NINMACH 2013

1st International Conference on Neutron Imaging and Neutron Methods in Archaeology and Cultural Heritage Research

Abstract Booklet

9 - 12 September 2013

Physik Department, Technische Universität München, Garching, Germany

NINMACH 2013 - Abstract Booklet

Editor:	Dr. Burkhard Schillinger
Local organising team:	FRM II: Dr. Burkhard Schillinger, Dr. Jürgen Neuhaus, Dr. Petra Kudejova, Elisabeth Jörg-Müller, Vladimira Vodopivec Prof. Dr. Rupert Gebhard (Archäologische Staatssammlung München)

Technische Universität München
Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II)
Lichtenbergstraße 1
85748 Garching, Germany
August 2013

Cover:	left:	Bronze relief by Lorenzo Ghiberti: Ancient Charme project
	right:	FRM II Ramona Bucher, TUM / MLZ
Cover USB-Stick:		Belt mount (7th century): Zsuzsanna Hajnal Radiography taken at FRM II: Ralf Schulze

Ladies and Gentlemen,

Let me give you a warm welcome here on the research campus for the ”International Conference on Neutron Imaging and Neutron Methods in Archaeology and Cultural Heritage Research“, which takes place for the very first time, and which will bring together so seemingly different fields of science like archaeology and material sciences. As patron of the conference, I am honored to welcome scientists from all over the world for this very special conference here at the Technische Universität München, where also the International Atomic Energy Agency participates as a partner.

I am especially delighted that it was scientists from the Technische Universität München who have joined these apparently so distant disciplines: Archaeology und Physics. It is and always has been the aim of Technische Universität München to join disciplines, because innovation happens at the interfaces between them, today more than ever before. They are the sources and seeds for synergies, new ideas and new cooperations.

Radiography, tomography and many other analyzing methods with neutrons have been state of the art for a long time on many, but preferably technical fields. Apart from some exotic examples, one has the impression that these methods have not been used in the investigation of the cultural heritage of mankind. But in spite of this first impression, vast knowledge, experience and progress have silently developed over the years in this particular field. It will be the merit of this conference and its participants to gather this treasure of experience, to make it visible and to make it available to the scientific community.

By hosting this conference, the FRM II reactor, which became critical in 2004 as Central Scientific Institute of the Technische Universität München, now adds an additional milestone to its history under the name Heinz Maier-Leibnitz Zentrum. The Instruments and Methods for Neutron Imaging which have been developed with significant contribution by scientists of the Technische Universität München have now become so sensitive that they can, at the high-flux neutron source FRM II, reveal details never known before. But they can now also deliver better results for standard measurements at medium and low power sources throughout the world. The objects are not destroyed or damaged during the examination, which is even more important in Cultural Heritage Research as the guardian of these important treasures of human history than in other disciplines. During this conference, archaeologists, art historians and conservators from renowned institutions will report about their experiences with neutron methods and how they benefitted from them. The interesting examples that you will hear about during this conference will certainly convince even more of your colleagues in the future.

In this sense, I wish you all much of success and a fruitful conference, which may be established, originating from the Technische Universität München, as a continuous meeting place for

the scientific exchange between neutron and cultural heritage researchers.
The implementation of the research neutron source is a culmination of my work as President of this university. With great enthusiasm and with conviction I have enforced the project for many years against the resistance of “self-proclaimed experts“, with the unreserved support of the Bavarian government under Prime Minister Edmund Stoiber. With the research neutron source, this government has made successful policy for Germany as well as for the international scientific community - my greatest respect for that also at the present day.

Therefore, I even more regret that it is not possible for me to be present today and to find out with you again, “Neutrons are light!“

Professor Wolfgang A. Herrmann
President of the Technische Universität München

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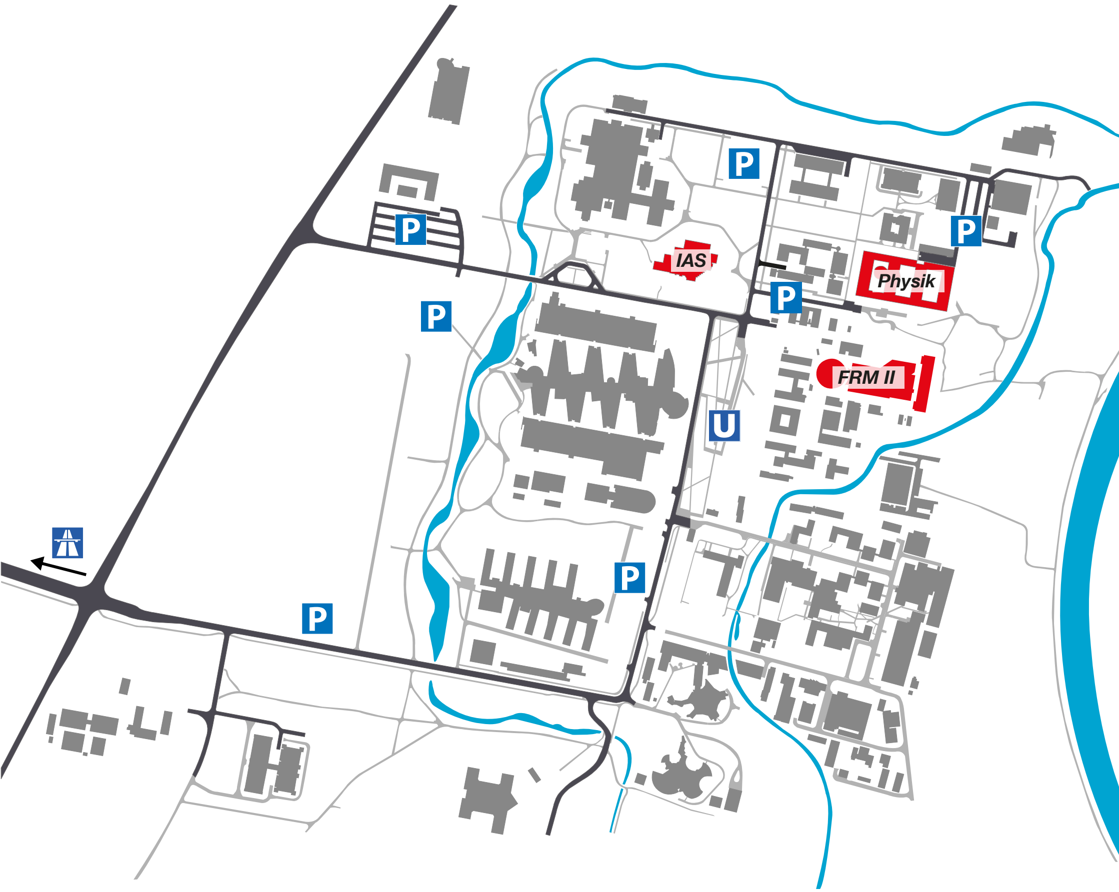
All sessions - except for the poster session - will be held at the Physics Department of Technische Universität München (address: James Franck-Straße, 85748 Garching near Munich). Coffee and lunch breaks will be hosted there, too.

The poster session will take place on Monday, 9 September 2013 at 6 pm, in the neighbouring Institute of Advanced Study (IAS) at the so called Faculty Club (4th floor).



Excursions on Tuesday, 10 September, afternoon:

Guided Tour through FRM II (limited number of visitors)

17:30 h For the guided tour through FRM II, we need to know your private address in advance and you need to present a valid personal identity card respectively passport for Non-Europeans in order to be granted access to FRM II. Staff at the NINMACH registration desk will assist you with details. The guided tour will take 2 to 2 ½ hours.

or

Visit of

Bavarian National Museum Restoration workshop (limited number of visitors)

www.bayerisches-nationalmuseum.de
(Oettingenstraße 15, 80538 München)

or

Neue Pinakothek München

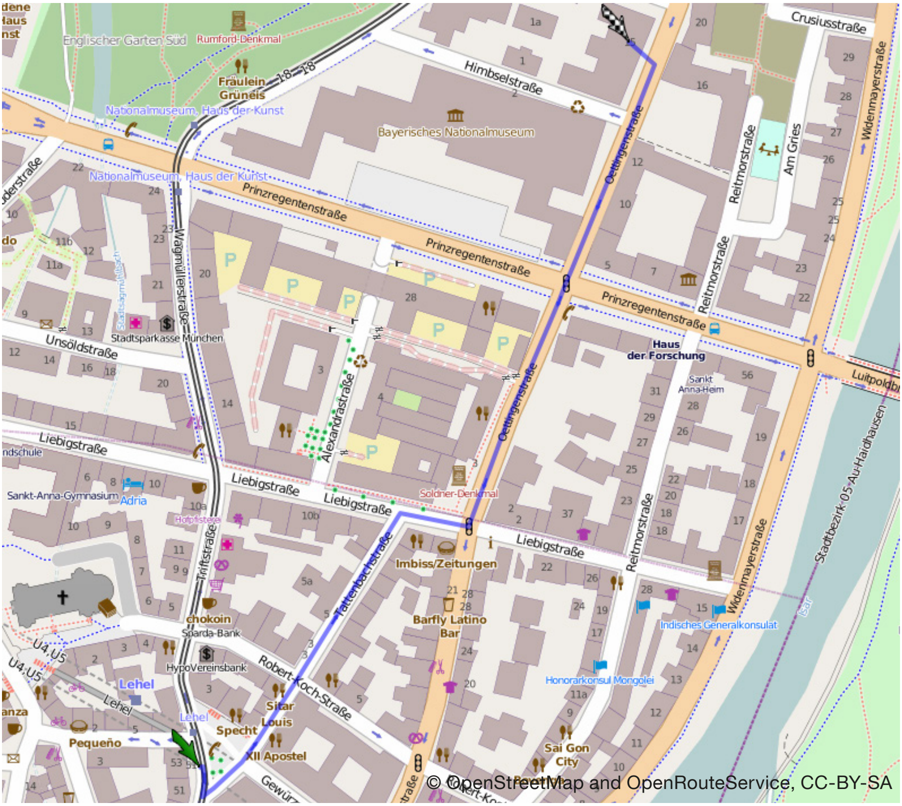
www.pinakothek.de/neue-pinakothek
(Bayerische Staatsgemäldesammlungen Kunstareal München, Barer Straße 29,
Eingang Theresienstraße, 80333 München)

For the latter two ones we will leave together at about 5 pm travelling to Munich downtown by public transport. According to the place you wish to visit you may join one of the two groups. Transport costs to Munich downtown and entrance tickets to Neue Pinakothek are on your own. The guided tours are free.

Every tour will end about 8 pm.

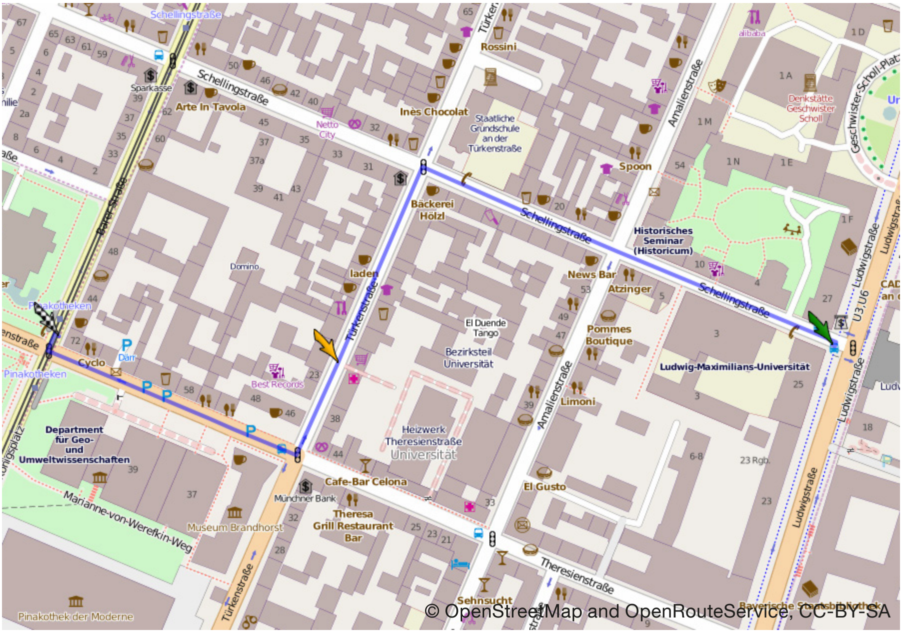
Destination: Bavarian National Museum - Restoration workshop

by metroline U6 from Garching Forschungszentrum to stop Odeonsplatz. Change to metroline U5, direction “Neuperlach Süd“, exit at stop “Lehel“. A walk about 10 minutes to the Restoration Workshop.



Destination: Neue Pinakothek

by metroline U6 from Garching Forschungszentrum to stop Universität. Then, a walk of about 10 minutes to Neue Pinakothek.



In order to get back to Garching in the evening please take metroline U6, direction Garching Forschungszentrum, exit

- at stop Nordfriedhof for Hotel Leopold
- at Garching-Hochbrück for Motel One
- at Garching for Hotel am Park / Hotel König Ludwig / Hotel Hoyacker Hof

Conference Dinner on Wednesday evening (11th of September)

A bus departing from Garching Maibaum (next to the Hotel am Park/Hotel König Ludwig) at **18:20 pm** and from the FRM II gate at **18:30 pm** takes you to Weihenstephan. The State Brewery of Weihenstephan is said to be World's oldest brewery. Tradition, expertise in brewery and hospitality makes Weihenstephan a renowned and popular place to visit.

Important – Information on Munich public transport:

If you wish to cover both the Garching area and Munich (downtown), the best ticket to buy

- is the single day ticket *Single Tageskarte München XXL* at EUR 7,80 per day for **one** person
- or the partner day ticket *Partner Tageskarte München XXL* at EUR 13,60 per dayfor groups of **up to five** persons (who have to stay together, of course).

For short trips from your Garching Hotel to the conference venue you may use the so called *Streifenkarte* stripe ticket

As to the number of stripes you have to validate, please ask at the reception desk of your hotel.

In any case, please do not forget to validate your ticket.

Haltestelle	Fahrzeit in Minuten
Garch.,Forschungsz.	
Garching	03
Garching-Hochbrück	05
Fröttmaning	09
Kieferngarten	11
Freimann	13
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Uhr	Montag - Donnerstag	Freitag	Samstag	Sonn- und Feiertag	Uhr
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4	16 ✕ V29	16 ✕ V29	16 ✕ V29	16 ✕ V29	4

✕ = bis Kieferngarten
■ = bis Münchner Freiheit
v = nur während der Vorlesungszeit der TU Garching (10.-21.12.2012, 07.01.-08.02.2013, 15.04.-19.07.2013 außer 21.05., 14.10.-23.12.2013)
V11 = Nicht in der Nacht vom 31.12.2012/1.1.2013 (siehe Sonderfahrplan)
V29 = nur Faschingsendspurt (Nächte 8./9. - 11./12.02.2013)
V93 = nicht Nächte vor Feiertagen, nicht 10./11. und 11./12.02.2013
V97 = Nächte vor Feiertagen, auch 10./11. und 11./12.02.2013

www.mvv-muenchen.de

Änderungen vorbehalten   SMS-Abfahrten: 460 an 0175 4321409   SWM/MVG   Tel.: 01803/442266 9Ct./M

Scan to see the next departure

Sponsors

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RGZM	www.rgzm.de
LOT QuantumDesign	www.lot-qd.de

The organizers would like to thank the following Sponsors for their support:

- The International Atomic Energy Agency (IAEA) for their patronage and for travel support of selected participants
- The Deutsche Forschungsgemeinschaft (DFG) for travel support of selected participants and support for conference rooms rental
- The Imaging company LOT Oriel for additional support

Why use neutrons?

An Overview of Neutron Imaging and Neutron Sources

Contrary to X-rays, thermal neutrons react not with the electron shell of atoms, but with the nuclei themselves. Huge contrast may appear even among isotopes of the same element. Neutrons react very sensitive to Hydrogen, while penetrating most metals easily; this makes neutron imaging a complementary tool to X-ray imaging, often showing the opposite contrast.
A few elements like Cadmium and Gadolinium show such high contrast that they can be used as contrast agents in liquid form to detect tiny cracks and hollows.

The downside of neutron imaging is that neutrons are not as easily obtained as X-rays – large facilities such as nuclear reactors or accelerators are required to obtain neutron radiation in intensities comparable to common X-ray sources.

The talk will give an introduction about the properties of neutrons and neutron imaging as well as scientific neutron sources.

Primary authors : SCHILLINGER, Burkhard ()

Co-authors : Mr. REIMANN, Tommy (FRM II) ; SCHULZ, Michael () ; Mr. BAUSENWEIN, Dominik (TUM/FRM2 ANTARES)

Presenter : SCHILLINGER, Burkhard ()

Neutron imaging of museum objects - the user‘s perspective

Research is one of the three principal activities of a museum, besides education and conservation of the collections. All three have to be understood in the widest possible sense.
Scientific research on the objects would in turn only represent a relatively small part of a museum's overall research activities, the focus being usually on historical, art historical, archaeological or other research directly in line with the museum's subject area.
Neutron imaging and analysis of cultural artefacts are relatively new methods and only represent therefore a marginal technique on the fringe of a museum's activities.

Why would under these circumstances neutron imaging be interesting to a museum? This paper presents examples from the Geneva Ethnographic Museum illustrating how looking inside an object contributes to our understanding of the museum's collections, and where, despite the exceptional logistical effort required for the analysis, neutron imaging becomes important or even indispensable.

Primary authors : Dr. ANHEUSER, Kilian (Musée d'ethnographie de Genève)

Co-authors :

Presenter : Dr. ANHEUSER, Kilian (Musée d'ethnographie de Genève)

Contribution to the knowledge about cultural heritage objects by means of neutron and X-ray investigations

Objects from cultural heritage have to be investigated mainly non-destructively or even non-invasively. In this respect, X-ray radiography has been used as a valuable and effective method since decades. The method provides art historians and experts active in cultural heritage studies with facts of interests to complete their vision of the studied topic.

In the talk, we want to introduce neutron imaging as an alternative tool for non-invasive material studies in respect to different classes of cultural heritage objects. Its power is given by the alternative attenuation behavior of neutrons in comparison to X-rays, where most of the heavy metals get transparent but organic materials show a high contrast due to the neutron scattering at hydrogen.

In some cases, neutron imaging is the only method to get the needed information either given by the transmission ability of neutrons or the high contrast from light elements, which are not visible with X-rays.

For other objects X-ray and neutron imaging can be applied sequentially and complementary at our beam line NEUTRA. We will provide some general findings from our long-term collaboration with museums partners who have often a different research approach. Natural scientific facts are only one part in the puzzle within cultural heritage research.

In our presentation, we will report about successful studies covering a lead-sealed violinist sculpture from Spain by the famous artist Pablo Gargallo, a stony altar table from Fribourg (CH), Tibetan Buddha sculptures from 15th century, casting attempts from experimental archaeology, combined utilization of X-ray and neutron tomography on a medieval sword recovered from Lake Zug (CH) and the study of block excavation from a Swiss region. The presented studies will give an overview on the broad spectrum of cultural heritage topics, which can be studied using neutron imaging methods.

The results should encourage potential users of neutron imaging to perform trials at our beam lines and to understand the potential for future dedicated studies.

