

# A NEW METHOD FOR STUDYING POTTERY? EXPLORING THE SCANNER TECHNOLOGY

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*Los análisis arqueométricos en cerámica suelen ser destructivos y costosos. En este artículo se propone un método alternativo basado en el escáner y el uso de imágenes 2D y 3D. Los resultados exploratorios obtenidos con fragmentos del Neolítico del Próximo Oriente son alentadores. Esta técnica permitirá comparar y superar los métodos utilizados por la arqueología tradicional.*

Arqueometría, Escáner, Imágenes, Cerámica, Próximo Oriente

*Archaeometric researches on ceramics are often destructive and costly. We explore here an alternative method based on scanner technology with 2 and 3D imaging. Exploratory results, obtained with Late Neolithic sherds from the Near East, are encouraging. This technique will allow and overcome the methods used by traditional archaeology.*

Archaeometry, Scanner, Image, Pottery, Near East.

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## INTRODUCTION

It is well known that pottery sherds are amongst the most numerous and durable remains found on archaeological sites. Ceramic is, with flint, one of the most studied artifacts of archaeology. This peculiar situation is related with the hardness of the material when fired, which hardly disappeared, and with the place of such artifacts in the daily life. Pottery is used for cooking, storage, but is not restricted to pots; it is also used for figurines or architectural elements such as tiles. Their basic material is clay. Found in the nature, clay is heterogeneous but has to be evenly mixed with other elements such as temper (vegetal or mineral) and water to be used as a raw material. Ceramic was one of the first

synthetic materials to be made by human communities in the past.

The observation of pottery in archaeology brings important information about technical knowledge (preparing the clay, firing temperature linked with the use of specific ovens sometimes) and decoration (style, nature of the motives, etc.). The classification of sherds or pots coming from stratified contexts allows us to interpret and assign dates. The contexts in which sherds are found give us an idea on the functions and uses of the pots and objects. However, if we want to go further, archaeometric researches are necessary. These analyses, which involve high technology and skilled scientific knowledge, are often expensive and destructive.

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New methods, based on scanning technology, are now available, making possible to work on images rather than on objects. For instance, a new method based on scanning technology, using an X-ray computed tomography scanner was tested successfully in the Mus e du Quai Branly by Christophe Moulh rat on Peruvian mummies<sup>1</sup>. New imaging technologies made possible to see through the objects, without destroying them, in other words, to make a virtual “autopsy” or “excavation”.

Within the frame of this conference, we tried to use this technology on sherds in order to see if the traditional investigation on pottery could be challenged. It was tested on Late Neolithic ware samples from Tell Turlu, Turkey (Jean Perrot’s excavations archives: Breniquet 1991 a)<sup>2</sup> and painted sherds from Tell es-Sawwan, Iraq (French excavations: Breniquet 1991b, 1992. See also Ippoltoni 1970-1971; Youkana 1997). All the samples used here come from official excavations and have been officially exported to France. These samples come from published excavations and were also studied by B atrice Robert with archaeometric analyses in the CRP2A Laboratory in the University of Bordeaux (Robert 2010, successfully defended in 2010). So, we expect to have a multifocal and comparative presentation of the general topic of ceramic investigation and of the different methods.

## GENERAL CONTEXT

Since the beginning of the XX<sup>th</sup> century, painted ware has been identified as a characteristic of the first agricultural settled villages in the Near East. The first painted ware found at Samarra by Hertzfeld (1911), which belonged to a set of grave goods, was found below the abbassid levels of the city. Other discoveries are reported in the same years below the neo-hittite layers of Tell Halaf, or at Tell el Ubaid during the British excavations in the twenties. At that time, the links between these painted wares and the other known wares were quite unclear and gave rise to speculation in interpreting their decorative motives (for instance *bucrania* or *swastika* or textile patterns). A better understanding of their stratigraphic positions was provided by the first stratigraphic excavations on major sites in the fifties, which set the whole chronological sequences of the Late Neolithic: Tell Hassuna, Eridu, Tepe Gawra and so on (Braidwood *et al.* 1952; Lloyd/Safar 1944; Safar/Mustafa/Lloyd 1981).

