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DIPLOMA THESIS:

"Investigation on weathering and surface depositions on marble Cycladic figurines"

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to my brother Manolis

The present MSc Dissertation was conducted at the Laboratory of Archaeometry, the Institute of Material Science at the National Center of Scientific Research "Demokritos" during the academic year 2015-2016 under the supervision of Dr Yannis Maniatis and Dr Vasilis Kilikoglou. The facilities and equipment of the institute was used during the research.

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Abstract

The island of Keros and the islet of Dhaskalio are two of the most important archaeological sites in the Cyclades, dating to the Bronze Age. According to Prof. Colin Renfrew these two sites are considered as the first maritime sanctuary in the world. This theory is based on the hundreds figurine fragments and marble vessels found in the Special Deposits South and North (looted) as well as the settlement of Dhaskalio.

In this work, a brief report on the history of the excavations and surveys at these areas is presented, as well as, the typology of the figurines and the geology of Keros Island and the geology and soil micromorphology of the Special Deposit South. A theoretical chapter of the marble weathering is discussed and the results of the preliminary research by Maniatis and Tambakopoulos in 2015 are reported.

The experimental part of the study is focused on the scientific examination and analysis of the weathering state of the marble and the depositions on the surface of a representative number of marble figurines and the red deposition of a fragment of a marble basin. The analyses were performed using a stereoscopic microscope, a petrographic microscope and a Scanning Electron Microscope with analytical equipment. The samples were examined and analyzed in two forms: (1) as received (ASR) and (2) in polished cross-sections. The examination and analysis showed cracks of different size going through whole marble grains and soil depositions formed at the surface of the grains entering at places inside the grain boundaries. A sequence of weathering events could also be detected. These indicate that the figurines were severely weathered under different environmental conditions and episodes. The intense red depositions on the interior of the marble basin were identified as the valuable mercury sulfide pigment Cinnabar.

For future research it is suggested that more figurines from Special Deposit South as well as from Special Deposit North, in combination with marble objects from Dhaskalio, should be examined and analyzed to see the difference in weathering in relation to the geological environment and soil. Other techniques such as FTIR, could give more details about the organic depositions on the grain surfaces.

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1. Introduction

Keros Island has an area of 15 km^2 and the highest point is 432 m. Keros was an important site during prehistory and mainly during the period of the Cycladic civilization, about 2500 BC. During the last decades the island is not inhabited. Excavations and surveys are organized since 60s until nowadays. Important settlements and findings came to surface (numerous fragments of Cycladic figurines, frying pans, marble vessels and metal objects). A number of artifacts are exhibited in the National Archaeological Museum of Athens and in the Archaeological Museum of Naxos. The majority of the material is stored at the Archaeological Museum of Naxos.

Fig. 1: Map of Keros and Small Cyclades (googlemaps.com).

The archaeological sites are mainly dated during Early Cycladic II period (2700-2300 BC) (dating Keros-Syros phase 2750-2550 BC, Early Kastri phase 2550-2400 BC, and ECII 2400-2300 BC at Dhaskalio). Among Keros, other important sites are establishing that period such as Kea, Ios, Naxos etc (Renfrew 2009).

Following the evidences and the findings, prof. Colin Renfrew came to the conclusion that the site may be the oldest maritime sanctuary in the world (Renfrew et al 2012). The combination of new technologies (radiocarbon dating, Bayesian analysis etc.) and the typology of the findings and the deposits in which the artifacts were buried, led the researchers to the conclusion that Keros should be a sanctuary like Delos Island (later period). The population of the sites Kavos and Dhaskalio was approximately 100 and 300 people (Broodbank 2000). Hundreds of broken marble figurines found at the areas of Special Deposit North and South, vessels and pottery show a ritualistic mission (Renfrew et al 2012).

Finally, Keros has suffered in the past from extensive looting and damaging of the sites causing the loss of valuable information which could have provided crucial information to the researchers (Marthari 2001). Thousands of objects had been sold illegally all over the world. Fortunately, the archaeologists manage to uncover areas that they have been unattacked by looters during the past centuries.

2. Keros Island: the history of the surveys

The last decades the archaeologists are interested to explore and learn more about the prehistory of the Aegean region, and especially in the area of Crete and Cyclades. Since the 1960s, archaeological investigations on Keros, held by foreign and Greek institutions, are bringing unique findings in the surface. Keros is defined as the oldest Maritime sanctuary (Renfrew et al. 2012). Colin Renfrew approached the island during 1963. Rescue excavations by Ch. Doumas and Ph. Zapheiropoulou were followed in 1987 in combination with systematic survey and excavation in the looted area. The leading team were Ch. Doumas, L. Marangou, and C. Renfrew (Renfrew et al 2007). It was the time that the special deposit was discovered. The Cambridge Keros Project of 2006 to 2008 led to the discovery of a new and previously undisturbed special deposit, now termed as "Special Deposit South". During 2013/2013, surveys were completed around the island, giving a clear image to the archaeologists about the habitation of the island.

2.1 The excavations during the years 2006-2008

The exceptional nature of Kavos and the importance of the settlement at Dhaskalio at Keros were well investigated during the excavations between 2006 - 2008.

The first excavation started during the spring of 2006. The University of Cambridge was leading the program (Cambridge Keros Project). They started excavating Kavos area and the Special Deposits (North and South). The structure of the Special Deposit South (fragmented materials - thousands of pieces –pottery marble figurines and marble vessels-) that could possibly mean that these materials were brought to Keros island from other islands of Cyclades perhaps also from the mainland of Greece (Renfrew et al 2007). During 2007, they started excavation on the rocky islet of Dhaskalio opposite to Kavos. The excavation revealed several structures such as prehistoric walls (dated on Early Bronze Age II). Dhaskalio was a major settlement of the Keros-Syros culture (Renfrew et al 2007).

Fig. 2: Fragments of figurines and marble vessels. (Renfrew et al 2007).

Special material were separated into categories, got a unique number and photographed in situ. Every object in the Special Deposit had been deliberately broken (only few small objects had escaped destruction). Flotation system was proceeded for collecting small finds and for the paleoenviromental research (Renfrew et al 2007).

Dhaskalio is 200m long islet, and rises to a height of 38 m above sea. The topography makes terracing desirable for building construction. During prehistory, probably there was a narrow natural path connecting Dhaskalio and Kavos, which automatically forms these areas to a desirable harbor both to north and south (Renfrew et al 2007). Other studies that were ongoing during the excavation were the geomorphological study, pottery, metal, marble, obsidian and stone studies, new technologies and dating studies.

2.2 Keros Island Survey 2012-2013

During these years the researchers managed to clarify in detail the extent of wider habitation on Keros. According to the results, the first evidence of habitation is dated to the middle/late Bronze Age, and continued to the archaic to Hellenistic, and the late Roman to early Byzantine period.

Fig. 3: Documentation procedure and walking distance between the walkers – Keros Island.

The information about this paper were taken from the publications on the "Annual Report 2011-2012" and the "Annual Report 2012-2013" published by BSA (British School at Athens). The project was held by C. Renfrew (Cambridge), M. Marthari and E. Dellaporta (Ephotrate of Cyclades).

First season

At the first season, the island was divided into 1584 one-hectare tracts, (764 tracts were walked). The aim of the survey was the identification of new areas with archaeological materials, possible settlements (Morgan and Sherratt 2012). The project was organized with GIS techniques.

The researchers were focused also on creating a new geological and geomorphic maps. Specialists organized the geological and geomorphological study, concentrated also on cross-sections of the island, and the recording of geomorphological features, soil types and land-use practices. Radio carbon dating (^{14}C) samples were taken where cultural material was noted. Soil micromorphology and OSL dating were applied. The extensive terracing systems were also investigated. Coring was conducted in specific terrace infills and foundations (Morgan and Sherratt 2012).

An important task for the archaeologists was to be able to understand the diachronic picture of the whole island. Terracing systems (water management systems, sediment capture systems, and agricultural terraces) underlined the diachronic picture of the island. The sea level was c. 3.5–5 m lower during the Early Bronze Age. Bronze Age material now on the shore (where not washed down) relates to areas formerly inland (Morgan and Sherratt 2012). On the southwest side of the island, levels of cultural material are generally low (closer to sea level).

Second Season 2012-13

During the second season, the team walked further 445 tracts, including some of the more remote and mountainous parts of the island. Intensive collection was carried out at 25 “polygons” (in 5 m^2 circles, with diagnostic ceramics and special finds collected in 100 m^2 squares) (Morgan and Sherratt 2013).

An enthnoarchaeological study was organized based on the toponyms the “modern” names of each area, to detect the use of land, the material culture and the abandoned architectural remains of the last century (Morgan and Sherratt 2012). Finally, due to the stories of looting that’s happening in the islands since 50s’ and 60s’ a search was organized for looted Early Cycladic cemeteries, recording cavities and structures, and relating them to finds from the intensive survey.

3. Cycladic Figurines

Early Cycladic marble figurines were found on different islands of the Cyclades as well as Crete and mainland. The figurines are made of white marble and commonly were decorated with painted patterns. The majority of the figurines represent female figures standing, with only few figurines of males (Hendrix 2003). Sometimes the female figurines are shown pregnant (dominates throughout), the male figurines account only the 5% of the total known number (Getz-Preziosi 1987). The interior details are emphasized, like neckline, pubic triangle, spine, buttocks, legs, knees and sometimes fingers and toes. However, facial features are not represented (usually painted). Most Cycladic figurines are quite generic, with feet angled downward, legs together and bent slightly at the knee, arms folded across the chest (left over right), and face forward and occasionally tilted upward (Hendrix 2003). The figurines vary in size from 5 cm to 1.5m (Renfrew 1969).

The archaeologists manage to recognize varieties and sub varieties while they were trying to keep a chronological order. The Cycladic figurines of Early Bronze Age (3200-2000 BC) are separated into three basic categories, schematic, naturalistic and hybrid (Sotirakopoulou 2005). Briefly, the main characteristics of each category are presented below[1]:

Schematic figurines

There are separated into twelve types, violin, violin–like, shouldered, spade, notch-spade, spatula, pebble, multipartite, Troy, Beycesultan, Apeiranthos and Phylakopi I or Agia Irini type.

Naturalistic figurines

This figurine category is divided into the types of Plastiras, Louros, precanonical, canonical, postcanonical and special type.

Plastiras type is the earliest approach of naturalistic representation of human figure, while *Louros type* is combining characteristics of the schematic and naturalistic categories.

The *canonical type* is distinguished into five varieties, Kapsala, Spedos, Dokathismata, Chalandiani and Koumasa, taking the name of the site where found first.

-Spedos variety is one of the large in terms of amounts of figurines of the different types. The variety is the most widely spread in Cyclades as well as outside the Cyclades (manufactured for a long period) and it is mainly dated to EC II period.

-Chalandriani variety is mainly a female variety. The main characteristic is the triangle head with produced nose and a sketchy appearance. The legs are generally short and the knees are straight.

[1] The reference is based on the study of Peggy Sotirakopoulou "The Keros Hoard, Myth or Reality". The study is focused on a large amount of figurines mainly looted and possibly coming from Keros and the Special Deposit North. Provenance studies by Maniatis et al. (2003) were complete in terms of identifying the marble sources.

-Koumasa variety is mainly found in Crete and are imitations of Dokathismata and Chalandriani variety.

The *postcanonical type* has similarities to Chalandriani variety and general the canonical type, however, there are difference between the positioning of the forearms.

At the *special type* figurines belong all the male figurines in action, for example the hunter/warrior, the musicians, the flutists, the harpists.

Hybrid figurines

The hybrid figurines are combing characteristics of the two other categories, schematic and naturalistic (early), and are dated between EC I and EC II.

A large archaeological enigma is the Kavos-Dhaskalio site in Keros and the deposition of hundreds of marble figurine fragments at the areas now called Special Deposits North and Special Deposit South. The study showed deliberate fragmentation of figurines, marble vessels as well as pottery and this possibly makes the area a special place for ceremonial purposes (Renfrew 2013).

Decoration with pigments

During Bronze Age, different types of pigments for representing parts of the body. More specifically, the following characteristics were marked: the eyes, the hair known as “polos”, dots or strips on face, painted grooves (spines, head and neck), jewelry (necklaces and arm bangles), zigzags/stripes on body. In some figurines there were more than one eyes painted. It is possible that they were repainted after the first painting had worn away. The colors that were used are mainly black, red, blue (rarely green and yellow) (Hendrix 2003).

Painting layers can be observed on the inner surfaces of marble vessels (bowls, basins etc.), with red, blue or black color. It is possible that these vessels were used during the preparation of the pigment (Hendrix 2003).

Pigments, Cinnabar

Bronze Age pigments are mainly blood-red (hematite-iron oxide), yellow ochre (limonite-iron hydorxides), bright-red (cinnabar-mercury sulfide), deep blue (lapis lazuli), (pale) blue (azurite), green (malachite) and black (charcoal or bone ash).

The most common used in the figurines are charcoal black, hematite, Egyptian blue (synthetic lapis lazuli), malachite green, azurite blue and cinnabar red.

Cinnabar is a toxic mercury sulfide mineral with a chemical composition of HgS. Cinnabar’s chemical composition is mainly 86.22% Hg and 13.78% S[2]. The most striking property of cinnabar is its bright red color. It has a Mohs hardness of 2 to 2.5 (very easily ground)[3]. People began using cinnabar for pigments thousands of years ago in Greece, Italy, Spain, China, Turkey, and the Mayan countries of South America. Cinnabar is one of a very small number of minerals that was independently

[2] http://webmineral.com/data/

[3] http://www.mindat.org/

discovered, processed and utilized by ancient people in many parts of the world. In prehistory was used as mined, with known source Laurion.

Later, Cinnabar was mined at the volcano, ground into a very fine powder and then mixed with liquids to produce many types of paint. The bright red pigments known as "vermilion" and "Chinese red" were originally made from cinnabar[4].

Cinnabar has also been used in powdered form for ritual blessings and burials. Powdered cinnabar was also used as a cosmetics. After the identification of its toxicity its use in pigments, paints, and cosmetics began to decline.

[4] http://www.trueart.info/

4. Geological References

In the following chapter a brief presentation of the geology of the Cyclades and Keros Island is reported as well as the geology of the area Special Deposit South, where the samples of this research were found.

4.1. Geology of the Cyclades

The central Aegean area is consisted by all the types of rocks, igneous, metamorphic and sedimentary (Dixon 2013). The complex of the Cycladic islands are located in the middle of the Aegean Sea (more than 200 smaller and larger islands). The mountains are parts of the mountain peaks of the Aegais landmass (submerged about 5 million years BP). They are part of the Cycladic massif, ancient tectonic unit (Dixon and Kinnaird, 2013).

The Cyclades are composed by three geological group: the volcanic islands (Melos and Thera) at the southern part, the limestone and sedimentary rocks group at the south-eastern part, and the rest of the islands (south –western and northern islands) that are parts of the metamorphic massif and composed of a variety of crystalline rocks (such as granite, gneiss, marble and schist) (Dixon and Kinnaird, 2013).

Melos was the center for the exploitation and trade of obsidian. Neolithic and Bronze age settlements were established around mid-5th millennium BC. Cyclades became famous for the white marble. During early Bronze Age marble masterpieces were created such as vessels, figurines, tools, etc. During the later periods Archaic, Classical and Hellenistic, marble quarries were open and a massive exploitation was happening. There are evidence for marble quarries in many islands such as Paros and Naxos (cutting- tool marks, semi-finished marble pieces etc.), however, there are no evidence about quarries during prehistoric era. Possibly the prehistoric people were using the natural flaws and cracks for taking pieces for marble objects (Tambakopoulos and Maniatis, 2012).

4.2 Geology of Keros Island

The geology of Keros is mainly composed of marble. The marble has different color varieties (white, dark grey). It can be from very fine-grained and semi-crystallized to coarse-grained (MGS>8.0mm). It is Calcitic marble, while containing about 10% dolomitic. The fine-grained is poor quality marble, considered semi-crystallized (crystallized limestone) and can be found on several locations on the island. The coarse grained marble can be found in large massifs, forming the natural bedrock on Kavos and on Dhaskalio islet and on the east sharp cliffs and the north side of the island (its characteristics distinguish it from any other Cycladic or Aegean marble) (Tambakopoulos and Maniatis 2012).

The geological map of Keros (IGME) shows that the geological consistence is similar at the larger area of Keros, with the major geological unit to be crystalline limestones to marble. No evidence of quarrying or working on marble has been found anywhere on the island (Tambakopoulos and Maniatis 2012). Few areas are consisted by calcitic metatuffites and carbonate unconsolidated materials. Finally, the last geological unit is the rubbles and cobbles of carbonate to schist composition. That is the unit where most of the sites are located.

Generally, the relief of Keros presents steep slopes, high altitudes (up to 433m) and V shaped valleys. The steepest relief is found in the southern part of the island (many coastal cliffs). The West-Northwestern part of Keros has smoother relief in comparison to the southwestern part. At the top of the hill Platia Rachi, in the north, a planation surface was discovered orientated Northeast-southwest. The large settlement, which is located on the center of the island, is on that plato.

4.3 Soil history of Kavos

The landscape of Kavos Keros has been studied. The results of the investigation showed that already during Bronze Age, the landscape was eroded, extensively modified and open area. In special Deposit South, there are only few spots with buried soil survival. A modern topsoil can be observed created by human disturbance in combination with excavation soil, natural slope effect and environmental phenomena (winter rains etc).

Only one of the third of the in situ soil appeared to be palaeosol. Two types of soil have been observed, one has been noticed on hard limestone bedrock in other location in Keros[5]. Also, a brown Mediterranean soil type has been observed (found in upper slopes)[6]. They have formed as a result of weathering of the carbonate sedimentary bedrock. However, the trenches B4 and G1 are consisted by silt, pure and impure clay illuviation and iron – enriched horizon of a red Mediterranean soil. While D2 and A1 have strong reddening with amorphous iron ad fine fabric (in andamaged among) the bedrock (indicative of a red Mediterranean soil) (French and Taylor, 2015).

Geomorphological analysis of the trenches.

Geomorphological analysis on the trenches B3, BA, D1-D3 (and RA) was proceeded. The geomorphological analysis of the trenches D2, RA (where three of the figurines were found showed that there was irregular, small blocky, reddish brown, calcitic sandy clay loam with alluvial clay and organic matter fragments (much disturbed by looting)[7] and aggregate, calcitic sandy, silt loam with very strong

[5] It is also known as Chromic Luvisols.
[6] These brown soils can also be found in Ano Koufonisi.
[7] Reddish-brown Mediterranean soil.

amorphous iron impregnation and coarse bedrock fragment[8]. Charcoal, bone fragments, plant tissues and cells, excremental fabric were also included on the soil (French and Taylor, 2015).

5. Weathering Procedure

On this chapter the short description of the soil formation is been made, and the different types of weathering of marble are discussed. In addition, the deformation mechanisms are analyzed, the alteration procedure of the crystals and grains (of stones, and marble) inside the soil.

5.1. Soils formation

Soil is a mixture of minerals, organic matter, gases, organisms, liquids, it is called pedoshere (natural body). There are four important functions, it is important for plant growth, water storage (supply and purification), and modifier of earth's atmosphere and the area of the living microorganisms (Earle 2010).

The parameters playing important role on the soil and ground formation are the following: the parent material (usually rocky), the climate and the duration of the climate events, the relief or topography of the area, the vegetation, and the time (Earle 2010). As the most important, the parent material and the climate can be marked. The parameters affect one the other and different type of soils can be observed at one spot[9].

5.2. Types of marble weathering

Weathering takes place when a rock is exposed to the "weather" (Earle 2010). There are two different types of marble. The calcitic ($CaCO_3$) and the dolomitic, a complex carbonate containing both calcium and magnesium ($CaMg(CO_3)_2$). There are also mixtures of these two types. The types of weathering are based on the environmental conditions that the marble artefact is exposed to. Briefly, a description of these types are mentioned below:

Acidic environment: Soil can include different types of acid, such as humic acids (from biodegradation of organic matter), nitric or sulphuric acids. These acids are penetrating the soil and dissolving the calcium carbonate crystals (first around the edges). That can cause detachment and loss of surface material. Other effects from acid attack can cause various other damages on the marble (depending on the concentration and the amount of the acid), such as pitting and needle-like erosion. Microorganisms can cause damages while attacking (they dissolve the marble) to the object. However, the microorganisms need air and light, so they can be found on the surface or in a low lever on the topsoil. (Maniatis and Tambakopoulos, 2015).

[8] Base of poorly developed red Mediterranean soil.

[9] The profile of the soil section is called Horizon, each of them have different names, such as Horizon O, O1, A etc. and have different characteristics.

Marine environment: The salty water and environment can cause damages to marble objects. The ions of Na, K, and Cl that exist in the water, they stay on the marble surface and its grains boundaries, after drying the salts are becoming crystallized[10]. Strong pressure is created due to the expansion of the crystalized salts and that can cause detachment. As a result of this procedure the removal of the first (and below) layer of marble (erosion and loss of surface material). In addition, structural cracking in the body (figurine). The exposition to the salty water spray causes faster changes from wet to dry conditions. Furthermore, chemical dissolution can be caused to the calcium carbonate crystals (Maniatis and Tambakopoulos, 2015)

Temperature changes: The cold temperature can cause problems to the marble objects, due to the absorbed water that can be found in the pores of the marble body. The freeze of it is causing expansion and micro and micro cracks on the body. In combination with the salty environment can cause larger and quicker damages (Maniatis and Tambakopoulos, 2015)

Exposure to fire: Fire can cause damages even to the objects that are buried few centimeters below the surface. In high temperatures (up to 750^{o}C) cracks can be created on the object's surface. In addition, the calcium carbonates crystals are converting to calcium oxide (CaO), depending of the duration of the exposure to fire. Finally, the conditions after the fire play an important role, by absorbing carbon dioxide and humidity the object can become brittle and powdery (Maniatis and Tambakopoulos, 2015).

Surface depositions: The depositions considered to be the additional weathering effect. The buried objects usually have soil depositions, which means change of their original color. The depositions in combination with the calcium carbonate crystallization can be compact and hard to remove (Maniatis and Tambakopoulos, 2015).

Preservation: The objects that were buried in deeper layer (no plant activity or microorganisms) and in low-acidity soil, rich in fine calcium carbonate, are better preserved[11]. In the case of this study, these areas and conditions could be found in some parts of Special Deposit North and in Dhaskalio (well preserved marble objects) (Maniatis and Tambakopoulos, 2015).

Different processes can be observed to marble monuments and artifacts which are exposed for a long period to open atmospheric conditions. Briefly, the following are mentioned: patina formations (microstromatolic, microlaminated, monolayered), destructive processes (biopitting, bioerosion), microbial colonization (of the marble surface), microclimatic conditions as well as the orientation and the position of the object (Garcia-Vallès et al. 2002).

Patina is a common weathering effect that can be caused under atmospheric or burial conditions. The word patina can be combined with the words crust or lacquer.

[10] NaCl, KCl, or $K_2(CO_3)$)

[11] A fine calcareous surface coating, can protect the object from further erosion.

The color of a patina can be different, depending on the environmental conditions, the material, the biological components and activities, etc. (Krumbein 2003). A typical burial patina mainly consists of quartz, iron oxides, clay minerals, calcite and dolomite (Garcia-Valles et al. 2010).

5.3. Deformation mechanisms (crystals and grains)

There are different parameters that effect the deformation of the crystals and the grains of a mineral, or stone. Some of the parameters are the lithological (mineral composition, composition of the pores, the grain size, the transparency, etc.), the external factors (temperature, pressure, etc.).

The mechanisms are: cataclastic flow (crumbling of the mineral's grains), pressure solution (pore fluid or intergranular fluid, the moving of the grains in different area caused interior transformation and change of the grain shape), intracrystalline deformation (dislocations or edge dislocations or screw dislocations of the grains, due to the bad state of the crystal), twinning (deformation twins or growth twins), recovery (one of the mechanisms which creating subgrains), recrystallization (grain boundary migration or subgrain rotation), solid-state diffusion creep (coble or nabarro-herring creep)[12], grain boundary sliding and superplasticity (mainly for fine grain rocks), grain boundary area reduction (the creation of polygonal grains), static recrystallization (Lozios 2003).

[12] Based on the temperature and the vacancies of the crystal, the grains are moving (and transforming) to a specific direction.

6. Previous examination

6.1 Weathering examination of marble artifacts from Special Deposit South.

Previous macroscopic examination was performed on the figures of the SDS by Maniatis and Tambakopoulos (2015). The weathering types of marble artifacts (figurines) were studied and classified. Firstly, the different environmental factors were identified: chemistry of the soil, environmental changes during the millennia (air, soil etc.), location changes (different area of exposition and burial) (Maniatis and Tambakopoulos 2015). To categorized the weathering degrees parameters where check such as: the marble type, the provenance (and the weathering), the figurines variety, the location as well as the burial depth (Maniatis and Tambakopoulos 2015).

A brief description of the weathering degrees categories are following[13]:

WD1: well preserved, color/transparency/surface feature clearly visible (possible thin localized beige soil deposition.

WD2: well preservation (relatively). Limited grain detachment (occasionally). Thin light brown deposition, mica (occasionally), limited to medium biological black spots.

WD3: limited to medium grain detachment and erosion, minor or fine cracking, structural damages in the bulk of the figurine, thin to medium light/dark brown soil deposition, localized calcitic encrustations (occasionally).

WD4: medium to extensive grain detachment and medium erosion, minor to medium cracking, structural damages in the bulk of the object, limited to medium biological black spots, localized calcitic encrustation (occasionally).

WD5: medium to extensive grain detachment and extensive erosion (loss of surface curvatures), minor to extensive cracking, major structural damages in the bulk of the object, thin to thick yellow brown (dark brown) deposition, limited mica deposition, limited to medium biological black spots, localized calcitic encrustations (occasionally).

A brief summary of the examination results is presented below:

- *Type of marble*: The maximum grain size (MGS) for each sample was measured (non-destructively) in combination with stereoscopic optical microscope and use of a strong light source. The MGS and the porosity are crucial parameters in the degree of absorbance of corrosive agents for the environment[14]. In the Special Deposit South, the burial environment is similar for all the figurines[15]. However, the results showed that the MGS played a minor role on the weathering procedure. In every category of weathering there are figurines with smaller and larger grains.
- *Provenance of marble and weathering*: Parameters of weathering based on provenance are the origin of the marble, the geological age, the formation and hence quality, as well as the cultural criteria such as the association with a cultural group and civilization, stylistic parameters (typology, variety etc.). However, no obvious

[13] The categories are based on the detailed examination of the weathering effects on the figurines.
[14] Difference of the MGS reflect different kind of marble and different origins.
[15] The figurines from Dhaskalio are excluded.

connection were found. Figurines that have the same provenance (Naxos-2 and Ios) are distributed in all weathering categories.

- *Variety and weathering*: the different varieties of the figurines and the weathering were examined in detail. The results showed that Apeiranthos, Chalandriani and Dokathismata have random degrees of weathering, coming from the microenvirmomental differences of the SDS, while Spedos variety has different characteristics, concentration of the majority of the Spedos figurines in particular location and depth (with the most unfavorable conditions), change of their depositional history (initial exposure to a different environment and then burial with the rest of the varieties at the SDS.
- *Location of finding and weathering*: Each trench versus the degree of weathering was investigated. The examination showed that the horizontal distribution of figurines in the trenches is in general similar for all varieties[16]. It is observed that for Spedos variety, in each at least one figurine was identified as WD5, that comes to the result that the weathering is not resulted only by the local soil conditions in which they were found. In Dokathismata variety all the degrees can be seen with WD5 to have the fewest examples. Similar results has been noticed with Chalandriani variety, fewest WD5 figurines. Finally, the Apeiranthos variety that found in Kavos are in better condition, between WD2 and WD3.
- *Burial depth and weathering*: each figurine's depth location and weathering was checked. Spedos variety was found in a wider spread in depth than other varieties, while Dokathismata variety there are no figurines deeper than 0.60m. The results showed that the depth and the local burial do not affect the weathering degrees dramatically. As characteristic example is that one figurine fragment (no.6410) is characterized as WD5, while the stones which were found around it, are not so weathered.

In conclusion, the investigation showed difference between the Special Deposit North, South and Dhaskalio (more corrosive environment in the Special Deposit South, less calcareous environment). Spedos variety figurines is the largest amount of the marble figurines and have much stronger weathering degree than the other varieties. The depths did not affect the weathering since at the same level different weathering degrees were marked. The figurines that belong to the WD4 and WD5 indicate the possibility that the figurines were exposed in different environment (wet, salty, hot or cold etc.) and then end up buried in the specific environment.

A large amount of figurines were examined for identifying the provenance. The results will be published the following year at the Vol. III.

6.2 Description of the figurines from which samples were taken

[16] In trenches C4, D1, D2, D3 and D4 all varieties are found in increased numbers. In trenches B1, B3, B4, C1 there was an increase accumulation of Dokathismata, Chalandriani and undetermined varieties. In trenches F2, F3, F4, J1 an increase in Spedos variety is marked.

Briefly, the macroscopic analysis included: the SF number, the type, variety, period, deposit, trench identifier, layer, part of the figurine, MGS (Maximum Grain Size), the color, the transparency, W.D. (Weathering Degrees), detachment, erosion, W. Cracking, Soil Deposition, Soil Color, Biological, Mica, Calcitic Deposition, calcitic color, Laminal, Splitting, Pitting[17].

Figurine - SF 1303

The SF 1303 was found in the trench D2 (Special Deposit South) during the digging period 2006, at the layer 5 (Renfrew et al, 2015).

Trench Information: D2

Trench D2 was excavated during the years 2006 and 2007. Large quantities of pottery and special findings were collected by the end of the digging season during 2006. On the eastern site, an aeolianite layer had been uncovered on the eastern side of the trench. In total, 36 layers were dug. Generally, the section at the north part (SF1303 – layer 5) is: topsoil, subsoil, mixture of subsoil and natural soil, natural soil with stones, light brown/orange little angular stones and finds, dark grey-brown pits, terra rossa/natural, brown sandy. The layers 2, 5, 6, and 7 are underlain by aeolianite bedrock that was uncovered later (Renfrew et al, 2015).

Macroscopic examination

The Special Find 1303 was identified as leg fragment of folded-arm figurine of Spedos variety (Renfrew et al, 2015).

Fig. 4: Photo of the SF 1303. A leg fragment of a folded-arm figurine of Spedos variety

[17] The photos were taken by Dr Y. Maniatis and Dr D. Tambakopoulos during their research at the Archaeological Museum of Naxos. The photo editing was completed by V. Anevlavi and S. Giannakidis on Adobe Photoshop CS6.

SF	1303
Type	FAF
Variety	Spedos
Period	K6
Deposit	S
Trench identifier	D2
Layer	5
Part	leg Frag.
MGS	1,0
colour	Pale grayish
transp.	No
W.D.	5
Detachment	Medium
Erosion	Extensive
W. Cracking	No
Soil Deposition	Thin
Soil Color	Brown
Biological	Limited
Mica	Limited
Calcitic Deposition	No
Laminal Spliting	No
Pitting	No

Table 1: Information and weathering characteristics of SF 1303

The SF 1303 has MGS 1.0, the color is pale grayish and it's not transparent. It is categorized as weathering degree 5. The erosion is extensive and it has a thin soil deposition (yellowish brown), with limited biological depositions (black spots). The figure is fragile and it has limited mica.

Figurine - SF2804

The SF2804 was found in the trench D2 (Special Deposit South) during the digging period 2006, at the layer 19 (Renfrew et al, 2015).

Trench information: D2

The SF 2804 was found at the trench D2 (same trench as the previous sample, SF1303), at the layer 19. Layer 19 was located in the north of the trench. Other parts of marble figurines (2) were found there, as well as part of marble basin and a large stone disk fragment (Renfrew et al, 2015).

Macroscopic analysis

The Special Find 2804 was identified as torso fragment of folded-arm figurine of Spedos variety.

Fig. 5: Photo of the SF 2804. A torso fragment a folded-arm figurine of Spedos variety

SF	2804
type	FAF
Variety	Spedos
Period	K6
Deposit	S
Trench identifier	D2
layer	19
part	Torso
MGS	0,9
colour	White
transp.	High
W.D.	5
Detachment	Medium
Erosion	Medium
W. Cracking	Medium
Soil Deposition	Thin
Soil Color	Brown
Biological	No
Mica	Limited
Calcitic Deposition	Localized
calcitic colour	White
Laminal Spliting	No

Pitting	No

Table 2: Information and weathering characteristics of SF 2804

The SF 2804 has MGS 0.9, the color is white and it had high transparency. It is categorized as weathering degree 5. The erosion is identified as medium and it has medium cracking. It has a thin soil deposition (yellowish brown), with no biological deposition. The figure has limited mica and a localized white calcitic deposition (Fig.2).

Figurine - SF6274

The SF6274 was found in the trench C4 (Special Deposit South) during the digging period 2007, at the layer 5 (Renfrew et al, 2015).

Trench information: C4

The trench C4 is among the richest trenches (in terms of findings) of the Special deposit South, C1 and D3. The excavation goals of the C4 trench was to be able to identify the limits of the cultural deposits. The C4 has only five layers (topsoil) and underneath is bedrock. However, a large amount of findings came out of this deposit (about 244 special finds). SF 6274 was found in the 5th layer. It is the lower level of the topsoil, paler in color soils. Some of the objects found in trench C4 (lamps, vessel, part of figurine) joined with trenches C3, D3 and C1 (Renfrew et al, 2015).

Macroscopic analysis

The Special Find 6274 was identified as waist, pelvic and upper legs fragment of folded-arm figurine of Spedos variety.

Fig. 6: Photo of the SF 6274. A waist, pelvic and upper legs fragment of a folded-arm figurine of Spedos variety.

SF	6274
type	FAF
Variety	Spedos
Period	K7
Deposit	S
Trench identifier	C4
layer	5
part	Waist, pelvic and upper legs
MGS	1
colour	White
transp.	No
W.D.	5
Detachment	Extensive
Erosion	Extensive
W. Cracking	Medium
Soil Deposition	Medium
Soil Color	Dark brown
Biological	Medium
Mica	Limited
Calcitic Deposition	Medium

calcitic colour	White
Laminal Spliting	Limited
Pitting	No

Table 3: Information and weathering characteristics of SF 6274

The SF 6274 has MGS 1.0, the color is white and it has no transparency. It is categorized as weathering degree 5. The erosion is extensive and it has medium cracking. It has medium thickness deposition (yellowish brown), with some spots of biological deposition. The figure has limited mica and a localized white calcitic deposition (Fig.3).

Figurine – SF 20167

The SF20167 was found in the trench D4 (Special Deposit South) during the digging period 2008, at the layer 7 (Renfrew et al, 2015).

Trench information: D4

The trench D4 includes 13 layer. The SF20167 was found in layer 7. The trench was a part of the continuing excavation of the area covered by the cairn. The layer 7 is in the northern corner of the trench, with a line of aeolinite bedrock to the northeast and bounded by stone alignment G to the southwest. Stone alignment G was up to 3.5m in length, 0.60m wide and c. 0.25m in height (Renfrew et al, 2015).

Macroscopic analysis

The Special Find 20167 was identified as upper legs fragment of folded-arm figurine of Spedos variety.

Fig. 7: Photo of the SF 20167. An upper legs fragment of folded-arm figurine of Spedos variety.

SF	20167
type	FAF
Variety	Spedos
Period	K8
Deposit	S
Trench identifier	D4
layer	7
part	Upper legs
MGS	1
colour	Grey/White
transp.	No
W.D.	4
Detachment	Yes
Erosion	Medium
W. Cracking	No
Soil Deposition	Thin
Soil Color	Brown
Biological	No
Mica	No
Calcitic Deposition	Limited
calcitic colour	White
Laminal Spliting	Limited
Pitting	No

Table 4: Information and weathering characteristics of SF 20167

The SF 20167 has MGS 1.0, the color is white (grey) and it has no transparency. It is categorized as weathering degree 4. The erosion is identified as medium. It has thin deposition (yellowish brown). The figure has localized white calcitic deposition (Fig.4).

