STRUCTURAL FEATURES OF ARCHAEOLOGICAL POTTERY

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Abstract: Investigations of archaeological pottery artifacts using Scanning Electron Microscope (SEM) provide information on pore structure of fired samples of shred belonging to Cris 1 culture. From long ago, the ancient art has been faked. We present a scientific analysis of the pore structure of ancient ceramic samples in order to establish a method that allows us to differentiate between the true and false ancient ceramic sample. The use of the technique is illustrated on both lot of authentic and fakes specimens. Measurement of pore sizes and established the statistical pore distribution in wall thickness of ceramics are highlighted as research area useful in the administration and tracking down the traffic with objects of patrimony.

Keywords: archaeological pottery, SEM, pore distribution.

1. INTRODUCTION

From ancient time the ancient art has been faked. Today there are some fakes that look so convincingly real, that they can deceive even the most experienced eye. The exact manufacturing conditions used for ancient ceramics have been reproduced, including the temperature ranges and the exposure in the kiln. Porous ceramics are produced within a wide range of porosities and pore sizes depending on the application intended. Porosity and pore size distribution can be carefully controlled by the choice of organic composite and the amount added.

The raw materials used in pottery (clay, water, and fuel) are widely distributed over the surface of the Earth. Clay has the property of being plastic even wet and thus it can be turned into the desired shape; but as it dries it becomes hard and when sufficiently heated its shape becomes permanent and cannot be made plastic again by the addition of water. Once fired, pottery is very durable. Pottery may be broken into shreds but it does not rot away and generally, it is not recycled.

We try to demonstrate the potential of SEM technique for the characterization of ancient ceramics. This non-destructive analysis offers a means of getting information on the process and even sometimes on the date of ancient artifacts [1]. Finally, we explore the relationship between statistical pore distribution in wall thickness of ceramics and authenticity of ancient ceramic samples [2, 3].

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2. EXPERIMENTAL

Ceramic fragments belonging to Cris 1 culture have been examined. Shreds from the pottery vessels have been found at Barbosi, Adancata – Imas1 site in southeastern Moldavia (Romania). The ceramic shreds are small, fragmentary and not decorated. Two main types of pottery were analyzed: the first group of samples consisted of 'true' ancient ceramic certified by experienced antiquity specialists (symbol GA). The second group of pottery samples consisted of 'false' ceramic (symbol GF) that appeared similar to first group, but could not be positively identified through archaeological characteristics. It was assumed that these samples might be imitations. This classification is made by professional archaeologists given the complex relationships between the physical objects/contexts/circumstance encountered in excavation. The authenticity of ceramic shreds is proved by the information based on archaeological indicators: repertory, period, ethnic group/culture, material/technique, dating and discovery area.

The samples were analyzed using SEM technique. SEM does not actually view a true image of the specimen, but rather produces an electronic map of the specimen that is displayed on a cathode ray tube. SEM used in our measurements is Quanta 200 FEI type and characterizes both conductive and non-conductive samples. SEM permits to obtain surface detail and perform statistical analysis to more fully characterized particle and/or pore size and shape distributions (statistically relevant data about particle size and shape with ability to view the actual image of the statistical outliers). For spherical particles and/or pores, size is defined by the diameter. However, for irregularly shaped constituents, characterization of size must also include information on the type of diameter measured as well as information on constituent shape. The samples must be clean, dry, vacuum compatible and electrically conductive. To avoid the samples contain any volatile components such as water, this will need to be removed by a drying process. The size of the specimens was around 6 mm in diameter and they can move 50 mm in the X and Y directions. SEM provides the necessary required sample space for the large and irregular specimens to navigate without requiring mechanical adjustments.

Analysis of the true and fake samples revealed discrepancies between their porosity structure, indicating that they were manufactured through different techniques and/or at different locations, despite their apparent similarity.

The equivalent size of the pores is calculated considering the diameter of a circle of spherical pores with a radius r.

The SEM images are imported into the AutoCAD program, for digital scaling and measurement of the pores dimensions. For the pores analysis, at each sample have been made 50 digital measurements on a scattered randomly distribution in the sample picture field [4, 5].

3. ANALYSIS

The fourteen fragments of true ancient ceramic samples (GA) and seven fake ceramic samples (GF) were analyzed. Figure 1 contains eight SEM images of the ceramic samples. Figure 2 contains the Gauss probability distribution of pore sizes of the samples.

4. DISCUSSIONS

SEM has facilitated the study of ancient ceramics by providing more accurate, less time-consuming profiles of all types of pottery as well as new insights into pottery-making techniques. The SEM images of the first group of samples (true samples symbol GA) differ significantly from that of samples of the second group (false samples symbol GF) indicating different formation conditions.

