

### Master of Science in Cultural Heritage Materials & Technologies





#### UNIVERSITY OF THE PELOPONNESE

### **AIMILIOS ZIKOURIDIS**

(R.N. 1012201602011)

#### **DIPLOMA THESIS:**

## NON-DESTRUCTIVE ACOUSTIC MICRO-TOMOGRAPHY OF TESSERAE FROM ANCIENT MESSINA

#### **SUPERVISING COMMITTEE:**

- Nikolaos Zacharias,

**Professor of Arcaheometry, University of the Peloponnese** 

- Dr. Georgios Karagiannis,

Scientific and technical head of the Diagnosis Centre of the «ORMYLIA» Foundation

#### **EXAMINATION COMMITTEE:**

- Nikolaos Zacharias
- Georgios karagiannis
- Theodosios Karamanos

**KALAMATA, JANUARY 2018** 

## Contents

INTRO	DDUCTION AND OBJECTIVE	4
СНАР	TER 1: HISTORICAL THROWBACK	5
1.	THE ANCIENT CITY OF MESSENE	5
Α.	THROWBACK	5
В.	THE SANCTUARY OF SARAPIS AND ISIS	8
I.	INTRODUCTION OF THE CULT OF ISIS TO MESSENE	8
CHAP	TER 2: SCANNING ACOUSTIC MICROSCOPY	10
2.	METHOD HISTORICAL REVIEW	10
Α.	ULTRASOUND THEORY - ACOUSTIC MICROSCOPY	11
В.	REFLECTION, DIFFRACTION AND ABSORPTION	12
c.	Sampling	13
D.	Analog Signal Converter to Digital (A / D Converter)	16
E.	Ways to obtain information using acoustic microscopy - ultrasound (A-B-C Scan modes)	17
СНАР	TER 3: S.A.M (SCANNING ACOUSTIC MICROSCOPY) CHARACTERIZATION OF TESSERAE FR	ЮМ
ANCIE	ENT MESSENIA	18
3.	CHARACTERIZATION OF TESSERAE FROM ANCIENT MESSENIA	18
Α.	TESSERAE: ARCHAEOLOGICAL BACKGROUND	18
В.	EXPERIMENTAL PROCESS WITH ACOUSTIC MICROSCOPY	21
c.	RESULTS FROM ACOUSTIC MICROSCOPY TOMOGRAPHY	22
CHAP	TER 4: C-SCAN 3D IMAGES OF THE ACOUSTIC MICROTOMOGRAPHY MEASUREMENTS – N	VI-
ROUG	GHNESS	30
СНАР	TER 5: CONCLUSIONS	33
RIRLINGPADUV		2/

## Table of Figures

Figure 1: The prefecture of Messenia (Google maps)	. 5
Figure 2: General topographic plan of ancient Messene (FROM P.THEMELIS, MESSENE.A	
MULTIPERIOD SITE)	. 6
Figure 3: The Asclepieion (From P.Themelis, 2010)	. 7
Figure 4: The sanctuary of Isis and Sarapis near the theater (from	
https://aristomenismessinios.blogspot.gr/).	. 8
Figure 5: Displacement of particles for a transmitted ultrasonic wave.	11
Figure 6: Reflection, absorption and propagation of ultrasound	12
Figure 7: Acoustic Microscope Layout Diagram of "Ormylia" Foundation	14
Figure 8: The SAM lens system [1] (a) Propagation microscope: 1 - piezoelectric converter, 2	2 -
diffuser, 3 - object, 4 - scanner, 5 - receiving lens, and 6 - transducer. (b) Reflection	
microscope: 1 - piezoelectric converter, 2 - lens, and 3 - object	15
Figure 9: The analog to digital converter	16
Figure 10: Tessera No11	19
Figure 11: Tessera No2	19
Figure 12: Tessera No 3	19
Figure 13: The observed layer layout No4	19
Figure 14: The porous surface of Tessera No 7	20
Figure 15: Tessera No 5	20
Figure 16: Tessera N°6	20
Figure 17: The SAM of "ORMYLIA" Foundation, in the background the container with Tesse	ra
is visible	22
Figure 18:Example of three-dimensional imaging of Tessera N°1	23
Figure 19: Illustration of tessera structure N°2	24
Figure 20:μTomography of tessera N°3	25
Figure 21: Three-dimension illustration of Tessera No4	26
Figure 22: Tessera No6 tomography of the structure	27
Figure 23. Three-dimensional imaging of Tessera N°7	28
Figure 24: 3D representation of Tessera N°2.	30
Figure 25: Representation of Tessera N <sup>0</sup> 3	31
Figure 26: Tessera N°6, Agglomerates and μ-Roughness	31
Figure 27: Tessera N°2 , Agglomerates-measurement of their size	32

#### Introduction and objective

The current master thesis has as target to analyze tesserae from ancient Messina mainly focused on the reveal nondestructively of the existence of agglomerates in the internal structure them. Agglomerates are the co-developments of crystals of a mineral so that they do not exhibit regularity to corresponding crystallographic elements. In order to achieve this target, tomographic techniques appeared to be the most appropriate in order to reveal the structures of the agglomerates from the one hand as well as identify if possible the material of the agglomerate structure. Taking into consideration the size and the distribution in the whole tesserae structure of the agglomerates the use of ultrasounds of high frequency was decided based also on similar resent and novel research efforts ([1]-[9]).

Since the sizes of the agglomerates are of the order of microns and decades of microns the frequencies of the ultrasonic that was used are of the order of 110-175MHz. Finally reconstructed tomographic images of the whole tesserae structures are displaying with high fidelity the distribution of the agglomerates in the whole structure. The resolution of the proposed and used method is of the order of several microns. The qualitative identification of the revealed agglomerates is the continuation of this work for the future research actions towards this direction.

#### CHAPTER 1: HISTORICAL THROWBACK

# 1. THE ANCIENT CITY OF MESSENE a. THROWBACK

Messene was an ancient city in the southwest of the Peloponnese in the Messenia prefecture (map1). Messene is one of the most important in size, form and maintained cities of antiquity.



Figure 1: The prefecture of Messenia (Google maps)

Messenia, like other parts of the Peloponnese, was inhabited by the upper Paleolithic (26000-9000 BC), according to recent research results. Particular efflorescence occurs during the Bronze Age, both in the Early Age (3200-2000 BC) and Middle (2000-1550 BC) and Late (1550-1050 BC).