Figurine – SF 25077

The SF 25077 was found in the trench RA (Special Deposit South) during the digging period 2008, (no layer mentioned) (Renfrew et al, 2015).

Trench information: RA

During the excavations in 2006 and 2007 four baulks remained intact in between the trenches D2, C1, B4, F3 as RAz, Ray, RAx, RB. In 2008, the baulks RA and RB were excavated for better understanding the conditions in between the D2, C1 and B4 trenches. The SF 25077 was found in trench RAz, in the first layer. Few finds were recovered even from the first layer. The RAz is consisted by 21 layers in total (Renfrew et al, 2015).

-Macroscopic analysis

The Special Find 25077 was identified as upper legs fragment of folded-arm figurine of Spedos variety. (Renfrew et al. 2015).

Fig. 8: Photo of the SF 25077. An upper legs fragment of folded-arm figurine of Spedos variety.

SF	25077

type	FAF
Variety	Spedos
Period	K8
Deposit	S
Trench identifier	RA
layer	-
part	Upper or Lower legs
MGS	1,5
colour	White (pale grey)
transp.	No
W.D.	5
Detachment	Yes
Erosion	Extensive
W. Cracking	Minor
Soil Deposition	Thin
Soil Color	Light brown
Biological	Limited
Mica	Limited
Calcitic Deposition	No
calcitic colour	-
Laminal Spliting	Medium
Pitting	No

Table 5: Information and weathering characteristics of SF 25077

The SF 25077 has MGS 1.5, the color is white (pale grey) and it has no transparency. It is categorized as weathering degree 5. The erosion is extensive and it has minor cracking. It has thin deposition (yellowish brown), with some spots of biological deposition. The figure has limited mica (Fig.5).

Vessel – SF 30006

The SF 30006 was collected during the excavation periods 2006-08 from the area of Special Deposit North (Renfrew et al, 2015).

Trench information: -

No systematic work has been conducted in the Special Deposit North since ‘60s. During the excavation periods 2006-08 few surface artifacts were noted and collected such as figurines, marble vessels, and metal objects (Renfrew et al, 2015).

Macroscopic analysis

The Special Find 30006 was identified as marble basin of diameter 380mm, and preserves faint red pigment in its interior (Renfrew et al. 2015).

Fig. 9: Photo of the SF 30006. A rim fragment of a marble basin with red pigment in it interior.

SF	30006
type	vessel
Variety	basin
Period	K6-8
Deposit	N
Trench identifier	Surface artifact
layer	-
part	rim
colour	White
M.Condition	1
Maximum Preserved Dimension	35
Estimated original diameter	380
Maximum Thickness	12
Breaks	3
Pigment	Red (faint)
Erosion	no

Table 6: Information of SF 30006 (Renfrew et al.2015)

The SF 30006 has white color. It is not eroded. It is categorized as weathering condition 1. It have red color pigment in its interior (Fig.6).

7. Experimental work

7.1. Samples and experimental techniques

In order to examine the preservation state of the Cycladic figurines and to characterize the pigment on the fragment of the marble vessel, a number of techniques were applied.

In total, 6 samples of figurines, found in Special Deposit South and one sample of a marble vessel with red pigment collected from the area of the Special Deposit North (Chapt. 6) during the excavation periods 2006-2008, were taken by Maniatis and Tambakopoulos during the previous examination at Naxos Museum[18].

The samples were examined in two forms, as received - ASR (first step for the identification of the deposition), but also in polished form, the cross sections of particles from each sample. The latter allow the tracing of the depth of grain alteration and the thickness of surface depositions, as well as the thickness of the pigment layer. For each samples certain types of analyses are presented in Chap. 7.1, deposition analysis of the surface from the ASR particles and one deposition analysis and one interior marble analysis from the polished sample. Inclusions from the polished sample were also analyzed.

a. **Optical Microscope examination** (stereoscopic microscope) of the samples: Microscopic examination of the samples, as a preliminary step of the examination, can provided information on various aspects of the preservation state of the sample. The color, the size, and the depositions were identified and each sample was photographed under Leica Microscope, under a magnification between 10x and 50x. After the microscopic observation and photographing the most representative particles were collected for further analysis under SEM (ASR, while one particle from each sample was chosen to be embedded in resin, polished – cross section).

b. **Petrographic Microscope examination of polished surfaces:** After the procedure of polishing, the samples were observed under Leica petrographic microscope (with different light sources) and photos of the cross sections were taken. A following step, was the procedure of carbon coating.

c. **Scanning Electron Microscopy**: SEM achieves a higher magnification with great depth of field than the optical microscope, thus enabling a more detailed examination of the sample. In addition, the analytical facility attached (EDX) allows the chemical analysis of the sample as a whole or of particular grains, inclusions, depositions etc. In addition, polished samples can give details of the condition of the grains in the interior and not only on surface, and the thickness of the deposition or the pigment layer. The method of SEM was chosen because of the combination of the image (high magnification photos), the ability to

[18] The artifacts have taken a special number. The figurines numbers in the present investigation are: SF 1303, 2804, 6274, 20167, 25077. The number of the vessel is SF 30006. Some of the samples were separated in samples A and B.

observe the cross sections of the small grains and finally, the chemical analysis that can give us information about the composition of the soil depositions, as well as the chemical composition of the pigment.

A more precise identification of the weathering state and the types, as well as the measurement of the bulk and the chemical composition was achieved through analysis by SEM-EDX (Philips 515 SEM, EDX & EBIC). The combination of high magnification and compositional analysis, which is provided by the SEM, is necessary to observe and be able to answer the question of the weathering situation of the Cycladic figurines.

Because of the small amount of particles from each sample, no other analyses were performed.

SEM was used in two ways. Firstly, the samples ASR where analyzed for identifying deposition on the sample and later the polished samples were examined for observing the weathering state on the interior of the grain. Chemical analysis was proceeded the oxides of Si, Al, K, Na, Fe, Mg, Ca and Ti were undertaken.

The main operating conditions for the SEM-EDAX were:

- Accelerating voltage: 25.00 KV
- Count time: 120 sec (approximately)

The samples in ASR form were analyzed under low vacuum, while the polished samples were analyzed under high vacuum. Many spectra were taking from each sample (grains) the most representative are discussed below. For the ASR samples one of the deposition analysis is discussed, while for the polished samples one analysis of the interior of the grain (marble) one for the depositions is presented. Few inclusions are also noticed (chemical analysis). The analysis with photo of the analyzed spot and the spectra are presenting on the following chapter.

7.2. Analyses and Results

Figurine - SF 1303

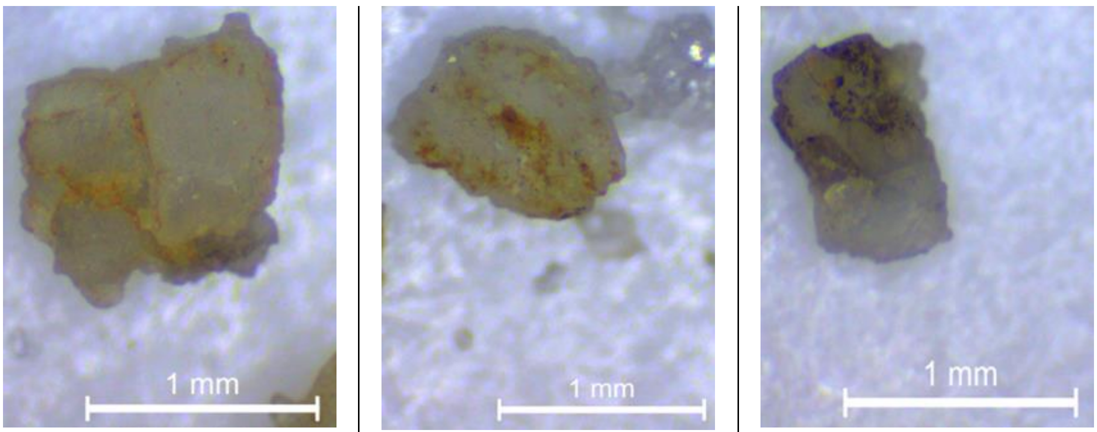
The Special Find 1303 was identified as leg fragment of folded-arm figurine of Spedos variety (see Fig. 4).

Optical Microscope Analysis

The sample was checked under Leica stereoscopic microscope (Fig. 10). It consists of a lose accumulation of smaller and larger particles produced by light scratching at the edge of the figurine which was badly weathered (Maniatis and Tambakopoulos 2015, Fig. 4) with marble particles falling apart. The particles consist of aggregates, containing 1-2 or more marble grains still joined together (Fig. 11). The color of the grains was mainly transparent white and grey. They were few particles with yellow-light brown depositions, mainly accumulated in the grain boundaries (Fig. 11 A, B). A small amount of grains had black spotty depositions (Fig. 11C).

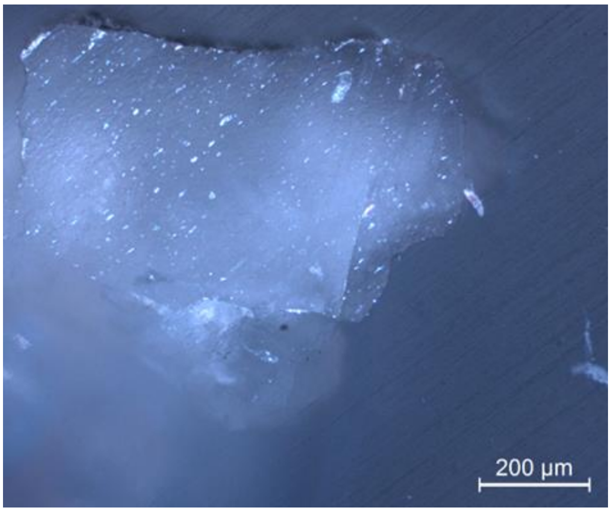
Fig. 10: General photo of the sample. Different sizes of aggregates, mainly of rounded shapes, containing a different number of grains of white, grey, yellowish color.

Photo taken with Leica Stereoscope.

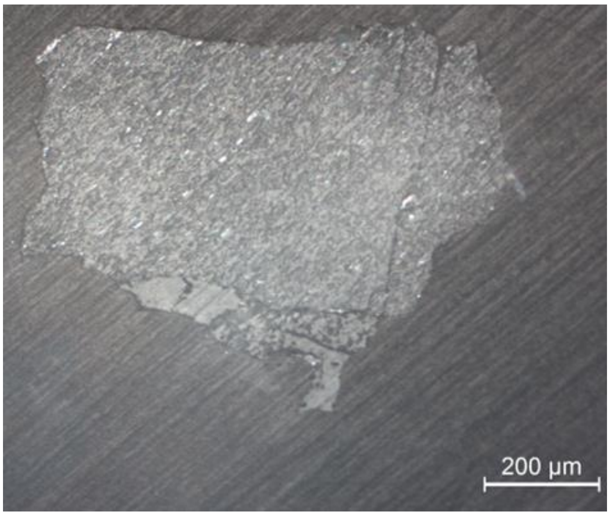


A B C

Fig. 11 (A, B, C): Characteristic marble particles/aggregates with yellowish - brown depositions (A, B) and black depositions C. These particles were selected for further analysis with the SEM. The particle C was embedded in resin.



A



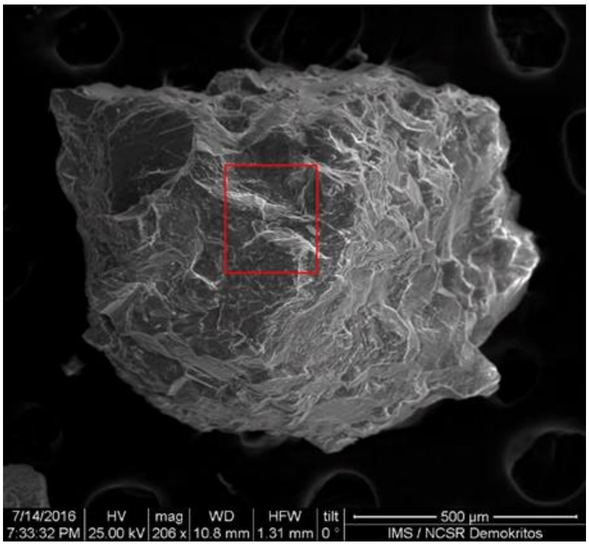
B

Fig. 12 (A, B): Cross section of the polished sample (Fig. 11 C) under the petrographic microscope in different polarization conditions. The grain is clean, some crucks can be observed. On the top of the grain a black spotty deposition can be observed.

The Fig. 11C was embedded in resin and the cross section is presented above (Fig. 12 A, B). It is a single grain of pure marble having a white-transparent color. Remnants of a black spotty deposition can be observed on the top of the grain (Fig. 12 A). Large cracks can be observed on the right side of the sample while broken parts of the grain can be noticed on the bottom (Fig. 11 B). The photos are taken under petrographic microscope.

SEM Examination and Analysis

Two grains (Fig. 11 A, B) were chosen for further examination and analysis under the SEM in the ASR form and the grain in polished cross section embedded in resin (Fig. 11 C, Fig. 12 A, B).



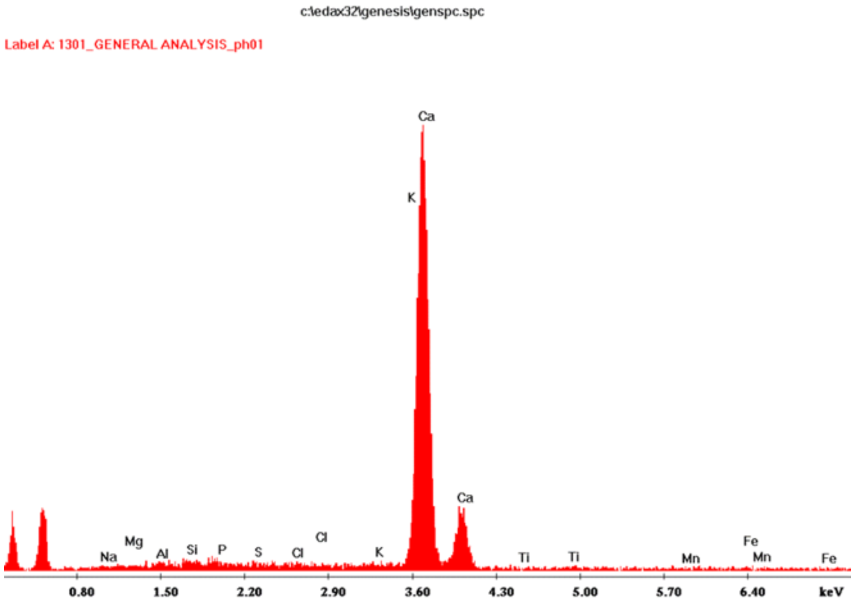


Fig.13: 1st spectrum (table 7): Left: The SEM image of a grain taken with the LFD detector at low vacuum. Right: the analysis spectrum taken with the EDX-ray microanalysis system of the SEM at the region shown with the red square. The elements detected are only Ca and C (pure calcite).

Figure 13 left shows the SEM image of one of the grains examined at low vacuum ASR. The spectrum on the right shows the analysis obtained with the ED X-ray analysis system of the SEM taken in the region indicated with the red square. As it can be seen the spectrum is dominated by a strong calcium peak plus oxygen and carbon with negligible traces of silicon and Aluminum. This indicates a pure calcitic marble. The amount of CaO calculated to 94% (Table 7). Similar analysis taken on other spots on the grain showed the same pure calcite composition with other elements detected in minimum quantities.

A second grain (Fig. 11 B), showed a thin layer of brown-yellowish deposition. Examination at the surface under the SEM at low vacuum ASR, looks grainy at the areas with the brown-yellowish deposition an indication of a fine particle composition (Fig. 14, left). The analysis at this surface (Fig. 14, right) showed apart from Ca, O, and C coming from the marble, some amounts of silicon, aluminum, Mg, Na, K, Fe, plus small amounts of Cl and S. This indicates that the brown-yellowish depositions on the surface of the grains consist of soil originating obviously from the burial environment.

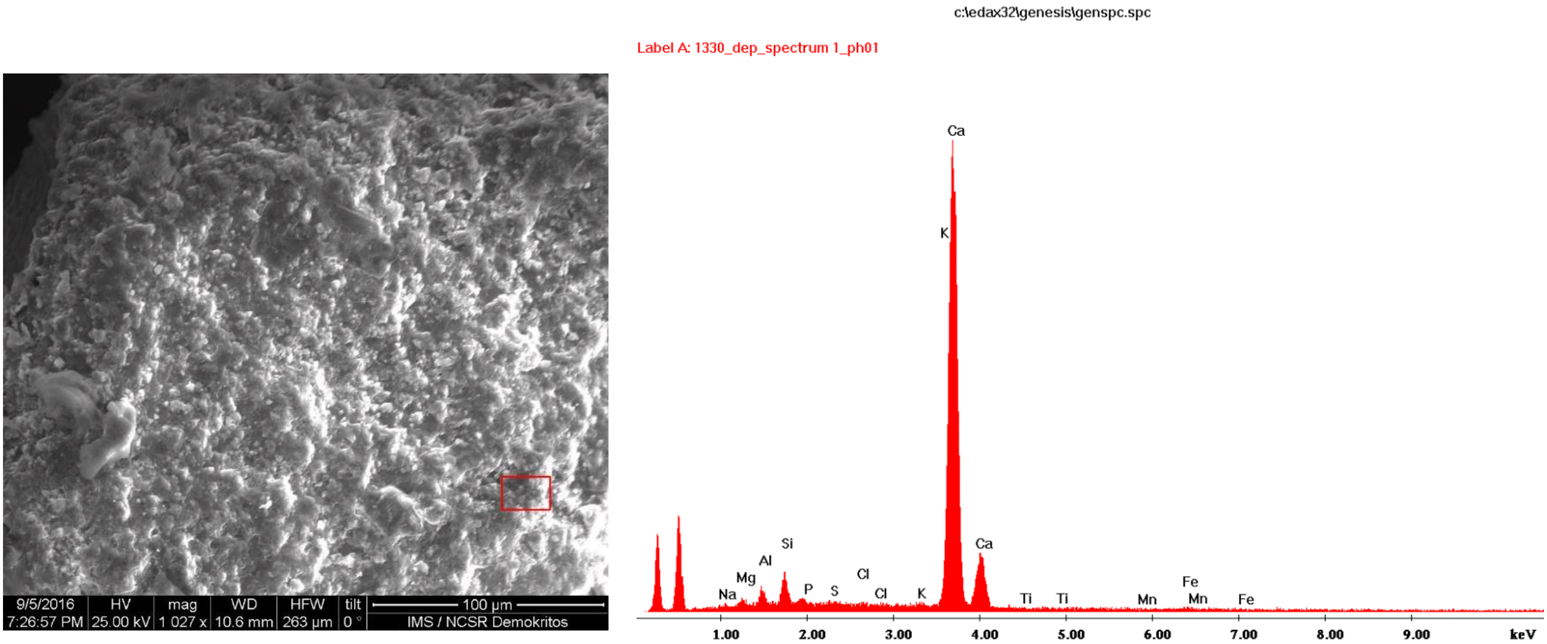


Fig.14: 2nd spectrum (Table 7): Left: SEM image of the surface with the brown-yellowish depositions. Right: Analysis spectrum at the area indicated with a red square.

The particle B (Fig. 11 B) was also examined and analyzed in several other surface spots. A typical composition of the surface depositions are shown in Table 7. The amounts of SiO_2 (7%), Al_2O_3 (4%), MgO (1.8%) and Fe_2O_3 (1.4%) are typical of soil deposition. The presence of a small amount of sulfur, SO_3 (1%), is also coming from the soil.

At a specific spot on the surface (Figs 15 & 16) an increased amount of sulfur was detected (SO3 =18%) / (3rd analysis) together with an amount of soil as witnessed from the increased amounts of Si, Al, Mg, Fe, Na, K (Table 7, no. 3). The area with increased sulfur does not show any typical form of crystallization (e.g. gypsum crystals). This is an indication that it is not so much a result of conversion of the calcitic marble to gypsum at the surface in an acidic environment containing sulfur but rather a result contact of the figurine in the soil with a localized source of sulfur, as animal or plant remains.

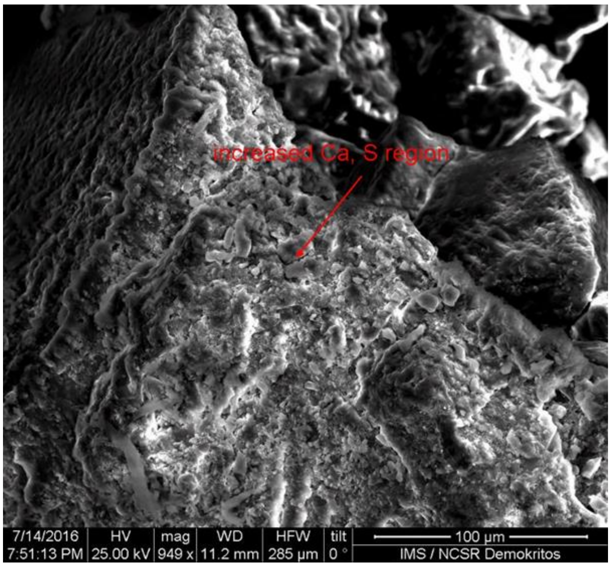


Fig. 15: On some areas an increase of SO_3 has been noticed. It could be a sign of gypsum formation, or contact with a localized sulfur source (animal or plant remains). Photo taken with SEM

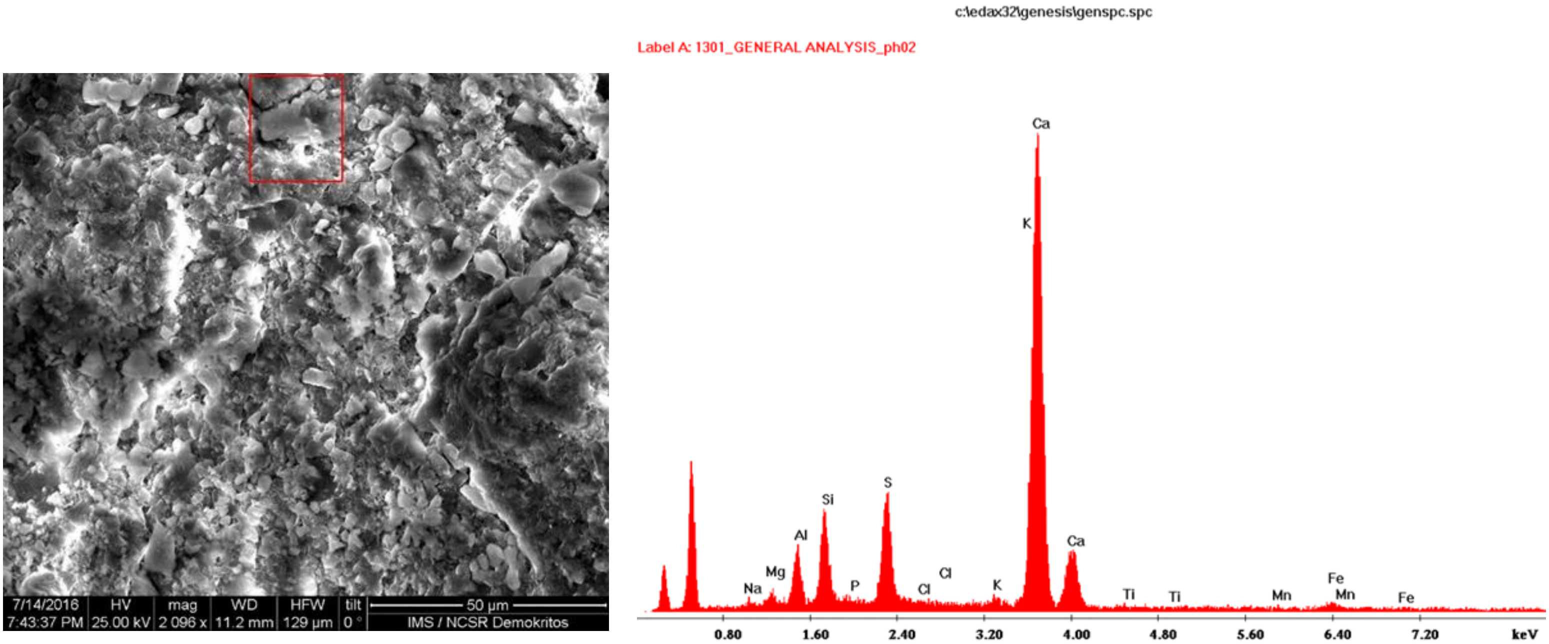


Fig. 16: 3rd analysis (Table 7): The spot analysis showed increased S (about 18%) as well as increased silicon and aluminum.

Polished sample

The polished cross-section vertical to the surface of the grain in Figure 11 C was coated with carbon and examined and analyzed under the SEM in high vacuum in a backscattered electron mode (Fig. 17, 18 and 19). Figure 17 shows the whole section consisting of a single marble grain. As it appears the black depositions (Fig. 12) could not be detected as a distinct layer at the surface due to its lack of continuity (spot-like depositions rather than a continuous layer) and to its thinness.

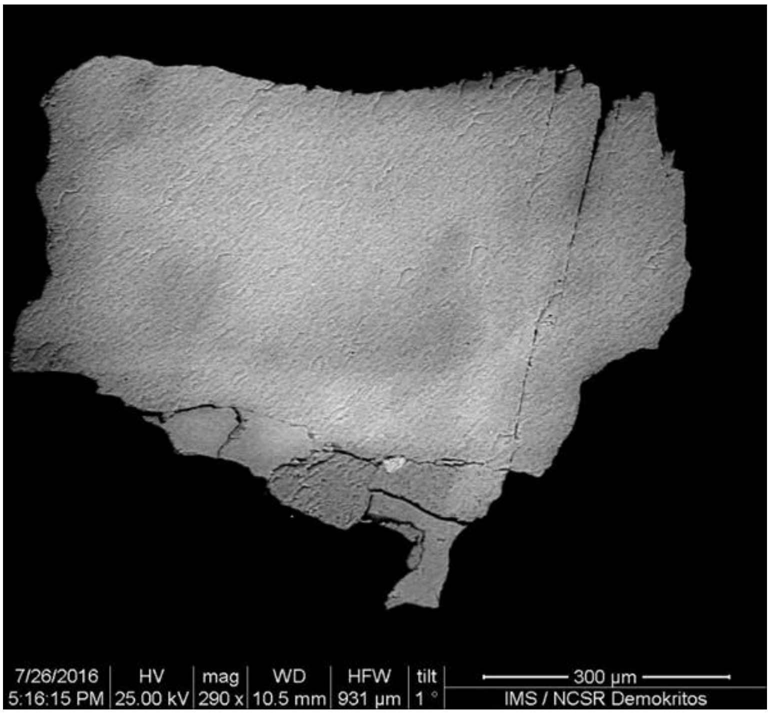


Fig. 17: General image of the polished sample, (Fig. 11 C). The natural surface is at the top and appears eroded. Large crucks on the right top part can be detected. More intense cracking can be observed at the lower part. An inclusion (brighter particle is shown trapped inside a crack in the lower part.

A closer image of the grain and its spectrum are shown in Figure 18. The inner part of the grain body shows the chemistry presented in Table 7, an.no. 4 (Fig. 18). It appears to be pure calcitic marble however containing a very small amount of dolomite as can be assessed from the small amount of Mg present. Traces of P, Fe, and other elements may indicate a minute absorption of elements from the soil.

Large and deep cracks can be detected close to the right part of the grain as well as at the bottom where fragments of the grain ready to be detached. The surface of the grain is eroded as can be observed from the shallow and deeper cracks and pitting, a result of differential dilution with tentacles extending above the surface and other anomalies (Fig. 17 and 18).

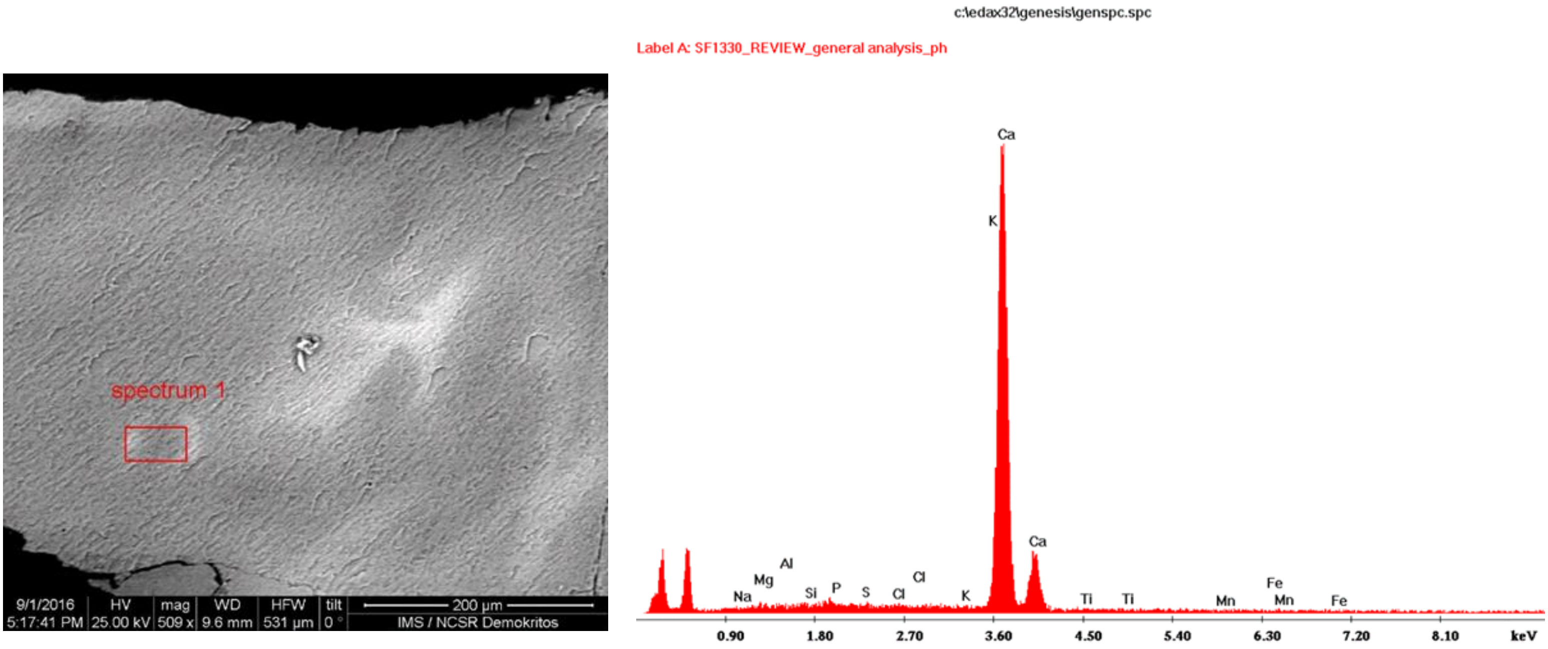


Fig. 18: 4th analysis (table 7): Left: a closer image of the inner part of the grain and the surface. Right: The analysis showed that the particle's consistence is pure marble, not other elements have been detected.

A white inclusion has been noticed on the lower part of the grain, between the cracks (Fig. 17, brighter particle lower part). Detailed examination and analysis is shown in Figure 19.

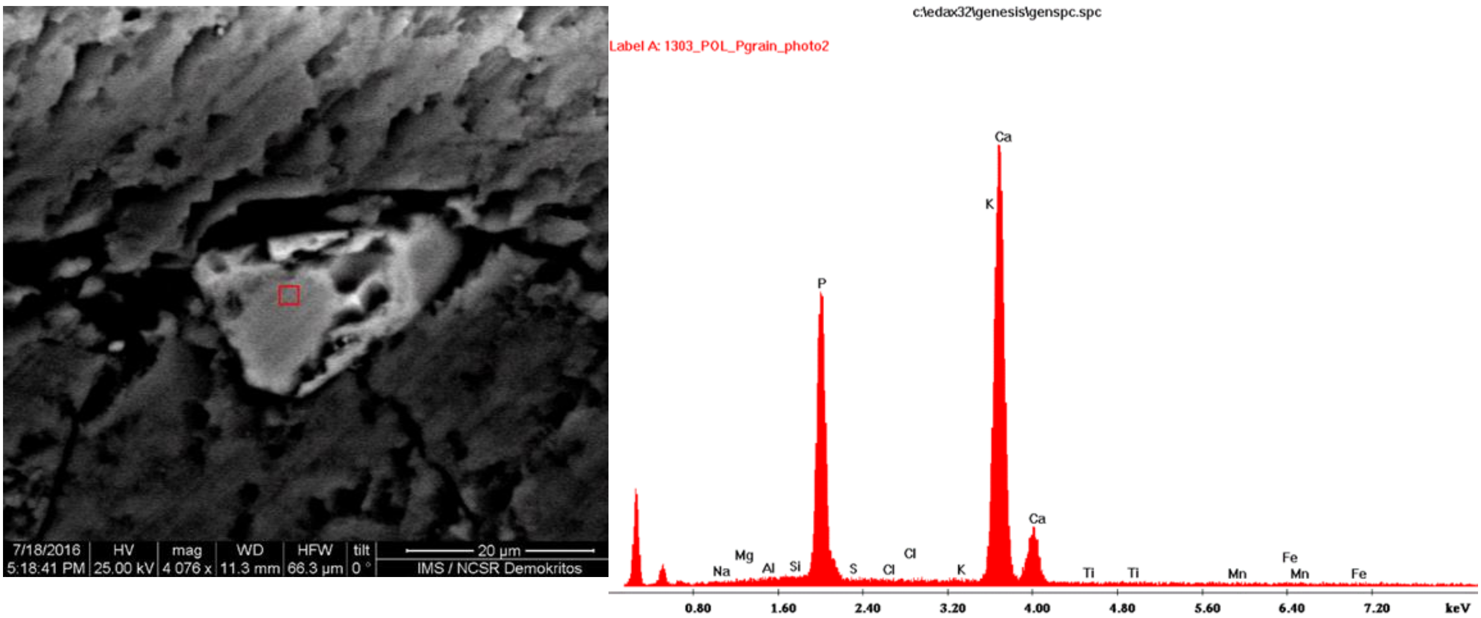


Fig.19: 5th analysis, (table 7): A white inclusion was observed between the cracks of the sample. The analysis showed that the inclusion is a fragment of a mineral apatite or a fragment of a bone. A voids pattern can be observed.

This inclusion has a size at its longer distance of about 20 μm (Fig. 19) and contains only phosphorus and calcium (Table 7, no.an. 5) in concentrations approaching the chemistry of apatite[19]. This could lead to the conclusion that this inclusion is either a fragment of a mineral apatite or a fragment of bone embedded in the marble from the soil through a crack. The voids observed in the inclusion fragment (Fig. 19) could tentatively point more to a bone fragment (spongy bone).

Table 7: Chemical analysis in oxides form (100%wt) - SF 1330

No An.	Spectrum	Na2O	MgO	Al2O3	SiO2	P2O5	SO3	Cl2O	K2O	CaO	TiO2	MnO	Fe2O3	total %
	ASR													
1	marble	0.52	0.43	0.94	1.24	0.60	0.90	0.37	0.40	94.00	n.d.	n.d.	0.49	100
2	deposition	0.77	1.87	3.74	7.02	0.88	1.00	0.4	0.50	82.00	n.d.	0.30	1.41	100
3	Sulfur dep	0.93	1.91	7.54	13.2	0.40	18.00	0.18	0.60	55.00	n.d	n.d.	1.88	100
	Polished													
4	marble	0.32	1.64	0.50	0.49	0.77	0.50	0.34	0.30	93.00	0.45	0.50	0.84	100
5	inclusion	0.31	0.21	0.32	0.29	43.2	n.d.	0.05	0.20	55.00	0.16	0.20	0.51	100

n.d. = not detected

Summary

The sample consists of smaller and larger particles representing aggregates of marble crystal grains, mostly white and transparent. Remains of yellowish brown depositions are common in several aggregates and more rarely black spotty depositions can be seen. The combination of the examination and analyses of the ASR grains and a polished cross-section under the SEM showed that the inner parts of the grains are pure and mainly calcitic marble with a very limited absorption of elements from the soil. However, the surfaces are eroded and brown-yellowish soil has been deposited on the surface and in the grain boundaries. Cracks and broken parts were noticed in the grains as well as inclusions from the soil wedged in the cracks. Also at places biological microorganisms were present that have left black sporia.