However, since the beginnings of the archaeological research in the Near East, ceramic (and by extension

painted ware which came soon after) is understood to be the last characteristic to appear in the Neolithic communities (after settled life, agriculture and herding). Moreover, the nature of the clay, the firing temperature, the pigments used for the decorative motives and the general style of the designs were considered relevant to their provenience and the “identity” of their fabricants and owners. Sometimes, pots were categorized as human groups (the famous never demonstrated adequacy between pots and people according to Kossina’s readings of the archaeological records, for instance: Demoule 2005) and their geographical area of distribution was supposed to be the area settled by specific groups. We learned that things are not so clear and that material culture is not the best way to identify ethnic groups. Cultural signification of the material differences between wares remains one of our biggest challenges.

Now, thanks to the previous great excavations, we understand that the last step of the Neolithic period is characterized by the introduction of pottery. At least, for the first time, in three different areas: northern Levant, Anatolia and Iran (and probably, elsewhere too). During the sixth millennium BC, Halaf painted pottery and the Halaf culture developed in northern Syria, from burnished ware, apparently after the contact of the Samarra culture (Nieuwenhyuse 2008). In the opposite part of Mesopotamia, Hassuna and Samarra represent a different evolution, as well as the Ubaid tradition (6500-3900 BC) from southern Mesopotamia (For an up-to-date view: Huot 1994; Nieuwenhyuse *et al.* 2013). Clays, technology, painting and firing are different.

The development of the laboratory and interdisciplinary archaeology since the fifties helped us focus on other aspects and on a more scientific platform. As stated above, from the archaeometric point of view, ceramic is a perfect material for conducting investigations: it is an inorganic, nonmetallic, and solid (because it was a fired) material and it is also a composite construction combining crystalline and non-crystalline phases, sometimes coated by thin layers of slip or paint. The whole process of transforming the clay into pot gives new physical properties to the object, perfectly suitable for archaeometric investigations. The physicists and chemists introduced new methods for dating and studying composition, determining the provenance or brought answers on technical data such the understanding of the “cha ne op ratoire” (manufacturing process, or operational sequence work), based on the physical properties of the constituting materials (especially atomic and electronic properties). The

1. For instance the exhibition held at the Mus e du Quai Branly, Paris, « Anatomie des chefs d’œuvre », 10 March 2015/17 May 2015.

2. We would like to thank J. Perrot for his generous help, many years ago.

availability and progress of computers bring a statistical perspective as well as image treatment possibilities. We are mainly concerned here by these last methods. Despite the progress made by archaeometry during the last decades, most (but by chance, not all) of the methods used (even the nano-analyses methods) are destructive and expensive. Obtaining authorization for exporting and destructing archaeological material, even for scientific reason, can be a huge problem.

## ARCHAEOLOGICAL INVESTIGATIONS

Such methods are varied and are now used in combination as they each have their appropriate applications in ceramic studies, one filling the weaknesses of another. In many phases of the studies, they complement each other in such a way that the final conclusions are better. They are often performed at different physical scales. Their main domains of application are: the characterization of the material, its structure, and the provenance of the clay or the temper in a wide perspective especially for prehistoric periods where textual data are unavailable (local products, exchanges...). This includes the technical knowledge of the potters, from the collection of the clay to the firing processes. Despite their cost and constraints, these methods provide valuable information.