The excavations that conducted for many years have started back in 1895 where the Archaeological society begun excavations at the site under the direction of the archaeologist Themistocles Sophoulis. Excavating activity resumed in 1909 and 1925 under the direction of G. Oikonomos. In 1957 Anastasios Orlandos who was the Secretary of the Archaeological Society and member of the Academy of Athens took

charge of the excavation project of Ancient Messene and worked until 1974. The excavation conducted by Anastasios Orlandos and his predecessors brought to light the greatest part of the building complex of the Asklepieion. All these excavators as well as the current excavator Petros Themelis<sup>1</sup>, give us historical evidence for the reconstruction of the city as well as for its fortifications and development.

The city was re-founded in 369 BC by the Theban general Epaminondas and the Argian allies, that owe its name to the first pre-Dorian queen of the country, Messini, the daughter of Argian king Triopa. It should also be noted that the efforts made by the Thebes to the establishment of the city were remarkable as heralds, on behalf of their name, were sent to Italy, Sicily and to the Libyan city of Eusperids and to all the exiled Messenians to return to their homeland.

The city was famous for its fortification walls, the monumentality of its public buildings and the Hippodamian town-plan, because all the buildings of Messini have the same orientation and are included to canvas created by horizontal (East-West) and Vertical (North-South) roads (Figure 2: General topographic plan of ancient Messene (FROM P.THEMELIS, MESSENE.A MULTIPERIOD SITE)).

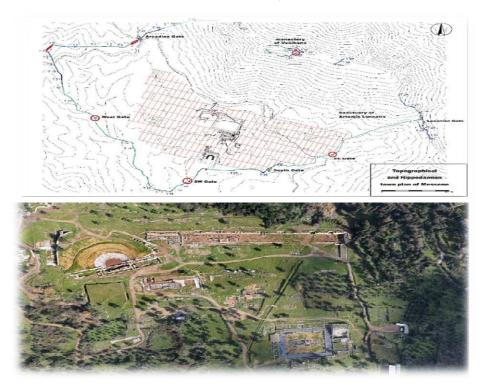


Figure 2: General topographic plan of ancient Messene (FROM P.THEMELIS, MESSENE.A MULTIPERIOD SITE)

\_

<sup>&</sup>lt;sup>1</sup>PetrosThemelis suggests that the systemic excavation of this site firstly took place by Georgios Oikonomouof the Athens Archaeological Society in 1909-1925.

Within the walls and the urban grid were built the most important buildings. Here there is among others the theater, the agora, the Asclepieion, the stadium, the Gymnasium, which also constituted the core of the city. Here, also, are situated the most important sanctuaries of Ithomi, of Demeter and Dioskouroi, the Limnatidos sanctuary as well as the sanctuary of Sarapis and Isis.

The city faced important changes during her long life existence up to late antiquity. In 14 AD the city suffered from a devastating earthquake and thus began an attempt to repair the existing buildings with contributions from affluent Greeks and Romans. At that time the Fountain of Arsinoe, the Ecclesiastical Hall, the Gates of the Gymnasium and stadium stables are repaired following a mission by Messinian Embassy to Emperor Tiberius for financial assistance.

The decline of the city, which for eight centuries flourished and was the cultural center of Messinia, appears in 365 AD when the last layers of destruction and abandonment of most buildings are dated. This is probably due to an earthquake as well as a fire. The decisive blow to this city, which was already in full decline, probably came from the Gothic raid of Alaricus in the Peloponnese in 395 AD [11].



Figure 3: The Asclepieion (From P.Themelis, 2010)

Archaeological research, however, revealed another building phase when an Early Byzantine settlement was founded over the ruins of the ancient city in 5<sup>th</sup> century BC

and existed until the years of Frankish and Venetian rule in the 15<sup>th</sup> century (P.THEMELIS, 2010).

#### b. THE SANCTUARY OF SARAPIS AND ISIS

i. Introduction of the Cult of Isis to Messene.

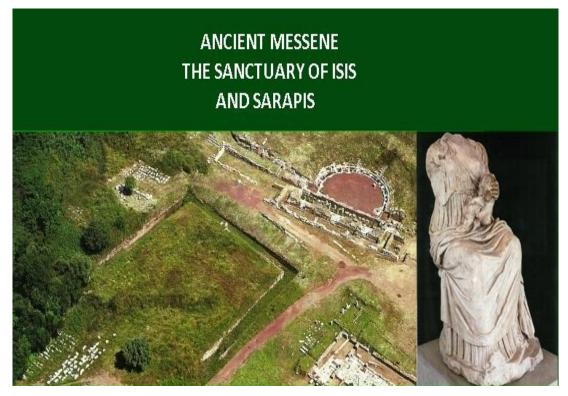


Figure 4: The sanctuary of Isis and Sarapis near the theater (from https://aristomenismessinios.blogspot.gr/).

The location of the sanctuary of Isis and Sarapis, as P. Themelis informs us, is known from Pausanias<sup>2</sup>. The northern part of 46.50 m in length, while the Eastern and the Western 35.50 m. the width is 3.25 m., while the depth reaches 3.50 m.

The cult of Isis in the ancient Messene probably originated from the trade between Messenian and Cretan merchants as well as the direct trade with Egypt. Petros Themelis suggests that based on the dating of the finds that are connected with the Sarapis and Isis sanctuary, the worship of the Egyptian gods seems to have been established at Messene in the 2<sup>nd</sup>century BC(P.Themelis, The Cult of Isis at Ancient Messene, 2011). The sanctuary survived until the 4th century when it was probably

<sup>&</sup>lt;sup>2</sup>Pausanias, (4.32.6), «τοῦ θεάτρουδὲοὂ πόρρω Σαράπιδός ἔστι καὶ Ἰσιδος ἱερόν». «Not far from the Theatre, there is a sanctuary of Sarapis and Isis».

destroyed by the devastating earthquake in 365 AD. The material studied and analysed for this thesis originated from this sanctuary and consists of seven (7) Tesserae.

#### **CHAPTER 2: Scanning acoustic microscopy**

#### 2. Method historical review

Acoustic microscopy is the type of microscopy that uses high frequency ultrasounds. Acoustic microscopes work non-destructively and "penetrate" most of the solids to reflect their internal characteristics.

