

# BOOK OF ABSTRACTS

# NINMACH

BUDAPEST 2017

2<sup>ND</sup> CONFERENCE ON

NEUTRON IMAGING AND NEUTRON METHODS IN ARCHAEOLOGY AND CULTURAL HERITAGE





2<sup>nd</sup> Conference on Neutron Imaging and Neutron Methods in Archaeology and Cultural Heritage

Budapest, Hungary

 $11^{\rm th}-13^{\rm th}$  October, 2017

Hungarian Academy of Sciences

# BOOK OF ABSTRACTS

NINMACH 2017 is organised by the Budapest Neutron Centre, a consortium of Centre for Energy Research and Wigner Research Centre for Physics of the Hungarian Academy of Sciences.

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### Preface

It is my privilege to welcome you as a participant of the second "International Conference on Neutron Imaging and Neutron Methods in Archaeology and Cultural Heritage Research" (NINMACH) 2017 conference, which takes place in the landmark building of the Hungarian Academy of Sciences, right next to the famous UNESCO world heritage river banks in Budapest, Hungary. The event is organized by the Budapest Neutron Centre, in collaboration with the International Atomic Energy Agency.

The mission of the conference is to create a stimulating environment to exchange ideas, to make a bridge between neutron scientists and cultural heritage experts and to compile the knowledge piece by piece, as it is symbolized by our conference logo, a fragmented pottery. Since the first NINMACH took place in Garching, Germany in 2013, this interdisciplinary field became even more mature, substantial experimental experience was accumulated, and an advanced community of specialists was formed.

The growing recognition of this topic is evidenced by the fact that it was declared as one of the Grand Societal Challenges (Reflecting societies) of the European Commission's H2020 framework programme. The success of the user access programs at the large neutron centres, as well as projects like ANCIENT CHARM, and more recently CHARISMA and IPERION CH, resulted in a sustained community that will likely evolve to a network of distributed research infrastructure, E-RIHS in Europe, and with regional partnerships, even worldwide. The Budapest Neutron Centre, with its 15+ years of experience, is committed to be a key participant of this initiative.

On behalf of the local organizing committee, the international advisory committee and our generous sponsors I wish you a very successful meeting, with the hope that you will take away good memories and new inspirations for the future.

Larl' Sendiniklos

László Szentmiklósi Conference chair

### Conference overview

Venue of the presentations: Hungarian Academy of Sciences, Central Building (1051 Budapest, Széchenyi István tér 9.), "Nagyterem" Lecture Hall

Wednesday, 11<sup>th</sup> October

12:00-13:30	Registration
13:30-13:45	Opening Ceremony
13:45-14:30	Keynote lecture 1.
14:30-15:50	Session I – Imaging 1.
15:50-16:10	Coffee break (Krúdy-terem)
16:10-18:00	Session II – Multi-technique approach
	and complementary methods 1.
18:00-21:00	Poster session & Welcome cocktail hosted by Mirrotron (Krúdy-
	terem)

Thursday, 12<sup>th</sup> October

09:00-09:45	Keynote lecture 2.
09:45 - 10:55	Session III – Imaging 2.
10:55-11:15	Coffee break (Krúdy-terem)
11:15-13:05	Session IV – PGAA & NAA
13:05-14:30	Lunch break (Krúdy-terem)
14:30-16:00	Session V – Imaging 3.
16:00-16:20	Coffee break (Krúdy-terem)
16:20-17:40	Session VI – Facilities
17:40-18:20	Workshop (Volume Graphics)
20:00-22:30	Conference dinner (Márványmenyasszony Restaurant)

Friday, 13<sup>th</sup> October

09:00-09:45	Keynote lecture 3.
09:45-10:55	Session VII – Neutron Scattering
10:55-11:15	Coffee break (Krúdy-terem)
11:15-12:35	Session VIII – Multi-technique approach
	and complementary methods 2.
12:35-13:20	Keynote lecture 4.
13:20-13:30	Closing ceremony
13:30-14:30	Lunch break
14:30-18:00	Optional visit to the Budapest Neutron Centre or to the Hungarian
	National Museum

### Conference programme

Wednesday,  $11^{\text{th}}$  October

12:00-13:30		Registration
13:30-13:45		Opening Ceremony
13:45-14:30	K-01	Using neutron imaging data for deeper understanding of cultural heritage objects – experiences from 15 years of collaborations <i>Eberhard Lehmann</i> (PSI)
14:30-15:50		Session I – Imaging 1. Chair: Burkhard Schillinger
14:30-14:50	O-01	A pulsed neutron transmission method for metal cultural heritage research Yoshiaki Kiyanagi (Nagoya University)
14:50-15:10	O-02	Neutron imaging study of 'pattern-welded' swords from the Viking Age Anna Fedrigo (STFC, ISIS Neutron Source)
15:10-15:30	O-03	Study of ancient metallic artefacts by using neutron imaging techniques, Ramanspectroscopy and SEM-EDS <i>Myriam Krieg</i> (Site et Musée Romains Avenches SMRA)
15:30-15:50	O-04	The neutron imaging study of cultural heritage items from Tver treasure Kuanysh Nazarov (Joint Institute for Nuclear Research)
15:50-16:10		Coffee break
16:10-18:00		Session II – Multi-technique approach and complementary methods 1. Chair: Thilo Rehren
16:10-16:40	I-01	Stone artefacts and Neutrons - Case studies from Hungary Katalin T. Biró (Hungarian National Museum)
16:40-17:00	O-05	Non-destructive determination of the manufacturing methods of ancient Indian blades and modern replicas through advanced applications of neutron tomography and neutron diffraction <i>Francesco Grazzi</i> (Consiglio Nazionale Ricerche)
17:00-17:20	O-06	Pre-historic funerary votive assemblages – stone vases provenancing using nondestructive neutron techniques <i>M. Isabel Prudêncio</i> (Centro de Ciências e Tecnologias Nucleares, Campus Tecnológico e Nuclear, Instituto Superior Técnico)
17:20-17:40	O-07	What is inside in the bronze weapons and tools? Archaeometallurgical Characterization of Late Bronze Age Metal Artefacts by Neutron Methods <i>Gábor János Tarbay</i> (Hungarian National Museum, Archaeological Department, Prehistoric Collection)
17:40-18:00	O-08	A multi-technique investigation of the incuse coinage of Magna Graecia Filomena Salvemini (ACNS-ANSTO, Sydney)
18:00-21:00		Poster session & Welcome cocktail hosted by Mirrotron

Thursday, 12<sup>th</sup> October

09:00-09:45	K-02	Science and Technology for Archaeology and Cultural Heritage <i>Thilo Rehren</i> (The Cyprus Institute)
09:45-10:55		Session III – Imaging 2.
		Chair: Eberhard Lehmann
09:45-10:15	I-02	Neutron imaging and PGAI methods applied to water absorption
		measurement on historic construction materials: first results and
		potentiality
		Francesca Sciarretta (Department of Design and Planning in Complex
		Environments, Università Iuav di Venezia)
10:15-10:35	O-09	Neutron tomography data fusion for visualization of South- and
		Southeast Asian bronzes
		Sara Creange (Rijksmuseum)
10:35-10:55	O-10	Recent uses of neutron imaging for the study of cultural heritage objects
		in Argentina
		Julio Marin (Centro Atómico Bariloche, CNEA, Av. Ezequiel Bustillo
		9500, Bariloche, Argentina)
10:55-11:15		Coffee break
11:15-13:05		Session IV – PGAA & NAA
		Chair: Heather Chen-Mayer
11:15-11:45	I-03	PGAI-NT, an integrated approach of element analysis and imaging, and
		its applications to cultural heritage samples
		$L\acute{a}szl\acute{o}$ Szentmiklósi (Centre for Energy Research, Hungarian Academy
		of Sciences)
11:45-12:05	O-11	Minimizing Sample Sizes while Achieving Accurate Elemental
		Concentrations in Neutron Activation Analysis of Precious Pottery
		Sheldon Landsberger (University of Texas)
12:05-12:25	O-12	Preliminary petrological and geochemical results of the possible source
		locations of HPmetaophiolitic polished stone artefacts
		Benjámin Váczi (ELTE Department of Petrology and Geochemistry)
12:25-12:45	O-13	Archaeometry at the PGAA facility of the MLZ
		Christian Stieghorst (Heinz Maier-Leibnitz Zentrum (MLZ), Technical
		University of Munich)
12:45-13:05	O-14	Quantitative chlorine analysis of archaeological iron objects by PGA
		Analysis
19.05 14.90		Britta Schmutzler (State Academy of Art and Design Stutttgart)
13:05-14:30		Lunch break
14.20 16.00		Continue V Incontinue III
14:30-16:00		Session V – Imaging III
14.20 15.00	1.04	Chair: Winfried Kockelmann
14:30-15:00	I-04	Neutron phase-contrast imaging for revealing armourers' marks
15.00 15.90	0.15	Nikolay Kardjilov (Helmholtz-Zentrum-Berlin)
15:00-15:20	O-15	Neutron imaging as tool for investigations on historical musical instruments