For one who wants to start an archaeological investigation on pottery, the first steps are to get information about the local clays by a geological survey and, if possible, to take different samples of a local formation. This is because the composition pattern of the clay should vary in the same area due to many natural factors. Analysis of clays from production centers is a direct approach in the study of sources of fine pottery. Macroscopic evaluation of the pottery should also be carried out. Many details such as pastes, temper, and color of firing, slip or paint can be observed. Usually this is completed with the binocular observations of thin sections from sherds. During the past, binocular microscopy was used to identify the traditional way of preparing the pastes, and to recognize their different features. The observation allows a quick description and classification of the pottery pastes and it is easy to identify "foreign" sherds by visual inspection when their surface features or treatment do not yield information about their provenance. Knowledge of petrography will bring information about the impurities and minerals included in the pottery and will allow the estimation of these major impurities in the pastes, even the finest ones, providing proof of the clay sedimentation process (and the craftsman's skill) and identifying different production centers. So, a preliminary typology can be proposed on the basis of petrographic observations.

Advances in technology have allowed petrography to be approached through cathodoluminescence analysis on thin sections. Cathodoluminescence is an evaluative method used when an electron beam (Bechtel and Schvoerer 1994: 256. Bechtel and Gourdon-Platel 2000: 36) impacts a material (sherd, glass, marble...) and luminescence is produced. Minerals absorb many colors which correspond to their mineralogical nature. For example, calcite returns red, quartz is often purple and feldspars appear blue or green. In archaeometry, cathodoluminescence is used to determine the internal structures of the samples studied, in order to get information on the composition, the distribution and quality of the material. In the case of pottery, information about the paste, the temper and its granulometry can, therefore, be gathered.

X-ray Diffraction is a crystallographic analysis used for identifying minerals present in the sherd (Mannoni 1984, 237-238) or other samples containing minerals. It can also be used to quantify them. This method runs on the atomic and molecular structure of a crystal: determining the size of the atoms, but also revealing the structure and function of many molecules. Since many materials can form crystals, X-ray crystallography plays a major role in the development of archaeometry as it allows the characterization of the materials with the same chemical composition but with different crystallization morphology. This is particularly useful for the study of ceramic temper, sometimes pastes (especially for inclusions of high-firing temperature because minerals transformations can be observed) or for identifying geological deposits.

The method is, however, destructive as the sample has to be transformed into powder. The crystal is then bombarded with a focused beam of X-Rays, which produces a diffraction pattern of regularly spaced spots. The images produced are then converted into a three-dimensional model using a mathematical model and matching the chemical properties of the sample.

Scanning Electron Microscope coupled with Energy Dispersive X-ray Spectrometry (SEM-EDX) is another surface analytical technique. It produces high resolution images of the surface of a sample by using scanning electron beam. From the archaeological point of view (Le Blanc 1984), SEM-EDX gives the opportunity to determine the elementary composition of the sherds' paste, minerals and also paintings. It also gives us detailed images of the mineral or organic material, making the elementary composition of organic temper such as chaff easy to identify.

X-ray fluorescence (XRF) is the emission of characteristic "secondary" X-rays from a material after having been bombarded with gamma rays. In archaeometry, the phenomenon is commonly used for elemental analysis

and the investigation of ceramics, especially, for their provenances (Schmitt 2003). This method is destructive because part of the sherd must be transformed into powder. Currently, specialists work on a non destructive perspective.

Last, Raman microspectrometry is a non-destructive method used for observing and characterizing both the molecular composition of a sample and its surface<sup>3</sup>. The best results for archaeology are the analyses of the painted decoration of pots.

### **ARCHAEO-METRIC INVESTIGATIONS ON LATE NEOLITHIC WARES FROM MESOPOTAMIA**

The archaeometric investigations conducted by Béatrice Robert combine several methods. Samples from Tell es-Sawwan and Tell Turlu were already studied (Robert 2000, 99-124), covering the last half of the 7th and the all 6th millennia. Several typological groups were concerned. All of them were representative of a culture (Burnished Ware, Samarra, Halaf) and come from well stratified layers and are well dated.

