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Ancient Charm: A research project for neutron-based investigation of cultural-heritage objects^(*)

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Summary. — The objectives and methodology of the Ancient Charm project are briefly described with reference to the state of the art in neutron-based analysis of cultural-heritage objects. The project is expected to develop a set of complementary 3D imaging methods, which will be applied to objects of high cultural-heritage significance selected as a result of a broad-scope archaeological research.

PACS 28.20.Fc – Neutron absorption.

PACS 29.30.Hs – Neutron spectroscopy.

PACS 61.12.Ex – Neutron scattering (including small-angle scattering).

PACS 82.80.Jp – Activation analysis and other radiochemical methods.

1. – Introduction

A large variety of chemical, physical and microstructural techniques are currently employed for the materials study of objects of cultural significance. Major and trace element analyses, which contribute to chemically fingerprint archaeological materials, play a key role in understanding the fabrication techniques, the distribution from the production areas and the use of artefacts in the past. Complementary information can be obtained by phase analysis through diffraction methods. The use of neutrons in this context has received increasing attention in recent years. Neutrons can penetrate layers of several centimetres without substantial attenuation depending on their energy. This property makes them an almost ideal probe for non-destructive analysis of undisturbed and unique objects made from materials as diverse as ceramics, glass, bronze and other metals [1, 2].

While the potential of neutron-based techniques is large, their development is recent. Phase-sensitive neutron diffraction (ND) techniques, used for the phase and microstructure characterization of archaeological ceramics [3, 4] and ancient bronzes [5, 6] clearly

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revealed the potential associated with the deep penetration of the probe. A promising ND application is texture analysis [7, 8] for recording the grain orientation distributions in a material which provide important clues to the deformation history, and thus to historic production steps [9-11] a prominent and first example being the neutron texture analysis of the Early copper age Iceman axe [12, 13]. A recent neutron application with high potential is the mapping of strain and texture in archaeological objects [14] based on the so-called Bragg-edge transmission technique [15].

Thermal or cold neutron tomographic installations provide within a relative short time (from few minutes to few hours) a three-dimensional inside view of few-centimeter thick objects with spatial resolution down to 100 micrometers [16-18]. Similar in principle to X-ray CT, the neutron tomography (NT) technique is based on the measurement of the attenuation of a neutron beam passing through an object. Rotation of the object in the beam and finally data analysis by reconstruction algorithms yields a 3D virtual map. The use of neutrons permits inspection of hydrogenous materials, which are transparent for X-rays. Neutrons allow one to distinguish between elements close in the periodic table of elements, which have similar contrasts with conventional X-ray imaging. Some heavy elements, such as lead, are on the other hand, quasi transparent to neutrons. Moreover, cold neutrons provide enhanced image contrast compared to thermal neutrons. While neutron tomography is an excellent technique for imaging objects of cultural heritage [19], it provides information on the elemental composition only indirectly via energy analysis of neutrons [20].

Neutron-induced prompt gamma activation analysis (PGAA) is a relatively new technique, which has gained importance with the advent of cold neutrons sources at nuclear research reactors [21]. Based on the detection of characteristic gamma-rays from cold neutron capture, it enables a non-destructive analysis for the determination of elemental and even isotopic composition of the irradiated samples on major as well as trace level. The numbers of applications are quite numerous [22].

Finally Neutron Resonant Capture Analysis (NRCA) has been developed in recent years [23] as a joint project of the Delft University of Technology in the Netherlands and the EC Joint Research Centre IRMM in Geel (Be). NRCA makes use of the unique resonance absorption properties of epithermal neutrons. These are commonly available at steady-state reactors and at accelerator-based pulsed sources. Many elements have neutron absorption resonances in the energy range below 500 eV. In this energy range suitable elements include copper, cobalt, silver, tin, antimony, gold just to name a few that are found in common archaeological materials. At higher neutron energies calcium, silicon, iron, nickel and sulphur can be mentioned. Their absorption cross-sections are well known and vary from one element to another and actually vary between isotopes of the same element [24]. Neutron absorption is followed by the prompt emission of a gamma-ray cascade, with total cascade energies up to about 8 MeV. The detailed energy spectrum of the gamma emission is also well known in the case of thermal neutron capture [25], but it can vary between resonances. NRCA has been explored at GELINA, the IRMM pulsed neutron source based on an electron accelerator, and used for analysis of archaeological artefacts to determine the concentration of neutron-sensitive elements [23, 26-33].