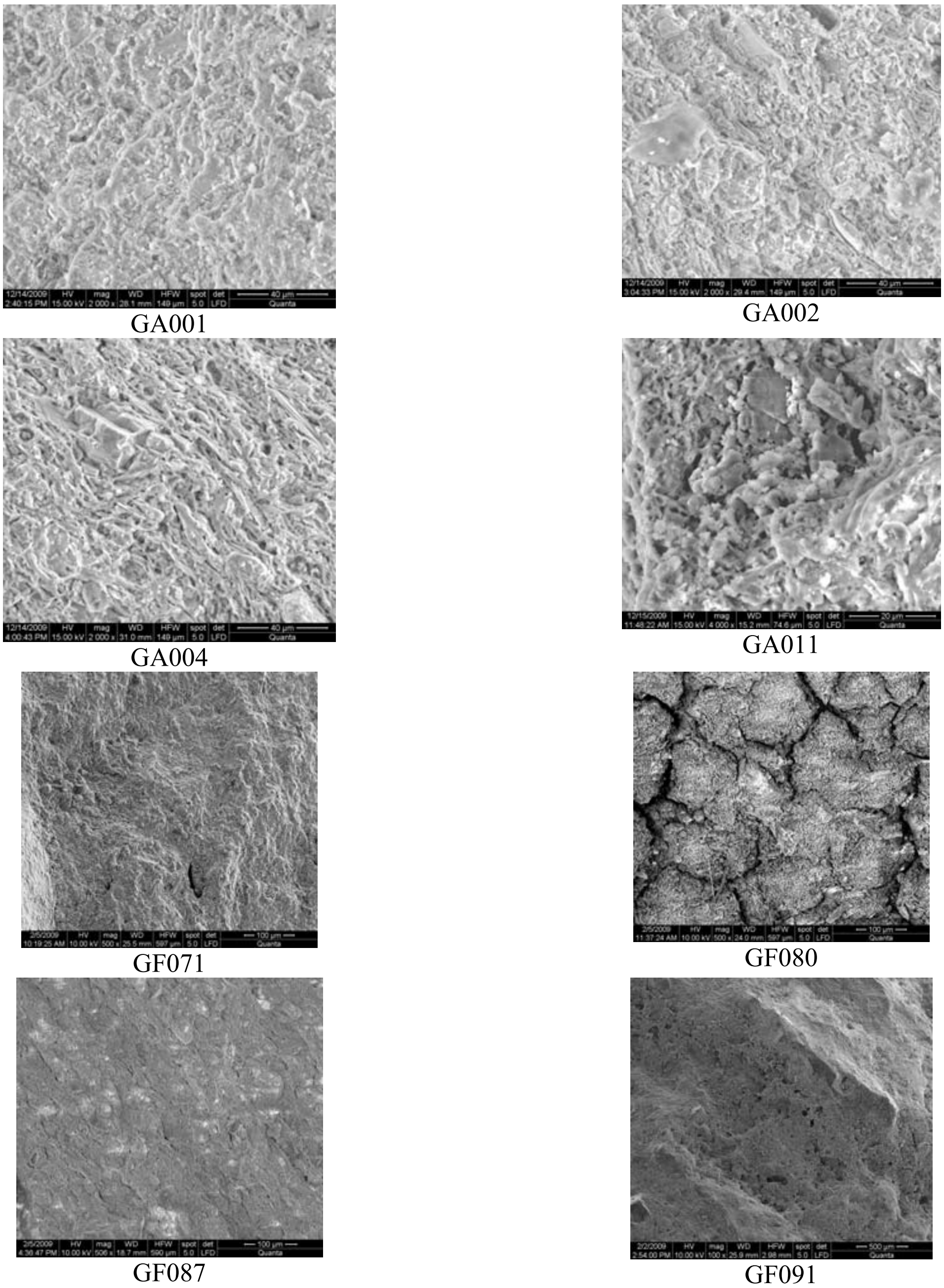

Fig. 1. SEM micrograph images of the true (GA) and false (GF) ceramic samples.

The SEM of true samples GA shows that the particles are irregularly shaped and varied in sizes. As it is seen in GA002, GA004 and GA011 the surface of the pottery comprises of volumetric plate like grains with a different crystallite sizes ranging from 20 μm to 40 μm. It allows interpreting that they were fired relatively at low temperature [6]. The examinations of micrographs of true samples reveal that the particles were heterogeneously shaped with horizontally aligned pores which may be attributed to coiling of pottery [7]. An encompassing distribution of pores structure in true sample reveals that this specimen was fired in multi-step technology at relatively high temperature [8, 9]. The fake samples present a different microstructure. The presence of small size pores with diameter of 1,2 to 4 μm indicates that the samples might have been fired at temperature below 1050ºC. According to Maniatis and Tite, the size of pores would become 10–50 μm around 1050–1100ºC [10].