Primary authors : Dr. MANNES, David (Paul Scherrer Institut) ; Dr. LEHMANN, Eberhard (Paul Scherrer Institut)

Co-authors :

Presenter : Dr. MANNES, David (Paul Scherrer Institut)

Advanced hard X-ray imaging techniques applied to archaeology and palaeontology: a tool complementary to neutron imaging

X-ray microtomography, synchrotron radiation, microfocus sources, phase-contrast imaging, quantitative analysis, cultural heritage applications.

Summary :

Imaging techniques play an important role in several research fields such as medicine, material science, geology, cultural heritage, food science, and in industrial applications. In recent years great interest has been posed on X-ray computed microtomography (m-CT) techniques, based on both microfocus and third generation synchrotron radiation sources. In fact, m-CT is a nondestructive characterization technique producing three-dimensional (3D) images of the internal structure of objects with a spatial resolution at the micron- and submicron- scale. The investigation of specimens can be performed directly in the 3D domain overcome the limitations of stereological methods usually applied to microscopy-based analyses. Moreover, m-CT techniques enable to get 3D images of the internal core of a sample in a non-destructive way, more suitable for further analyses and for precious or unique samples (fossils, archeological finds, etc..). An intriguing challenge lies on the extraction of quantitative measures directly from these kinds of images. However, accurate image processing and analysis methods for an effective assessment of morphological and textural parameters are still an open issue in several applications.
The talk will illustrate a short overview of the potentialities of hard X-ray imaging techniques applied to archaeology and palaeontology, and their complementary aspect with respect to neutron imaging.

Primary authors : Dr. MANCINI, Lucia (Elettra - Sincrotrone Trieste S.C.p.A.)

Co-authors :

Presenter : Dr. MANCINI, Lucia (Elettra - Sincrotrone Trieste S.C.p.A.)

Computed tomography meets highlights of the state archeological inventory – virtual excavation and reconstruction

The Archäologische Denkmalpflege Baden-Württemberg has used computed X-ray tomography (XCT) routinely since 2009. This non-destructive method is particularly advantageous in the case of excavated blocks containing finds of different materials. Some astonishing results have been achieved with it over the past years, especially on composites of metal and organic materials. One of the exigencies of daily routine at the archeological heritage preservation authority is that objects have to be processed by a specified deadline. Not only does the preparation of archeological finds for evaluation have to be carried out within an ever shorter time, the volume of finds is also increasing year by year. This made it necessary to look for alternative, yet also highly precise processing methods that could be used alongside conventional approaches. XCT has proved helpful here both in regard to its costs in time and the
insights it offers.

Due to finds having lain the ground, especially in the case of burials, their materials are often found compressed in layers only a few centimetres thick. Separating individual layers of finds often leads to destruction of their context. XCT is well suited for documenting individual find layers because it permits their virtual separation. To be able to visualise organic structures it is necessary to have a knowledge of the specific characteristics of the material in question, since they cannot be identified otherwise.
When objects are so poorly preserved from having lain in the ground that they can hardly be removed from the surrounding earth XCT often provides the only way to obtain a three-dimensional image of their appearance. In special cases neutron computed tomography (NCT) is used, which has been found to produce some very good results in the area of mineralised textiles. The lecture deals with the possibilities of and limits to the innovative methods of X-ray and neutron computed tomography and shows different results.

Primary authors : Mrs. EBINGER-RIST, Nicole (Fachgebietsleitung Archäologische Restaurierung Regierungspräsidium Stuttgart Landesamt für Denkmalpflege Referat 84/ Archäologische Restaurierung)

Co-authors :

Presenter : Mrs. EBINGER-RIST, Nicole (Fachgebietsleitung Archäologische Restaurierung Regierungspräsidium Stuttgart Land

Non-invasive characterization of ancient Japanese helmets through Neutron Imaging

Japanese swords and armours have always been very attractive to the western culture because of their distinctive styles and technological features, which are considerably different from the corresponding objects familiar to western culture. Among the various components of the samurai's armour, the kabuto assumes, for obvious reasons, considerable importance. The kabuto of the traditional samurai armour is a kind of helmet, typically made of steel components, assembled in ways peculiar to the particular manufacturing school [1]. Here, the technological skill of the craftsman might reach the best results in joining lightness and effectiveness to defend the most important organ of the samurai's body: the head. In addition, being the most visible part of the warrior from a distance, the helmet assumed also the role of the distinctive sign of a leader in battle. Thus, not only effectiveness, but also elegance and visibility became necessary qualities for the samurai's helmet [2].

Much literature exists about Japanese swords, but far less is known about the technology of Japanese helmets. So, an international team of scientists and curators decided to work together, to investigate the construction of one of the most critical components of the Japanese armour. These objects are quite rare and, when found in museums, are usually in an excellent state of conservation, being considered masterpieces representative of Japanese culture. For this reason, any detailed study of these artefacts must rely on non-invasive techniques and it was decided that thermal and cold neutron techniques should be employed for this investigation.

Here, we present novel results from a non-invasive examination, conducted thought neutron imaging techniques, of two kabuto attributed to the 17th Century. The two chosen helmets are antithetical in their complexity: while the first one is characterized by a unusually complex shape, the second is made of a large set of relatively simple components, assembled in a very complex structure. Preliminarily, neutron diffraction techniques have been applied to the study of kabuto to obtain detailed information on bulk properties (e.g. phase composition, texture, residual strain distribution) [3]. Complementary, neutron imaging experiments (radiography and tomography) carried out at the ICON and NEUTRA beamlines, operating at the neutron source SINQ (CH), have allowed to determine the inner metal structure and manufacturing techniques of these beautiful examples of past technology, revealing some otherwise invisible details [4].

1. Kozan Sakakibara 1800 The manufacture of armour and helmets in 16th century Japan:(chûkokatchû seisakuben),; translated by T. Wakameda, revised by Koop, A. J., and Hogitarô Inada, 1912; revised and edited by Russell H. Robinson, The Holland Press, London 1963.
2. C. Sinclaire 2004 Samurai: The Weapons and Spirit of the Japanese Warrior (The Lyons Press, GUILFORD).
3. A. Fedrigo et al. 2013 Neutron diffraction characterization of Japanese armour components, J. Anal. At. Spectrom., 286, 908 – 915.
4 F. Salvemini et al. 2013 Revealing the secrets of composite helmets of ancient Japanese tradition (accepted on EPJ plus).

Summary :

Primary authors : Mrs. SALVEMINI, Filomena (Istituto dei Sistemi Complessi-Consiglio Nazionale delle Ricerche)

Co-authors : Mrs. FEDRIGO, Anna (ISC-CNR) ; Dr. GRAZZI, Francesco (CNR-ISC) ; Dr. ZOPPI, Marco (Consiglio Nazionale delle Ricerche, Istituto dei Sistemi Complessi, 50019 Sesto Fiorentino (FI), Italy) ; Dr. WILLIAMS, Alan (Wallace Collection, W1U 3BN London, United Kingdom) ; Dr. CIVITA, Francesco (Museo Stibbert, 50100 Firenze, Italy) ; Dr.

SCHERILLO, Antonella (Science and Technology Facility Council, ISIS Neutron Source, OX11 0QX Didcot, United Kingdom) ; Dr. VONTOBEL, Peter (Paul Scherrer Institut, SINQ Neutron Source, 5232 Villigen, Switzerland) ; Dr. HARTMANN, Stefan (Paul Scherrer Institut, SINQ Neutron Source, 5232 Villigen, Switzerland) ; Dr. LEHMANN, Eberhard (Paul Scherrer Institut, SINQ Neutron Source, 5232 Villigen, Switzerland)

Presenter : Mrs. SALVEMINI, Filomena (Istituto dei Sistemi Complessi-Consiglio Nazionale delle Ricerche)

Neutron imaging, a non-destructive method for the study of mobile cultural heritage. A close collaboration with the Neutra team at the PSI

The scientific staff at the laboratory for conservation research at the Swiss National Museum performs non-destructive or minimal invasive analyses of cultural heritage by means of micro X-ray fluorescence spectrometry, atomic absorption spectrometry and infra-red and Raman spectrometry, in order to determine the composition of metal alloys, adhesives, pigments, colouring, precious and semi-precious stones, corrosion products and preservatives.

For specific studies, other methods are required to get knowledge about inner hidden structures or the state of conservation. The common approach for this kind of investigations is to use thermal X-rays and/or cold neutrons rays.

In close collaboration with the PSI we already performed studies within several projects.

The flanged axe of Thun-Renzenbühl, dated to the Early Bronze Age, is decorated with numerous inlays of a golden metal and was investigated by neutron tomography in order to obtain virtual cuts of the axe in all three dimensions. This allowed studying the casting and decoration technique [1].

The sword of Oberwil, dated to the 15th century, was excavated from the lake of Zug in 2010. This sword, remarkably preserved, made of iron with a precious elaborated wooden grip and cross-guard, was also investigated at the PSI in order to study the sword and the decoration technology.

[1] Daniel Berger, Katja Hunger, Sabine Bolliger-Schreyer, Daniel Grolimund, Stefan Hartmann, Jan Hovind, Felix Müller, Eberhard H. Lehmann,

Peter Vontobel and Marie Wörle; New insights into Early Bronze Age damascene technique of the Alps.

The Antiquaries Journal, 93, 2013, pp 1 of 29 r The Society of Antiquaries of London, 2013; doi:10.1017⁄s0003581513000012.

Summary :

M. Wörle1, V. Hubert1, K. Hunger1, K. Schmidt-Ott1, E. Lehmann2, F. Schmid2, D. Mannes2, D. Berger3, F. Müller4, S. Hartmann4 and J. Frey5.

Swiss National Museum 1

Paul Scherrer Institute 2

Independent researcher in archaeometallurgy 3 Historical Museum Berne 4

Archaeology Department of Canton Zug 5

Primary authors : Dr. WOERLE, Marie (Swiss National Museum)

Co-authors : Dr. MANNES, David (Paul Scherrer Institut)

Presenter : Dr. WOERLE, Marie (Swiss National Museum)

Investigating Production Technology with Neutron Tomography

Style has played a major role in how archaeologists view material culture. Visual style, over technological style, has often dominated the study of materials due to the destructive nature of many investigative methods. Metallographic investigations, used to analyze metal production technology, are destructive and invasive; they require removing a section of the object. Neutron imaging, on the other hand, provides a non-destructive and non-invasive method of analyzing materials on the basis of production technology. Neutron tomography has been applied to a bronze Roman oinochoe (a wine pouring vessel) to better understand the methods of its production. This allowed for the study of the internal structure of the bronze vessel, revealing the porous nature of the metal. This paper will examine the variability of the porosity within the structure and investigate the methods by which the vessel was constructed. These results will highlight how neutron imaging may provide otherwise inaccessible details of production technology.

This study was performed on the CG-1D prototype neutron imaging beamline at the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory in Tennessee, USA.

Primary authors : Ms. HERRINGER, Susan (Brown University) ; Prof. RYZEWSKI, Krysta (Wayne State University)

Co-authors : Dr. BILHEUX, Hassina (Oak Ridge National Laboratory) ; Dr. BILHEUX, Jean-christophe (Oak Ridge National Laboratory) ; Prof. SHELDON, Brian (Brown University)

Presenter : Ms. HERRINGER, Susan (Brown University)

NIPS-NORMA: A new PGAI-NT setup at the Budapest Research Reactor

Efforts are being made at laboratories worldwide to develop Prompt Gamma Activation Analysis (PGAA) towards a position-sensitive technique. It was proven earlier that the complete scanning with a few-mm-resolution is only practical on small objects due to constrain of experiment time and neutron flux. A feasible alternative is the combination of neutron radiography with prompt gamma activation analysis. Radiography, or even a full tomography of the complex sample, as a first step, can be completed in minutes, but often provides enough information to set up regions of interests inside the sample. The detailed element analysis by PGAA is carried out then only at these spots, saving substantial beam time. This novel combination of methods is best used for real samples which consist of a few homogeneous parts.

The technique proved its usefulness and raised enough interest in the user community. Based on these experiences the first permanent radiography-driven PGAI facility, NORMA (Neutron Optics and Radiation Measurement for element Analysis) was constructed and put into operation in the first months of 2012.

The sample chamber has dimensions of 20×20×20 cm3. By removing one or more side panels, larger objects up to 5 kg weight could be analyzed (such as a sword, vase, stones, etc.). Samples can also be loaded to the chamber manually from the top side. The positioning table has a nominal travel distance of 200 mm. The gamma radiation is detected with a Compton suppressed system, that consists of a central Canberra GR2318/S HPGe detector surrounded by a Bismuth Germanate (BGO) scintillator made by Scionix. The cylindrical and exchangeable lead collimators can be mounted into a socket of the 10-cm thick lead shielding. The gamma events are collected with a Canberra DSP-2060 digital signal processor, in anti-Compton mode. The imaging system comprises a 100 um thick Li-6/ZnS scintillator, a silver-free quartz mirror set in 45 degree to the neutron beam and a cooled ANDOR iKon-M CCD camera (16-bit ADC and 1024×1024 pixel resolution), mounted to a light tight aluminum housing. Integrated data acquisition software operates the moving table, the gamma-ray spectrometer and the camera.

The facility became open to the international user community through the EU-funded access programs. The first results of this non-destructive technique will be presented here from various fields of applications, such as archaeometry, safeguards and material science.

Primary authors : Dr. SZENTMIKLÓSI, László (Centre for Energy Research, Hungarian Academy of Sciences)

Co-authors : Dr. KIS, Zoltán (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. BELGYA, Tamás (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. RÉVAY, Zsolt (Technische Universität München, Forschungsneutronenquelle Heinz Maier-Leibnitz (FRM II))

Presenter : Dr. KIS, Zoltán (Centre for Energy Research, Hungarian Academy of Sciences)

Neutron computed tomography for determining the spatial distribution of carbolineum in wooden artefacts of cultural heritage

Some wooden artefacts of cultural heritage were treated with carbolineum as a preservation agent like the baroque epitaph Reyer (1704) at the St. Laurentius church in Tönning (Schleswig-Holstein, Germany). The subsequent constant migration of carbolineum through the layers of paint to the surface has had a detrimental effect on the aesthetic appearance of the epitaph.

Carbolineum is an oily, water-insoluble, flammable, dark brown mixture of coal tar oil components. Due to its content of polycyclic aromatic hydrocarbons (PAH), which are classified as carcinogenic and harmful to the environment, the use of carbolineum has been widely forbidden.

The aim of the project is to develop an exemplary conservation treatment that will reduce the toxic residue within the historical wooden object. Thus, the spatial distribution of carbolineum inside the artwork was determined by neutron computed tomography (CT) at the NECTAR facility of the Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM-II) of the Technische Universität München. The neutron CT revealed a heterogeneous density distribution with enriched concentrations near the surface. These results will be compared with X-ray CT showing more spatial details and small metal reinforcements.

Primary authors : Dr. NUSSER, Amelie (Rathgen-Forschungslabor)

Co-authors : Dr. OSTERLOH, Kurt (BAM Federal Institute for Materials Research and Testing)

Presenter : Dr. NUSSER, Amelie (Rathgen-Forschungslabor)

“Elemental analysis of Minerals content of Some Ayurvedic Medicinal Plants from India by Nondestructive Instrumental Neutron Activation Analysis (INAA) and Atomic Absorption Spectroscopy (AAS) Techniques”.

ABSTRACT: Ethno medicine practices are becoming a rising new trend in urban areas. Their healing processes generally consist of botanical therapies, herbal remedies and native ethomedical knowledge. Traditional medicinal plants are enriched with number of minerals and vitamins ,provide not only the natural nutrition but also that are useful in the treatment of different diseases without /less side effects, less expensive and easily available in India .

Medicinal plants were purchased from medicine shops and were analyzed by Non-destructive Instrumental Neutron Activation Analysis (INAA) using ^{252}Cf spontaneous fission neutron source available at Department of Chemistry, University of Pune, INDIA. The induced activities were counted by γ-ray spectrometry and Atomic Absorption Spectroscopy (AAS) techniques using Perkin Elmer 3100 Model) for the measurement of major, minor and trace elements. 15 essential major, minor and trace elements Al, K, Cl, Na, Mn by INAA and Cu, Co, Pb Ni, Cr, Ca, Fe, Zn, Hg and Cd by AAS were analyzed from different Indian herbals .

A critical examination of the data shows that all these elements are present in the five herbals at major, minor and trace levels. The elements Ca, K, Cl, Al and Fe are found to be present at major levels in most of the samples while the other elements Cu, Co, Ni, Cr, Ca, Fe, Zn are present in minor or trace levels. Pb ,Cd and Hg are below the permissible levels. These medicinal herbs are safe to consume as The differences in the concentration of the elements are attributed to soil composition and the climate in which the plant grows.

Summary :

The elemental concentrations in different herbals from India are discussed.

The data is useful to medical practitioners, pharmacists as well as food analysts, for the synthesis of new Ayurvedic herbal formulations as well as in deciding the proportion of various active constituents and managing dose of a particular herbal formulations . and the researchers in the areas of Ayurvedic and alternative medicines.

Primary authors : Dr. BHARATI, Pardeshi (B.G.College.Sangvi Under University of Pune,INDIA)

Co-authors :

Current status of neutron radiography in Thailand

For the past few years, neutron radiography in Thailand has been dramatically developed since the IAEA Coordinated Research Project titled “Application of 3D Neutron Imaging and Tomography in Cultural Heritage Research” was initiated and Thailand was chosen to participate in the project. In order to preserve the original characters of cultural heritage for our future generations, it is significant to perform all investigations on object non-destructively. Neutron radiography serves to meet the requirement. Neutron radiography of several objects has been taken at TRR-1/M1 using conventional film method and reusable imaging plate. Even the facility has been operated for more than a decade; the current status is still under developing. Several difficulties as a result of limited facilities including lack of neutron camera and its components lead to 3D neutron imaging are likely impossible to achieve. In the early state, a DSLR camera assembled with an in-house light-tight-box and a prototype computer controlled rotary table were set up for Buddha sculpture analysis. Subsequently, the first near real-time digital neutron imaging was established in Thailand in 2012. Furthermore, the combination image of neutron and X-ray provides complete inner structure information that helps better understand the past manufacturing technology as well as to obtain an appropriate conservation method. The authentication proofs and relative dating using structural profile along with elemental analysis by NAA and XRF will be studied further to implement the cultural heritage interpretation. In order to achieve 3D imaging capability, the current neutron radiography facility is scheduled for upgrading in various aspects including exposure station, shielding wall, and beam shutter. In parallel to the upgrade of the hardware, image reconstruction techniques and software are currently investigated and optimized to fulfill the information that is difficult to achieve by 2D imaging. The upgraded facility (hardware and software) will not only contribute to research and advanced application of neutron imaging techniques in Thailand, but will also contribute to human resource development in the area of neutron imaging technology in this region. In addition, the renovated facility will be further possible to establish routine approaches for archaeological service and wide range of applications.

Primary authors : Dr. KHAWEERAT, Sasiphan (Thailand Institute of Nuclear Technology)

Co-authors : Mr. RATANATONGCHAI, Wichian (Thailand Institute of Nuclear Technology) ; Dr. WONGLEE, Sarinrat (Thailand Institute of Nuclear Technology) ; Dr. PICHA, Roppon (Thailand Institute of Nuclear Technology) ; Mr. SILVA, Kampanart (Thailand Institute of Nuclear Technology) ; Ms. PROMPING, Jiraporn (Thailand Institute of Nuclear Technology) ; Dr. LIAMSUWAN, Thiansin (Thailand Institute of Nuclear Technology)

Presenter : Dr. KHAWEERAT, Sasiphan (Thailand Institute of Nuclear Technology)

PGAA analysis of some Neolithic obsidian samples from Romanian regions

Thirty archaeological samples from different regions of Romania and from different prehistorical periods were analyzed at the PGAA facility of the Budapest Neutron Centre: Iclod, Tzaga, Silagiu sites in Transylvania, Neolithic period; Cuina Turcului site at Iron Gates (on Danube border, between Romania and Serbia), Early Neolithic (Neolithisation) and Neolithic period; Magura site in Teleorman County (South of Bucharest, near Danube), Early Neolithic (Neolithisation period).