2. – The Ancient Charm project

Ancient Charm is a project financed by the European Community “New and Emerging Science and Technology” programme. The project’s title is an acronym for “Analysis by

Neutron resonant Capture Imaging and other Emerging Neutron Techniques: new Cultural Heritage and Archaeological Research Methods". Aim of the project is to develop NRCA and the other neutron-based analysis techniques into non-invasive methods for 3D tomographic imaging of the elemental and phase composition of cultural heritage objects. The interest for this kind of analysis of complex archaeological objects will be investigated with a broad theoretical scope to provide the cultural heritage foundations of ANCIENT CHARM. The new imaging methods that will be developed are:

I) *Neutron Resonant Capture Imaging combined with Neutron Resonance Transmission* (NRCI/NRT). This is the most challenging task in the project. Both gamma emission and neutron transmission measurements of resonant epithermal neutrons will be used to determine the 3D elemental composition of an object.

II) *Prompt Gamma Activation Imaging combined with cold Neutron Tomography* (PGAI/NT). NT is a well-established technique; combined with gamma emission measurements (presently performed only in bulk) it will determine the 3D elemental distributions thus complementing NRCI/NRT.

III) *Neutron Diffraction Tomography* (NDT). At present the most advanced measurements of this kind provide 2D (radiography-type) maps of strains and phases of rather simple metal objects. NDT will be 3D (tomography).

The imaging methods will be applied for an integrated neutron-based 3D imaging of objects of high cultural heritage significance selected as a result of a broad scope archaeological research.

Four large neutron facilities will be used in the project:

1) The Prompt Gamma Activation Analysis (PGAA) facility at the Budapest Research Reactor, which operates with cold neutron source since 2000. The current neutron beam intensity is $5 \cdot 10^7$ n/cm² s.

2) The PGAA and NT facility at the FRM-II Research Reactor in Garching, Germany. A neutron beam of 10^9 n/cm² s will be available from 2007.

3) The pulsed neutron beam of GELINA, Geel, Belgium is obtained from a 150 MeV LINAC accelerator, which has already been used for NRCA.

4) The 800 MeV pulsed spallation neutron source at ISIS, UK is regularly used for Time of Flight Neutron Diffraction (TOF-ND) experiments.

3. – Workplan and methodology

The overall objectives of the project will be achieved through a sequence of more specific tasks as described below.

3.1. Cultural-heritage foundations of neutron-based imaging. – The number of analysis techniques available to cultural heritage researchers is constantly growing, but so is the demand for non-invasive methods to unveil the secrets hidden inside archaeological objects. The analysis methods that will be developed in this project will provide a unique opportunity for a detailed characterisation in terms of the elemental and phase compositions of complex objects, which would be hardly accessible by other diagnostic techniques. Ultimately it will be the availability of the new technique that will provide new opportunities for archaeological research if the project is successful. However archaeological research is also needed in order to highlight the motivations for the new imaging methods and for specifying the requirements for the methods in terms of sensitivity and spatial resolution. In other words, it is the current archaeological re-

search problems that should drive the selection of objects for analysis, which, in turn, set the requirements on the measurement methods. The selected objects should be representative of different classes of objects (*e.g.*, objects with voids, composite materials, composite objects containing a core, jewellery with inlays and multi-layered objects), geographical provenance (throughout Europe) and archaeological/historical periods (*e.g.*, Neolithic, Roman, Early medieval, Late medieval/Renaissance). The information output from neutron-based methods should be unique and not available by other routine archaeometric tools.