These all are indications that the marble grains or group of grains have been detached by acidic attack and exposure to changing environmental conditions and then buried in the ground. During this burial soil was deposited on the surface of the already detached grains or inside the grain boundaries and cracks making the disintegration even faster

This particular figurine as it was mentioned on Chapter 6 has been categorized by macroscopic examination as a heavily weathered figurine (WD = 5, Maniatis and Tambakopoulos 2015). The detailed examination and analysis of the grains in the

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[19] Apatite's chemical formula (oxides) consists: CaO = 55.07%, P_2O_5 = 41.28% and Cl_2O = 2.32%. The colors of the mineral are white, yellow, green, red, blue and it's transparent to translucent.

sample obtained from this figurine confirms the extensive weathering and determined that the damage extends to the individual grains. It also identified the origin of the depositions on the surface and the fact that the marble was first weathered and then buried.

Figurine - SF2804

The Special Find 2804 was identified as torso fragment of folded-arm figurine of Spedos variety (Fig. 5) (Renfrew et al 2015).

The figurine appeared to have a crust at specific places on its surface, such as the part of the right shoulder (Fig. 5). The color of the crust is yellowish white. On top of the crust a yellowish brown deposition can be also observed.

Different size particles were collected as sample ranging from 0.5 to 1 mm.

Optical Microscope examination

The sample consists of different particles that can be seen in Figure 20 under a stereoscopic optical microscope.

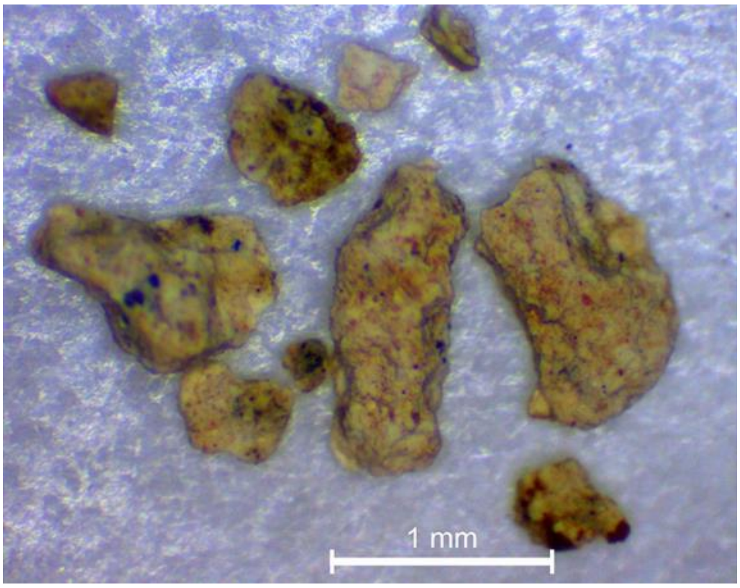


Fig. 20: General photo of the sample under a stereo-optical microscope. White crust, light brown and black depositions on the crust can be seen.

Two characteristic grains have been chosen to be examined and analyzed without treatment (ASR) under the SEM, and a third grain had been embedded in resin and polished to produce a cross-section perpendicular to the outer surface.

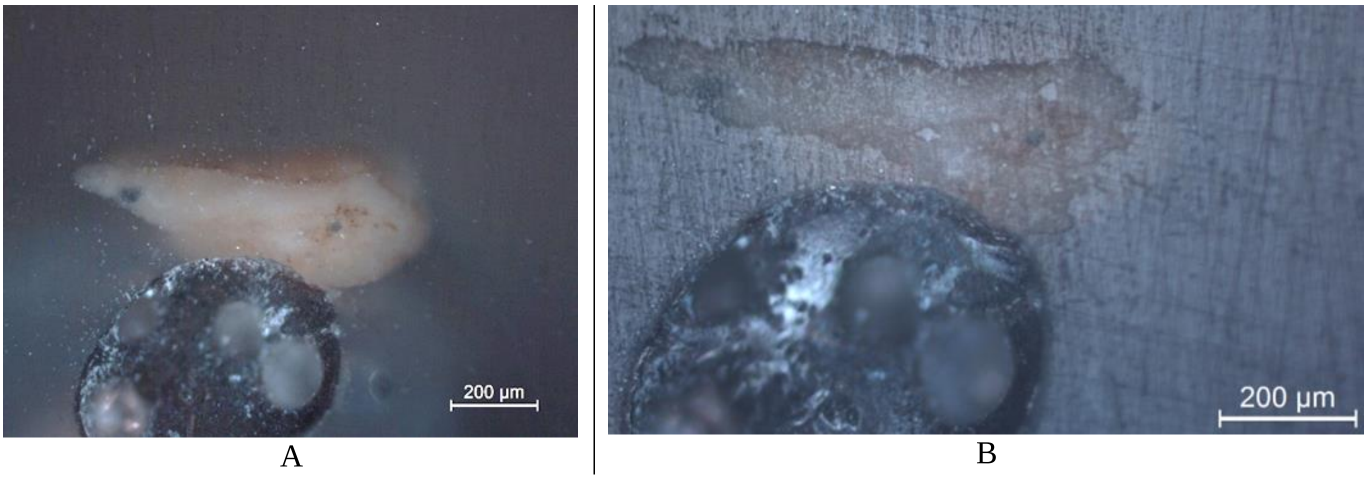


Fig. 21 A, B: Polished cross-section under the petrographic microscope of a small particle close to the outer surface bearing a white crust and a brown-yellowish deposition on top. A: Under non-polarized light showing the white crust and a thin layer of the brown-yellowish deposition on top,

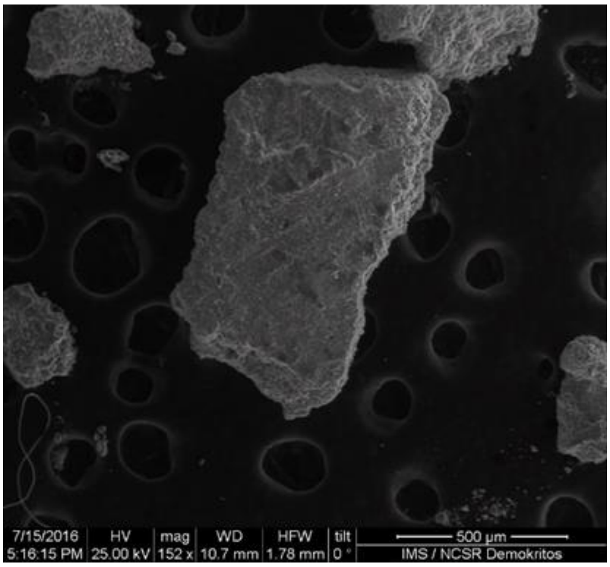
penetrating also inside the white crust. B: The sample at higher magnification and with polarized light. Different sizes of inclusions can be observed in the crust of white and dark color). The circular feature below the sample is a bubble in the resin.

The particle embedded in resin and polished to produce a cross-section was a small fragment mostly containing the white crust and the brown-yellowish layer on top of it. Figure 21 shows the polished section under the petrographic microscope with non-polarized (A) and polarized (B) light. The white crust appears quite thick, over 200 μm. Penetrations of the brown-yellowish layer from the outer surface inside the white crust layer can be seen in Figure 21 A. Also angularly shaped inclusions seem to be contained in the white crust layer (Fig. 21 B) pointing to a mixed crust layer.

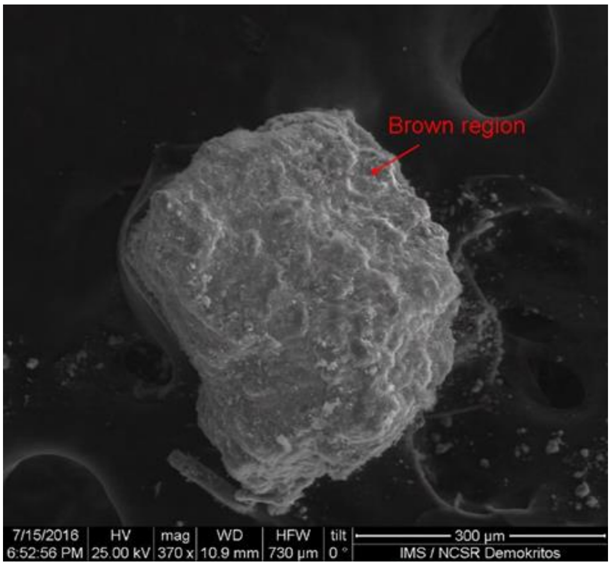
SEM examination and analysis

-Two particles were analyzed in the ASR state under the SEM.

The general images of these particles are presented below (Fig. 22 A, B). The first one (Fig. 22 A) is a rectangular piece of a fragment of the crust with approximate dimensions of 300x700 μm. While the second one (Fig. 22 B) is a marble particle (about 400x400 μm) with brown-reddish deposition. EDX_ray analyses was performed on both particles.

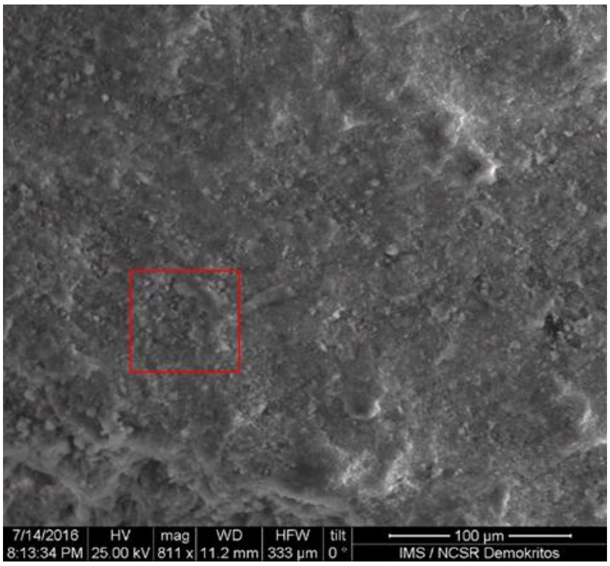


A



B

Fig. 22 A, B: Images of the two particles that have been analyzed under SEM. A: Fragment of the white crust. B: a marble particle with brown-reddish deposition.



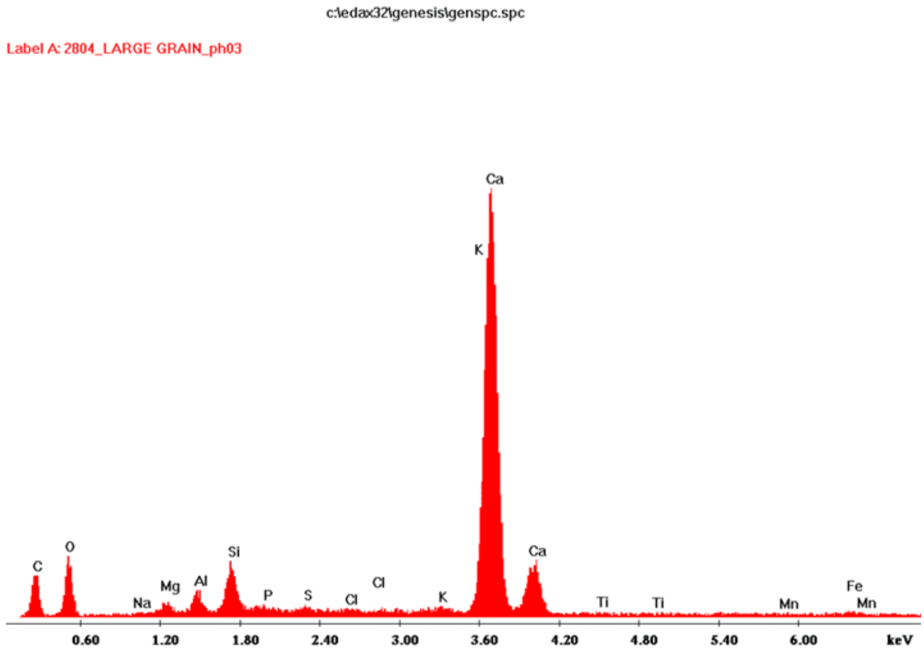


Fig.23: (1st analysis, Table 8): Left: SEM micrograph of the surface of the white crust of fragment shown in Figure 22 A. Right: EDXA spectrum of the area of the surface indicated with red square.

The morphology of the white crust surface (Fig. 23, left) indicates that it consists of small rounded particles in a compact layer. The general analysis of the surface (Fig. 23, Table 8, no.1) showed composed mainly of calcium (CaO = 77%). A certain amount of soil seems to have been absorbed in the white crust as indicated by the presence of elements such as Si, Al, Mg and Na. The increased Mg is possibly coming from the soil materials.

The second particle which is marble with a brown-reddish deposition of its surface is shown in Figure 24 left. The analysis spectrum of the brown deposition (red square) is shown in Figure 24 right. The quantitative elemental composition is listed in Table 8, no.2.

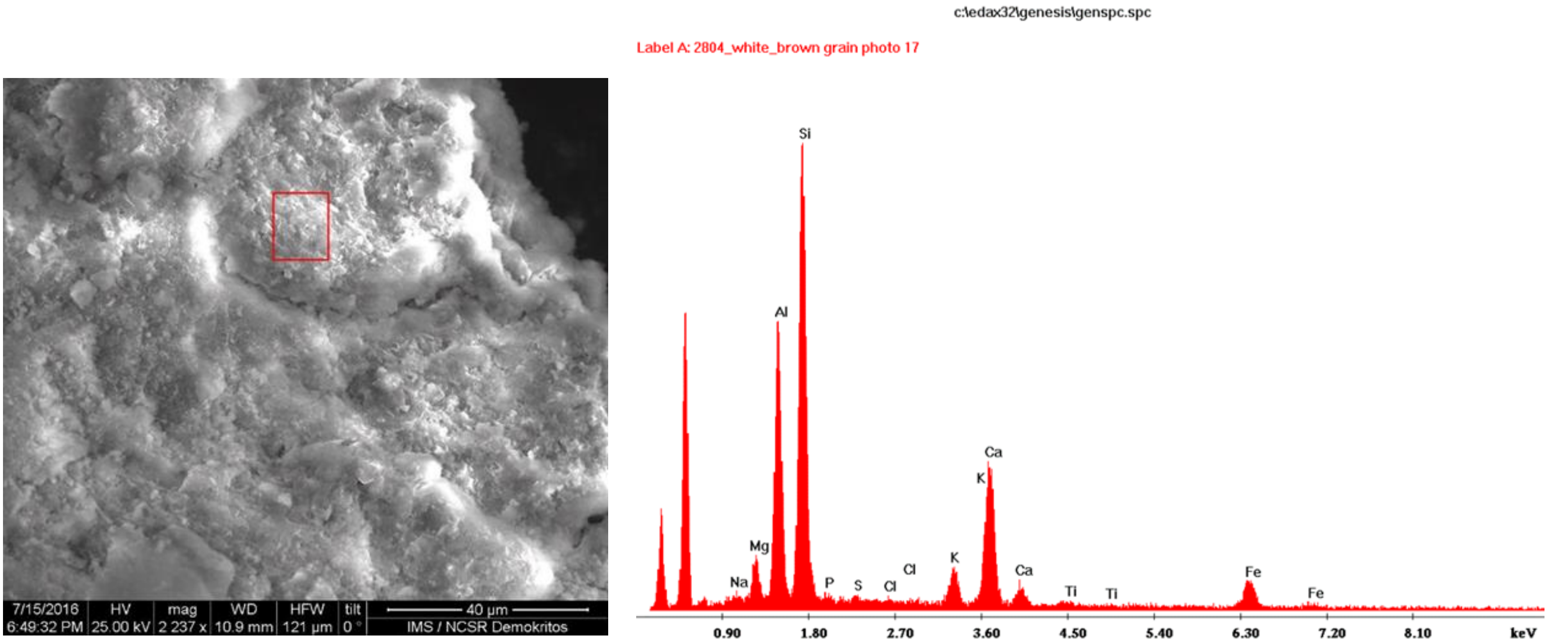


Fig.24: (2nd analysis, Table 8): Left: Brown-reddish deposition on a marble particle. Right: The EDX spectrum taken at the red square region. A high concentration of soil elements is observed (Table 8, no.2). The amount of calcium detected originates from the marble substrate.

The brown-reddish deposition consists of a fine particle layer on the surface of marble (Fig. 24, left). The analysis taken in the region of the red square showed high concentrations of Si, Al, Mg, Na, K and Fe (Table 8, no. 2) typical of a brown-red clay deposition, which has been observed on the majority of the grains of most samples and it is most likely coming from the soil in the burial environment.

-Polished sample

A small particle of the white crust has been embedded in resin and polished (Figs 21 A, B). The SEM image of the cross section is shown in Figs 25, 26 and 27. As it appears the white crust is not a homogenous layer but containing inclusions and clay layers (Fig. 25 A, B) shows its irregular layer structure. Layers of different composition, texture and color are marked with red lines. At least 5 different layers are identified in this fragment. Large or smaller inclusions, minerals of different shapes can be noticed in the whole section of the particle. Clear marble crystals cannot be detected, so all this fragment is a crust shown a maximum thickness of about 300 μm.

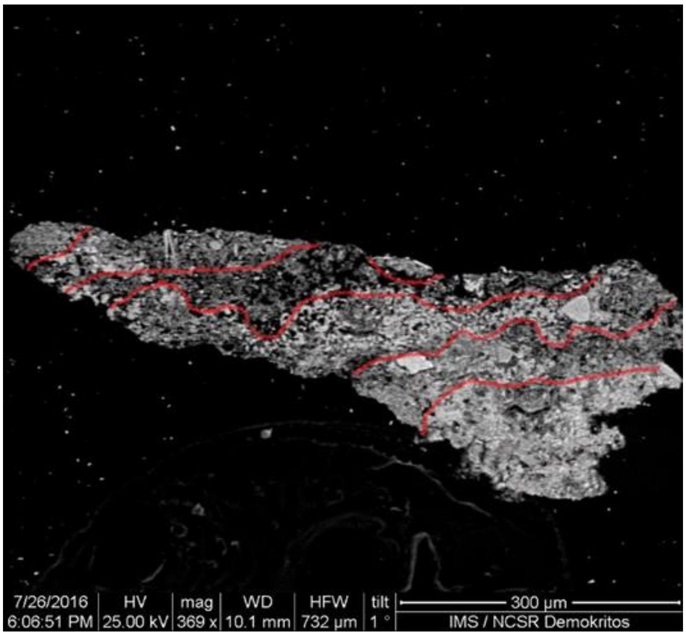


Fig. 25 A: Different layers are detected. Each layer is separated by red line and variations in color of the layers can be observed. Different sizes inclusion can be notices. The layers are marking different environmental episodes during the burial of the figurine underground.

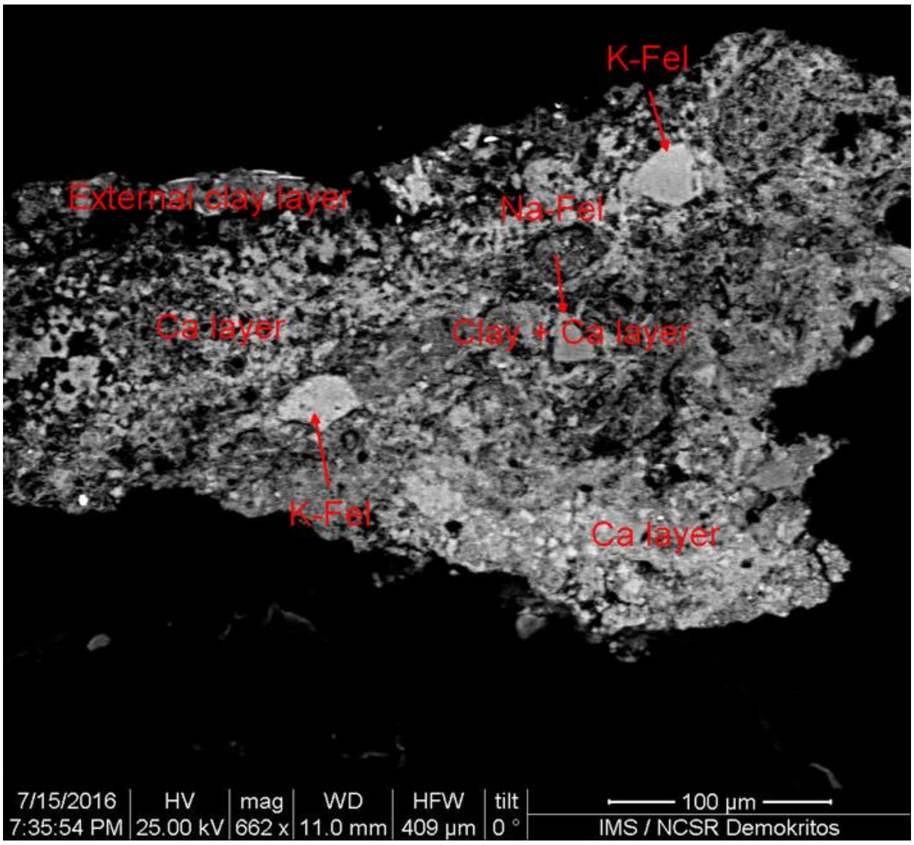


Fig. 25 B: SEM image of cross section at higher magnification and identification of inclusions and layers. The larger inclusions are K- and Na-feldspars.

Figure 25 B shows in higher magnification a part of the cross-section. The lowest layer (lower right of photo, brighter region) consists of a high amount of Ca layer and composed of fine particles. It is obviously an almost pure calcite layer (Table 8, layer 1) formed by dilution and recrystallization of the marble at the surface of the figurine itself or deposited from dilution of other neighboring marble figurines or calcitic rocks. The layer on top of it consists also mainly of calcite but it contains a higher amount of soil than the first layer (Table 8, layer 2). It also contains various inclusions such as potash and sodium feldspars (Fig. 25 B and 26). Above it is again a layer richer in calcite and contains also inclusions

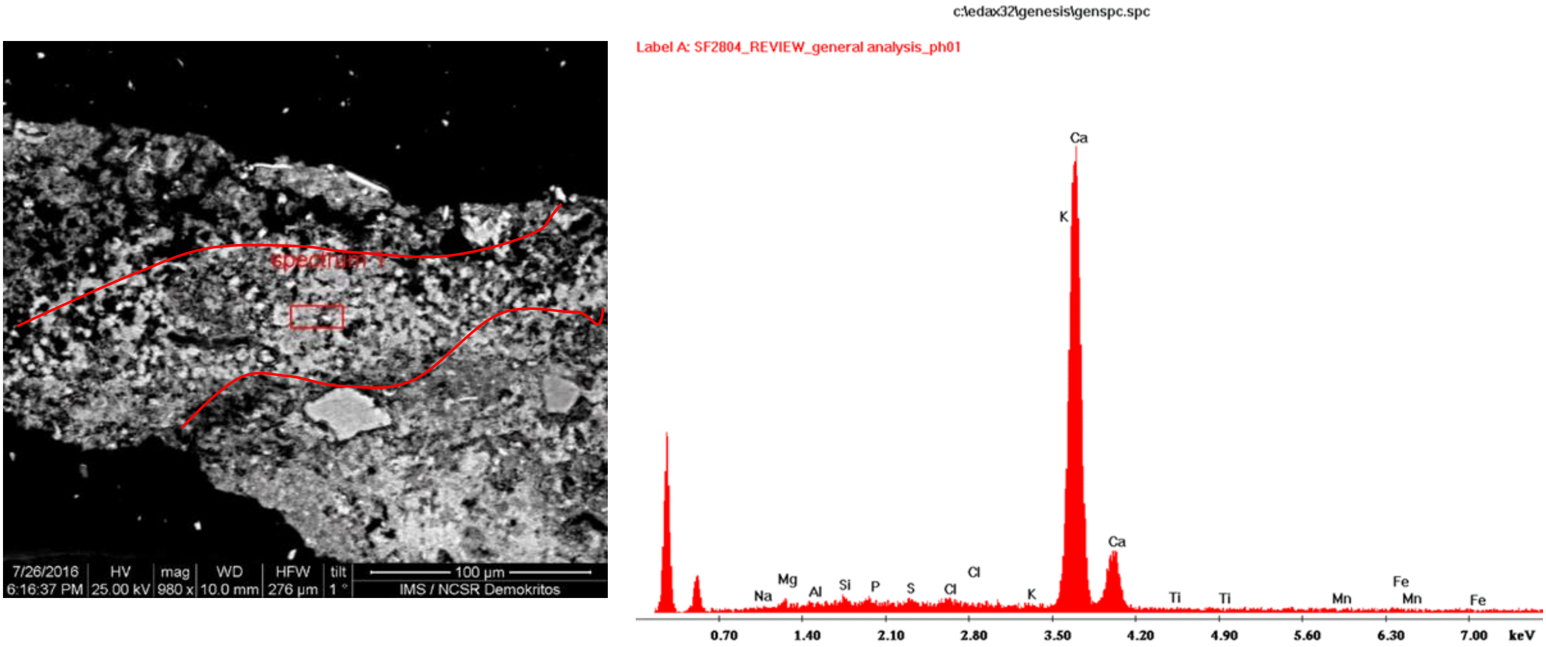


Fig. 26: 3rd analysis (Table 8): The analysis showed a pure calcium layer, with no other inclusions. The high pick of C originates from the carbonates and the carbon coating of the section. The cross section showed rounded marble crystals. The analyzed layer is marked with red lines.

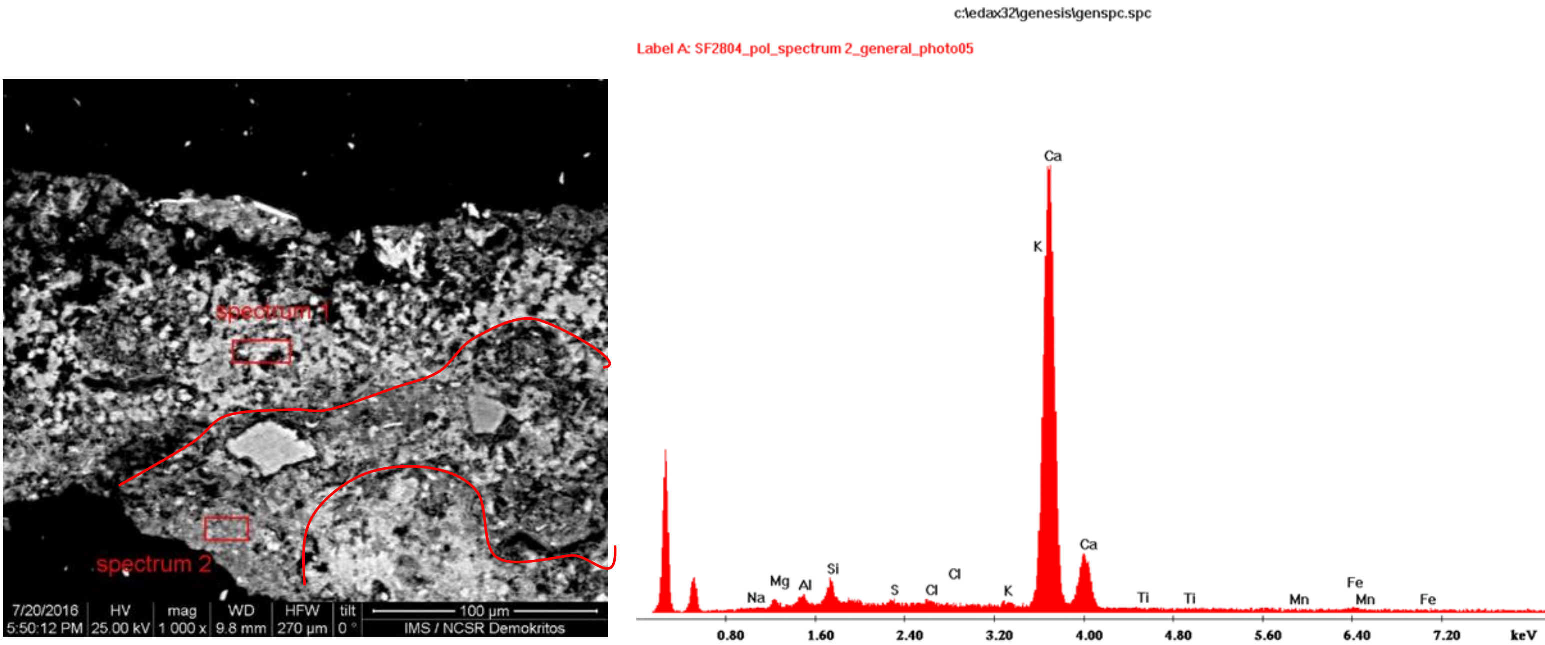


Fig. 27: 4th analysis (table 8): The third layer has darker color than the previous one (Fig. 26). The analysis showed an increase of Si, Al and Mg. The analyzed layer is marked with red lines.

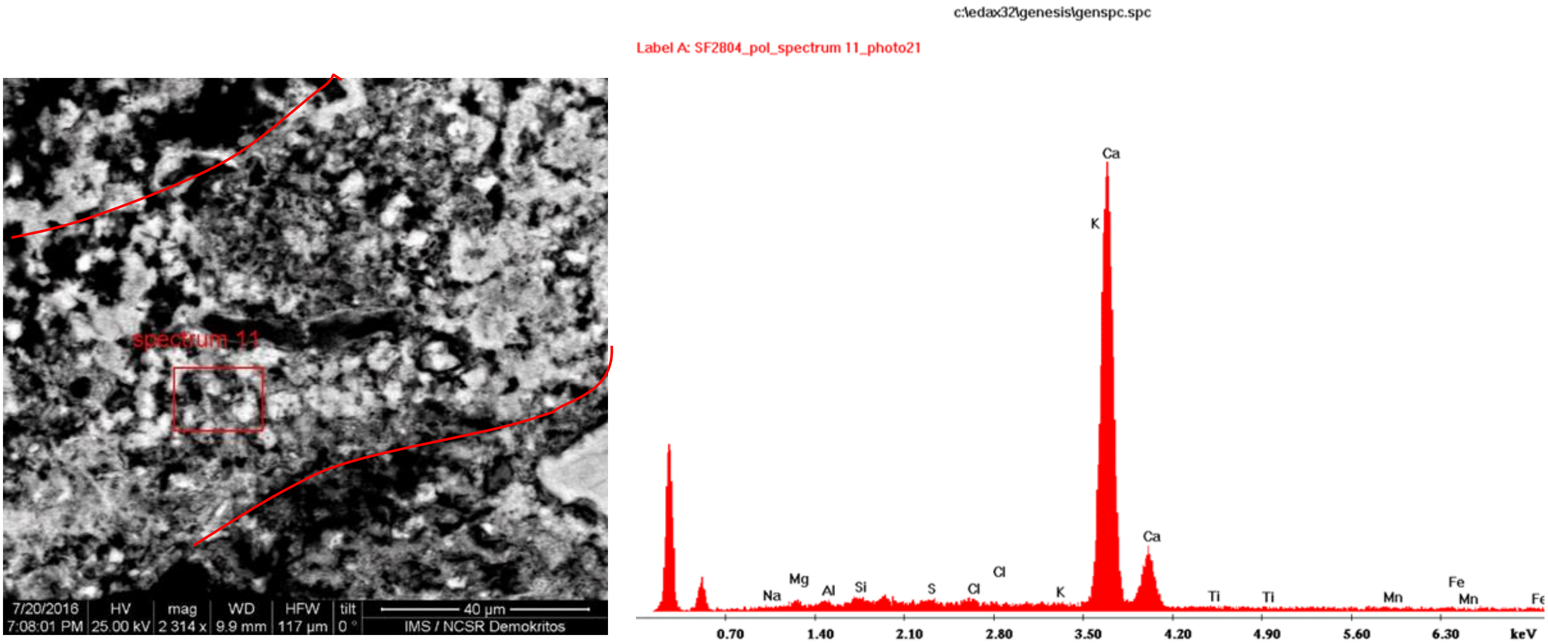


Fig. 28: 5th analysis (table 8): The analysis of the last layer (4th) showed an area with pure calcium element. At higher magnification the rounded marble crystals can be observed. The analyzed layer is marked with red lines.

The 5th analysis (Fig. 28, table 2) showed similar chemical concentration with the second layer (Fig. 26). The layer is consisted with pure calcium, no other elements can be detected. In higher magnification the rounded marble crystals can clearly be noticed (recrystallization). Not many inclusions can be noticed.

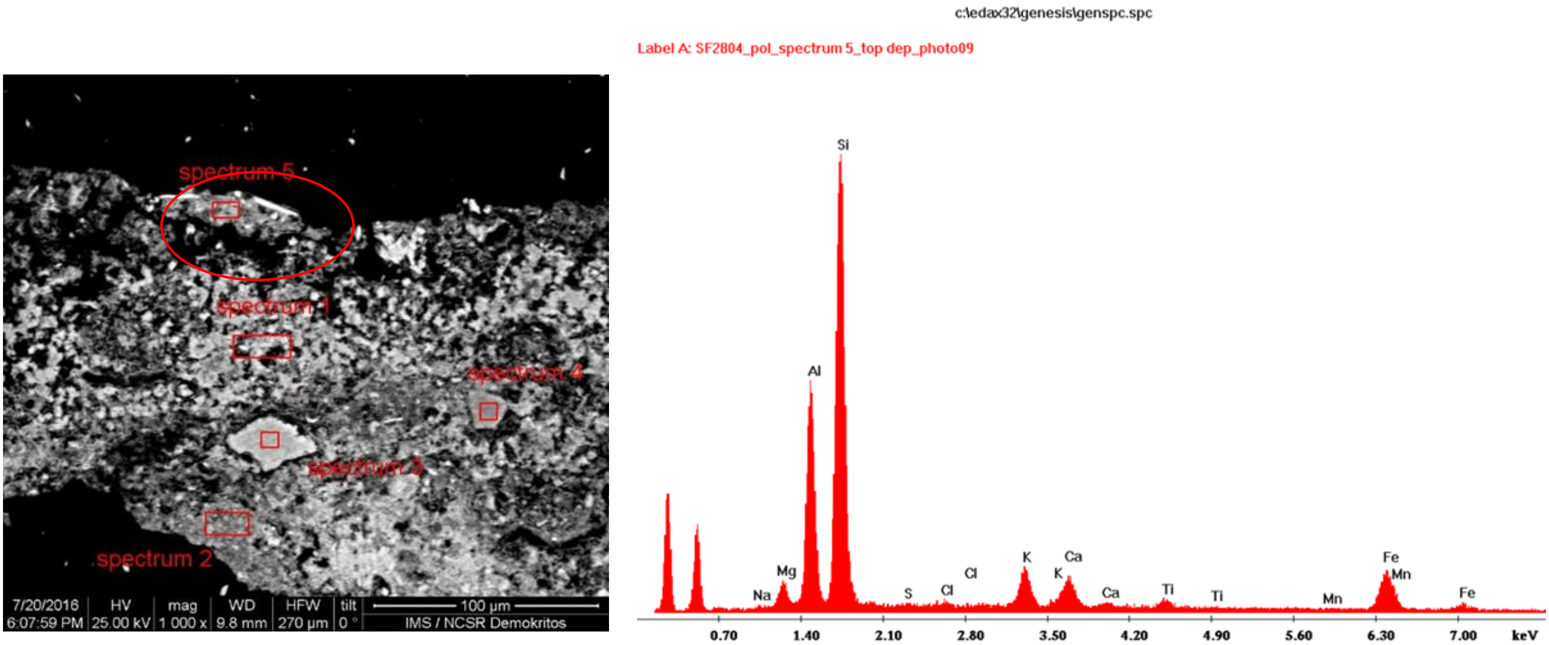


Fig.29: 6th analysis (table 8): The analysis of the external layer showed a typical clay layer. High concentration of Si and Al is detected as well as the elements of Mg, K and Fe. Two thin horizontal mineral can be noticed at the highest point of the layer. The analyzed layers is marked on a red circle.

