alabaster" (Fig. 14A-B)

As has been stated above, "Egyptian alabaster" vessels could not be used for storing liquids due to the high porosity of the mineral's structure. Therefore, the black (burned?) organic substance present on the external walls of many vessels (Fig. 14A) found at the site wasn't originally filling them. As the remains of the aforementioned matter occur only occasionally inside the Egyptian alabaster artifacts, the process of coating must have been of a later date and focused mainly on the outside of the vessels. There are grounds for supposing, that the organic matter under investigation is a natural product of decomposition of the primary organic substance, currently unidentified. One can observe that this secondary organic matter spread in various directions and coated various objects present in the tomb.

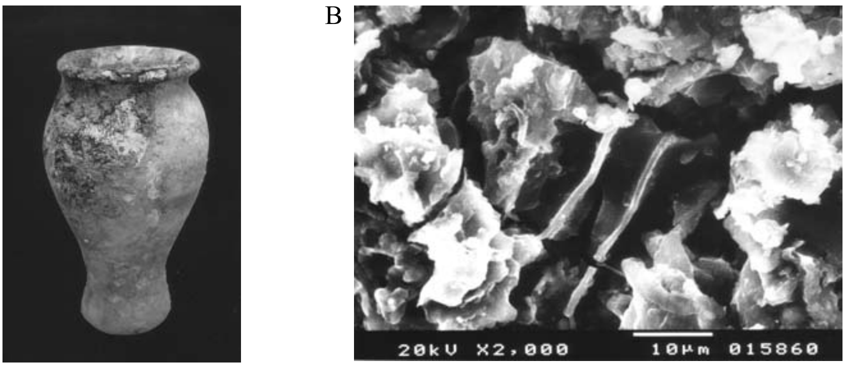
A


Fig. 14 A – vessel made of "Egyptian alabaster" with the visible part of its external wall coated secondarily with black organic substance. B – SEM picture of the black organic substance from the vessel's wall. One can see the organic mass in a structure containing small unidentified organic fragments (central part of the photo)

13. Copper harpoon and products of corrosion of copper points
(Fig. 15A-C)

An investigation of the copper pike heads has been conducted, with the analysis of both the fresh metal, as well as the products of corrosion by using SEM and EDS methods.

Examination of the unweathered metal present inside of the objects, showed heterogeneity, both in the structure and chemical composition. Apart from the pure copper, inclusions of Cu, containing admixtures of As and Ni in form of dark inclusions, appear (Fig. 15B, 15 I). Also, small crystals of sulphides (Fig. 15C, 15 II) can be distinguished. Those inclusions, as well as the very form of the pikes confirm, that the metal used was not melted but rather hammered. Forms of inclusions and their chemical and mineral composition exclude the process of copper melting because of the presence of not oxidized sulphides.

The above observations suggest the probability of pure natural copper being used in the making of the pikes, instead of copper minerals. On the other hand small fragments of slag were also discovered in the same tomb. Detailed explanation of the interrelations between slag and the pikes needs further investigation.

Transition zone between copper and the products of corrosion

Investigation of this zone within copper objects is of great importance for our understanding of the corrosion processes occurring in the specific environment of a tomb. As the body tissues undergo decomposition, a large number of secondary components is formed, some of which may react with metals. Analysis of the zone mentioned by means of SEM (Fig. 15C) showed irregularity of the border line between the two spheres.

Differences observed are due to the heterogeneous structure of the metal, containing various inclusions and admixtures. As a result, the oxygenated (corroded) outer layer, formed during the process, is of various thickness in different parts of the pikes surface.

Products of copper corrosion

Green colour of the faience beads, observed under the microscope, suggest the presence of copper sulphates and carbonates as major products of metal corrosion. SEM investigation of the altered layer confirms the crystalline character of the corrosion products (Fig.12 E,F). Crystals represent mainly oxides and chlorides of copper i.e. paramelaconite, paratacamite and atacamite (Fig. 12 IV, V, VI). Also, corrosion products of amorphous structure are present.

Both the products of copper corrosion and the conditions found in the tomb are specific. Oxides of copper are dominating but the presence of copper chlo-

Fig. 15. A – harpoons discovered in tomb No 55. One can see small variations in the shape of artifacts due to the technology of production

