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Characterization of Ancient Ceramic Matrices with High Resolution Microscopy Methods

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1. Introduction

Potteries, ceramics and their production methods in the ancient world are one of the most important items for researching about the cultural heritage materials (Riederer, 1994). The processing of material being used for making ceramics and mineralogical investigations for understanding the production process, mainly firing, are among the most important goals of the present investigations.

Scientific studies on archaeological ceramics, based on laboratory methods carried out over decades and are known as ceramography (All-Saad, 2002). Studying the old objects give not only substantial information about the manufacturing in the past but also provides information about the processing of structures in such primitive materials (Letsch & Noll, 1983). Recognition of the evolution of ceramics is among the most important questions that has attracted the focus of archaeological researches. Apart from them, these ceramics being one of the oldest industries and arts, has become nowadays as one of the most modern research sciences for human being. In archaeological researches is also important to know about the procedure of manufacturing (in this case ceramics) in the daily life of nations (Fazeli et al, 2001). Beside archaeological investigations, cramography methods are useful and suitable ways for classification ancient ceramics and could help archaeologists to classify them with high accuracy. This investigation resulted in more accurate classification of some ceramics found in Persepolis World Heritage Site and showed high quality manufacturing process which used to make these ceramics (Maggetti, 1982).

The great consideration on these kinds of ceramics is due to their variation of raw materials which have been used for manufacturing, and additionally ceramics from Achaemenian period are recognized as transfer materials between two considerable dynasties in Iran, namely Elamite to Achaemenian. This transfer is marked as transfer through out of evidences and prominently also technical evidences.

To characterization ceramic matrices, some samples of ancient ceramic pieces from Persepolis (Takht-e Djamshid) world heritage site were selected. These samples were examined with Atomic Force microscopy (AFM-PFM) (Emami et al, 2008 & Maggetti, 1982).

The experiments get information about the topography of ceramic's surface and generating of the surface. Also grain boundaries were recognized and AFM pictures gave sufficient information on sintering around a grain and the spreading of this process from the grain boundary into the matrix of the ceramic. The sintering has occurred complimentary well in these samples. To characterization of the raw materials that used for making of these ceramics, QXRD method is recognized. The calculated and measured peaks are refined by Ritveld refining methods to realize about the crystalline structures. According to XRD and polarization microscopy studies the research materials can be divided in one main group considering their matrix structure. This group proves siliceous matrix which mainly consist of SiO₂ and Pyroxene component which show us the stability of the pyroxene solid solution in the area of 750 - 870°C. Calcareous matrices which mainly consist of CaO, CaCO₃ and a minor content of SiO₂ are also observed as non complimentary sintered ceramics.

In fact by using of chemical-mineralogical methods, one seeks is to find the using technique of crude material in the past by means of laboratory methods and not merely through comparative comparison of the potteries of a particular investigated area. Investigations based on material characterization also let to creating a technical chronology with respect to the technique that practised in each area.

2. Experimental procedure

For chemical- mineralogical investigation and characterization of the ceramic matrices, there are different disciplines which used in the following chapters. These methods and the aim of each method describe schematically in the Figure 5.

The main purpose of this investigation's discipline is to pay attention to the textural formation of the material as well as finally sinter structure of traditional ceramics based on mineralogical- chemical properties in such system.

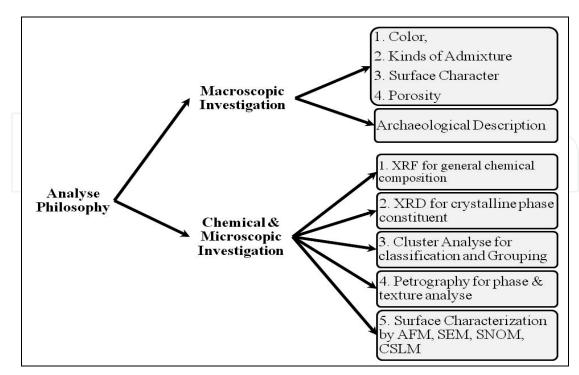


Fig. 5. Analyze discipline for ceramic investigation

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3. Sample descriptions

3.1 Macroscopic investigation

The shape, typology and the color of the objects are drastically variable and can be grouped as follows:

- 1. Potteries without paintings, having a very single form, the color of which being red, green or buff.
- 2. Yellow, bright brown potteries having very fine designs in the form of parallel lines.

