NEUTRON IMAGING FOR ARCHAEOMETRY

A NEUTRONOS KÉPALKOTÁS ARCHEOMETRIAI ALKALMAZÁSA

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Abstract

This paper gives an overview of neutron imaging applied to cultural heritage science. After introducing the potential of non-destructive imaging techniques (both with neutrons and X-rays), principles of neutron absorption radiography (NR) and tomography (NT) are described and compared to the corresponding X-ray radiography (XR) and tomography (XT). Examples of archaeometric applications made with NR/NT and presented hereafter are related to three major topics: (1) The technological studies investigate the details of the manufacturing method. (2) The functional studies determine the utilization of the objects by examination of the internal content (residues). (3) The corrosion or conservation studies focus on the preservation state and methods of the object. Neutron imaging is proved to be a powerful non-invasive tool in archaeometry, especially for the visualization of organic contents inside metals or less-dense matrices. Synergies between X-ray and neutron imaging are also highlighted.

Kivonat

Munkánk áttekintést nyújt a neutronos képalkotási módszerek archeometriai alkalmazási lehetőségeiről. A hagyományos (neutrongyengülésen alapuló) neutronradiográfia (2D, NR) és -tomográfia (3D, NT) a neutronok anyagbeli relatíve nagy behatolási mélységén alapul, így lehetőség nyílik (elsősorban szerkezeti, másodsorban közvetetten anyagi minőségre vonatkozó) információt szerezni a tárgyak belsejéből roncsolás nélkül. Tanulmányunk nem tekinti mindenhatónak a neutronos képalkotást, sokkal inkább – ahogyan arra a bemutatott példák is rámutatnak – egy, a már hagyományosnak mondható röntgenes képalkotási módszerek mellé kiválóan társítható módszerként mutatja be, kiaknázva mindkét sugárzástípus előnyeit.

Az archeometriai kutatásban elsősorban a fémtárgyak képi megjelenítése elterjedt, de van példa könnyebb anyagok (kőzet, kerámia, üveg, fa, papír, csont, bőr) vizsgálatára is. A leghatékonyabb képalkotás azoknál az összetett tárgyaknál érhető el, amelyekben szerves anyagok nehezebb (nagyobb rendszámú, tehát pl. szilikát vagy bizonyos típusú fém/ötvözet) mátrixba ágyazva jelennek meg, mivel ez esetben nagy a neutrontranszmissziós kép kontrasztja. A fő kérdéskörök (1) a technológiára, azaz a készítés módjára; (2) a funkcióra, azaz a tárgy használatára; illetve (3) a tárgy korrodáltságára és konzerválhatóságára irányulnak. Mindhárom témakörben több sikeres neutronos képalkotási tanulmányt tekintünk át, amelyek közül azok tekinthetők a legeredményesebbeknek, amelyek egyszerre alkalmazzák a röntgenes és a neutronos technikákat.

KEYWORDS: NEUTRON RADIOGRAPHY, NEUTRON TOMOGRAPHY, TECHNOLOGY, FUNCTION, CONSERVATION

KULCSSZAVAK: NEUTRONRADIOGRÁFIA, NEUTRONTOMOGRÁFIA, KÉSZÍTÉSI TECHNOLÓGIA, HASZNÁLAT, KONZERVÁLÁS

Introduction: Potential of neutron imaging

Imaging is a powerful technique to non-destructive and non-invasive investigation of complex samples. Looking through and into objects made of a few different materials, created by complex manufacturing processes and used for different purposes is a common objective in archaeometry. Diversity of materials analyzed by imaging methods ranges from metals (e.g. Lehmann et al. 2005b, Lehmann 2006, de Beer et al. 2009, Berger et al. 2013, Agresti et al. 2016) through stones (e.g. Jacobson et al. 2011), ceramics (e.g. Stanojev Pereira et al. 2013, Abraham et al. 2014), glasses (e.g. Fiori et al. 2006), wood (e.g. Osterloh et al. 2015) or other organic matters to pigments (e.g. Boon et al. 2015). Complexity of manufacturing techniques amenable to imaging methods means forging/casting of metals (e.g. van Langh et al. 2009, 2011, Gravett 2011, Rehren et al. 2013); polishing or carving stones (e.g. Jacobson et al. 2011); tempering/mixing/kneading ceramic raw materials and coiling/wheeling of pottery (e.g. Latini et al. 2013, Miśta et al. 2016); molding/blowing of glass (e.g. Fiori et al. 2006); carving wood (e.g. Masalles et al. 2015); joining/soldering of the different parts of a complex object (e.g. Mannes et al. 2015). Function identification of artefacts can involve the mechanical use, the storage of dry or liquid materials (e.g. Stanojev Pereira et al. 2013), cooking, production at normal or high pressure and temperature conditions, etc. In addition, from an archaeological perspective, aging or deterioration is also a relevant factor and the preservation state of the objects is an observable feature of the object. Imaging techniques can reveal exactly these features: external morphology, internal structure, inhomogeneities (e.g. fake complements), and corrosion.

Neutrons can be used for imaging because they give high-resolution information from deep layers of the material in a completely non-invasive way. Neutron radiography (NR, 2D) and neutron tomography (NT, 3D) could provide information about the inner structure of the sample. This way, interesting details can be observed without destructive sampling and important areas of the sample can be selected for further investigation.

Being interdisciplinary, cultural heritage science relies on the knowledge of many disciplines of natural sciences, such as physics, chemistry, geology and biology. It is not surprising that imaging techniques that have been successfully applied in all these research fields for many decades, found their application niche in the expanding methodological repertoire of archaeometry. However, it is our intention to emphasize that neutron imaging is not omnipotent, and that its complementary application together with X-ray imaging and other neutron techniques (diffraction, elemental analysis) is usually the most attractive way of investigation. This paper aims to present the conventional (absorption) imaging possibilities with neutrons, and to give an overview of its worldwide applications in cultural heritage science.

Methods

Principles and conditions of neutron absorption radiography and tomography

Radiography literally means 'draw with radiation'. It is a direct imaging technique, where the visual representation of an object is obtained non-destructively by detecting the modification of an incident beam as it passes through matter. Radiography transforms the interaction of an invisible radiation with the material into tangible images (**Fig. 1**). By applying this method, information is revealed about internal features of an object which are otherwise invisible from outside. The visibility of an image depends on the contrast, i.e. the difference between the intensities (grayscale values) observed at two adjacent areas of the image. Mathematically it is the ratio of the difference over the sum of adjacent intensities. In practice, the grayscale values span a range from black to white with various (12-16 bit) information depth; 12-bit and 16-bit images have 2^{12} (4096) and 2^{16} (65536) native gray level values, respectively.

Tomography is an extension of radiography, where the 3D visualization of the object is achieved using computational algorithms from a series of radiographic projections acquired as the object is rotated in small angular increments. Through digital processing, a virtual-reality representation can be created by reconstruction and rendering of the 3D image (**Fig. 1**).

Neutrons are electrically neutral particles, so they easily penetrate into the sample, and the interactions (e.g. nuclear reactions, scattering) that take place there cause the attenuation of the incoming neutron beam (lowering its intensity along straight trajectories from the source towards the detector).