The specific objective can be realized using standard methods in archaeological research, such as search in archaeological literature and collections. The project will have access to museums and archaeological sites throughout Europe including the Hungarian Museum, Villa Adriana near Rome, and the National Museum for Antiquities of Leiden. Objects for analysis within the project may come from these institutions or other museums in Europe.

As an illustration of ongoing research on archaeological materials of the Carpathian Basin [34], a preliminary survey identified a number of objects and archaeological problems:

- Production technology, authentication and conservation of hollow objects with small, or not easily accessible apertures (8th century AD cast bronze strap ends, 6-7th century belt mounts [35], toilet knives in a bronze or silver sheath from 7th century AD, 7-8th century metal earrings with globular metal pendant, neolithic vessel filled with red paint, Roman age bronze statuettes).
- Crystalline or composite natural and artificial materials where destruction of the object for petrographical thin section is out of question (Neolithic smoothed stone axes, palaeolithic stone artefacts).
- Solid composite objects with complex structure but simple raw materials: casts containing a core, jewellery with inlays and multi-layered objects (Celtic and Roman coins, iron belt mounts with silver, copper and stone inlays from 6-7th centuries AD, iron swords in an organic—wood and leather—sheath with metal fittings, silver cast fibulas covered with golden plate with stone inlays).

One particular interesting object illustrates the complexity that can be tackled with Ancient Charm. Its image is shown in fig. 1. It is an iron disc fibula with almandine inlays from Kölked-Feketekapu [36], grave A279, from the early Avar period in the Carpathian Basin is a characteristic Merovingian type of dress accessory. A detailed material analysis in terms of elements and phases would be crucial for supporting the dating and the provenance of this fibula. At first sight this object was made out of iron with silver cells and stone inlays although the iron parts may have been just used to repair the original silver fibula. One open question is therefore if iron or silver is of the original fibula. X-ray photographs detect silver cells on the front of the object whereas a possible silver ground plate remains invisible. A fibula originally made of iron could imply an earlier date of the burial site (middle or second half of 6th century AD) and indicate a direct import from a smaller production site in Northeastern France or Northwestern Germany. If the fibula was made out of silver, the repair would indicate a long term use (second half of the 6th, beginning of the 7th century). As a widespread object of Frankish origin the fibula could be a product of a local, of an Alamannic or of a Lombard workshop. The filling material—cementkitt—underneath the stone inlays and a possible gold foil could identify the very workshop of fabrication among several Merovingian producing centres.



Fig. 1. – Photo of the disc fibula with almandine inlays, from the collection of the Hungarian National Museum (origin: Kölked, Hungary; 2nd half of 6th c. AD, Grave A 279; 76.1.45). The diameter of the fibula is about 25 mm.

3'2. Development of the PGAI/NT method for 3D elemental imaging of complex objects. – The PGAA-NIPS facilities [37, 38] of Institute of Isotopes are constructed at the end of a guided cold neutron beam from the 10 MW Budapest Research Reactor. In the past few years, the bulk elemental composition of many archaeological objects have been studied such as Roman bronze objects [39], silver coins [40], chipped stone tools from the Paleolithic [40], polished stone tools from the Neolithic [41] and provenance of Amerindian pottery figurines [42].

To demonstrate the applicability of PGAA for 3D mapping of elemental composition, a pilot 2D mapping experiment was performed on a composite demonstration object (copper wire in Teflon cylinder filled with quartz powder) showed encouraging results and indicated some necessary hardware improvements that would be required in order to develop an elemental tomographic imaging technique. The spatial resolution achievable could be at the millimetre level, considering that the product (Acquisition time \times Spatial resolution) will be limited by the neutron flux and the number of photon detectors around the object.

Neutron tomography (NT) provides images with much higher resolution but has no specific elemental sensitivity. An effective experimental procedure for combined 3D PGAI/NT imaging will have to be developed. In order to improve the sensitivity, the neutron beam could be collimated in one direction only and a tomography of the object performed by scanning the other direction accompanied by a rotation of the object.