The idea of acoustic microscopy dates back to 1936 when S.Ya. Sokolov, proposed a device that would produce extended faces of a structure using 3 GHz acoustic waves. Because of the technological limitations of time, such an instrument could not be produced. This was overturned when Dunn and Fry showed their first experiments on acoustic microscopy in 1959, though they did not work at such high frequencies (Maev, 2008)

After the first experiments, acoustic microscopy developed slowly until 1970, when two scientific groups appeared: one from C.F. Quate from Stanford University and the other from A.Korpel and L.W.Kessler from Radio Zenith. They dealt with the construction of a functional acoustic microscope. The Quate team began in 1973 to develop an idea that led to the creation of the SAM (Scanning Acoustic Microscope) model, which achieved a high resolution image in original ways. SAM was sold by Leitz and Olympus. It then followed SLAM (Laser Acoustic Microscope Scan), which had a laser detection system for the acoustic microscope, which only works with the transmitted wave. In 1984, Kessler's team completed the C-SAM instrument model, which, on contrary to SLAM, operated with the reflected wave and the transmitted wave.

These microscopes are the ancestors of modern microscopes and their development is the beginning of many future developments such as the three-dimensional acoustic image. The ultrasonic frequencies in the acoustic microscopes range from the low of 5-10 MHz to 2GHz and more. "By the length" of this spectrum there is a compromise between penetration and analysis. Low frequencies have better penetration, while the high ones have much better resolution in the final result. The frequency selected for the appearance of a particular sample depends on the geometric characteristics and the materials that are composing the sample.

#### a. Ultrasound Theory - Acoustic Microscopy

If ultrasound is characterized by acoustic waves from 20 KHz to 20 GHz, the ultrasonic function is pretty much the same as the radar, but in the case of ultrasound it is used a mechanical wave, instead of an electromagnetic wave. The standard ultrasonic frequency range used in non-destructive testing is from 100 KHz to 100MHz.

In 1883 the first supersonic siren was made by Francis Galton. Ultrasound is now widely used in engineering applications, in medicine and in many other areas, and is gradually being developed in the analysis and examination of objects of cultural heritage. The wave propagates like a particle disruption of the medium that sustains the propagation of the wave as shown in the Figure 5.

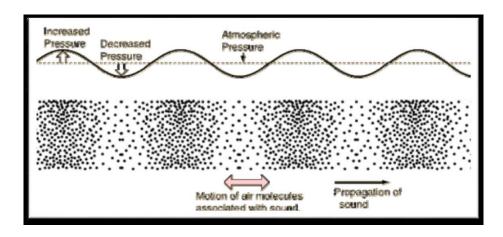


Figure 5: Displacement of particles for a transmitted ultrasonic wave.

The particles of the medium are initially at rest and evenly distributed if there is no wave interruption. Due to the presence of ultrasonic wave, the particles will rotate around their position. The oscillation occurs along the wave propagation direction, making it a longitudinal wave. There is almost no total particle displacement and mass transport. The ultrasonic wave acts as a simple disorder in the medium. In particular, the distance traveled by the particles during sound propagation is called "particle displacement" and is usually in the order of 1 nm.

Consequently, the velocity of the oscillating particles is called "particle velocity". It should be stressed that this velocity differs from the energy transmission rate in a medium, which is actually defined as "phase velocity" and which receives much higher values than "particle velocity". Finally, despite the fact that the particles are

moving at some nm, the breakdown that is caused is transmitted to other particles of the medium at a much greater distance [15].

Acoustic microscopy is a non-destructive method of analysis that aims at collecting information through the emission of high frequency sound waves. Since the sound is essentially a mechanical wave, has the ability to interact with material that meets and uncover useful information about elastic properties. Acoustic microscopy uses the principle that sound waves reflected when, when multiplied, encountering material interfaces with different typical/specific acoustic resistances (where  $\rho$  is the density of the medium and the speed the sound in this way). So, it appears almost absolute reflection interfaces means big difference on acoustic elements between them, while the reflection is minimal when two materials/instruments have similar acoustic elements.

#### b. Reflection, diffraction and absorption

When an ultrasonic wave falls on a wall or generally on a two-media separation surface, part of the wave transferred is reflected, while another part is absorbed or propagated through the wall on the other side of Figure 6. From the various interfaces a reflected echo is generated due to the different acoustic impedances of the two materials. The acoustic impedances of each material is provided from the following equation:

Z=p\*c, where p is the density of the material and c the acoustic velocity of it.

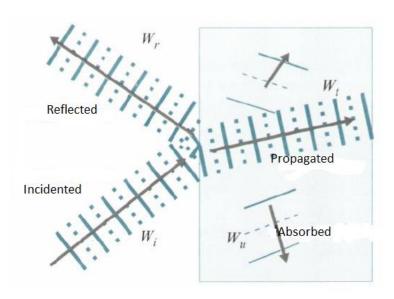


Figure 6: Reflection, absorption and propagation of ultrasound

#### **Absorption from materials**

The absorption of ultrasounds from various materials is mainly due to two phenomena:

- a) As the ultrasonic wave penetrates into the material that is usually porous, the propagation medium (usually air) in the friction with the material loses energy. So we have a sound conversion to thermal.
- b) The ultrasonic wave stimulates the molecules of the absorbent material so that the energy of the ultrasonic wave decreases.

Absorbent materials can be divided into three main categories: porous, membranetype absorbents and resonators.

#### c. Sampling

The basic arrangement of the acoustic microscope is presented in Figure 7. The piezoelectric transducer is activated by a 200Vp-p amplitude pulse from a transmitter having a range of 400MHz. The selected mode is broadcast and receives (Pulse / Echo mode) with controlled piezo-inductive excitation rate. The signal received from the piezoelectric transducer is amplified using a preamplifier and digitized by the A / D converter (analogue to digital converter). An analog to digital converter has a sampling rate of 2GSamples / s.

The resultant waveform is the useful signal through which, by the appropriate techniques of scanning or processing, the fluctuations in the acoustic characteristics of the measured structure result. The position and movement of the piezoelectric transducer is continuously controlled by a suitable high precision moving system.

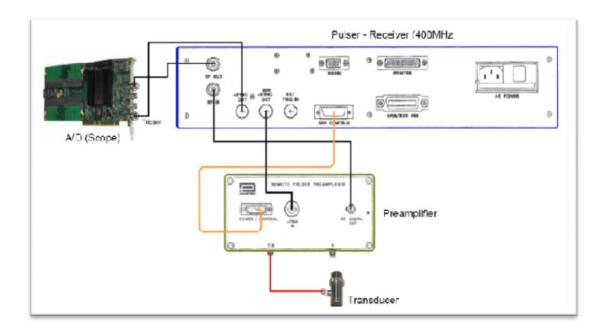


Figure 7: Acoustic Microscope Layout Diagram of "Ormylia" Foundation

SAM uses a focused audio signal to research, measure, or visualize an object, a process known as Scanning Acoustic Tomography. Used in error analysis and non-destructive evaluations. It has a wide range of applications, including biological and medical research. Also the semiconductor industry uses it to detect gaps, faults and failures in integrated circuits.