With two samples from Turlu (BDX6535 and BDX6566), a complete characterization was obtained. The pottery has a black core and a brown surface which is linked with a first reductive and then oxidizing firing atmosphere. Manufacturing was not fully under control: the ware is porous and vegetal temper was abundant, probably mostly straw (Fig.1).

Observations with a binocular microscope and cathodoluminescence confirm the first impressions and show a siliceous paste with a natural mineral temper made of quartz and calcites. Few potassic feldspars were noticed too. These facts were confirmed by SEM-EDX analysis and a good estimation of the elementary composition which characterized the paste could be performed: silico-aluminous paste (around 73% SiO<sub>2</sub>, 8% Al<sub>2</sub>O<sub>3</sub> and 8% Fe<sub>2</sub>O<sub>3</sub>). Low levels of potassium, calcium, titanium, magnesium, manganese and sodium were also found.

For the second sample, the temper is entirely anthropic. It was composed by large white minerals (more than 600 µm) and fragments (around 150 µm). Crystallographic and microscopic methods show that the temper here is made of quartz and calcite inclusions, mixed probably with some siliceous elements.

Returning to the first sample, crystallographic analysis with X-ray diffraction confirms the presence of quartz

and calcite and helps determine the nature of the clay, which was an illite-montmorillonite, whose minerals are not destroyed by the firing.

The two sherds from Turlu belong to the burnished wares family. Clay used in both cases seems the same as well as the surface-treatments. However, the manufactures are different, corresponding probably to different uses: in the first case, a cooking-pot used on fire and maybe linked with the use of liquids, while storage was probably used for the second pot.

Regarding the two Samarra samples from Sawwan (BDX6536 and BDX6568), both are beige in color and fired in an oxidizing atmosphere. Round homogeneous inclusions (around 400 µm) seem to be more concentrated at the peripheral margins than in the center (Fig. 2).

The temper is mineral, made of quartz with few feldspars, calcite, iron oxides inclusions and probably mudstone. Crystallographic analysis allows us to add the presence of gypsum and diopsides to the list. These elements are linked with a high firing temperature, at least 900 °C. Scanning electron microscope observations show a calcareous paste (14%) with a high percentage of silicium (around 51%) and aluminum (around 14%). Iron, magnesium, potassium follow, but sodium, titanium and manganese are present in a low percentage. Temper is also made of micas which were invisible by eyes. Black pigments in the paint were identified as iron (magnetite) applied in a thick layer (100 à 200 µm).

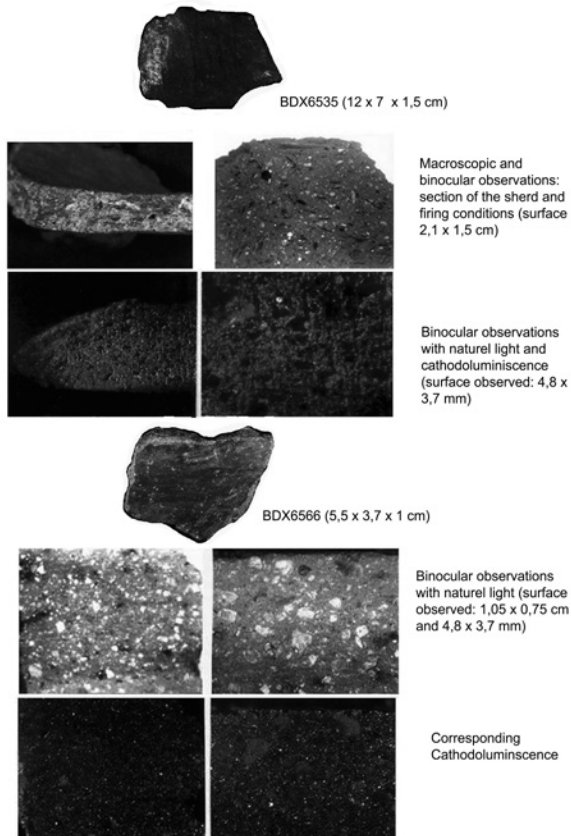
In conclusion to these Samarran ware analyses, pastes are always light beige, calcareous, non siliceous (contrary to naked eye immediate observation), with an abundant mineral temper (quartz calcites, feldspars, iron oxides, few micas and mudstones). These wares were fired at high temperatures (900°C) bringing, sometimes, a greenish color to the paste. Black thick layers of paint were made with iron pigments. Those two samples show similar technical features, quite different from the previous Halaf ones. They recall the technical characteristics of the Ubaid ware and, probably, the manufacturing processes are similar. However, in order to keep in mind that we are in front of different cultures (and people), the composition of the pastes are probably different. Ubaid pastes are siliceous and black paints are made with manganese pigments as Liliane Courtois previously shown (Courtois unpublished; Courtois/Velde 1987, 160-162, 1996, 345, 1991, 286 and 292).