The aim of the study was to identify obsidian geological sources used in each region and period. Neolithisation is the process of transition from hunting-fishing-based society to agriculture, process related to an important populations movement. The most accepted theory is “Ex Oriente Lux”, the migration of “Neolithic model” (and population) from Mesopotamia, Anatolia, Greece - through Aegean Islands, Balkans, Central Europe - via Danube.

Two main geological regions are presumed to be the obsidian sources for Romanian territory: Tokaj Mountains (Carpathian I – now in Southern Slovakia and Carpathian II – now in Northern Hungary) and Greek Islands – especially Melos (Aegean Sea). PGAA proved to be the most convenient method to quantify the major components and some characteristic trace elements in the bulk material, most of all B and Cl, in a non-invasive way. In order to determine the provenance of the archaeological objects, we have investigated several elements’ contents. Compositions of archaeological objects were compared with our own reference database including the major European and Mediterranean sources. B/SiO_2 vs. Cl/SiO_2 ratios and Principal Components Analysis (PCA) proved to be the most indicative in determination of different groups.

Our results indicate all the Transylvanian Neolithic samples fit the Carpathian I pattern. The same pattern can be attributed to Neolithic Cuina Turcului samples. A special situation is for the Neolithisation period, both for Cuina Turcului and Teleorman. These samples fit Carpathian II pattern, however, based on K_2O content, these samples are very similar to those from Yali Island (Aegean Sea). Since the latters are known to show weak mechanical quality, it has been less probably used for tools production. By increasing the number of fingerprinting elements, using additional analytical methods, one can further confirm or disprove the current theories of Neolithisation.

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Summary :

Non-destructive Prompt Gamma Activation Analysis was applied to perform provenance study of Neolithic obsidian artefacts. Elemental compositions of archaeological objects from Romanian sites have been compared with reference measurements of the most important geological sources in

have been compared with reference measurements of the most important geological sources in Central Europe and in the Mediterranean region. Based on the measured concentrations, especially on B- and Cl content, the samples proved to be either ‘Carpathian I’ (North of Tokaj mountains, Slovakia) or ‘Carpathian II’ (South of Tokaj mountains, Hungary) types. However, further methods are recommended to identify more fingerprint-like trace elements in obsidians.

Primary authors : Dr. CONSTANTINESCU, Bogdan (National Institute for Nuclear Physics and

Co-authors : Mrs. CRISTEA-STAN, Daniela (National Institute for Nuclear Physics and Engineering, Bucharest, Romania) ; Dr. KASZTOVSZKY, Zsolt (Centre for Energy Research, Hungarian Academy of Sciences) ; Ms. MARÓTI, Boglárka (Centre for Energy Research, Hungarian Academy of Sciences)

Elemental Analysis of Smithsonian Building Stones and Brick by Prompt Gamma and Delayed Gamma Neutron Activation Analysis

A systematic characterization was undertaken of the lithology and physical properties of eight building stones used in the monumental architecture of the Smithsonian Institution in Washington, D.C., from 1847 onward. The Smithsonian building stones make up a representative sample of the American dimension stone industry with respect to style, quarrying techniques and geology. The stones selected include Holston marble, Mt Airy granite, two types of Vermont marble, Seneca sandstone, Mankato dolomite, Salem limestone and hydraulic pressed brick. This characterization included both instrumental neutron activation analysis (INAA) and prompt gamma neutron activation analysis (PGNA). The former is typically used for provenance studies, while PGNA is of interest because it can be used in a nondestructive method mode, either at a reactor or, with portable neutron sources, used on site. The characterization of the same set of samples by both methods makes comparisons possible. Over the entire set of specimens INAA detected a total of 33 elements, and PGNA, 29 elements. For the silicate rocks and brick in the rare earth elements group typically used for provenance studies, INAA detected 9 of the 14: La, Ce, Nd, Sm, Eu, Tb, Dy, Yb and Lu. PGNA detected 4 of these 9: Nd, Sm, Dy and Yb, and in addition, Gd. Of the fourth period transition metals INAA detected all except Cu, and PGNA detected all except Ni, Cu and Zn. PGNA also detected Si, which INAA is unable to measure. For the carbonate stones, both INAA and PGNA detected the elements Mg, Ca, Sr and Mn, which are major formers of carbonate minerals. In addition, PGNA detected B, which could be useful for limestone and marble provenance.

Primary authors : Prof. LIVINGSTON, Richard (University of Maryland)

Co-authors : Ms. ALOIZ, Emily (Smithsonian Institution) ; Ms. GRISSOM, Carol (Smithsonian Institution) ; Dr. PAUL, Rick (NIST)

Presenter : Prof. LIVINGSTON, Richard (University of Maryland)

Neutron investigation of an exceptional zinc lamp from the Academia Georgica Treiensis archaeological collection (Italy)

The Academia Georgica Treiensis is one of the oldest Italian Academies, created in the XV century for the main interests of poetry and literature. In the XVIII century, influenced by the Enlightenment ideas, the Academy decided to renovate its interests "encouraging rational and practical studies to improve agriculture and industry and to honour sciences, literature and arts" [1]. The Archaeological Collection of Academia Georgica Treiensis was put together by the noble family of Teloni, Counts in Treia, Italy, between the end of the XVIII and the end of the XIX century. The Collection has been recently reorganized and inventoried: it is composed of about 517 pieces (e.g. armours, table-ware, jewels, ceramics and varied tools), mostly of unknown origin but belonging to prehistoric and ancient periods, nevertheless it is not excluded that some objects are earlier [2, 3].

A few metallic objects from this collection have been selected for a neutron based archaeometric investigation. The primary goal of the analyses was to advance the accurate technological and material description of the objects providing scientific data for a further and more comprehensive comparative analysis also covering the find material from the close archaeological sites [4, 5]. The involved complementary techniques are Prompt Gamma Activation Analysis (PGAA), Time-of-Flight Neutron Diffraction (TOF-ND) and Neutron Radiography (NR). The neutron investigations allowed us to determine the bulk composition, also providing a qualitative and quantitative assessment of the phase composition and the structural properties of the constituents, as well as radiographic images, finally to identify possible manufacturing techniques. An additional examination, carried out by external beam particle induced X-ray emission (PIXE) spectroscopy, provided a quantitative analysis of major and trace elements and supplied data on the near-surface elemental composition complementary to the results characteristic for the bulk.

The present case study is focussing on one of the investigated objects, a not common, so called polilicnes lamp, which proved to be unique in several aspects drawing more attention to its production and dating. The lamp is characterised with six ogive-shaped projections of which three are decorated with different bearded faces. Based on the type of the artefact as well as its supposed bronze material, it was considered as an ancient product from the Imperial Roman period. The obtained results, however, showed surprisingly that it is mainly made of metallic zinc. It raises several questions, since this material is not to be expected from the classical antiquity. Because of the high volatility of metallic zinc and the reactivity of the zinc vapour, producing zinc metal was a technological challenge before the industrial discoveries of the early modern period. Considering the few relevant historical and archaeological sources about the ancient and medieval metallurgical processes, metallic zinc could be available until the beginning of the XIX century only as a by-product of the zinc-rich lead ores smelting [6, 7]. The gained zinc, however, could be of a very small amount practically insufficient for manufacturing large objects.

With respect to metallic artefacts from archaeological periods, the presence of zinc among the alloying components (both in brass and zinc objects) is of great significance as the way of its use may indicate the possible earliest date of the production. In the case of the polilicnes lamp the identified elemental and phase composition, i.e. the high pureness of the zinc material, opens different hypotheses about its origin querying its ancient production and genuineness. With the archaeological interpretation of the analytical results we aimed to clarify and reconstruct the context and history of this peculiar piece belonging to the investigated collection.

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Primary authors : Dr. ROGANTE, Massimo (Rogante Engineering Office)

Co-authors : Dr. MITCSENKOV-HORVÁTH, Eszter (Institute for Solid State Physics and Optics, Wigner Research Centre for Physics, Hungarian Academy of Sciences) ; Dr. KÁLI, Gyorgy (Institute for Solid State Physics and Optics, Wigner Research Centre for Physics, Hungarian Academy of Sciences) ; Dr. KASZTOVSZKY, Zsolt (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. KIS, Zoltán (Nuclear Analysis and Radiography Department, Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. KOVÁCS, Imre (Institute for Particle and Nuclear Physics, Wigner Research Centre, Hugarian Academy of Sciences) ; Dr. MARÁTI, Boglárka (Centre for Energy Research, Hungarian Academy of Sciences) ; Prof. ROSTA, László (Institute for Solid State Physics and Optics, Wigner Research Centre for Physics, Hungarian Academy of Sciences) ; Prof. SZÕKEFALVI-NAGY, Zoltán (Institute for Particle and Nuclear Physics, Wigner Research Centre, Hugarian Academy of Sciences)

Presenter : Dr. ROGANTE, Massimo (Rogante Engineering Office)

Neutron diffraction measurements for the characterisation of Italian Celtic coins

The silver coinage of the ancient Celtic peoples settled in northern Italy is, even now, a topic with many unsolved problems, such as chronology, attributions, relationships between series and emissions through time (from IV to I century B.C.). In order to provide numismatists with new data for metrological studies, which require average weights of the emissions and the knowledge of their compositions, several samples have been analyzed with scientific techniques.

Our research group at the Physics Department (University of Torino, Italy) has carried out neutron diffraction measurements at the ISIS facility (Rutherford Appleton Laboratory), to analyze structural properties and composition of these coins. Neutron diffraction is actually a powerful tool for the analysis of bulky metal objects that cannot be sampled. The instrument used is the TOF diffractometer of the Italian Neutron Experimental Station (INES), equipped with 144 3He squashed detectors, grouped in 9 banks, covering a range of about 160 degrees on the horizontal scattering plane. This experimental setup allows to measure a wide d-spacing range (from 0.2 to 12 Å), and is then suitable for most metals. In the analyzed coins two main phases were detected, accordingly to the biphasic diagram for silver-copper alloys, while minor phases (> 0.5 wt.%) are due to alteration phenomena. The d-spacing of the silver (beta) and the copper (alpha) rich phases calculated on the diffraction patterns from the backscattering bank are different from the ones of the pure elements, being smaller and bigger respectively. Thanks to literature data, obtained on certified samples, the relationship between d-spacing and composition has been investigated in order to estimate the elemental composition and then obtain a silver/copper ratio useful for numismatic studies.

The first results presented here allow us to suggest a new organization of the entire north-Italian Celtic coinage, based on the scientific data obtained. The most ancient coins of the series are made of almost pure silver, while coins attributed to Insubres and Boico-Cenomani peoples, which belong to a second stage and are contemporary to each other (III century B.C.), show a lower average silver beta phase composition. Later coins (200-125 B.C.), attributed to the Celtic tribe of the Libui, show an even lower silver content average.

In conclusion, information achieved show the importance of the neutron diffraction technique, which provided data to demonstrate a silver content decline due to inflation and, furthermore, to give elements for the study of metrological relationships with the contemporary Roman Republican currency.

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Primary authors : Mr. CORSI, Jacopo (University of Turin, Physics Department)

Co-authors : Dr. GRAZZI, Francesco (CNR-ISC) ; Dr. SCHERILLO, Antonella (Rutherford Appleton Laboratory (ISIS Facility)) ; Dr. LO GIUDICE, Alessandro (University of Turin, Physics Department) ; Dr. RE, Alessandro (University of Turin, Physics Department) ; Dr. BARELLO, Federico (Soprintendenza per i Beni Archeologici del Piemonte e M.A.E)

Presenter : Mr. CORSI, Jacopo (University of Turin, Physics Department)

Preserve Historical Paintings by Means of Neutron Imaging:

A Complete Guideline.

The art of Autoradiography with neutrons were described in various papers through examples of valuable paintings being successfully analysed – however, this paper describes a step-to-step procedure for the complete process to act as a guide line for the practice and procedure to the technique with the purpose to perform the process of Autoradiography with neutrons successfully. Although neutron autoradiography might be applied for many years, only a limited number of facilities in the world are equipped to perform a successful autoradiograph of a precious painting. The cultural heritage community on the other hand needs to be confirmed that the neutron dose received in a typical autoradiography scanning sequence does not causes permanent if any damages to the pigments within the painting. The aspect of damage to the pigments and irreversible damage to the painting is always a concern to historians and collectors and this paper attempts to address this aspect in detail.

Primary authors : Mr. DE BEER, Frikkie (Necsa)

Co-authors : SCHILLINGER, Burkhard () ; Dr. KARDJILOV, Nikolay (Helmholtz-Zentrum Berlin)

Presenter :

Neutron techniques applied to better define conservation strategies of 16th – 18th centuries Portuguese glazed tiles

The city of Lisbon has in situ sixteenth-eighteenth -century tiles that sometimes were decontextualized of its original architectural framework and landscape, owing to the deep urban changes operated in the following centuries.

There are several examples in the various city gardens and ancient buildings all over Lisbon and other cities such as: (i) 16th century tile panel near the floor in the Madre de Deus Church (belongs to the National Tile Museum) in so advanced state of degradation that have been taken off and replaced by new ones; (ii) 17th century set of tiles in the Torel hill, near the Campo de Santana, with advanced state of degradation and in the rear part of a new condominium; and (iii) 18th century glazed tile from the “Nossa Senhora da Conceição dos Aflitos” Church (Elvas, Portugal).

These glazed tiles from different centuries have been placed in diverse environments along the last centuries. The 16th tiles were indoor close to the floor and given the proximity of the river and the low water level, they were seriously damaged by the effects of water ascendance by capillarity. The 17th tiles are outdoor exposed and without any kind of protection. The 18th tiles are indoor and are the less deteriorated.

The main objective of this work is to identify the degradation state and the main degradation processes of glazed tiles, so that they can be better overcome or at least reduced, giving tools to conservators so they can better choose the intervening strategy. So, in order to contribute to better design future actions of conservation/restoration of Portuguese glazed tiles, a study including diverse approaches has been performed, especially comprising neutron techniques, namely neutron tomography and neutron activation analysis. These methods were complemented with X-ray diffraction to identify the mineral phases of ceramic body.

The results obtained showed that geochemical patterns together with mineralogical assemblage became a useful approach for the knowledge of nature of these works of art, as they characterized the original materials and the alteration products. These results allowed identifying the chemical and physical conditions which favoured endogenous and/or exogenous processes of decay. The neutron tomography, enabling an inner visualization and the light elements content, particularly hydrogen, was particularly useful to evaluate the degradation state of each tile. NT also showed that brushing technique to apply consolidants appears to be more efficient than immersion technique, used in the National Tile Museum.

Thus this work allowed establishing a methodological approach to help conservators to select conservation practices of tiles in different environmental conditions. Brushing technique appears to be a suitable technique for consolidation, which favours the conservation procedures of the tiles in situ, like in the Quinta do Torel panel.

Primary authors : Prof. PRUDÊNCIO, Maria Isabel (Campus Tecnológico e Nuclear, Instituto Superior Técnico, Universidade Técnica de Lisboa) ; Prof. DIAS, M. Isabel (CAMPUS TECNOLOGICO E NUCLEAR. IST, Univ. Técnica de Lisboa)

Co-authors :

Presenter : Prof. PRUDÊNCIO, Maria Isabel (Campus Tecnológico e Nuclear, Instituto Superior Técnico, Universidade Técnica

The use of Monte Carlo Code for radiation transport and dosimetry calculation for neutron radiography exposure room facility at reactor triga mark it puspati (RTP)

A Monte Carlo simulation of photon and neutron flux at the neutron radiography exposure room (NuR II) with various shielding material and with different sizes of beam port at Agensi Nuklear Malaysia was performed using the MCNP5 computer program. The objective of the work is to model the NuR II beam port with different sizes of holes and various materials to obtain radiation transport and dosimetry calculation result. The code was used to calculate of photon and neutron flux and dose distributions using the KCODE card. This paper describes the use of the Monte Carlo Code to generate a radiation transport and dosimetry calculation for the actual configuration and discusses the potential of applying the method to more complex radiation protection problems. The simulated results using the variance reduction technique were very good agreement with the measured data.

Primary authors : Mr. JAMRO, Rafhayudi (Malaysian Nuclear Agency)

Co-authors : Mr. M. ZAIN, Muhammad Rawi (Malaysian Nuclear Agency)

Presenter : Mr. JAMRO, Rafhayudi (Malaysian Nuclear Agency)

Eximination of the roman treasure find by neutron and gamma radiography and I-NAA

By the end of 2003 during the archaeological excavations near the village Drnovo on the construction site of future European highway Ljubljana-Zagreb (Croatia) an intact Roman ceramic pot assumingly containing a treasure find was unearthed The ceramic pot was dated into the 2nd half of the 3rd century A.D. In order to get some preliminary information for the archaeologists and to properly conduct the opening and salvaging the suspected precious contents of the find non-destructive radiographic examinations by both conventional radiographic techniques (X-ray and Ir-192 gamma ray radiography), by thermal neutron radiography (NR) and non-invasive instrumental neutron activation analysis (I-NAA) were performed. The pot was completely filled with earth, with total weight of over 9 kg and quiet large - out. dia 20 cm, height 24 cm) and hence presented a demanding task for radiographic examination. The radiographic techniques provided clear evidence on the presence of a hoard of coins and jewelry and confirmed the assumptions about the treasure contents. The I-NAA gave a clue about the elemental composition of the hoard. The NR complemented the conventional radiography since it revealed that the coins were hoarded in the 3 separate purses made of organic materials, probably leather.

Summary :

Key words: archaeology, neutron radiography, Ir-192 radiography, instrumental neutron activation analysis, Ljubljana TRIGA reserach reactor

Primary authors : Prof. RANT, Joze Rant (ret. from J.Stefan Institute, Ljubljana, Slovenia)

Co-authors : Dr. MIŠKEC, Alenka (National Museum of Slovenia) ; Dr. JAČIMOVIČ, Radojko (J.Stefan Institute)

Presenter : Prof. RANT, Joze Rant (ret. from J.Stefan Institute, Ljubljana, Slovenia)

Applications of Imaging Techniques in Cultural Heritage: Examples and survey on recent developments

In this work we provide a broad survey of developments in radiation transmission imaging techniques for archaeological and cultural heritage studies purpose.
In the domains of archaeology and cultural heritage, the imaging practitioner interprets the produced images to conclude about origin, chronology and making of technique of an ancient art object. In addition to this survey, we present some examples of applications developed around 35 kV & 1 mA X-ray Tomograph and 1.6x106 n/cm2/s neutron imaging facility. Applications examples concern fossils, bones, stones and biological objects.

Primary authors : Dr. KHARFI, Faycal (Deparmtent of Physics, Faculty of Science, University Ferhat Abbas-Sétif, Algeria)

Co-authors :

Presenter : Dr. KHARFI, Faycal (Deparmtent of Physics, Faculty of Science, University Ferhat Abbas-Sétif, Algeria)

3D Neutron Imaging of a XVIIIth Dynasty Egyptian Sealed Pottery

Forever, art scientists, historians and museum curators are interested in the precise determination of the material composition and substructure analysis of historic objects. This scientific approach is essential as it can be employed to reveal the history of the sample, investigate the original material properties and even discover how the object has been restored in the past.