15:20-15:40	O-16	Eberhard Lehmann (Paul Scherrer Institut) Searching for "the needle in the haystack": reconstructing the brain and vascular system of the mammalian forerunners Burkhard Schillinger (Heinz Maier-Leibnitz Zentrum (FRM II))
15:40-16:00	O-17	Crystallographic characterization on different types of structure (Tsukurikomi) of Japanese swords using pulsed neutron imaging and diffraction methods <i>Kenichi Oikawa</i> (Japan Atomic Energy Agency)
16:00-16:20		Coffee break
16:20-17:40		Session VI – Facilities, techniques and data processing Chair: Nikolay Kardjilov
16:20-16:40	O-18	The neutron imaging beamline in Delft and future plans Lambert van Eijck (TU Delft Appl. Sci. dep. RadiationSciTech)
16:40-17:00	O-19	PGA-Imaging and Neutron Tomography at MLZ's PGAA-facility (FRM-II). A position-sensitive spectroscopy-visualization method with neutrons <i>Eschly Jan Kluge</i> (Institute of Nuclear Physics, University of Cologne) Christian Stieghorst (Maier-Leibnitz Center, Technical University Munich)
17:00-17:20	O-20	Neutron imaging options with fission and thermal neutrons at the NECTAR facility Malgorzata Makowska (Heinz Maier-Leibnitz Zentrum (MLZ), FRM II, Lichtenbergstr. 1 85748 Garching, Germany; Bavarian Research Institute of Experimental Geochemistry and Geophysics (BGI), University of Bayreuth, Germany)
17:20-17:40	O-21	Neutrons and complementary methods for Cultural Heritage research at the Budapest Neutron Centre <i>László Rosta</i> (Wigner Research Centre for Physics)
17:40-18:20		Workshop (Volume Graphics) Chair: László Szentmiklósi
20:00		Conference dinner (Márványmenyasszony Restaurant)

### Friday, 13<sup>th</sup> October

09:00-09:45	K-03	Neutron Imaging Methods for Cultural Heritage at the ANTARES
		facility
		Burkhard Schillinger (Heinz Maier-Leibnitz Zentrum (FRM II))
09:45-10:55		Session VII – Neutron Scattering
		Chair: Francesco Grazzi
09:45-10:15	I-05	The Metallurgical Texture of gold artefacts from the Bronze Age
		Rampart of Bernstorf (Bavaria) Studied by Neutron Diffraction
		Friedrich Wagner (Technical University of Munich)

10:15-10:35	O-22	IMAT: A new neutron imaging and diffraction beamline at ISIS Winfried Kockelmann (STFC-Rutherford Appleton Laboratory)
10:35-10:55	O-23	Neutron diffraction for cultural heritage studies: the Italian Neutron Experimental Station INES@ISIS Antonella Scherillo (STFC – ISIS, UK)
10:55-11:15		Coffee break
10.00 11.10		CONCO DICUN
11:15-13:05		Session VIII – Multi-technique approach and complementary methods 2.
44.45.44.05	0.04	Chair: Katalin T. Biró
11:15-11:35	O-24	Neutron and laboratory x-ray characterization of excavated Napoleonic artefacts from the Berezina Battlefield in Belarus
11 05 11 55	0.05	Jérôme Beaucour (Institut Laue Langevin)
11:35-11:55	O-25	Investigation of a Simulated Chinese Jade Dagger by Neutron
		Radiography and Prompt Gamma Activation Analysis Heather Chen-Mayer (NIST)
11:55-12:15	O-26	Investigating beads from Chalcolithic funerary cremation contexts of Perdigões, Portugal
		M. Isabel Dias (Centro de Ciências e Tecnologias Nucleares, Campus
10.15.10.05	0.07	Tecnológico e Nuclear, Instituto Superior Técnico)
12:15-12:35	O-27	Multi-technique archaeometallurgical investigations of metal objects from the Marche Region, Italy
		Massimo Rogante (Rogante Engineering Office)
12:35-13:20	K-04	Chemical imaging of paintings: X-rays and ions as complementary probes to neutrons
		•
		Thomas Calligaro (Centre de recherche et de restauration des musées de
10.00.10.00		France C2RMF)
13:20-13:30		Closing ceremony
13:30-14:30		Lunch break
14:30-18:00		Optional visit to the Budapest Neutron Centre or to the Hungarian
		National Museum

Poster presentation programme

Imaging:	$\operatorname{P-01}$ to $\operatorname{P-05}$
Multi-technique approach and complementary methods:	$\operatorname{P-06}$ to $\operatorname{P-09}$
PGAA – NAA:	$\operatorname{P-10}$ to $\operatorname{P-17}$
Facilities, techniques and data processing:	P-18 to P-19
Neutron Scattering:	$\operatorname{P-20}$ to $\operatorname{P-21}$

P-01 Nondestructive evaluation of the microstructure of iron parts of traditional Japanese sword manufacturing process by pulsed neutron transmission spectroscopy *Genki Hori* (Nagoya University)

- P-02 Detector Development for Mobile Fast-Neutron Radiography *Florian Reisenhofer* (RWTH Aachen University – Nukleare Entsorgung und Techniktransfer)
- P-03 Microstructural characterization of European historical swords through neutron imaging

Francesco Grazzi (Consiglio Nazionale Ricerche)

P-04 A Feasibility Study of Cultural Properties in Hidden Relics using Neutron and Xray Imaging Technique

Taejoo Kim (Korea Atomic Energy Research Institute)

P-05 Examining pottery forming techniques through combined petrographic analysis and neutron tomography

John Gait (Fitch Laboratory, British School at Athens)

- P-06 Preliminary result of investigation of the metal composition of coins from Phanagoria's treasure by method of neutron resonance capture analysis *Almat Yergashov* (Joint Institute for Nuclear Research)
- P-07 Production and use of bronze spirals in the 2nd Millennium BC Carpathian Basin *Géza Szabó* (Wosinsky Museum, Szekszárd)
- P-08 Neutron characterization of ancient and modern textiles Massimo Rogante (Rogante Engineering Office)
- P-09 PIXE investigation of ancient linen fabrics dated from Old Kingdom to Ptolemaic ages (2200-300 B.C.) Massimo Rogante (Rogante Engineering Office)
- P-10 Application of Prompt Gamma Activation Analysis to Provenance Study of the Korean Obsidian Artefacts

Yong-joo Jwa (Gyeongsang National University)

- P-11 PGAA as an analyzing tool for glass archaeological samples Bogdan Constantinescu (National Institute for Nuclear Physics and Engineering "Horia Hulubei", Bucharest)
- P-12 PGAA as an analyzing tool for obsidian archaeological samples Bogdan Constantinescu (National Institute for Nuclear Physics and Engineering "Horia Hulubei", Bucharest)

- P-13 Determination of the chlorine content of archaeological iron artefacts by prompt gamma activation analysis *Friedrich Wagner* (Physics Department, Technical University of Munich)
- P-14 Time and frequency domain analysis of pulsed cold neutron beam PGAA Danual Turkoglu (NIST)
- P-15 Lithic material of Tata-Porhanyó investigated by prompt-gamma activation analysis and petrographical methods *Viola T. Dobosi* (Hungarian National Museum)
- P-16 Erősd and related sites investigated by prompt-gamma activation analysis and petrographical methods

Sándor Sztáncsuj (Székely National Museum, Sfântu Gheorghe)

P-17 Monte Carlo Simulation of Compton Imaging Tomography for Prompt Gamma Activation Analysis

Benjamin Riley (University of Kentucky)

- P-18 Inverse Iteration Algorithm for Neutron Depth Profiling *Cong Shi* (Chengdu University of Technology)
- P-19 NeXT-Grenoble, a novel Neutron and X-ray Tomography characterisation facility Duncan Atkins (Institute Laue Langevin)
- P-20 Neutron texture analyses of Bronze Age swords from the Alpine region: benchmarking neutron diffraction against laboratory methods *Marianne Mödlinger* (Excellence-Cluster TOPOI, Deutsches Archäologisches Institut, Eurasien Abteilung, Berlin, Germany)
- P-21 Determination of firing temperature of clay pottery wares by Small Angle Neutron Scattering

Katalin Bajnok (Wigner research Centre for Physics, Hungarian Academy of Sciences)

## ORAL COMMUNICATIONS

### K-01

### Using neutron imaging data for deeper understanding of cultural heritage objects – experiences from 15 years of collaborations

<u>Eberhard Lehmann<sup>1,\*</sup></u>, Marie Wörle<sup>2</sup>, Eckhard Deschler-Erb<sup>3</sup>, Robert van Lang, Anne Pury-Gysel<sup>4</sup>, Michael Henss, Jan Hovind<sup>1</sup>, David Mannes<sup>1</sup>

\* eberhard.lehmann@psi.ch

Neutron imaging techniques can be used quite efficiently for non-invasive investigations in particular when other common methods like X-rays come to the physical limit. This is true with respect to transmission for many metals and with respect to sensitivity for small amounts of organic materials such as moisture, resins, lacquer and corrosion products.

Since the knowledge about the power of neutron imaging is not generally around in the community of museums experts, conservators and archaeologists, it has been necessary to convince them by successful studies of objects with relevance. We got the experience that scientists operating neutron imaging facilities (mainly physicists and engineers) and the community mentioned before have a quite different working style and approach in specific studies. Resulting publications may differ than in many respects when the main authors define the outcome.

Nevertheless, it was possible to perform many interesting studies in collaboration with museums people and archeologists, which give reason to go ahead for similar other such attempts in the future. In this framework, it was also important to get support from funding bodies like National Science Foundation or networks within the European Community (COST, Framework Programs), at least for travelling or even for paying manpower.

From the many different investigations we performed over the years, we want to highlight the following and will give feedback for the particular outcome of the studies:

- · The collection of Roman bronze sculptures found in parts of Switzerland
- $\cdot$   $\quad$  Bronze statues from the Renaissance period taken from the Rijksmuseum exhibition
- · Investigation of the inner content of Tibetan Buddha sculptures
- The structure and the manufacturing process of the Marc Aurelius bust from the Avanches region

<sup>&</sup>lt;sup>1</sup> Paul Scherrer Institut, Villingen, Switzerland

<sup>&</sup>lt;sup>2</sup> Swiss National Museum, Sammlungszentrum, Affoltern a. Albis, Switzerland

<sup>&</sup>lt;sup>3</sup> Archaeologisches Institut, Universität zu Köln, Germany

<sup>&</sup>lt;sup>4</sup> Musée romain d'Avenches, Switzerland

The objects are very different in composition, size and content why a specific strategy for the investigations had to be defined in advance to the investigations and for the final analysis. It was important from the beginning to accept that the neutron imaging data can only contribute to the description of the artifacts, their history, manufacturing process and conservation state, but are not a self-standing study alone. The whole pre-knowledge and the expertise of the involved museum experts were needed to complete the picture and the interpretation.