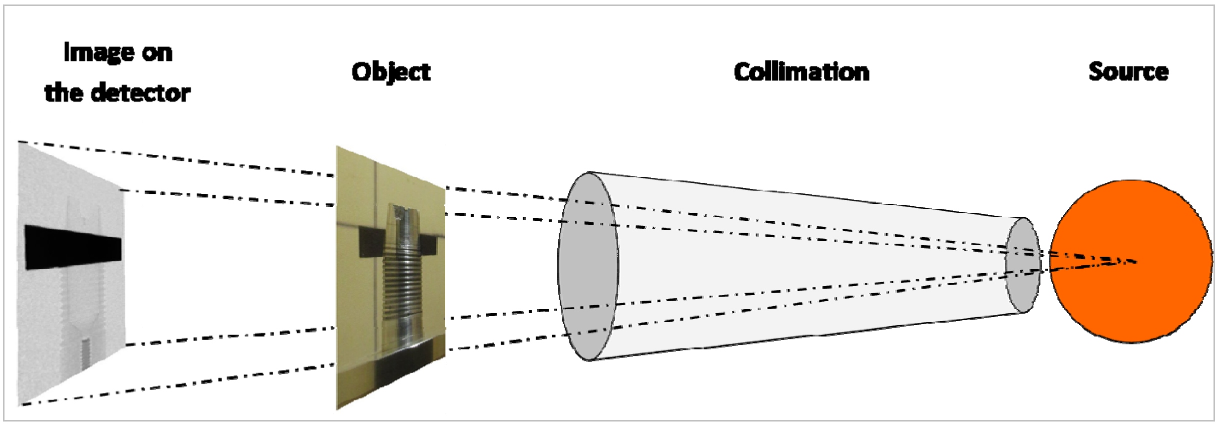

Fig. 1.: Simplified setup of a neutron absorption radiography experiment

1. ábra: A neutronabszorpciós radiográfiai berendezés egyszerűsített felépítése

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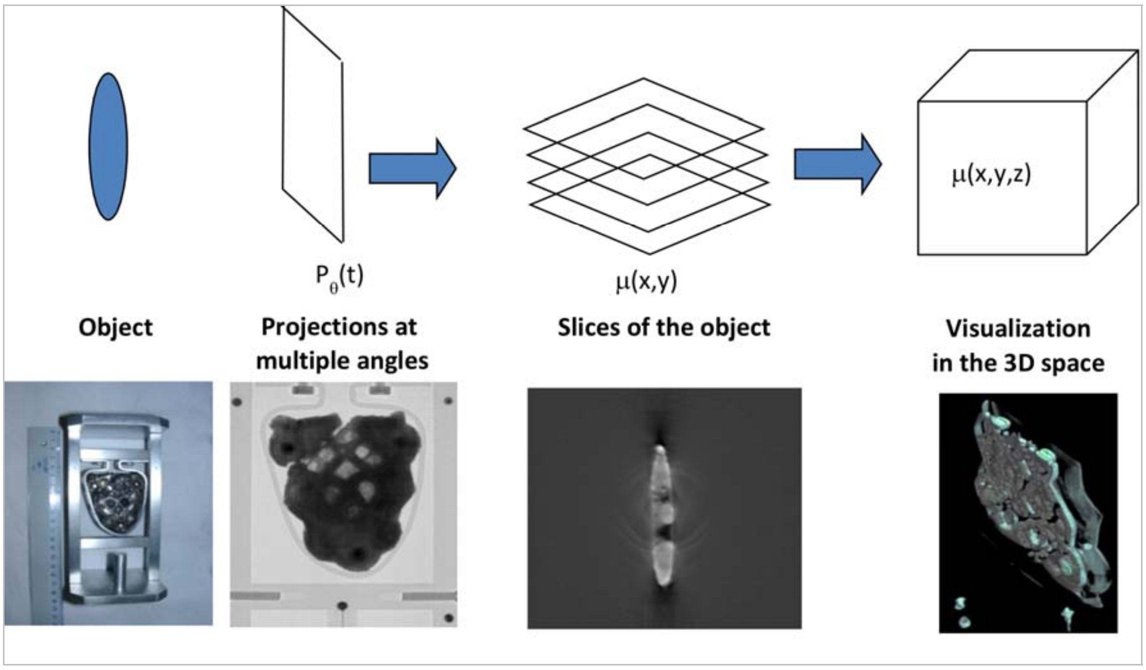

Fig. 2. Transformation of a set of 2D neutron radiographic projections into a 3D tomographic image (example in the lower row is taken from the EU FP7 ANCIENT CHARM project)

2. ábra: A 2D neutronradiográfiai projekciók átalakítása 3D tomográfiai képpé (az alsó sorban bemutatott példák az EU FP7 ANCIENT CHARM projekt keretében készültek)

According to the Beer-Lambert law, in a material with a thickness $\boldsymbol{d}$, the intensity of the unmodified beam ($\boldsymbol{I_{tr}}$) decreases exponentially relative to the incident beam ($\boldsymbol{I_0}$):

$$\frac{I_{tr}}{I_0} = \exp\left(-\mu^{tot} \cdot d\right),$$

where $\boldsymbol{\mu^{tot}}$ is the linear attenuation coefficient (cm^{-1}) of the sample material. In case of thermal and slow neutrons (i.e. their kinetic energies are relatively low), there are two dominant types of interactions: absorption and scattering. The attenuation of the neutron beam while travelling through the objects is, in general, a collective effect of these two processes; and for their visual representations radiography (NR, 2D image) and tomography (NT, 3D image) are appropriate methods. The depth of penetration and the probability of an interaction depend strongly on the energy-distribution of the incident neutron beam and the material of the object in its path. Neutrons are able to penetrate through several cm thick materials, so the inside of even a larger object can be investigated, too. A generally accepted condition is that the transmission should be between 2% and 98%, from which the corresponding thickness can be calculated as follows:

$$d_{\max} < \frac{-\ln(0.02)}{\mu^{tot}} = \frac{3.91}{\mu^{tot}}$$

$$d_{\min} > \frac{-\ln(0.98)}{\mu^{tot}} = \frac{0.02}{\mu^{tot}}$$

According to the basic idea of imaging, the object placed in the path of the neutron beam shadows on the neutron-sensitive screen, i.e. neutrons interacting with the object's material will not impinge the screen (in practice, unfortunately, this is not necessarily true). The image (i.e. a contrast pattern) is developed by the different number of unattenuated neutrons arriving on each single pixel of the detector screen in a unit time.

In the mid-1930s, the first system to record neutron radiography pictures of was created by H. Kallman and E. Kuhn (Chankow 2012). In the 1950s, with the appearance of intense neutron sources (by developing research reactors), better quality pictures were recorded. The scientific and industrial applications of radiography reached a more intensive development phase when the detection of neutrons became more productive. Film detection had an ultimate role for tens of years, but with the appearance of digital techniques and better detectors it was supplanted in the 2000s. Today, besides traditional radiography, special imaging techniques (e.g. tomography, energy-selective imaging, use of polarized neutrons, grating-interference imaging) are becoming more common.

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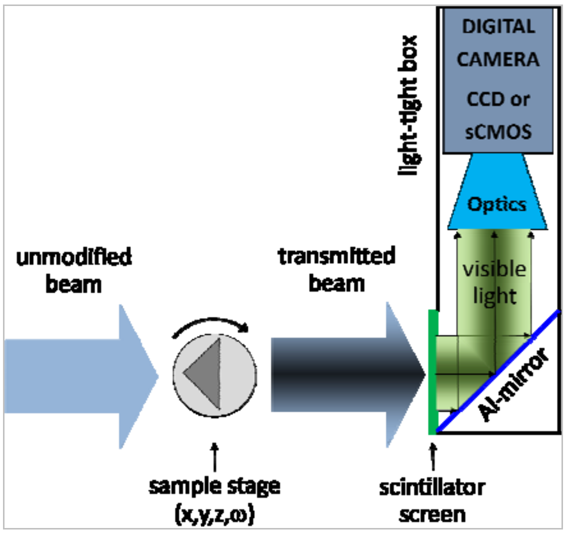

Fig. 3. Major components of a modern radiograph / tomograph

3. ábra: Egy modern radiográfiai/tomográfiai berendezés fő alkotórészei

Modern radiography/tomography setups (**Fig. 3.**) consist of a neutron sensitive, visible-light emitting scintillator screen, a mirror, optics, a digital camera and image processing software and hardware. Modern devices applying digital imaging have several advantages. Applying cold and thermal neutrons gives better detectability and good contrast for most materials. For better detection of neutrons scintillators with ZnS/^{6}LiF and Gd-oxide are widespread. Creating an image is based on the different amount of visible light that is emitted by certain points of the scintillator screen. The emerging (usually green or blue) light is then directed towards the optical system (objective + digital camera) by a mirror set in 45° angle to the beam. More parallel beam geometry (increasing the so-called L/D ratio) improves the spatial resolution, even for parts of the object located opposite to the screen, on the expense of a longer exposure time. It means that using a beam with a large L/D-ratio the inherent resolution of the screen could be approached for a wider distance from it. Nowadays a routinely achievable spatial resolution is in the order of several tens of microns (Kaestner et al. 2016). The best spatial resolution reported in the literature is around 5 μm at the moment (Trtik et al. 2015, Trtik & Lehmann 2016). The light-sensitive CCD or sCMOS chip in the camera collects the incoming light in its pixels during the exposure, and after readout it provides a set of grayscale values, i.e. the intensity at each (x,y) pixel. The image processing software and hardware do the necessary calculations with the image treated as a data matrix. During data collection it is possible to follow the processes in real time, dynamically, e.g. water absorption, by consecutive recording of snapshot images.