Dedicated analysis software will have to be developed for combining PGAI and NT data. For instance the existing PGAA gamma peak analysis software should be modified

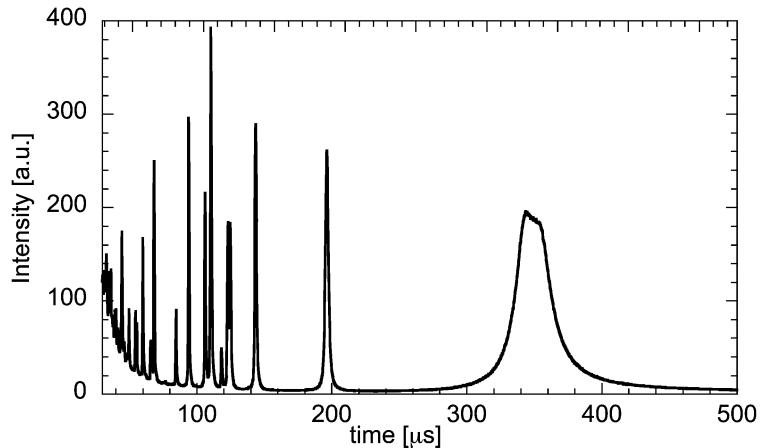


Fig. 2. – Time-of-flight spectrum recorded from a silver sample in a pilot NRCA experiment performed on the VESUVIO spectrometer at the ISIS spallation neutron source. A prototype YAP (YAlO_3) scintillator was used as gamma detector.

so as to include automated fitting of a large number of peaks where only the position and intensity of the peaks is required for PGAI.

3.3. From NRCA to NRCI/NRT. – The step from NRCA to the new NRCI, and the integration of NRCI and NRT will require the development of new NRCI and NRT detector systems as well as of new shielding and beam collimation. New simulation and analysis tools for Neutron Resonant Capture Imaging data will also be developed starting from the present state of the art in NRCA.

The new imaging technique builds on the experience gained in NRCA on the GELINA neutron source. Energies (E_n) of captured neutrons are determined by a time-of-flight (TOF) arrangement commonly available at pulsed neutron sources. That is $E_n = (1/2)m(L/T_n)^2$, where m is the neutron mass, L the distance of flight and T_n the TOF between source and object. Typically the available time span is of order 1 to 20 milliseconds depending on the number of neutron pulses per second. The neutron energy range depends on the length of the flight path (L). For instance for $L = 10$ m the energy range is of order 0.2 eV to 30 keV. The detection of prompt gamma rays provides a time signal for the TOF. A typical NRCA spectrum on GELINA [29, 32, 33] features a large number of resonance peaks of different elements, corresponding to neutron energies in the range of 1 to 2500 eV. The energies and intensities of the resonance peaks characterize the elemental composition of the sample.

Pilot NRCA experiments have also been performed on the VESUVIO spectrometer at the ISIS spallation neutron source. The beam geometry and the neutron energy spectrum on VESUVIO are similar to those at GELINA. Main differences are a higher neutron flux and a higher gamma background on VESUVIO, the latter of which limits the sensitivity of the measurements. The practical limit for quality measurements will not be much above a maximum peak energy of 300 eV, as observed in pilot experiments where reference samples were analysed in a NRCA-like setting using a prototype YAP (YAlO_3) scintillator as gamma detector. The pilot experiment shows that gamma background needs to be reduced, as expected. An example of recorded TOF spectrum is shown in fig. 2.