In this field of cultural heritage research, we recently investigated a sealed Ancient Egyptian pottery from the XVIIIth Dynasty (New Kingdom period), stored in the Museum of Aquitaine in Bordeaux, France. The object (inventory number 8608) is 97 mm in height with a maximum diameter of 64 mm. The original use of this jar remains a mystery for curators. Without any specific decoration, it looks like a simple red clay vessel hermetically sealed with a clay cork. Its shape (a jar with a neck) indicates that it was most likely used for liquids, not ointment, perfumed cream, or unguent, because it would not allow easy access to these less liquid contents for skin application. However, the existence of Menkheperre Tuthmosis III's cartouche (1479–1425 B.C.) on the pottery's clay cork allows us to infer that this object was probably very important, perhaps linked with the funeral ritual of this pharaoh. Indeed, a sticker under the object indicates that it was purchased in 1861 in Gournah, on the famous necropolis of Thebes (on the west bank of Luxor). According to experts, this nearly 3500-year-old pottery bottle probably did not contain viscera — usually preserved in canopic jars — but most likely offerings of food to the dead during his funeral.

In the framework of the CHARISMA project (Cultural Heritage Advanced Research Infrastructures, Synergy for a Multidisciplinary Approach to Conservation/Restoration), we analyzed the Egyptian jar with complementary techniques such as X-ray, terahertz (THz) radiation and neutron beam. The use of non-invasive THz radiation, 2D and 3D imaging revealed an internal mobile content inside the jar. This content was also visualized using an X-ray scanner, with the limitation that the measurement can alter the integrity of the object owing to the irradiation action.

Recently, we analyzed the jar at the Budapest Neutron Centre using the NIPS-NORMA instrument installed at a guided cold neutron beam, in order to determine the physical properties of the object with a complementary physical approach. The experimental setup consists of the Neutron Induced Prompt Gamma Spectrometer (NIPS), equipped with "NORMA" neutron radiography and 3D tomography system. The neutron beam with a cross section of 40×40 mm2 has been used to perform 3D neutron imaging of a selected region of the sample. Although the object was about 10 cm tall, by systematically moving the object, we were also able to create a composite 2D image of the whole jar from a set of tile images, in order to visualize its structure with a spatial resolution of 0.3 mm. The main results are the precise identification of the cork stopper hidden by a clay cork. 3D neutron imaging clearly reveals that this stopper is made of a ball of linen or any other string-like organic material. This observation was not possible with both X-rays and THz radiation owing to insufficient spatial resolution and contrast. Neutron imaging also confirmed the presence of cracks in the wall of the pottery bottle, as already discovered with X-rays. 3D neutron imaging also provides some representations of the mobile content which is constituted of inhomogeneous dried materials. By semi-quantitative analysis of the (n,γ) spectra taken at NIPS, we concluded that the jar content is mainly composed of H, C, N, S and Cl elements, which supports the assumption about its organic nature. Without certitude, we can assume that this content could consist of germinated seeds of any other dried organic material.

As a result, these measurements performed at the Budapest Neutron Centre provide complementary information about this Ancient Egyptian jar: the way it has been closed by a double stopper (clay and probably linen) and the organic and inhomogeneous nature of the mobile content. These partial conclusions might be a prime importance for historians and museum curators.

The authors gratefully acknowledge François Hubert, director of the Museum of Aquitaine, Anne Zieglé, curator at the Museum of Aquitaine, and the city of Bordeaux for the authorization to perform the measurements at the museum and in Budapest. This project has been supported by the CHARISMA project, contract nr. 228330. The authors acknowledge Michel Menu who fostered the scientific contact among the collaborators.

Summary
3D and 2D neutron imaging and non-destructive position-sensitive elemental analysis by PGAI were performed at the Budapest Neutron Centre on a XVIIIth Dynasty (15th c. B.C.) Egyptian sealed pottery. The result of the neutron tomography revealed that a ball of linen was most probably used to seal the jar. Furthermore, finer structural details of the inner content were made visible, which was not possible to find out in earlier attempts using X-ray and THz imaging methods. In addition, with the help of highly penetrating and well collimated neutron beam, local elemental composition was determined that allowed us to conclude about the nature of the inner content, which is supposed to be a dried organic material.

Primary authors : Dr. ABRAHAM, Emmanuel (LOMA)

Co-authors : Dr. HERVÉ, Marie-Christine (Museum of Aquitaine, Bordeaux) ; Dr. BESSOU, Maryelle (PACEA, Univ. Bordeaux, CNRS, UMR 5199, Talence, France) ; Dr. KIS, Zoltán (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. SZENTMIKLÓSI, László (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. KASZTOVSZKY, Zsolt (Centre for Energy Research, Hungarian Academy of Sciences)

Presenter : Dr. KASZTOVSZKY, Zsolt (Centre for Energy Research, Hungarian Academy of Sciences)

Investigation of carbonate deposits of ancient roman aqueduct systems in the mediterranean area via lase-icp-ms ans INAA

Terrestrial carbonate deposits such as speleothems, travertine and tufa are important archives for paleoclimate. Carbonate deposits also form in Roman aqueducts, and these have recently been proposed as an alternative high-resolution climate proxy. The number of layers in the carbonates can be used to establish how long an aqueduct functioned, and the stable oxygen isotope distribution in the material gives information on temperature in the channel, which is related to external temperature. Besides information on paleoclimate, the carbonate deposits can also give information for archaeology and earthquake research. Many Roman cities, such as Rome, Constantinople, Lyon and Ephesus are fed by several aqueducts, and it would be interesting to know how the water distribution in the city worked. If the source water was of different chemical composition, it may be possible to determine which source fed the different parts of the water supply system. This can be investigated by detailed chemical analysis of trace elements in the carbonate deposits. The same applies to aqueducts that were fed by more than one source: in this case, it may be possible to establish how the source water was used, and if both sources functioned all the time. Finally, changes in land use such as deforestation will gradually change the composition of source water of an aqueduct, and such changes may therefore be detected in the trace element record.
Trace element chemistry of calcium carbonate as occurs in the aqueducts was analysed by Laser-ICP-MS and INAA. Laser-ICP-MS comes up with a good spatial resolution respecting the seasonal variation of the trace element ratio in the deposits, whereas INAA delivers concentration profiles on a wide spectrum of trace elements. INAA data should be analysed with multivariate statistical methods to carry out provenance analysis in order to deliver insights into the function of the former aqueduct system. Previous results indicate that the homogeneity of trace elements in the collected samples is high enough for this purpose. Further samplings and measurements to confirm this assumption are in progress. The Laser-ICP-MS measurements were performed at the Institute for Geosciences of the Johannes-Gutenberg University of Mainz, Germany. For INAA the research reactor TRIGA Mark II of the University of Mainz was used as neutron source. The reactor provides multiple irradiation facilities for INAA purposes: The rotary specimen rack allows simultaneous long time irradiation of up to 80 samples, a higher flux for detecting low element concentrations can be reached in the central irradiation tube, and the pneumatic transfer system is available for activation products with shorter half-life. Results of the detailed seasonal behaviour as well as the ongoing provenance investigation via INAA will be presented.

Primary authors : Mr. STIEGHORST, Christian (University of Mainz - Institute of Nuclear Chemistry)
Co-authors :
Presenter : Mr. STIEGHORST, Christian (University of Mainz - Institute of Nuclear Chemistry)

Combination of 3D visualisation techniques and nuclear analysis methods

Non-destructive analysis has become more and more important for archaeology, especially in the case of ELD (extra-long distance) trade items in prehistory. One of the potential highlights for such objects is high pressure metamorphites („jadeites”) and related rocks playing a crucial part in European prehistoric long distance trade networks. The extremely rare, attractive and prestigious objects were spread all over the western half of Europe (D'Amico et al. 2003) and recently the eastern borderline is seemingly shifted essentially (Szakmány et al. 2013, Petrequin et al. 2011). These rocks can be optimally studied in petrographic thin section, but as they are invaluable proofs of prehistoric communication networks and trade, any invasive analytical treatment for their studies is beyond question.

A combination of non-destructive techniques might be useful in this case. Non-destructive SEM-EDS of high-polished surfaces can be a good choice (Bendő et al. 2012, 2013). In this paper we use geochemical fingerprinting by PGAA (Szakmány et al 2011) coupled with density measurements derived from 3D scanning models. Density is a characteristic feature of the HP/HT rocks being very high (3300-3500 kg/m3). It has been used for characterisation of jade axes already using traditional laboratory techniques. 3D scanning and calculation of volume on the basis of geometrical shape might extent the applicability of this non-destructive and highly informative technique to very small objects where traditional laboratory techniques are hard to control.

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Summary :

Non-destructive PGAA and SEM-EDS measurements coupled with density measurements derived from 3D scanning have been used to characterise high pressure metamorphic rocks, often called 'jadeites'. The aim was to study their role in European prehistoric long distance trade networks.

Primary authors : Dr. SZENTMIKLÓSI, László (Centre for Energy Research, Hungarian Academy of Sciences)

Co-authors : Dr. SZABÓ, Lárinc (Semmelweis University) ; Dr. T. BIRÓ, Katalin (Hungarian National Museum) ; Dr. BENDÕ, Zsolt (Roland Eötvös University, Dept. of Petrology and Geochemistry) ; Dr. KASZTOVSZKY, Zsolt (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. KIS, Zoltán (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. SZAKMÁNY, György (Roland Eötvös University, Dept. of Petrology and Geochemistry)

Presenter : Dr. KIS, Zoltán (Centre for Energy Research, Hungarian Academy of Sciences)

Neutron Activation Autoradiographs – the Technique

In collaboration with the research reactor in Berlin, the Helmholtz-Zentrum für Materialien und Energie, the Gemäldegalerie Berlin is the only institute worldwide, which systematically employs the method of Neutron-Activation-Autoradiography to analyze paintings. Today we have investigated about 70 paintings. This is an effective, non-destructive, however, exceptional method for the investigation of paintings and paint techniques. It allows the visualization of structures and layers beneath the top surface and, in addition, enables the identification of elements contained in the pigments. The instrument B8 at the research reactor BER II is dedicated to this research. It allows to irradiate and activate artistic, technical, or geological items (foils, stones etc.) and other materials with cold neutrons having a flux of Φn=1•109 cm-2s-1 and to investigate it afterwards with imaging plate technique and/or to analyze it with gamma-spectroscopy.
The irradiation process, the investigation of the induced radioactivity and the possible identification of the pigments will be presented.

Primary authors : Dr. DENKER, Andrea (Helmholtz-Zentrum Berlin für Materialien und Energie)

Co-authors : LAURENZE-LANDSBERG, Claudia (conservator at the Gemäldegalerie, Staatliche Museen Berlin)

Presenter : Dr. DENKER, Andrea (Helmholtz-Zentrum Berlin für Materialien und Energie)

The examination of paintings by Rembrandt

with Neutron Autoradiography

For over thirty years, neutron autoradiography is in use for the examination of paintings from the Gemäldegalerie, Staatliche Museen Berlin, in cooperation with the Helmholz Zentrum Berlin. It expands the spectrum of colours by a multiple whose distribution in deeper layers of paint can be made visible. Together with the X-ray photograph and infrared reflectography a comprehensive insight into the genesis of a painting is made possible. By additional application of γ spectroscopy the darkenings on the autoradiograph can be assigned to special isotopes. The use of film allows a particularly good reproduction of the brush stroke, the technique of paint application and the conservation status (condition) of the painting. Autoradiography therefore provides art historians and conservators with a wide scope of possible interpretations.

By now about 70 paintings have been examined, mainly those by Rembrandt and his circle, but also works by Vermeer, Titian, Jan Steen, Frans Hals, Rubens. The paintings by Rembrandt hold a special interest, because he is a painter who repeatedly makes changes during the creative process in search of satisfactory composition. The progress of his quest can be read on the autoradiographs. The interpretation of the achieved goal, which we see as a finished work before us is thus provable and no longer subject only to personal hypothesis.

A problem in the interpretation of determining the genesis of a painting, however, is that the darkening of the film illustrates a summation of the radiation from all layers of paint.

Difficult is the determination of the depth of the radiation within the paint layer structure and how many layers of paint are involved. It is therefore imperative for the evaluation of the autoradiographs to perform a simultaneous examination of the painting with the stereoscope.

Thanks to funding from the Andrew W. Mellon Foundation the autoradiographs will be evaluated in a wholly interdisciplinary manner. The results will then be made accessible to the broader public in digitalized format within the context of the Rembrandt Database.

In the talk, some examples will be presented of already fully examined Rembrandt paintings that prove the painter's ingenuity and creativity.

Also there will be a brief report from a test: For several years deeper layers of paint can be made visible with the help of a XRF scans. Matthias Alfeld of the University of Antwerp has scanned paintings in the Gemäldegalerie, of which there are already autoradiographs available. The goal was a direct comparison in order to determine the advantages and disadvantages of both methods.

Primary authors : LAURENZE-LANDSBERG, Claudia (conservator at the Gemäldegalerie, Staatliche Museen Berlin)

Co-authors : Dr. DENKER, Andrea (Helmholtz-Zentrum Berlin für Materialien und Energie)

Presenter : LAURENZE-LANDSBERG, Claudia (conservator at the Gemäldegalerie, Staatliche Museen Berlin)

The new neutron imaging station DINGO at OPAL

A new neutron imaging instrument will be built to support the area of neutron imaging research (neutron radiography/tomography) at ANSTO. The instrument will be designed for an international user community and for routine quality control for archaeology, geology, defence, industrial, mining, space and aircraft applications. It is also a useful tool for assessing hidden features from corroded samples and magnetic imaging of core drill sample to understand human impact in different areas of Australia[2]. The instrument is fully installed and will be operational by end of 2013. The designated instrument position for DINGO [1] is the beam port HB-2 in the reactor hall. The estimated flux for an L/D of approximately 500 at HB-2 is calculated by Mcstas simulation in a range of 4.75 * 107 [n/cm2s]. A special feature of DINGO is the in-pile collimator place in front of the main shutter at HB-2. The collimator offers two pinholes with a possible L/D of 500 and 1000. A secondary collimator will separate the two beams and block one. The whole instrument will operate in two different positions, one for high resolution and one for high speed.

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Primary authors : Dr. GARBE, Ulf (ANSTO)

Co-authors :

Presenter : Dr. GARBE, Ulf (ANSTO)

The new Neutron Imaging Beam Line ANTARES at FRM II

Neutron imaging is a method which is being used in many different applications in the context of nondestructive testing. After six years of operation during which excellent experiments have been performed, the neutron imaging beam line ANTARES at FRM II has been completely rebuilt in the last two years and is now nearing completion. Together with the rebuild a major upgrade has been performed and the beam line now comes back to operation with new features, lower background and higher flexibility.

ANTARES now offers three separate chambers along the beam. The first chamber contains the collimators, instrument shutter as well as all beam formation devices such as filter crystals, double crystal monochromator, velocity selector, etc. Following this chamber the user can choose between two experimental chambers. The first one offers a smaller beam for high resolution / high flux and low background experiments with an adjustable L/D ratio of 100 - 3600. A maximum flux of 1.9E9n/cm2s can be achieved for time resolved imaging. The second chamber has a larger beam size and ample space for even large sample environment. Here the L/D ratio can be varied between 200 and 7100.

In this presentation the new ANTARES beam line and its performance and features will be shown.

Primary authors : SCHULZ, Michael ()

Co-authors : SCHILLINGER, Burkhard () ; Mr. BAUSENWEIN, Dominik (TUM/FRM2 ANTARES) ; SCHMAKAT, Philipp () ; Mr. REIMANN, Tommy (FRM II)

Presenter : SCHULZ, Michael ()

Archaeological Ceramics studied by NAA at the FRM-1

In our studies of archaeological ceramics we employ a number of physical methods, mainly neutron activation analysis, Mössbauer spectroscopy, optical thin section microscopy and X-ray diffraction, in an effort to reach a holistic view of ancient ceramic production and its socio-economic context. The selection of the material for the studies depends on the detailed knowledge of the archaeological situation. The existence of unfired local material for model firing experiments in the laboratory is desirable.

As an important first step in the investigation of a complex of finds, neutron activation analysis followed by a cluster analysis of the data can be used to obtain information on the provenance of the ceramic material, which may not have been produced at the site of excavation. To illustrate both the possibilities and the difficulties of this method, the results obtained at the FRM-1 reactor for two groups of material will be described as examples. One group is from the excavations of the Celtic settlement of Manching (ca. 300 BC - 50 BC). The other group pertains to the Sican civilisation in Northern Peru (ca. 950 - 1050 AD). The material was only recently recovered from the West Tomb at Huaca Loro (ca. 1000 AD) and from a middle Sican multi-craft workshop at Huaca Sialupe (ca. 950 – 1050 AD).

Primary authors : Dr. WAGNER, Ursula (Physics Department E15, TU München)

Co-authors : Dr. WAGNER, Friedrich (Physik-Department E15, TU München) ; Prof. GEBHARD, Rupert (Ltd. Sammlungsdirektor Archäologische Staatssammlung München)

Presenter : Dr. WAGNER, Ursula (Physics Department E15, TU München)

Investigation of archaeological ceramics from the Brazilian colonial period by k0 – neutron activation analysis

The archaeological sites of high historical value, Bota-Fora and Hiuton are located in State of Espírito Santo, southwest of Brazil in a region of old Jesuitical Missions built in the XVI century in the colonial period. In this work, the elemental composition of several pottery fragments from both sites were investigated by the neutron activation analysis and the concentration of the elements Al, As, Ba, Br, Ce, Cl, Co, Cr, Cs, Dy, Eu, Fe, Ga, Hf, I, K, La, Mn, Na, Nd, Rb, Sc, Sm, Ta, Tb, Th, Ti, D, V, Yb, Zn, Zr were determined. Statistical analyses of the elemental concentration were performed using the software R which is associated to archaeological interpretation. The results pointed out that the pottery from both sites were made by clay from different places and the earthenware from the site Bota-Fora was made by clay of different compositions pointing out different provenances. These results will support future archaeological studies of works of art produced during the Brazilian colonial period by the group of natives Tupi-Guarani.

Summary :

Keywords: Neutron Activaton Analysis, Archaeology, Pottery, Tupiguarani

Primary authors : Dr. SOARES LEAL, Alexandre (Centro de Desenvolvimento da Tecnologia Nuclear (CDTN) (SERTA))

Co-authors :

Presenter : Dr. SOARES LEAL, Alexandre (Centro de Desenvolvimento da Tecnologia Nuclear (CDTN) (SERTA))

From the cathedral of Augsburg to the old Chinese vase A short report of the neutron activation analysis @FRM II

The neutron source Heinz Maier-Leibnitz (FRM II) is currently the most powerful neutron source in Germany. During the 10 years reactor operation, the modern neutron source provides researchers not only numerous possibilities for the neutron scattering experiments, but also offers opportunities for trace element analysis in the archaeological samples.

The "classical" neutron activation analysis (NAA) at Garching has already written a successful history in the last half century. The so-called “fingerprint” method of analysis of trace elements can bring light into the darkness of the puzzles of the archaeological objects and give us more details about their origins and relations between them.