Advantages of digital imaging are the high sensitivity and productivity, the fast readout, the high frame rate and the outstanding linearity within a wide dynamic range. This way, an acceptable (but still improving) spatial resolution and digital information suitable for quantitative evaluation can be collected allowing the possibility of subsequent image processing, easy operation and data transfer, and possibility of tomographic image processing.

Comparison of neutron and X-ray imaging

Neutron imaging, similarly to X-ray imaging, is successfully applied in science and industry. Successful scientific applications in nuclear technology, materials research, wood and soil physics, geology-palaeontology and archaeology-cultural heritage are remarkable. In the industrial sector, non-destructive testing (welding, soldering and brazing; structural integrity and performance; adhesive connections), two-phase flow modelling, characterizing combustion engines and fuel cells are the most relevant topics.

When comparing X-ray and neutron imaging, the following facts has to be considered. The neutron attenuation coefficients of elements show rather large variations even between neighbouring elements, sometimes even up to orders of magnitudes, unlike with X-rays, where the attenuation coefficients increase monotonically and smoothly with the atomic number. The reason is (neglecting the details) that the neutrons interact with the atomic nucleus, whereas X-ray photons interact with the electrons of an atom. For neutrons, however, hydrogenous materials deliver high contrast, and many metals can easily be trans-illuminated. It can be beneficial in visualizing objects, including organic material (the beam attenuation is significant due to the hydrogen content), and for discriminating between elements with similar atomic numbers (which do not give appropriate contrast in X-ray radiography). For X-rays, light elements (e.g. organic materials) have low contrast, and heavy elements (e.g. metals) are difficult to trans-illuminate. This way, the complementary character of the two radiations can easily be understood. In addition, laboratory-based X-ray radiography systems are more easily accessible in many labs, while neutron-imaging facilities have been established only at a few dedicated institutions. It is also an important aspect that XR/XT could have much better spatial resolution than NR/NT (see **Fig. 4.**) due to the more localized light conversion of the scintillator screen.

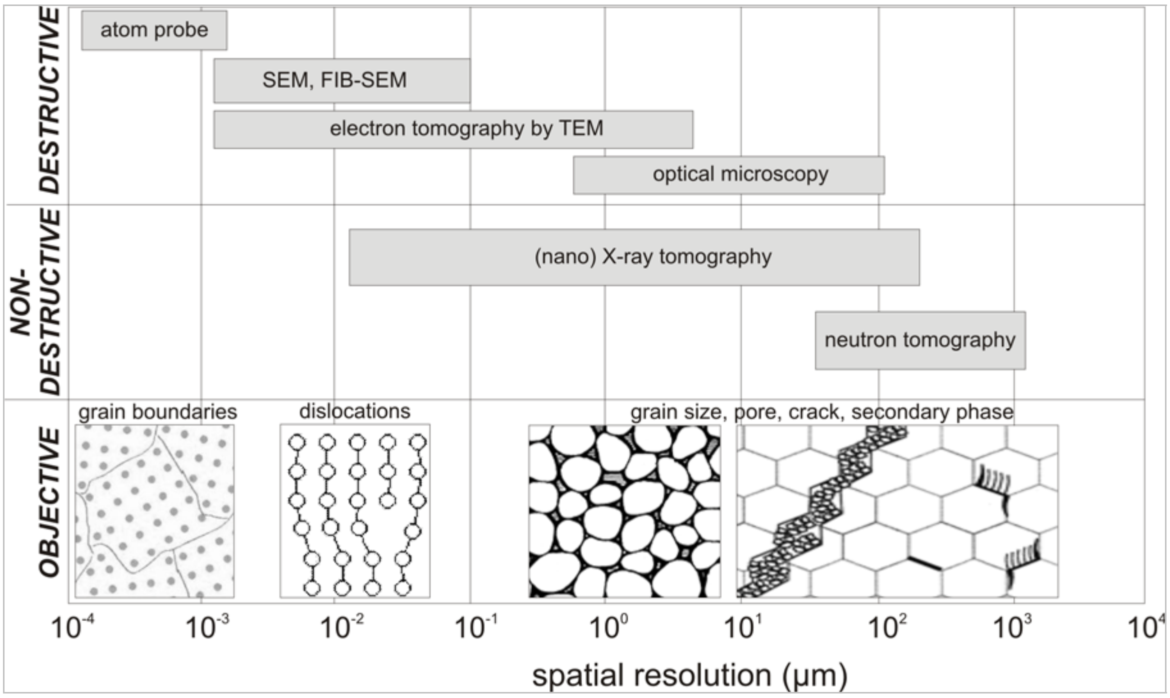

Fig. 4. Comparison of some methods for 3D imaging applied to material study (after Salvo et al. 2010)

4. ábra: Néhány, az anyagok 3D-s képalkotásában alkalmazott módszer összehasonlítása (Salvo et al. 2010 nyomán módosítva)

NR and NT facilities around the world

As it was mentioned above, there are a handful of dedicated state-of-the-art neutrons imaging facilities around the world. The most advanced "user facilities" are located in Europe (Germany, Switzerland, Hungary), North America (US, Canada) and the Far East (Japan, Korea, China, Australia) as can be seen in **Fig. 1g. 5.** There are additional facilities in South Africa, Russia and Brazil as well, serving basically the local needs. For details see Lehmann et al. (2011).

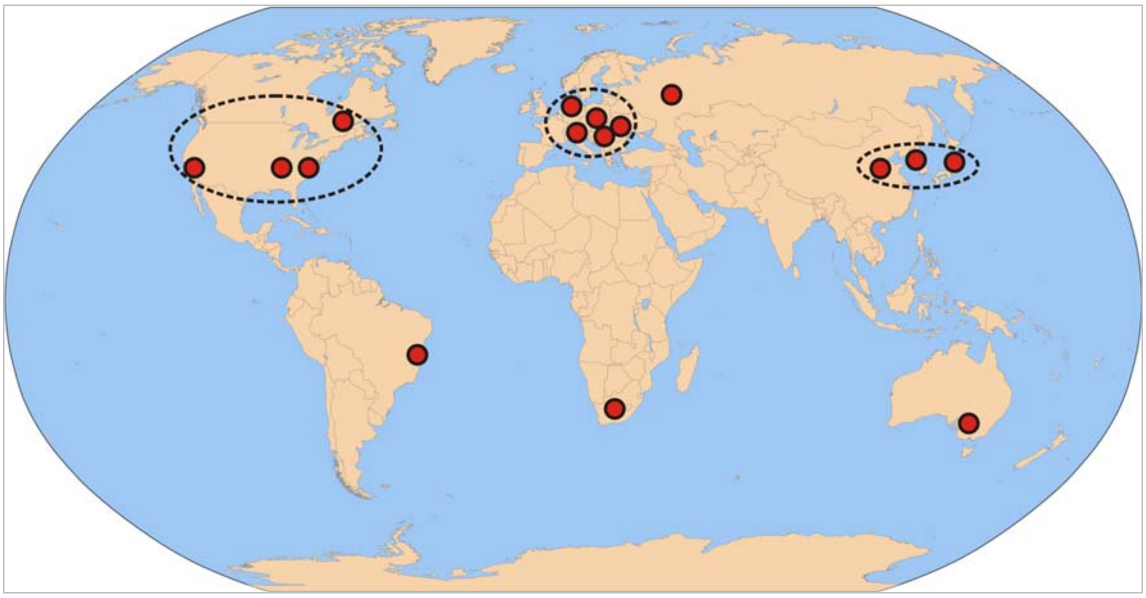

Fig. 5. State-of-the-art neutron imaging facilities in the world (data taken partly from Lehmann et al. 2011)