In summary:

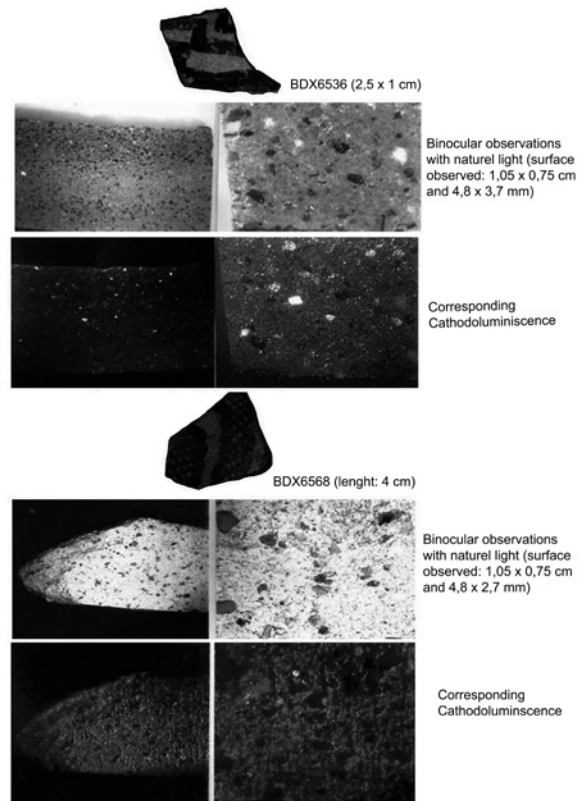
By using the different archaeometric methods present-

3. <http://www.sciencefrance.com/techniques/RAMAN/RAMAN.html>, consulted 15/04/2016.

TELL TURLU NEOLITHIC BURNISHED SHERDS



TELL ES-SAWWAN SHERDS



**Figure 1.** Manufacturing process: macroscopic, microscopic and luminescence observations. Tell Turlu Burnished sherds, Turkey. (Robert, 2010 © Robert, B. CRP2A Bordeaux).

**Figure 2.** Manufacturing process: macroscopic, microscopic and luminescence observations. Samarran sherds, Tell es-Sawwan, Iraq. (Robert, 2010 © Robert, B. CRP2A Bordeaux).

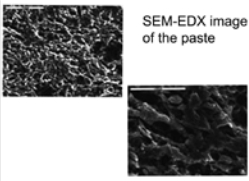
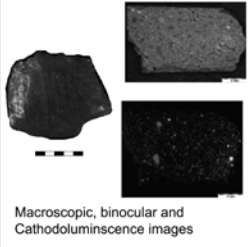
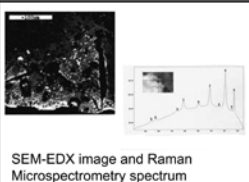
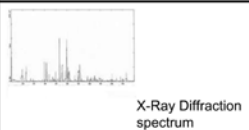
ed above, results concerning ceramic technology have been highlighted. The entire manufacturing process has been brought to light from the collection of the clay to the firing:

- The collection of the clay was approached by X-ray Fluorescence and SEM-EDX.
- The manufacture was studied through macroscopic observations with a binocular microscope, cathodoluminescence and X-ray diffraction.
- The drying process and the pigments identification were obtained through observations with SEM-EDX and Raman microspectrometry.
- The firing, then, was evaluated by X-ray diffraction as some minerals get a new physical structure at certain temperatures. (Fig. 3).