The lens system is the most important tool of a SAM. As mentioned, a high frequency acoustic signal is produced in the lens system by a transducer. The signal is propagated through a medium having a high acoustic resistance and then focuses, with the help of a lens, on the other end of the medium. This is immersed in liquid to fill the gap between the lenses and the object under consideration. Immersion in the liquid provides a kind of "acoustic continuity" as it achieves a smooth transition between the medium and the object to be examined.

The focused beam, interacting with the object, is partially reflected and diffused by the object and partly propagated through the object. If the reflected wave is detected, the microscope operates by reflection. When the transmitted acoustic wave is recorded by a second lens, then the microscope operates with propagation (Figure 8) [14].

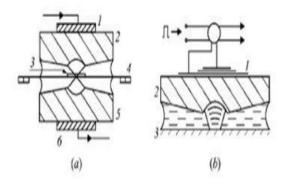


Figure 8: The SAM lens system [1]
(a) Propagation microscope:

1 - piezoelectric converter, 2 - diffuser, 3 - object, 4 - scanner, 5 - receiving lens, and 6 - transducer.
(b) Reflection microscope:

1 - piezoelectric converter, 2 - lens, and 3 - object.

An acoustic wave is usually produced by a piezoelectric transducer. Intense, small electrical pulses from the ultrasonic machine cause the transducer to be tuned to the desired frequency. The acoustic wave is focused by the lens located in front of the transducer and produces a spherical acoustic wave from the surface of the sensor. The wave travels to the body and focuses on a desirable depth.

The return of the acoustic wave to the inverter has the same effect as the sending of the acoustic wave but from the reverse. That is, the returned acoustic wave causes the transducer to vibrate at its frequency and then convert that vibration into electrical pulses, which are processed and transformed into images.

The greater the difference between the acoustic resistances is, the greater the reflection. If the pulse "collides" with gas or solid, then the difference in density or rather better in the Young modulus is so great that most of the sound energy is reflected and it becomes impossible to penetrate the wave deeper. For this reason a special dispersion medium (water, gel) is also used to make a smooth change of Young modulus<sup>3</sup>.

-

 $<sup>^{3}</sup>$ Young modulus is the ratio of stress to tension. The tension is dimensionless, so the Young modulus has pressure units, which in the system SI is the Pascal (1Pa = 1N / m2).

The Young modulus size value is not always the same for all the addresses of hardware. If the material is anisotropic, then the Young modulus value varies depending on the direction in which the force is applied.

#### d. Analog Signal Converter to Digital (A / D Converter)

The analog to digital converter (Figure 9), supports the input of two analogue signals (2 channels A and B) with 8 bit resolution at a sampling rate of up to 2.0G Samples / sec. The resulting data is input to a Field Programmable Gate Array (FPGA), where they can either be processed in real-time or stored using the built-in memory in the converter. Real-time processing is very important so that the user has the ability to observe the phenomenon during the download process and to react when the process has been diverted from the desired target. This can happen very easily since the resonance activity of the layers lasts only a few tens of nsec, so if the relief of the object changes to "height" a few tens of small, echoes cannot be taken in the observation window that is set.

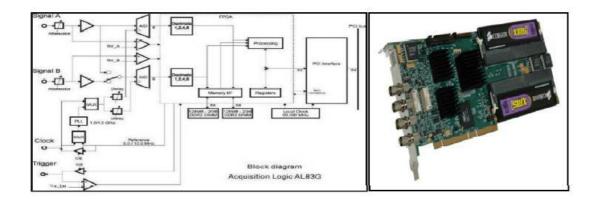


Figure 9: The analog to digital converter

The resulting data is transferred to the PC's main memory via a 64-bit / 66MHz PCI data passage, using Direct Memory Access (DMA) to ensure that other data transfers are not delayed by any latent operating system situations or application programs to provoke.

The output data is transferred to the PC's main memory using DMA transfers to the PCI data pass. The PCI interface used on this card supports 64 bit 66MHz data rate operation. All control functions on the card are accessed through a 256-digit address window in the address space and I / O devices and memory. These functions use only part of the available resources in the FPGA. The rest part can be used to implement a specific processing algorithm [5].

## e. Ways to obtain information using acoustic microscopy - ultrasound (A-B-C Scan modes).

There are 3 ways that are used to visualize the information provided by the acoustic microscopy: A-, B- and C-scan:

A-scan is nothing more than the (digitized) waveform of the signal received by the piezoelectric converter from a single measurement point. The inverter remains stationary and sounds as it is at a distance preferably equal to the focal length of the project. A waveform is taken, one axis referring to the amplitude of the received signal, and the other the time of arrival of each point of the waveform. Since the sound velocity of the sounding material is known, one can convert the time of arrival away from the converter, so it receives the information of the "Time of Flight" (TOF). The A-scan and in particular the width of the 1st echo can provide information about the nature of the outer surface of the measured material. Porous or granular materials cause more scattering and absorption in the incident sound wave than a smooth and solid material, and this difference is reflected in the width of the measured signal [10].

In B-scan, the piezoelectric converter moves along a line where it performs multiple A-scans. Thus, a 2-dimensional image is created, in which one axis is the one in which the piezo-electric converter moves and the other the TOF information from each point being emitted. The width of the signal at each point of the 2-dimensional image is designed / marked as a different intensity of that point, ranging from white to black [10].

In the C-scan, piezoelectric converter moves onto axy plane scanning an entire region of interest (ROI). At each point in this area it performs an A-scan measurement. In this way, the user has at his disposal the signal width and the TOF information from each point in the area and has the ability to construct 3-dimensional representations of the area being emitted at different distances from the piezoelectric transducer (and hence different depths within the material).

# Chapter 3: S.A.M (Scanning acoustic microscopy) Characterization of Tesserae from Ancient Messenia

#### 3. Characterization of Tesserae from Ancient Messenia

This chapter lists the results of analyses of tesserae with the non – destructive techniques in order to qualify the existence of agglomerates. The term agglomerates means the development of crystals of a mineral so that they do not exhibit regularity to corresponding crystallographic elements.