3. Potteries with simple forms and paintings produced by exerting pressure on the body. In general potteries in the region are mostly produced by clay, soil and earthen material. The color of these potteries is classified as bright, having very low porosity, and for the same reason, have a rather high toughness. The color of some samples varies from the corn to the surface because of uncontrolling firing temperature (Fig. 1). In many samples typical admixtures are visible on the texture of the ceramics (Fig. 2). They could be the remains of





Fig. 1. Ceramic with different colors in the matrix as a function of uncontrolling firing.

Fig. 2. A part of a Vase structure varies from the surface to the core and also different admixtures



Fig. 3. Sandwich structure with different matrix texture in ceramics.

Fig. 4. Salt recrystallization on the surface through capillary effect of high porosity ceramics.

organic materials such as hairs, straws. In some cases the sandwich- and glassy structure are also noticeable (Fig. 3). These kinds of ceramics consist of three layers, the outer layers are reddish and the inner part of the ceramic structure is dark. These ceramics have glassy echo by knocking on their surface. The corrosion effects and the salt recrystallization on the surface of the ceramics are mostly observed. These could occur as a function of high porosity of ceramic's body which is very important for traditional ceramics (Fig. 4).

Their typology mostly includes long mouths and in some cases spherical shapes. In addition to these, there exist some bowls with a flat bottom and bevelled rims.

3.2 Chemical- mineralogical investigations3.2.1 Chemical composition of potteries

Identification of the chemical composition of samples has been carried out by X-Ray Fluorescence Analyses (XRF). The aim is to find the correct chemical formula and combination of the samples which could also obtained an statistical distribution of elements that consist the pottery's matrix in system CaO+MgO – SiO₂ – Al₂O₃ (Noll, 1991). In later phases, some complementary methods will be used for grouping the materials. Among them one can mention by the "Scanning Electron Microscope" methods.

The chemical composition of all the samples is given in Fig. 6. From the whole range of samples, there are only 6 sub-samples that will be discussed in this paper. From the chemical standpoint, the samples are characterized by high SiO₂ and approximately amount of CaO contents. Another important composition in these samples is Al₂O₃. The ceramic's matrix is classified in general into two groups; the samples of group 1 are defined as sandy matrix, and the second group is defined as a calcareous matrix. The origins of these components depend on the raw materials which were used for manufacturing. Sand is the most common raw materials that make up the main clay mineral deposits in this area. The application of quartz had two considerable advantages with respect to its physical and chemical properties;

Firstly, Quartz is a hard mineral (H=7 on the Mohs scale), secondly, Quartz shows a high physico-chemical resistance against acidic conditions (acidic solution or acidic atmosphere). Indeed, here is another point that made quartz unfavourable for such procedures. Quartz changes its structure three times (decomposition) while reaching its melting point, and after this point it will cross over to the glass form. This could to be a hypothesis, why have the oldest ceramics still good texture as well as body for research purposes, and why optically they seem to be glassy (with uncontrolled temperature in the open furnace) (Emami et al, 2009). Quartz rich wares create a glassy rattle by the knocking on their surface. Perhaps this might be a way to distinguish quantitatively the age difference between some ceramic wares. The existence of carbonate could have three reasons. Firstly, the carbonate content seems to originate from calcareous stone, such as calcite and dolomite that contain the main geological formation on the field; secondly, the calc produced as a secondary phase through the firing process as a result of contamination of ceramic fragments (Ca²⁺, Fe²⁺, Mg²⁺) and third, calc is used in the ancient time as flux.

XRF results proved that these kinds of ceramics are SiO_2 rich ceramics (Fig. 6) and there exist large distinction between them and the ceramics from south-west Iran (Emami et al, 2008). Their matrices are glassy and consist of many crystal fragments that have a magmatic origin. In some samples there exist also sandwich structures in the ceramics as layering structure with different colors. The inner layer is darker than the other and it comes through high SiO₂ content (Emami et al, 2009).