The bronze portal of the Augsburg Cathedral dated the first half or at the latest the middle of the 11th century is one of 12 major Roman bronze portals in Europe and one of the best examples of medieval casting art north of the Alps [1]. The relief panels of the two non-wide door leaves show mythological, symbolic and several Biblical scenes. There are still a lot of questions about panels. The relations of the images and their meanings, the place of manufacture and the original arrangement of the panels, are still widely unknown [2]. The elements compositions in samples from relief plate frames are analyzed at the FRM II recently. Together with other material science investigations, the NAA should help the archaeologists getting additional information about the classification of the plates. More than 20 trace elements could be quantitative detected by using instrumental neutron activation analysis [3]. A new method is developed to compare the similarity of the objects according to their multi-elements compositions. With the detection of trace of gold, the previous open question whether the door originally gilded or not, could be answered. The differences in concentration of As and Sb in the bronze samples could provide ideas for a new interpretation of the plate classification.

Some other archaeological samples and antiques, such as old jewels of gold and ceramics from the Far East, were also analyzed. The results will be also discussed in my presentation.

Primary authors : Dr. LI, Xiaosong (FRM II / TU München)

Co-authors :

Presenter : Dr. LI, Xiaosong (FRM II / TU München)

„Into the past“ : application of neutron imaging to paleontology

The Cradle of Humankind World Heritage area is a richly fossiliferous karstic system found in Krugersdorp region, Gauteng, South Africa. These karstic systems date from the Plio-Pleistoce (ca 1-4 mya) and have yielded many fossils, including hominids. Fossils are embedded ir cemented limestone matrix of sediment, known as breccia the density and composition of wh varies greatly. Neutron imaging can aid by pre-preparation analysis of breccia blocks to priorit those containing the most promising and/or complete fossils. Currently breccia blocks prioritised for preparation by surface examination and preparing a standard 2kg block of brec can take up to 6 months. Neutron imaging by allowing internal details of breccia blocks to be ea visualised would reveal the contents of a given block and fast-track preparation of promis blocks. A second impo! rtant area where neutron imaging has advantages over clas paleontological methods is the examination of internal or hidden structures of the fossil specim themselves. To see internal features, traditional methodology often entail destructive sampling irreplaceable material. Neutron imaging would be advantageous by being non-destructive revealing (internal) details. Neutron imaging can be used to study specific morphological featu (e.g. trabecular details) inside specimens that is not possible by classical methods. Examples successful application of neutron imaging to fossils will be discussed such as density variations trabecular structures (i.e bipedal locomotion vs. quadrupedal locomotion). Application of neut imaging has already stimulated new directions and research avenues in paleontology and the fut promises further exciting results and insights.

Primary authors : Ms. POTZE, Stephanie (Plio-Pleistocene Palaeontology Section | Departmen Vertebrates DITSONG: National Museum of Natural History)

Co-authors : SCHILLINGER, Burkhard () ; Mr. DE BEER, Frikkie (Necsa)

Presenter : Ms. POTZE, Stephanie (Plio-Pleistocene Palaeontology Section | Department Vertebrates DITSONG: National Mu

Imaging with cold and with fast fission neutrons on breccia containing animal and hominid fossils

A string of limestone caves spans more than half the African continent. When ceilings caved in, the holes formed a deadly trap for animals and hominids. Fossils mixed over time with soils and limestone into a conglomerate called breccia, sealing the fossils away from oxygen. The Ditsong Museum in Pretoria, South Africa, has a research program to retrieve fossils from breccia both mechanically and chemically, which is a very time-consuming process of many weeks and months even one block. However, some of the most valuable fossils, e.g. Ms. Ples, the oldest known hominid skull, were later found in blocks of breccia that had already been sorted out as worthless, and dumped. Pre-examination of the blocks before processing is thus desireable. X-rays deliver no contrast between fossils and breccia, so for the first time, examinations were done with cold, low energy neutrons, and fast fission (high energy) neutrons at the imaging facilities ANTARES and NECTAR of the esearch reactor FRM II of the Heinz Maier-Leibnitz Zentrum of Technische Universität München, Germany. This talk will explain the setup of the facilities and experiments, present the results and determine the penetration depths for slow and for fast neutrons. The results will be used for serial investigations at the upgraded neutron imaging facility SANRAD at NECSA, South Africa, and possibly influence the design of the future replacement reactor at NECSA.

Primary authors : Dr. SCHILLINGER, Burkhard (FRM II)

Co-authors : Ms. POTZE, Stephany (DITSONG National Museum of Natural History) ; Mr. DE BEER, Frikkie (Necsa)

Presenter : Dr. SCHILLINGER, Burkhard (FRM II)

Neutron Imaging Integrated into the Development and Activities of the South African Palaeo-Scientific Community

The South African Nuclear Energy Corporation (Necsa) is situated within the Pelindaba complex at the North East corner of the "Cradle of Human Kind". It is on the same dolomitic band that houses the fossil deposits and would have been included within the Cradle were it not a nuclear facility. Geologically it is part of the Cradle and spatially it is right next to the Cradle. The Radiation Science (RS) Department of Necsa, which is part of its R Division, has recently designated heritage and heritage materials studies as one of the core focus areas of its activities in its support of the National System of Innovation and Necsa's mandate to promote research in radiation sciences. Necsa provide access of its scientific expertize and facilities to the Cultural heritage communities to exploit the potential and capabilities. In this regard, Necsa provide capacity to exploit neutron- and micro-focus X-ray tomography scanning services (non-diagnostic) to many researchers in a variety of scientific research fields – also to the palaeosciences and archaeological sciences. The complementary nature of neutron and X-ray tomography allows for a full understanding on a non-destructive basis of the internal information of e.g. fossil bearing rock, through the creation of detailed 3D virtual images of fossil material – critical to the scientist in evaluating internal aspects of fossils.

Palaeontologists and palaeoarcheologists increasingly utilise these techniques to investigate critical internal detail of valuable specimens in a non-invasive manner with no damage to the specimens.

It makes scientific sense for a fully staffed acetic acid laboratory to be physically linked to a tomography facility and Necsa has embraced the concept and allocated on-site premises in the same building as the tomography unit for the establishment of an acetic acid laboratory. This paper will outline the needs of palaeo-researchers and how Necsa fulfils those needs.

The satellite lab will constitute an independent processing facility with full time staff to be used communally by South African and International palaeontologists and palaeonthropologists. It has significant advantages:

- Standardizes processing and curatorial protocols.
- employment for staff drawn from the Cradle itself (vs. grant-based, term-limited employment)
- Development of a critical and unique skills base tied to stability of employment that ensures professional and consistent results.
- Minimizes the duplication of resources by South African and foreign granting agencies.
- Encourages collaborations between different scientific teams, Necsa, DAC, DST and COH WHS Management Authority
- Provides a South African based mechanism for training students, young scientists and field researchers in methods used in palaeontology and palaeoanthropology
- In the longer term the Satellite lab provides an opportunity to spread the tourism focus to another area of the Cradle (Maropeng/Sterkfontein towards the South West , and Necsa in the North East). Necsa already has an impressive visitor centre.

Primary authors : Mr. DE BEER, Frikkie (Necsa)

Co-authors : NOTHNAGEL, Gawie (Radiation Science Department, Necsa, SA) ; FRENCH, Glyn (private (Landowner))

Presenter : Mr. DE BEER, Frikkie (Necsa)

Problems and limitations of X-ray microtomography for the endostructural characterization of fossil tooth tissues

Dental remains usually represent the most common available evidence attesting of the life history of extinct taxa. Advances in comparative developmental morphology and microanatomy of extant and extinct primates show that a significant amount of valuable information for assessing their taxonomy, adaptive strategies, evolutionary pathways and phylogenetic relationships are preserved in the structure of tooth crowns and roots. In response to the potentially conflicting requirements of safeguard vs. fruition/exploitation of this record, the available technologies based on X-ray microfocus tubes (μCT) and synchrotron (SR-μCT) microtomography allow the quantification of the meso-microstructural signature stored in the mineralized tissues through high-resolution "virtual histology". Nonetheless, depending on their taphonomic history and degree of diagenetic alterations, fossil tooth tissues do not systematically provide a distinct inner signal, preventing the extraction of crucial paleobiological information.

In our record, the extent of such problems and investigative limitations is well illustrated by two spatially and chronologically distant fossil dental assemblages whose taxonomic and phylogenetic status is a matter of contention since over a century: the Late Miocene ape Oreopithecus bambolii and the Lower-Middle Pleistocene hominids from Java, Indonesia.

O. bambolii is peculiar in many aspects, with a typical hominoid postcranial skeleton, a very specialized dentition, and an unusual cranial morphology, developed under insular conditions. Craniodental anatomy has always played an important role in discussions of the phylogenetic relationships of this fossil primate. Albeit reported as the "enigmatic anthropoid", Oreopithecus is currently broadly accepted as a hominoid belonging to the great ape and human clade (Hominidae s.l.). In order to assess its intraspecific variation, we used SR-μCT to image two dental samples from Sardinia and Tuscany, Italy. However, while the record of the Sardinian specimens revealed sufficient contrast among the mineralized components, thus allowing a reliable quantification in terms of tissue proportions and enamel-dentine junction surface morphology, it was not possible to confidently retrieve any information from the assemblage from Tuscany despite the application of advanced post-processing imaging techniques.

Most of the fossil hominid dental remains collected at Java have been attributed to Homo erectus. However, due to the strong convergence in external size and morphology between the human and the pongine (orangutan-like) molar crowns, a larger taxonomic diversity has been suspected since the early discoveries. In our sample from the Sangiran Dome, only less than 50% among the fifteen fossil molar crowns imaged by μCT provided a signal suitable for endostructural assessment, the remaining cases revealing a contrast threshold enamel-dentine-sediment inhibiting any reliable quantitative analysis.

Diagenetic alterations capable to variably hide the inner structural morphology are quite common in the tooth fossil record. Besides phase-contrast SR-μCT, neutron microtomography (n-μCT), whose absorption profile differs from X-rays by being more strongly attenuated by organic than mineral material, could represent an effective investigative tool for imaging the fossil material with a better contrast resolution despite the variably hazy appearance of the inner structural signal.

Primary authors : Dr. ZANOLLI, Clã©ment (Multidisciplinary Laboratory, The 'Abdus Salam' International Centre for Theoretical Physics, Trieste, Italy)

Co-authors : Dr. MANCINI, Lucia (Elettra-Sincrotrone Trieste S.C.p.A., Basovizza (Trieste), Italy) ; Dr. KULLMER, Ottmar (Department of Palaeoanthropology and Messel Research, Senckenberg Research Institute) ; Prof. MACCHIARELLI, Roberto (UMR 7194 CNRS, Muséum National d'Histoire Naturelle, Paris, France; Département Géosciences, Université de Poitiers, France) ; Prof. ROOK, Lorenzo (Dipartimento di Scienze della Terra, Università di Firenze, Italy) ; Dr. SCHILLINGER, Burkhard (FRM-II, Technische Universität München, Garching, Germany) ; Prof. SCHRENK, Friedemann (Department of Palaeoanthropology

and Messel Research, Senckenberg Research Institute) ; Prof. TUNIZ, Claudio (Multidisciplinary Laboratory, The 'Abdus Salam' International Centre for Theoretical Physics, Trieste, Italy; Dipartimento di Biologia Ambientale, Università di Roma "La Sapienza", Italy; Centre for Archaeological Science, University of Wollongong, Australia) ; Dr. VODOPIVEC, Vladimira (FRM-II, Technische Universität München, Garching, Germany)

Presenter : Dr. MANCINI, Lucia (Elettra-Sincrotrone Trieste S.C.p.A., Basovizza (Trieste), Italy)

Neutron radiography studies of the Przeworsk culture objects from Czersk

The remnants of Przeworsk culture (from 2th century BC to 3rd century AD) have been found in many sites of Poland (Lower Silesia, Greater Poland, central Poland, and western Mazovia and Lesser Poland) for almost a century. Since the Przeworsk culture objects have some features of the Roman and Scandinavian products the studies of these artefacts are of unquestionable importance for European cultural heritage.

The results of the studies performed on several objects found at the Przeworsk culture burial site at Czersk (central Poland) are presented. This ancient cemetery has been thoroughly searched since 2008. The main experimental tool was the thermal neutron (white beam) radiography supplemented with X-ray, neutron diffraction, SEM and laser ablation techniques. The study dealt with few clearly identified objects like metal parts of an ancient shield and burial urn as well as few initially unidentifiable aggregates of many different objects found at the excavation site. The neutron imaging helped in identification of their components.

The main objects investigated came from a shield excavated at Czersk and comprised the central part and the handle with an unique silver cladding. Elemental composition of the artefacts was determined SEM with Energy-dispersive X-ray analysis and Laser Ablation Inductively Coupled Plasma Mass spectrometry. The bronze alloy was confirmed as a main component of the objects' body and the silver was identified in the handle cladding. The results were compared with tests carried out for two fibulas found at the same site. The ornaments on the surface of the handle were revealed with neutron imaging despite significant corrosion decay. The phase composition of the bronze alloy forming the handle was determined with X-ray and neutron diffraction. In particular the small angle neutron scattering revealed an advanced phase decomposition stage of the bronze in nano-scale.

Three heavily corroded aggregates of the initially unidentifiable objects were carefully studied with neutron imaging. The inspection of the neutron images revealed the presence of the spear or arrow heads, clip and spur parts clumped together inside of the objects.

Primary authors : Dr. MILCZAREK, Jacek (National Centre for Nuclear Research)

Co-authors : Dr. FIJAŁ-KIREJCZYK, Izabela (National Centre for Nuclear Research (NCBJ))

Presenter : Dr. MILCZAREK, Jacek (National Centre for Nuclear Research)

High Flux Prompt Gamma Activation Analysis: a non-destructive technique for determination of elemental composition of cultural heritage objects

Possible applications of the PGAA based techniques for cultural heritage objects will be presented in this overview talk. The (n,gamma) capture reaction is used for non-invasive determination of the elemental composition in samples while irradiated by high intensity flux of cold neutrons. Generally, the PGAA method is used in applications like archaeometry and archaeology, geology, environmental science, new material research…

In frame of the Ancient Charm project we have performed a feasibility study for 3D mapping of elemental composition of archaeological objects up to size of ca. 5cm x 5cm x 5cm. For that purpose, low-resolution neutron tomography (NT) set-up was combined with the spatially resolved PGAA technique called Prompt Gamma Activation Imaging (PGAI). With PGAI, the neutron beam is collimated to a pencil beam of 2 mm x 2 mm and the detector sees only a 2 x 2 x 2 mm3 voxel of the sample. While scanning the object in the pencil neutron beam, the elemental composition can be given in dependency on the position in the sample. The gained position information is then compared and combined with the internal structure information of the object determined by the neutron tomography 3D image which was performed before the actual PGAI scan.

A large variety of experiments at the PGAA instrument at FRM II and some prominent results will be described and discussed. Possible ways how to improve e.g. the detection limits for better determination of trace elements will be proposed.

Summary :

An overview talk explaining the PGAA method and PGAA based techniques. Some examples of PGAA experiments on archaeological objects will be given.

Primary authors : KUDEJOVA, Petra ()

Co-authors : SOELLRADL, Stefan () ; REVAY, Zsolt ()

Presenter : KUDEJOVA, Petra ()

Non Destructive elemental and mineralogical evaluation

of Greco-Roman Bronzes.

Non-destructive PGAA and ND evaluation measurements were conducted on several coins representative of a 1st century AC Greco Roman copper coin collection along with a number of bronze artifacts from a horse harness found in the course of archaeological excavations at the site of Lithochori-Kavala, North East Greece and currently preserved in Kavalla Museum. The aim of this work was : On the one hand to determine the elemental compositions of Cu-Sn-Zn, and identify traces of secondary elements dissolved in the primary matrix. On the other hand to obtain, through microstructural analysis, bulk information of the metallic phases present in the samples, second to collect data of the structure and compositions of the corrosion products and third to diagnose uncommon additions / growths, observed on the bronzes that may have been deposited during burial.

Spectral analyses of the PGGA patterns showed that the bulk metallurgical distributions with the corresponding weight fractions of the primary elements Cu-Sn - Zn and the Fe may be determined rather accurately. Furthermore traces of secondary elements dissolved in the primary matrix, that have not been discovered in the SEM investigation, can also been identified within the experimental limitations.

Analysis of the diffraction patterns suggests that the main phases are Cu-alloys namely α-CuSn / CuZn with significant additions of Pb. Additionally, significant amounts of cuprite and nantokite confirm the high degree of corrosion observed on the measured objects. Secondary - traceable phases of pyromorphite, chalcopyrite, and Fe-oxides suggest extensive environmental interaction with either phosphorous enriched or anaerobic and humus reach soil .The corresponding tin/zinc content in the Cu-alloy for each item has been estimated from the refined lattice parameters and it is in fair agreement with the value determined by PGGA. The diffraction profiles indicate that there are three distinct categories of coins. The first one is a set of three coins consisting mainly of copper/tin alloy Cu alloy phases with high quantities of Cu_2O , CuCl and Pb . The second group is formed of coins with high Zinc/ copper alloy ratio and the third is a collection of four coins consisting of very high amount of Cu with small tin inclusions. The comparison between the diffraction profiles of the original coins to those of the replicas present distinct variations that may be used to differentiate between authentic and fake pieces. As the results of this preliminary inquiry are encouraging, further analyses will probably help to further the comprehension of Thracian coinage during the first century.

Primary authors : Dr. SIOURIS, Ioannis (Democritus University Thrace)

Co-authors : Dr. KATSAVOUNIS, Stefanos (DEMOCRITUS UNIVERSITY OF THRACE) ;
Mrs. SPYROPOULOU, Evmorfoula (Democritus University of Thrace) ; KUDEJOVA, Petra () ;
Dr. HOSER, Andreas (Helmholtz-Zentrum Berlin für Materialien und Energie GmbH)

Presenter : Dr. SIOURIS, Ioannis (Democritus University Thrace)

NIPS-NORMA: a new neutron-based element-mapping and imaging facility at the Budapest Research Reactor

Numerous non-destructive techniques utilize neutron attenuation, scattering or capture to gain morphological, structural or elemental information about the material under study. However, few attempts have been made so far to use neutron-induced gamma radiation for 3D element imaging. A novel non-destructive method is being developed to determine the distribution of major elements and visualize internal arrangement of heterogeneous objects, by combining position-sensitive prompt-gamma activation analysis (PGAI) and neutron radiography/tomography (NR/NT).

Though conventional PGAA provides limited or no spatial resolution, a strong collimation of the neutron beam and the gamma-detection reduces the sampling volume, i.e. the origin of the detected gammas is localized. This provides the basis to construct the element distribution of the sample by systematic scanning, i.e. measuring a set of gamma spectra at different positions. This technique is called Prompt Gamma Activation Imaging (PGAI). While PGAI gives a 3D distribution of the elements, NR/NT produces high-resolution 2D/3D images to survey the structure of the object. The two datasets can be merged into a common coordinate system for a sound chemical and structural interpretation.

It is often sufficient to measure the elemental compositions only at selected spots of the object and link them with the structure revealed by NR/NT. This makes the technique, called tomography/radiography-driven PGAI, much more feasible and cost-effective.

The first ever facility using direct element mapping in combination with neutron imaging was constructed at the Budapest Research Reactor in 2007. Recently a permanent facility, called NORMA, was commissioned. The setup consists of a variable neutron collimator, sample chamber, neutron tomograph, xyzw sample stage, gamma collimator, a Compton-suppressed and well shielded high-purity germanium gamma-ray (HPGe) detector and acquisition software.