On the external layer (Fig. 29, Table 8, an. no 6) the analysis showed high concentration of Si (57%) and Al (22%), as well as high level of Fe (9%) oxides. An increased amount of Mg (2.71%) was detected, possible coming from the soil materials. It is the typical clay composition that appears to be on the grains of each sample. However, a long shaped mineral - inclusion, has been observed in horizontal position at the surface (Fig. 30). The analysis of the horizontal mineral is presented below:

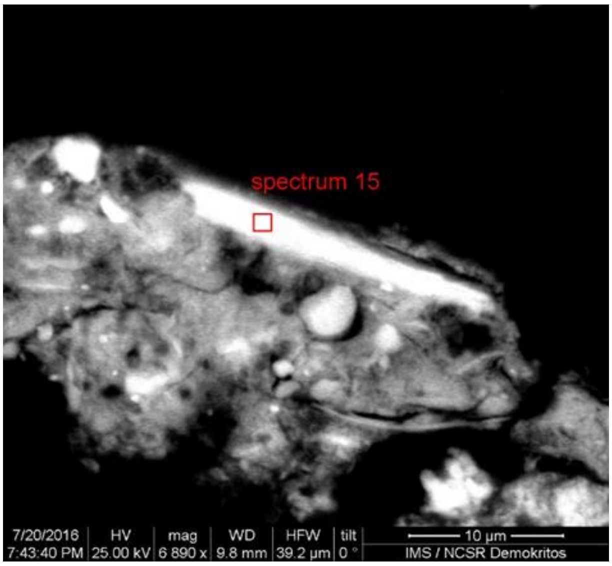


Fig. 30: (Table 8, an.no 7): The analysis showed that this mineral has increased amounts of K, Mg, Fe. It is most likely a mineral belonging to the mica group.

The analysis of an elongated inclusion (Fig. 30, Table 8, an.no 7) showed that this inclusion has increased amounts of Si, Al, Mg, Fe and normal K. Due to its flake like shape and chemistry we can conclude that it must be a mica flake from the soil. This group of philosilicates compose of sheets of silica tetrahedrons and alumina octahedrons and contain various elements as impurities. Such minerals are phlogopite, muscovite and biotite. The chemical formulas of these mineral had been checked. In the case of the 7^{th} analysis (table 8) it is possible that the inclusion belong to biotite group. The theoretical composition of biotite is: K_2O = 10.86 %, MgO = 23.24 %, Al_2O_3 = 11.76 %, FeO = 8.29 %, SiO2 = 41.58 %.

The whole structure and analysis of the crust on this figurine points to a layer formed composed of calcite and soil on the surface of the figurine in multiple layers. It is possible that this is a result of various environmental episodes where diluted calcite, either from the surface of the figurine itself or from the surrounding calcitic objects or rocks is deposited on the figurine together with soil containing also natural soil inclusions. The fact that this crust is not deposited on the whole surface of the figurine but in specific locations indicated that it was created inside the soil at the side of the figurine lying closer or in contact with a source of diluted calcium carbonate or a high acidity region of soil. Depending of the weather and the climatic conditions and the procedures inside the soil, such as underground water rich in diluted carbonates, fires, plant roots, microorganisms etc. different results can be seen on the surface of the figurine. The marble crystals of the figurine itself may have been diluted and recrystallized as calcite plaster at the surface encasing soil from the environment

Table 8: Chemical analysis in oxides form (100%wt) - SF 2804

No An.	SPECTUM	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl_2O	K_2O	CaO	TiO_2	MnO	Fe_2O_3	total %
	ASR													
1	general	0.16	2.48	4.60	10.8	0.86	0.87	0.29	0.68	76.90	0.18	0.28	1.94	100
2	deposition	0.68	3.89	23.79	49.3	0.86	0.92	0.22	2.49	12.00	0.41		5.42	100
	Polished													
3	1st layer	0.83	1.91	0.93	2.02	0.65	1.68	0.97	0.43	88.80	0.18	0.47	1.17	100
4	2nd layer	0.37	2.1	3.01	6.05		1.34	0.76	0.78	83.30	0.37	0.42	1.53	100
5	4th layer	0.87	2.53	1.90	2.55		1.39	0.89	0.46	86.80	0.44	0.92	1.23	100
6	External l.	0.14	2.71	22.16	57.30		0.31	0.3	3.36	2.93	1.40	0.29	9.12	100
7	Inclusion	0.35	14.00	17.43	47.40		0.36	0.38	5.77	1.04	1.06	0.87	11.35	100

Summary

The sample consisted of few grains of marble and some fragments of white crust of different sizes. This crust appears to be on the right arm of the figurine and on a back part. The crust is white with some spots of brownish depositions (clay depositions). The microstructure and the chemical composition of the white crust layer indicates that it is made by diluted calcium carbonate deposited on the surface of the figurine in different environmental episodes and trapping different amounts of soil in the different layers. It is possible that the crust has been created in underground conditions.

On other places on the figurine a brown-reddish soil layer has been deposited the marble surface. The composition of this soil layer indicates a fine clay with a typical and characteristic chemistry found on most of the figurines examined.

The SF 2804 as it is mentioned on the chapter 6 has been categorized as WD5 (Maniatis and Tambakopoulos 2015). The SEM analysis identified that the macroscopic observations for the degree of weathering and depositions on this figurine (category WD5, Maniatis and Tambakopoulos 2015) are due to a prior exposition to weathering conditions which damaged the surface and then a depiction of fine soil with a characteristic chemistry, rich in iron. The analysis also identified the nature of the white crust on certain parts of the surface of the figurine and revealed the sequence of events that led to its formation.

Figurine – SF 6274

The Special Find 6274 was identified as waist, pelvic and upper legs fragment of folded-arm figurine of Spedos variety (see Fig. 6).

Optical Microscope analysis

The samples (A and B) were checked under Leica stereoscopic microscope (Fig. 31 A, B). The sample A consists of a white color crust (recrystallized particles), with spotty yellow depositions on its surface (Fig. 31 A). The sample B consists of accumulation of large and smaller size particles that appeared to be heavily weathered. The majority of the particles have brown, dark brown depositions (Fig. 31 B) and are fragile. The Fig. 31 C, D was embedded in resin and the cross section is presented below (sample B). The photos are taken under petrographic microscope.

Fig. 31 A

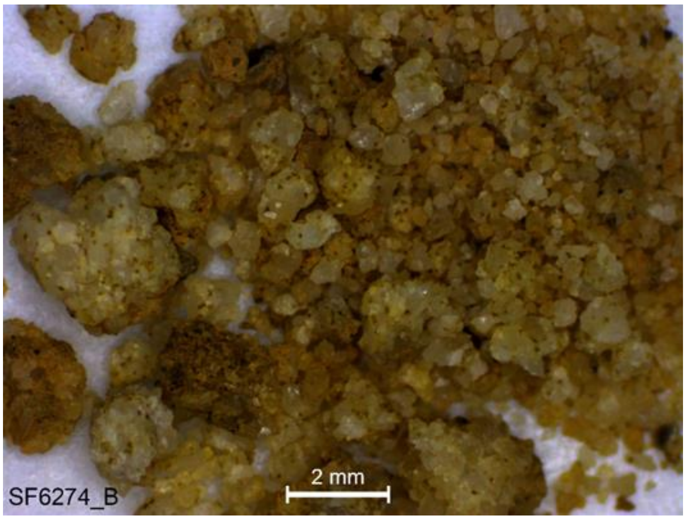


Fig. 31 B

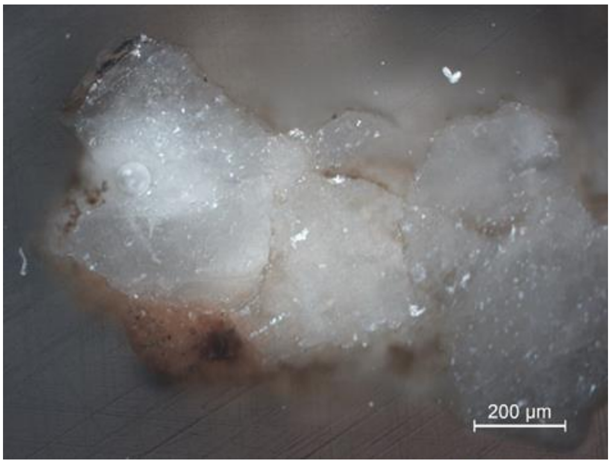


Fig. 31 C

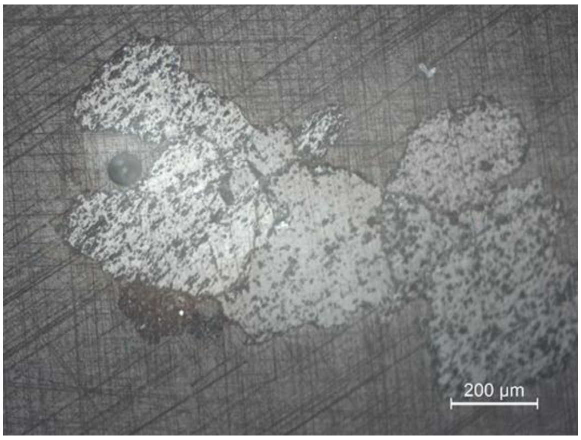


Fig. 31 D

Fig. 31 A, B: General photos of the sample A and B. The Fig.31 A shows a piece of the white crust (about 300mm), with yellowish brown spotty depositions. The Fig. 31 B shows different size particles with brown and dark brown depositions.

Fig. 31 C, D: Cross section of the polished particle (Sample B), under petrographic microscope in different polarization conditions. The detached particle can be observed, as well as the voids (Fig. 31 D).

The cross section of the polished sample showed yellowish – brown depositions at the lower part of the particle (Fig. 31 C). Voids can be noticed on the particle's surface (Fig. 31 D).

SEM Examination and Analysis

- Sample A

The sample A was analyzed in the ASR form (Fig. 31 A). The SEM image (high magnification) showed that the particle is composed by small rounded marble crystals, creating a crust (Fig. 32). The crystals are about 1-2μm.

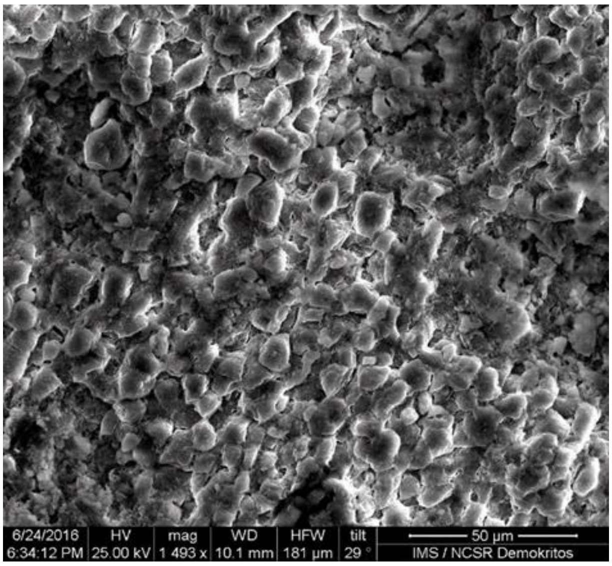
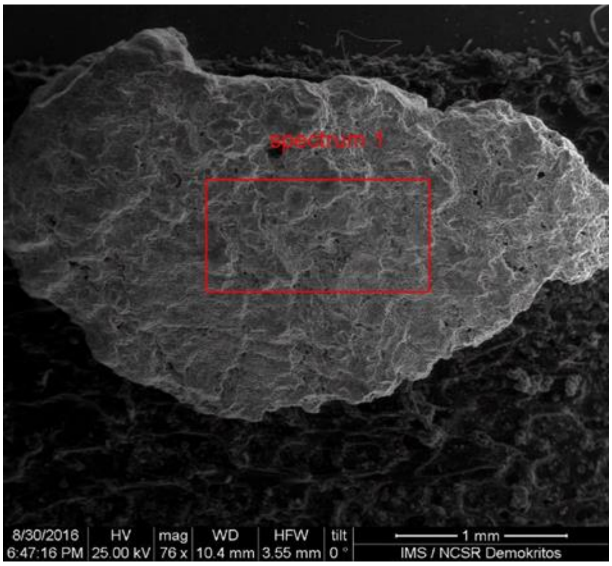


Fig. 32: Rounded marble crystals have created the crust (recrystallization). Each crystal is about 1-2μm.



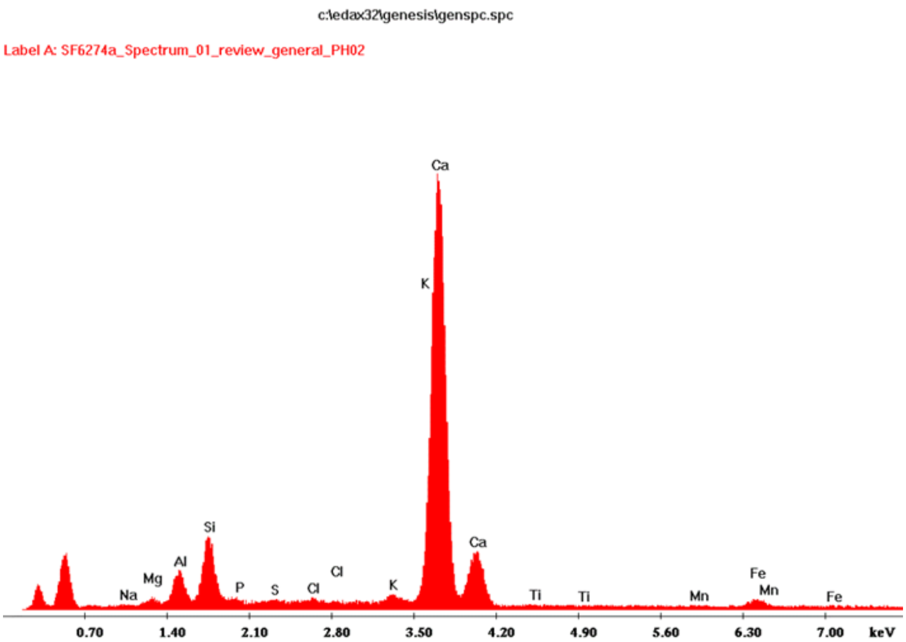


Fig. 33: 1st analysis (table 9): Left: The SEM image of the grain taken with the LFD detector at low vacuum. Right: the analysis spectrum taken with EDX-ray microanalysis system of the SEM at the region shown with the red square. The elements detected are Ca, while Si, Al and small amounts of Mg and Fe have been detected.

Figure 33 left shows the SEM image of one of the grains examined at low vacuum ASR. The spectrum on the right shows the analysis obtained with the ED X-ray analysis system of the SEM taken in the region indicated with the red square. As it can be seen the spectrum is dominated by a strong calcium peak plus oxygen and carbon with traces of silicon and Aluminum. This indicates a calcitic marble. The amount of CaO calculated to 88%, while the Al is 8% and the Si is 15% (Table 9).

The 2nd analysis (table 9) showed the spot of brown-yellowish deposition. Examination at the surface under the SEM at low vacuum ASR, looks grainy at the areas with the brown-yellowish deposition an indication of a fine particle composition (Fig. 34, left). The analysis at this surface (Fig. 34, right) showed apart from Ca, coming from the marble, some amounts of silicon, aluminum, Mg, Na, K, Fe. This indicates that the depositions on the surface of the grains consist of soil originating obviously from the burial environment. A high peak of O is also detected.

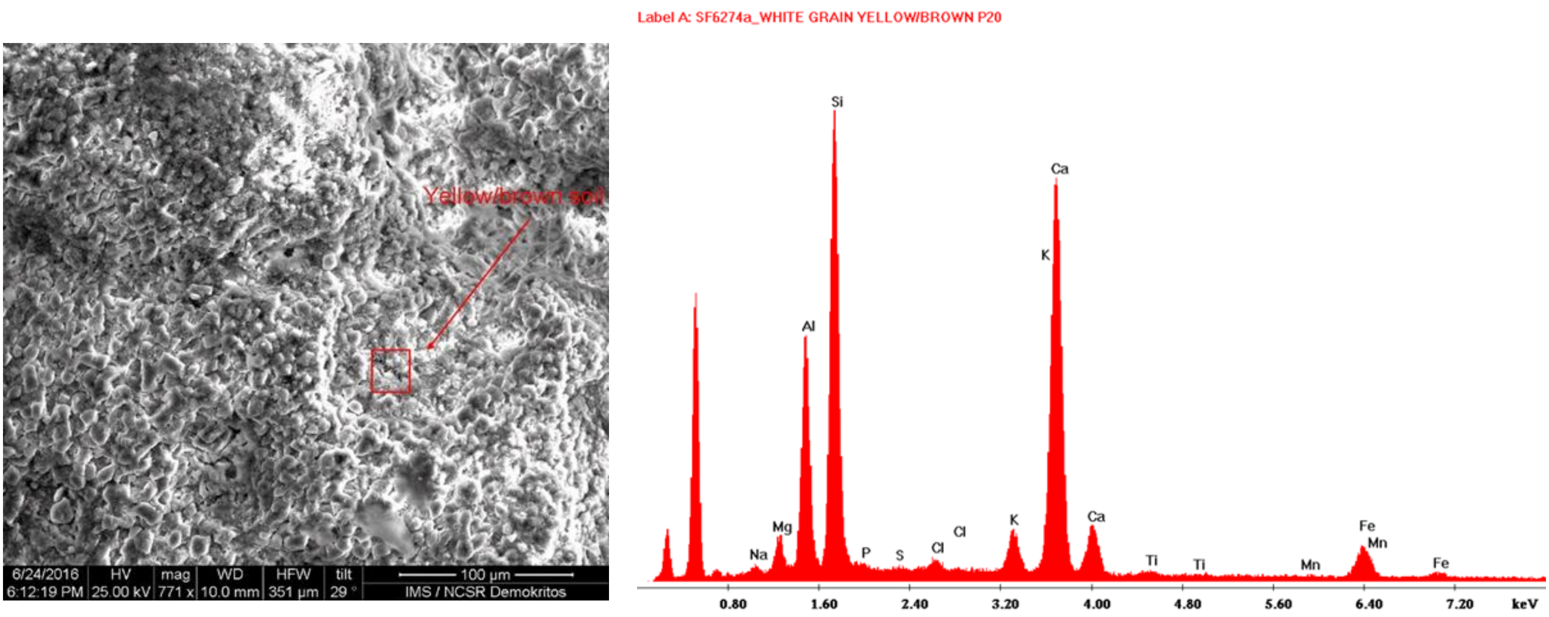


Fig. 34: 2nd analysis (Table 9): Left: The SEM image of the surface with yellowish-brown depositions Right: Analysis spectrum at the area indicated with a red square.

- Sample B

One representative particle from the sample B in the ASR form was examined and analyzed (Fig. 35).

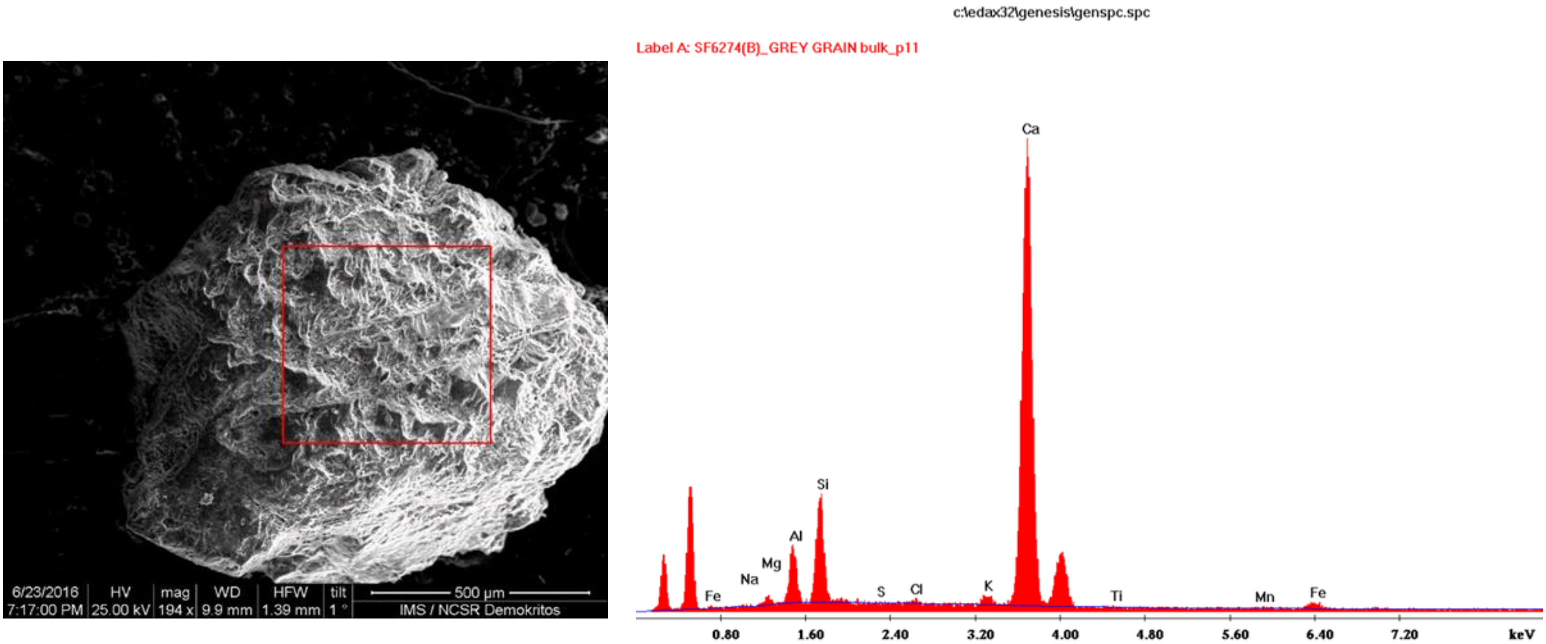


Fig. 35: 3rd analysis, (Table 9): Left: The SEM image of a grain taken with LFD detector at low vacuum. Right: the analysis spectrum taken with EDX-ray microanalysis system of the SEM at the region shown with red square. The elements detected are Ca, Si, Al, and O.

Figure 35 (table 9) left shows the SEM image of one of the grains examined at low vacuum ASR. The spectrum on the right shows the analysis obtained with the EDX-ray analysis system of the SEM taken in the region indicated with the red square. As it can be seen the spectrum is dominated by a strong calcium peak plus oxygen and carbon with traces of silicon and aluminum and oxygen. The high concentration of Ca consists thin layer of yellowish-brown deposition. The amounts of Si (19%), Al (10%) and Fe (2.8%)

A general analysis of the particle have be taken (3rd analysis, table 9). The yellow deposition is covering the marble particle. However due to its thinness, large concentration of Ca can be detected. The typical clay deposition showed 19% of Si and 10% of Al, while the is Fe is about 2,8% are typical for soil depositions.

- Polished Sample B

The polished cross-section was coated with carbon and examined and analyzed under the SEM in high vacuum in a backscattered electron mode (Fig. 36, 37, 38).

Fig. 36 shows the whole section consisting possibly of a single marble grain. A corroded grain had been chosen to be embedded in resin. Under the SEM, the photo showed that the grain has been detached and the surface of the grain is deeply weathered. Cracks have been observed around each part of the grain. Voids are observed to the upper left part and lower right part of the grain. Clay deposition can be seen around the grain (in different thickness).

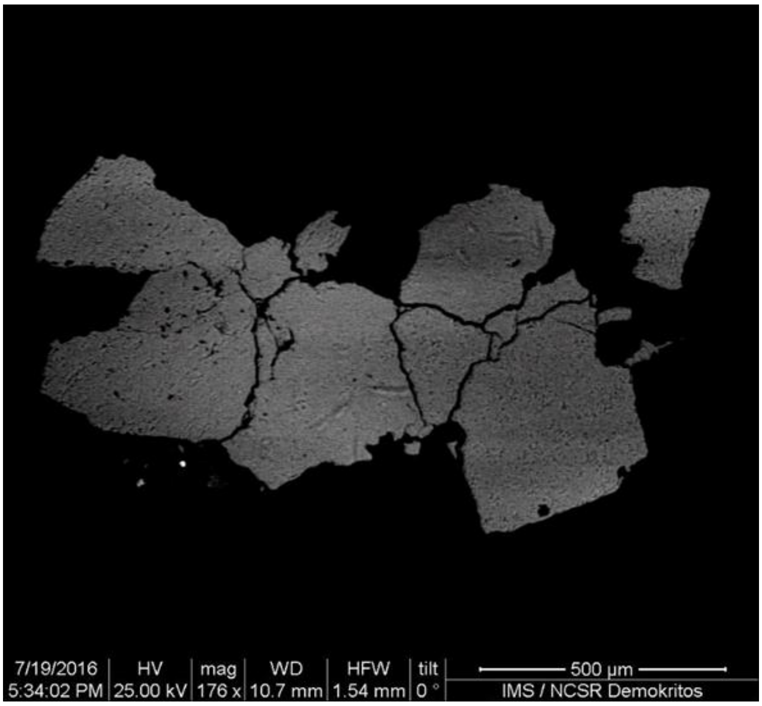


Fig.36: A general image of the polished sample SF 6274_B. The particle is detached in many large and smaller pieces. The surface is severely weathered. Voids can be noticed at the left part. Deposition can be observed at the lower left part of the particle

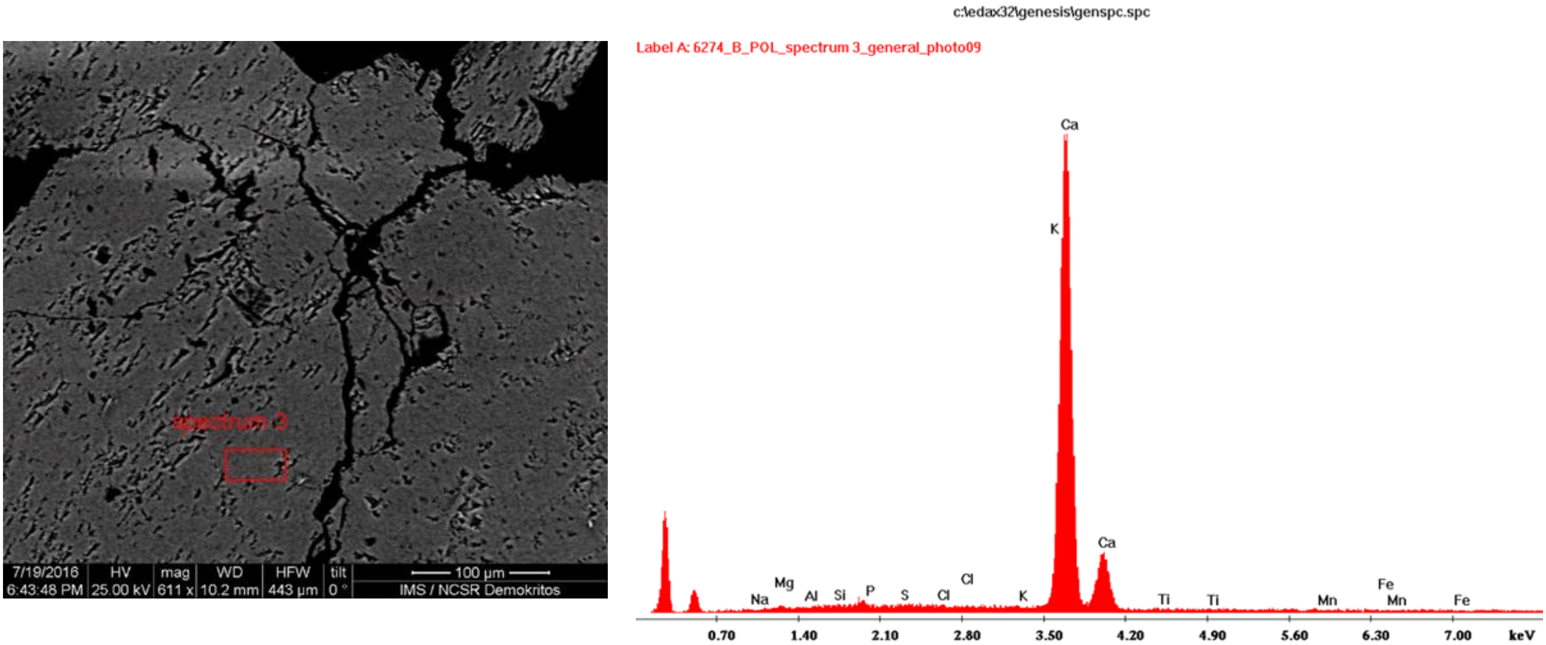


Fig 37: 4th analysis (Table 9): Left: a closer image of the inner part of the grain and the surface. Right: the analysis showed that it is pure Ca consistence, not other elements have been detected.

The analysis (no.an.4, table 9) showed that the particle is mainly consisted by Ca (95%). No other elements could be detected. Small and larger crucks can be seen. Voids can be noticed all over the particle surface.

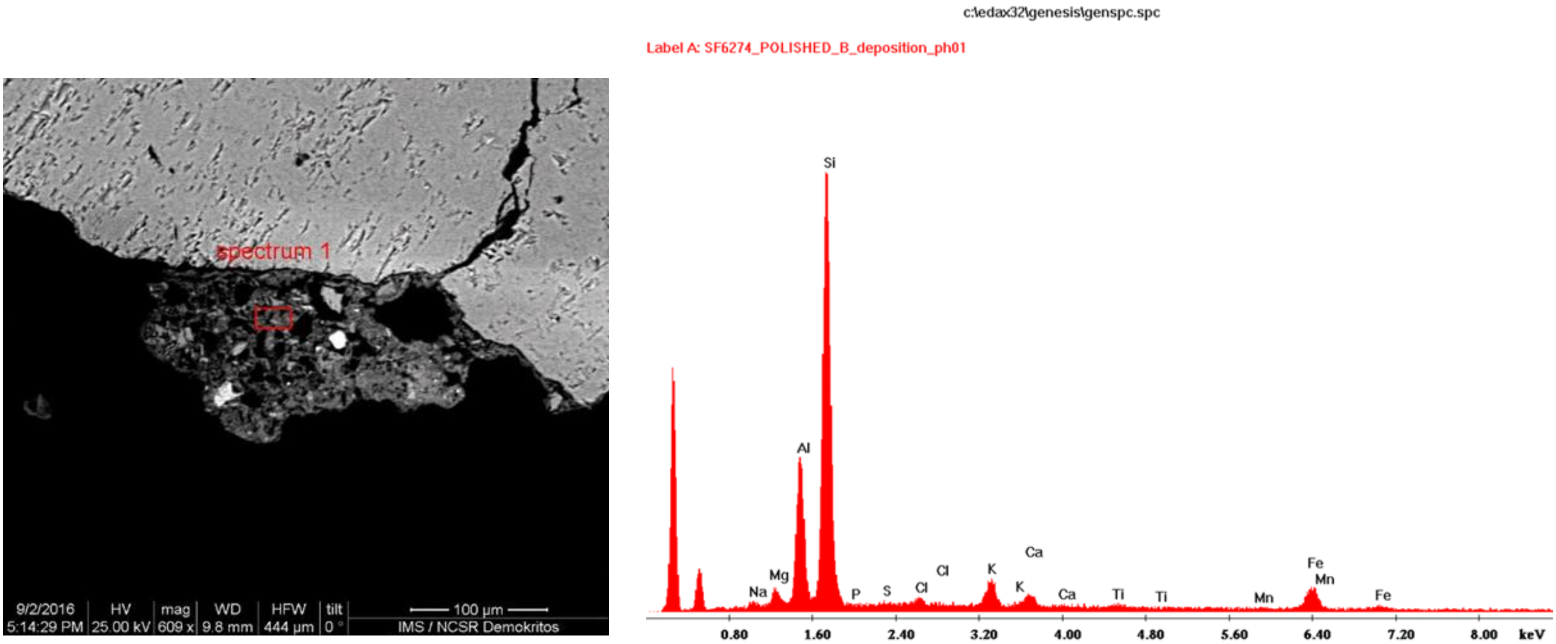


Fig 38: 5th analysis (Table 9): Left: SEM image of the surface with the yellowish-brown deposition. Right: Analysis spectrum at the area with a red square. A high peak of Si and C can be noticed. The thickness of the deposition is 126.26μm.

The analysis (no. an.5th, table 9) showed the typical clay deposition with increased Si (64%) and Al (18%). Other elements detected are Mg (2%), K (3%), Fe (6,6%) and minimum amounts of Na and Cl. The thickness of the deposition is 126.26μm. The larger inclusions which are part of the depositions are iron minerals, quartz, and marble pieces.

Table 9: Chemical analysis in oxides form (100%wt) - SF 6274

No An.	Sample	SPECTUM	Na2O	MgO	Al2O3	SiO2	P2O5	SO3	Cl2O	K2O	CaO	TiO2	MnO	Fe2O3	total %
		ASR													
1	**A**	marble	1.00	1.95	8.05	15.29	0.98	0.83	0.44	0.89	66.41	0.39	0.53	3.23	100
2	**A**	deposition	0.73	2.90	17.31	41.04	0.45	0.21	0.59	2.48	27.81	0.54	0.30	5.66	100
3	**B**	deposition	0.54	1.81	10.01	19.46	n.d.	0.14	0.40	1.03	63.14	0.46	0.39	2.63	100
		Polished													
4	**B**	marble	n.d.	1.14	0.32	0.50	0.58	0.89	0.37	0.30	94.73	0.49	0.37	0.31	100
5	**B**	deposition	0.70	2.06	18.31	64.17	0.35	0.89	0.98	2.99	1.66	0.85	0.39	6.65	100

Summary

The Special Find 6274 is separated in two parts. The first part (Sample A) is only one large, white grain with yellowish brown spotty depositions. It is part of a crust (recrystallized marble). The second part (Sample B) is a large amount of weathered marble particles, mainly with thick brown and dark brown depositions. The combination of the analysis from the sample A and B showed that the preservation state of the figurine is bad. The crucks are possibly created due to chemical or temperature changes. Possibly, the figurine was exposed to the environmental conditions (sun, sea, salt) and the marble absorbs salt from the sea, dry and the crystallized salts are destroying the marble grains. After a repetitive action, different layers of marble surface could be corroded. However, this phenomenon could be reduced after the figurine buried in the soil, due to the stable underground environmental conditions. The layer of clay around the grains, constitutes the last "environmental episode" of the weathering procedure.

This particular figurine as it was mentioned on Chapter 6 has been categorized by macroscopic examination as a heavily weathered figurine (WD = 5, Maniatis and Tambakopoulos 2015). The extensive weathering has been confirmed and each individual grain has been damaged. It also identified the origin of the depositions on the surface and the fact that the marble was first weathered and then buried.

Figurine – SF 20167

The Special Find 20167 was identified as upper legs fragment of folded-arm figurine of Spedos variety (See Fig. 7).

Optical Microscope Analysis

The sample was separated in two samples, A and B. The sample was checked under Leica stereoscope (Fig. 39 A, B, C, D). The sample A (Fig.39 A, C) consists small particles, white-transparent color, with yellowish brown. In few of the particles spotty black depositions can be observed. The sample B (Fig. 39 B, D) consists a small parts of white crust. On top of the crust a yellowish-brown deposition can be observed. Above the crust, pure marble grains can be noticed. On top of the crust spotty black depositions can be observed.