5. ábra: A világ legkorszerűbb neutronos képalkotási berendezései (az adatok forrása részben Lehmann et al. 2011)

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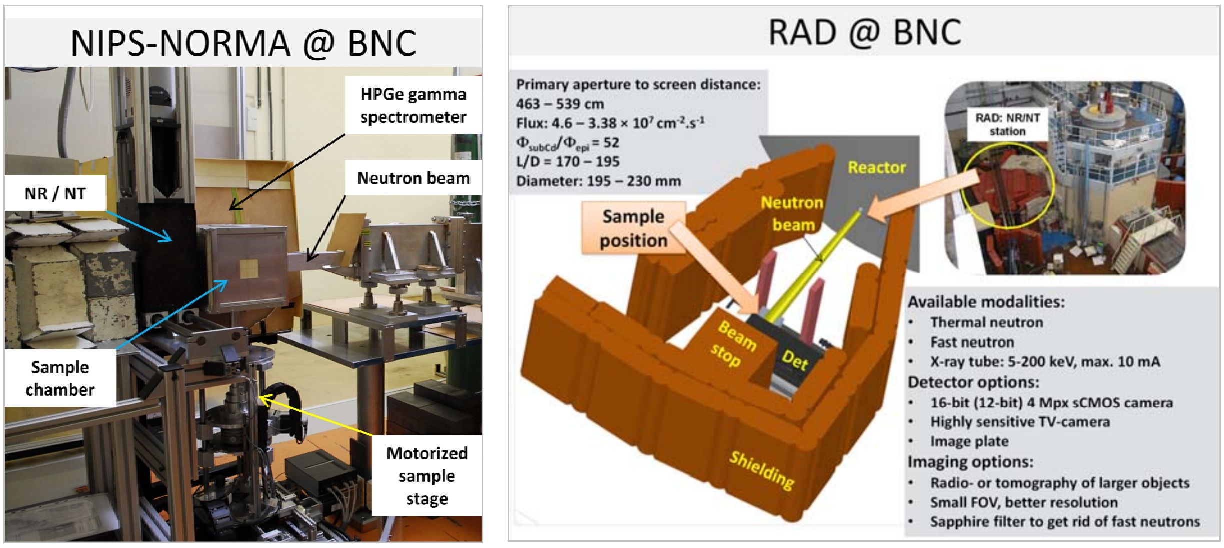

Fig. 6. The two neutron imaging stations at the Budapest Neutron Centre (BNC): (a) NIPS-NORMA station and (b) RAD station

6. ábra: A Budapesti Neutron Centrum (BNC) két neutronos képalkotási berendezése: (a) NIPS-NORMA mérőállomás és (b) RAD mérőállomás

The Hungarian neutron imaging facilities (NIPS-NORMA station and RAD station, see **Fig.Fig. 1 6)** are located at the Budapest Research Reactor. The two stations operate under the umbrella of the Budapest Neutron Centre (BNC), which serves as an entry point for users interested in such investigations. The RAD facility is intended to study larger objects with bimodal (neutron, X-ray) imaging, even in real-time. The NIPS-NORMA facility is for neutron imaging of smaller objects in combination with position-sensitive element analysis by Prompt Gamma Activation Imaging (PGAI).

Risk of adverse effects of the neutron exposure

Any objects put in a neutron beam for imaging will absorb a proportion of the traversing neutrons because this creates the shadow image in the detector. We must be however aware of the potential risk of exposures as a side-effect (Bertrand et al. 2015). Theoretically, the risk increases with longer neutron irradiation times and the use of more intense beams, resulting in transient radioactivity and possible alteration of the objects. During imaging experiment, absorption of a neutron in the atomic nuclei could generate radioactive isotopes which reaction is called activation. As the number of affected atoms is negligibly low, it has no macroscopic effect on the physical integrity of the object, neither on its composition. A few days of decay after the imaging experiments can therefore be necessary to get rid of the induced radioactivity in the object.

The alteration of the object could be recognized in form of e.g. visible (colour) or integrity (structural) changes. Any form of tangible damage, in turn, needs several orders of magnitude higher neutron fluence than used in a typical experiment, so on a routine basis no degradation is observed. If the level of the induced radioactivity is kept at a reasonable level, it will automatically guarantee that the radiation damage of the object is avoided.

If there are a priori information available about the elemental composition of the sample, calculation of the induced activity under the given circumstances could be assessed already at the feasibility check of an experiment (Kis et al. 2017). Then an upper limit of the irradiation time can be defined which keeps the activity level of radioisotopes within the objects below the legal exemption levels. Fortunately, the usual exposure times in imaging (minutes, hours) are well below these limits; therefore any item that has a radioactivity below the exemption levels (which could be different for different isotopes) are practically and legally considered as inactive material. These objects could be given back to the owner and can be used without any restriction. If the radioactivity level incidentally exceeds the exemption levels, the object should be kept at measurement site for a while to let the radioisotopes decay out, of course under conditions appropriate for valuable artefacts, if needed. Fortunately, for typical materials in cultural heritage studies the so-called half-life of most created radioisotopes is so short (less than some hours) that this safeguarding is not needed or does not exceed a few days.

Archaeometrical case studies

Theoretically, neutron radiographic and tomographic studies follow the same protocol (from the irradiation of the sample through the detection of the contrast image to the basic processing of the data). However, the main difference lies in the time needed for the measurement (some or hundreds of projections to be taken) and the subsequent image-processing procedure (e.g. beam-profile and detector noise correction, mathematical reconstruction of 3D images). Considering the archaeometric applications, sometimes it is not necessary to observe the complete 3D morphology of the object to interpret the data. However, observing the complete 3D structure of an artefact can help to understand the connections among the different (structural or functional) parts of a given object in many situations. For instance, radiographic images can be applied for investigation of the manufacturing process (e.g. casting of metal sculptures) but tomography is essential when complexity of the materials' distribution has to be demonstrated (e.g. corrosion state of an object for a conservation study, internal content of a closed/deformed object without damage).

Considering the material types of the neutron imaging studies in archaeometry, the most frequent objects are made of metal (especially bronze and iron). Lighter substances like stone, ceramic, glass, wood, bone, textile or leather are much less common. This phenomenon is probably due to (1) the emphasized role of metal objects in many archaeological contexts, (2) the usually higher contrast between fresh and corroded metallic phases compared to that in lighter materials or (3) the scale of textural features relevant in the identification of manufacturing techniques which is more appropriate for the investigation by neutrons (which is usually 50-500 μm for metals, while 1-30 μm for ceramics or glasses) (e.g. Jacobson et al. 2011).

Observing the topic of the archaeometric investigations made with neutron imaging techniques, the three main groups are (1) *technological studies* which analyze the details of the manufacturing method; (2) *functional studies* which determine the utilization of the objects by examination of the internal content (residues); and (3) *corrosion or conservation studies* which focus on the preservation state of the object. These will be discussed hereafter.

Technological studies

Details of the manufacturing process provide important information for archaeologists and restoration-specialists. Identification of the manufacturing technique is crucial since it can (1) be characteristic to the archaeological era (and culture) and thus can be considered as an indirect dating method, or (2) simply characterize the development of handicraft in a less known era.