The step from NRCA to resonant neutron imaging cannot be achieved by a quantitative extension of the technique already developed such as could be achieved by scanning a collimated neutron beam across an object. Instead we intend to develop a new approach of combining gamma emission measurements with resonant neutron absorption measurements. In this way the mapping of chemical element distributions by means of NRCA will be achieved by the combined use of two detection techniques, i) recording the emitted gamma cascade that follows neutron capture with a collimated “pencil” beam, and ii) recording the transmission time of flight spectrum with a 2D position-sensitive neutron detector (PSND) in a straight-through collimated but broad primary neutron beam. Gamma and neutron radiation, respectively, are measured as a function of the neutron flight times, *i.e.* the neutron energies. Most of the gamma emission will occur at those times when resonant neutrons are absorbed in the sample. That is to say, peaks in the gamma TOF spectrum correspond to dips in the transmitted neutron spectrum. The collimated pencil beam for gamma detection defines a cord inside the object over which integrated information is obtained. A 2D-image of the element distribution is achieved by (YZ)-scanning the object across perpendicular directions to the incoming neutrons, using the intensity of the gamma peaks as a function of scan variables. For 3D imaging, spectra are recorded for a series of object orientations by an additional omega (ω) rotation scan. In principle gamma measurements should have a better sensitivity whereas neutron measurements will benefit from a substantial boost in signal intensity due to the open beam geometry. The combined use of the two measurements is what we call Neutron Resonant Capture Imaging combined with Neutron Resonance Tomography (NRCI/NRT). There is a strong similarity here with PGAI/NT, which will be developed in the project before NRCI/NRT. At the same time the two techniques are complementary because they have different sensitivity to different elements because of the underlying cross-sections. Thus PGAI is best for the elements with high thermal neutron absorption cross-section, especially for some light elements like H, B, Cl, Ti and also Cd, Hg, Nd, Sm, Eu and Gd. On the other hand, NRCI is best for heavy elements like As, Ag, Sb, Sn, Au, Cu. Also NRT is an element-sensitive tomography. Compared to cold neutron tomography, the spatial information of NRT will be limited to millimetre scale due to the complexity of the detection system. Usually, there are several resonant energies for the same element, with different absorption cross-sections. Thus, construction of 3D maps with different resonances would be an immediate use of the unique penetration properties of resonant neutrons mentioned earlier.

Similarly to conventional NT, NRT with a broad primary neutron beam requires good angular collimation, and a 2D position-sensitive neutron detector (PSND) behind the sample. For 3D imaging, time-of-flight spectra are recorded for a series of object orientations by an omega (ω) rotation scan. PSND are in regular use at ISIS for thermal neutron measurements. A new detector will have to be developed for epithermal neutron detection.

A related requirement in NRCI is the modelling neutron and photon interactions in large, complex archaeological objects. Neutrons suffer from scattering in the sample before resonance capture, and gamma-rays suffer from interaction in the sample after emission. These processes will be modelled to the level of detail required to match the desired experimental accuracy. Moreover, the gamma and neutron detector geometries will be included in the simulations. We will simulate NRCI data for both set-up modes and reconstruct 3D images. Thereafter, it can be decided which set-up is the most efficient, what the limitations are, and whether a differentiated approach with coarse and fine spatial resolution can be employed.

For the computer simulations we will use the MCNP code [43] and, especially, the GEANT4 code, a well-developed, well-supported and validated code for radiation transport calculations [44]. For the simulated and measured resonance capture data analysis the code REFIT is at present the most appropriate [45]. This programme can fit measured time-of-flight spectra after suitable data reduction. It corrects for neutron-energy-dependent self-shielding in the sample, capture of neutrons after scattering in the sample, and Doppler broadening of the resonances due to thermal motion of the capturing nuclei. However, large irregular objects cannot be treated well with REFIT. Furthermore, for the large amounts of data recorded in NRCI we cannot handle all the resonances as a function of object position/orientation individually. Therefore we will improve the data analysis by introducing a routine for the data reduction, adapting REFIT to the large-size objects, and making the analysis automatic, at least in the lower neutron-energy range (1–500 eV) where resonances are in general well separated. Finally the data resulting from the user-friendly “New REFIT” code will be used as input to a 3D reconstruction code.