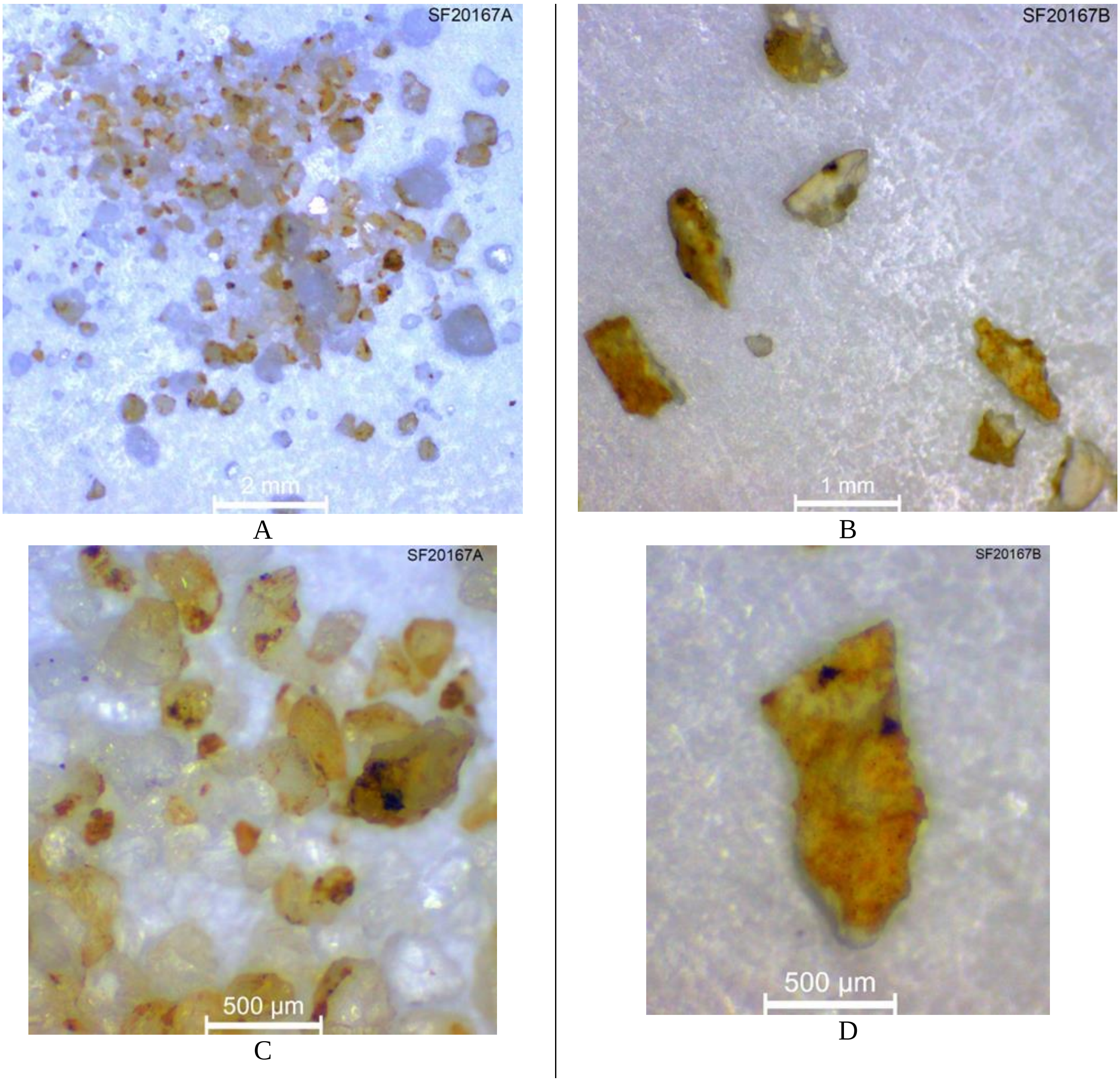
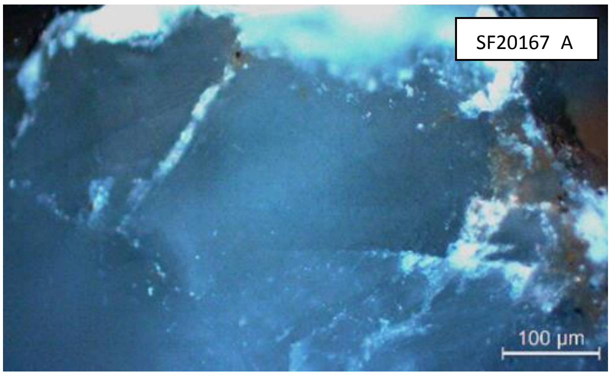
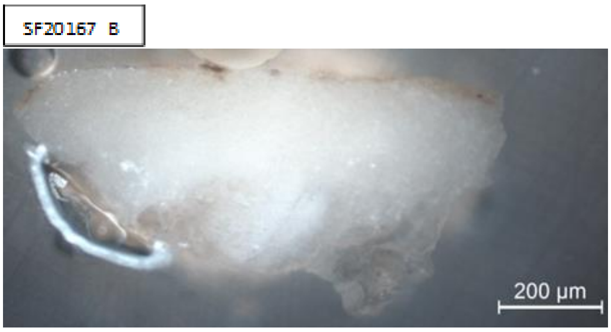


Fig. 39 A, B, C, D: General photo of the samples A and B. Sample A (Fig. 39 A, C): Different sizes of grains, white transparent color and yellow/brown depositions. Sample B (Fig. 39 B, D): Parts of the

white crust in different sizes, white crust with yellowish-brown depositions on top. Photo taken with Leica Stereoscope.



A



B

Fig. 40 A, B: Cross section of the polished samples under the petrographic microscope in different polarization conditions. Left: ***The Sample A*** *(Fig. 40 A) is a clean marble grain, a large cruck can be observed. Right:* ***The Sample B*** *(Fig. 40 B) is a part of the whit crust. On top a yellowish brown deposition can be noticed (black spot on the top-middle.*

Two particles of each sample were embedded in resin and the cross sections are presented above. The Fig. 40 A is a small particle of marble (about 350μm). A large cruck on the surface of the sample can be noticed on the left part of the particle. Yellowish brown deposition can be observed in few areas of the particle. The Fig. 40 B is part of the white crust, about 500μm. A layer of yellowish brown deposition can be observed on the top of the particle. A small black spot can be seen on the middle top of the grain.

SEM Examination and Analysis

- Sample A

One grain (Fig. 41 A, B) was chosen for further examination and analysis under the SEM in the ASR form and the grain in polished cross section embedded in resin (Fig. 41 A).

A yellowish-brown deposition can be seen on the surface of the grain. Black deposition is been observed next to the yellow deposition (Fig. 41 B).

Fig. 41 A

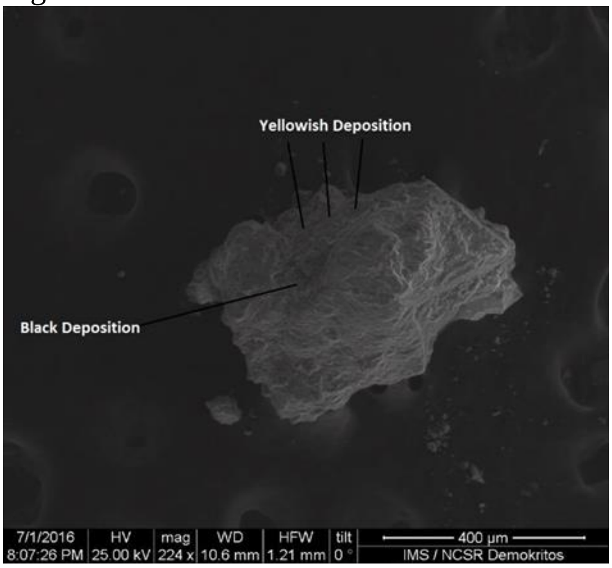


Fig. 41 B

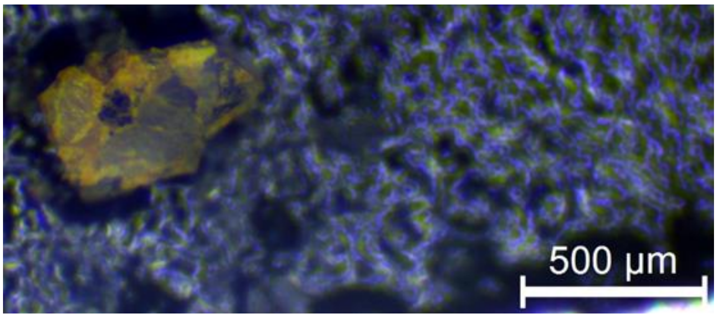


Fig.41 A, B: A grain is examined and analyzed (about 300μm). Yellowish deposition is covering a large area of the particle and with a black spot in the center.

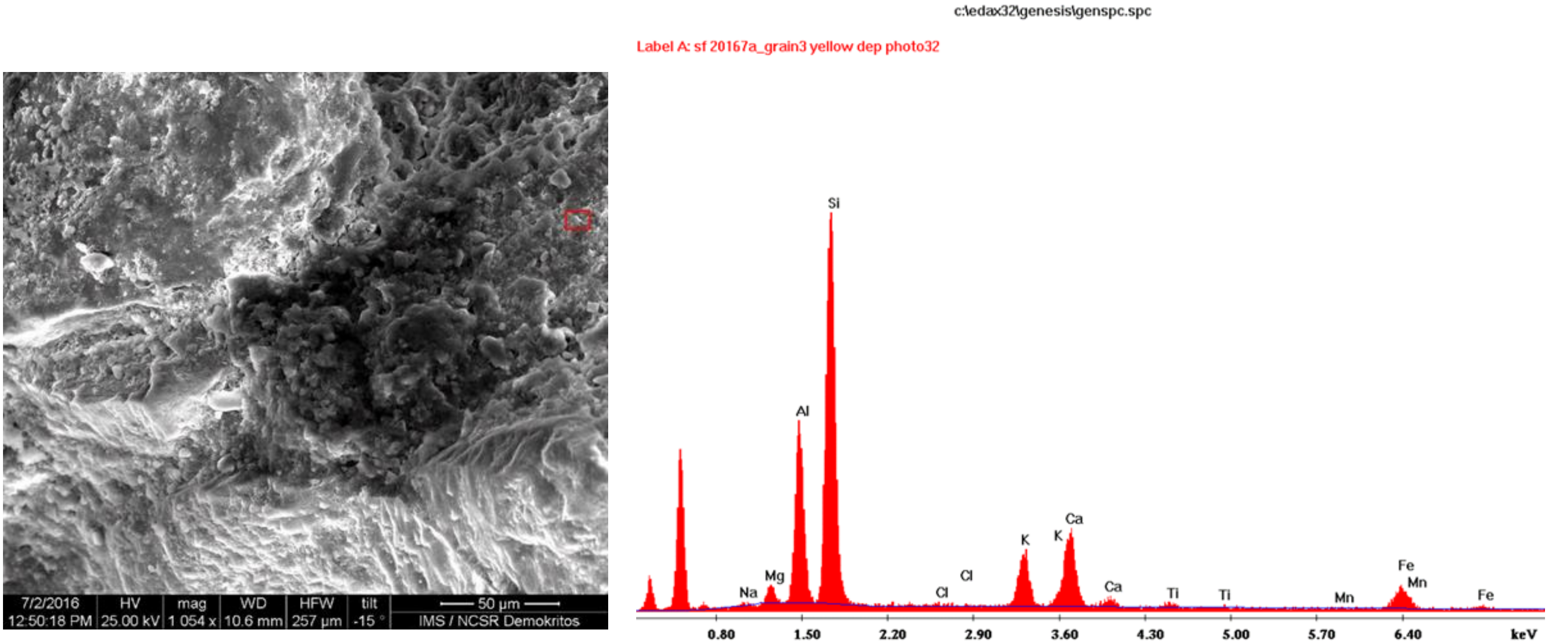


Fig.42: 1st analysis, (table 10): Left: The SEM image of a grain taken with the LFD detector at low vacuum. Right: the analysis spectrum taken with EDX-ray microanalysis system of the SEM at the region shown with the red square. The elements detected are Si, Al and O (higher peaks).

The Fig. 42 (no. an 1, table 10) the typical clay deposition has been observed. The soil depositions consists also a large amount of Fe_2O_3, almost 6% and K 5.14%. The amount of Ca comes from the marble substrate. A darker area has been spotted next to the yellow deposition.

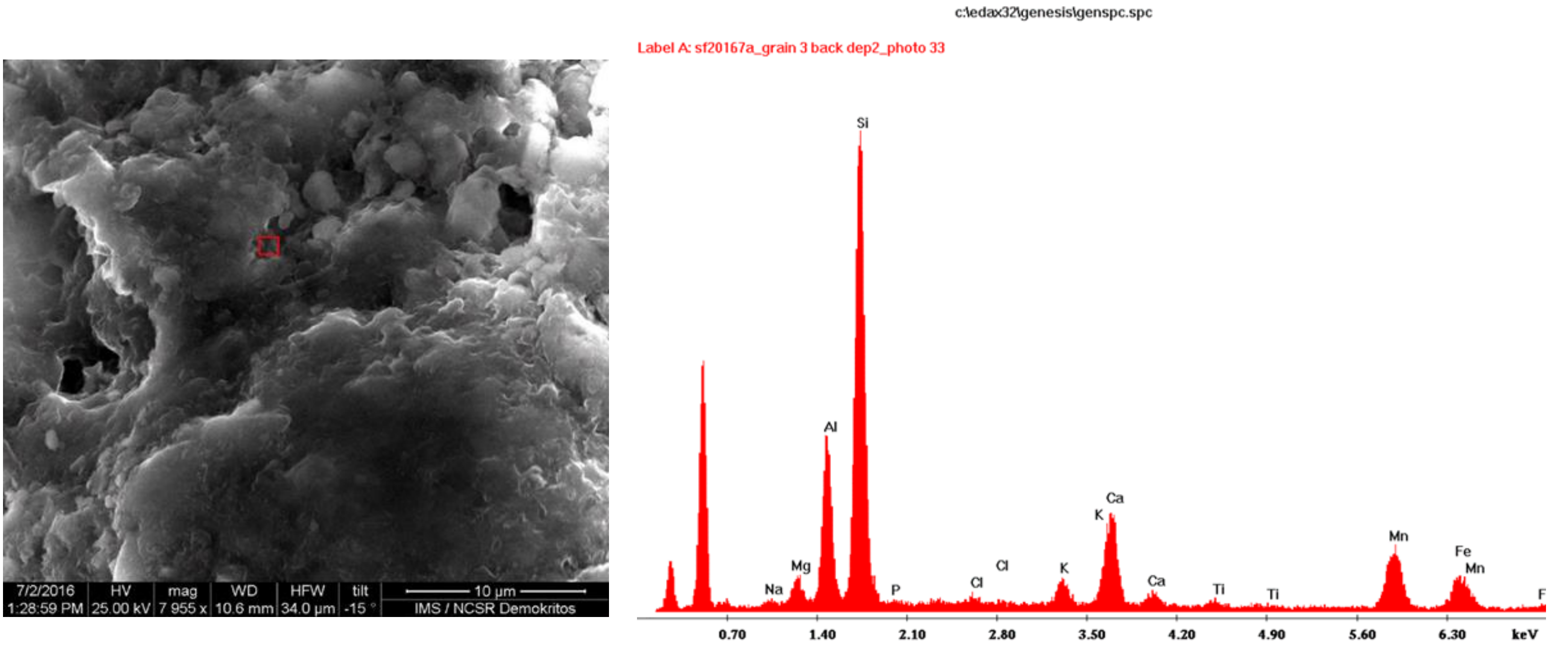


Fig. 43: 2nd analysis, (table 10): Left: SEM image of the surface with the black deposition. Right: the analysis spectrum at the area indicated with a red square. High concentration of Mn (11%) has been detected.

The 2nd analysis (table 10) showed an area with high concentration of Si (50%) and Al (19%) as well as increased Mn (11%) and Fe (6.5%). The color of the deposition caused by the iron and manganese oxides. The deposition possible is resulted by soil inclusions.

- Sample B

A piece of the crust (Fig. 44) chosen for further examination and analysis under the SEM in the ASR form and the grain in polished cross section embedded in resin (Fig. 39).

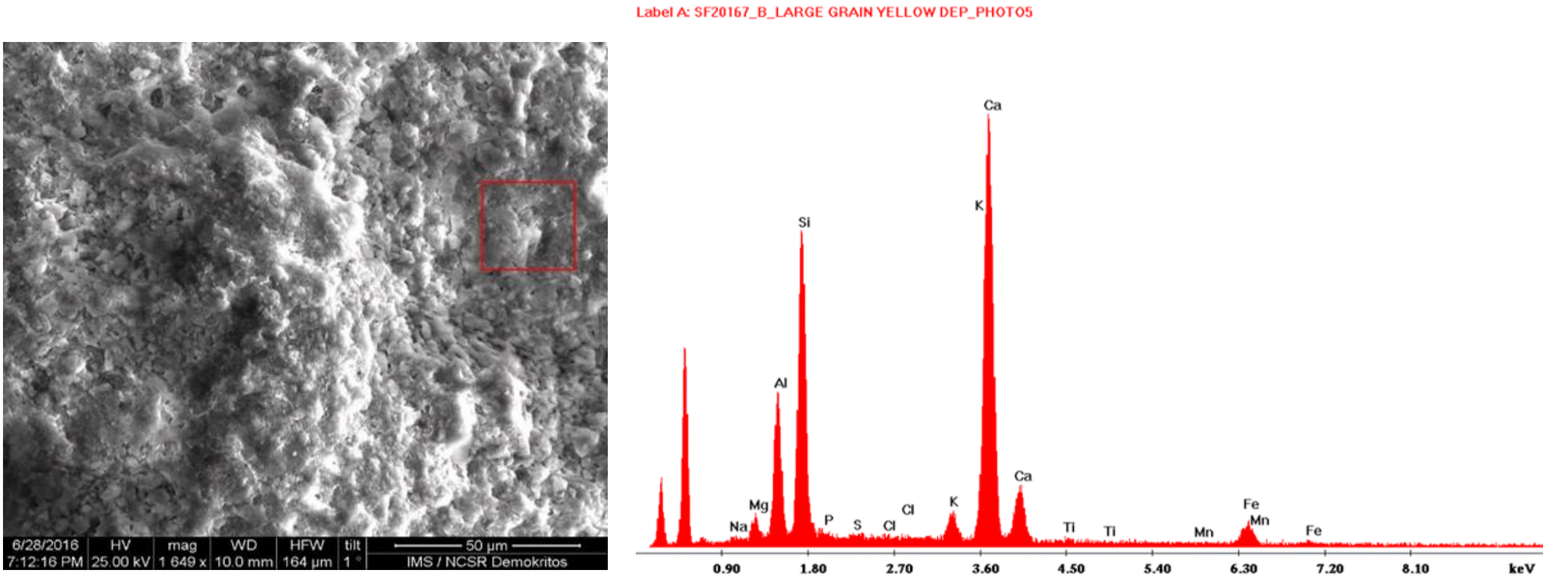


Fig. 45: 3rd analysis, (table 10): Left: SEM image of the surface of the crust with the yellowish-brown layer. Right: Analysis spectrum at the area indicated with a red square. High peaks of Ca, Si, Al and O can be observed.

The typical clay chemical composition has been detected (no. an. 3, table 10). The high concentration of Ca comes from the marble substrate (thin deposition). High amounts of Si (36%) and Al (15%) have been detected. Low amounts of other elements have been detected such as Mg, K, and Fe.

- Polished Sample A

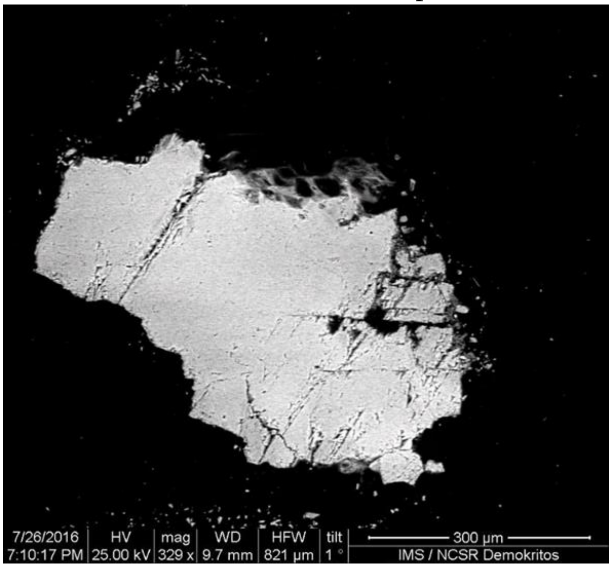
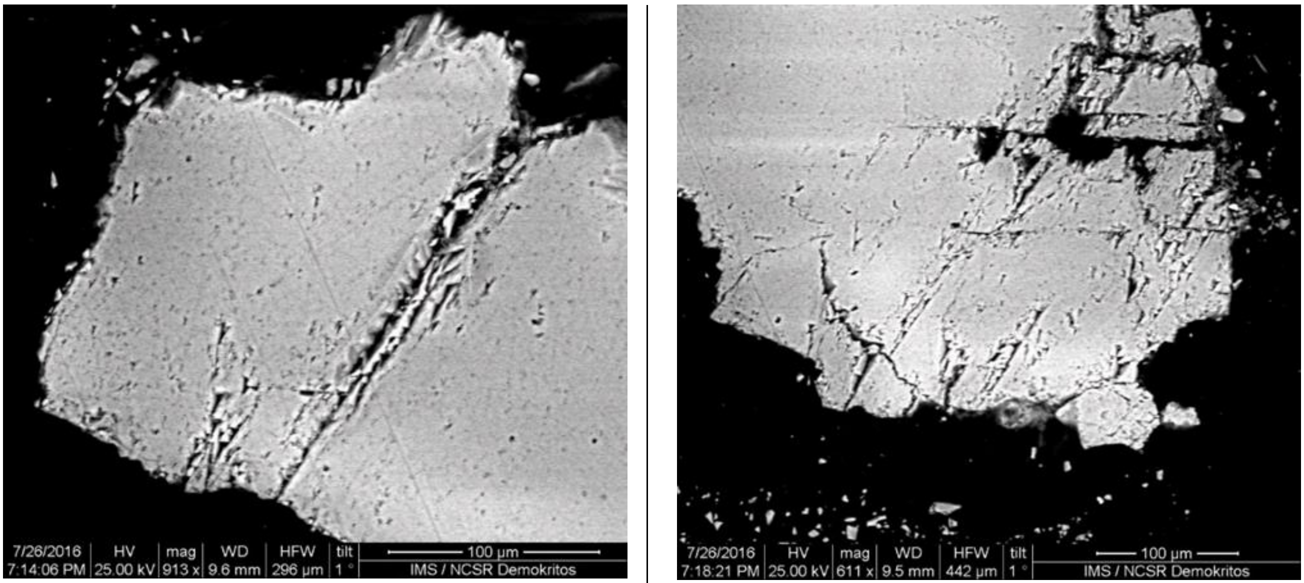


Fig. 45 A: Image of the cross section of the polished particle. Large and smaller crucks can be noticed. On the right part of the grain broken part of the particle can be observed. Thin deposition spots can be seen at the right part.

The Fig. 45 was coated with carbon and examined and analyzed under the SEM in high vacuum in a backscattered electron mode. A closer image (Fig. 45 B, C) shows that the inner part of the grain. Voids, small and larger crucks can be observed.



B C

Fig.45 B, C: Detailed imaged of the cross section of the polished particle. Voids can be observed in the surface of the particle mainly on the right part (Fig. 45 C).

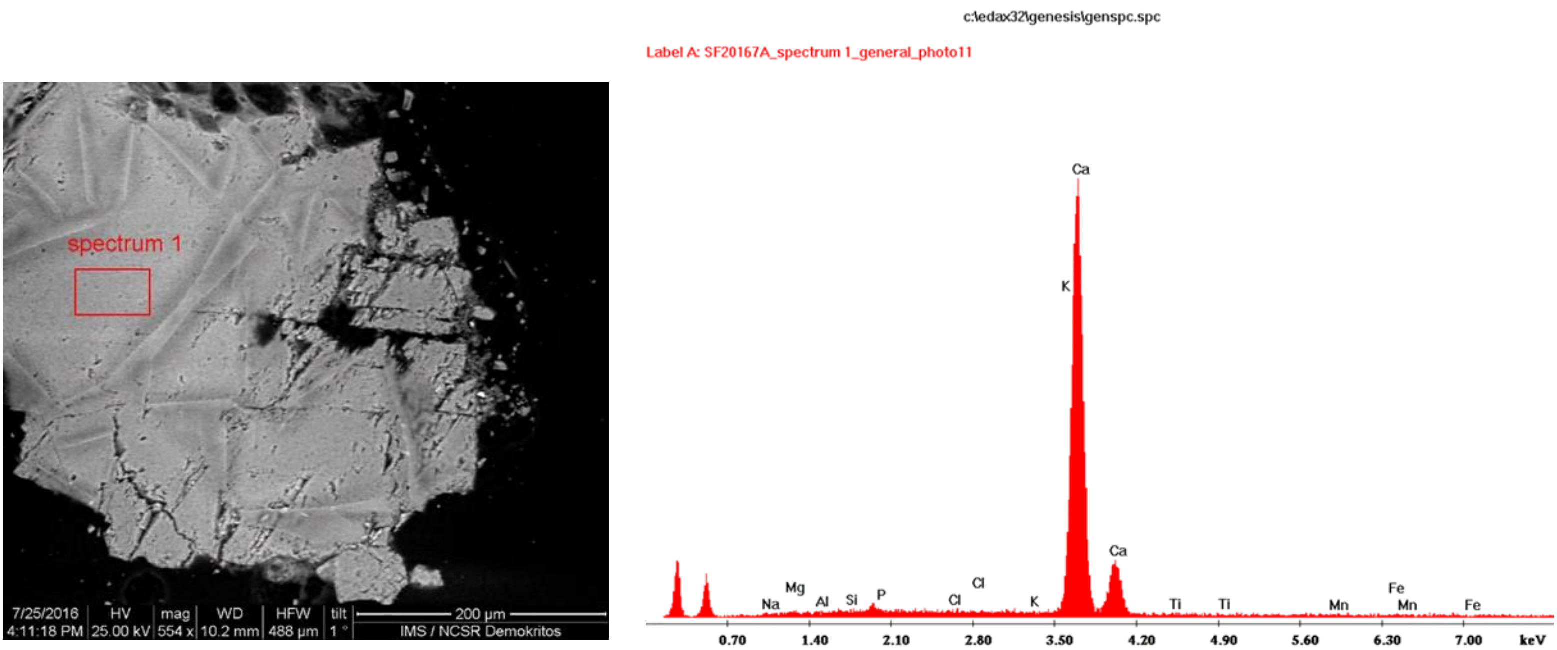


Fig. 46: 4th analysis, (table 10): Left: a closer image of the inner part of the grain and the surface. Right: the analysis showed that the particle's consistence is pure marble, not other elements have been detected.

A closer image of the grain and its spectrum are shown in Figure 46. The inner part of the grain body shows the chemistry presented in Table 10, an.no. 4. It appears to be pure calcitic marble however containing a very small amount of dolomite as can be assessed from the small amount of Mg present.

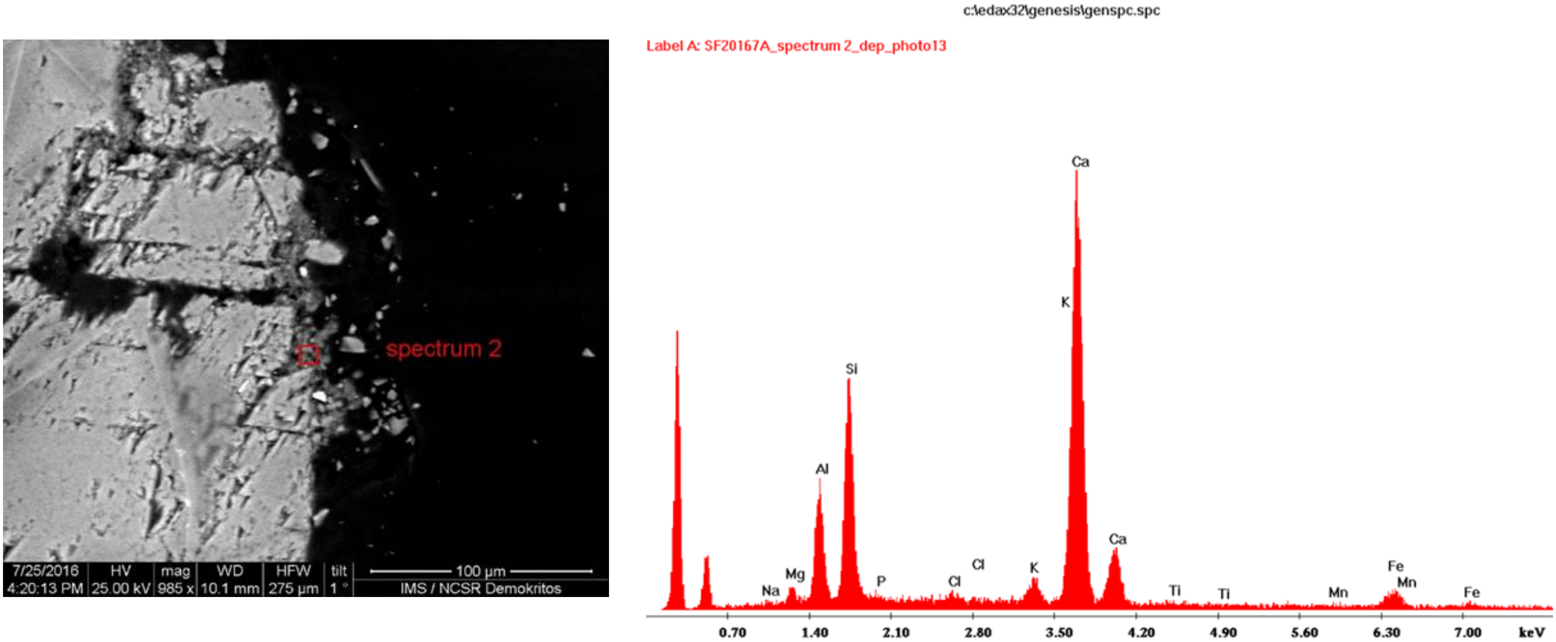


Fig. 47:5th analysis, table 10: Left: a closer image of the deposition. Right: the analysis showed the typical clay deposition. The high concertation of Ca is coming from the surrounding marble particle.

The 5th analysis (table 10) showed the typical clay deposition, consisted by Si and Al. A high peak of Ca can be noticed (detected by the surrounding marble grain). Parts of the marble were broken and stacked on the soil deposition (white inclusions on the soil, Fig. 47). Other deposition's inclusions are K-feldspars and quartz grains.

- Polished Sample B

The polished cross-section (Fig. 49) was coated with carbon and examined and analyzed under the SEM in high vacuum in a backscattered electron mode (Fig. 49, 50, 51). Figure 48 shows the whole section consisting of a crust particle with marble crystals on the bottom. Two long rectangular inclusions are observed on the right part of the grain (top and bottom) On top of the grain a thin layer of clay deposition can be noticed (Fig. 48).

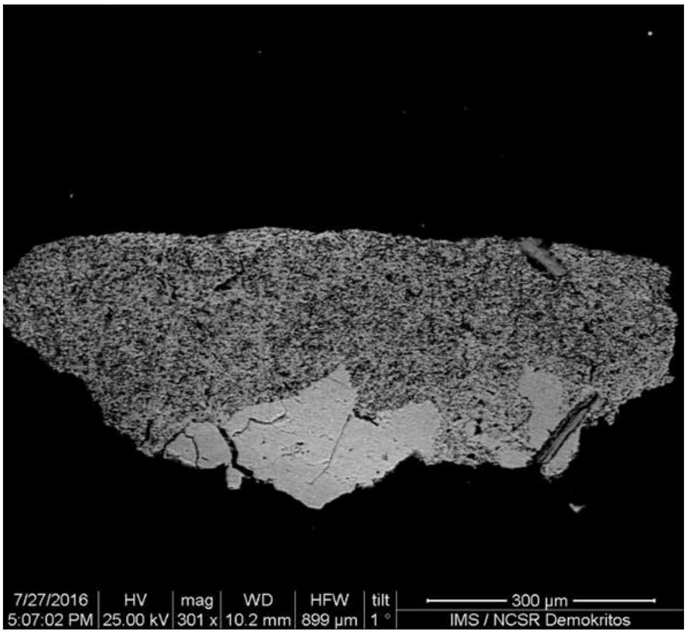


Fig. 48: General Image of the polished sample 20167_B. A thick crust can be seen (maximum thickness 150µm). Clear marble crystal can be observed under the crust. Two large inclusions were noticed on top and bottom on the right part of the particle.

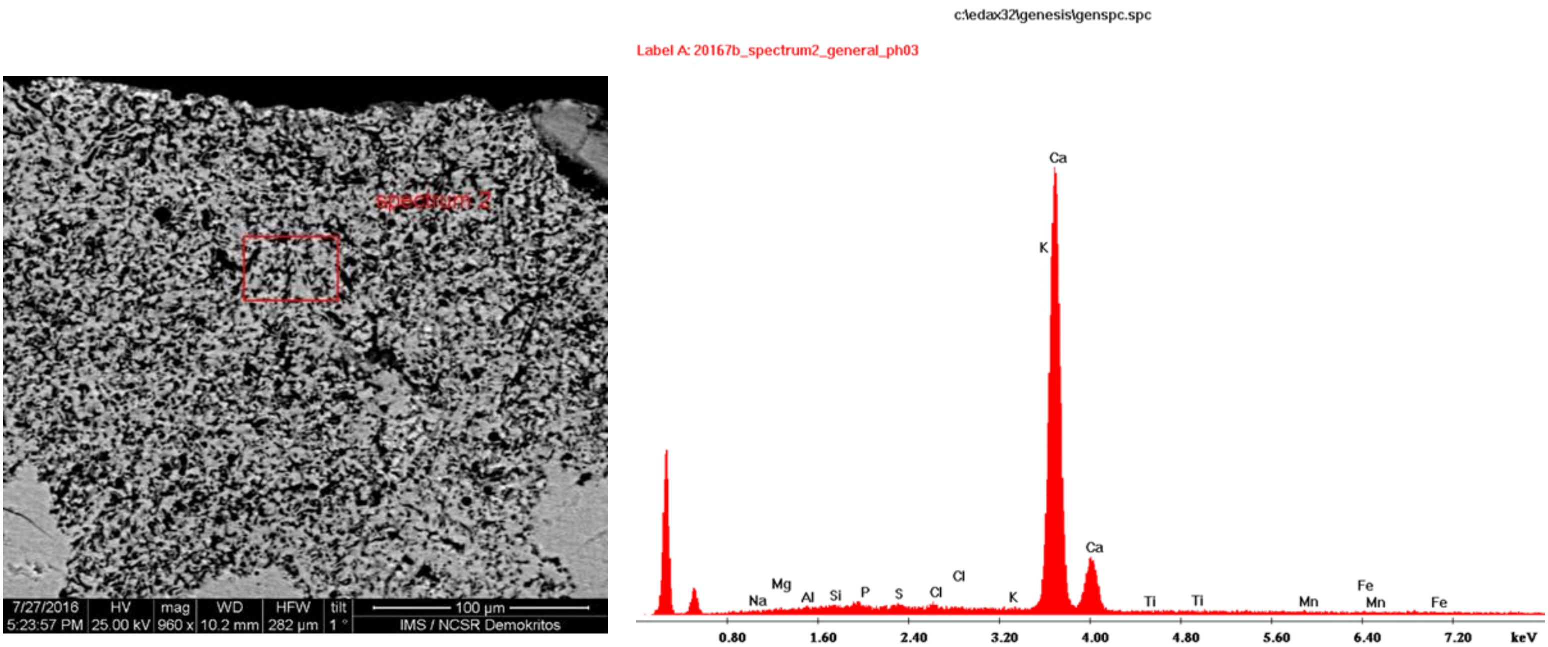


Fig. 49: 6th analysis, (table 10): Left: a closer image of the inner part of the crust. Right: the analysis showed that the crust is pure Ca. Rounded and different shapes of marble crystals create the crust (recrystallization).

The Fig. 49 shows image of the inner part of the crust. The analysis (an.no.6, table 10) shows that the grain consists pure calcium. The white crust is about 200µm thick. Possibly, it has been dissolute and recrystallized. No other elements could be detected.