Among metals and alloys, the most common objects of radiographic studies are bronze (e.g. Lehmann 2006, Lehmann et al. 2010a, Ryzewski et al. 2013, Kiss et al. 2015, Agresti et al. 2016) and iron (e.g. Rant et al. 2005, Godfrey & Kockelmann 2011, Koleini et al. 2012, Rehren et al. 2013), but copper (Koleini et al. 2012, Figueiredo et al. 2016), tin, lead (Masalles et al. 2015), gold and silver objects have also been successfully investigated. In the case of metals (and alloys) the fundamental imaging problems are the identification of the forming technique (forging or casting) and the exploration of the complexity of composite objects (made with the combination of more materials besides metal). Evidence of cast manufacture (e.g. armatures and cores, cavities due to casting faults and porosity due to trapped gases) (Gravett 2011, Figueiredo et al. 2016, Mannes et al. 2014a) or forging (e.g. hammer marks, chisel cuts, weld lines and fibrous microstructures) in finished artefacts could be detected by radiography (e.g. Koleini et al. 2012, Rehren et al. 2013). Tracking of mounting artefacts from single or multiple components, and methods of joining (e.g. pins, studs, rivets, seams, casting-on and soldered joins) are also observable. Lehmann et al. (2005b) investigated a replica of the famous 'Heavens Plate of Nebra' and determined an adhesive layer fixing the gold layers on the base metal plate. The investigation of the complexity of objects (e.g. weapons) is an ideal task for the combined X-ray and neutron radiography / tomography, where both the metallic and the non-metallic parts can be studied in details. Composite offensive weapons (sword, scabbard) were investigated by Deschler-Erb et al. (2004), Mühlbauer et al. (2007) and Mannes et al. (2015) to visualize fine internal details. The X-ray images highlighted all metallic parts, including the ornamental inlays on the surface, while the neutron images focused on the wooden structure as well as the main metal parts. Due to the complementary imaging techniques, the entire structure of the scabbard consisting of fur, wood, leather, cord and bronze could be reconstructed (Mühlbauer et al. 2007).

Neutron and X-ray imaging of an Early Iron Age bronze object from the Hungarian National Museum (Hungary) supervised by G. Tarbay was investigated at the RAD imaging station of the Budapest Neutron Centre (Hungary). It was an Early Iron Age bronze shield found amongst other confiscated objects, which supposedly originated from the territory of the Balkans. A part of the object (**Fig. 7.**), where the rim and the adjacent layer is held together, was supposed to contain materials invisible from outside.

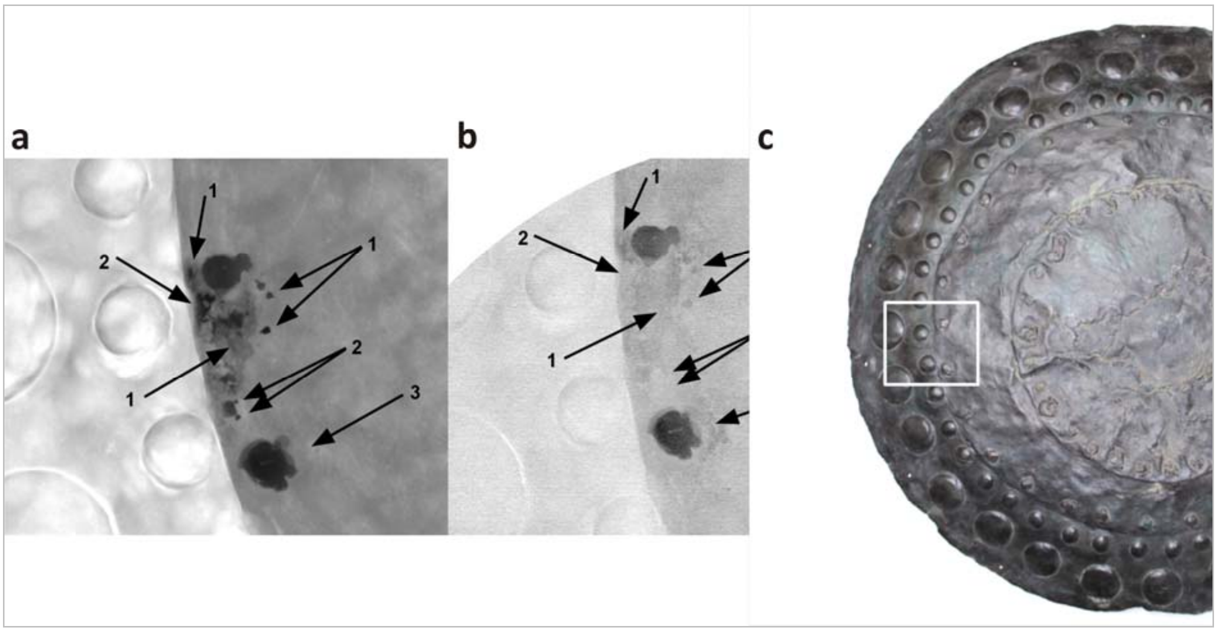

Fig. 7.: The (a) X-ray and (b) neutron transmission images of (c) a detail of a bronze object (shield?, Early Iron Age, Hungarian National Museum, Budapest, Hungary). See the interpretation of the numbered spots in the text.

7. ábra: Egy bronz tárgy (pajzsdudor?, kora vaskor, Magyar Nemzeti Múzeum, Budapest) részletének (a) röntgen- és (b) neutrontranszmissziós felvételei. A számozott pontokra vonatkozó értelmezést lásd a szövegben.

Darker spots giving contrast in both the X-ray and the neutron images (arrows labelled by 1 in **Fig. 7.**) are presumably the broken-down parts of the rivets. Darker spots, which give contrast in the X-ray image, without giving contrast in the neutron image (arrows labelled by 2 in **Fig. 7.**), could be materials with higher atomic number (e.g. lead, which have high and low attenuation for X-rays and neutrons, respectively). A darker spot, which gives contrast in the neutron image, without giving contrast in the X-ray image (arrow labelled by 3 in **Fig. 7.**), could be organic material.

Mannes et al. (2015) presented an excellent case study investigating the complexity of an object, utilizing the advantages of the combined neutron and X-ray imaging. The analyzed object, a 15th century short sword found in lake Zug (Switzerland), is a composite of metal (e.g. blade, pommel, metallic ornaments of the hilt) and organic material (e.g. the wooden parts of the hilt, wooden disc concealed by the metal cap of the pommel), which are rather transparent for one type of radiation, while yielding at the same time high contrast for the other (see **Fig. 8.**). The organic material parts yield a very high contrast in the neutron image, while it is barely visible in the X-ray image. Larger metal pieces, such as the tang, the central metal piece of the hilt, as well as the metal pins connecting grip and guard are visible in both the XT and the NT images.

A lot of strong reconstruction artefacts appear in the X-ray CT slices as a result of the dominant metal parts, caused by the signal from the metal outshining the signal from the organic parts. As a consequence the wooden structure cannot be reconstructed in the images. Nevertheless, the distribution and position of the small metallic ornaments can solely be determined with help of the X-ray data.

In the case of ceramics, it is a very important question how the pottery body was built up. Differentiation among the primary forming techniques (e.g. drawing, coiling, slab building, molding, throwing) can be essential in certain archaeological contexts. The mechanical stress put on the plastic raw material can be tracked in the texture of the fired final product. The criteria for identifying the primary forming techniques were established by Rye (Rye 1977, 1981), and are based on the orientation of voids and elongated temper particles (see Berg 2008, 2009 for a detailed discussion). In most of the investigated ceramic assemblages, identification of the forming technique was possible (Magrill & Middleton 2001, 2004; Berg 2008, 2011). Similarly to the metal artefacts, mounting of attachment (e.g. spouts, handles) can be investigated (Berg 2011).