Such studies were always started with simple transmission imaging measurements from several directions to find out if a further tomography run seems to be reasonably – or not. The dimensions of the objects also define the specific beam and detector size, ranging at PSI from 40 cm down to only 3 cm in both directions across the beam. Accordingly, the spatial resolution in the studies can be tuned between 200 and 10 micro-meters.

The data and the derived conclusions are published and available in different format (papers, animated videos via YOUTUBE, books and catalogues).

### A pulsed neutron transmission method for metal cultural heritage research

### Yoshiaki Kiyanagi<sup>1,\*</sup>

<sup>1</sup> Nagoya University, Japan

\* kiyanagi@phi.phys.nagoya-u.ac.jp

The pulsed neutron transmission method is a unique method that can give crystallographic information non-invasively over a relatively wide area by analyzing the transmission spectrum of a sample reflecting the crystallographic characteristics [1]. The transmission spectrum includes information on crystal structure, lattice spacing, strain, preferred orientation, and crystallite size. By applying this method to a Japanese sword, we successfully obtained such information. Trend of spatial changes of crystallite size and texture suggested that they were affected by hammering. Furthermore, we recently found that the Vickers hardness is proportional to widening of the lattice spacing distribution in martensite phase produced by quenching [2]. This relation was applied to the cutting edge of a Japanese sword and obtained the Vickers hardness. The values were reasonable compared with the hardness obtained by a hardness meter for other swords. Sometimes we observed coarse grains in a metal and they gave single crystal-like dips in a transmission spectrum. Such coarse grain orientation will be determined by the dip analysis [3]. Coarse grains may appear also in a Japanese sword and they will be analysed by a similar method. The pulsed neutron method is one of powerful tool for the research of metal cultural heritages to elucidate crystallographic characteristics and furthermore to guess unclear making process. Here, we present established and recent developed features of the method and some examples applied to Japanese swords.

- H. Sato, et al., Materials Transactions, 56, pp.1147 (2015).
- [3] H. Sato, et al., submitted to J. App. Crystal.

O-01

Y Kiyanagi, H Sato, T Kamiyama and T Shinohara, J. Physics, Conference Series 340, 012010 (2012).

# Neutron imaging study of 'pattern-welded' swords from the Viking Age

<u>Anna Fedrigo<sup>1,\*</sup></u>, Alan Williams<sup>2</sup>, Peter Pentz<sup>3</sup>, Kim Lefmann<sup>4</sup>, Poul Erik Lindelof<sup>5</sup>, Burkhard Schillinger<sup>6</sup>, Francesco Grazzi<sup>7</sup>, Markus Strobl<sup>8</sup>

<sup>1</sup> Science and Technology Facilities Council, ISIS Neutron Source, Oxford, UK

<sup>2</sup> The Wallace Col lection, London, UK

<sup>3</sup> The National Museum of Denmark, Copenhagen, Denmark

<sup>4</sup> Nanoscience Centre, Niels Bohr Institute, University of Copenhagen, Denmark

<sup>5</sup> University of Copenhagen, Copenhagen, Denmark

<sup>6</sup> FRM-II, Heinz Maier-Leibnitz Zentrum (MLZ), Garching, Germany

<sup>7</sup> CNR, Istituto dei Sistemi Complessi, Sesto Fiorentino, Italy

<sup>8</sup> European Spallation Source ESS ERIC, Lund, Sweden

\* anna.fedrigo@fi.isc.cnr.it

Scientific investigations and archaeometric studies have played a major role in the field of archaeology, especially with regard to materials transformed through human activity. Until recently, metal artefacts have been mainly studied through standard analytical techniques like metallography and Scanning Electron Microscopy (SEM), which required the samples to be taken from the artefact and provided only punctual information. On the contrary, neutron techniques allow measuring bulk properties in a non-invasive way. Scandinavians from the Viking Age (800–1050 AD) were famous for being brave seafarers and explorers, and their weapons represented an indispensable tool in their travels. Sword from the Viking age often showed 'pattern-welding', made by welding together thin strips of iron and steel that were twisted and forged in various ways, producing a decorative pattern on the surface. Such a process introduces a differentiated distribution of the steel related phases (ferrite, cementite and slag inclusions) in the different parts of the blade. In this work we present a neutron imaging study of three sword blades from the Viking age belonging to the National Museum of Denmark. In particular, white beam and energy selective neutron tomography have been applied, in order to study the morphology of the blades, detect information on the forging techniques, and map the distribution of steel related phases in such composite 'pattern-welded' structure. The measurements have been carried out at the ANTARES beamline at FRM II (Garching, DE).

### Study of ancient metallic artefacts by using neutron imaging techniques, Raman-spectroscopy and SEM-EDS

Anika Duvauchelle<sup>1</sup>, <u>Myriam Krieg<sup>1,\*</sup></u>, Eberhard Lehmann<sup>2</sup>, David Mannes<sup>2</sup>, Laura Andrey<sup>1</sup>, Sandra Gillioz<sup>1</sup>

 $^{1}$ Site et Musée Romains Avenches SMRA

<sup>2</sup> Paul Scherrer Institute, Villingen, Switzerland

\* myriam.krieg@vd.ch

This presentation describes the investigation of two objects from a necropolis (Nécropole à la Montagne) in Avenches (VD, Switzerland): a bracelet and a sanitary tool made of copper alloy and presenting different questions. In both cases it was necessary to get sight of the interior of the objects. For that purpose, completely non-destructively methods were needed. Although methods with X-rays are very common and easily available, they often fail when larger amounts of metals are involved. In such cases, neutron imaging methods can be very useful since the penetration of neutrons through metals is much higher and clear information about the inner content and structure of inspected objects can be obtained. In the same way, the state of preservation of the metal can be studied very carefully due to the high sensitivity of neutrons for hydrogen, a component in the corrosion products. Therefore, both objects were analysed by neutron imaging and X-rays with the facility NEUTRA at the Swiss spallation neutron source of the Paul Scherrer Institut. Energy-dispersive X-ray spectroscopy (SEM-EDS) was done at the University of Fribourg, Department of Geosciences. In addition, a non-metallic part of one object was studied by Raman-spectroscopy at the School of Engineering and Architecture of Fribourg. Based on these analyses it becomes possible to describe these two objects in a broad manner and to understand its manufacturing method.



The bracelet (left) and the sanitary tool (right) from the necropolis "A la montagne" (images: Paul Scherrer Institut)

### O-04

# The neutron imaging study of cultural heritage items from Tver treasure

### Kuanysh Nazarov<sup>1,\*</sup>

 $^{\rm 1}$ Joint Institute for Nuclear Research, Dubna, Russia

 $^*$  quanysh@list.ru

One of the most important tasks of archaeology and other human sciences is comprehensive study of the cultural heritage items, which takes us into the deep past and allows understanding the formation and development of civilizations and ethnic groups. The special value and uniqueness of such items requires application of advanced nondestructive methods for their studies [1,2]. One of such methods is neutron radiography and tomography, which gives detailed information about the internal structure of the investigated objects. The difference in the total absorption cross sections of neutrons for different elements allows visualizing the distributions composition or structural heterogeneities in the studied materials, obtaining a three-dimensional model spatial resolution of a portion of a millimeter [3]. In this paper, the results of the studies of cultural heritage items found in Tver treasure are discussed [4]: fragment of two-leaved bracelet and the pendant. In order to obtain information about the internal structure of those cultural heritage items, neutron tomography experiments have been performed. These studies were prepared at the neutron radiography and tomography facility on beamline 14 of the IBR-2 the high-flux pulsed reactor [5]. According to the observed neutron tomography data, the 3-D analysis of radial pendant revealed that the thick ring with edges bent outside was used as a main fastening element; the studied fragment of the two-leaved bracelet was made in the technique and style of the first Kiev workshop which elevates the Tver treasure to the rank of other unique and composition rich treasures dating of the pre-Mongol period.

- Janssens K., Van Grieken R. Non-Destructive Micro Analysis of Cultural Heritage Materials. 1st ed. Imprint: Elsevier Sci., 2005, p. 828.
- [2] Anderson I.S., McGreevy R.L., Bilheux H.Z. (Eds.). Neutron Imaging and Applications: A Reference for the Imaging Community. Springer: New York, 2009, p. 341.
- [3] Middleton A., Tum J., Lang J. (Eds.). Radiography of Cultural Material. Routledge, 2 edition, 2005.
- [4] Khokhlov A.N., Kungurteeva S.A. Tver Treasure 2014 (pre-publication) (Ed. A.N. Khokhlov) Tver, Tver Region ond Neighbouring Territories in the Middle Ages. V.9. Tver, 2016, p.113-123.
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### Stone artefacts and Neutrons - Case studies from Hungary

<u>Katalin T. Biró</u><sup>1,\*</sup>, Zsolt Kasztovszky<sup>2</sup>

<sup>1</sup> Hungarian National Museum, Budapest, Hungary

<sup>2</sup> Centre for Energy Research, Hungarian Academy of Sciences, Budapest, Hungary

\* tbk@ace.hu

The application of large scale facility analytical techniques in archaeology, especially that of non-destructive methods has important new results in Hungary due to recent National Research Development and Innovation Projects. The objects investigated include various categories of cultural heritage including prehistoric finds (lithic artefacts, pottery, metal etc) and finds from more recent periods including glass as well. The present lecture will summarize new achievements on the field of lithic analysis using neutron techniques. The basic question for lithic analysis is provenancing, i.e., identifying the geological source of archaeological implements by the help of scientific techniques. For this purpose we need representative comparative material and wide geological, geochemical knowledge on the source areas. Some of the raw material categories are relatively easy to characterise and supply important and directly relevant data for archaeologists; some categories, however, are difficult to interpret and need more analytical work, sometimes complementary methodology. The selection of these techniques should consider the following aspects:

- they should be, preferentially, non-destructive
- the features observed (e.g. range of elements measured) should be complementary
- the categories (groups) distinguished should be evaluated using simple and clear statistical techniques

I-01

### Non-destructive determination of the manufacturing methods of ancient Indian blades and modern replicas through advanced applications of neutron tomography and neutron diffraction

<u>Francesco Grazzi<sup>1,\*</sup></u>, Burkhard Schillinger<sup>2</sup>, Alan Williams<sup>3</sup>, David Edge<sup>3</sup>, Francesco Cantini<sup>4</sup>, Davide Coco<sup>5</sup>, Filomena Salvemini<sup>6</sup>, Anders Kaestner<sup>7</sup>, Weimin M. Gan<sup>8</sup>, Antonella Scherillo<sup>9</sup>

<sup>1</sup> Consiglio Nazionale Ricerche, Sesto Fiorentino, Italy

- <sup>4</sup> Università di Firenze, Firenze, Italy
- <sup>5</sup> Università di Roma "La Sapienza", Roma, Italy
- <sup>6</sup> Australian Nuclear Science and Technology Organisation, Bragg Institute, Lucas Heights, Australia
- <sup>7</sup> Paul Scherrer Institut, Neutron Imaging and Activation Group, Villigen, Switzerland
- <sup>8</sup> German Engineering Materials Science Centre at MLZ, Helmholtz-Zentrum Geesthacht, Garching, Germany

\* francesco.grazzi@isc.cnr.it

The analysis of the micro-structural features of ancient Indian blades has been carried out by neutron tomography and neutron diffraction. The results provide a clear identification of the different types of steel used to produce such weapons. Among them, only a small proportion of the large number of swords produced in India is made of hypereutectoid textured steel, namely wootz steel also known as "Damascus steel". The others present characteristics very similar to the European swords produced in the same period including composite low and high carbon steel assembled together and the application of thermal treatments. The ancient swords and daggers, provided by the Wallace Collection in London and the Bernisches Historisches Museum in Bern, as well as the modern replicas made by a professional swordsmith, were analysed using neutron tomography both in white beam and energy selective configurations and neutron diffraction to get quantitative phase analysis and pole figure reconstruction of the texture in cementite phase. The results permitted to determine the spatial distribution of the iron and steel components inside the swords and the size and orientation of the microstructure of the ferrite and cementite grains in the wootz steel. These results are an important starting point to lead to the comprehension of the metal preparation and the forging procedure to produce swords made of wootz steel. This kind of results is a further proof of the validity of the use of neutron techniques for non-destructive and quantitative authentication and characterization of ancient metal artefacts.

### O-05

<sup>&</sup>lt;sup>2</sup> Heinz Maier-Leibnitz Zentrum (FRM II)

<sup>&</sup>lt;sup>3</sup> The Wallace Collection, London, UK

### O-06

### Pre-historic funerary votive assemblages – stone vases provenancing using non-destructive neutron techniques

### <u>M. Isabel Prudêncio<sup>1,\*</sup></u>, M. Isabel Dias<sup>1</sup>, Zsolt Kasztovszky<sup>2</sup>, Boglárka Maróti<sup>2</sup>, Ildikó Harsányi<sup>2</sup>, Imre Kovács<sup>3</sup>, Zoltán Szőkefalvi-Nagy<sup>3</sup>, A.C. Valera<sup>4</sup>

<sup>1</sup>Centro de Ciências e Tecnologias Nucleares, Campus Tecnológico e Nuclear, Instituto Superior Técnico, Lisbon

<sup>2</sup> Centre for Energy Research, Hungarian Academy of Sciences, Budapest, Hungary

<sup>3</sup> Wigner Research Centre for Physics, Hungarian Academy of Sciences, Budapest, Hungary.

<sup>4</sup> Era Arqueologia, Núcleo de Investigação Arqueológica – NIA. Cç. de Santa Catarina, 9C, 1495-705

Cruz Quebrada – Dafundo – Portugal. Interdisciplinary Center for Archaeology and Evolution of

Human Behavior (ICArHEB, Universidade do Algarve, Campo de Gambelas, Faro, Portugal

\* iprudenc@ctn.tecnico.ulisboa.pt

The Perdigões archaeological site is one of the largest known Portuguese Late Neolithic and Chalcolithic ditched enclosures (Neolithic and Chalcolithic) in southern Portugal. A variety of exogenous objects such as pottery, lithic artefacts, stone and bone and ivory idols, pectin shells, were found during excavations of several funerary contexts. In this work small stone vases from these funerary environments were studied by prompt-gamma activation analysis (PGAA), neutron radiography and external milli-beam particle induced X-ray emission spectroscopy (PIXE) at the Budapest Neutron Centre. In addition, several samples from carbonated rocks quarries of central and southern Portugal were analysed by using the same methods aiming the identification of the geographic region of the raw materials that were used to produce these objects thus contributing to the understanding of the interaction network in which Perdigões was involved [1]. The macroscopic observation of the vases showed a porous whitish stone. The outlier layers present a brownish colour, certainly the result of alteration during burial. Also in the interior of the vases brownish aggregations of soil particles and bones remains were found. The chemical composition obtained by PGAA enhances the calcite rich material constituent of the vases. PIXE analyses in different points (whitish, brownish and aggregates), showed an enrichment of Si, K, Ti, Fe and Zn in the outer layers; the brownish terrigenous material has higher content of P, certainly due to the bones contribution. In addition, iron enrichment was found in some points of these terrigenous materials. Neutron radiography allowed to accurately visualizing the vase morphology. The chemical composition obtained for the archaeological stone vases and local and regional raw materials point to the resource of Estremadura limestones (Central-western Portugal), pointing to long distance provenance of the vases production. It is important to enhance the significant results obtained for the archaeological discussion of Perdigões interaction network during Chalcolithic, by using non-destructive neutron techniques.

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### What is inside in the bronze weapons and tools? Archaeometallurgical Characterization of Late Bronze Age Metal Artefacts by Neutron Methods

### Gábor János Tarbay<sup>1,\*</sup>

 $^1$  Hungarian National Museum, Archaeological Department, Prehistoric Collection, Budapest, Hungary \* tarbayjgabor@gmail.com

The aim of the presentation is to show the results of a cooperative work between the Hungarian National Museum, Archaeological Department, Prehistoric Collection and the Hungarian Academy of Sciences, Centre for Energy Research, Nuclear Analysis and Radiography Department, which focused on the bronze alloy artefacts from the Late Bronze Age (LBA, ca. 1500-1000 BC) Carpathian Basin. In the presentation, three different minor and major projects will appear: 1. Spearheads, 2. Warrior Equipment, 3. Project Axe. Although, the samples of these projects are different, they are all linked by the same analytical methods (PGAA, neutron radiography and imaging) and the same research questions, such as how the objects were made, what kind of casting defects their bare.

Within project Spearheads, four spearhead with the remains of the prehistoric wooden shaft were investigated with the above non-destructive methods which allowed us to provide a completely new data on the LBA spear-use in the Carpathian Basin. Project Warrior Equipment focuses on an unparalleled assemblages of defensive and offensive weapons (e.g. pair of greaves, cap helmet) as well as rare artefacts (e.g. metal cup). By the aid of the applied analytical methods, the manufacturing of these objects as well as their prehistoric manipulation during deposition was evaluated in details. Project Axe is a brand new approach for the detailed characterization of casting defects of prehistoric cast artefacts. Non-destructive neutron based methods as well as destructive ones (metallography) and even experimental casting was involved in this project. As a results, detailed characterization and classification of visible and invisible defects in prehistoric bronze artefacts was possible, as well as the description of the main causes which allowed us to interpret the LBA casting defects from a different point of view than before.

### A multi-technique investigation of the incuse coinage of Magna Graecia

<u>Filomena Salvemini</u><sup>1,\*</sup>, Scott Olsen<sup>1</sup>, Vladimir Luzin<sup>1</sup>, Maxim Avdeev<sup>1</sup>, Joel Davis<sup>2</sup>, Kenneth Sheedy<sup>3</sup>

<sup>1</sup> ACNS-ANSTO, Sydney, Australia

<sup>2</sup> Nuclear Materials-ANSTO, Sydney

<sup>3</sup> Australian Centre for Ancient Numismatic Studies-Macquarie University, Sydney

\* filomena.salvemini@ansto.gov.au

This paper presents a synergic combination of different neutron techniques applied to characterize the manufacturing process of 'incuse' coins minted by Greek colonies in South Italy during the 6th and 5th centuries BC. After the invention of coinage in the Asia Minor kingdom of Lydia around 630-620 BC, the manufacture of coins spread all around the Mediterranean. The Greek colonies in Southern Italy developed their own method of minting, the so-called incuse coin technique, around the mid-6th century BC. The distinctive feature of this coin is that the image on one side is repeated on the other side in reverse (or intaglio) – and the alignment is always extremely close. While the production techniques for the coinage minted by the majority of Greek cities on the mainland and in Asia Minor are relatively well understood, the incuse method developed by cities in South Italy is still debated.

In order to shed new light on the processes and the advantages of the incuse manufacturing method, a non-invasive multi-technique approach based on neutron imaging and diffraction methods was applied to investigate a set of 30 silver coins produced in the communities of Metapontum, Kroton, Sirinos/Pyxoes, Sybaris, Caulonia and Taras in South Italy. All samples are part of the collection of the Australian Centre for Ancient Numismatic Studies (ACANS), Macquarie University in Sydney (AU).

Neutron tomography was exploited to virtually reconstruct the bulk of incuse coins in 3D. It is useful as a means to study aspects such as morphology, porosity, inclusions, the presence of composite structures (plating). Neutron diffraction and SEM-EDS analyses were applied to define their composition. Complementary neutron texture analysis was performed to determine the distinctive texture patterns, the crystallographic preferred orientations in the alignment of the atomic lattice of metal grains induced, by the manufacturing process. This archaeometric investigation is part of the Cultural Heritage project carried out at ANSTO. This scientific program aims to interface and synergize the suite of nuclear methods available across facilities to provide a non-invasive approach for the study and conservation of heritage materials. In close collaboration with the cultural heritage community, the research is focused on several areas ranging from the characterization of Aboriginal pigments, through the chronology, origin and conservation of Australian Rock Art, to the study of archaeometallurgy.