Other methods such as, for instance, differential thermal analysis (or DTA) could have been used. This is a thermo-analytic technique where the material under study and an inert reference are made undergo identical thermal cycles, while recording any temperature

difference between sample and reference. It could be combined with thermo-gravimetric analysis (TGA), where changes in physical and chemical properties of materials are measured as a function of increasing temperature, or as a function of time. They can provide data about decomposition, and solid-gas reactions (e.g., oxidation or reduction) which are particularly suitable for ceramic studies. They can shed new light into characteristic decomposition patterns, evaluation of firing temperatures, and the determination of the organic content in a sample. The main constraint remains the cost of the technical material involved.

In this context, archaeometric investigations are considered a fundamental tool applied to uncover aspects regarding the technical parameters necessary for pottery production and to understand various aspects of the ancient pottery technology.

STEP	METHOD	ILLUSTRATION
<b>Collect of clay</b>	SEM-EDX (composition of paste only)  X-Ray Fluorescence	 SEM-EDX image of the paste
<b>Manufacturing process</b>	Macroscopic Observations  Macroscopic observations with binocular  Cathodoluminescence	 Macroscopic, binocular and Cathodoluminescence images
<b>Drying process</b>	XRD SEM-EDX Raman Microspectrometry	 SEM-EDX image and Raman Microspectrometry spectrum
<b>Firing</b>	XDR	 X-Ray Diffraction spectrum

**Figure 3.** Archaeometric methods used to study the pottery's "chaîne opératoire". (Robert, 2010, unpublished CRP2A Laboratory Bordeaux).

## SCANNER TECHNOLOGY: NEW INVESTIGATIONS AT THE MUSÉE DU QUAI BRANLY

### CBCT SCANS

In order to study ceramic fragments (Late Neolithic Near Eastern Halaf polychrome pottery) we first used a Cone Beam CT scanner. Cone beam computed tomography (or CBCT) has become increasingly important in treatment planning and diagnosis in implant dentistry and interventional radiology, among others. This medical imaging technique uses a divergent source of X-rays, which form a cone-shaped beam.

In contrast, the conventional scanner uses a narrow fan beam which rotates several times around the object acquiring thin axial sections at each rotation. The CBCT scanner however, acquires the whole volume within a single rotation with its conical beam. This technology offers more freedom regarding the size of the acquisition field, which allows for a better resolution than conventional scanners, but is limited to smaller objects. In fact, CBCT scanners are not suitable for objects larger than 18x16x16 cm (Fig. 4).

Acquisitions were taken after a few minutes at a CBCT scanner equipped hospital; the data was then transferred on a USB key. The data provided by the scans was later processed with the Vizua software in order to obtain 2D and 3D images. Some appear very similar to those obtained by thin section observations and provide some insight as to the repartition, the granulometry (shape, size) of the inclusions, and the nature of the temper (mineral or vegetal).

In B&W images, it is hard to identify the minerals and to distinguish quartz and calcites as a stereo microscope does. However, it is possible to improve the processing by using colors and density. For instance, physicians use "Hounsfield units" (HU), named after the inventor of the CT. HU represent the relative density of the scanned substance through measuring the attenuation (brightness) of each pixel. It ranges from -1000 (air) to +1000 (bone). The medical range can be used to improve the approach to the study of archaeological artifacts. Densities of different ceramics analyzed by Cone-beam computed tomography, using human studies for calcium, air water, and corporal fat as a reference provide a good starting point for using CBCT in archaeology (Photo). 3D images of sherds, sections and surfaces, are quite good and offer many possibilities for studying the shape (fits or not in the typology), the paint, the way it was applied, the many layers, the errors made by the potter etc. These images could easily be compared with stereo microscope observations, which could be especially interesting in the case of the Halaf polychrome pottery. Additional processing allows comparing the composition of a few sherds on the same 3D image, in order to make their own "identity card" visible immediately, as regards to their cultural background and the technical knowledge.