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Fig. 8. 3D-visualisation of the combined X-ray and neutron CT data sets of a short sword (Zug, 15th c., Switzerland); information of the small metal pieces such as ornaments pins and nails originate from the X-ray CT; the wooden parts and larger metal parts originate from the neutron CT. (from Mannes et al. (2015); a YouTube video is accessible, too: NIAG - Neutron Imaging and Activation Group 2015)

8. ábra: Egy rövid kard (Zug, 15. század, Svájc) kombinált, röntgen és neutron CT méréséből készített 3D-s megjelenítés. A röntgentomográfiai mérés szolgáltatott információt a kisebb fém elemekről (pl. díszítések, tűk, szögek), míg a neutrontomográfiai mérés a fa és a nagyobb méretű fém részeket azonosította. (kép forrásai: Mannes et al. 2015; a NIAG-Neutron Imaging and Activation Group 2015 videója a YouTube-on)

It is important to note that due to the average grain size of ceramics (besides clay particles, 15-2000 µm), spatial resolution of X-ray imaging is more appropriate for their study than neutron imaging (Jacobson et al. 2011). In addition to the above mentioned points, in relation to the technological aspects of the *chaîne opératoire* approach, XR can also help with the characterization of clay fabrics and this can provide information on the provenance (NR: Latini et al. 2013, XR: Berg 2011). Repairs and breaks invisible to the naked eye can be also identified with confidence. However, identification of secondary forming techniques and surface treatments is not possible. One can see well the glaze of the pottery as well.

An example of neutron and X-ray imaging of ancient ceramics (a ceramic pot from Budakalász, Hungary from the 7th century AD), in the form of a neutron transmission tomographic image, can be seen in **Fig. 9a**. The object is made up of original ceramic sherds and a modern complementary material (gypsum) for the missing parts. As one can see the different parts of the pottery can easily be separated based on their grayscale values. During the rendering of the 3D dataset there is a possibility to show just that range of the grayscale values which is interesting for the researcher. For example, in **Fig. 9b** parts with higher neutron attenuation values are visualized. This image shows the gluing material between the fragments, the tiny grain-like material in the body of the clay, and some gypsum. A vertical cut of the X-ray transmission image of the pottery can be seen in **Fig. 9c**. This image shows the different structures of the original clay and the modern gypsum. In **Fig. 9d** only the higher attenuation value parts of the materials are shown. The gluing material is not visible because it is a high H-containing material, which gives negligible contrast for X-rays. The more dense parts, however, are more or less clearly seen.

In the case of glasses, the detection of defects, inhomogeneities or voids can help to interpret the melting and fashioning processes (Fiori et al. 2006). Internal structure of artefacts made of other materials like stone (Jacobson et al. 2011), wood (Osterloh et al. 2008, 2015) or bone (Miśta et al. 2016) can also be investigated. During NR/NT of wood, the resulting images show even the annual rings in the wood, not to be mistaken with the circular rings artificially produced during the tomography reconstruction process (Masalles et al. 2015). These authors identified the internal wood core of a lead sculpture to be pinewood which was carved out of a block of 3 different vertical planks gluing together. It was due to that some straight joints could be spotted at different heights in the horizontal tomograms all through the sculpture, thanks to the change of direction of the concentric wood structure, creating the vertical joins visible in the tomograms. Wavy carving marks can be seen on the wood sculpture by NR and NT, proving the carving with gouges. Chips of wood are trapped behind the lead sheet and in elbows.

Utilization and 'inside' studies

In this chapter, intended use of the artefacts, or the role of 'hidden content' within the objects is discussed. In many cases this 'content' cannot be considered as evidence on the real utilization of the finds but rather gives information on their underlying (e.g. religious, ritual) meaning/role. Direct utilization studies focus on the content of closed vessels or other containers (e.g. Stanojev Pereira et al. 2013, Abraham et al. 2014).

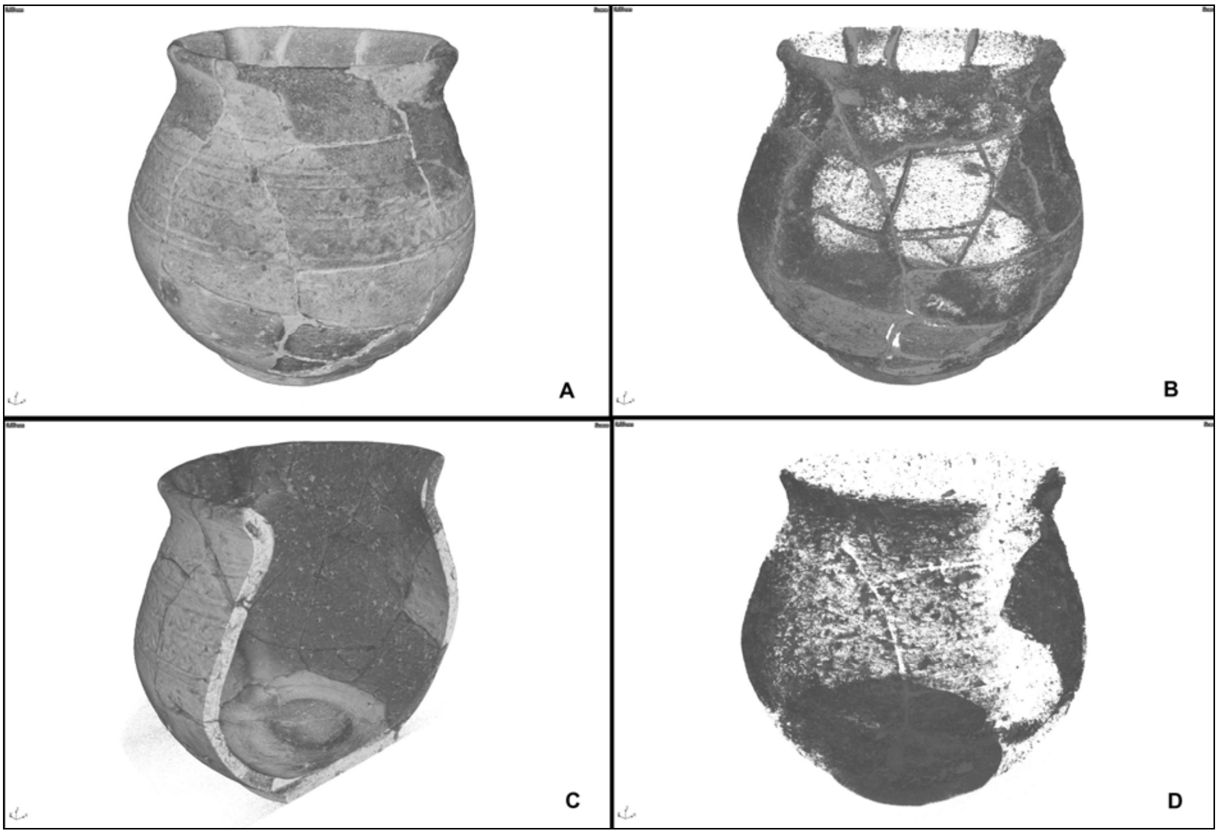

Fig. 9. (a-b) The neutron and (c-d) X-ray transmission tomographic images of an Early Mediaeval pottery

9. ábra: Egy régészeti kerámia (Budakalász, 7. század) (a-b) neutron- és (c-d) röntgentranszmissziós felvételei

Indirect or 'hidden content' studies deal with internal materials of objects which originally were made to be sealed and never to be opened up. These can be hollow sculptures (e.g. Mannes et al. 2014b) or sacred installations, like an altar table in Fribourg (Switzerland) investigated by (Mannes et al. 2014a). It was proved to contain three objects as embedded reliquary. The neutron transmission images showed that the objects show contrast and high attenuation, while they were almost invisible in the X-ray image. It was assumed that they were organic material, such as bone fragments.

A detailed study of a sealed container, an Egyptian sealed pottery (Abraham et al. 2014) successfully represents how imaging techniques can help to understand the usage of a certain object. The pottery was thought to be of some importance, directly linked with the funeral ritual of a pharaoh. This is not a canopy vase, containing viscera of the dead body. It was a hypothesis that the pottery was related with the offerings of food to the dead during the pharaoh's funerals **(Fig. 10a)**. Non-destructive THz computed tomography **(Fig. 10b)** revealed the presence of a mobile and deformable content. XR **(Fig. 10c)** and NR **(Fig. 10d)** visualized the fabrication process and conservation of the pottery more precisely, i.e. the presence of many cracks and damages could be detected in the red clay body. NT determined the sealing method of the jar (lower string-like substrate (linen ball?) and upper clay stopper) and the finer structure of the inner content (organic matter, germinated seeds?).