O-08

### Science and Technology for Archaeology and Cultural Heritage

### Thilo Rehren<sup>1,\*</sup>

<sup>1</sup> The Cyprus Institute

\* th.rehren@ucl.ac.uk

Advanced scientific methods play an ever-increasing role in the study of the human past. The topic of this conference is just one example of such highly-specialised fields, offering a uniquely sharp and powerful tool for a wide range of situations. While Archaeology has from the beginning evolved through merging different specialist disciplines, it has also from the beginning grappled with the problem how best to integrate these often seemingly incompatible, even incomprehensible approaches to achieve real advances for the field as a whole.

With this lecture I want to shine a light at the sometimes opposing forces within Archaeometry / Science-Based Archaeology / Science and Technology in Archaeology. On the one hand, there is the trend to develop ever-cleverer tools and methods, while on the other hand, routine application of what looks like pedestrian science is often all that can be done, or needs to be done. I will offer a personal view on how best to keep these two forces in a productive balance, even make them work together like two horses drawing one carriage.

This balance needs to be kept within an often turbulent academic and social environment which makes it difficult to reach anything resembling equilibrium, or a pleasant trot through that beautiful country called 'The Past'. Instead, it is a very real race in competition with many other academic fields vying for public attention (and money), and its playing field often resembles an obstacle course more than a country lane. In such an environment it is imperative that the two (metaphorical) horses of technical development and routine application work together as closely as they possibly can, while watching closely what the competition is up to. As in any good sport, competing for talent is an important part of the game, and there are opportunities here which Archaeology and Cultural Heritage can usefully exploit in order to attract the best people, even in the fields of science and technology.

K-02

### Neutron imaging and PGAI methods applied to water absorption measurement on historic construction materials: first results and potentiality

### Zoltán Kis<sup>1</sup>, <u>Francesca Sciarretta<sup>2,\*</sup></u>, László Szentmiklósi<sup>1</sup>

<sup>1</sup> Centre for Energy Research, Hungarian Academy of Sciences, Budapest, Hungary

<sup>2</sup> Department of Design and Planning in Complex Environments, Università Iuav di Venezia, Italy \* scifra@iuav.it

There is a strong demand for knowledge and diagnostics on architectural heritage buildings to be obtained mostly with non-destructive techniques, especially for large and complex assets. The motivation is to physically preserve materials and get an information picture as extensive as possible. From this viewpoint the modelling of a heritage building, be it numerical or physical or both, is recognized as a powerful tool to investigate and/or predict the real structure's mechanical response to service and ultimate state loads. Models implement mechanical properties, which may rely on empirical data yielded from the building itself or from laboratory measurements. We aimed at establishing a multidisciplinary procedure to characterize porous historic construction materials, whose properties (physical and mechanical, as well as chemical composition) can be affected by the presence and action of water content and salt ions. The whole procedure's objective is to relate qualitative to quantitative information, to get an evaluation of the selected material's mechanical properties. We developed a neutron-based investigation technique to assess the condition of historic buildings' construction materials. Neutron radiography and tomography, as well as prompt-gamma activation analysis and imaging were applied to various types of stone blocks (which can be characterized e.g. with different levels of liquid permeability in saturated conditions) to detect the uptake of water and salt ions in porous construction materials of cultural heritage significance. The results enlighten the accurate water intrusion patterns, the evaluation of the water content in unsaturated conditions, the movement of water and salt contents inside the stone samples. The established methodology may find its application niche in the non-destructive assessment of historic and contemporary building construction materials. The results of the neutron imaging (NR/NT) and elemental mapping obtained by position-sensitive neutron-based analytical techniques (PGAI, NR/NT-driven PGAI) could answer relevant scientific questions in porous cultural heritage construction materials. Among them there are the structural information for water uptake patterns from tomographic images, a time-dependence of water or salt ion solution flow in the medium, and the quantitative estimation of the spatial distribution of the water content in unsaturated conditions. The measurement and visualization of the spatial and temporal distribution is deemed useful to test and/or predict e.g. (1) the service time-dependent behaviour of a weather-exposed structure or cladding, (2) the performance of materials used for restoration, for instance local substitution of cladding, (3) possible alternatives for restoration works. The time- and spatially-resolved visualization would provide a clear and immediate comprehension of stone properties even to the non-specialist, e.g. clients, accomplished or general audience interested in cultural heritage preservation.

### Neutron tomography data fusion for visualization of South- and Southeast Asian bronzes

<u>Sara Creange</u><sup>1,\*</sup>, Francesco Grazz<sup>2</sup>, Nikolay Kardjilov<sup>3</sup>, Robert Erdmann<sup>4</sup>, Francesco Cantint<sup>5</sup>

<sup>1</sup> Rijksmuseum

<sup>2</sup> Consiglio Nazionale Ricerche, Sesto Fiorentino, Italy

<sup>3</sup> Helmholtz-Zentrum Berlin, Germany

<sup>4</sup> Rijksmuseum, University of Amsterdam, Radboud University Nijmegen

<sup>5</sup> Conservation of Cultural Heritage Program, University of Florence

\* s.creange@rijksmuseum.nl

A group of four South- and Southeast Asian bronze religious statues in the collection of the Rijksmuseum were subjected to white beam neutron tomography and energy selective scans at Helmholtz-Zentrum Berlin (2016) and are proposed for further tomography-guided neutron diffraction studies at ENGIN-X (projected 2018). Neutron techniques are well-suited for this study group, because of the ease of penetration of neutrons in large bronzes, and because the different attenuation coefficient of the various phases is advantageous for locating corrosion, core and restoration materials. The four bronzes under study embody a variety of questions related to conservation and technical art history. Neutron tomography was undertaken to investigate corrosion of iron armatures and bronze, crack systems, repairs and separately cast elements and casting methods including orientation and gating system. The tomographic reconstructions provided significant insights regarding the sculptures and their fabrication. Where questions remain, a number of other non-destructive (or micro-destructive) analytical techniques can be used to clarify and supplement the results. The complexity of the tomographic images and the related information from other analyses and imaging methods prompts reflection on the process of generating and presenting complex data, first within an interdisciplinary research team and subsequently to a broader public. Therefore preliminary results of the neutron investigations the four statues will be briefly presented here, along with a new method being developed at the Rijksmuseum for integrating neutron tomography images with other layers of information generated from a variety of sources including visual light photography, 3D scans, x-ray radiography and x-ray fluorescence. The aim is to create an intuitive, engaging, web-based interactive viewer for clear presentation of complex data sets related to each sculpture.

### Recent uses of neutron imaging for the study of cultural heritage objects in Argentina

### Julio Marin<sup>1,\*</sup>, Fernando Sánchez<sup>1,2</sup>

<sup>1</sup> Centro Atómico Bariloche, CNEA, Bariloche, Argentina

<sup>2</sup> Instituto Balseiro, Universidad Nacional de Cuyo-CNEA, Argentina

\* jhmarin@cab.cnea.gov.ar

The only neutron imaging facility in Argentina is placed at the RA6 research reactor which is located in the Bariloche Atomic Centre, Argentina. In 2010, the reactor's core was upgraded and its power increased to 1 MW, for that reason all its associated facilities were redesigned. The neutron beam of the neutron imaging facility uses a radial extraction tube and has a double cone collimator with L/D=100. The sample is placed in a shielded space of 40 cm x 40 cm x 40 cm. The back wall of this space is an aluminium sheet that supports a 20 cm x 20 cm 6LiF/ZnS:Ag scintillation screen. The spatial resolution of the facility is of about 200 microns.

After two years employed characterizing the beamline, tomographic capabilities were incorporated. 3D reconstructions using a locally developed inexpensive system were performed, being these the first of its kind done in Argentina (see figure included).

In this work we present our recent experience in the application of neutron radiography and neutron tomography to objects belonging to the cultural heritage. We have worked with objects from excavations on gardens and dumps of historical houses of the city of Buenos Aires provided by the Heritage and Historical Institute of the City of Buenos Aires. Another collaboration was established with Adam Hajduk, a local archaeologist. We studied a sceptre made of wood and silver. In addition, we have used the facility to study fossils provided by two local institutions: the Bariloche's Paleontological Association and the Lago Gutiérrez Museum. Finally, we analysed the homogeneity of a metal medallion by combining gamma spectroscopy with neutron imaging.

### PGAI-NT, an integrated approach of element analysis and imaging, and its applications to cultural heritage samples

László Szentmiklósi<sup>1,\*</sup>, Zoltán Kis<sup>1</sup>, Boglárka Maróti<sup>1</sup>, Tamás Belgya<sup>1</sup>, Zsolt Kasztovszky<sup>1</sup>

<sup>1</sup> Centre for Energy Research, Hungarian Academy of Sciences, Budapest, Hungary

\* szentmiklosi.laszlo@energia.mta.hu

Most widespread element analysis techniques are not suitable for bulky heterogeneous or structured objects. Prompt-gamma activation imaging (PGAI), in combination with neutron tomography (NT), has proven its potential to fill this niche, thanks to the nondestructive nature and the high penetration depth of neutrons and gammas. This is of utmost importance for valuable cultural heritage objects. Following the success of the demonstration measurements within the ANCIENT CHARM EU FP7 NEST project, NIPS-NORMA, the single operational facility for this purpose worldwide, was set up in 2012 at the Budapest Neutron Centre.

Neutron imaging and local chemical compositions from PGAI-NT technique can be combined even with high-definition X-ray imaging, or a laser-scanned point cloud, in order to achieve a comprehensive characterization of macroscale structure and the in-depth, but still localized elemental composition. Detailed computer simulations can be used to derive corrections for self-absorption and self-shielding. The joint interpretation of these so-far separately treated datasets offers significant benefits.