3.4. Neutron Diffraction: from bulk analysis to tomography. – Additional, and complementary, information can be obtained by diffraction techniques, *e.g.*, on the microstructural properties of the object. This additional 3D information can be gathered using the same instrumentation developed for NRT but using thermal neutrons.

For low neutron energies, typically outside the range of resonances, the neutron transmission varies as a result of the crystalline structure exhibiting so-called Bragg-edges. Bragg-edge pulsed neutron transmission measurements are an existing technology, already used for residual strain mapping on ENGIN-X at ISIS [15]. The neutron transmission detector developed for NRT will record Bragg edges and thus will provide information on structural variations within the object. Moreover, neutron diffraction patterns can be recorded with the multi-detector banks of existing TOF diffractometers such as GEM and POLARIS. Thus the same device used for NRCI/NRT will also provide the simultaneous 3D mapping of the mineral or metal phase composition, of the crystallographic texture of each phase as well as of microstructural properties. This is what we call neutron diffraction tomography (NDT), which is a novel extension of a diffraction analysis of archaeological materials.

Most neutron diffraction analyses on cultural heritage materials performed in recent years would have benefited from extra bulk elemental information. It has to be seen, however, if the two techniques “see” the same material in the sample. Diffraction is phase and structure sensitive and provides information from the crystalline part of a sample. Activation analysis is element sensitive and provides information from crystalline and non-crystalline portions of the object, however it does not see common elements (Ca, C, Al, Si, O) and oxides, carbonates, and sulphates of metal corrosions (O, C, S). The methods are thus to be considered complementary.

3.5. Integration of imaging methods. – A specific project objective is the comparison of the different imaging techniques and their combination. This is achieved by a comparative study of the involved techniques, the definition and realisation of calibration tools, the implementation of iterative reconstruction algorithms and the development of methods for integration of the data from different techniques before reconstruction.

The state of the art in reconstruction algorithms is quite developed and textbooks have been available for a long time [46, 47]. Medical imaging is often the driving force as is the case, for instance, of Single Photon Emission Computed Tomography (SPECT) [48-50].

The reconstruction code for tomographic images can indeed be based on the filtered back projection algorithms or the maximum likelihood expectation maximization as used in medical imaging.

Commercial software packages for 3D visualization exist [51]. The innovative aspect here will not be in the reconstruction or visualization methods but rather in the simultaneous and comparative visualization of the 3D maps from PGAI/NT, NRCI/NRT and NDT. Even more important, it is the combination of the data before reconstruction that can be much more fruitful. Two examples:

i) The NT results can be used as a first draft to define the region of high interest that will be scanned with PGAI and NRCI. These smaller regions can thus be imaged faster and with a better spatial resolution that would be chosen if the full object had to be scanned.

ii) NT yields the 3D distribution of the neutron attenuation. This 3D distribution can directly be used to correct PGAI data for neutron attenuation. Such correction, similar to matrix correction in other methods (like PIXE) is required for determining the spatial distribution of the elements rather than the average composition of the object.

Duration of measurements and neutron beam times can be kept to a minimum by limiting the analyses to parts and object regions that are of relevance for addressing the archaeological questions. Finally, the results from the different techniques will be integrated to construct 3D elemental and phase distributions of the whole object or part of it.

4. – Conclusions

The last activity in the project will be the analysis of real cultural-heritage objects selected as suitable candidates for NRCI/NRT and other neutron-based imaging techniques. The samples will be transported to the neutron facilities for the measurements. The individual techniques will be applied to the objects and their information output will be integrated. The provision of 3D spatially resolved images of elemental and phase composition of archaeological objects will mark the conclusion of the project.

Hopefully the 3D mapping analysis of the selected objects will not only represent a first real application of NRCI/NRT and of the other neutron-based techniques developed in the project, it will also provide important clues for the archaeologists to work on. This would represent a success of the new analysis techniques beyond the technical achievements of the hardware and software tools.

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APPENDIX

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