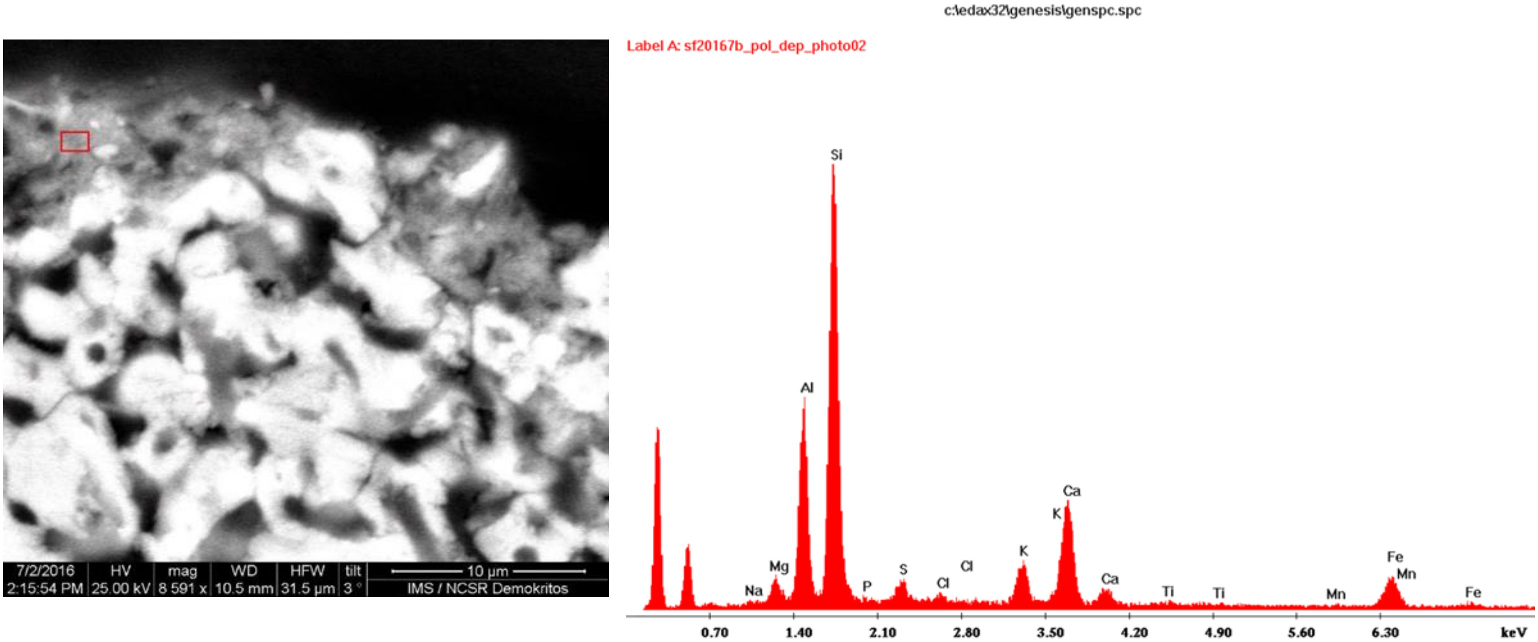


Fig. 50: 7th analysis, (table 10): Left: a closer image of the clay deposition. Right: the analysis showed the typical concentration of the clay deposition. High peak of Si, Al and C can be observed.

On top of the crust, a clay deposition has been detected (Fig. 50). The deposition has the typical chemical components, almost 52% Si and 19% of Al. Increased iron oxides, almost 7%, have also been detected. The amount of Ca is detected from the surrounding area. The clay deposition on the crust was thin (14μm.) and it appeals to the last environmental episode inside the soil.

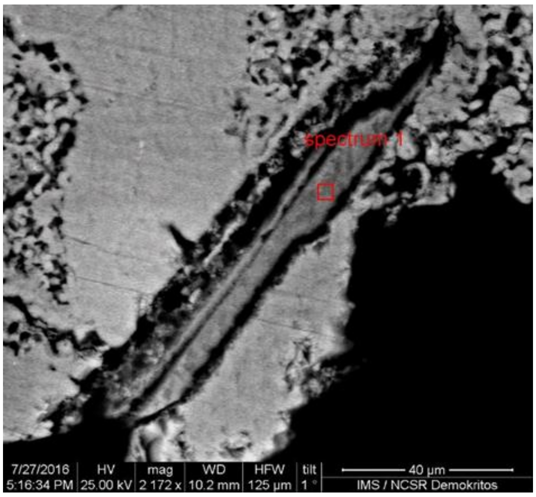


Fig. 51: 8th analysis, (table 10): The large inclusion at the bottom of the crust has been analyzed. The mineral has long rectangular shape. The analysis showed K-feldspar possibly categorized at the group of orthoclases.

This grain (Fig. 51) and the upper grain have similar chemical composition. Both of the grains are belonging to the group of K-Feldspar minerals and more specifically at the group of orthoclases, However, in this case the Al oxides are much higher amounts compared to the orthoclases chemical formula (formula aluminum oxides: 18-19%).

Table 10: Chemical analysis in oxides form (100%wt) - SF 20167

No An.	Sample	SPECTUM	Na2O	MgO	Al2O3	SiO2	P2O5	SO3	Cl2O	K2O	CaO	TiO2	MnO	Fe2O3	total %
		ASR													
1	**A**	deposition	0.46	2.30	20.61	55.77	n.d.	n.d	0.26	5.14	8.54	0.96	0.21	5.74	100
2	**A**	deposition	0.47	2.94	18.98	49.55	0.24	n.d.	0.63	2.56	5.58	1.24	11.25	6.57	100
3	**B**	deposition	0.55	2.52	14.77	36.06	0.55	0.58	0.14	1.94	37.48	0.53	0.25	4.64	100
		Polished													
4	**A**	marble	0.89	1.63	0.64	0.77	1.03	n.d.	0.37	0.35	93.11	0.37	0.28	0.56	100
5	**A**	deposition	0.83	2.45	13.98	29.78	0.88	n.d.	0.84	2.23	43.26	0.95	0.36	4.44	100
6	**B**	marble	0.45	0.71	0.87	1.11	1.11	1.49	0.78	0.38	91.87	0.42	0.41	0.39	100
7	**B**	deposition	0.37	2.56	19.17	52.41	0.32	3.41	0.61	3.38	10.13	0.58	0.20	6.85	100
8	**B**	inclusion	0.62	1.52	33.00	48.66	0.19	0.63	0.47	11.01	3.31	0.13	0.11	0.35	100

n.d.=not detected.

Summary

The Special Find 20167 is separated in two parts. The first part (Sample A) is including medium and small marble particles with yellowish brown depositions. The second part (Sample B) is including parts from white crusts, probably a pieces of recrystallized marble grains. The chemical analysis showed similar chemical compositions with increased iron oxides. In addition, the polished samples showed that the clay layer is consisted by soil, feldspars inclusions, quartz minerals etc. Polished samples showed the great damage of the marble crystals of the figurine. Large cracks have been observed on the both polished samples. The cracks are possibly made due to the acidic soil. The Polished sample B is a thick crust of recrystallized marble grains.

This particular figurine as it was mentioned on Chapter 6 has been categorized by macroscopic examination as a heavily weathered figurine (WD =4, Maniatis and Tambakopoulos 2015). The detailed examination and analysis of the grains in the sample obtained from this figurine confirms the extensive weathering and determined and the creation of a crust layer in combination with the clay absorption and the creation of a thin clay layer on top of the crust.

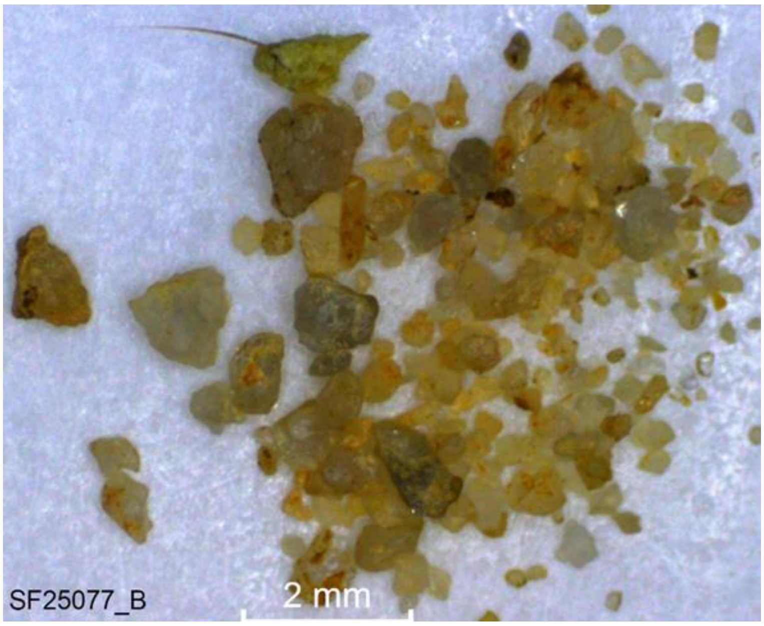
Figurine – SF 25077

The Special Find 25077 was identified as upper legs fragment of folded-arm figurine of Spedos variety (see Fig. 8).

Optical Microscope Analysis

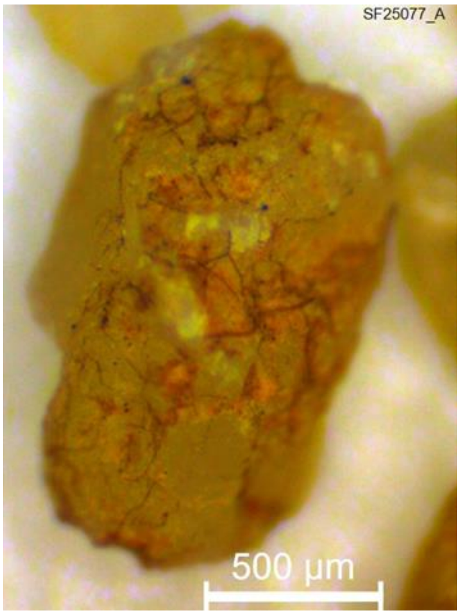
The SF25077 is divided in two samples, A and B. The sample was checked under Leica stereoscope (Fig. 52 A, B). The samples consist small and larger particles, mainly the color is white transparent with yellowish and yellow-light brown depositions. A small amount of particles had black depositions.

A

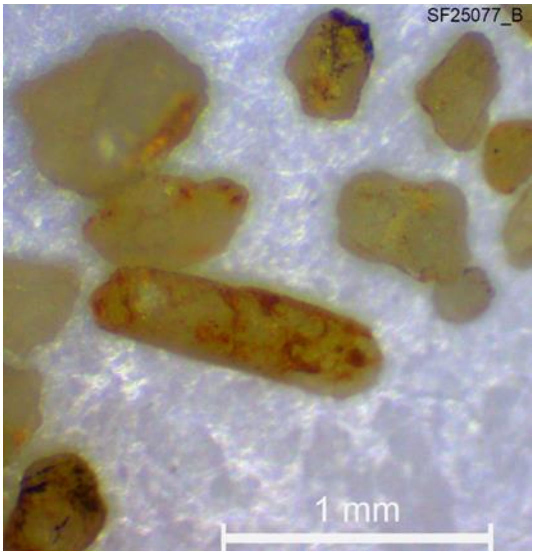


B

Fig. 52 A, B: ***General photo*** *of the samples A and B. Different sizes of particles, white transparent color with yellow/brown depositions (thin and thick) Black spotty depositions were observed on the surface of some particles. Photo taken with Leica Stereoscope.*



C

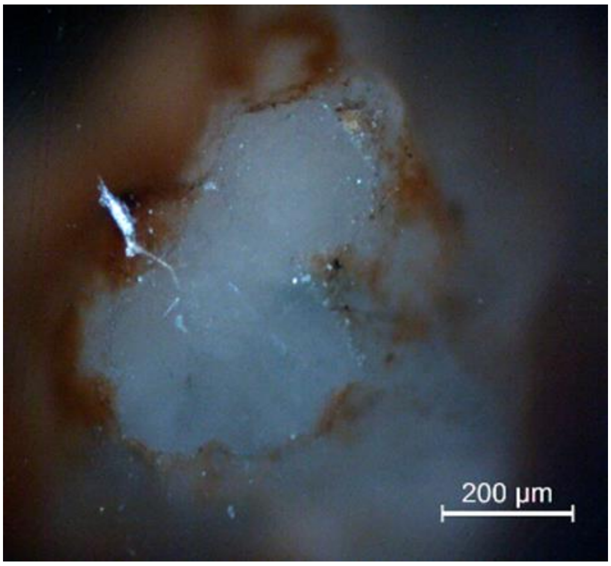


D

Fig. 52 C, D: Characteristic marble particles with yellowish-brown depositions. These particles were selected for further analysis with the SEM in ASR form.

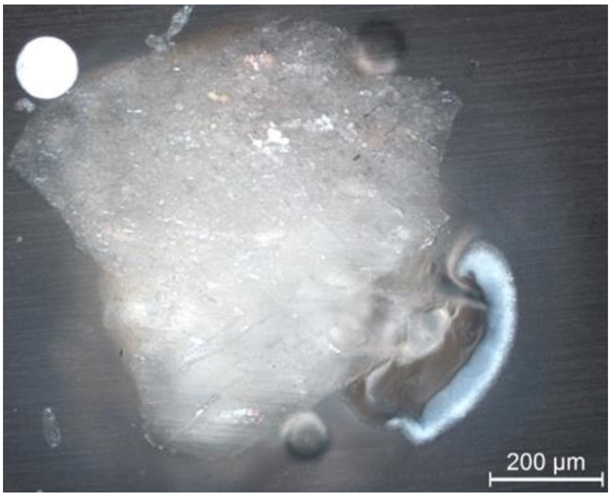
Two particles were chosen to be embedded in resin are presented below. The particle of sample A (Fig. 53 A) is a pure marble grain with orange brown deposition. The particle of sample B (Fig. 53 B) is a clean marble grain with a thin layer of yellow deposition on top of the sample.

SF25077_A



A

SF25077_B

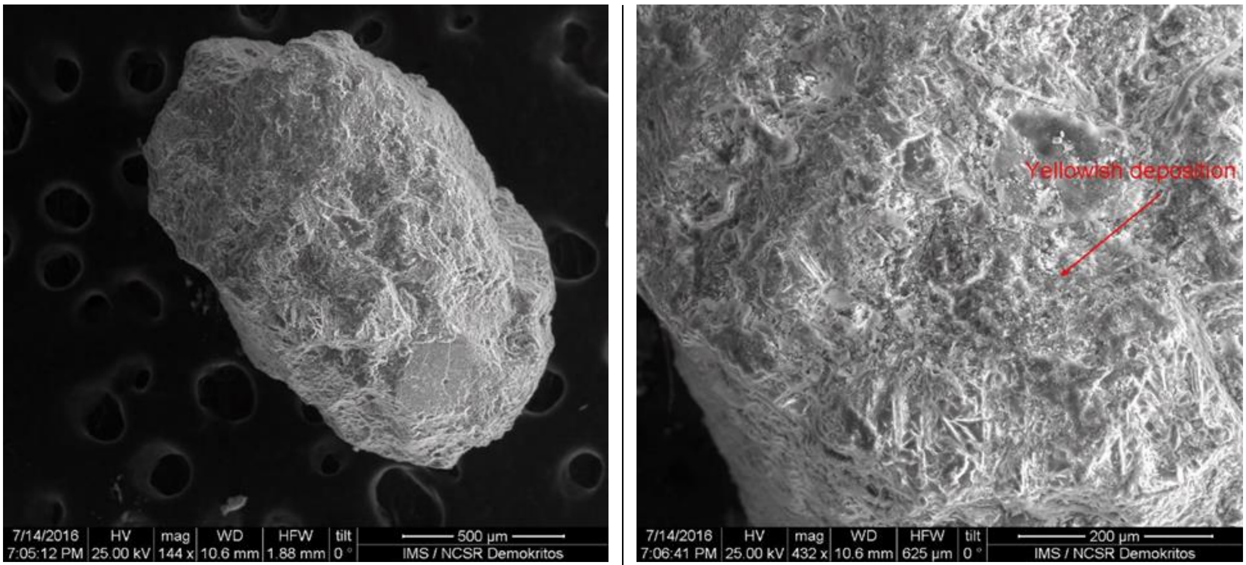


B

Fig. 53 A, B: Cross section of the polished sample under the petrographic microscope in different polarization conditions.

SEM Examination and Analysis

- Sample A



A B

Fig.54 A, B: General and detailed photo of the particle of Sample A (Fig. 52 C). The analyses were focused on the yellow brown depositions.

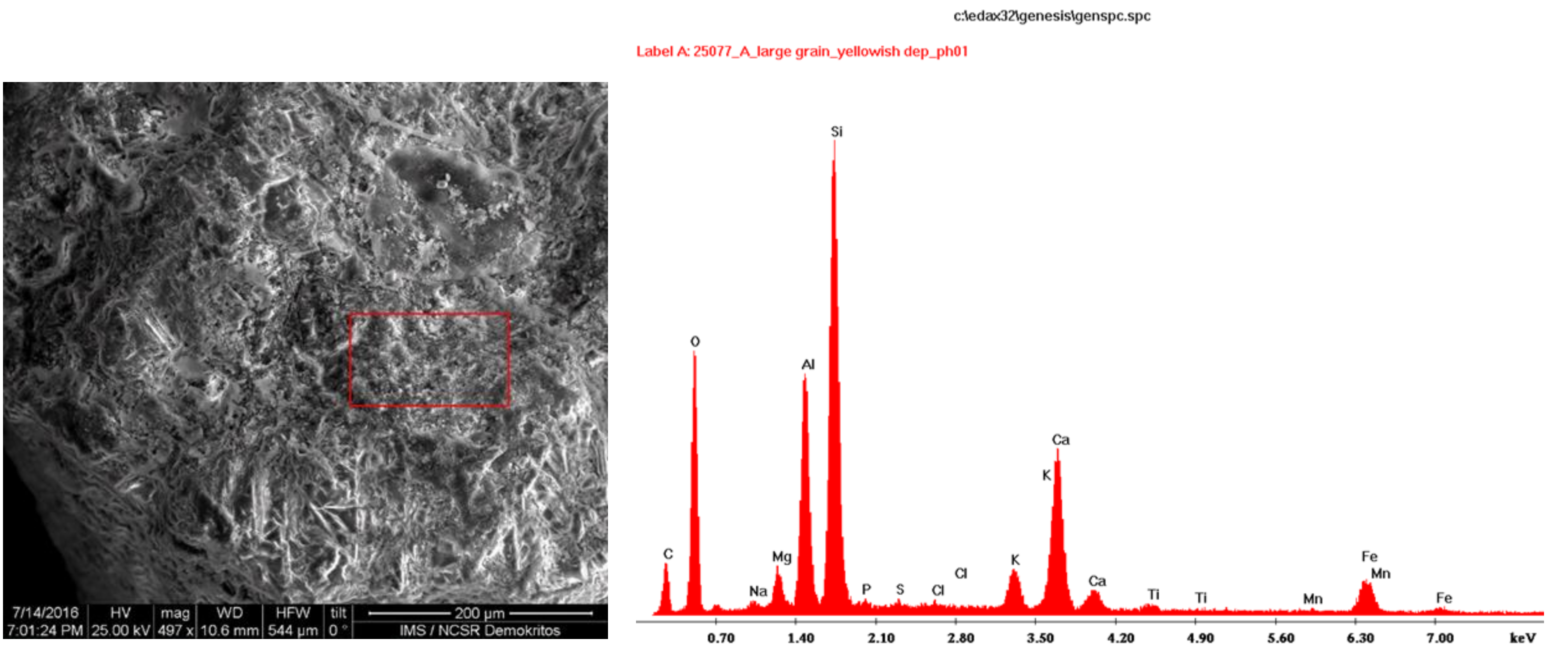
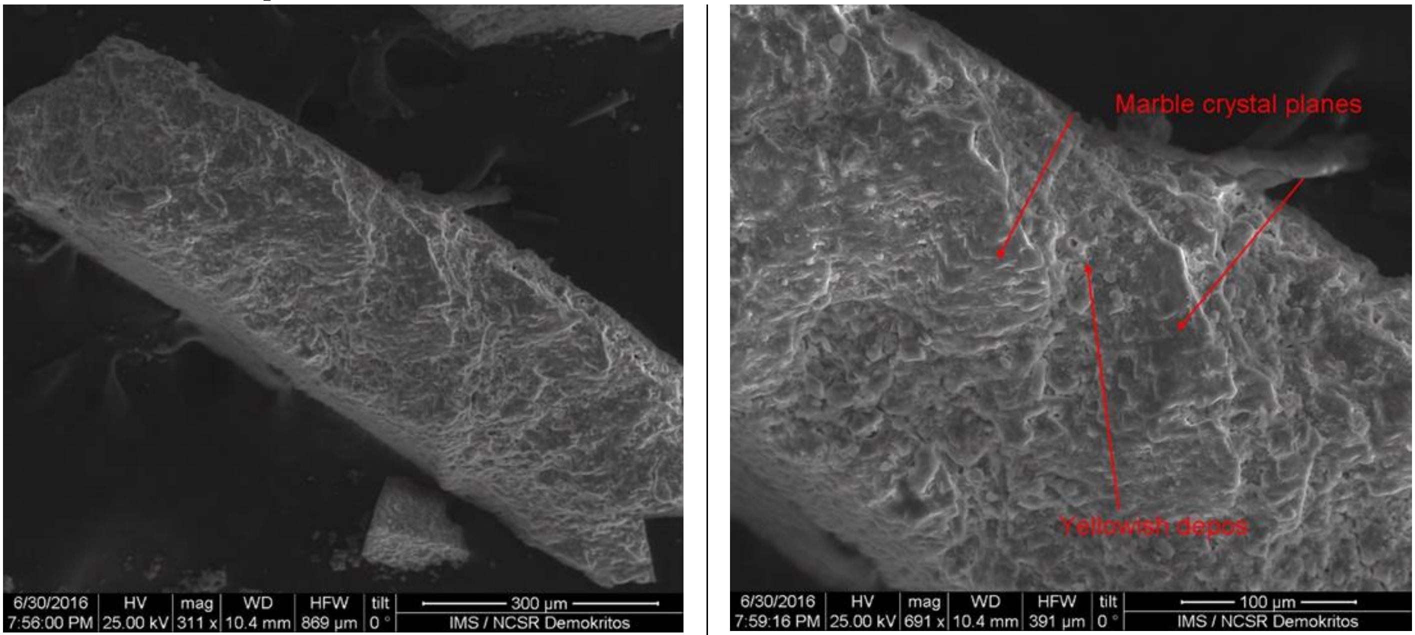


Fig. 55: 1st analysis, (table 11): Left: The SEM image of a grain taken with the LFD detector at low vacuum. Right: the analysis spectrum taken with the EDX-ray microanalysis system of the SEM at the region shown with the red square. The chemical analysis showed high concentration of Si and Al, as well as elements such as Mg, K, typical clay concentration.

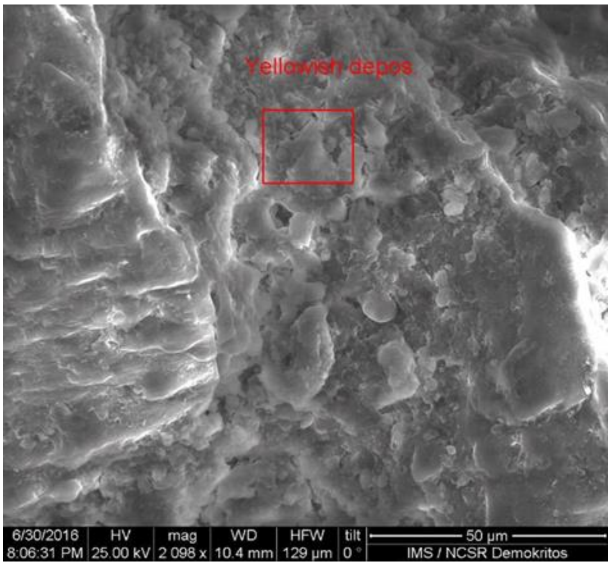
Figure 55 left shows the SEM image of one of the grains examined at low vacuum ASR. The spectrum on the right shows the analysis obtained with the ED X-ray analysis system of the SEM taken in the region indicated with the red square. As it can be seen the spectrum is dominated by a strong Si and Al. The amount of Ca comes from the marble substrate. An increased amount of Fe has been detected, about 7%.

- Sample B



A B

Fig. 56 A, B: A general and detailed image of the marble particle (Fig. 52 C). The size of the grain is about 900μm.



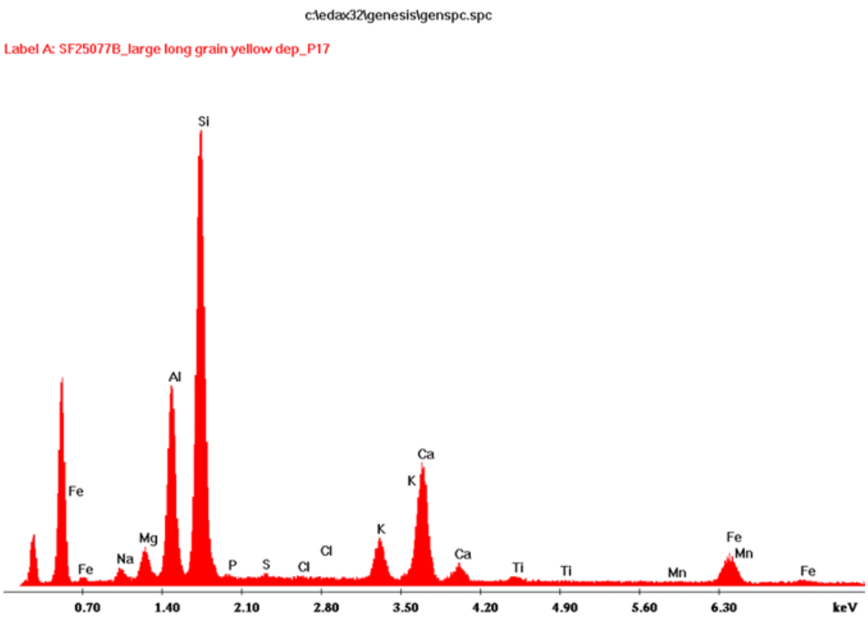
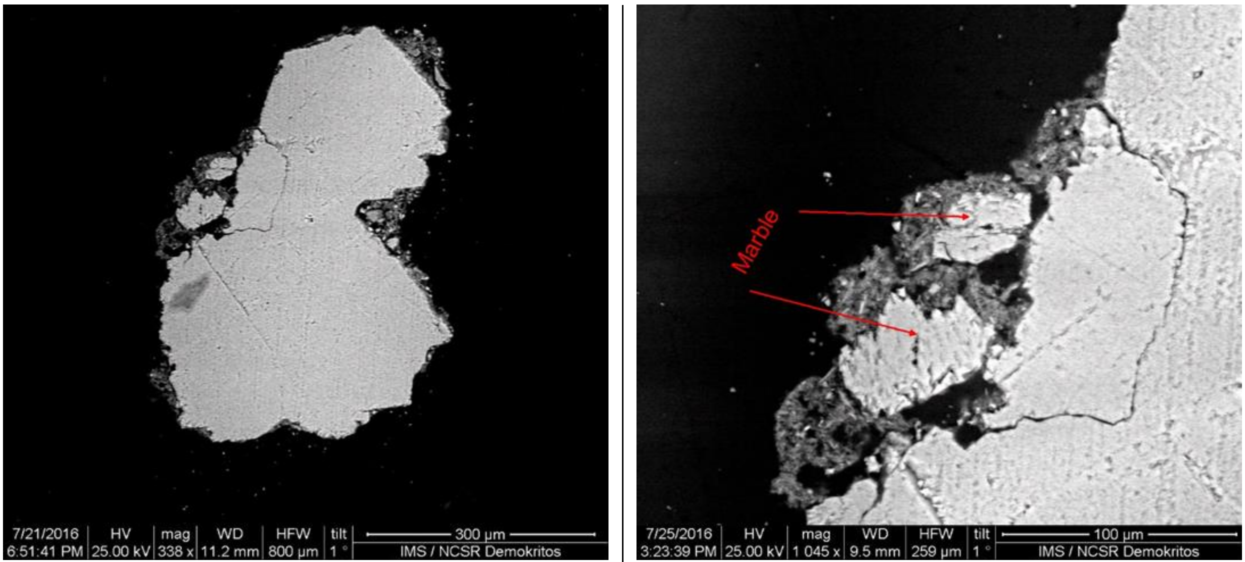


Fig. 57: 2nd analysis, (table 11): Left: SEM image of the surface with the yellowish-brown deposition. Right: Analysis spectrum at the area with a red square. The chemical analysis showed the typical clay deposition with increased Si and Al.

The Fig. 57 showed the typical clay deposition. Increased amounts of Si (54%) and Al (19%) and small amounts of other elements such as Na, Mg, K and Fe. The amounts of the elements are similar with the 1st analysis of Sample A (table 11).

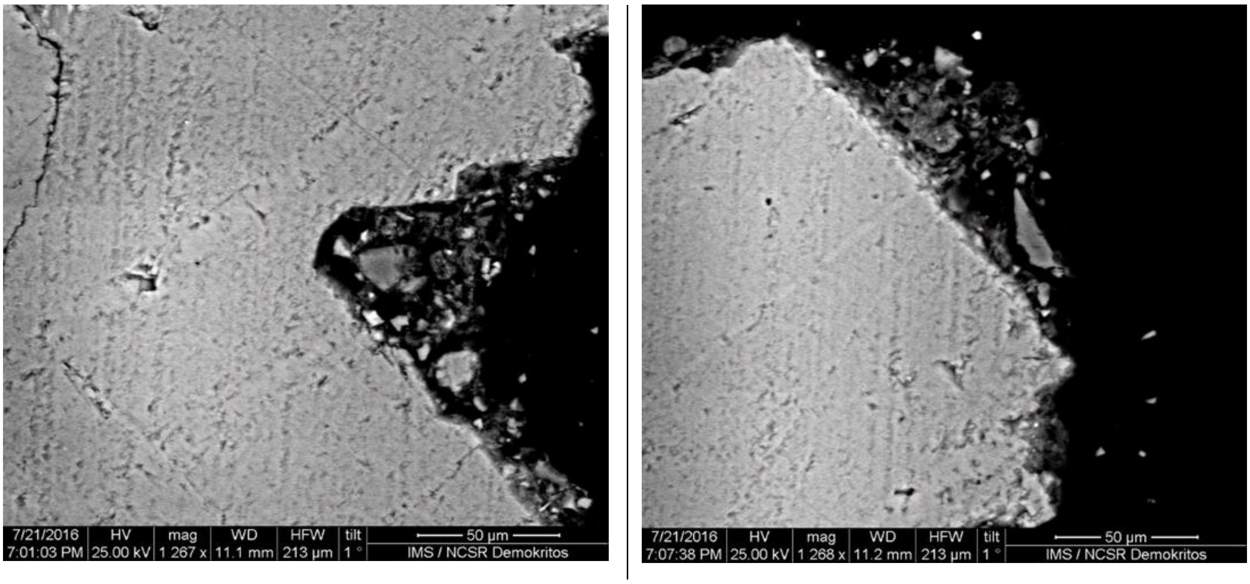
- Polished sample A

The polished cross-section was coated with carbon and examined and analyzed under the SEM in high vacuum in a backscattered electron mode (Fig. 58, 59). Figure 58 A shows the whole section consisting of a single marble grain with clay depositions around it.



A B

Fig. 58 A, B: A general and detailed image of the polished marble grain. At the Fig. 60 deposition spots can be seen on the top and at the middle left and right part of the particle. At the Fig. 60 B a detailed photo of the middle left deposition is presented.



C	D

Fig. 58 C, D: It shows the inclusions of the clay deposition. Different size minerals. Chemical analysis showed iron inclusions, marble pieces, quartz, K-feldspars etc. It is remarkable that the actual marble body has not been affected.

The Fig. 58 A the depositions of the grain can be seen. However, the marble surface has not been affected (Fig. 58 C, D). On the left part of the grain (Fig. 58 B), two marble pieces have been detached and kept together with the clay matrix. Large cracks can be seen on the marble surface, possible caused due to environmental conditions (wind, sea water, sun etc.)

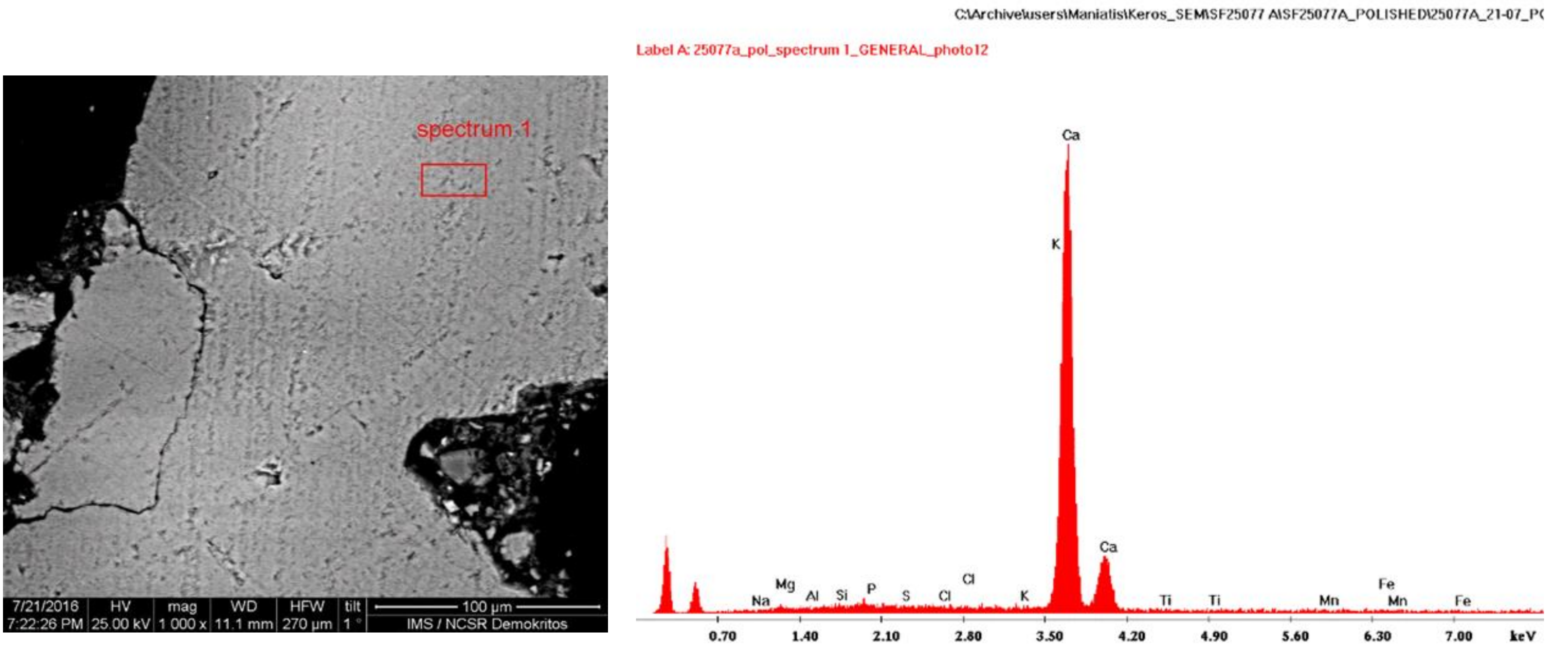


Fig. 59: 3rd analysis, (table 11): Left: a closer image of the inner part of the grain and the surface. Right: The analysis showed that the particle's consistence is pure marble, not other elements have been detected.

The analysis (no.an.3, table 11) showed clear marble grain, about 94% Ca and a small amount of Mg (1.2%), possible dolomite. No other elements were detected.