For the purposes of the present study, a total of seven (7) tesserae were studied. The tesserae originally were studied with the Optical microscope, and subsequently with the method of acoustic microscopy (SAM). Also it should be noted that an attempt was made to study the tesserae with the Raman method, FTIR and XRF but the results will not be included in this work but will be used for future research.

All tesserae that were analyzed and studied originated from the temple of Serapes and Isis that is located in Ancient Messini, which is extensively mentioned in a previous chapter (introductory chapter).

#### a. Tesserae: Archaeological background

Ancient glasses were generally obtained from a mixture of naturally occurring materials containing silicon, alkali and lime. Beach sand and a source of alkali were typical ingredients, both with sand and with alkali containing enough lime or magnesia to give a sufficient amount of chemical stability. From 1000 BC in eastern Mediterranean glasses used Natron (hydrated Na<sub>2</sub>CO<sub>3</sub>) this availability can be found in the north Egypt as a favorite source of alkali. This practice continued also through the Mediterranean region until Late Antiquity [12].

Glass is usually marred by adding small amounts of certain salts (mainly copper, iron and manganese). This addition of dyes was perhaps the first example of using small ingredients by changing the properties of the glass to produce the desired effect. Opaque glasses were taken with crystalline suspension, quite insoluble in the internal glass matrix, such as calcium, lead antioxidants, and tin oxide [20].

Ancient mosaic tesserae are a range of materials of very varied and complex nature, including pottery, stone and glass. The advantages of a glass tesserae in contrary to stone or ceramic material were the ability to offer a range of colors and a glamorous quality that could increase the bold contrasts in iconographic representations[19].

Tessera  $N^{\circ}1$  (Figure 10), glass tessera, in which we can discern its structure and bubbles that are found in.

The only yellow colored tessera is N°3 (Figure 12), which is of a great interest.

Tessera N°2 (Figure 10), is one of a great interest and will be presented more extensively. As it is previously mentioned all tesserae were found and located in the temple of Sarapis and Isis in the archaeological site of Ancient Messini. Its color is green. Both macroscopically and microscopically there are observed some gaps that are probably due to the firing method. It is very important because when examined with the acoustic microscope showed a large number of agglomerations. Same to N°2 is the tessera N°4 which seems to have a double layer.



Figure 10: Tessera No 1

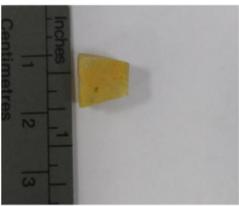


Figure 11: Tessera N° 3



Figure 10: Tessera N°2



Figure 12: The observed layer layout N°4



Figure 13: The porous surface of Tessera No 7

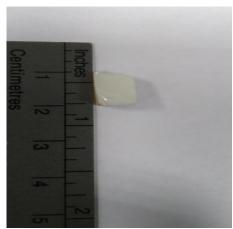


Figure 14: Tessera No 5

There were examined also two white-beige colored tesserae (Figures 14, 15). Macroscopically the tessera No7 due to its porous surface, shows several cavities, where also agglomerations are observed. The tessera No5 shows good homogeneity with minimal bubbles.



Figure 15: Tessera N°6.

Tessera Nº6 (Figure 16), presents several cavities resulting from bubbles as well as agglomerates. The particular tessera due to the lack of smooth surface was the most difficult to measure by the technique of acoustic microscopy.

#### b. Experimental process with acoustic microscopy

Acoustic microscopy is a new method in the field of art analysis. In the future, it will be a powerful non-destructive imaging tool for the internal structures of each object, which will provide information to scientific staff of all specialties, which will relate to conservation status, depth of wear, and manufacturing details.

Using acoustic microscopy, it is possible to accommodate the three-dimensional internal structure of the mosaics, capture the roughness of the surface, imprint the multilayer structure, and calculate the thickness of the different layers. Ultrasound microscopy uses frequencies up to 200 MHz to examine structures.

The information coming from the acoustic microscope is quite difficult to uncover, since ultrasonic high frequency waves (100MHz and above) ensure the resolution required but at the same time show significant losses when propagated into the multilayer structures of the data chips that the material which forms the glass is not homogeneous, bubbles and impurities are contained. In addition, the not flat surface makes the transport of the wave more difficult. Therefore, the discovery of small resonances (of the order of magnitude  $\mu V$  and mV), often overlapping one another, which translates into local changes of the signal phase in means of nsec, requires processing of signals by the use of special tools.

A prerequisite for taking measurements is to transfer the wave from the transducer to the sample without being attenuated, for this purpose, the test pieces were placed in a container and covered with water. For the experimental procedure the 100 MHz transducer was used without delay line and mapping was performed on a representative section of all Tesserae.

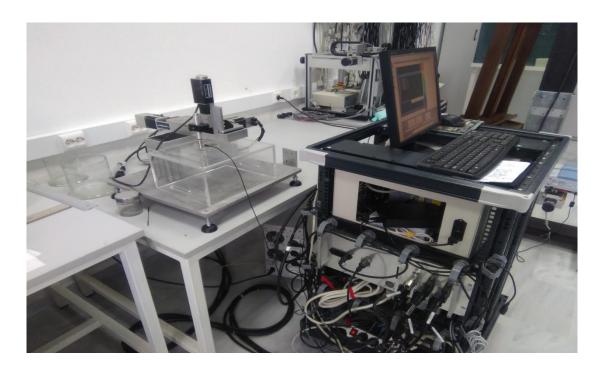


Figure 17: The SAM of "ORMYLIA" Foundation, in the background the container with Tessera is visible.

The initial data from the acoustic microscope must be processed as the visual study of signal waveform is not sufficient to reveal and certify the existence and number of agglomerates. For this process, all one-dimensional A-Scans measured in a region of interest (ROI) were collected and stored in a suitable file format on PC to represent the three-dimensions of the area (c-Scan) through a program. For this reason, the results presented below were derived from the 3D Slicer program

#### c. Results from Acoustic microscopy tomography

On this chapter the results of acoustic tomography will be presented. For a better understanding of the images that will be shown below, an example will be used as a reference (Figure 18). The tab 1 and 4 shows the vertical B-scans. On the tab 3 there is presented the C-scan, where the point of interest will be presented by F (1, 2, etc), and in the second tab (2) we observe the A-scan from the point of interest which will be within the circle.