An example to of an investigation of 'hidden content' of a composite object, a bronze Buddha statue (statue (Buddha Sakyamuni from Bhumisparsa Mudra, West Tibet, 14^{th}-15^{th} c., height 17.1 cm, largest diameter 13.3 cm) is presented by Lehmann et al. (2010b). The filling of the statue is very individual in terms of structure and composition. It has a wooden stick in the middle, which is surrounded by a rolled textile or paper layer and fixed with a metal wire. Flowers or other plants with buds have been filled in the lower part of the sculpture (it is less transparent for neutrons close to the bottom). The sealing plate (wax, resin or gemstone) is more or less absorbent for neutrons. The NR and NT investigations proved that the Buddha statue contains the religious organic articles (central pole with the religious text paper scrolls wrapped around it) essential for a genuine, sanctified Tibetan Buddhist statue. This visualization clearly proves the genuineness of the statue.

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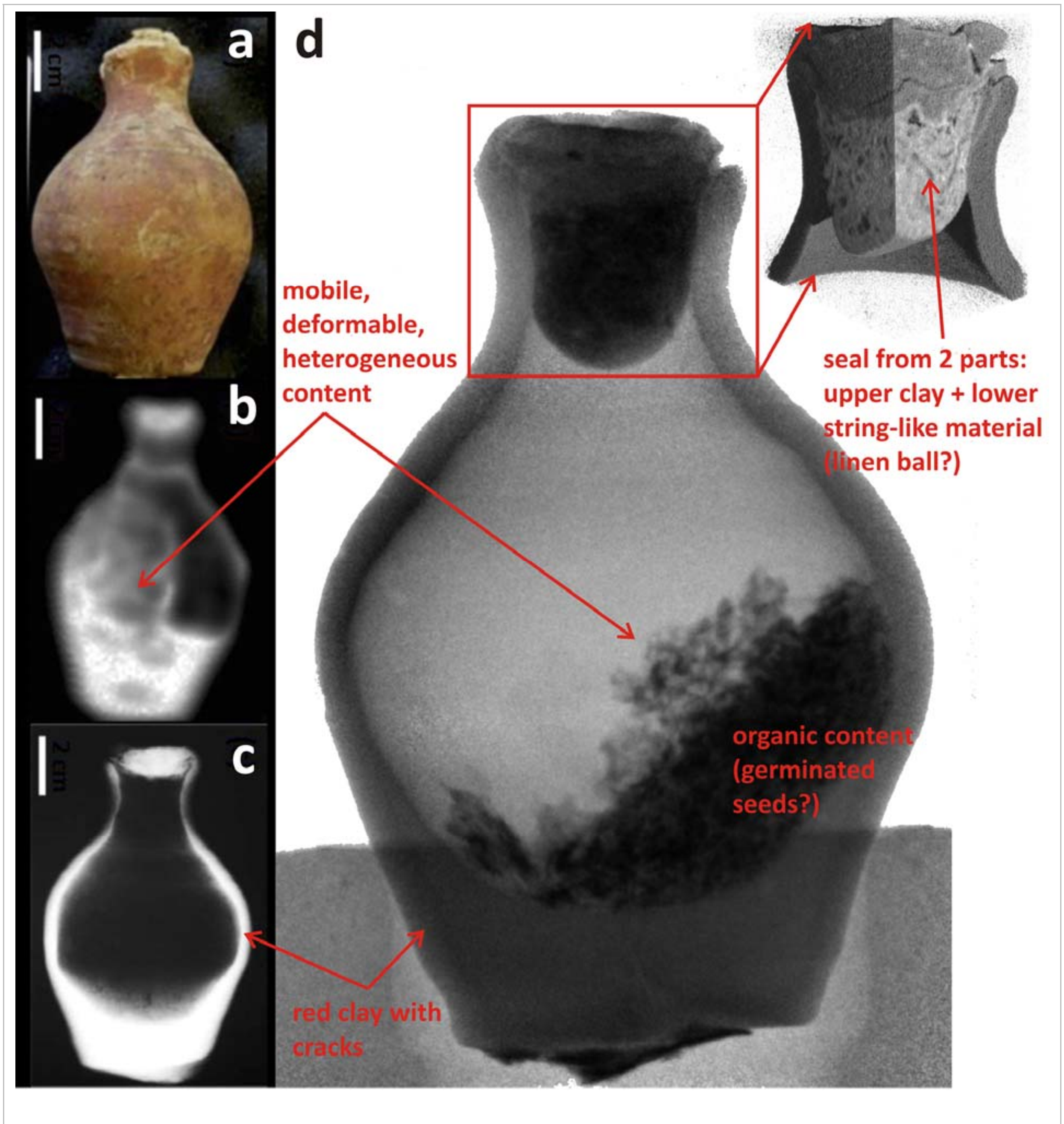

Fig. 10. Imaging study of a closed pottery (15th c. BC, Eighteenth Dynasty Egyptian sealed bottle stored at the Museum of Aquitaine, Bordeaux, France; size: 97 mm×64 mm, scale bar 2 cm). (a) photograph, (b) terahertz transmission image, (c) X-ray radiograph of the whole vessel and (d) neutron radiograph of the vessel and its sealed neck (adapted from Abraham et al. 2014).

10. ábra: Egy lezárt edény (18. dinasztiabeli egyiptomi lepecsételt edény az i.e. 15. századból, Aquitániai Múzeum, Bordeaux, Franciaország; mérete: 97 mm x 64 mm, méretlépték 2 cm) képalkotási esettanulmánya. A teljes edény (a) fényképe, (b) terahertz transzmissziós felvétele, (c) röntgenradiográfiai felvétele, illetve (d) a teljes edény és a nyakrész neutronradiográfiai felvétele (Abraham et al. 2014 nyomán).

Corrosion and conservation studies

The two main topics of such investigations are ‘diagnostic’ and ‘testing studies’. Diagnostic studies focus on the preservation state of artefacts (e.g. is there still original material under the corrosion layer?) (Casali 2006) and the metallic element distribution (Watkinson et al. 2014). Pilot studies model the behaviour of the material under different environmental conditions (e.g. temperature, humidity, pH, pressure, luminosity) and time spans, and also its reaction with conservation preservatives.