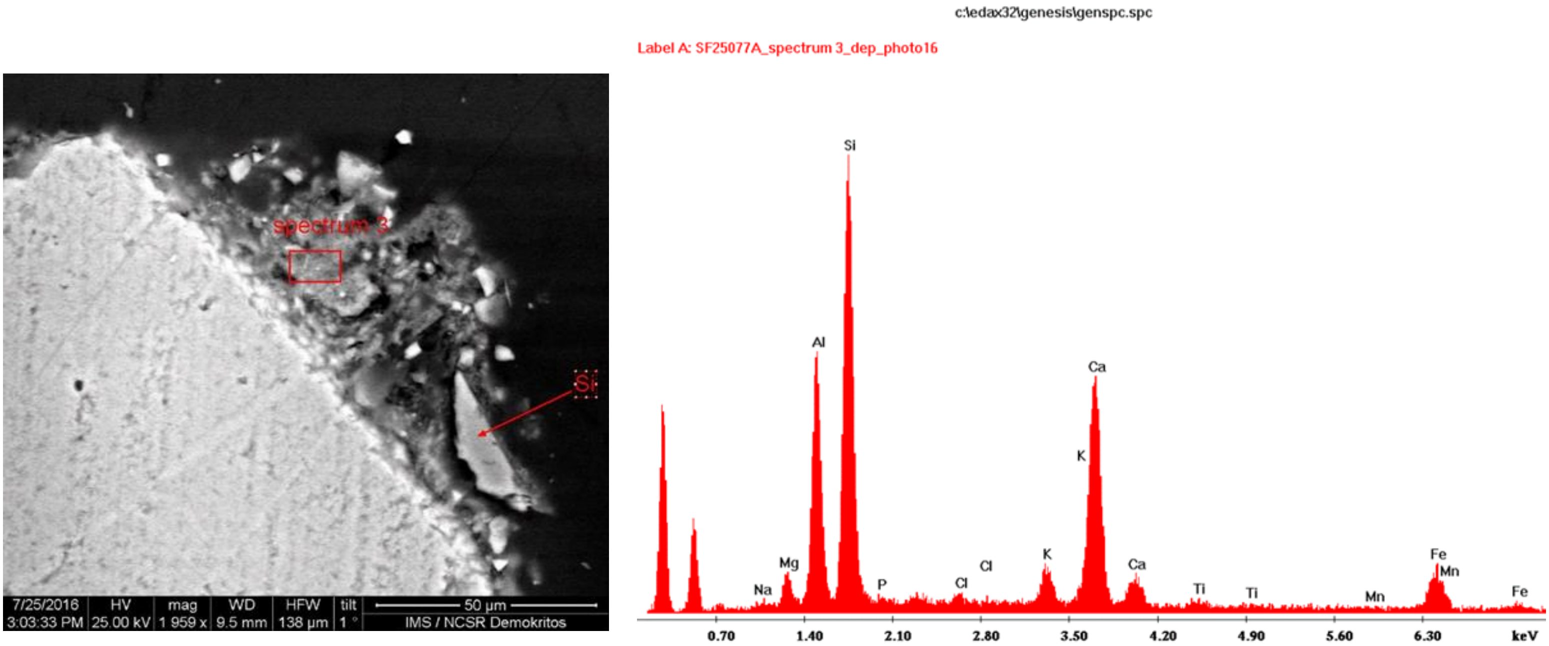


Fig. 60: 4th analysis, (table 11): Left: Closer image of the clay deposition. Right: Spectrum analysis showed the typical clay deposition, with high peak of Si and Al.

The 4th analysis (table 11) showed the typical clay deposition of Si (45%) and Al (21%), as well as increased quantities of Fe (7%). The large inclusion on the deposition is mineral quartz. The increased Ca comes from the marble substrate.

- Polished sample B

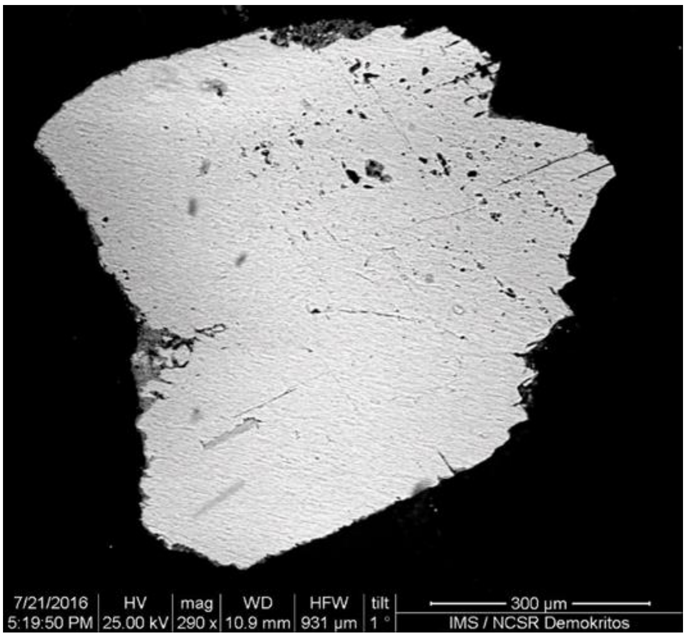


Fig. 61: A general image of the polished marble particle. Voids can be noticed mainly at the upper part of the particle. Only few spots with depositions can be observed (on the top and left middle part). The parallel inclusions can be seen on the lower part of the particle.

The polished cross-section was coated with carbon and examined and analyzed under the SEM in high vacuum in a backscattered electron mode (Fig. 62, 63). Figure

61 shows the whole section consisting of a single marble grain. It is a grain with maximum size of 600μm. The exterior surface of the grain is in a good condition. However, voids can be seen mainly on the upper part of the particle. This could be a result of corrosion from environmental conditions (acidic soil). Clay depositions are thin and only in two spots (on top and on the middle left part of the grain). Large cracks can be observed on the surface of the grain (mainly on the right part). Two long parallel grains can be observed on the bottom of the grain.

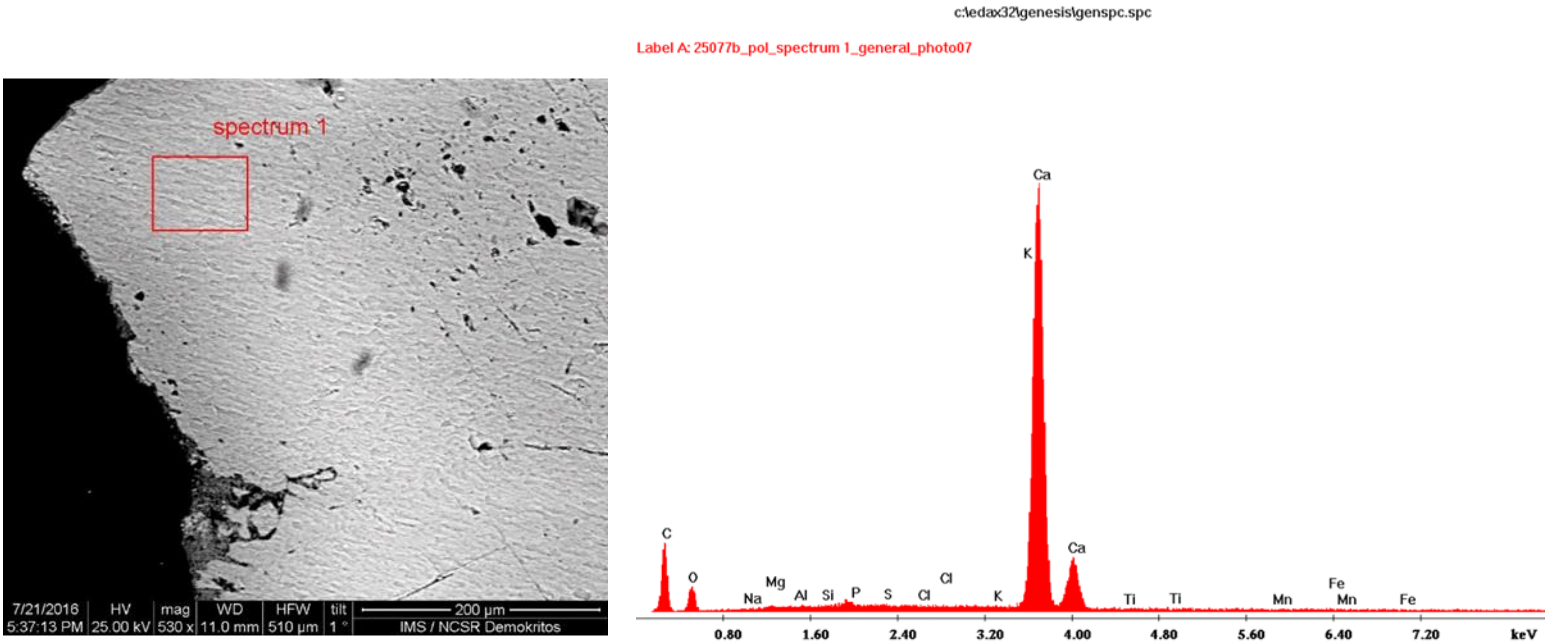


Fig. 62: 5th analysis, (table 11): Left: a closer image of the inner part of the grain and the surface. Right: The analysis showed that the particle's consistence is pure marble, not other elements have been detected.

The 5th analysis (table 11) showed the typical concentration of Ca, without other contaminants. However, the surface is in a bad condition, with wavy appearance and different size voids (Fig. 62).

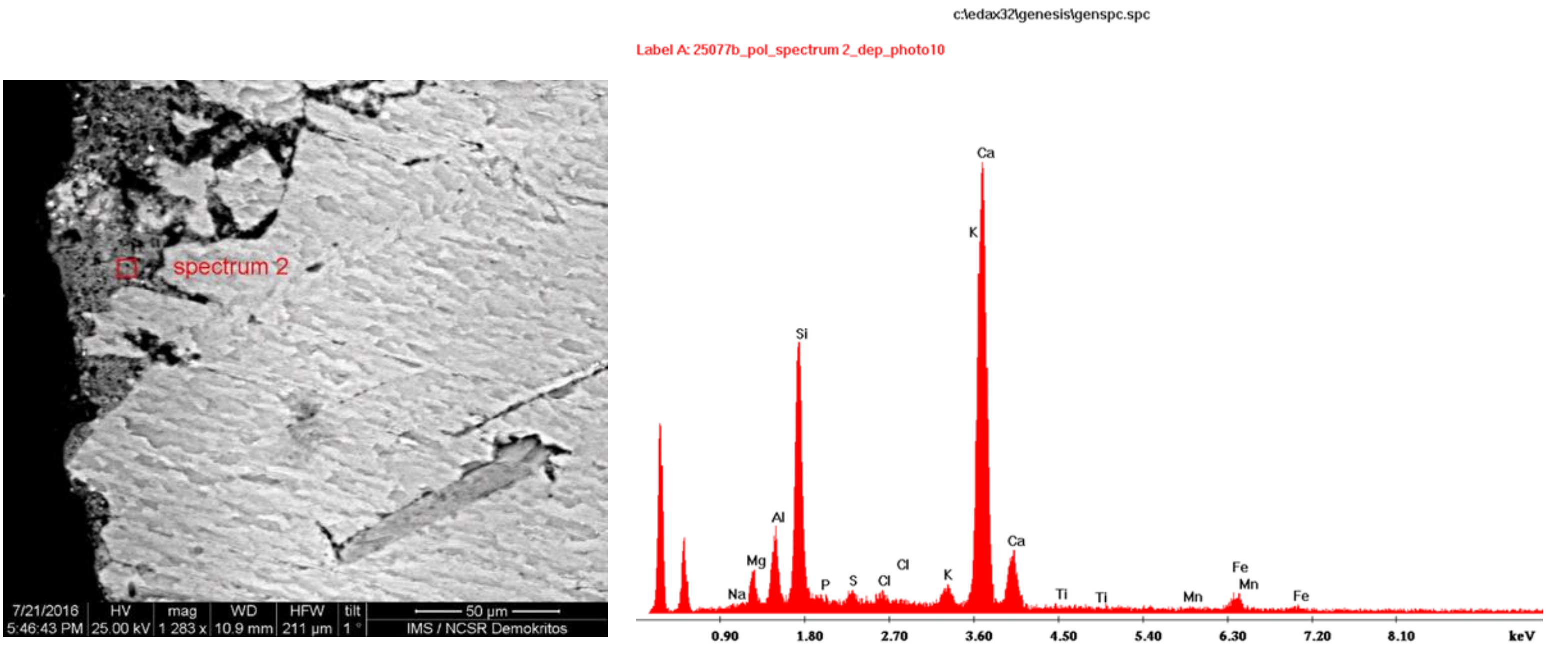
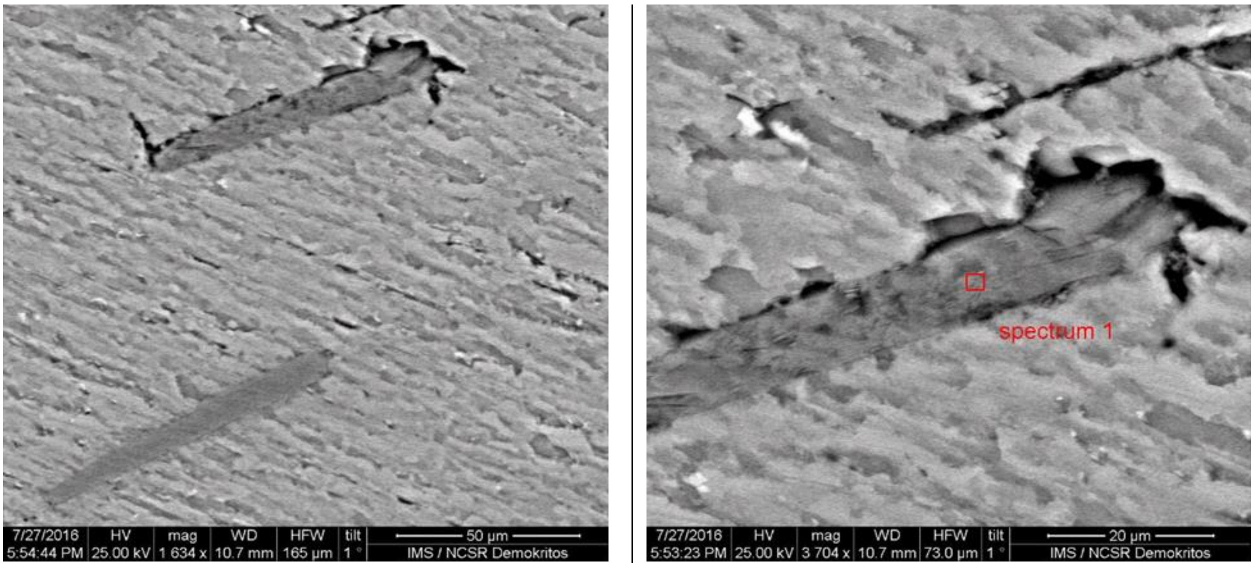


Fig. 63: 6th analysis, (table 11): Left: Closer image of the inner surface and clay deposition. Right: the chemical analysis showed the typical chemical composition of the clay deposition with increased Si and Al.

The deposition analysis shows (no. an. 6, table 11) the typical chemical composition with Si (31%) and Al (8%), and increased oxides of iron, potassium, chlorine. The amount of Ca comes from the marble substrate. The Mg (4,9%) is resulted by soil inclusions.



A B

Fig. 64 A, B: 7th analysis, (table 11): The Fig. 66 A is showing in detail the parallel mineral that are located at the bottom of the sample in the marble matrix. At the Fig. 64 B the spot of the analysis is marked with a red square. It showed the mineral possibly is Kaliophilite / Kalsilite.

These two grains should probably have been included during the transformation of the sedimentary rock to metamorphic rock (marble). Possibly these minerals belong to the group of K-Feldspars, a variety of orthoclase. These feldspars are mostly colorless, white or grey. However, due to the high concentration of Al oxides could not fit with the formula of orthoclase[20], sanidine or microcline due to the low concentration of Al oxides. These inclusion on the sample B could be considered as Kaliophilite / Kalsilite: K_2O 29.78 %, Al_2O_3 32.23 %, SiO_2 37.99 %. The amount of K oxides are more than the analysis of the sample B, although, their ratios approaching closer the analysis of sample B.

Table 11: Chemical Analysis in oxides form (100%wt) - SF 25077

No An.	Sample	SPECTUM	Na2O	MgO	Al2O3	SiO2	P2O5	SO3	Cl2O	K2O	CaO	TiO2	MnO	Fe2O3	total %
		ASR													
1	**A**	deposition	0.68	3.42	20.58	49.55	0.68	0.63	0,28	2.83	13.35	0.88	0.37	6.77	100
2	**B**	deposition	1.48	3.14	19.11	53.84	0.21	0.48	0,14	3.33	11.06	0.84	0.14	6.25	100
		Polished													
3	**A**	marble	n.d.	1.23	0.44	1.04	0.64	0.37	0,16	0.40	93.64	0.74	0.53	0.83	100
4	**A**	deposition	0.58	3.02	20.87	45.27	0.82	n.d.	0.62	2.52	18.06	0.87	0.30	7.06	100
5	**B**	marble	0.26	1.08	0.53	0.72	1.07	0,67	0.25	0.42	93.40	0.42	0.37	0.79	100
6	**B**	deposition	0.39	4.29	8.20	31.60	0.73	1,99	0.93	1.73	45.36	0.51	0.38	3.88	100
7	**B**	inclusion	0.74	1.49	33.60	47.65	0.52	0,38	0.19	10.58	3.41	0.52	0.20	0.74	100

n.d. = not detected

Summary

The Special Find 25077 is separated in two samples. Both of the samples are including medium and small marble grains with yellowish brown depositions. The analyses of the ASR particles showed the typical clay depositions (Si and Al). The polished particle of sample A, is not very corroded. On the polished particle of the sample B, the surface of the crystal is corroded and there are few voids, in addition there are two rectangular parallel minerals.

This particular figurine as it was mentioned on Chapter 6 has been categorized by macroscopic examination as a heavily weathered figurine (WD = 5, Maniatis and Tambakopoulos 2015). The detailed examination and analysis of the grains in the sample obtained from this figurine confirms the extensive weathering and determined that the damage extends to the individual grains. It also identified the origin of the depositions on the surface and the fact that the marble was first weathered and then buried.

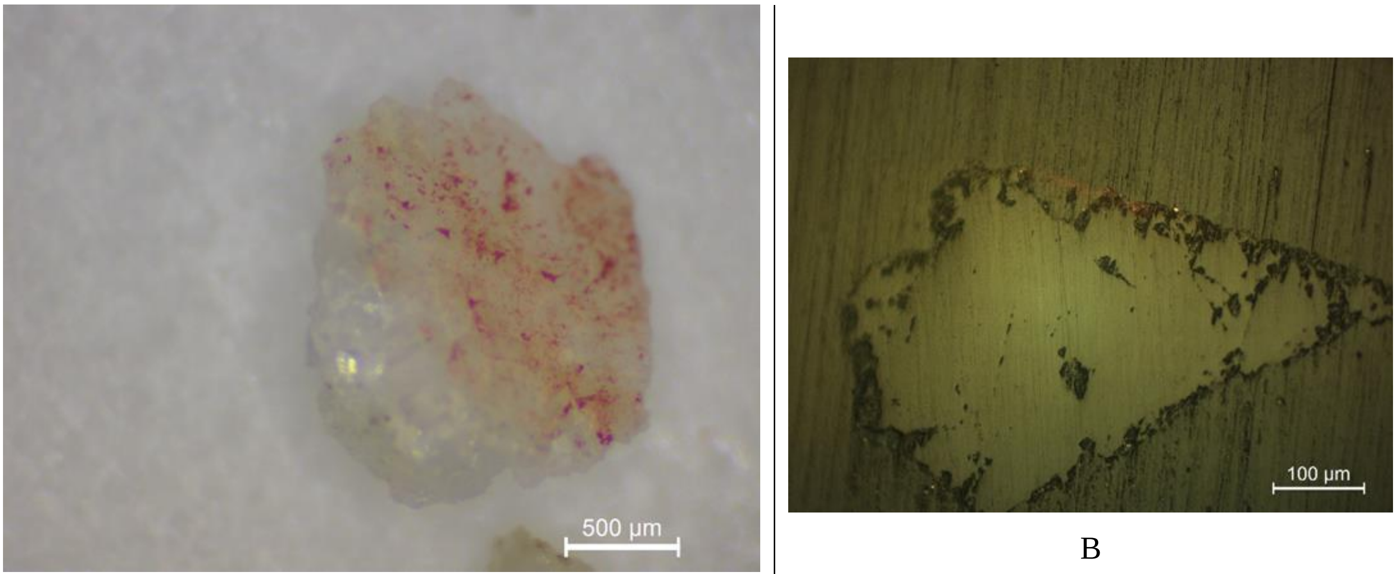
[20] Orthoclase chemical formula: 16.92 % K_2O, 18.32 % Al_2O_3, 64.76 % SiO_2

Vessel – SF 30006

The Special Find 30006 was identified as marble basin, with red pigment on the interior surface (see Fig. 9).

Optical Microscope Analysis

The sample was checked under Leica stereoscope (Fig. 65 A). The color of the marble particles is white-transparent. Red pigment remains in the form of spots can be observed on each particles surface. One of the few marble particles available was chosen to be examined and analyzed in ASR and polished cross-section embedded in resin in order to assess the thickness and penetration of the pigment layer (Fig. 65 B).



A B

Fig. 65 A, B: Left: The color of the main particle is white transparent, red spot pigment can be observed. Right: The Fig. 65 B is the image of the cross section of the polished particle under the petrographic microscope.

SEM Analysis

The chemical composition of the pigment was analyzed and determined as **Cinnabar** (HgS). Three analyses are discussed below:

- **Sample ASR form**

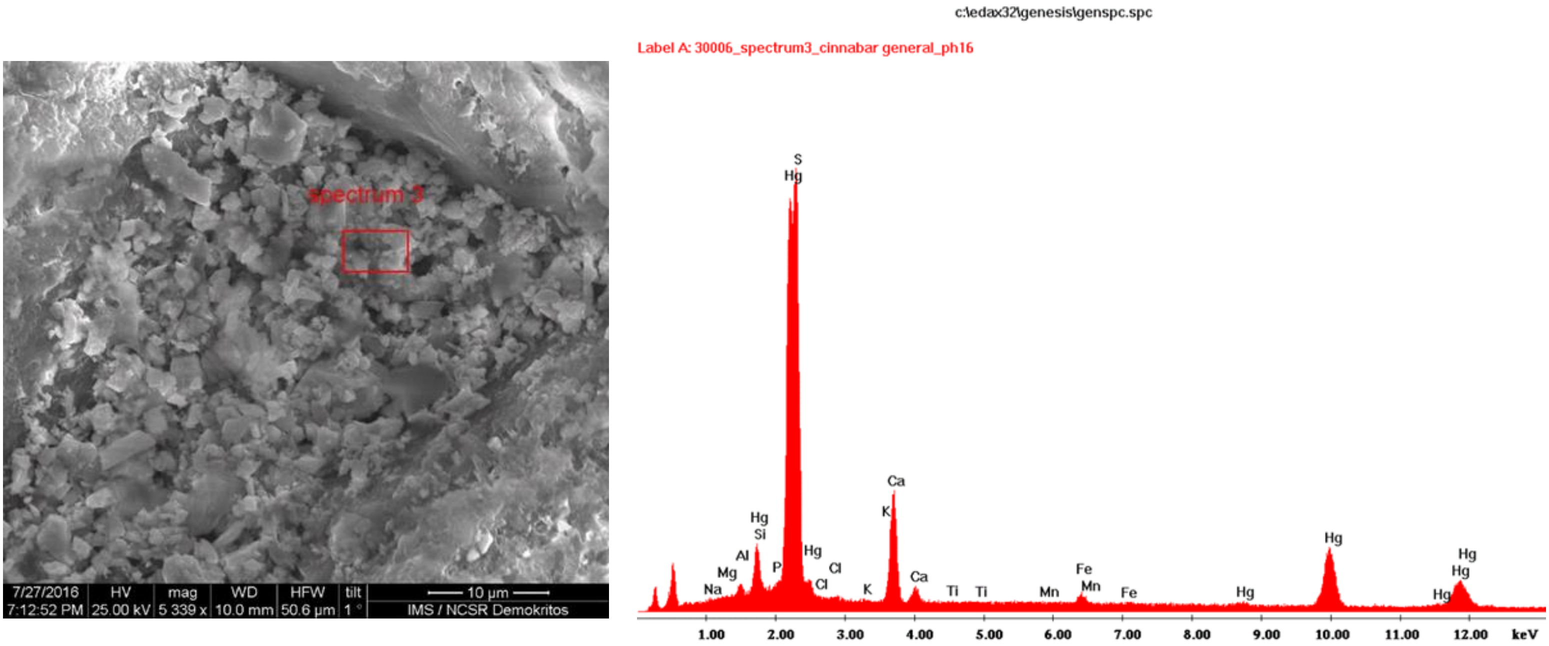


Fig. 66: 1st analysis, (table 12): Left: A closer image of the red depositon on a cut. Right: The analysis showed that the red pigment is Cinnabar, with high concentration of S and Hg.

The pigment deposition is concentrated on a cut of the marble surface (Fig. 66). The 1st analysis (table 12) showed 10% of Ca (coming from the marble substrate). In addition, traces of Si, Al, Mg, and Fe oxides have been detected due to the burial of the sample in the soil.

- **Polished sample**

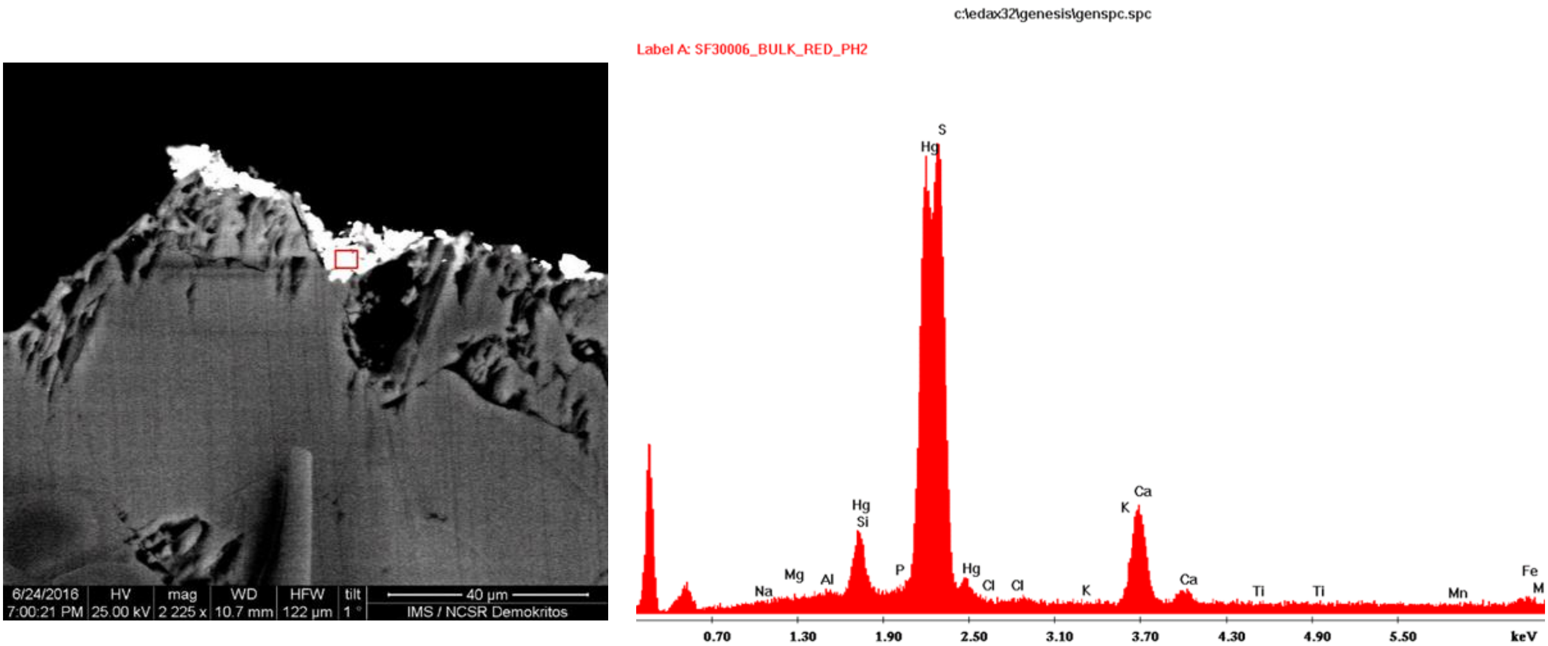


Fig. 67:2nd analysis, (table 12): Left: a closer image of the pigment deposition. Right: the analysis showed the cinnabar chemical composition. No other elements were detected. The deposition is thin, 3-4μm.

The cross section showed a thin layer of cinnabar (3 to 6 μm) (Fig. 67), the thickness of the pigment layer at the surface is about 10μm, while the marble surface under the pigment shows a certain degree of weathering and absorption of cinnabar to a depth of 35μm to 55 μm.

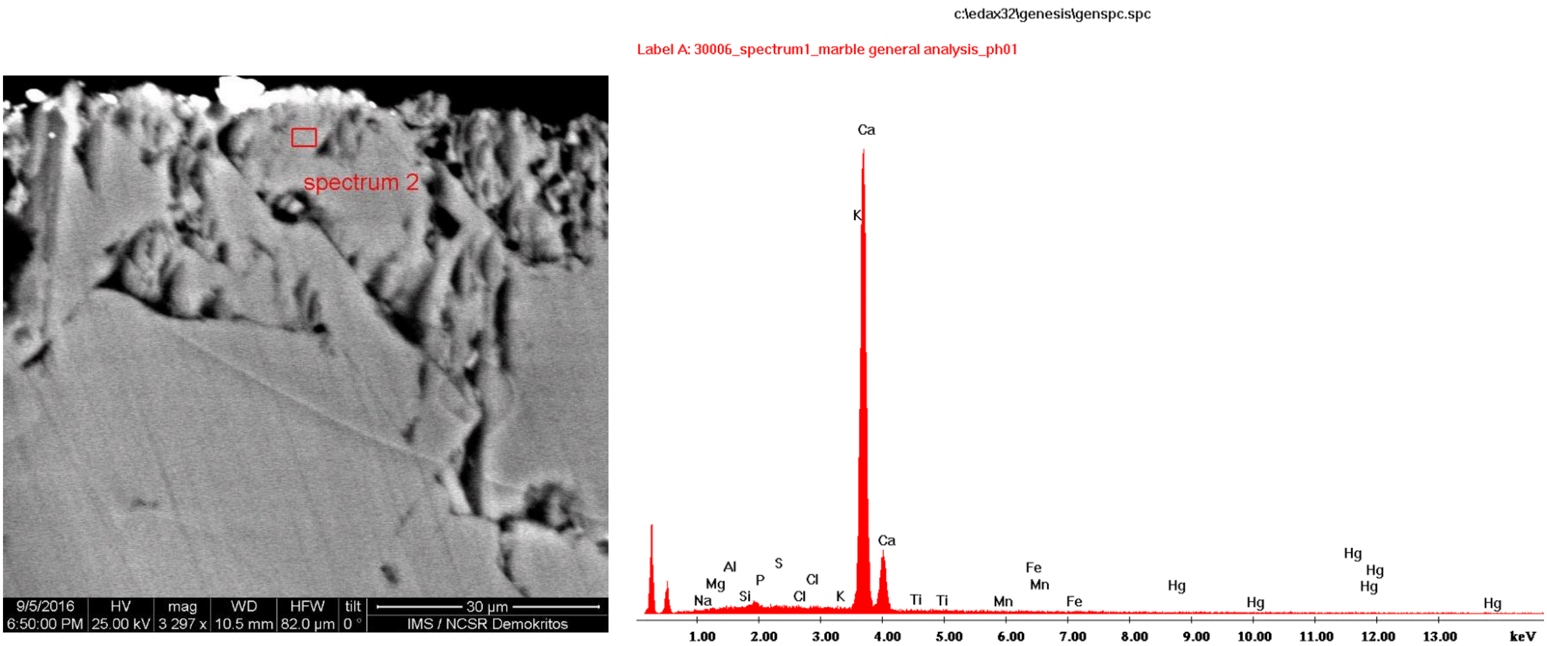


Fig. 68:3rd analysis (table 11): Left: Closer image of the area below the cinnabar layer. Right: The analysis showed pure Ca consistence, with minimum quantities of Hg and S.

The 3rd analysis performed at the surface under the cinnabar layer (Table 11), showed a very high concentration of Ca with a very a very small amount of Mg (pure calcitic marble. The presence of small amounts of Hg and S is the result of absorpion from the overlaying cinabar pigment. The marble is in a better preservation state compared to the figurines because of the different conditions of burial (Special Deposit North).

Table 12: Chemical Analysis in oxides form - SF 30006

No An.	SPECTRUM	Na2O	MgO	Al2O3	SiO2	P2O5	SO3	Cl2O	K2O	CaO	TiO2	MnO	Fe2O3	HgO2	total%
	ASR														
1	pigment	0.42	0.51	1.25	3.95	1.17	18.14	0.23	0.28	10.23	0.38	0.45	1.92	61.07	100
	Polished														
2	pigment	0.29	0.29	0.50	5.47	0.90	19.11	n.d.	n.d.	9.55	0.28	0.30	1.58	61.72	100
3	marble	0.16	0.76	0.39	0.36	0.85	0.71	0.30	0.27	93.62	0.21	0.40	0.58	1.40	100

Summary

The SF 30006 is a marble basin with red pigments on the interior surface of the vessel. The SEM analysis showed that the red pigment is cinnabar (HgS). The layer of the pigment is thin (3-6μm). Larger amounts of cinnabar are deposited on the cut on the basin surface. The cross section showed a cinnabar absorption, however that analysis showed minimum amounts of Hg and S (3^{rd} an. table 12).

8. Conclusion

Optical and scanning electron microscope examination coupled with ED analysis was performed on five marble figurines from Keros excavation on the Kavos Special Deposit South in 2006-2008 in order to assess and document the different types and degrees of weathering their preservation state. In addition, a marble vessel (open basin) from the Kavos Special Deposit North, with traces of red pigment in its interior was examined and analysed and the red pigment identified. The samples were examined and analyzed in two states; as received (ASR) and in polished cross-sections.

The detailed examination and analysis of the marble crystal grains obtained from each sample in each sample obtained showed that a thin or thicker clay depositions have been deposited on the surface of each sample, which was identified as an iron-rich fine clay. The polished sample showed clearly an extensive damage by weathering extending to the individual grains which were detached due to this weathering from the surface of the figurine. The grains in many instances exhibited severe cracking caused most probably by rapid alternations of low and high temperature and salt crystallization. In other cases soil with calcite crystallization, probably from dilution of the marble and redisposition, had formed crusts on the surface in a sequence of layers indicating more than one weathering episodes. These facts indicate exposure of the figurines first to open air conditions (salt spray from the sea, exposure to sun, cold weather, etc) and later burial in the soil. These results confirmed previous macroscopic observation of the differential weathering of the Keros figurines (Maniatis and Tambakopoulos 2015) and provided more in depth documentation of the severity of conditions and alternating exposure of the figurines prior to their final burial at Kavos Special Deposit South.

Finally, the red pigment detected on the interior of the marble basin was identified as Cinnabar (HgS) a rare material/pigment however well-known during the Early Bronze Age period. The condition of preservation of this vessel and the pigment was much better that the figurines because the conditions in SDN are very different.

As future research, more figurines from Keros Special Deposit South as well as from Special Deposit North, in combination with marble objects from Dhaskalio would need to be examined and analysed in order to see the effect of difference geological environment and soil and conditions of environmental exposure. Other techniques such as FTIR could be applied to detect mineralogical changes and give more details about the organic depositions on the grain surfaces.