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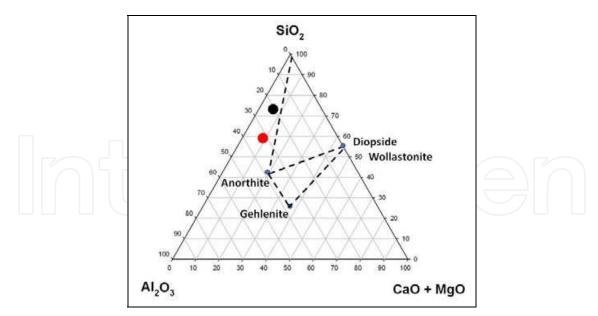


Fig. 6. Classification of two ceramic groups from Persepolis in system $Al_2O_3 - SiO_2 - CaO + MgO$. According to this classification the ceramics fit in to the high $SiO_2 - Al_2O_3$ category.

3.2.2 Identification of crystalline phases in pottery's texture with Rietveld refining method

XRD analysis carried out for identifying Mineralogical phases in the ceramic matrices. To gain a high-quality interpretation from X-Ray Diffraction and the refining of the results according to the Rietveld Method, the chemical and mineralogical investigations and phase analyses of ceramic samples carried out qualitatively and either quantitatively. The Rietveld method allows the option of classification of phase identification and compensates for line broadening induced by particle size effects. This method of research using Powder Diffraction was recorded by the International Union of Crystallography Commission (Mccusker et al, 1999). The topics from these kinds of refinements are as follows; (i) classification of data, (ii) peak shape description, (iii) background determination, (iv) Fourier analysis and (v) refinement of profile parameter (Mccusker et al, 1999). In this application, the results will be presented and discussed based on the final calculated diffractogram. For these samples, the X-ray generator operated with a copper target at 45kV and 40mA. The powder pattern of ceramics was measured between $2\theta = 3^{\circ}$ and $2\theta = 61.80^{\circ}$, using a scanning rate $2\theta = 0.01^{\circ}$ per minute. This large scanning area was required because the important clay minerals, such as illite, kaolinite and layer silicates such as biotite and muscovite, show the major peak intensity over these ranges. On the other hand, some phases, such as quartz and calcite, that, indeed, play a large role in our research, show the typical peaks for matching their identity between $2\theta = 51^{\circ}$ to $2\theta = 58^{\circ}$. The samples showed the minimum intensity of 5 - 58 counts, and a maximum intensity between 1029 - 2828 counts. These differences occurred as the origin of materials and the methods for pottery production were different (Fig. 7).

Quartz, calcite and some chain silicates, such as pyroxenes, are the major minerals which are contained in the ceramic bodies. Feldspars, plagioclases and some modification of melilite groups are the minor phases in the ceramics. Micas and clays minerals in different modifications, and some other minerals, such as nepheline, are present as trace phases, which are calculated in these materials. Quartz appeared mainly as trigonal/rhombohedral in form of α - quartz. This structure of quartz is stable at low temperatures under 573°C, and

the best crystallographic preferred orientation appeared at d [Å] = 3,34 (101), 4,26 (100) and 1,54 (211). The last peak is the important strong line of quartz because it does not overlap with another mineral phases, such as calcite, analcim or orthoclase. Over these ranges and with this strong line, it was possible to identify the quartz exactly. In many cases, Qz + Mus or either Qz + Ana showed a sharp overlapping over the main peaks at $2\theta = 26.64$. For using the Rietveld method in order to obtain a better quality of fit in the calculation such, it is better to refine other phases such as muscovite or analcim, with quartz. Calcite appeared with the best orientation at d [Å] = 3,04 (100), 2,28 (111) and 1,92 (011) and $2\theta = 29,2^{\circ} \& 39,4^{\circ} \& 47,0^{\circ}$ (Emami et al, 2008). The next important group within these materials are the chain silicates, such as pyroxenes. Miscibility gaps of pyroxenes in general were apparent between calcic-clinopyroxenes and pigeonite, and therefore an intermediate composition may be possible at high temperature (Emami et al, 2009).