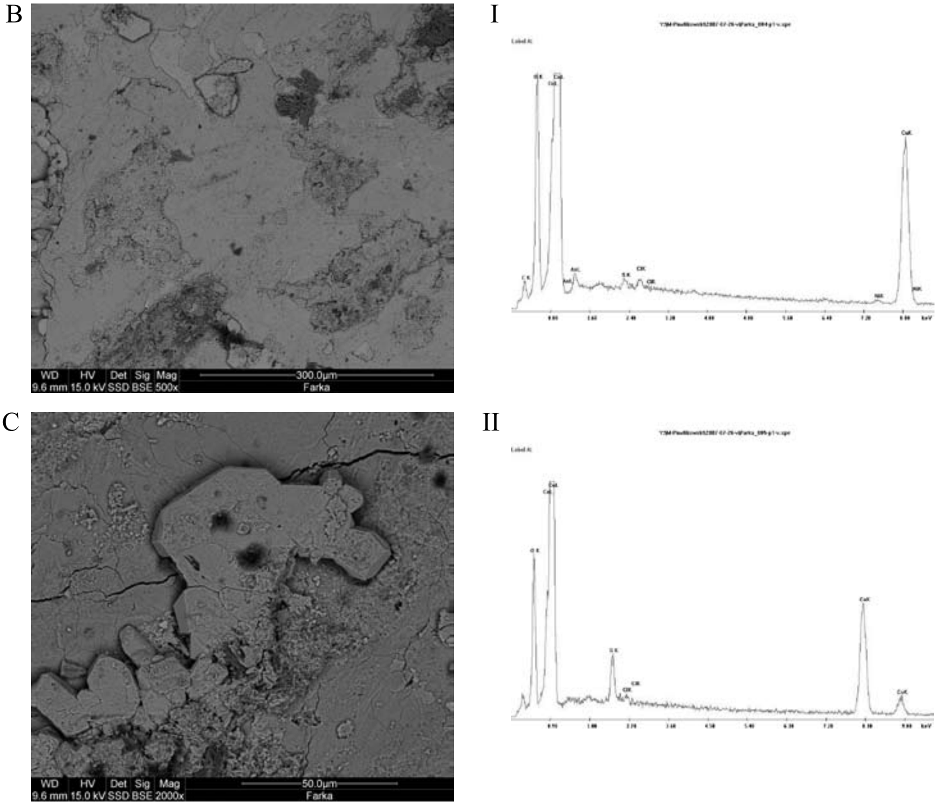
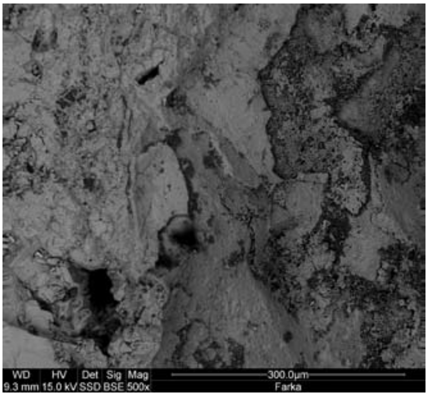


Fig. 15. B – pure copper with inclusions of dark grains containing As and Ni; C – crystal of sulphide present as an inclusion in the copper. SEM; I – EDS energetic curve of dark inclusions present in the copper. One can see small peaks of As and Ni; II – EDS energetic curve of the sulphide crystals – inclusion in the copper

D



III

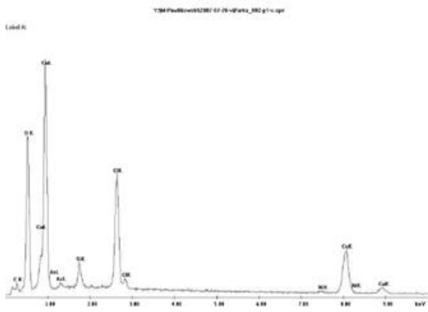
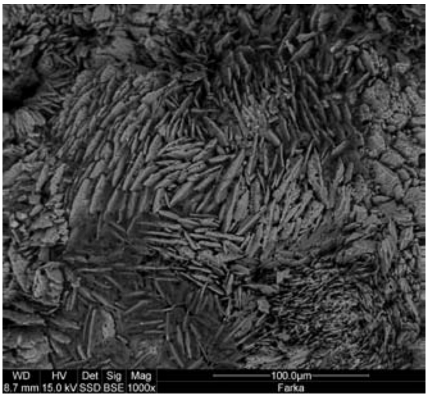
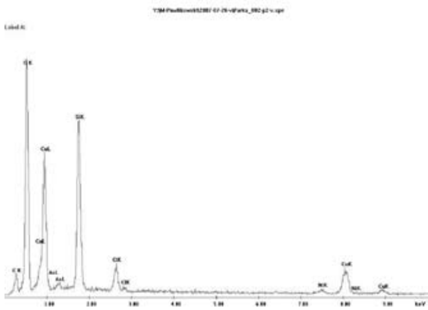


Fig 15. D – Contact zone between pure copper and corrosion products. Copper – left, lighter part of the photo; products of corrosion, dark “islands” and “peninsula”, right part of the photo; SEM; III – EDS energetic curve of the copper corrosion zone. One can see peaks of Cl and S