The facility is open to the scientific community via EU-funded access programs related to the Budapest Neutron Center in the fields of industrial, cultural heritage, nuclear data and material science applications. Results of this non-destructive technique will be presented here from various fields of applications.

Primary authors : Dr. KIS, Zoltán (Centre for Energy Research, Hungarian Academy of Sciences)

Co-authors : Dr. SZENTMIKLÓSI, László (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. BELGYA, Tamás (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. RÉVAY, Zsolt (Technische Universität München, Forschungsneutronenquelle Heinz Maier-Leibnitz (FRM II))

Presenter : Dr. KIS, Zoltán (Centre for Energy Research, Hungarian Academy of Sciences)

In-Situ elemental analysis and provenance study of terengganu´s historic stone using neutron induced prompt gamma-ray techniques

Batu Bersurat Terengganu (Terengganu's inscribed stone) is the oldest artifact with Jawi writing on it. The artifact proves that the Kingdom of Terengganu exist earlier than 1326 or 1386. It was accidentally discovered near Tersat River at Kampong Buluh, Kuala Berang, Terengganu, Malaysia by a gold & tin trader after a flash flood hit Kuala Berang. He was then presented the stone to the sultan (Zainal Abidin Ill). The sultan had it placed in the fort on Bukit Puteri, and there it remained until 1922 when a British colonial official had it sent to the Raffles Museum in Singapore for examination.The inscription turned out to be a proclamation issued by the "Sri Paduka Tuan" of Terengganu. The significant is the date on the first face of the inscription, which is given as the year 1303 AD.

The stone itself is still shrouded in historical mysteries. What happened to the first kingdom of Terengganu? What indeed was its origin and why did this kingdom subsequently disappear? Was the inscribed stone made of stone from local deposit? And if the stone was made locally, where was it made on the Terengganu site? There is no scientific data or reports available in opened literatures up to now. It is a most treasured heritage.

Due to invaluable historical values, at the UNESCO International Advisory Committee meeting in Barbados on 13 July 2009, the 700-year-old inscribed stone was listed as an item eligible for world heritage recognition. A lot of methods is being developed by scientists, archaeologists and historians to study different aspects of the inscribed stone. The first scientific project is to develop a non-intrusive analytical inspection based on nuclear techniques. Conventional neutron activation analysis (NAA) is a well established nuclear technique, which may be used to investigate elemental composition of materials. However, a sample needs to be taken before irradiating with neutrons in nuclear reactors. Due to high historical values of Terengganu's inscribed stone, taking samples from it is restricted by the Terengganu State Museum Authority. X-ray flourensence (XRF) method using a portable system is another choice but it is limited to the surface analysis. Neutron induced prompt gamma-ray technique (NIPGAT) using isotopic neutron source in conjunction with portable gamma-ray spectroscopy system can be applied to studies a large variety of samples. Indeed, it may be used to address the above-mentioned questions in non-destructive manner. Neutrons are useful as probes for non-destructive examination of extended media because neutrons can travel relatively long distances before interacting with the nuclei of the media. It is for this reason that NIPGAT is being developed at the Malaysian Nuclear Agency for in-situ quantitative elemental analysis and finally for provenance study of the Terengganu's inscribed stone.

This paper describes the experimental set-up and presents some of the results of NIPGAT obtained from many samples from two types of stones, namely Dolerite and Granite which are most likely the river boulders used for making the inscribed stone. The MCNP code developed by Los Alamos Laboratory was also used to design and optimise the laboratory and field experimental facilities. Based on these results a portable system based on neutron-induced prompt gamma-ray techniques was constructed. Using this system, the inscribed stone can be investigated in a volumetric manner and non-destructively thus increasing measurement accuracy. The scientific data or technical evidence obtained from this project will be of great benefits not only to curators and archaeologists but also to scientists, historians and many others.

Primary authors : Dr. ABDULLAH, Jaafar (Malaysian Nuclear Agency)

Co-authors :

Presenter : Dr. ABDULLAH, Jaafar (Malaysian Nuclear Agency)

Neutron scattering analyses of cultural heritage objects

Neutron scattering is a vital research and analysis technique for exploring the structure and dynamics of materials and molecules. It provides unique and complementary information to that available from other diagnostic techniques. The main advantage of neutron-based analytical methods is that compositional and structural information can be obtained on the intact object without cutting out a sample of the material. In the past few years, increasing numbers of cultural heritage materials characterisation projects using neutron diffraction have been carried out at the pulsed neutron spallation source ISIS, UK. Neutron diffraction is used for quantitative analysis of metal, mineral, and intermetallic compounds present in a sample. In metals, residual strains, microstrains, and grain orientations can be analysed giving evidence of manufacturing techniques like casting, hammering, and annealing. The structure data are evaluated to obtain estimates of chemical concentrations. For instance, in steel, the cementite content can be analysed by TOF-ND and then used to calculate the bulk chemical composition in terms of weight percent of carbon. Lead added to copper or bronze does not go into solution in the copper and can be directly and accurately determined. The possibility of collecting structural data non-destructively from many points on one and the same object is of advantage for many cultural heritage studies especially if neutron scattering is combined with other non-destructive techniques such as X-ray or neutron imaging.

Primary authors : Dr. KOCKELMANN, Winfried (STFC Rutherford Appleton Laboratory)

Co-authors :

Presenter : Dr. KOCKELMANN, Winfried (STFC Rutherford Appleton Laboratory)

Non-invasive characterization of ancient Japanese helmets through ToF-Neutron Diffraction

In historical times, armours and weapons were generally made from the best available materials and using the best known technologies. Japanese armours have always been considered a wonderful example of the union of elegance and functionality, becoming a symbol of command and an expression of social status.

From the 15th century, with the advent of large armies of foot soldiers and the beginning of a decline in fighting on horseback, the Japanese helmet, the kabuto, became one of the most important elements among an armour's constituents, because it allowed the samurai to stand out in the battlefield. Nonetheless, scientific and technological research into this category of artefacts has scarcely developed. Very few Japanese historical armours have been thoroughly investigated, owing both to the rarity of the available armours and to their extraordinary beauty. In addition, because of their generally excellent state of preservation, the traditional (invasive) methods of analysis did not suit these objects. For these reasons, the present investigation has been carried out using a non-invasive approach, namely thermal neutron diffraction [1]. The study focussed on seven iron and steel helmets, belonging to different periods and different levels of quality, kindly provided by the Stibbert Museum in Florence and by a British private collector.

The experiments were carried out at the INES diffractometer at ISIS, the pulsed neutron source in the UK. Thanks to the small interaction of thermal neutrons with other nuclei and to the consequent high penetration power, neutrons represent the ideal probe for dense matter investigation, and can be effectively used to study the microscopic properties of metal artefacts, with thickness of several centimetres [1].

Similarly to X-ray diffraction, the present analysis has been carried out in order to determine, through Rietveld refinement of the neutron diffraction patterns [2], the phase composition of the different parts, to evaluate the quality of the steel (i.e. the carbon content level), and hence gain information on the smelting procedure and the present conservation status of the metal [3]. Moreover, the analysis of the shape and relative intensities of the main phase diffraction peaks [2] provided indirect information on the thermal treatments and shed some light on the likely working techniques [4,5].

Further neutron techniques have been applied, on a kabuto of particular value (both from the artistic and technological point of view) among the analyzed samples. The helmet has been characterized using the instrument for thermal neutron radiography and tomography NEUTRA (SINQ, Paul Scherrer Institut, CH) [6].

This work represents the starting point for the characterization of materials and technology used to make a particular class of artefacts of great interest from the point of view of anthropology, art and technology. In light of the results of this thesis work, the combined use of neutron techniques (imaging and diffraction) is one of the best approaches for a non-destructive characterization of the entire volume of metal artefacts of artistic interest.

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Summary :

In this work we present an extensive time of flight neutron diffraction (ToF-ND) study on seven Japanese helmets (kabuto) made between the 16th and 17th Century. The experiments were carried out at the INES diffractometer at ISIS, the pulsed neutron source in the UK. By this non-invasive approach we have been able to determine quantitatively the phase composition and the micro-structural properties of these artefacts, confirming that the use of ToF-ND represents one of the most suitable non-destructive approaches for the characterization of metal archaeological artefacts.

Primary authors : Mrs. FEDRIGO, Anna (CNR-ISC)

Co-authors : Mrs. SALVEMINI, Filomena (CNR-ISC) ; Dr. GRAZZI, Francesco (CNR-ISC) ; Dr. ZOPPI, Marco (CNR-ISC) ; Dr. WILLIAMS, Alan (Wallace Collection) ; Dr. SCHERILLO, Antonella (STFC-ISIS) ; Mr. CIVITA, Francesco (Museo Stibbert)

Presenter : Mrs. FEDRIGO, Anna (CNR-ISC)

Combining neutron methods – a unique science tool kit in artefact analysis

Nowadays growing interest is devoted to exploration and protection of artefacts belonging to our treasure of Cultural Heritage. The application of analytical tools of natural sciences has become an important field of archaeology by now. The investigations usually concern dating, provenance, manufacturing techniques, workshop affinities, as well as fake identification or preservation of objects. Neutrons are perfect tools of archaeometrical studies due to their non-destructive and non-invasive nature. Various kinds of neutron techniques can be used to explore the compositional or structural features of the samples. Based on the detection of characteristic γ-photons produced in (n, γ) reaction, one can determine the 'bulk' elemental composition of the objects. Or, investigation of neutron scattering patterns gives information on the atomic, molecular or nano-scale structural properties: crystalline/amorphous morphology, phase composition, mechanical strains, impurities, etc. Furthermore, neutron imaging techniques (tomography/radiography) play an important role in the exploration of the deep bulk topology or in-side content of artefacts.

The Budapest Neutron Centre (BNC) has long traditions in application of neutrons for archaeology research. In particular, the Prompt Gamma Activation Analysis (PGAA) group has made a pioneering work to apply this technique to archaeometry. Small Angle Neutron Scattering (SANS) and Neutron Diffraction (ND), Neutron and Gamma Radiography (NGR) facilities have been involved in various archaeological research project, both at national and European level [1]. Since 2009 BNC contributes to the EU FP7 Cultural Heritage Research project called CHARISMA. The project gathers large European museums as well as provides access to a network of large scale facilities; BNC is one of the transnational access providers by the services offered at its neutron facilities: diffractometers (ND), SANS, PGAA stations and imaging facilities. Complementary measurements are offered by the use of External Beam milli-PIXE and compact XRF Spectrometers, microscopes, mass spectrometers etc. also at BNC site.

Several case studies as comprehensive analysis of archaeological objects by combined neutron techniques will be given. For example, manufacturing processes of the earliest Hungarian Bronze Age defensive armour such as helmets, greaves and cuirass were studied by PGAA, PIXE and ToF-ND. Another highlight resulting from a series of neutron studies is the establishing the meteoritic origin of the earliest known iron objects, a set of elongated iron beads, excavated a hundred years ago at Gerzeh, Egypt in a tomb dated to circa 3300 BC. Predating the invention of bloomery smelting by nearly two millennia, they are commonly assumed to be made from meteoritic iron. Our combined neutron measurements have provided unambiguous results to discuss and offer a comprehensive interpretation of the origin and fabrication of these beads and their significance for the history of iron working development [2].

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Primary authors : Dr. ROSTA, László (Budapest Neutron Centre)

Co-authors :

Presenter : Dr. ROSTA, László (Budapest Neutron Centre)

The manufacturing of Japanese swords: a non destructive quantitative analysis of steel composition and microstructure through neutron diffraction and neutron imaging techniques

In this work we present the characterization of the peculiar compositional and micro-structural properties of the different kinds of steel used to forge ancient Japanese swords according to the five historical different forging traditions (Gokaden) and to the period [1-2]. In order to achieve such a goal, a large number of samples needs to be studied and, due to the unique nature of the artifacts, only a non-destructive approach is now possible. Japanese swords were analyzed in the past through traditional methods [3-6] aiming to characterize the steel composition and microstructure, always applying a destructive methods (metallography and electron microscopy) and the results were of great interest but this kind of approach is impossible to be applied at a large extent. Neutron diffraction and neutron imaging represent the ideal method to reach the proposed goal in a non destructive way since they permit to obtain quantitative information about composition, microstructure, morphology and spatial distribution of the material in the samples.

A total of seven intact Japanese swords and nine broken ones, pertaining to different periods ranging from 14th until 19th century, have been analyzed through neutron diffraction and neutron imaging techniques [7-10]. The samples have been made available by the Stibbert Museum and Wallace Collection staff and by private collectors.

Neutron experiments have been performed in two different facilities: ISIS (UK) and SINQ (CH). Diffraction measurements have been applied on all the selected samples by using the INES [11], ENGIN-X [12] and POLDI [13] diffractometers while neutron imaging measurements have been performed on the ICON [14] beam-line. Neutron imaging measurements covered the full body of most of the samples while neutron diffraction experiments were focused on specific parts of the swords as the tip, the core of the blade (differentiating among the cutting edge, the centre and the back) and the tang to determine the quantitative distribution of the metal and non metal phases. The comparative analysis of the phase distribution and the tomographic reconstruction of the samples permitted to identify peculiar characteristics related to the forging traditions and periods of the Japanese history and to determine the inner metal phase distribution thus confirming the differentiate specialization of the single parts of this kind of swords.

Due to the high quality level of the results in terms of spatial resolution and quantification of phases and microstructures, this multi-methodological non-destructive approach presents an incomparable potential in the field of historical metallurgy.

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Primary authors : Dr. GRAZZI, Francesco (CNR-ISC)

Co-authors : Mrs. SALVEMINI, Filomena (Istituto dei Sistemi Complessi-Consiglio Nazionale delle Ricerche) ; Ms. BARZAGLI, Elisa (CNR-ISC) ; Dr. SCHERILLO, Antonella (STFC-ISIS Neutron Source / CNR) ; Dr. WILLIAMS, Alan (Wallace Collection) ; Mr. PEETERMANS, Steven (PSI-SINQ) ; Dr. DAVYDOV, Vadim (PSI-SINQ) ; Dr. PARADOWSKA, Anna Maria (ANSTO) ; Dr. CIVITA, Francesco (Museo Stibbert) ; Mr. FRANCI, Riccardo (Museo Stibbert) ; Dr. LEHMANN, Eberhard (PSI-SINQ) ; Dr. ZOPPI, Marco (CNR-ISC)

Presenter : Dr. GRAZZI, Francesco (CNR-ISC)

Energy-selective neutron imaging in cultural heritage

Neutron imaging is a method of non-destructive investigation for objects of scientific and technological interest. Within the last decade, neutron tomography and radiography have significantly gained importance among the neutron science community. One of the reasons is the fast development in digital image recording and processing technology, which has allowed overcoming of some previous limitations in spatial and time resolution. Another reason is that in addition to the attenuation contrast technique, new innovative methods for neutron imaging are being implemented. Using monochromatic neutrons for imaging, complementary contrast due to the coherent scattering in polycrystalline materials can be obtained, providing information about structural changes or composition heterogeneity. The energy-dependence of the neutron imaging can be used for localization of Bragg edges with high precision which helps for mapping of residual stresses and textures in metallic samples. Tomography investigations with monochromatic beams allow for phase separation in heterogeneous samples. The application of these methods to cultural heritage objects provides information about the manufacture process and the trade paths in the ancient times.
Examples of performed studies at the imaging instrument CONRAD(HZB) at the steady state (reactor based) neutron source BER-2 will be presented and discussed.

Primary authors : Dr. KARDJILOV, Nikolay (Helmholtz-Zentrum Berlin)

Co-authors : Dr. HILGER, André (HZB) ; Dr. MANKE, Ingo (HZB) ; Mr. WORACEK, Robin (UT) ; Prof. PENUMADU, Dayakar (UTK) ; Dr. VISSER, Dirk (Department of Physics, Loughborough University) ; Prof. BANHART, John (HZB)

Presenter : Dr. KARDJILOV, Nikolay (Helmholtz-Zentrum Berlin)

Application of a pulsed neutron transmission method to a cultural heritage study

A pulsed neutron transmission imaging method is a unique method which can give spatial dependent information on physical parameters of materials, such as crystallographic characteristics, elements, magnetic field, and so on. These data are obtained by analyzing the transmission spectra dependent on the neutron wavelength observed at each pixel of a 2-D position sensitive detector. We succeeded in obtaining the crystallite size, preferred orientation, lattice spacing, and element information of some metal materials. We have considered this method would be useful for studying the cultural heritage materials. As the first test samples we have studied Japanese swords. The Japanese swords are well known for their special characteristics. We performed experiments to reveal what kinds of information could be obtained by exploiting pulsed neutron transmission methods. We examined four Japanese swords. Three of them are fragments of swords and one is a full size. Two fragments were produced in Okayama prefecture in 16 and 17 century, and the third one in Kanagawa prefecture. We used a GEM detector for the fragments and a MCP detector for the full size.

Summary :

From the fragment sample we obtained crystallite size information and preferred orientation information. The crystallite size of the swords in Okayama is almost the same, about 3.0 m, and that in Kanagawa is about 3.7 m. Degree of the anisotropy is similar for the old Okayama fragment and the Kanagawa one. The new Okayama one is rather isotropic. We obtained the lattice spacing distributions from edge to back for the full size sword. There are clear differences between middle position and edge position of the sword. Martensitic phase was observed at the edge position and it disappears in the tang (nakago) area. These results indicate that the pulsed neutron transmission method is one of useful non-destructive inspection methods for cultural heritages.
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Primary authors : Prof. KIYANAGI, Yoshiaki (Hokkaido University)

Co-authors : Mr. AYUKAWA, Naohiko (Hokkaido University) ; Dr. SHIOTA, Yoshinori (Hokkaido University) ; Dr. KINO, Koichi (Hokkaido University) ; Mr. SATO, Tomoya (Hokkaido University) ; Dr. SATO, Hirotaka (Hokkaido University) ; Dr. KAMIYAMA, Takashi (Hokkaido University) ; Dr. GRAZZI, Francesco (Consiglio Nazionale delle Ricerche Istituto dei Sistemi Complessi) ; Dr. ANTONELLA, Scherillo (Rutherford Appleton Laboratory) ; Prof. UNO, Shoji (High Energy Accelerator Research Organization) ; Dr. TREMSIN, Anton (University of California at Berkeley)

Presenter : Prof. KIYANAGI, Yoshiaki (Hokkaido University)

Neutron Computed Tomography as a Basis for Concepts in Preserving Wooden Works of Art

Since any wooden object is prone to biological degradation some artworks were treated with carbolineum as a preservation agent like railway sleepers. However, this substance reappeared with time on the surface through the painting layers. This not only had an impact on the aesthetics and the stability of the coatings but also exhibited a release of a carcinogenic pollutant into the environment. This made new concepts for preservation measures necessary for both, extracting the pollutant carbolineum and novel protective measures.

An essential prerequisite to remove the impregnation now undesired due to its noxious nature is the knowledge of its distribution inside of the object. The method of choice for this purpose is computed tomography (CT). However, it has to be expected that the impregnant consisting of a carbon hydrogen compounds might be diffusely distributed within a matrix composed of the same elements. Therefore, a radiological method is required which is sensitive to density contrasts of carbon and hydrogen. This makes neutron CT the method of choice. The size of the objects to be interrogated makes it necessary to apply fast neutrons, as they are available at the NECTAR facility of the Heinz Maier-Leibnitz Forschungsreaktor of the Technische Universität München (fission neutrons, 1.8 MeV mean energy). Several steps in image processing have been applied and will be discussed as well as the method of CT reconstruction with parallel beam geometry. Wooden sculptures from an epithaph in the St. Laurentzius Church in Tönning (Holstein, Germany) showing the problems as described above have successfully investigated by neutron CT. Densities have been distributed heterogeneously with high concentrations underneath the surface. However, the nature of these dense areas remains to be elucidated.