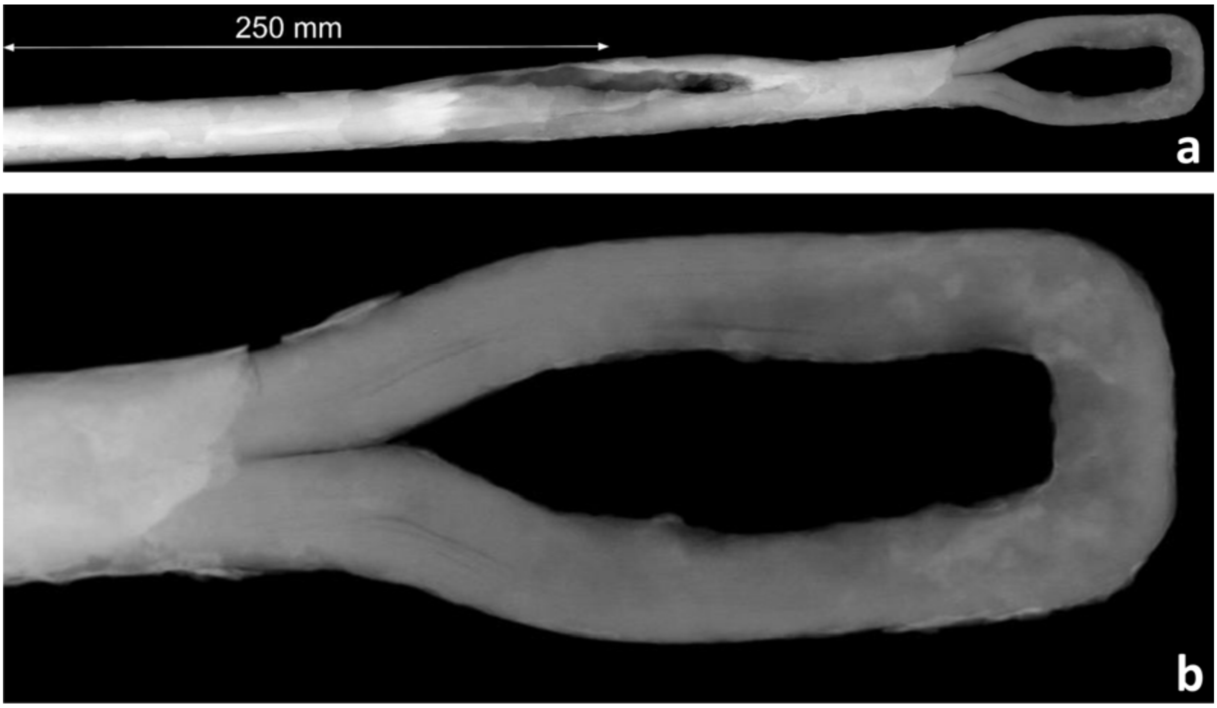

Fig. 11. Neutron imaging study of a wrought iron tie-rod (Milan Cathedral, Italy), (a) The image of the long rod created by as a mosaic of overlapping tiles shows a damaged part and gives some hints about the fabrication technique, (b) the elongated cracks in the material of the long rod are well recognizable.

11. ábra: Egy megmunkált vasrúd (Milánói Dóm, Olaszország) neutronos képalkotási tanulmánya. (a) A mozaikszerűen egymásra lapolt felvételekből készített képen jól azonosítható a hosszú rúd sérült része, illetve a megmunkálás néhány jellemzője is. (b) A rúdban a megnyúlás irányában kialakult, elnyújtott repedések figyelhetők meg.

Metal corrosion is well seen in the radiographic image because the items become less dense and differently textured where corroded, and the corrosion products may diffuse outwards around the object or into the surrounding soil. On the other hand, the neutron attenuation coefficient may decrease due to the presence of oxygen, or increase due to the hydroxyl-groups, relative to the base alloy. In particular this applies to ferrous alloys, which corrode differently from other metals (often a fuzzy halo forms around a more solid core, which can still reveal the shape of the original object in the radiograph).

Masalles et al. (2015) investigated the extension of the corrosion and established the recommendations for the future conservation procedure of a lead sculpture. The wall of the statue contained different lead sheets, nails and some wood grains. Corrosion was visible in NR and NT images, even the surface texture of the corrosion product (lead carbonate) layers from the inside could be seen. These layers appear as fine white bands attached to the inside of the lead sheets. The thorough analysis of the tomographic images can help to obtain an accurate mapping of the areas affected by carbonation and to estimate the percentage of the total internal lead surface affected (about 10-15%). The thicknesses of both the sound lead (1.5 - 2.5 mm depending on the degree of hammering) and the thicker corrosion product layers (0.4 – 20 mm) could be reliably measured. Based on these studies, a better understanding of lead carbonation dynamics became possible. It is clear in the images that the lead sheets begin to show symptoms of plastic deformation only when the growth of the carbonate layer has reduced the thickness of the sound lead to approximately 25%. Corrosion started behind the lead surface from its creation, and with every rise of relative humidity a new fine layer has grown on the interface with the sound lead.

Di Martino et al. (2016) investigated medieval iron nails and structural elements of buildings to understand their corrosion mechanisms and preservation states. It is fundamental, especially in the case of supporting structures, where the continuous mechanical stress enhances the evolution of weakening areas and corrosion spots. A subsequent study, supervised by Di Martino and realized at the RAD imaging station of the Budapest Neutron Center (Hungary), investigated the tie-rods of the Milan Cathedral (Italy) (**Fig. 11.**) in the framework of the IPERION CH project. The iron tie-rods were applied outside of the Cathedral as reinforcement against sideways forces in the cathedral's walls. The exposition to atmospheric agents was the main reason for their replacement. Subsequent investigation of their damages, the deeper material characterization and identification of local defects, was carried out by neutron imaging to complete former studies (metallographic and hardness tests) which revealed extremely heterogeneous structure of the rods. Thermal neutron imaging of long tie-rods and a large iron slab was performed. In **Fig. 11a**, a highly-damaged part of a rod is shown. The corroded parts are separated from each other showing how they were originally assembled. It gives some hints about the fabrication technique, and the way how other rods could principally be attacked by corrosion. The spalling surface layers visible to the naked eye and neutron imaging are mixtures of corrosion products and painting materials. In **Fig. 11b** the elongated cracks in the material of the long rod are well recognizable. These cracks were not visible to the naked eye. The path of the voids follows the curvature of the iron body. It hints that the tensile stress on this part of the tie-rod could have been high enough to considerably weaken the strength of the material.

Lehmann et al. (2005a) observed by means of NR/NT the conservation effect of different oils, waxes, natural and synthetic resins, for the stability-enhancement of degraded wood samples. Due to the high sensitivity of neutrons in detecting hydrogen-containing materials, the visualization of the distribution of the most common conservation preservatives can be obtained by NR with a good spatial resolution. A variety of solvent mixtures (Paraloid B-72 combined with different solvents) was applied to coniferous wood samples and with their investigations the uptake and loss processes were determined, and the 2D-distribution over a period of about 20 hours. The resin uptake depends strongly on the solvent, as well on the proportion of the resin in the mix. The best mixture-composition is where the largest proportion of the resin is retained and its distribution seems to be the most homogeneous.

Prudêncio et al. (2012) applied NT to visualize the inner structure of ancient Portuguese glazed tiles undergoing conservation treatments. To evaluate the efficiency of two different methods of treatment (brushing and immersion in solution), the distribution of the consolidant Paraloid B-72 inside tiles was investigated. The results confirmed that NT is indeed a useful tool for visualization of the inner structure of ancient glazed tiles, to assess penetration depth of consolidant and its distribution inside the tile. Brushing with a solution of 10% Paraloid B-72 in acetone appears to be more efficient conservation technique than immersion. NT showed a greater and more uniform retention of resin inside the tile if the brush is used to apply the consolidant, to increase the cohesion of the object. Thus, brushing appears to be the most appropriate way to apply the consolidant in order to improve structural strength in a homogeneous way through ancient glazed tiles, especially undergone serious degradation (exfoliation).

Conclusions

Neutron imaging is a powerful tool that can be used in archaeometry, whenever the presence, shape and arrangement of organic contents, wrapped in metal or less-heavy matrix, needs to be investigated in situ and in a non-invasive way. Other archaeological materials (e.g. stone, ceramic, glass) can be visualized by this method less effectively. Case studies applying both X-ray and neutron imaging methods confirm their implied complementary nature. The data sets acquired with the two radiation types, when evaluated together, are more useful than the individual subsets. While organic materials (e.g. wood, other plants and textile) yield a very high contrast in the neutron image, they are barely visible in X-ray images. In contrast, metal artefacts show much higher transparency in neutron images, while they give a high contrast for X-rays.

The experimental effort for radiography and tomography is relatively moderate, which makes possible tests with larger series of artefacts. Objects of dimensions up to 20-30 cm can be observed in one single frame; larger objects can be studied in tile mode, by merging images taken with different sample offsets. The ultimate limitation of the object size comes from the sample thickness and the attenuation of the contained materials. Starting with simple transmission images, it can be decided on a case-by-case basis whether a more time-demanding tomography run should and could be completed.

Based on the abovementioned arguments it can be concluded that imaging techniques found their valuable role in the wide (and continuously expanding) methodological repertoire of archaeometry.

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