Extensive solid solution is possible among members of the calcic, calcic-sodic and sodic pyroxenes. The best preferred orientation is over $2\theta = 29-31^{\circ}$ and $34,5-36,5^{\circ}$ (Emami et al, 2008). The next important calculated phases are the framework silicates, the so called feldspars & plagioclases. These phases appeared in all miscibility formations. The main minerals in these groups are alkali feldspars, such as Orthoclase, sanidine & microcline. The best-preferred orientation of plagioclases occurred between Qz and Cc. The greatest intensity of these groups of minerals occurs between $2\theta = 27-30^{\circ}$ and $2\theta = 35-40^{\circ}$. In our case study, gehlenite (Ca₂[Al₂SiO₇] as a common minerals from melilite miscibility appeared, and this structure required substitution at some compositions as MgSi₂ \leftrightarrow Al, AlSi to MgSi₂ \leftrightarrow Si, AlAl.

According to XRD studies, and with respect to available phases, the potteries from Persepolis can be divided in two groups as follow:

Group I, Mostly consist from quartz and granitic fragments; among them one can specify as Muscovite and Microcline. This kinds of samples includes % 54,16 of the samples. Other important phases in this texture are magnetite, hematite, enstatite, diopside, anorthite, orthoclase and nepheline. Hematite and magnetite are the high temperature phases in the traditional ceramic technology (Maggetti & Galetti, 1982). Enstatite and diopside are the secondary phases that occurred during firing

Group II., This group in general includes Calcite and Quartz. The chemical composition of this group of potteries varies drastically depending on pyroxene, muscovite and plagioclase. Stone fragments and calcareous fragments also exist in this group which is an evidence for different techniques in pottery making. This group includes some % 37,5 of the whole. In general it should be concentrate that Pyroxene is more abundant in potteries which are an evidence for the use of different admixtures in the soil of that region due to the variation in available quarries of the field. The variation of the crystalline phases in these ceramic textures based on XRD-Patterns demonstrate on Fig. 8.

3.2.3 Petrological and petrographical investigation through polarized light microscopy

The aim of "thin width samples" studies is to confirm the existence of identified phases through XRD method as well as relation between these phases and surrounded matrix from mineralogical point of view. Of greatest importance for these studies, is to investigate the binding process on the grain boundaries that accomplished the structure of the pottery at least as well as determine the kind of used temper and pasta. These kinds of phases and structures within them they reveal themselves in the background structure are twining, color, corona at the boundary of grains, double refraction of light, crystal network and

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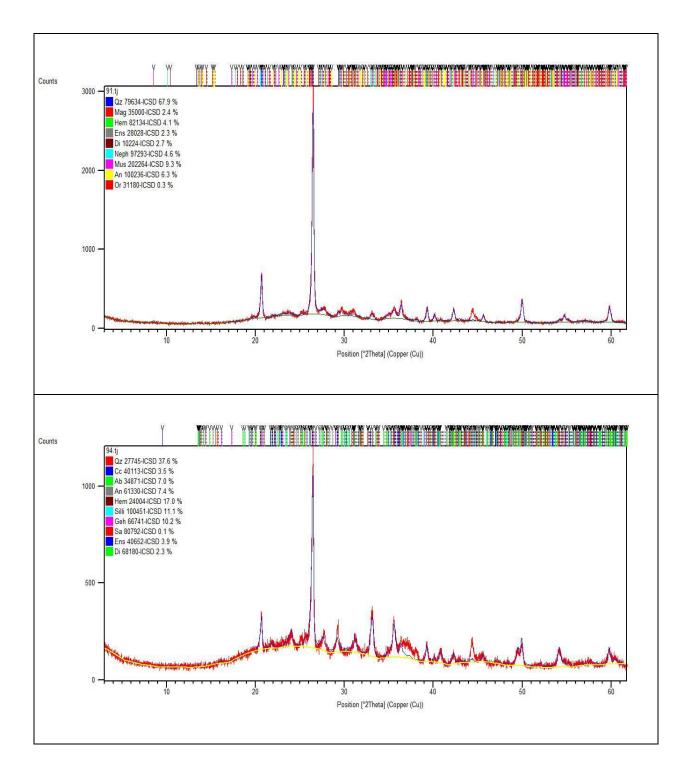
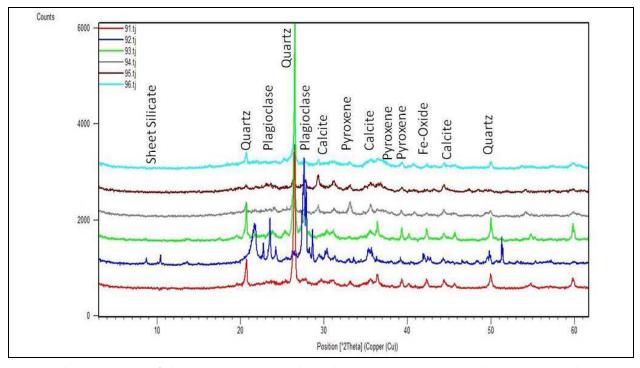
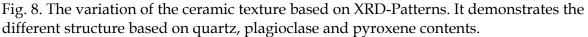