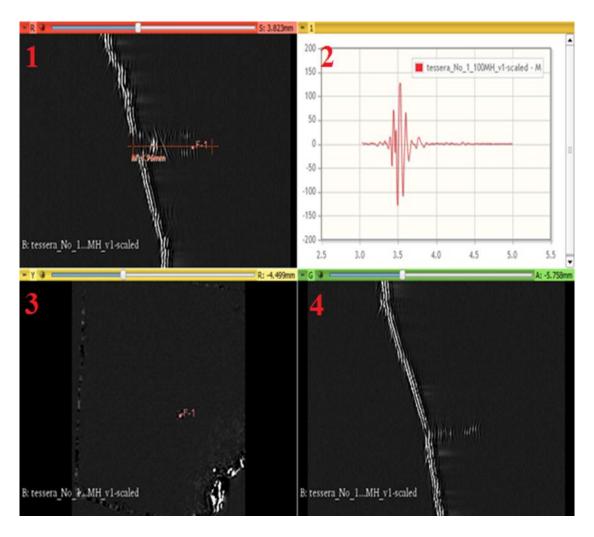


Figure 18:Example of three-dimensional imaging of Tessera N°1. Tabs: 1,4 B- scans, 3 C-scan, 2 A-scan.

In the representation of the structure of the 1<sup>st</sup> tessera (Figure 18) the existence of echoes shows the existence of agglomerates. After thorough scrutiny no more agglomerates were observed.

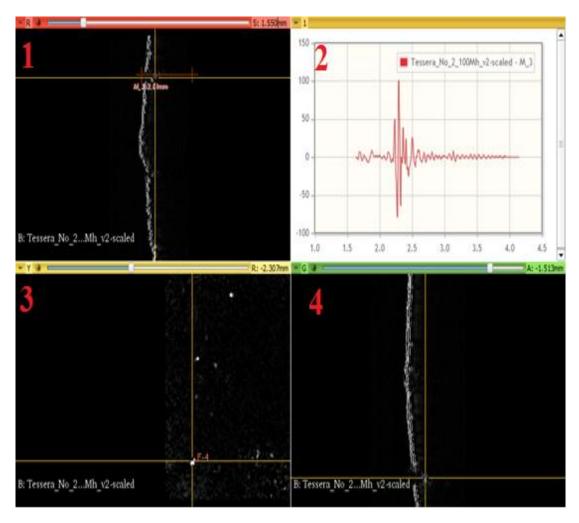


Figure 19: Illustration of tessera structure N°2.

Unlike the 1st, the 2nd Tessera has several reflections within the structure, which lead us to the idea that the material does not appear homogeneous, but there are several variations that lead to resonances of the sound signals. This indicates the result that there are many agglomerates, as there are a large number of them that are observed in

Figure 19.

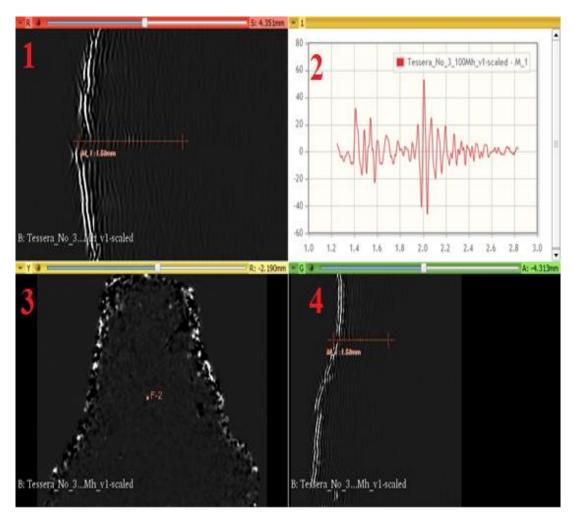


Figure 20:μTomography of tessera N°3.

Similar to the second we observe that the material does not appear homogeneous and the existence of resonances in the  $3^{rd}$  tessera. This leads to the conclusion that there are agglomerates that are visible, acoustically in the internal structure.

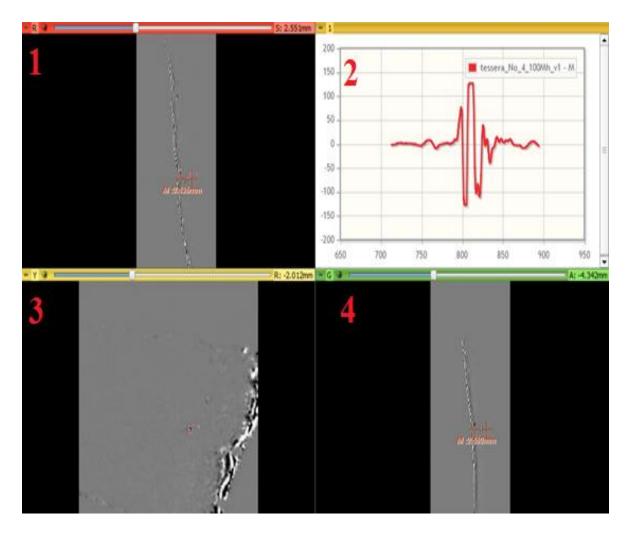


Figure 21: Three-dimension illustration of Tessera No4

In the next Tessera (Figure 21), there is also the suspicion of agglomerates, in C-scan it is pointed the Region of Interest (ROI) and in the A-scan it is observed the echo from the agglomerate.

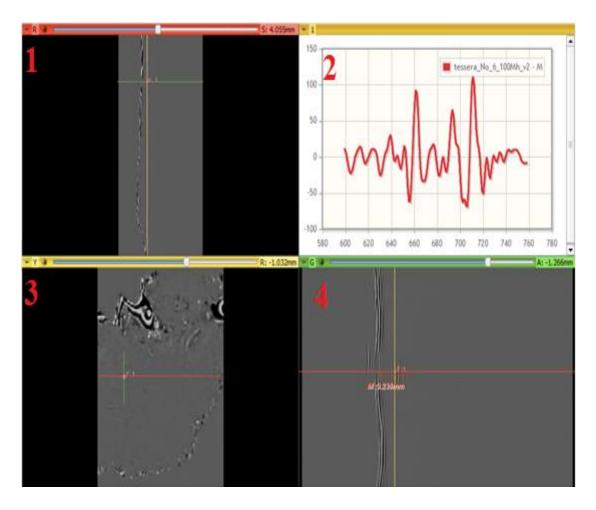


Figure 22: Tessera No6 tomography of the structure.

In the tessera  $N^{\circ}6$ , and specifically in the tab 4, fragmented echoes are distinguished in the structure, which are due to the internal heterogeneity of the material, which indicates the existence of agglomerates.