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### Minimizing Sample Sizes while Achieving Accurate Elemental Concentrations in Neutron Activation Analysis of Precious Potterv

Sheldon Landsberger<sup>1,\*</sup>, Joseph Yellin<sup>2</sup>

<sup>1</sup> University of Texas, USA

<sup>2</sup> The Hebrew University of Jerusalem, Israel

\* s.landsberger@mail.utexas.edu

Manufacturing containers, body adornments, paintings, sculptures, etc. are one of the major characteristics of human endeavor into art. Being able to achieve provenance of these objects gives modern humans the link to past lives of major cultural groups and civilizations. The field of archaeological forensics has been very active for the past century and a half. When analyzing pottery, ceramics, and other archaeological finds, neutron activation analysis (NAA) is one of the two preferred methods for elemental analysis due to the large number of elements that can be measured precisely and accurately using a small sample size. An excellent overview of nuclear techniques for cultural heritage research has been published by the International Atomic Energy Agency [1]. The current methodologies usually require about 100 - 400 mg of the specimen to acquire representative results. Typically these quantities for NAA are relatively large when dealing with precious pottery or sherds from broken pottery vessels, the most common artefacts found during excavation of archaeological sites. NAA can be done with much smaller samples but a sample too small may not reflect the bulk composition. Very recently the application of NAA to micro gram scale of solid samples has been explored for extraterrestrial samples [2]. To test this hypothesis in a pilot project 10 mg samples from Tunisian pottery were examined by NAA to ascertain if samples at such small quantities could give representative and meaningful results.

Typically sample sizes less than 100 mg are considered to be subject to inhomogeneity. We have demonstrated in this study that the combination of micro-sampling and various methods in NAA including is an ideal method of quantifying trace elements in pottery. Methodologies in thermal and epithermal NAA in conjunction with Compton suppression has resulted in a wide range of elements with sample weights of only 10 mg. These results will help develop new and improved methods of analysis of precious pottery and ceramics while preserving as much of the pottery pieces as possible.

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### Preliminary petrological and geochemical results of the possible source locations of HP-metaophiolitic polished stone artefacts

Benjámin Váczi<sup>1,\*</sup>, György Szakmány<sup>1</sup>, Zsolt Kasztovszky<sup>2</sup>, Elisabetta Starnini<sup>3</sup>

<sup>1</sup> Department of Petrology and Geochemistry, Eötvös Loránd University, Budapest, Hungary

In NW-Italy most of the Neolithic polished stone artefacts (axes, adzes, chisels) are manufactured from high pressure and low temperature metaophiolites – greenstones – but they widespread everywhere in Europe also [1, 2, 3]. In the last 10 years in Hungary 25 prehistoric polished stone tools were identified during the re-examination of the Museum historical collections [4]. The specialty of these artefacts is in their really uncommon raw material for East Europe until now. There are only few territories in whole Europe where we can find LT-HP metaophiolites in primary form: Monviso, Western Alps (near Torino)[5] and Syros and Tinos, Greece [6]. Na-pyroxenites, eclogites and their retrograde metamorphic derivates appear also in the Oligocene conglomerate on the northern side of Voltri [7] and in the recent alluvium of river Po, Curone and Staffora. The researchers agree in: the raw material of these artefacts originated from the W-Alps and the N-Apennines, but the precise source location is not specified so far. The aim of this research is to find significant difference between the potential source locations. We selected 3 possible source locations: the southern part of Monviso, the alluvium of Po, and the alluvium of Curone, to make a detailed petrographic and geochemical study to clarify the provenance of these special artefacts. During our work we analysed the samples by PGAA and SEM-EDX by electron-microscope to compare directly our results with the results of the earlier analysed polished stone tools

The main rock forming minerals of Na-pyroxenites and eclogites are Na-rich monoclinic pyroxenes (jadeite-omphacite), the eclogites also contain a great quantity of garnets. Destructive analytical methods couldn't be applied on archaeological specimens with high value, therefore to make the comparison relatively easy between the possible raw materials and the archeological samples we used non-destructive methods during this work. By the chemical composition of HP meta-ophiolites, D'Amico created 9 main groups: jadeitites, Fe-jadeitites, omphacitites, Fe-omphacitites, mixed jade, Fe-mixed jade, Mg-eclogite, intermediate eclogite and Fe-eclogite [3]. This type of classification simplifies the comparison and provides a basis for the further investigations. From the three possible source location the specimens can fit into 7 geochemical groups.

From Monviso south most of the samples are Fe-mixed jades, but jadeities, mixed-jades and an omphacitite can be found among the specimens too. Furthermore this is the only locality where we found Fe-jadeitite. Specimens originated from the alluvium of Po cover

<sup>&</sup>lt;sup>2</sup> Centre for Energy Research, Hungarian Academy of Sciences, Budapest, Hungary

<sup>&</sup>lt;sup>3</sup> School of Humanistic Sciences, Department of Historical Studies, University of Torino, Italy

<sup>\*</sup> vbeni9305@gmail.com

wide range of these chemical groups, there are two omphacitites and one sample from nearly every chemical groups. Some of the samples from the Curone sites fit into some of the eclogite chemical groups but most of the specimens cannot fit into any of these, because of the great amount of retromorphous minerals that slightly change the chemical composition of the rocks. The accessory minerals are the best tools to prove that there is significant difference between the different source locations, and that they can be used as markers to trace the provenance of prehistoric artefacts. Our preliminary results showed that the main Ti-, and Fe-bearing mineral is ilmenite in the Monviso area and rutile appears in minor quantity. The greenstones from Curone and Po river sediments contain rutile as a Ti-mineral and pyrite as Fe-mineral. There is remarkable difference between the localities about the REE-minerals. Monviso and Po alluvium samples contain often allanite, furthermore monacite and xenotime also appear. In Curone samples the REEminerals almost absent and the rocks from this site are characterized by strong retrograde alteration compared to samples from the W-Alpine localities.

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### Archaeometry at the PGAA facility of the MLZ

<u>Christian Stieghorst<sup>1,\*</sup></u>, Zsolt Révay<sup>1</sup>, Petra Kudějová<sup>1</sup>, Hans Peter Kuhnen<sup>2</sup>, Cornelis Willem Passchier<sup>3</sup>, Gül Sürmelihindi<sup>3</sup>, Rupert Gebhard<sup>4</sup>, Friedrich Wagner<sup>5</sup>, Britta Schmutzler<sup>6</sup>

<sup>1</sup> Heinz Maier-Leibnitz Zentrum (MLZ), Technical University of Munich, Germany

<sup>2</sup> Institut für Altertumswissenschaften, Johannes Gutenberg University Mainz, Germany

<sup>3</sup> Institute of Geosciences, Johannes Gutenberg University Mainz, Germany

<sup>4</sup> Archäologische Staatssammlung München, Germany

<sup>5</sup> Physics Department, Technical University of Munich, Germany

<sup>6</sup> Staatliche Akademie der Bildenden Künste Stuttgart, Germany

\* christian.stieghorst@frm2.tum.de

Instrumental neutron activation techniques such as prompt gamma activation analysis (PGAA) and instrumental neutron activation analysis (INAA) are well-suited for archaeometrical applications. A bundle of advantages make these methods attractive for archaeometry and cultural heritage, for instance low destructiveness, straightforward sample preparation and good detection limits for many elements. Panoramic bulk analysis of the elemental sample composition is ideal for provenance studies of various materials. Due to the fact that PGAA and INAA are complementary for several elements, a combination of both methods is used to further increase the number of the detectable elements. This can help to distinguish geologically similar deposits, which are very close to each other. At the PGAA facility of the Heinz Maier-Leibnitz Zentrum (MLZ) we have various archaeometry and cultural heritage projects.

1. Provenance studies of Gallo-Roman limestone objects of the Rhine-Main-Moselle area allow an insight into the ancient transport system, the history of quarrying and the reutilization of building material in the later times (so-called spoils).

2. The measurement of the chlorine content in archaeological iron objects is important for conservation-restoration, because these objects often suffer from chloride induced post excavation corrosion. The effectiveness of different approaches to remove the chlorine was tested at our facility.

3. Another project is about reverse engineering of ancient aqueduct systems in the Mediterranean area. It is possible to get information about the (re-)construction and operation history of these systems by analysing the variations of the elemental composition in the layering of the sinter material, because modifications in the system lead to changes in the water composition and further in the sinter composition as well. Variations of the elemental composition in the layering of sinter material were measured with a narrow collimated beam (stripe profile of 2-3 mm thickness).

We will give an overview about the results of these current projects and present some new developments at the PGAA facility and their potential use in archaeometry.

### O-14

### Quantitative chlorine analysis of archaeological iron objects by PGA Analysis

Britta Schmutzler<sup>1,\*</sup>, Zsolt Révay<sup>2</sup>, Petra Kudějová<sup>2</sup>, Christian Stieghorst<sup>2</sup>

<sup>1</sup> State Academy of Art and Design Stutttgart, Germany

<sup>2</sup> Heinz Maier-Leibnitz Zentrum (MLZ), Technical University of Munich, Garching, Germany

\* britta.schmutzler@gmx.de

### Background

Archaeological iron objects usually suffer from post excavation corrosion, induced by chlorine, which diffuses into iron objects during burial. Objects conservation has the task to extract the chlorine by e.g. washing methods since the destruction of this corrosion form is drastic and hard to control by climate conditions. Several approaches were developed the last decades, but "Alkaline Sulphite Method" has become to an international standard method in conservation for archaeological or marine objects. It is based onto alkaline washing solutions of half molar sodium hydroxide, which contain half molar sodium sulphite for deaerating the aqueous solutions, heated up to 70 °C and stirred. Several variations of this protocol were examined by conservation science the last years due to restrictions in time, budget and personnel in the conservation departments, creating needs for simpler and cheaper possibilities in mass treatment. For example, diluted or cold solutions were tested in the empirical research project "Saving from Rust" (funded by DBU Deutsche Bundesstiftung Umwelt / Osnabrück / Germany), using original archaeological material as specimens. The key for meaningful results is the chlorine resp. chloride analysis, which could formerly be conducted with destructive methods only. So there was the need for non-destructive methods for chlorine analysis in further projects what could be realized by PGA analysis at the Heinz Maier-Leibnitz Zentrum (MLZ). The residual chlorine, which is still present inside the treated objects after conservation, is the hard criterion in the evaluation of the conservation success – the less, the better.