Fig. 7. XRD Patterns from two different samples from Persepolis after selective Dissolutions of Phases.

Qz = Quartz, Cc = Calcite, Di = Diopside, En = Enstatite, Ab = Albite, An = Anorthite, Or = Orthoclase, Sa = Sanidine, Mag = Magnetite, Hem = Hematite, Neph = Nepheline, Mus = Muscovite, Sili = Silimanite & Geh = Gehlenite





- 1. *Quartz;* which in most cases represent itself in the form of spherical as well as edge aggregates with different sizes. Structure of quartz with their specific "dark angel" is observed as undulatory extinction (Fig. 9). Quartz is seen in many cases together with Ferric ores. Potteries consist of abundant amount of quartz in their structure proved dark color than the others. The color becomes more inclined towards to reddish according to heating range and also kiln atmosphere.
- 2. *Calcite;* is observed in all samples and demonstrates different specification from optical aspect. Investigations have shown that calcite reveals itself in spherical or broken form. Collectively with calcite there is aspect of ferric ore in form of hematite layers (Fig. 10). This layer seems to be rebuilt from MgO, FeO or other secondary oxidation products. Calcite is among phase which is observed in the pottery's matrix and determined also the region's soil conditions as well as alteration.
- 3. *Feldspar*; appear in different modification as a function of unlike conditions; and for the same reason, reveal their phase in different structures. Feldspars appear mainly in form of albit as well as serisite-albit, or as plagioclase with microlcline modification. One can identify these structures through the specific twining structures or through detecting light direction in them. Plagioclase has seen less frequently in the pottery's structure; while serisite has been seen much more frequently as alteration products of albite (Fig. 11, 12). In addition to mentioned ores, some other phases observed also in pottery's textures; among them biotite and pyroxene (Fig. 13) are the typical phases in high temperature range. According to the microscopic observation these phases don't categorised as primary phases in pottery's structure. The structure of consist phases and sub-minerals get information about the authenticity of the soil used for pottery making. According to the microscopic investigations the soil that used for pottering has the same origin.

- 4. The group of clay minerals are among the most important constituents in the ceramic texture. The clay minerals provide their structure twice, firstly by addition of water to clay and secondly come through firing as secondary phases in the texture. Apart from them, one should consider that the size of the clay minerals is not coarse enough for discovering their structures with light microscopy. These minerals studied mainly through scanning electron microscopy as well as atomic force microscopy.
- 5. Admixtures that added to the clay source for pottering mainly consist of organic shale as well as straw and agricultural remains from the surrounding environments. In many cases fossils are the fingerprint for characterization of the soils (Fig. 14). The qualities of material as well as the unique similarities between these soil reservoirs nearby Persepolis and those which were used for pottering suggest them to be one of the sources which used for pottering in Persepolis complex.