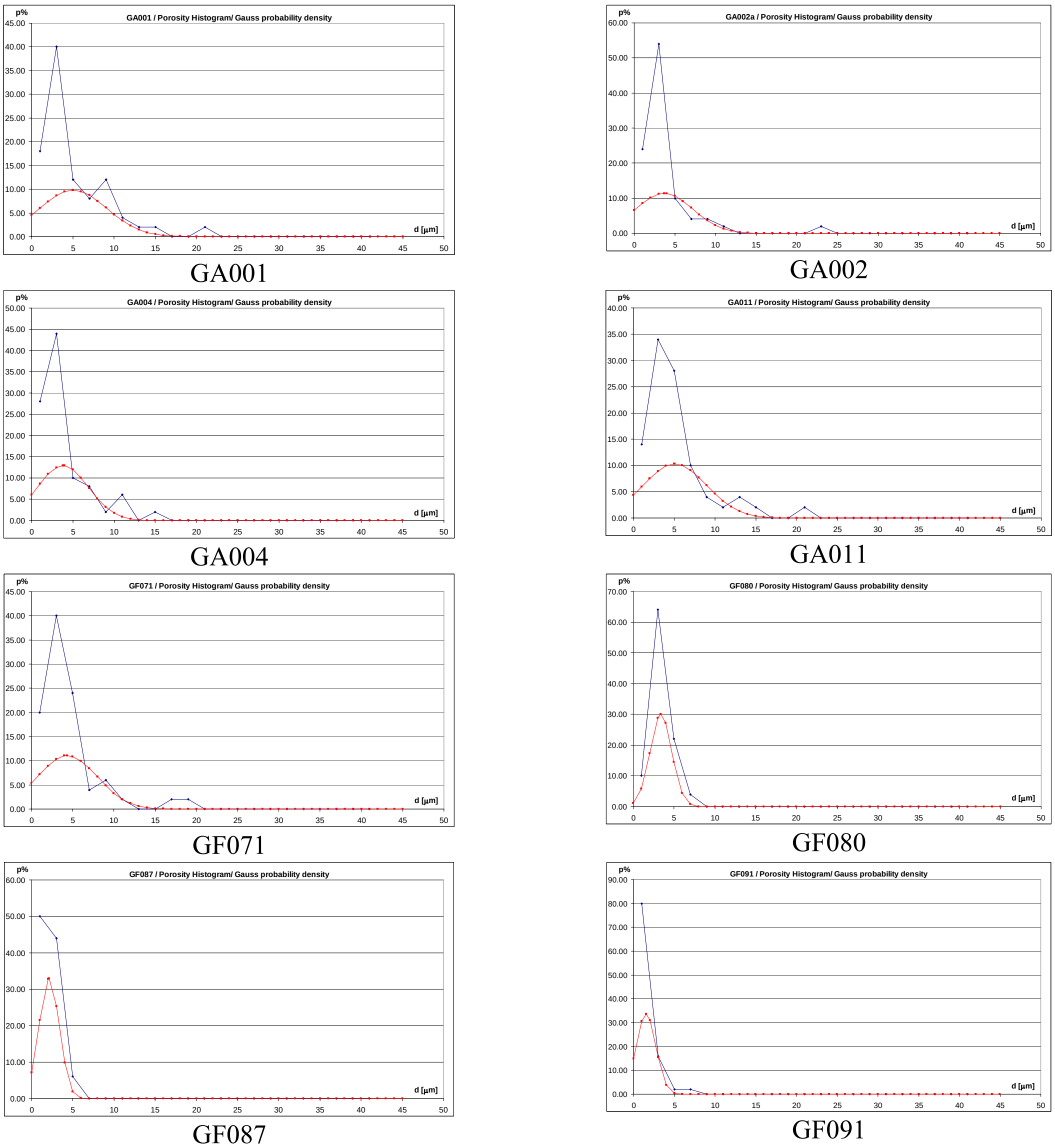

Fig. 2. The Gauss normal distribution refer to pores sizes.

SEM analysis reveals air bubbles in the walls, a finding that indicates poor handling of the clay. SEM profiles make it possible to test the theoretic explanation for pottery-making techniques. This can be carried out with each individual piece of ceramic.

Figure 1 shows SEM images of the microstructure of the porous ceramic samples. These experimental data were evaluated considering size distribution of the interconnected pores. It can be observed that the pores show circle shapes. The true ancient ceramic samples present some bigger pores that indicate poor handling of the clay. Bigger interconnected pores are observed due to release of gases formed during burning of organic material. The false ancient ceramic samples present smaller pores and sample number GF080 shows some kind a needle shape pore that indicate a better handling and burning of the clay.