### PGAA in the framework of conservation research

The research project "Besonderes Eise(r)n bewahren" (DBU funded) is presented with data from chlorine analysis by PGAA, complementary checked by oxygen consumption as a second non-destructive testing for corrosion by residual chloride (the more chlorine a treated object contains, the more oxygen is consumed in accelerated corrosion tests). The conservation treatment was conducted by use of alkaline solutions with varying physical parameters (e.g. temperature; ultrasonic assisted extraction). PGAA data were used for comparison of the impact of these different treatment parameters. The huge advantage is the possibility of repeated measurements before, during and after treatment as well as control measuring (reproduction). The main goal of the research work is to diminish the residual chlorine to very low or zero level for optimum stabilization of the archaeological iron objects against post treatment corrosion. And to preserve as much as archaeological iron objects in their actual state of condition for upcoming research approaches.

## Neutron phase-contrast imaging for revealing armourers' marks

Alan Williams<sup>1</sup>, David Edge<sup>1</sup>, Francesco Grazzi<sup>2</sup>, Nikolay Kardjilov<sup>3,\*</sup>

<sup>1</sup> The Wallace Collection, London, UK

<sup>2</sup> Consiglio Nazionale Ricerche, Italy

<sup>3</sup> Helmholtz-Zentrum-Berlin, Germany

\* kardjilov@helmholtz-berlin.de

High-quality armour made during the Middle Ages, such as the products of 15th century Milan, which were exported in some quantity, was frequently marked by the craftsmen who made pieces of plate armour, as well as by the "traversator" who assembled the suit. These marks can identify both the craftsman and the city and, sometimes also, the arsenal where it might have been stored. Much of the history of Medieval and Renaissance technology and trade can be reconstructed from the study of the marks, especially where they can be correlated with the purchases of customers and the written records of guilds and courts. These marks were stamped on the finished plates but frequent use and polishing has worn away the top layer of steel and, in many cases, only traces of the marks are now visible. However, if these marks had been struck in cold metal, there should still be layers of strained grains below the outermost polished layer. Forensic scientists usually etch metal artefacts such as gunbarrels in order to reveal hidden serial numbers by using strong acids to dissolve away the polished layers. This approach is, of course, ruled out for museum objects. We believed that advanced neutron imaging techniques would provide a non-invasive alternative. In this research, we performed radiographic experiments exploiting polychromatic phase-contrast imaging, a method for contrast enhancement in transmitted neutron beams by measuring a sample made with a homogeneous material but exhibiting a different microstructure induced by mechanical actions. We were able to reveal the armourers' marks now obscured by polishing that are present in these examples of medieval armour.

#### O-15

# Neutron imaging as tool for investigations on historical musical instruments

### David Mannes<sup>1,\*</sup>, Eberhard Lehmann<sup>1</sup>

<sup>1</sup> Paul Scherrer Institut, Villingen, Switzerland

\* david.mannes@psi.ch

Musical instruments represent a multifaceted and rather delicate section in cultural heritage objects. They can consist of simple piece of wood with holes or represent a complex mechanical apparatus made of a wide combination of materials. Beside the preservation of the physical integrity of musical instruments the preservation of the sound is a very important topic, as it represents a unique feature of every historical instrument. Non-destructive investigations, such as neutron imaging help to handle these manifold issues, by providing information on the construction material composition and condition of the musical instruments. Neutron imaging proves to be an important tool for such investigation as many instruments consist of a mixture of metallic and organic materials. It allows beside the simple documentation of the instrument's shape to investigate small amounts of hydrogen-containing materials (e.g. corroded areas on the inside of the instruments, wax, laquer, moisture...). In our presentation, we give an overview on different projects which have been carried out at the neutron imaging facilities of the Paul Scherrer Institut (PSI) in Villigen (Switzerland). Some of the presented projects aim at a better understanding of the construction, shape and dimensions of certain historical instruments, comprising wooden instruments as well as brass wind instruments in order to allow for accurate copies of the instruments, which could be played again. Another project aims at the playability of historical brass wind instruments and the impact on the instruments condition i.e. internal corrosion. Historical instruments were played during a period of one year and the condition of the instruments, with special focus on the development of corroded areas, was monitored by means of neutron imaging, carrying out tomography scans at the beginning and the end of the project. With the before mentioned projects and additional case studies, we will show the possibilities and limitations of neutron imaging in the area of investigations of historical musical instruments.

# Searching for "the needle in the haystack": reconstructing the brain and vascular system of the mammalian forerunners

## Michael Laaß<sup>1</sup>, Burkhard Schillinger<sup>2,\*</sup>

<sup>1</sup> University Duisburg/Essen, Germany

<sup>2</sup> Heinz Maier-Leibnitz Zentrum (FRM II), Germany

\* burkhard.schillinger@frm2.tum.de

Only little is known about the brain anatomy of the mammalian forerunners, the nonmammalian therapsids, because the brain reconstruction is faced with several problems. The first is that soft-tissue is not preserved. Consequently, information about the shape of the brain can only be deduced from the surrounding bony cavity. A second problem is that the brain cavities of most therapsids were only incompletely ossified. Therefore, the forebrain of most therapsids is only known from slight impressions on the underside of the skull roof. Moreover, it seems likely that the brain often did not fully fill in the braincase as it is also the case in modern turtles and reptiles. Furthermore, nerves and blood vessels were often covered by the meninges and did not leave impressions in the braincase. Consequently, the vascular system of the therapsid brain is almost completely unknown. Finally, fossil skulls are usually filled with sediment and cannot be studied from external. Mechanical preparation is often impossible to avoid destruction of the valuable specimens. For this reasons it is useful to search for therapsid species with well ossified braincases, in which the brain was in close contact to the surrounding bones by means of non-destructive methods for investigation such as neutron radiography.

One "needle in the haystack" we found was the dicynodont Diictodon feliceps. A specimen of Diictodon investigated at the facility ANTARES at FRM2 in Munich clearly showed impressions of several blood vessels on the inner surface of the braincase. This is an indication for a close contact between the brain and the braincase and enabled us to reconstruct hitherto unknown parts of the vascular system. In this context, the nature of a controversially discussed depression in the braincase, the "unossified zone", could be clarified. The neutron tomographic images clearly showed that the unossified zone was a terminal chamber of several blood vessels at the level of the hindbrain. Furthermore, the course of another blood vessel, the prootic sinus, could be reconstructed and several passages of cranial nerves could be identified, which penetrated the braincase [1].

Another example is the dicynodont therapsid Kawingasaurus fossilis. Neutron tomography showed that Kawingasaurus possessed an almost completely ossified braincase. Brain structures such as the cerebral hemispheres left sharp impressions in the brain cavity, which enabled a less hypothetical reconstruction of the forebrain for the first time [2]. Finally, it can be stated that neutron tomography is a very useful tool in palaeoneurology for the investigation and virtual reconstruction of cranial endocasts of fossil skulls.

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# O-17

# Crystallographic characterization on different types of structure (Tsukurikomi) of Japanese swords using pulsed neutron imaging and diffraction methods

<u>Kenichi Oikiwal</u><sup>1,\*</sup>, Yoshiaki Kiyanagi<sup>2</sup>, Takenao Shinohara<sup>1</sup>, Tetsuya Kai<sup>1</sup>, Kenichi Watanabe<sup>2</sup>, Akira Uritani<sup>2</sup>, Genki Hori<sup>2</sup>, Yuho Hirata<sup>2</sup>

<sup>1</sup> Japan Atomic Energy Agency

<sup>2</sup> Nagoya University, Japan

\* kenichi.oikawa@j-parc.jp

The Japanese swords are very attractive not only as a work of art but also a metallurgical point of view. There are various kinds of the structure combining the Japanese swords materials, i.e., the steel combining method of making Japanese sword blade (Tsukurikomi). Among them, Honsanmai was popular in Edo period (1603–1867) and is also popular in modern Japanese swords. However, at rather early stage of the Japanese sword history, Mukukitae (formula without combining different steels) was adopted. Various kinds of Tsukurikomi swords were already studied by using destructive methods, such as microscope, chemical analysis, X-ray diffraction, etc. This observation style was possible in the past, but at the present time when Japanese vintage swords became valuable, it is indispensable to establish non-destructive analysis method to identify the type of Tsukurikomi.

Non-destructive experiments such as pulsed neutron imaging and diffraction are powerful tools to study metallic cultural heritages due to their high penetrating power and capability to give crystallographic information. For example, the strained and the no strained samples show different shape of the Bragg edge spectra as well as the Bragg peak patterns, which indicates difference in treatment methods of steel. For the first trial of our study, three kinds of Japanese swords were made by a swordsmith. Two of them are Mukukitae and one is Honsanmai. Pulsed neutron imaging and diffraction experiments were performed at RADEN and NOBORU at J-PARC, respectively. Each sword samples were cut in three parts to be measured simultaneously by using the  $100 \times 100 \text{ mm2}$  area detector, nGEM. Neutron diffraction experiments were focused on specific parts of the swords to see the difference in combination of the steels. We are now analysing the measured 2D-transmission spectra using RITS code to obtain spatial distribution of the crystallite size, the texture variation and the edge shift and broadening (hardness) mapping for each sample. Complementary diffraction data analysis is also on going. Detailed analysis results will be presented.

Acknowledgments: The authors would like to thank Naohiko Sasaki from the Japan Steel Works, Ltd. for forging sword samples and valuable comments on them.