3.2.4 Application of SEM investigation on interpretation of sinter structure

SEM investigation and observation lead to find some interesting results about the mechanical failures. In traditional ceramics, sintering occurs around 1100 - 1200°C, depends on the raw materials as well as the firing atmosphere. Sinter structure is mainly a compact structure with high porosity effect. The constituent minerals change their textures during sinter and followed by don't looked like the origin shape. One of the most evidently fractures, are the damaging effect on the surface of investigates materials.

Effectively, the origin of the fracture could be traced to the defect in the structure of the ceramics through the pottery manufacturing process. The cracks pass from their origin toward to the other side of the samples surface. The cracks could be classified as a capillary cracks, which are very fine and can be defined as microcracks, or shrinkage cracks, which happened by changing the physical- or thermodynamically coefficients of the surroundings environments.

According to Fig. 15, the structure of Aluminosilicates observed that reacted with the environment. Such disruption of aluminosilicate suggests that these kinds of minerals tend to change its structure easily. In this example the Matrix is mainly consist of lime or clay-rich raw material (Maggetti & Schwab, 1982). Alkaifeldspars with typical crossed cleavage are also observed as the high temperature phases in the quartz-rich matrix (Fig. 16) (Mathé, 2007). The bright structures suggest probably salt recrystallization on the surface of crystal. The conversion of quartz- α (low-quartz) to quartz- β (high-quartz) will take place with 5% volume expansion namely; quartz splitting. For the reason that this operation occur in a very short period, such cracks increased in the mainly part of the ceramics (Fig. 17).

The cracks appeared in a material with high density. Such cracks on the surface of collected grains could happen only after changing the ambient decomposition. In the middle of the samples, there are tiny cracks which proceed and propagate themselves from diagonally from the bottom to the top left hand on the surface. These cracks could be identified as secondary cracks, which appeared around the grain after weathering or diffusion (Fig. 18).

As a matter of fact, during the firing and burning process the quartz rich matrix and the calcareous matrix show different damage models. In the calcareous matrix, deformations normally increase over a large area on the samples and the cracks are wide enough to have secondary reactions among them. According to figure 18 in argillaceous and quartz rich matrices, the cracks normally are found on the quartz grain and demonstrate internal extension during the decomposition of α -quartz to β -quartz. In many cases, the crack's opening accomplish if another aggregate dropped out from the matrix.

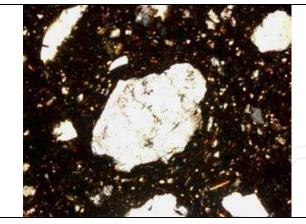


Fig. 9. Quartz with different size and typical undulatory extinction. In the matrixconsist of stone fragment. x 10. Pol + Ana.

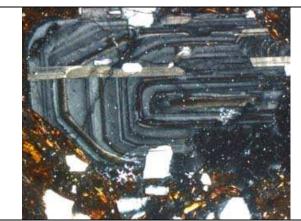


Fig. 11. Albit with typical zoneing structure as myrmekite texture. This structure appeared through rebuild of radiactive elements in the interatomic places of crystal structure. x 10. Pol +Ana.

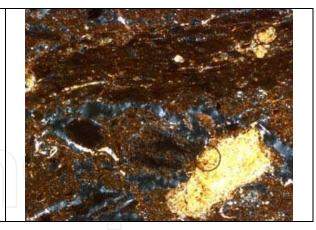


Fig. 10. Calcareous matrix consist of spherical calcitic aggregate sarounded with Ferro- material. x 5. Pol + Ana.



Fig. 12. Sericitization in Albit as a fine lamel structure. In a matrix fron stone fragment quartz is seen too. x 20. Pol +Ana.

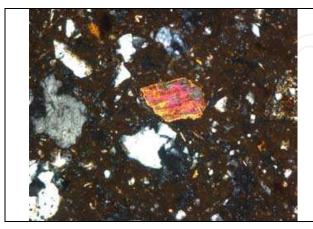


Fig. 13. Biotite as mineral aggregate whit characteristic red color. x 20. Pol +Ana.

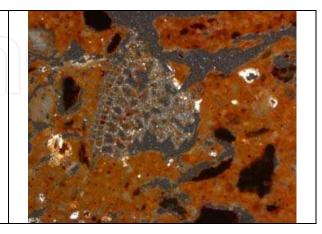


Fig. 14. Characteristic high Ca-matrix with organic admixture as fossils shale that comes through the soils. x 20. Pol +Ana.