This new approach can still be improved but the first results are very encouraging. The aspects speaking in favor of the method are: the low cost of the analyses, the fact that they are non-destructive and routinely available. These images are probably also a very good starting point for studying the way that various liquid substances penetrate the ceramic walls, especially milk, grease or beer. It can thus provide interesting information on the use made of the pots. In near future, we hope to improve and develop the method. Every scholar interested in this new field of research is welcome to contact us.

One way to improve the results obtained by X-ray imaging would be to enhance resolution. However, the power used for devices dedicated to medical purposes has to be kept relatively low in order to limit the patient's level of irradiation. To overcome this hindrance, we turned to Industrial Computed Tomography Scanners that provides high resolution data but, however, implies more constraining operating conditions.



**Figure 4.** CT scanner (X-ray computed tomography). Copyright: C. Moulhérat.

## INDUSTRIAL COMPUTED TOMOGRAPHY PROCESS

Basically any type of industrial CT system uses three principal components: an X-ray Tube, an X-ray Detector, and a rotational stage. Everything is enclosed in a radiation shielding Steel/Lead/Steel cabinet. This allows for the use of the system in a public environment without any additional safety concerns. Adjacent to the enclosure is a computer workstation, consisting of a 2D X-ray console for the set up and acquisition steps, and a 3D CT supercomputer workstation for volume reconstruction and visualization.

Micro-computed tomography (MicroCT) is primarily the same as a standard CT except it uses a microfocus tube instead of a traditional tube. A MicroCT scan yields resolutions in microns due to the fact that the focal spot of a microfocus tube is only a few microns in size. For comparison, MicroCT resolution is about 100 times better than the best CT scan in the medical field. High quality industrial X-ray detectors used for CT often use a new generation Amorphous Silicon Flat Panel Area Detector, offering very high sensitivity, resolution, and bit depth. The resulting 2D X-ray images are very clear and the contrast is unparalleled (Fig. 5).

Acquisition from the Samara Sample: CT is a non-destructive evaluation method that doesn't heat the specimen. In operation, it generates x-ray photons that either travel through the object or are attenuated by it. The photons which penetrate the object enter the imaging detector, carrying detailed information (see image be-

low) with them. The degree of photon attenuation depends on the amount of energy applied and the thickness or density of the material that the photon passes through. A modern high-end CT scan consists of taking several 2D X-ray images around the object, preferably covering 360 degrees (complete rotation).