Primary authors : Dr. OSTERLOH, Kurt (BAM Federal Institute for Materials Research and Testing)

Co-authors : Dr. NUSSER, Amelie (Rathgen-Forschungslabor, Staatliche Museen zu Berlin) ;
Mr. SCHWABE, Andreas (Rathgen-Forschungslabor, Staatliche Museen zu Berlin) ;
Dr. KOLKOORIE, Sanjeevareddy (BAM Federal Institute for Materials Research and Testing) ;
Dr. BÜCHERL, Thomas (TU München)

From Neutron Imaging to Neutron Activation Analysis: Neutrons in Cultural Heritage Applications

The McClellan Nuclear Research Center (MNRC) is owned and operated by the University of California, Davis (UC Davis). MNRC's mission is to advance research and applications in the nuclear sciences, including educational and training opportunities for the next generation of nuclear scientists. The MNRC operates a 2 MW TRIGATM reactor, which is currently the highest power TRIGATM reactor in the United States. The MNRC reactor can also be pulsed to approximately 400 MW for 30 milliseconds. The MNRC was originally developed by the US Air Force to detect hidden defects in aircraft structures using neutron radiography. UC Davis took ownership of the reactor in the year 2000, following the closure of the McClellan Air Force Base. Today, MNRC continues to provide and expand the use of its unique neutron radiography capabilities while expanding its applications to various research disciplines. An overview of research at the MNRC will be shown in this presentation.

The Center offers the largest neutron radiography capability in the World: samples as large as 10.00 m long, 3.65 m high, and weighing up to 2,270 kg can be accommodated. Two of the four radiography bays are laboratory-size rooms where smaller samples can be inspected using both radiography and tomography. The maximum usable neutron beam size is 22-50 cm in diameter with intensities of approximately 1x105 n/cm2·s to 1x107 n/cm2·s, depending on the bay used. The highly collimated (L/D up to 350) neutron beams provide high spatial resolution and quality. Dynamic radiography can be taken at 30 frames/second; the film neutron radiography system can produce images with spatial resolutions of 50 μm; and MNRC's neutron tomography systems have an overall spatial resolution of approximately 200 μm.

In addition to neutron imaging, Neutron Activation Analysis (NAA) can be performed at the center. NAA is an analytical method for the determination of elemental concentrations in a material; it has good sensitivity for over 60 elements. The method is based on gamma spectroscopy after sample irradiation with neutrons in a reactor. The detection limits depend on the specific element and the available neutron flux; at MNRC they range from a few micrograms to less than a nanogram independent of the total sample weight. Due to its high achievable accuracy, NAA has recently been identified as a primary method, i.e., a standard method for the certification of reference materials.

Neutron imaging and NAA have already been used extensively in archeology and cultural heritage. Both techniques are non-destructive. Imaging artifacts, such as metal cast sculptures, allows determining their inner structure and how they were manufactured, which may also reveal knowledge transfer between different cultures.

NAA can be used in archeology to source artifacts, pottery and pigments. NAA analysis of the elemental composition of artifacts made of clay, for example, can provide invaluable knowledge about the geographical origin of the clay and can therefore reveal migration and trading routes.

Summary :

We will present neutron imaging and neutron activation analysis based research opportunities at the McClellan Nuclear Research Center (MNRC)in the field of cultural heritage.
MNRC is owned and operated by the University of California, Davis (UC Davis); it operates a 2 MW TRIGA reactor, which is currently the highest power TRIGA reactor in the United States as well as one of the newest research reactors in the US.

Primary authors : Dr. LERCHE, Michael (University of California, Davis)

Co-authors :

Presenter : Dr. LERCHE, Michael (University of California, Davis)

Fifteen years of archaeometry research at the Prompt-gamma activation analysis facility of the Budapest Neutron Centre

In 1995, the prototype of the Budapest prompt-gamma activation analysis facility has been installed on a guided thermal neutron beam of the Budapest Research Reactor. After precise engineering of the detector- and data acquisition system, designing proper shielding and establishing our PGAA library by series of standardisation measurements, the system was ready to explore possible analytical applications by 1996. Following the installation of a Cold Neutron Source in 2000, the thermal equivalent intensity of the neutron beam has increased from 2.5•106 cm-2s-1 up to 5•107 cm-2s-1. As a consequence of further beam adjustments and installation of supermirror guides, we have reached the 1•108 cm-2s-1 intensity by now, while the beam background was simultaneously kept low at about 20 cps [1].

Recognizing the advantages of non-destructivity and the lack of sampling, one of the most promising candidates of applied research was to investigate valuable Cultural Heritage objects, i.e. unique archaeological findings, museum pieces, etc. Since 1997, we studied the possibilities in analysis of various metals, rocks and minerals, ceramics and also glass.

One of the first studies was to investigate the alloying components of late Roman (2nd to 4th c. AD) fibulae found in Hegyeshalom, Hungary. Based on the major components (Cu, Sn, Pb and Zn) we were able to define groups of objects referring to the changes of casting techniques. Besides fibulae, occasionally other bronze objects like helmets, shields, sculptures, etc. were studied, too. In another study, by determination of decreasing silver content of Roman coins, we have followed the course of inflation (decreasing Ag/Cu ratios during reigns of various emperors) [2]. Due to the complexity of bronze and silver spectra and to the phenomenon of recycling of historic metals, provenance research of metal objects is not favoured by PGAA.

PGAA proved to be the most successful in provenance of prehistoric stone tools and of some gemstones [3]. In fortunate cases, some rocks show fingerprint-like elemental composition characteristic for their provenances. In the last 10 years, we have built significant databases containing data of geological references and archaeological objects made of obsidian, flint, felsitic porphyry, greenschist, etc. Provenance study of obsidians is very successful, whereas high-silica flints and other silexes are more problematic. A special study is devoted to provenance of lapis lazuli, the most popular ancient precious stone in the Near-East [4].

Although, ceramics are considered as composite materials containing clay and temper, in some instances bulk elemental analysis can help in determination of raw material sources [5]. If possible, other – sometimes destructive – methods are often required.

Well measurable major components of glass, i.e. Si (sand), Na or K (plant- or wood ash) and Ca or Mg (flux) are characteristic for provenance or workshop and indirectly for age. Colorants like Co, Cu, Mn or Fe can be also quantified. A special attention is paid to B, which was applied on purpose since the 18th century [6].

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Summary :

Following the design, engineering, installation and standardisation procedure, the Budapest PGAA facility became ready to carry on analytical applications in 1996. Thanks to the absolutely non-destructive feature of the method, archaeometry applications seemed to be one of the most adequate topics. Since 1997, we have started to explore the capability of the method on the most frequent types of historical materials, such as metals, rocks, ceramics and glass. The most successful studies and also the limitations are presented in this paper.

Primary authors : Dr. KASZTOVSZKY, Zsolt (Centre for Energy Research, Hungarian Academy of Sciences)

Co-authors : Dr. BELGYA, Tamás (Centre for Energy Research, Hungarian Academy of Sciences) ; Ms. GMÉLING, Katalin (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. KIS, Zoltán (Centre for Energy Research, Hungarian Academy of Sciences) ; Ms. MARÓTI, Boglárka (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. REVAY, Zsolt (Centre of Energy Research, Hungarian Academy of Sciences) ; Dr. SZENTMIKLÓSI, László (Centre for Energy Research, Hungarian Academy of Sciences) ; Dr. SZILÁGYI, Veronika (Centre for Energy Research, Hungarian Academy of Sciences)

Presenter : Dr. KASZTOVSZKY, Zsolt (Centre for Energy Research, Hungarian Academy of Sciences)

Feasibility Study for Detecting the Lost Leonardo Mural by Prompt Gamma Neutron Activation

There has been considerable interest in the application of nondestructive test methods for finding the lost mural of the Battle of Anghiari by Leonardo da Vinci in the Palazzo Vecchio in Florence, Italy. This mural was thought to have be painted in 1505, but it was subsequently covered over by another mural in 1560. Most recently, it has been proposed to use a neutron-based elemental analysis technique, prompt gamma neutron activation (PGNA), to detect elements characteristic of the pigments that Leonardo may have used. To determine the feasibility of this method for finding the lost mural, a preliminary analysis of the probability of detection (POD) has been made using the Currie Equation for estimating the minimum detectable limit for a given element. The first step was to determine the pigment most likely to be detected by PGNA. This involved identifying the palette that Leonardo may have used and then screening them for detectable elements by neutron capture cross-section, gamma ray energies and yields. In addition, interferences from elements in the materials surrounding the mural had to be taken into account. Based on this analysis, mercury, an element in the red pigment vermilion, was the most favorable candidate. The next step was to estimate the total mass of mercury in the field of view based on vermilion layer thickness and surface distribution. Finally, the mercury count rate was modeled using the operating specifications of existing portable prompt gamma neutron activation systems, including thermal neutron flux and gamma ray detector efficiency. It was concluded that the count rate would be too low to be practical.

Primary authors : Prof. LIVINGSTON, Richard (University of Maryland)

Co-authors :

Presenter : Prof. LIVINGSTON, Richard (University of Maryland)

Lapis lazuli: The stone of the Antiquity and their origin

Archaeological objects, like beads, gems, seals and small decorative objects made of lapis lazuli are widely distributed in the ancient East and some date back as early as the second half of the fourth millennium B.C. in Central Asia.

The chemical composition and mineralogy of lapis lazuli has been given considerable attention in the last decades. Non-destructive analytical techniques of archaeological and historical artefacts have gained more attention in archaeometry research. Commonly methods, such as polarising microscope investigations and analytical methods (XRF, INAA) could not solve the lapis lazuli provenance problem since they are more or less destructive. Although XRF can be applied also non-destructively, it is usually restricted to the study of smooth surfaces of restored archaeological objects. However, the non-destructive bulk neutron-based analytical techniques, such as Prompt Gamma Activation Analyses (PGAA), and TOF-Neutron Diffraction supply further potentials for provenance studies. Knowledge of the elemental composition, including major and trace elements, as well as exploring the mineral composition may provide clues concerning the provenance and raw materials. In this project we have systematically investigated lapis lazuli samples from the largest and - from archaeological point of view - the most relevant quarries. Rock samples from Afghanistan, from Lake Baikal, from Pamir Mountains, from Chile and from Ural Mountains have been collected and investigated by instruments of the 10 MW Budapest Research Reactor and the dedicated external milli-beam PIXE (Particle Induced X-ray Emission) spectrometer of Wigner RC. Due to the high penetrability of the neutron, both PGAA and TOF-ND will give the average composition of the bulk material, i.e. of the object as a whole. The unsurpassable advantages of PGAA, TOF-ND and PIXE measurements that they do not require sample preparation; the artefacts can be positioned directly in the neutron beam; they are absolutely non-destructive, Based on some characteristic chemical elements (i.e. S, Cl, Ca, Fe, Si) we may succeed to distinguish between the most relevant quarries and to determine the provenance of some archaeological objects from the near East. Unfortunately, groups of different provenance defined by characteristic elements, overlap on the discrimination diagram, especially for specimens originated from Afghanistan and from the Baikal Lake. In order to clarify this doubtfulness, TOF-ND was recently tested for non-destructively identification of mineral composition characteristic for the provenance. The diffractograms show the structural changes in the lazurite crystals and the mineral composition of the lapis that could be typical for provenance. Also PIXE analyses have been carried out, and information were obtained on the lateral inhomogenity of the elemental composition on millimeter scale.

The experiments at the Budapest Neutron Centre have been supported by NMI3 and CHARISMA projects of the EC.

Primary authors : Dr. ZOELDFOELDI, Judit (MPA, Uni Stuttgart)

Co-authors : Dr. KASZTOVSZKY, Zsolt (Nuclear Analysis and Radiography Department, MTA Centre for Energy Research) ; Dr. KÁLI, György (Research Institute for Solid State Physics and Optics) ; Dr. SZÕKEFALVI-NAGY, Zoltán (Nuclear Analysis Group, Department of Biophysics,Hungarian Academy of Science) ; Dr. KOVÁCS, Imre (Nuclear Analysis Group, Department of Biophysics, Hungarian Academy of Science) ; Dr. NÖLLER, Renate (Bundesanstalt für Materialforschung und -prüfung)

The evolution of mineralogical phases during firing of ancient Greek ceramic pots

The preset work is part of a systematic study, using polychromatic and monochromatic neutrons, to characterize pottery, from the ancient Greek provinces of Phillipi, Topiros and Abdera. The aim of this project is to supply data on the constituent minerals, firing and weathering conditions, to both archaeologist and scientists in the field. The experiment was performed in two steps. First, a single piece of very low fired archaic pottery was fired in a furnace. In-situ TOF -ND patterns were collected from room temperature to 950 ˚C in steps of 25 ˚C. During the second stage seven ceramic pieces of approximate dimensions 3 - 15 cm2 and 0.7 – 1.5 cm thick, were illuminated with a wide monochromatic beam for several hours each at RT and the patterns were collected.The initial TOF experiments were carried out at ISIS, followed by sets of measurements with monochromatic neutrons using the E6 and E2 instruments at the BER II reactor site in Berlin, Germany.

Diffraction pattern Rietveld type analysis showed that the dominant phases are quartz (54%) and feldspars (20%). Diopside, orthoclase, calcite and iron oxide phases were also identifiable. The diopside content is found to decrease with increasing quartz - feldspar compositions. Furthermore the plagioclase content is increased almost linearly with the temperature. The hematite content in the sample remains almost constant above 700 ˚C and is in excess of 7%. Based upon the data collected in the second step, the firing temperatures of the pottery samples were determined to be within 850 ˚C to 950 ˚C.

Primary authors : Dr. SIOURIS, Ioannis (Democritus University Thrace)

Co-authors : Dr. KATSAVOUNIS, Stefanos (DEMOCRITOS UNIV. THRACE) ; Ms. CHARITOU, Soultana (DEMOCRITUS UNIVERSITY OF THRACE) ; Dr. HOSER, Andreas (Helmholtz-Zentrum Berlin für Materialien und Energie GmbH) ; Dr. KOCKELMANN, Winfried (Rutherford Appleton Laboratory, Chilton, Didcot, Oxfordshire OX11 0QX, UK.)

Presenter : Dr. SIOURIS, Ioannis (Democritus University Thrace)

Detection of Cu-Sn intermetallic compounds on tinned archaeological bronzes using diffraction methods: an evaluation of XRD and TOF-ND methods

This paper reports results of our systematic investigation of detection of tinning on low tin copper alloy using diffraction methods. Many ancient bronzes are likely to have been tinned in antiquity for their protection or aesthetics. Corrosion of low tin bronze can result in grey, tin-rich surfaces that are deceptively similar to tinning, which makes tinning difficult to identify on archaeological bronzes. During tinning, silver-colour Cu-Sn intermetallic compounds of characteristic composition are formed; their presence on low tin bronzes is evidence of tinning. Current methodology relies strongly on examination of cross-sectioned sampled using scanning electron microscopy and composition analyses. Corrosion can change the characteristic composition of intermetallics; taking a representative sample from an archaeological object is equally challenging. XRD and time-of-flight neutron diffraction offer the opportunity for non-destructive identification of tinned archaeological bronzes. To further understanding of how best to identify tinning on corroded bronzes using diffraction methods, model tinned copper analogues were prepared to establish limitations of detection of Cu-Sn intermetallics. Results showed that XRD as a surface technique has an advantage over TOF-ND. Rietveld analysis of the TOF-ND data showed a great sensitivity of TOF-ND to detect the δ-phase and ε-Cu_3Sn depending on the thickness of the metal coating and its volume fraction but was unsuccessful in detecting η-Cu_6Sn_5. Considering these limitations, neutron diffraction can be a useful non-destructive diffraction method for analysis of archaeological tinned bronzes.

Primary authors : Dr. MANTI, Panagiota (Cardiff University, Department of Archaeology and Conservation)

Co-authors : Dr. KOCKELMANN, Winfried (STFC Rutherford Appleton Laboratory)

Presenter : Dr. MANTI, Panagiota (Cardiff University, Department of Archaeology and Conservation)

The evolution of mineralogical phases during firing of ancient Greek ceramic pots

The preset work is part of a systematic study, using polychromatic and monochromatic neutrons, to characterize pottery, from the ancient Greek provinces of Phillipi, Topiros and Abdera. The aim of this project is to supply data on the constituent minerals, firing and weathering conditions, to both archaeologist and scientists in the field. The experiment was performed in two steps. First, a single piece of very low fired archaic pottery was fired in a furnace. In-situ TOF -ND patterns were collected from room temperature to 950 ˚C in steps of 25 ˚C. During the second stage seven ceramic pieces of approximate dimensions 3 - 15 cm2 and 0.7 – 1.5 cm thick, were illuminated with a wide monochromatic beam for several hours each at RT and the patterns were collected.The initial TOF experiments were carried out at ISIS, followed by sets of measurements with monochromatic neutrons using the E6 and E2 instruments at the BER II reactor site in Berlin, Germany.

Diffraction pattern Rietveld type analysis showed that the dominant phases are quartz (54%) and feldspars (20%). Diopside, orthoclase, calcite and iron oxide phases were also identifiable. The diopside content is found to decrease with increasing quartz - feldspar compositions. Furthermore the plagioclase content is increased almost linearly with the temperature. The hematite content in the sample remains almost constant above 700 ˚C and is in excess of 7%. Based upon the data collected in the second step, the firing temperatures of the pottery samples were determined to be within 850 ˚C to 950 ˚C.

Primary authors : Dr. SIOURIS, Ioannis (Democritus University Thrace)

Co-authors : Dr. KATSAVOUNIS, Stefanos (DEMOCRITOS UNIV. THRACE) ; Ms. CHARITOU, Soultana (DEMOCRITUS UNIVERSITY OF THRACE) ; Dr. HOSER, Andreas (Helmholtz-Zentrum Berlin für Materialien und Energie GmbH) ; Dr. KOCKELMANN, Winfried (Rutherford Appleton Laboratory, Chilton, Didcot, Oxfordshire OX11 0QX, UK.)

Presenter : Dr. SIOURIS, Ioannis (Democritus University Thrace)

Neutron imaging studies within the neu_ART Cultural Heritage project

Content :

The potential of imaging by thermal and fast neutrons has been assessed in the Cultural Heritage field, given the fact that this technique is non invasive and neutrons can penetrate many layers of thick objects. This method provides valuable information to assist preservation and restoration activities and to help dating or attributing through the understanding of the manufacturing techniques.

An investigation of the imaging capabilities of the Italian Neutron Experimental Station (INES at the ISIS pulsed neutron source of RAL laboratories, UK) and the Neutron Computerized Tomography and Radiography laboratory (NECTAR at the FRMII reactor in Munich) applied to artwork objects made of metal alloys was carried out. For this purpose custom samples have been prepared, following size and composition of typical ancient works of art. Through neutron tomography the internal structure of the testing metal objects was reconstructed and millimetric scale details inside them were resolved.

Moreover several measurements have been performed to fully characterize the performance of both apparatus. Their imaging response has been analyzed in terms of linearity, signal to noise ratio, dynamic range and resolution by the usage of scintillator screens of different chemical composition and thickness.

A detailed description of these studies will be presented.