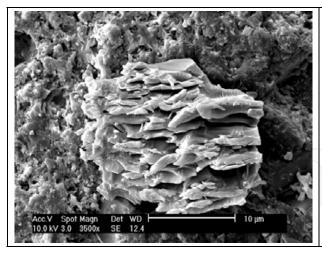


Fig. 15. Decomposition of aluminosilicate in a quartz rich matrix through high sintering

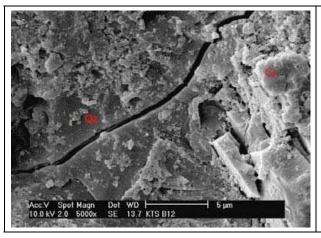


Fig. 17. Quartz splitting by 550°C and the primary cracks that happen during firing process.

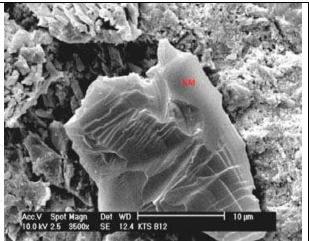


Fig. 16. Alkalifeldspar with typical twinning structure with calc as well as salt recrystallization.

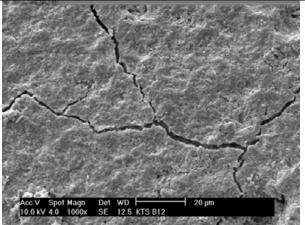


Fig. 18. Secondary cracks on the surface of the ceramic's matrix as a function of firing process.

3.2.5 Confocal and atomic force microscopy

Confocal as well as AFM microscopy methods divided us to find out about the characterization and to obtain some physical and topographical properties of the surface. The homogeneous or inhomogeneous of the material's surface, and the frequent distribution of such elements such as Fe, Mg, Ca, can give us a result about the discolouring of the ceramic bodies and, on the other hand, the roughness and therefore potential diffusions under the microscope (Emami et al, 2008).

Ceramic texture been used to characterize many aspects of atomic and molecular arrangements. Confocal microscopy allows one to show the properties of the surface and textures of the materials, which includes a three-dimensional reconstruction of the specimen's surface and fractures over large time scales. The ceramic surface consists of a solid state with many phases in equilibrium. The structure and hardness of the surface and the character of mineral boundaries depend strongly on the type of the particles and their physical properties. Cracks, which happened through shearing forces or internal shrinkage or external forces, could be a reason for deformation and damage. Such microscopic observation helps one to connect the surface dynamics to the macroscopic damage behaviour. By use of the AFM method, it is be possible to measure the interaction between a solid surface and extremely fine pike (Emami et al, 2009). In general, this involves scanning an area in non-connected mode, which is carried out by a kind of sensor called a cantilever. In this case, a laser beam is focused on the cantilever and reflected to the detector. The detector measures the deflection the cantilever and, in the following, of the surface. To observe the cracks and deformation areas, it is important to have a fresh break on the surface and, for this reason, it is better to have a piece or sample instead of the thin sections. Physical and mechanical interferences on the surface have been interpreted by AFM method through topographic and phase images (Fig. 19).

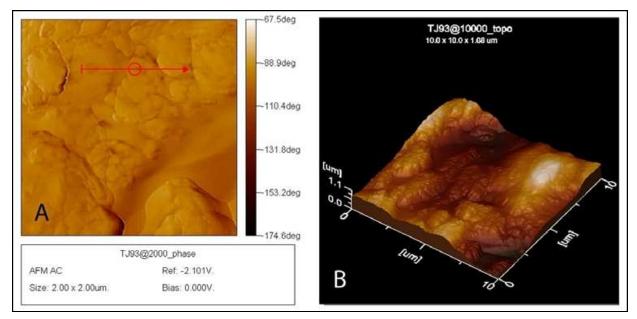


Fig. 19. AFM image of the topography on the surface in Non Contact Mode. A: is the phase image that illustrate a homogeneous matrix depends on candilevel flections factor. B: Topography image and the born of cracks on the surface of grains.