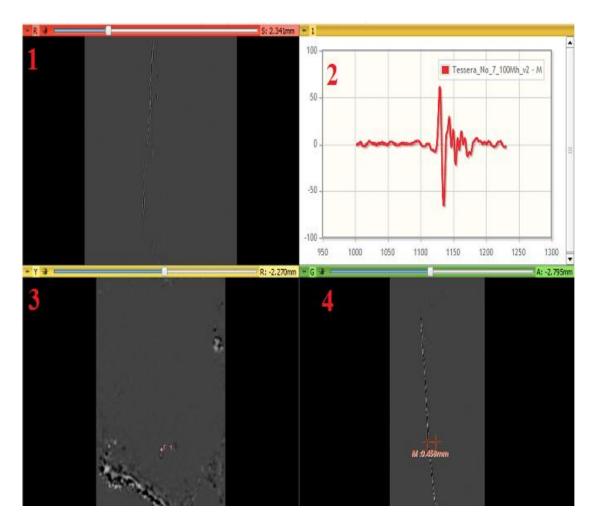


Figure 23. Three-dimensional imaging of Tessera N°7

The tessera N<sup>o</sup>7, is of particular interest as through echoes it is perceived the existence of agglomerates.

In thumbnails 3 and 4, we can observe the agglomerates that exist in the structure of each tessera, suggesting that they constituted a manufacturing defect of the material during production.

The information provided from the presence of echoes can lead us to estimate the number of agglomerates and their thickness. Through the three-dimensional imaging program 3D Slicer, as we will see below, we can select structures that can provide us with information about the length, width, thickness or even the radius, area or diameter of a circle. In any way, to calculate the thickness of agglomerate, it is necessary to know the propagation of the acoustic velocity in the material to be analyzed. Knowing the sound propagation velocity in glass, which is 3950, we can

measure the dimensions at a given moment with a low probability of error as the limits are not clearly distinguishable. The results shown in every Figure, from the agglomerate measurement do not require conversion because the measurement result does not concern the acoustic wave propagation distance but the echoes of the given moment.

# CHAPTER 4: C-Scan 3D images of the acoustic microtomography measurements – μ-Roughness

With the program mentioned previously, 3D slicer, we can create 3D images from the acoustic results. As we will see in the images the depiction is similar to the Tesserae studied. This ability is also the power of acoustic microscopy as we have a live image from the inside of the various objects that we can study with this technique. Also from the following pictures we can draw a number of conclusions regarding the roughness of the surface. In the 3D images there is the possibility to measure with high fidelity and precision the size of the agglomerates structures. For that purpose an acoustic velocity of the materials was assumed to be equal to 3950m/sec like the velocity of the glass. This was done since the specific studied tesserae are made from glass. This is an issue for further research in the future.

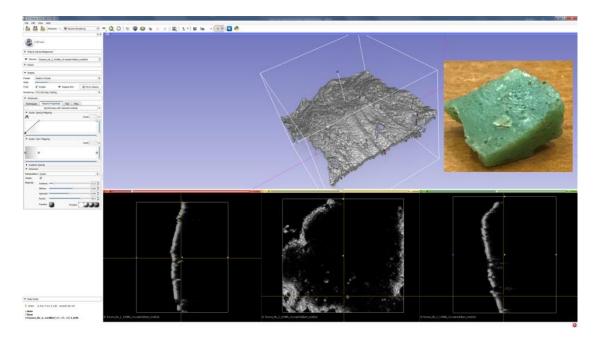


Figure 24: 3D representation of Tessera N°2.

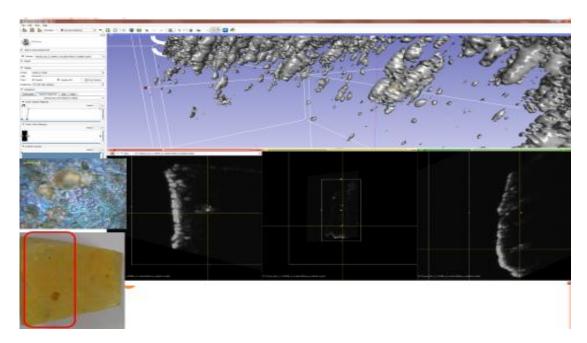


Figure 25: Representation of Tessera N<sup>0</sup>3.

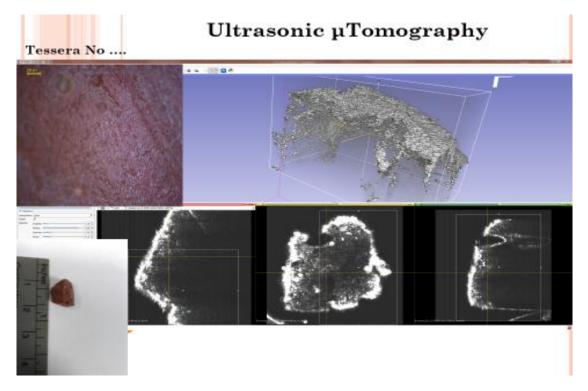


Figure 26: Tessera N°6, Agglomerates and μ-Roughness

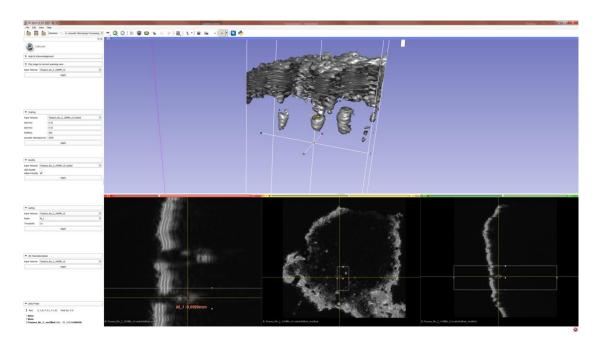


Figure 27: Tessera  $N^{\circ}2$ , Agglomerates-measurement of their size. An acoustic velocity of 3950 was assumed since the main material is glass.

#### **CHAPTER 5: Conclusions**

In the current master thesis, the integration of acoustic microtomography is proposed in order to analyze tesserae as far as the reveal nondestructively of the existence of agglomerates in the internal structure them. Due to the capability of the ultrasounds with operating frequency of the order of 75MHz-175MHz to penetrate the whole structure of the tesserae the following capabilities in the presented µtomographic images were provided by the proposed method:

- "Roughness" between the various interfaces between different materials in the internal structure.
- Agglomerates distribution in the internal structure with high fidelity 3D reconstruction and measurement of their dimensions with resolution of the order of several microns depending on the acoustic velocity of the local material. In this case an acoustic velocity of 3950 was taken for granted for the specific materials.
- Various internal defects were also revealed.
- The ultrasonic μtomography is also a "3D tracing imaging" capable to reveal the pathology and technology or ways of creation of the tesserae.