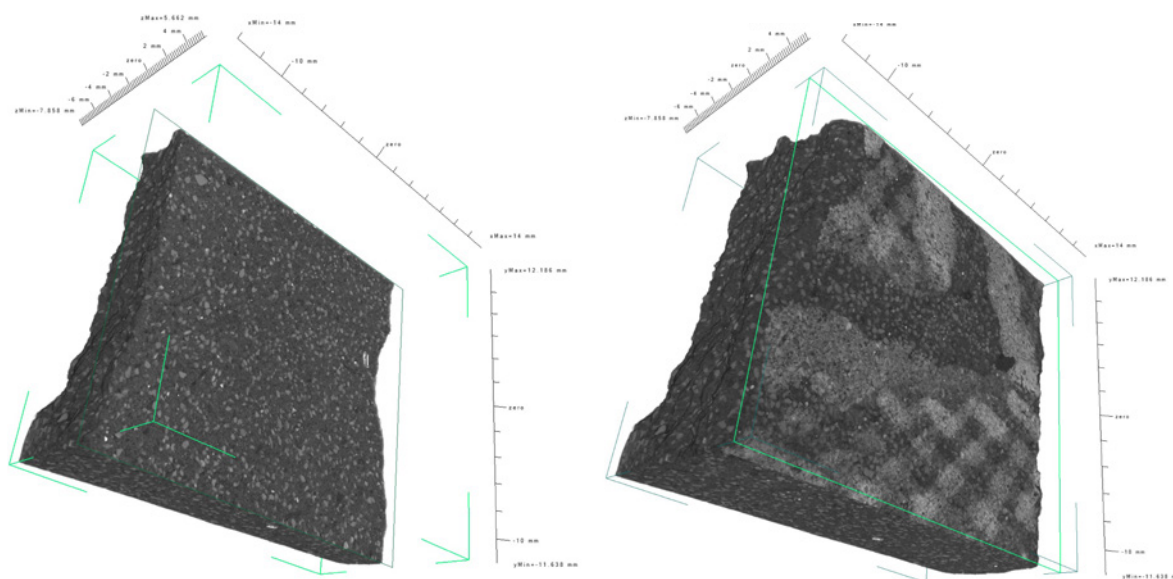
On the Samara ceramic sample, 1200 images have been acquired (1 image every 0.3 degree). Each image is 8 Megapixels and it is also averaged and filtered to reduce noise. The 2D digital images taken during this step are saved directly into a single folder which will be used in the next step of the CT process.

## RECONSTRUCTION AND VISUALIZATION

Once the acquisition process of the CT scan is completed, CT calibration and CT reconstruction algorithms are used to reconstruct the 3D CT volume. These 3D images are made of Voxels (three dimensional Pixels), and, with the use of visualization software, the 3D volume can be manipulated in real time. Therefore, it is possible to slice through anywhere inside the object, inspecting and looking for key features, taking accurate measurements, reconstructing a surface model, etc. Industrial CT technology is improving very quickly. While a few single CT slices could take hours to generate years ago, it is now possible to reconstruct complete 3D models with billions of Voxels in just a few seconds or minutes. In that regard, industrial CT has become a very competitive technology for 3D scanning (Fig. 6 a and b).



**Figure 5.** View a sherd in Industrial micro-computed tomography. Copyright: C. Moulhérat.



**Figure 6 a and b** Views of the 3D CT volume. The NSI 3D CT viewer allows that volume to be sliced in every direction and analyze both internal and external features.

The principal benefit of using 3D CT for scanning or digitization is that we obtain a complete model of an object, with both external and internal surfaces, without destroying it. Moreover, CT works with any surface, shape, color or material (up to a certain density and/or thickness penetrable with X-rays). Also, due to the penetration of X-rays, CT scans are unaffected by certain

object characteristics such as dark, reflective or transparent surfaces and/or shaded zones on the item which can difficult reading with other 3D scanning methods. Generally, a modern start-to-finish CT scan can be as fast as a few seconds or take longer than an hour, depending on resolution requirements and the size and/or density of the object. Overall, the resolution is excellent



both internally and externally. On the Samara Ceramic Sample for instance, the resolution achieved was 16µm (Fig. 6 a and b).

Within the frame of this conference addressed to ceramic specialists, we presented the exploratory investigation conducted on sherds from Late Neolithic sites of the ancient Near East, using two CT methods nowadays available for such type of studies. In summary, 3D CT is now accessible to Museums as a viable tool for scanning objects. User-friendly interfaces, increased scan speeds, resolution and image quality have all contributed to the rapid development of this technology. CT is quite unique for accessing especially accurate imagery data without destroying the item. There are no shaded zones, it works with all kinds of shapes and surfaces, there is no post-processing work needed – or very little, depending on the application – and the resolution is excellent. Most of all, the greatest benefit is the ability to nondestructively obtain the internal structure of the object, which only CT technology is able to provide.

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