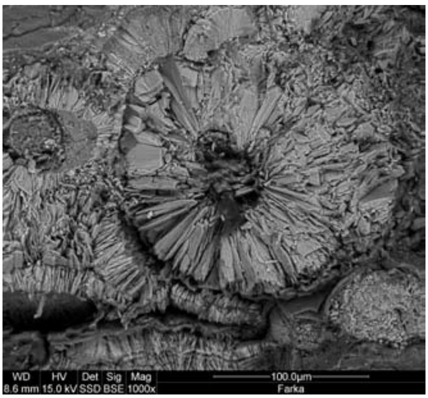
E



IV



F



V

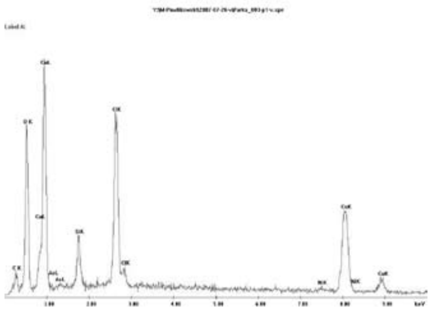


Fig. 15. E – Crystals of corrosion products (mixture of paratacamite and atacamite) covering the surface of the copper pikes; F – spheroidal form of the copper corrosion product of the secondary crystallization– atacamite; IV – EDS energetic curve of the crystals shoved in photo 12D; V – EDS energetic curve of atacamite

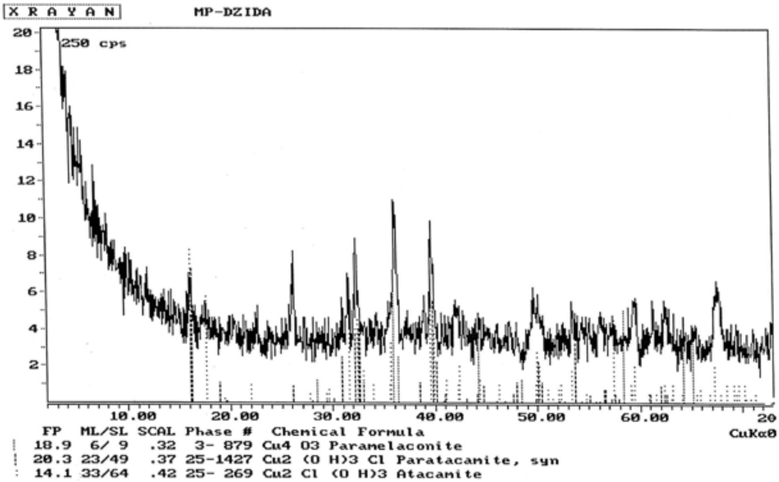


Fig. 16. Xray diffraction pattern of the corrosion products coating the copper pike from Tomb No 55, Kom E

rides is very interesting due to their presence at the Tell el-Farkha site. The absence of Na suggest that primary chlorides did not represent halite (NaCl). Natural salinity of the underground waters have to be therefore excluded. Another important source of the chloride are human bones, where parts of the OH groups in the structure of hydroxyapatite are substituted by Cl anions. It is highly probable, that the chloride liberated from the bone structure during the body decomposition process, reacts with atmosphere oxygen and with copper. Therefore, the secondary products of copper corrosion could be of a mixed human-metal origins. Nevertheless, one must consider a possibility of chlorides being used in order to preserve the human body discovered in tomb No 55 (a simple, very early mummification). Confirmation of this hypothesis requires further, more detailed examination. While discussing the results of Cu – harpoon investigation, the problem of the copper's primary sources has to be considered. There are no large copper deposits in the Western Desert, the Nile Valley and Delta, while in the Eastern Desert and Sinai the copper is present only occasionally and only in small number. The biggest deposits of copper are known from Wadi Fayran – Jordan[3] which supplied Mesopotamia and Egypt. Mining and metallurgical tools had been in use there as early as 5000 BP[4].

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[3] Klaein S., Hauptmann A., *Iron Age Leaded Tin Bronzes from Khirbet edh-Dharih*, *Jordan*, Journal of Archaeological Science 26, 1999, p. 1075-1082.

[4] Hauptmann A., *Zur frühen Metallurgie des Kupfers in Fayran/Jordanien*, Deutsches Bergbau Museum, Freiberg 2000, p. 238.

Conclusions

Our investigation brought to light a number of facts concerning both the human activity and also natural processes occurring at the site after it was abandoned. All the results additionally obtained, confirmed the necessity of engaging advanced scientific methods while working on the details which traditional archaeological methods cannot fully examine.

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