Primary authors : Mrs. MILA, Giorgia (Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mrs. DURISI, Elisabetta (Dipartimento di Fisica, Università di Torino, Italy & Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mr. VISCA, Lorenzo (Dipartimento di Fisica, Università di Torino, Italy & Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mr. SACCHI, Roberto (Dipartimento di Fisica, Università di Torino, Italy & Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mr. RE, Alessandro (Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mr. LO GIUDICE, Alessandro (Dipartimento di Fisica, Università di Torino, Italy & Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mr. STAIANO, Amedeo (Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mrs. ROMERO, Alessandra (Dipartimento di Fisica, Università di Torino, Italy & Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mrs. BRANCACCIO, Rossella (Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mr. DUGHERA, Giovanni (Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mr. FERRARESE, Walter (Dipartimento di Fisica, Università di Torino, Italy & Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mr. COTTO, Giorgio (Dipartimento di Fisica, Università di Torino, Italy & Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy) ; Mr. NERVO, Marco (Centro per la Conservazione ed il Restauro dei Beni Culturali "La Venaria Reale", Italy) ; Mr. DEMMELBAUER, Marco (Centro per la Conservazione ed il Restauro dei Beni Culturali "La Venaria Reale", Italy) ; Mrs. GIOVAGNOLI, Annamaria (Centro per la Conservazione ed il Restauro dei Beni Culturali "La Venaria Reale", Italy) ; Mr. BÜCHERL, Thomas (Institut für Radiochemie, TU-München, Garching, Germany) ; Mr. GRAZZI, Francesco (Consiglio Nazionale delle Ricerche, Istituto dei Sistemi Complessi, Sesto Fiorentino, Italy) ; Mrs. SCHERILLO, Antonella (Consiglio Nazionale delle Ricerche, Istituto dei Sistemi Complessi, Sesto Fiorentino, Italy & Science and Technology Facility Council, ISIS Neutron Source - Didcot, United

Mrs. PASTRONE, Nadia (Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy)

Co-authors :

Presenter : Mrs. MILA, Giorgia (Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Italy)

Neutron Imaging of Archaeological Waterlogged Wood

When waterlogged wood is found, the main challenge for the curator is to dry the wood without deteriorating the object. This is often done by replacing some of the water by polyethylene glycol (PEG), and then removing the remaining water by freeze--- drying, with the PEG serving as a consolidant. The present study is an attempt to locate the PEG by means of neutron imaging. Eighteen samples of waterlogged alder with a size of 2 x 1 x 6 cm treated in different ways have been studied. If the PEG cannot be distinguished from the wood, the relative amount of PEG soaked in each sample, depending on the duration of impregnation, and its distribution in the wood can be clearly determined. Furthermore, owing to the large difference in neutron attenuation between the wood and the cracks (air), neutron tomography can be used very effectively to study the distribution and amount of such defects after the conservation treatment.

Primary authors : Mr. DEMOULIN, Thibault (ETH Zurich)

Co-authors : Prof. GEBHARD, Rupert (Dr.) ; Dr. SCHILLINGER, Burkhard (FRM II)

Presenter : Mr. DEMOULIN, Thibault (ETH Zurich)

Investigation of the cultural heritage by neutron tomography at INR, RO

The neutron and gamma imaging facility placed at the tangential channel of the TRIGA-ACPR from INR was used to investigate Dacian treasure pieces of silver and ancient statues of clay from the Arges County Museum of History. This collaboration is done within a research contract with IAEA with title “The neutron and gamma imaging method combined with neutron-based analytical methods for cultural heritage research” that helps curators to reveal the internal structure and composition of the objects. This is a good beginning for the dissemination of the investigation methods based on neutrons for cultural heritage and beyond this area. It is presented the work done with imaging methods for revealing the morphology of the objects.

Primary authors : Dr. DINCA, Marin (Institute for Nuclear Research)

Co-authors : Dr. MANDESCU, Dragos (Arges County Museum)

Presenter : Dr. DINCA, Marin (Institute for Nuclear Research)

Lost Letters found. A Neutron tomography study of Medieval

The more modest funeral effigies in Medieval England of the period 1250- 1400 AD were made of incised tomb slabs with simple crosses. This type of monument evolved in slabs inlayed with Lombardic letters made of laiton (brass). Finally these letters where joined up in brass strips with the letters incised. Finally this process resulted in the monumental brass slab monuments of which many can still be found in the Medieval English Churches.

Only a few individual letters have survived and are mostly in museum collections. However, recently metal detector finds of Lombardic letters are regularly reported. One of these letters was found in the bank of a brooke in South Stoke, Oxfordshire. We have been able to trace this Letter L back to the incised slab of Vicar Robert de Esthall (died 1274), still present in chancel of the Medieval Church of North Stoke,

A second example is a small brass strip of lettering which was removed from the Church of Shottesbrooke. This strip dates to the period of around 1370. The strip was spotted in an antique shop and recognised to be from one of the minor brass funeral monuments of St John the Baptist Church in Shottesbrooke Park, Berkshire.

We report a neutron tomographic study, performed at ANTARES, on both objects in order to study the corrosion and homogeneity of the objects. The information on the depth of the patina, the mineralisation and corrosion of the objects provides essential information for the conservation of these objects.

Summary :

The information from neutron tomographies are use for the conservation of Earl Medieval Brass Letters and Brass plaques

Primary authors : Dr. VISSER, Dirk (Dept of Physics, Loughborough University)

Co-authors : Dr. MUEHLBAUER, Martin (FRM2,TUM, Garching) ; Ms. BADHAM, Sally (Monumental Brass Society)

Presenter : Dr. VISSER, Dirk (Dept of Physics, Loughborough University)

Compact Neutron Imaging System for the Investigation of Large and Dense Objects

The aim of our project - financed by BMBF and undertaken in cooperation by RWTH Aachen University, Forschungszentrum Jülich, and SIEMENS AG - is to study the feasibility of a compact neutron imaging system. The system will be based on a 14 MeV neutron source and an advanced detector system, with the first concept consisting of a commercial aSi-flat-panel-detector linked with an exclusive converter/ scintillator for fast neutrons (currently under development). The compact system could be used for the characterization of large and compact like archaeological or cultural artefacts. Subject matters for the investigation are especially the structure of objects capsuled in concrete, clay or lead matrices. Detailed simulations of the neutron and photon transport are part of the current development program. Experiments are scheduled to start in the middle of 2013. The actual status of the project as well as the application spectrum will be presented.

Primary authors : Dr. KETTLER, John (RWTH Aachen - Institut für Nuklearen Brennstoffkreislauf)

Co-authors : Dr. MAUERHOFER, Eric (Foschungszentrum Jülich) ; Mr. SCHUMANN, Manuel (Forschungszentrum Jülich) ; Mr. ENGELS, Ralf (Forschungszentrum Jülich) ; Dr. KEMMERLING, Günter (Forschungszentrum Jülich) ; Dr. SCHITTHELM, Oliver (Siemens) ; Mr. HAVENITH, Andreas (RWTH Aachen - Institut für Nuklearen Brennstoffkreislauf (INBK)) ; Dr. FURLETOV, Sergey (Forschungszentrum Jülich) ; Dr. FURLETOVA, Julia (Forschungszentrum Jülich) ; Mr. NEIKE, Daniel (RWTH Aachen - Institut für Nuklearen Brennstoffkreislauf) ; Dr. VASQUES, Richard (RWTH Aachen - MATHCCES) ; Prof. FRANK, Martin (RWTH - MATHCCES)

Presenter : Dr. KETTLER, John (RWTH Aachen - Institut für Nuklearen Brennstoffkreislauf)

The upgrade imaing facility conrad-2

The neutron imaging facility CONRAD at Helmholtz-Zentrum Berlin (HZB) was upgraded as a part of the “Upgrade program for the cold neutron instrumentation” at HZB. The flight path of the facility was increased up to 10 m providing a larger beam size (field of view) of 20x20 cm2 with higher neutron flux density due to the new neutron guides with super mirror coating.
The characteristics of the upgraded facility like neutron flux density, neutron spectral distribution and beam size at the sample position will be presented. The benefit for different experimental methods will be discussed and examples from the first experiments performed at the upgraded facility will be shown.

Primary authors : Dr. KARDJILOV, Nikolay (Helmholtz-Zentrum Berlin)

Co-authors : Dr. HILGER, Andrã© (HZB) ; Dr. MANKE, Ingo (HZB) ; Prof. BANHART, John (HZB)

Presenter : Dr. KARDJILOV, Nikolay (Helmholtz-Zentrum Berlin)

Simulation studies for the development and construction of a demonstration facility for radiography with fast neutrons

In cooperation between University, Research Center and Industry we develop a compact demonstration facility. A D-T neutron generator with energies of 14.1 MeV was selected as neutron source. Neutrons with high energy are able to penetrate large and dense objects, like concrete structures. Several simulation studies are in progress to identify critical aspects of neutron and photon transport. One of the main tasks is to investigate and simulate the effect of neutron and gamma self-shielding in the objects, which is a challenge for non-destructive structural analysis of large objects like archeological artifacts, i.e. sarcophagus or large metallic objects with cavities like bronze statues. Therefore the feasibility to distinguish between different material groups like heavy metals and organic substances has to be verified. Furthermore, detailed simulations of the neutron and photon transport in the system will be performed in order to determine the best approaches to minimize the noise and optimize the contrast of the radiographic pictures. The simulations will be performed using the simulation tools MCNP5, MCNPX and FLUKA.

Primary authors : Mr. NEIKE, Daniel (Institute for Nuclear Fuel Cycle, RWTH University, Germany)

Co-authors : Dr. KETTLER, John (Institute for nuclear Fuel Cycle, RWTH Aachen, Germany)

Presenter : Mr. NEIKE, Daniel (Institute for Nuclear Fuel Cycle, RWTH University, Germany)

Penetrating Corrosion on Ancient Coins using Neutron-CT at ANTARES

Large numbers of coins from the Greco-Roman period have been excavated, and continue to be excavated. They can, particularly in sealed contexts, provide crucial information about the dating of stratigraphic layers in-as-much as most coins can be dated by their legends and iconography to relatively narrow brackets of time. Unfortunately these coins frequently suffer from the build-up of corrosion products on their surface during hundreds of years of burial. Removal of this corrosion by chemical or mechanical means often produces disappointing results. As a complement to X-ray CT, experiments were conducted at the ANTARES in 2010 to explore the possibility of using high-spatial resolution Neutron Computed Tomography to “see through” these corrosion products non-destructively. On the small sample of coins imaged during the pilot project several positive observations are possible. First, neutron radiographs were not a reliable predictor of the contrast produced by Neutron CT. In cases where almost none of the legend could be read on the Neutron radiograph, significant improvements were seen on the examination of CT slices. Second, Neutron CT equalled or exceeded X-ray CT in allowing the identification of otherwise wholly occluded features. Finally, the markedly different mass attenuation coefficients of neutrons meant that conservators could examine the layers of corrosion product on the coins in cross-section, whereas with X-ray CT the energies required to achieve 40% penetration of the copper bulk of the coin meant that the less dense corrosion was invisible.

Primary authors : Dr. BEVAN, George (Queen's University)

Co-authors : Mr. GABOV, Alexander (Conservation of Sculptures, Monuments & Objects) ; Dr. SCHILLINGER, Burkhard (FRM II)

Presenter : Dr. BEVAN, George (Queen's University)

An imaging technique to enhance cadmium in oil paints as a demonstration of element selective neutron imaging

Neutron radiography has been utilized for a non-destructive imaging taking advantage of high transmission properties of neutrons. New imaging techniques utilizing the neutron energy (wavelength) dependence of the transmission rate have attracted attention over the years. Such two-dimensional information of a specimen as lattice, elements, temperature and magnetic-field are obtainable by analyzing the changes of transmission rate with neutron energy. In this presentation, recent activities of the element selective imaging and the dedicated imaging beam line in the Japan Proton Accelerator Complex (J-PARC) are reported.

Experimental studies of the element selective imaging in J-PARC have been performed in the beam line NOBORU which is often used to develop new techniques. The element enhanced imaging were demonstrated by using metal foils of cobalt, silver, cadmium, indium, tantalum and gold, sodium in glass, copper and zinc in coins and iridium in catalyst of a satellite. Most recently, a study of the element enhanced neutron imaging have performed to visualize cadmium in oil paints. Neutron transmission rate of cadmium exhibits local minimum at 0.2 eV which is specific for cadmium. It was estimated that the neutron transmission rate locally decreased by 20% (it was expected to give clear image of cadmium) at 0.2 eV assuming 5 mg/cm2 of cadmium was included in an oil paint. For example the cadmium thickness of 5 mg/cm2 is equivalent to the oil paint thickness of 0.2 mm if cadmium content is 3wt% and the oil paint density is 1.0 g/cm3. A preliminary measurement was performed by using oil paints labeled cadmium-yellow, vanadium yellow, cadmium orange, cadmium red and two kinds of cadmium green. Each oil paint was put on a 15 x 15 mm aluminum plate of 1 mm in thickness, and was kept at 80 degrees C for about 100 hours. The dried weights were between 58 mg and 93 mg. Shadows caused by cadmium were recognized in the neutron transmission image of 0.2 eV at the positions of oil paints except the vanadium yellow and one of the cadmium green. The cadmium contents in these two oil paints were indicated to be much less than those in the others. Quantitative analysis will be performed in the next step. The authors wish this technique is helpful in some research fields.

A dedicated neutron imaging beam line is in construction now, and will start user programs in 2015. Even before that, proposals of the neutron energy selective imaging and conventional radiography are welcomed for NOBORU beam line in J-PARC.

Primary authors : Dr. KAI, Tetsuya (Japan Atomic Energy Agency)

Co-authors : Prof. KIYANAGI, Yoshiaki (Hokkaido University) ; Dr. FURUTA, Etsuko (Ochanomizu University)

Presenter : Dr. KAI, Tetsuya (Japan Atomic Energy Agency)

Della Robbia Sculptures in Portugal: neutron techniques applied to provenance issues

An interdisciplinary study is running in glazed terracotta sculptures ascribed to the Della Robbia workshop and nowadays exhibited at the most significant museums of Lisbon, like the National Museum of Ancient Art, the Calouste Gulbenkian Museum, the National Tile Museum and The Jerónimos Monastery of Belém.

The artworks are mainly glazed terracotta medallions with floral/fruit motifs on the edges and diverse types of centerpieces and also glazed terracotta statues.

Complementing the art historian's research, neutron techniques were used to characterize the ceramic body of several sculptures engaged by Della Robbia and eventually other rival workshops in the late 15th and through the 16th century. The main objective is to better understand the questions and problems deriving from the history, iconography and the artistic work of the main sculptors, particularly the process of their manufacture and assemblage, contributing to a better identification of the history and techniques used by the renowned Della Robbia family.

Micro-invasive sampling in hidden parts of the objects was performed so as not compromise the integrity of the piece. Chemical patterns were obtained by instrumental neutron activation analysis using the Portuguese Research Reactor (Sacavém, Portugal) as neutron source. The chemical analysis was complemented by X-ray diffraction.

Geochemical patterns, especially trace elements, emphasized subtle compositional features within sculptures that together with iconographic art historian studies contribute to Della Robbia workshop characterization. Quartz dominates in general and is associated with gehlenite, diopside, calcite, K feldspars. Plagioclase and hematite may occur in trace amounts. The mineral phases point to firing temperatures within a range of 800ºC-900ºC and to carbonate rich raw materials.

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Primary authors : Prof. DIAS, M. Isabel (CAMPUS TECNOLOGICO E NUCLEAR. IST, Univ. Técnica de Lisboa) ; Prof. PRUDÊNCIO, Maria Isabel (Campus Tecnológico e Nuclear, Instituto Superior Técnico, Universidade Técnica de Lisboa)

Co-authors :

Presenter : Prof. DIAS, M. Isabel (CAMPUS TECNOLOGICO E NUCLEAR. IST, Univ. Técnica de Lisboa) ; Prof. PRUDÊNCI

Modifying PGAA for the measurement of bronze samples

Bronze and other copper-based alloys have been used in the human history for a few thousand years already as construction material in arts and technology. Usually Sn and/or Zn can be found as main alloying elements together with some minor components as Al, P, Mn, Fe, As and Pb. Prompt gamma activation analysis is a multi-elemental and non-destructive nuclear analytical technique used for determination of major, minor and some trace elements in the whole volume of valuable objects. The detection limits for the minor elements strongly depend on the matrix of the object material. Since copper represents a difficult matrix for PGAA analysis, the method is not as popular for the determination of composition of various copper alloys.

Irradiating already a few grams of copper alloys with neutrons leads to the emission of a broad-energy-range gamma-rays (from 90 keV up to 8 000 keV) and so creating high count-rates, high background signal and long dead times in the detecting system during the PGAA measurement. The main gamma emission from copper is in the low energy range up to about 650 keV. However, also at energies above 7000 keV characteristic gamma rays are emitted. Tin has its characteristic gamma rays at energies between 1100 keV and 2300 keV. Various minor elements that may be found in bronze alloys as aluminum, manganese, iron, nickel, zinc, and lead have also characteristic gamma rays in the same range and above 7000 keV. Thus, if attenuating strongly energies below 1000keV, we can avoid saturating the detector with unnecessary signal.

The standard PGAA instrument was modified with the introduction of a lead attenuator in front of the gamma-ray detector and so reducing the intensity of gamma rays with lower energy. Having the advantage of a high-intensity cold neutron beam at the PGAA facility at MLZ in Garching, we can irradiate the objects with high neutron flux and detecting only the high energy gamma-rays. With decreasing the count rate and the dead time through the lead attenuator we obtain reasonable values for detailed sample analysis.

Based on a recent study while using a 10 mm thick lead attenuator for the PGAA measurement of samples with high boron concentrations, this method is tested for the analysis of bronze samples. First results will be presented during this presentation.

Primary authors : SOELLRADL, Stefan ()

Co-authors : RÉVAY, Zsolt () ; KUDEJOVA, Petra () ; Prof. TÜRLER, Andreas (Paul Scherrer Institute, Laboratory for Radiochemistry and Environmental Chemistry)

Presenter : SOELLRADL, Stefan ()

Advances in high resolution neutron computed tomography:

Adapted to the Earth materials

The application of state-of-the-art detector systems at the Advanced Neutron Tomography And Radiography Experimental System (ANTARES) has led to significant improvements in spatial resolution and contrast for geomaterial imaging. Resolutions of approximately 16 μm → 100 μm are now possible with fields of view of 33 mm → 205 mm, a level which is now comparable with X-Ray Computed Tomography (XCT) for which a micro XCT at the institute for mineralogy, crystallography and material science at the University Leipzig was used. Fine pixel resolution comes at the cost of image quality and increased exposure time, so that the optimum configuration for each sample must be determined on a case-by-case basis. Our interdisciplinary approach has yielded an efficient system of data acquisition, processing and quantification that is well suited for geomaterial imaging. It is now expected to find application in a much wider spectrum of geomaterial research, including: the formation of natural glasses, the characterisation of limited / precious samples such as scientific drill cores, and biomaterials (e.g. tooth) studies.

Primary authors : Dr. HESS, Kai-Uwe (Ludwig-Maximilians-Universität)

Co-authors : SCHILLINGER, Burkhard () ; Dr. FRANZ, Alexandra (3Institut für Mineralogie, Kristallographie und Materialwissenschaften, Universität Leipzig) ; Prof. DINGWELL, Donald B. (1Geo- und Umweltwissenschaften, Ludwig-Maximilians-Universität München)

Presenter : Dr. HESS, Kai-Uwe (Ludwig-Maximilians-Universität)