Based also to the fact that using the ultrasonic infrastructure described in the current thesis the tracing of the first echo generated from the surface of the under-study object the measurement of the surface  $\mu Roughness$  is also capable.

Since the limits and potentials of the ultrasonic µtomography have not yet been fully studied and it is certain that in the future it will be a diagnostic tool that will yield important conclusions, different from the other methods, broadening the scope of research.

The forthcoming and continuation of the current work is the use of the raw data of the ultrasonic data named a-scans the qualitative analysis of the agglomerates can be performed and thus identify the materials of them.

#### **Bibliography**

- [1] Georgios Karagiannis, Dimitrios Alexiadis, Argirios Damtsios, Georgios Sergiadis and Christos Salpistis, 3D non destructive "sampling" of art objects, IEEE instrumentation and measurements, vol.60, issue 9, Pages 1-28, September 2011.
- [2] G. Marchioro, G. Apostodolis, G. Karagiannis, M. Galeotti, C. Daffara, Surface and subsurface layers' characterization in artworks using conoscopic laser holography and acoustic microscopy, SPIE O3A, Munich, June 2017.
- [3] Georgios Karagiannis, "3D "spectracoustic" system: a modular, tomographic, spectroscopic mapping imaging, non-invasive, diagnostic system for detection of small starting developing tumors like melanoma", Proc. SPIE 10064, Photons Plus Ultrasound: Imaging and Sensing 2017, 100645Z (April 24, 2017); doi:10.1117/12.2271498; http://dx.doi.org/10.1117/12.2271498
- [4] Georgios Karagiannis, Scientific and Technical Head, Diagnosis Centre of the ORMYLIA Foundation, Innovative instruments and methods for integrated approaches to CH analysis and diagnostics), International Symposium on 'Cultural Heritage and Data: The Role of Research Infrastructures', December, 5-6 2016, S. Dillon Ripley Center, Smithsonian Institution (1100 Jefferson Drive SW, Washington DC 20560).
- [5] G. Karagiannis, G. Apostolidis, S. Sotiropoulou, K. Fragoulis, D. Minasidis, A. Mentzos, Application of a non-destructive testing mobile lab for in situ analysis of archaeological objects from the "Late Antique house" in Dion, Greece: results and data interpretations, ISA2016, May 2016 Kalamata, Greece.
- [6] G. Karagiannis, Non-destructive endoscopy of cultural heritage objects of various kinds and materials, ISA2016, May 2016 Kalamata, Greece.
- [7] G. Karagiannis, 3D spectroscopic mapping tomography applied to art objects diagnosis, InART, International conference on innovation in art research and technology, Ghent Belgium, 22-25th March 2016.
- [8] G. Karagiannis, Spectroscopic mapping tomography, SPIE Photonics Europe 2016, SQUARE Brussels Meeting Centre, Brussels, Belgium 4 7 April 2016.
- [9] Georgios Karagiannis, Georgios Apostolidis, Christos Salpistis, Semeli Pingiatoglou, Aristotelis Mentzos, "Acoustic Microscopy applied to archaeological objects", Technart 2015, April 27 30, 2015, Catania, Italy.
- [10] Anon., n.d. *Pvatepla*. [Online]

  Available at: <a href="http://www.pvateplaamerica.com/acoustic\_microscope/acoustic\_microscopy-scanningmodes.php">http://www.pvateplaamerica.com/acoustic\_microscope/acoustic\_microscopy-scanningmodes.php</a>
- [11] Papahatzis, N., 1995. Παυσανίου Ελλάδος Περιήγησις ΙΙΙ. Μεσσηνιακά-Ηλιακά. Αθηνα: ΕΚΔΟΤΙΚΗ ΑΘΗΝΩΝ.
- [12] Freestone, I. C., 2005. The Provenance of Ancient Glass through Compositional Analysis. *Materials Research Society*, Issue 852, p. 008.1.1–008.1.14.

- [13] Khuri-Yakub, B., 1993. Scanning acoustic microscopy. *Ultrasonics*, pp. 361-372.
- [14] Maev, R. G., 2008. Scanning Acoustic Microscopy. Physical Principles and Methods. Current Development. In: *Acoustic Microscopy: Fundamentals and Applications*. Weinheim, Germany: Wiley-VCH Verlag GmbH & Co. KGaA.
- [15] Miller, D. L. S. N. B. B. M. R. C. G. J. H. K. M. I. R. S. a. B. C. o. t. A. I. o. U. i. M., 2012. Overview of Therapeutic Ultrasound Applications and Safety Considerations. *Journal of Ultrasound in Medicine*, 1 April, 31(4), p. 623–634.
- [16] P.THEMELIS, 2010. *ΑΡΧΑΙΑ ΜΕΣΣΗΝΗ ΙΣΤΟΡΙΑ ΜΝΗΜΕΙΑ ΑΝΘΡΩΠΟΙ*. Αθήνα: ΜΙΛΗΤΟΣ.
- [17] P.Themelis, 2010. ΠΡΟΪΣΤΟΡΙΑΚΑΙΙΣΤΟΡΙΑΤΗΣΑΡΧΑΙΑΣΜΕΣΣΗΝΗΣ. In: *ΑΡΧΑΙΑ ΜΕΣΣΗΝΗ ΙΣΤΟΡΙΑ ΜΝΗΜΕΙΑ ΑΝΘΡΩΠΟΙ*. ΑΘΗΝΑ: ΜΙΛΗΤΟΣ, p. 59.
- [18] P.Themelis, 2011. The Cult of Isis at Ancient Messene. In: L. B. &. R. Veymiers, ed. *Bibliotheca Isiaca*. Bordeaux: Ausonius Éditions, pp. 97-109
- [19] Papageorgiou, M., Zacharias, N. & Beltsios, K., 2009. TECHNOLOGICAL AND TYPOLOGICAL INVESTIGATION. ASSOCIATION INTERNATIONALE pour l'HISTOIRE du VERRE.
- [20] Verita, M., 2000. Tecniche di fabbricazione dei materiali musivi vitrei. Indagini chimiche o mineralogiche.. In: *M edieval Mosaics: Light, Color, Material.* Florence: s.n., p. 47–64.