4. Results and discussion

The chemical analysis of investigated materials concludes that the materials respond as high quartz (argilleous), materials. The physicochemical properties of their materials depend on the components included in them. The chemical components also control the kinds of weathering as well as damaging process. There are many important factors during the firing process of ceramics that have influences on the reaction conditions; increasing and decreasing the temperature range and the chemical composition of the gases in the kiln. The twin-oxidation reaction in manufacturing of potteries ($C/CO/CO_2 \& Fe_2O_3/Fe_3O_4$) will proceed under high oxygen fugacity and a wide range of temperature. According to these reactions, it is necessary to consider that firstly, water delivered from the system and, based on the free lime constituents the CO_2 concentration will increase in the atmosphere of kiln. These two reactions suggest to the dehydration of clay materials or dissociation of carbonates.

Apart from them, the ceramics that have a high amount of quartz, demonstrated a higher damaging process rather than the materials which contain a calcium or magnesium rich matrix. The ceramics with a high content of Mg proved refractory properties in their structure.

The effect of different carbonate decompositions tend to understanding about the pottery production process. The best wares and products were those wares which were fired and burned rapidly, and cooled very slowly. These procedures took place in closed kilns with exactly controlled temperature during the manufacturing process.

5. Conclusion

Analytical chemistry and petrological-mineralogical investigation as well as microscopy methods, such as polarization, scanning electron microscopy (SEM), confocal laser scanning microscopy (CLSM) and atomic force microscopy (AFM) have a great role on the study about ancient materials as interdisciplinary subjects.

Apart from them prominent damage features have been reliably identified and characterized, showing that mineralogical- chemical investigations can involve other techniques in archaeology in form of experimental archaeology or archaeometry. In general, phase analytical methods (XRD, and the refinement with the Rietveld methods as complimentary methods) showed meaningful results to explain as well as illustrate ancient technologies. Microscopic methods are complementary studies to reinforce the interpretations and observations.

The varieties of experiments and the reproducibility of the results make these analyses as good fundamental choice for distinguishing and explaining questions in archaeological problems as well as material science discipline.

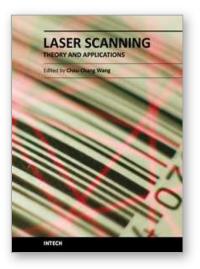
Research methods regarding to the archaeological feature based on the technical and scientifically interpretations are interdisciplinary way to connect gaps between archaeology and material science.

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Laser Scanning, Theory and Applications Edited by Prof. Chau-Chang Wang

ISBN 978-953-307-205-0 Hard cover, 566 pages Publisher InTech Published online 26, April, 2011 Published in print edition April, 2011

Ever since the invention of laser by Schawlow and Townes in 1958, various innovative ideas of laser-based applications emerge very year. At the same time, scientists and engineers keep on improving laser's power density, size, and cost which patch up the gap between theories and implementations. More importantly, our everyday life is changed and influenced by lasers even though we may not be fully aware of its existence. For example, it is there in cross-continent phone calls, price tag scanning in supermarkets, pointers in the classrooms, printers in the offices, accurate metal cutting in machine shops, etc. In this volume, we focus the recent developments related to laser scanning, a very powerful technique used in features detection and measurement. We invited researchers who do fundamental works in laser scanning theories or apply the principles of laser scanning to tackle problems encountered in medicine, geodesic survey, biology and archaeology. Twenty-eight chapters contributed by authors around the world to constitute this comprehensive book.

How to reference

In order to correctly reference this scholarly work, feel free to copy and paste the following:

S. Mohammadamin Emami, Johannes Volkmar and Reinhard Trettin (2011). Characterization of Ancient Ceramic Matrices with High Resolution Microscopy Methods, Laser Scanning, Theory and Applications, Prof. Chau-Chang Wang (Ed.), ISBN: 978-953-307-205-0, InTech, Available from:

http://www.intechopen.com/books/laser-scanning-theory-and-applications/characterization-of-ancient-ceramic-matrices-with-high-resolution-microscopy-methods



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