9. Appendix

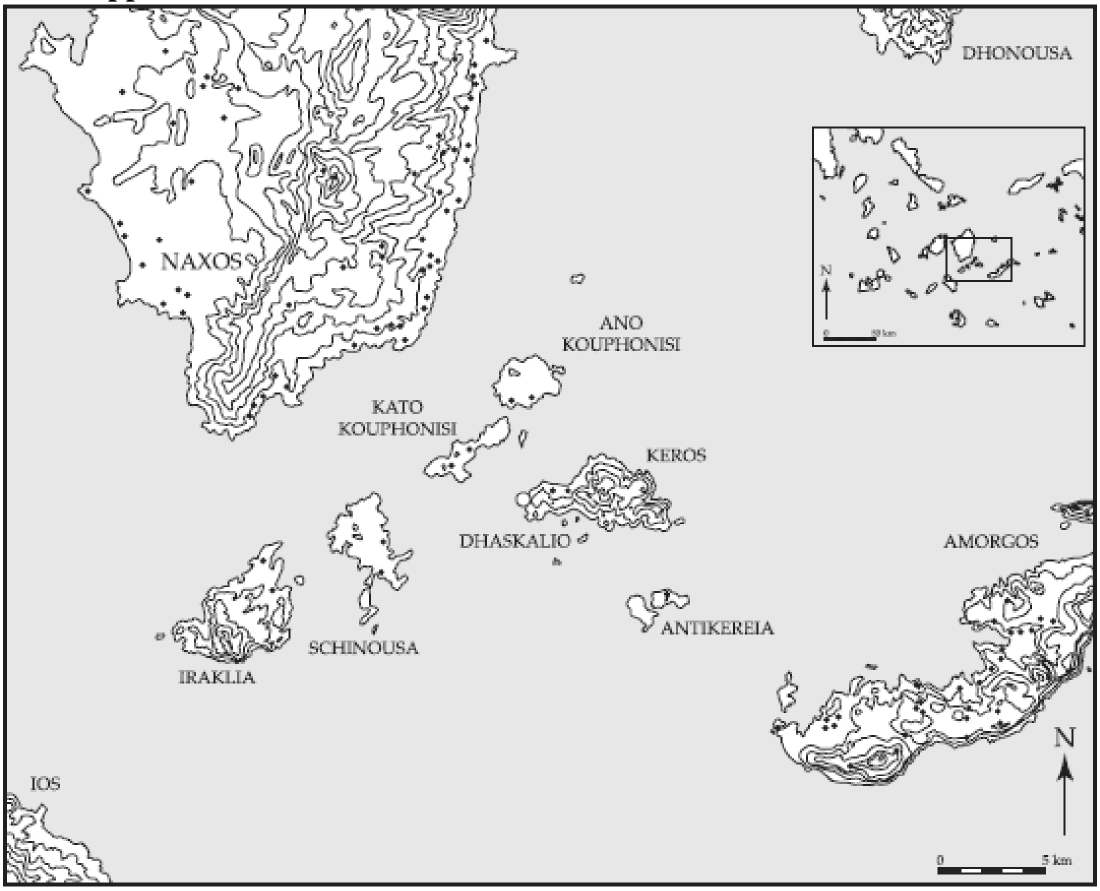


Fig. 69: Map of Small Cyclades (in the middle is Keros) (Renfrew et al. 2015)

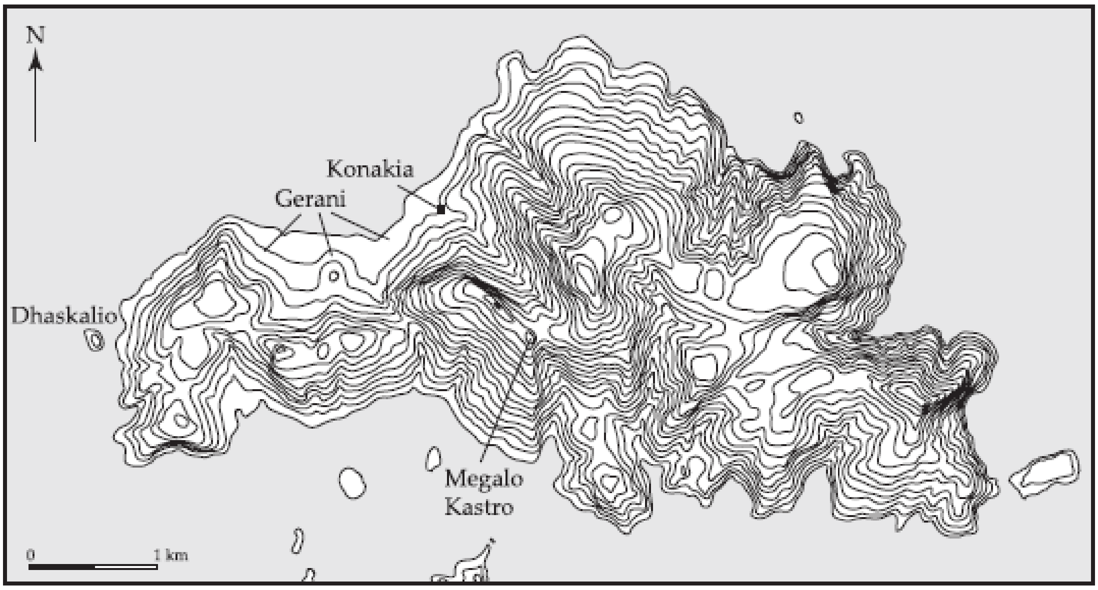


Fig. 70: Keros Island. On the left part is Dhaskalio and opposite is Kavos (Renfrew et al. 2015).

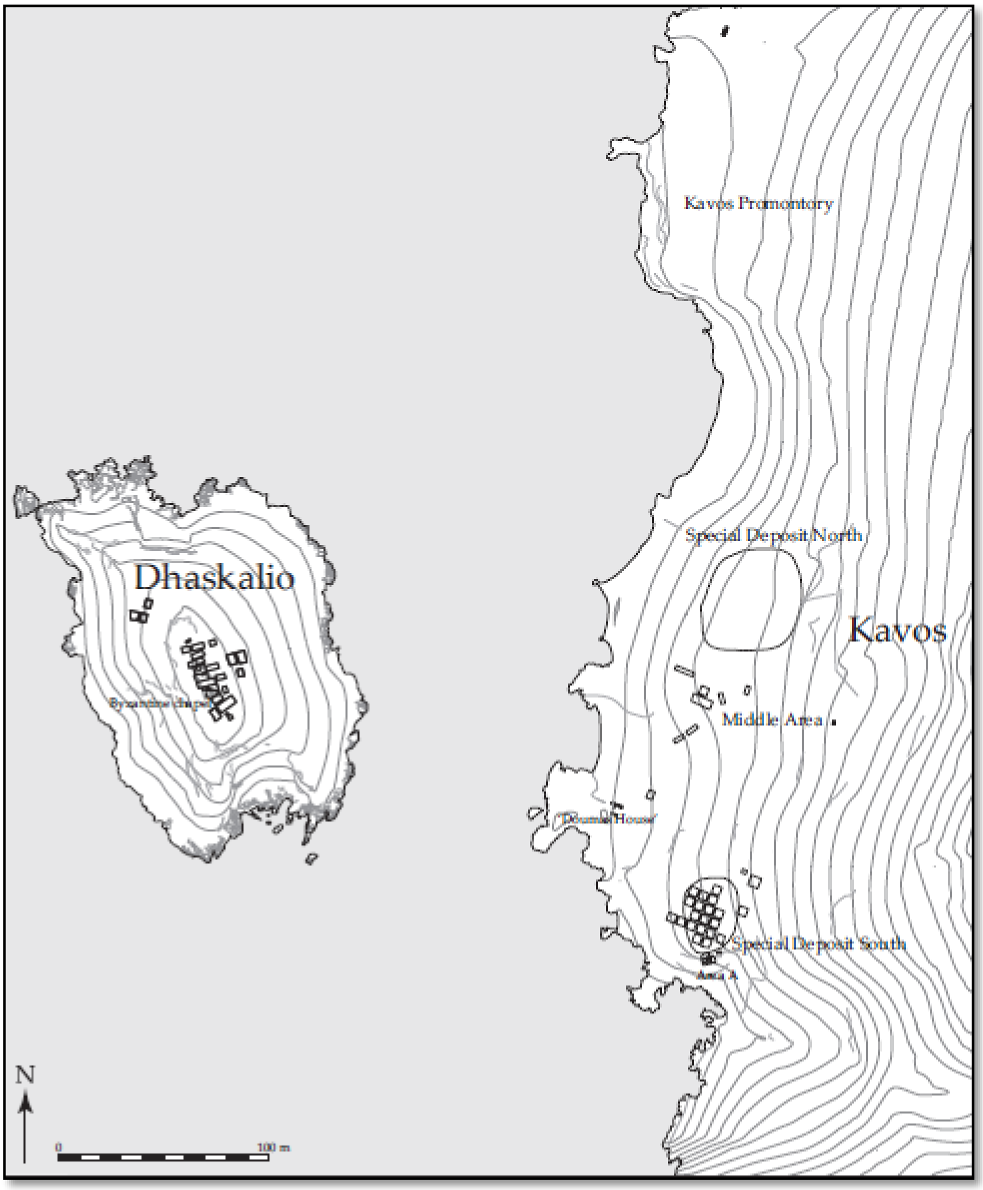


Fig. 71: Map of Dhaskalio and Kavos, the Special Deposits are marked. The excavation of Special Deposit South was conducted during the years 2006-2008, while the Special Deposit North was looted in the past. (Renfrew et al. 2015).

Fig. 72: The Special Deposit South (Excavation period 2008). (Renfrew et al. 2015).

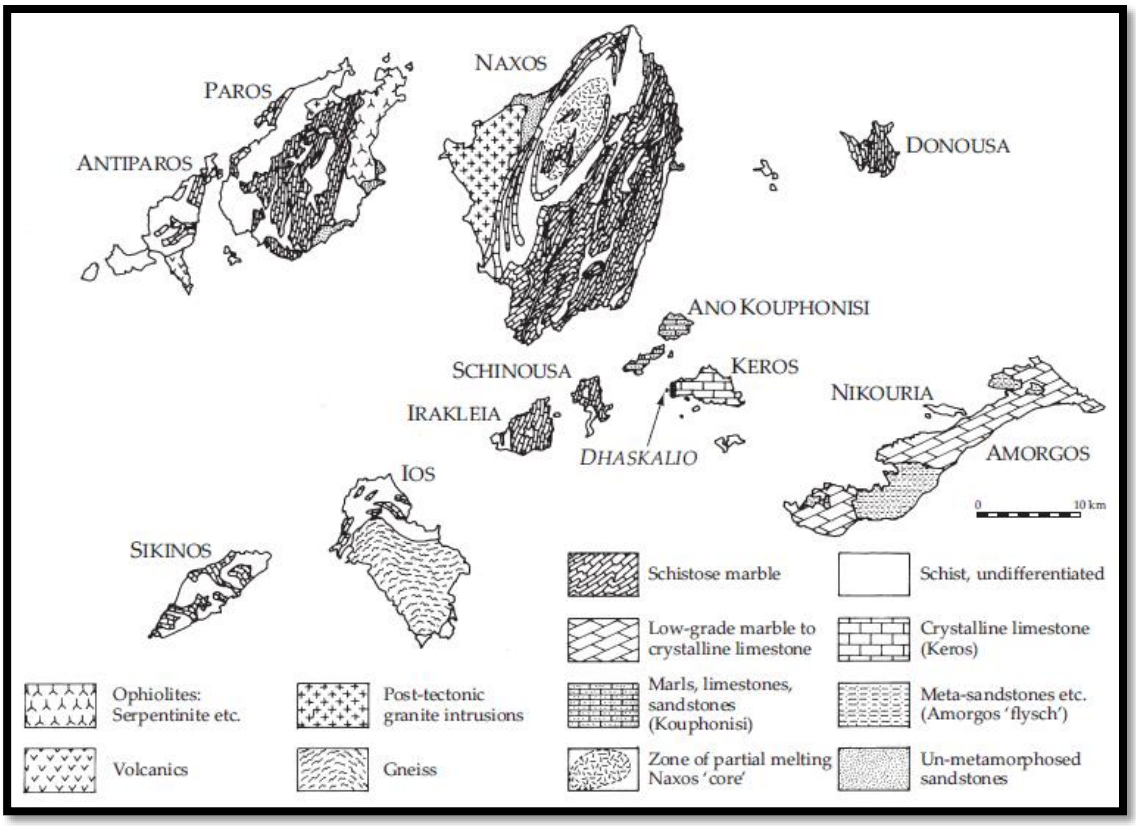


Fig. 75: Geological map of Cyclades

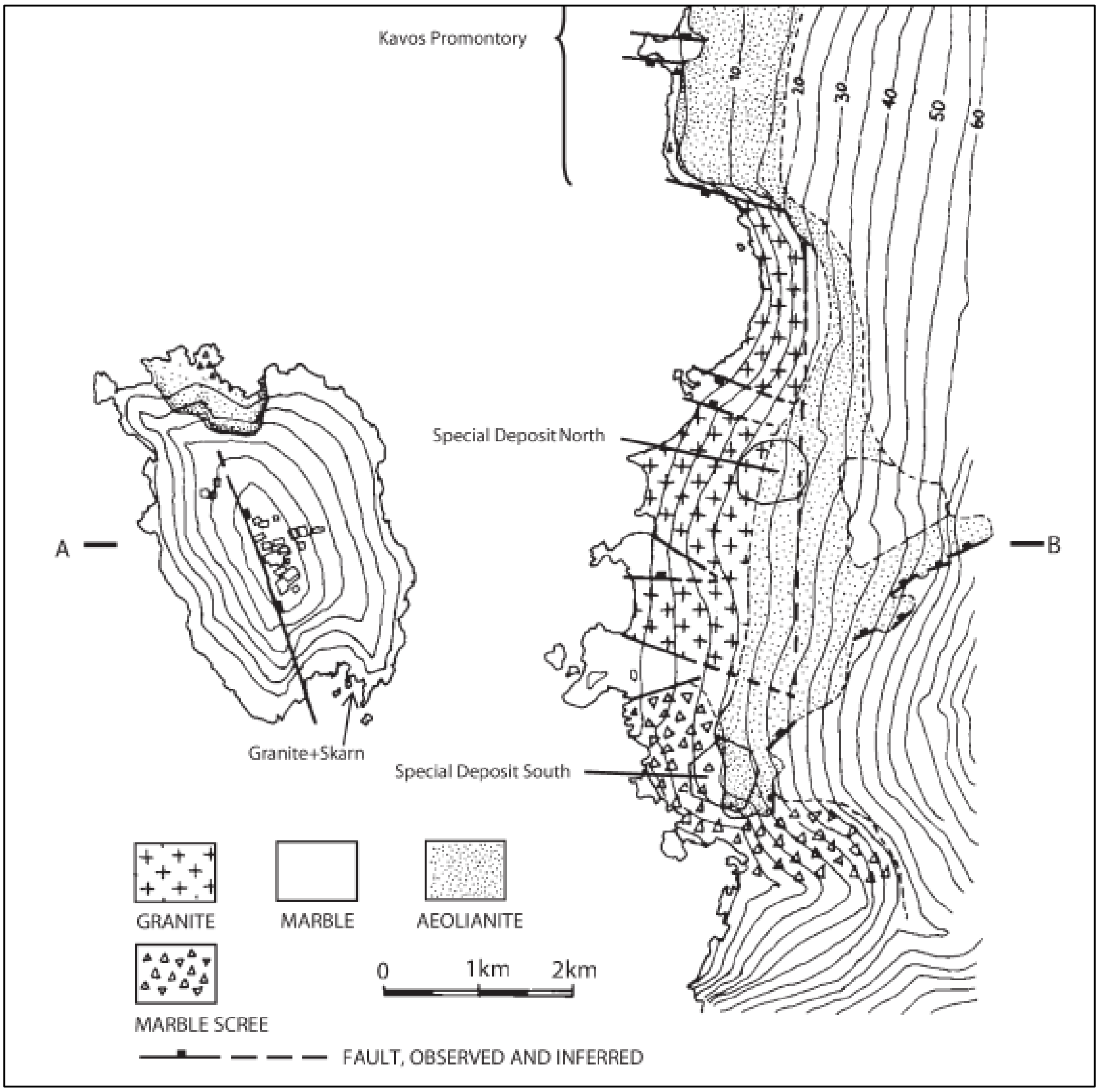


Fig. 74. Geological Map of the area of Dhaskalio and Kavos (Renfrew et al. 2015).

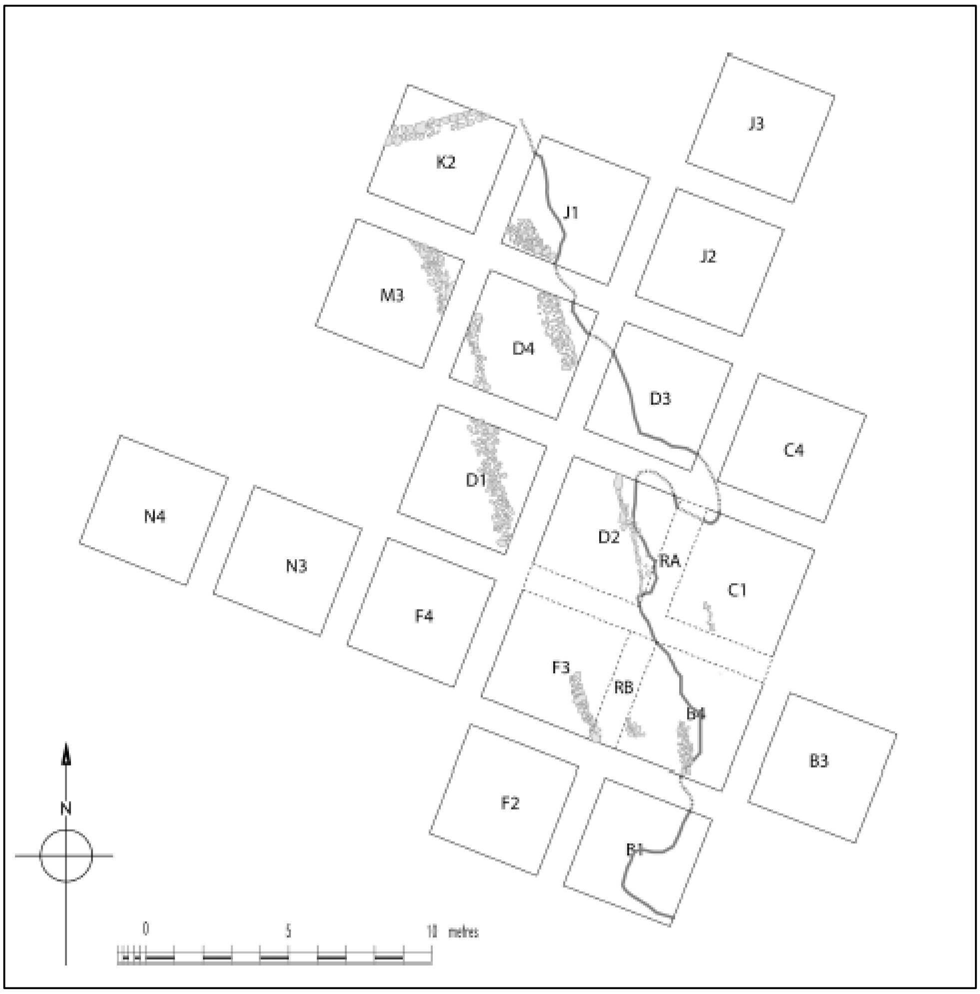


Fig. 75: The excavation plan of the area of Special Deposit South (Renfrew et al. 2015).

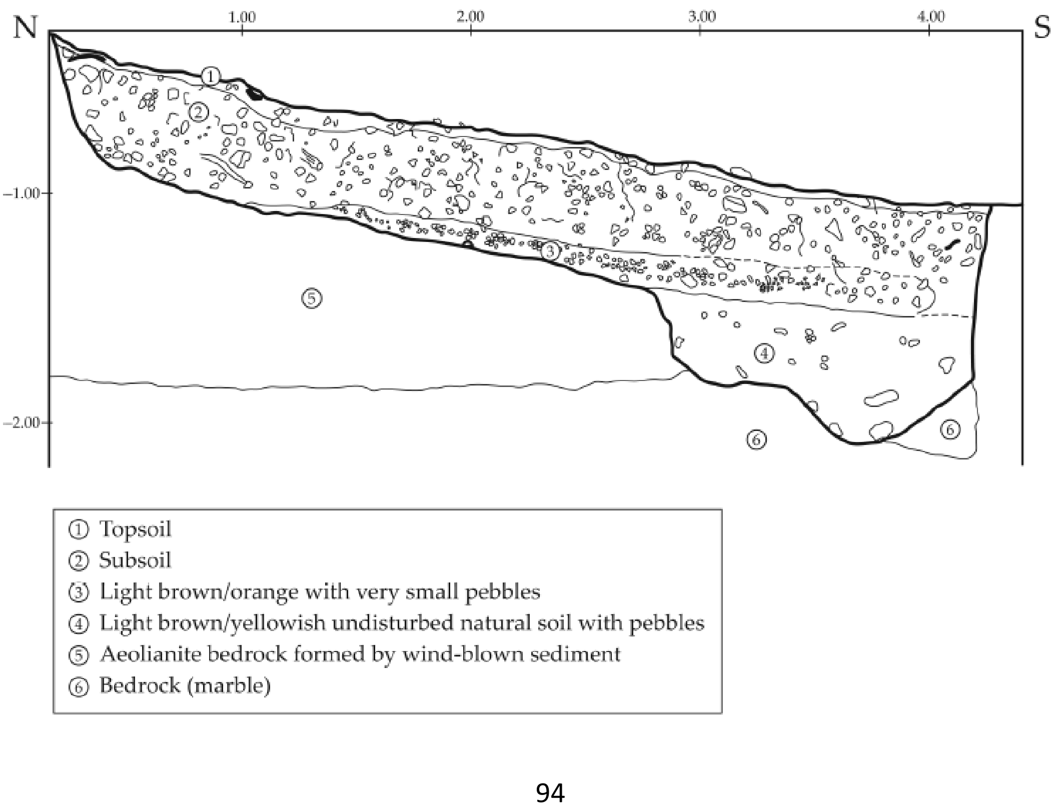


Fig. 76: Section of east baulk of trench D2 (Renfrew et al. 2015).

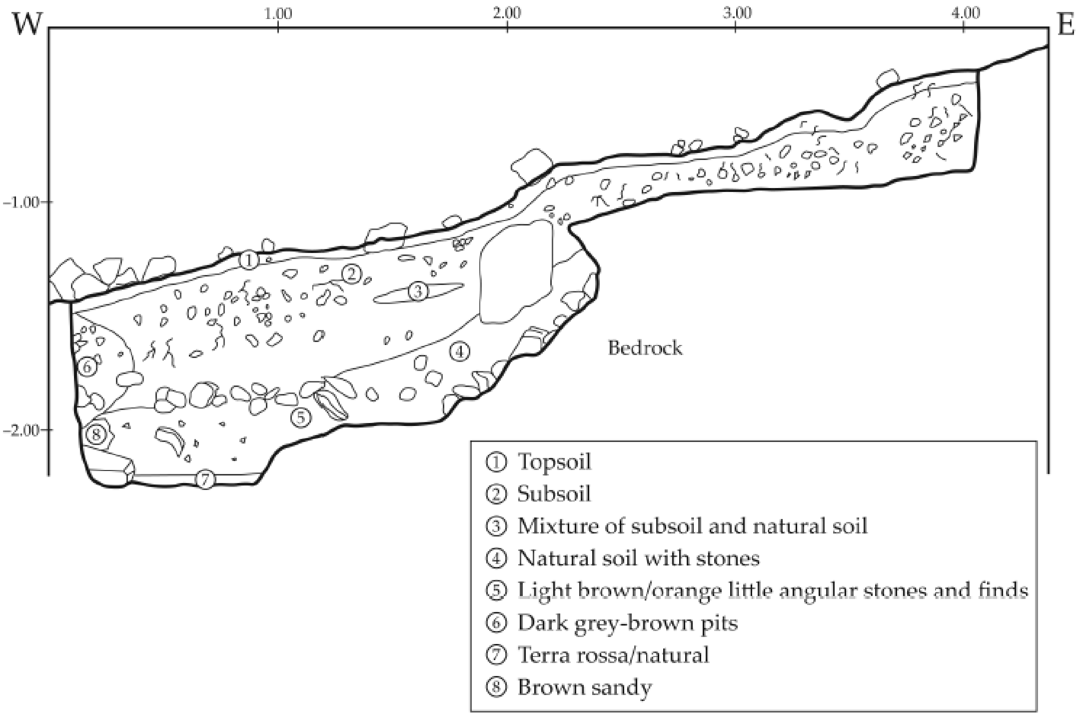


Fig. 77: Section of north baulk of trench D2 (Renfrew et al. 2015).

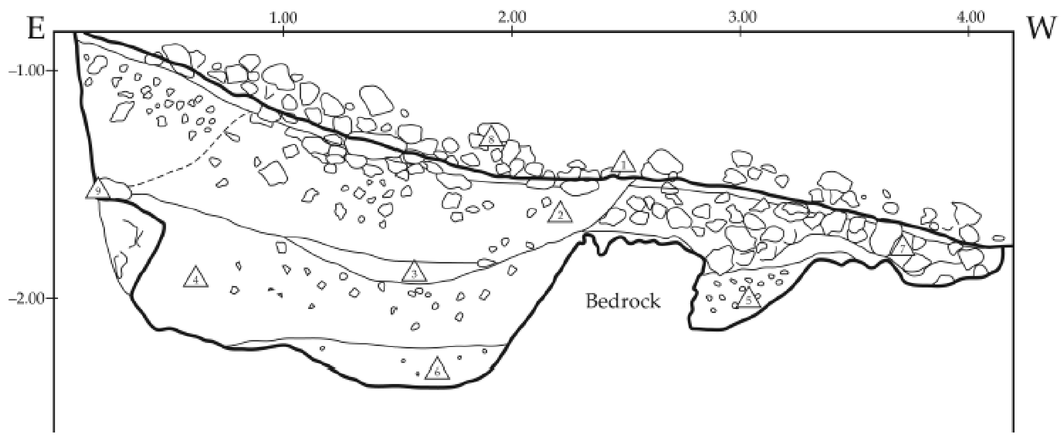


Fig. 78: Section of south baulk of the trench D2 (Renfrew et al. 2015).

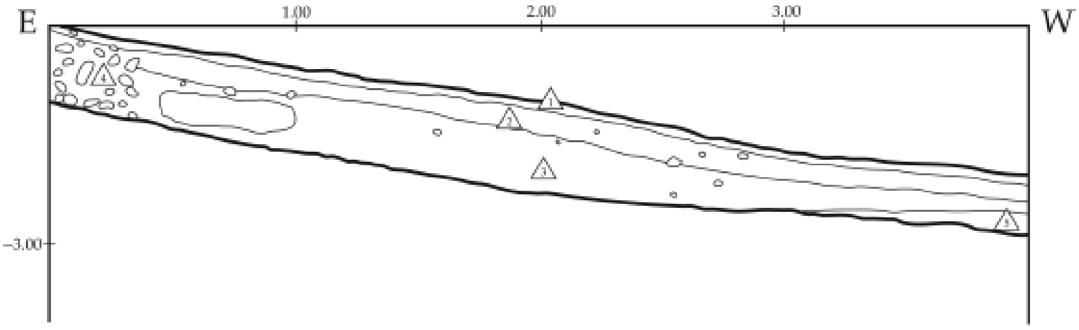


Fig. 79: Section of south baulk of trench D2 (Renfrew et al. 2015).

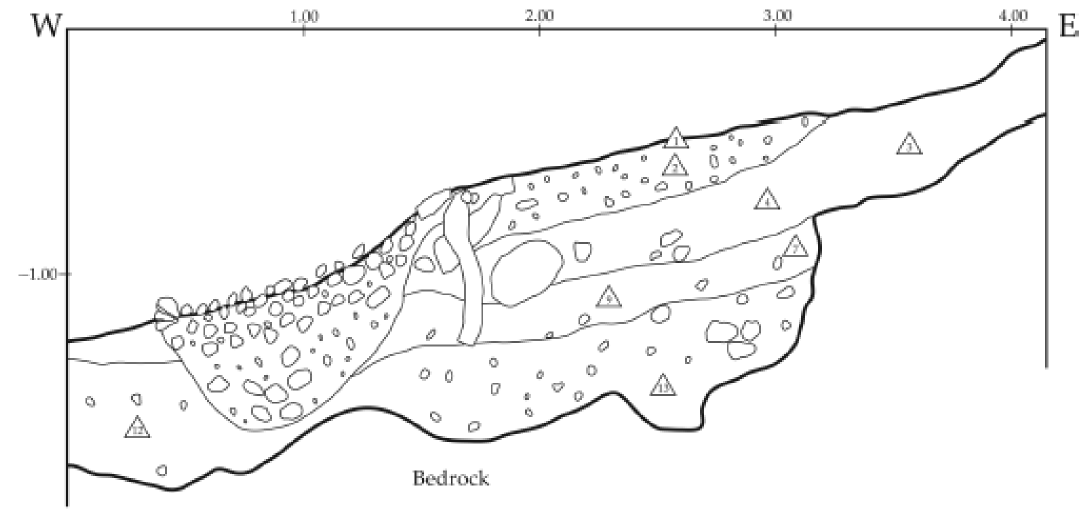


Fig. 80: Section of the north baulk of trench D4 (Renfrew et al. 2015).

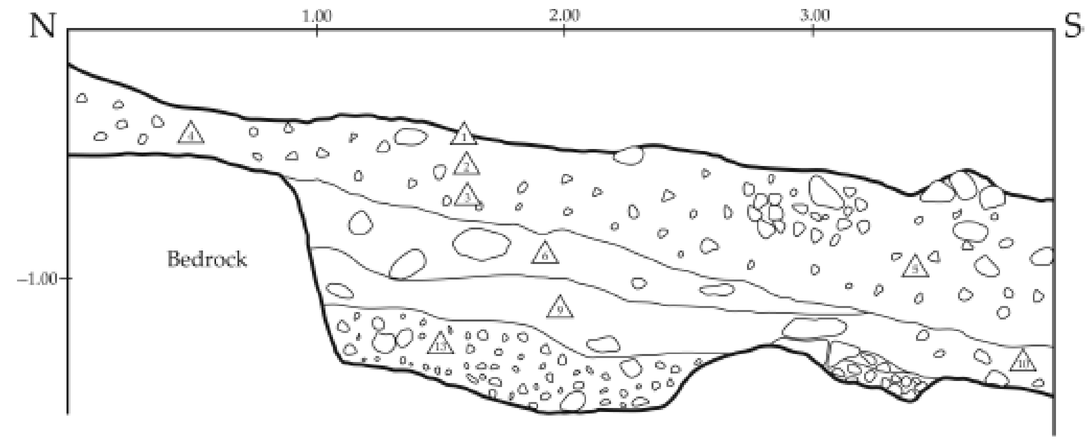


Fig. 81: Section of the east baulk of trench D4 (Renfrew et al. 2015).

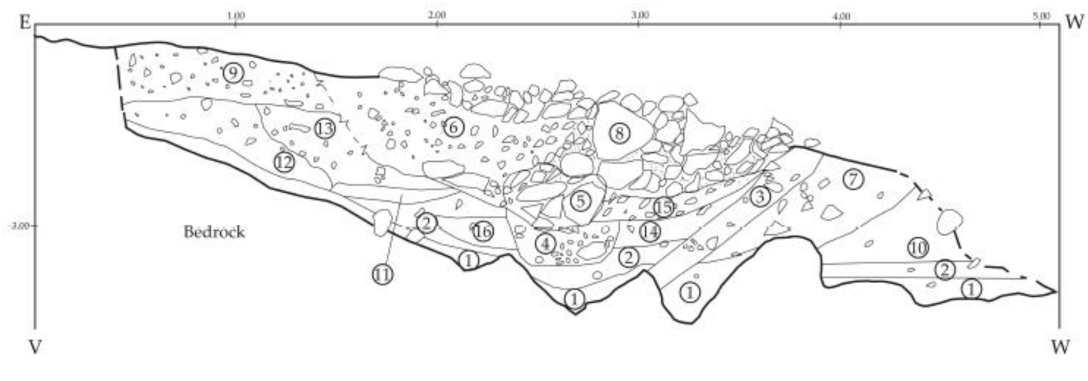


Fig. 82: The section of trench Ra, the different contexts are mentioned (Renfrew et al. 2015).

Fig. 83: The Special Deposit South (photo from the Southeast) (Renfrew et al. 2015).

Fig. 84: An amount of the figurines that have been found in Special Deposit South. The photo was taken during the study at the Archaeological Museum of Naxos (Renfrew et al. 2015).

Table 13: Photos of the used equipment, the stereoscopic microscope (on the left), the SEM (on the right).

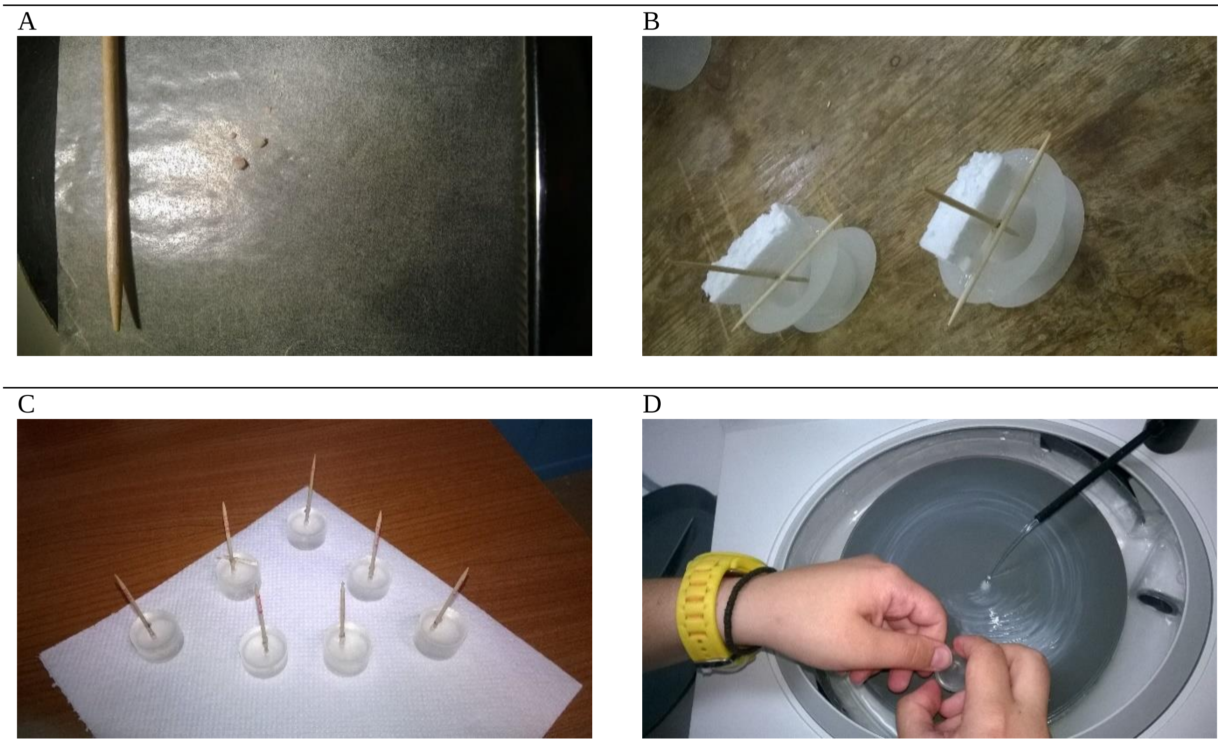


Table 14: Photos during the preparation of the polished samples. A: Selection of the representative particles under stereoscopic microscope. B: The particles are embedded in resin. C: the sample after the dry procedure. D: the samples during the polishing procedure.

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