When considering the Gauss probability distribution of group GF each of the samples present different shapes of distribution curves of pore sizes in comparison with those of first group GA. Except one sample (GF-71, see Figure 1 and 2) all of the other samples can be identified successfully with regard to their authenticity by their porosity, pore sizes and distribution of pore sizes. This means that the groups of samples we have studied have different porosity statistically, which are determined by their manufacture materials and/or batch composition.
Table 1 presents the statistical mean value and standard deviation for the pore dimensions, based on SEM analysis [4, 5], cumulated for all the „true" and „false" ceramic samples, as presented in Figures 1 and 2.

Table 1. The statistical mean value and standard deviation for the pore dimensions, based on SEM analysis (Figures 1-4), for all the „true" and „false" ceramic samples (sampling resolution 0.2 μm).

Sample	GA001	GA002	GA004	GA011	**GA**
Mean (μm)	5.03	3.68	3.85	5.15	**4.92**
Std.dev(μm)	4.06	2.75	3.01	3.79	**3.82**

Sample	GF071	GF080	GF087	GF091
Mean (μm)	4.31	3.30	2.26	1.60
Std.dev(μm)	3.50	1.17	0.89	0.97

In Figures 3a and 4a are presented the pores dimension histogram cumulated for all the „true" and „false" ceramic samples.

In Figures 3b and 4b are presented the pores dimension Gauss normal distributions cumulated for all the „true" and „false" ceramic samples.

In the case of „true" ceramic samples, the pores dimension distribution is extended on a wide significant values range 1 – 40 µm.

In the case of „false" ceramic samples, the pores dimension distribution is grouped around a narrow significant values range 1 – 15 µm.

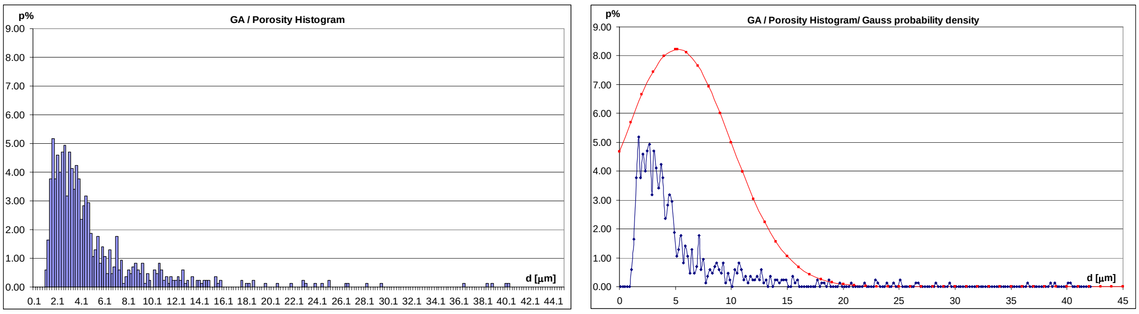

Fig. 3.a. The pores dimension histogram cumulated for all the „true" ceramic samples.

Fig. 3.b. The pores dimension Gauss normal distribution cumulated for all the „true" samples.

Fig. 3. The pores dimension histogram cumulated for „true" ceramic samples.

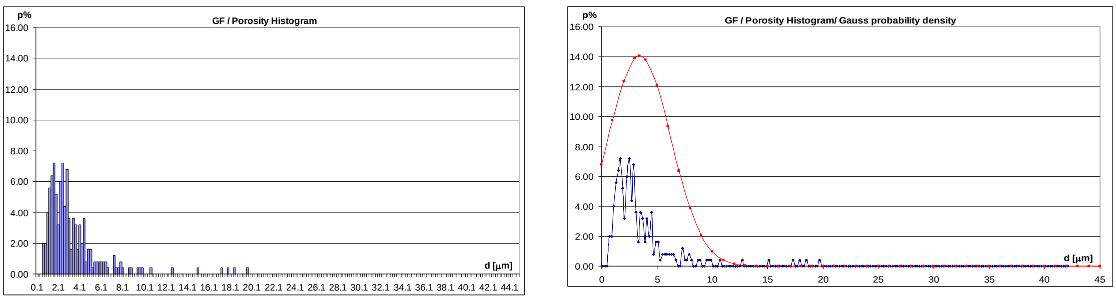

Fig. 4.a. The pores dimension histogram cumulated for all the „false" ceramic samples.

Fig. 4.b. The pores dimension Gauss normal distribution cumulated for all the „false" samples.

Fig. 4. The pores dimension histogram cumulated for „false" ceramic samples.

5. CONCLUSIONS

SEM has facilitated the study of ancient ceramics by providing more accurate, less time-consuming profiles of all types of pottery as well as new insights into pottery-making techniques. SEM profiles make it possible to test the theoretic explanation for pottery-making techniques. This can be carried out with each individual piece of ceramic.

We demonstrate the potential of SEM technique for the characterization of ancient ceramics. This non-destructive analysis offers a way to get information on the process and even sometimes on the date of ancient artifacts.

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