## The neutron imaging beamline in Delft and future plans

Lambert van Eijck<sup>1,\*</sup>, Zhou Zhou<sup>2</sup>, Jeroen Plomp<sup>2</sup>, Catherine Pappas<sup>2</sup>

 $^1$  TU Delft Appl. Sci. dep. RadiationSciTech

<sup>2</sup> TUDelft / fac. Appl. Sci. / dep. RadiationScienceTechnology

\* l.vaneijck@tudelft.nl

The technical university of Delft runs a 2MW research reactor which is used for neutron activation analysis, research on isotope production schemes, and for positron and neutron research, mostly in food and materials science. We have recently built a competitive neutron imaging beam line with limited means, based on which we now have several collaborations running. Here we will present the benchmarking experiments that we have performed with our generous colleagues from PSI, the first experiments with musea and several other universities in the Netherlands, as well as our plans for the near future.

#### O-18

#### O-19

# PGA-Imaging and Neutron Tomography at MLZ's PGAA facility (FRM-II). A position-sensitive spectroscopy-visualization method with neutrons

Eschly Jan Kluge<sup>1,\*</sup>, Jan Jolie<sup>1</sup>, Zsolt Révay<sup>2</sup>, Christian Stieghorst<sup>2,\*</sup>

<sup>1</sup> Institute of Nuclear Physics, University of Cologne, Germany

 $^{2}$  Maier-Leibnitz Centre, Technical University of Munich, Germany

\* ekluge@ikp.uni-koeln.de; christian.stieghorst@frm2.tum.de

## Introduction

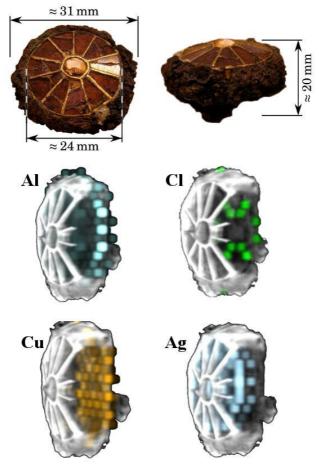
Thanks to the increase of more diverse and research dedicated neutron sources as well as advancements in digital recording techniques, the range of available spectroscopy and imaging methods utilizing neutrons has significantly expanded in recent years. Especially the relatively new Prompt Gamma Neutron Activation Analysis Imaging and Neutron Tomography (PGAI-NT), a method to obtain and effectively visualize ever-higher resolving position-sensitive spectroscopy data sets, shows promising results for material research and archeometry.

### The PGAI-NT method

PGAI-NT is the combination of a position-sensitive three-dimensional extension of the Prompt Gamma Neutron Activation Analysis (PGNAA) of a sample and the sample's Neutron Tomography (NT) for the purpose of an easy assignment and clear representation of spectroscopy data. The via position-sensitive PGNAA obtained distribution of the various elements within a sample is visualized by differently coloured voxels, hence the term PGA-Imaging. The relative abundance of a specific element itself is visualized by different intensities of the colour assigned. The resulting voxel-matrix is finally embedded into the tomography-volume of the bulk sample. A PGAI sub-volume of a sample is established by the intersection of the collimated neutron beam and the collimated field of view (FOV) of one or more gamma detectors positioned in 90 degrees to the beam.

## The Current Instrument

Because PGAI-NT is a method-combination with a strived application area, it should be as user friendly and cost effective as possible. For this reasons we opted for an all-in-one instrument approach by conducting the NT recoding at the same instrument site. To quicker parallelize the elliptically tapered beam of the facility for PGAI, a boron-lead collimator with a 2x2 cm2 profile is used. The pinhole-aperture used in combination with said collimator is a 3 mm thick boron-carbid plate with a 2 mm diameter. The resulting beam profile at sample position has a flux of  $1.34 \times 10^{9}$  /cm2s and a PGAI-resolution determining diameter of 3.2 mm. The previously used gamma collimator was replaced by two 50 mm longer heavy-lead collimators with a conical 2x8 mm2 profile. In this way, the  $\gamma$ -energy dependent resolution in beam direction was improved by an average of 42%. The measuring time can be reduced significantly by using two collimated detectors simultaneously. The parallel-beam tomography flight-tube system was replaced by a conebeam setup, realized by introducing a pinhole-aperture into the focal point of the even further elliptically tapered beam. The boron-carbide pinhole-aperture for this usage is of 3 mm diameter and thickness, resulting in a 47% greater L/D ratio of 247(2). The current 5.5 megapixel camera in combination with a 100  $\mu$ m LiFZnS(Ag)P scintillator achieves a 130% improved object resolution of 175(4)  $\mu$ m. The reconstruction under cone-beam algorithms resolves now 140  $\mu$ m details for negative and 40  $\mu$ m details for positive contrast differences.



#### O-20

# Neutron imaging options with fission and thermal neutrons at the NECTAR facility

Malgorzata Makowska<sup>1,2,\*</sup>, Thomas Bücher<sup>B</sup>, Martin Mühlbauer<sup>1,4</sup>, Michael Schulz<sup>1</sup>

<sup>1</sup> Heinz Maier-Leibnitz Zentrum (MLZ), FRM II, Garching, Germany

<sup>2</sup> Bavarian Research Institute of Experimental Geochemistry and Geophysics (BGI), University of Bayreuth

<sup>3</sup> Technische Universität München, ZTWB Radiochemie München RCM, Garching, Germany

<sup>4</sup> Institute for Applied Materials (IAM), Karlsruhe Institute of Technology (KIT), Germany

\* malgorzata.makowska@frm2.tum.de

NECTAR [1] (neutron computed tomography and radiography) next to ANTARES is one of the two neutron imaging facilities located at the FRM II neutron source at Heinz Maier-Leibnitz Zentrum (MLZ). While Antares offers a broad range of versatile neutron imaging techniques utilizing a cold neutron spectrum, NECTAR, in contrast to most of the existing imaging beamlines, makes use of fission neutrons. This makes it a unique facility world-wide. The highly energetic spectrum with its mean energy at about 1.8 MeV is obtained via fission reactions taking place in the so-called converter plates placed in front of the window of the SR10 beam tube. The converter plates consist of 2 slabs of highly enriched uranium (93% 235U)-silicide with a total weight of 540 g. While the converter plates are not in the 'working position', a thermal neutron beam is available [2].

Due to the possibility of switching between fission and thermal spectra, NECTAR can be used for a broad variety of applications. The non-destructive inspection performed by neutron radiography and tomography using these two ranges of neutron energy can provide complementary information about the investigated objects. Penetration depth of fission neutrons is much higher as compared to cold or thermal neutrons, and thus gives more insight in large objects and samples containing strongly attenuating elements. In contrast, thermal neutrons provide a much better spatial resolution while still showing higher penetration depth than the cold neutrons available at ANTARES. Thus, due to high penetration depths, NECTAR is a well-suited instrument for investigation of inner structure of large, i.e. archaeological or paleontological objects. Because of the high sensitivity to light elements many applications are related to hydrogen or ammonia storage systems [3,4] and observation of water distribution in e.g. large wooden samples [5]. Neutron imaging capabilities and specification of the NECTAR facility as well as examples of typical applications will be presented.

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# Neutrons and complementary methods for Cultural Heritage research at the Budapest Neutron Centre

## László Rosta<sup>1,\*</sup>

<sup>1</sup> Wigner Research Centre for Physics, Hungarian Academy of Sciences, Budapest, Hungary

\* rosta.laszlo@wigner.mta.hu

In the past decade neutron methods have become and now constitute an important and unique part of the science tool kit for exploiting information about objects of Cultural Heritage. Neutrons are perfect tools of archaeometrical studies due to their nondestructive and non-invasive nature. Neutron methods can be used to explore compositional, structural and topological features of the samples. The application of various experimental techniques in the investigations of artefacts usually concern dating, provenance, manufacturing techniques, workshop affinities, as well as fake identification, conservation or preservation of objects. Intense neutron beams are produced in nuclear reactors or accelerator based neutron sources. The Budapest Neutron Centre (BNC) operates the 10 MW Budapest Research Reactor, it is a research organisation for the open access utilisation in science and technology development for its 15 experimental stations; this is a unique infrastructure of this kind in the Central European region. BNC has long traditions in archaeometry research, it has made part of EU projects on Cultural Heritage like CHARISMA and IPERION-CH. The usefulness and complexity of investigations with neutrons is based on the interaction of neutrons with matter by three major ways: 1) Imaging by passing neutron beams through objects – neutron radiography and tomography; this can reveal internal parts or hidden objects inside bulky materials. 2) Absorption of neutrons by nuclear reaction with atoms of studied materials – radiative capture via  $(n,\gamma)$  reaction; this technique gives information on the elemental composition of objects. 3) Week interaction with atoms – changing in the trajectory and velocity of neutrons passing through solid or liquid materials – neutron scattering (elastic or inelastic); measuring intensity variation of scattered neutrons from the sample reveal information on microstructure. BNC offers a complex approach of studying artefacts at its neutron facilities: diffractometers (ND), SANS, PGAA stations and imaging facilities. Complementary measurements are offered by the use of External Milli-Beam PIXE and compact XRF spectrometers, microscopes, mass spectrometers etc. also at BNC site. Some case studies as comprehensive analysis of archaeological objects by combined neutron techniques will be given.

#### K-03

# Neutron Imaging Methods for Cultural Heritage at the ANTARES facility

<u>Burkhard Schillinger<sup>1,\*</sup></u>, Michael Schulz<sup>1</sup>, Alexander Backs<sup>1</sup>, Tobias Neuwirth<sup>1</sup>, Marc Seifert<sup>1</sup>

<sup>1</sup> Heinz Maier-Leibnitz Zentrum (FRM II), Munich, Germany

\* burkhard.schillinger@frm2.tum.de

The ANTARES neutron imaging facility at Heinz Maier-Leibnitz Zentrum (FRM II) of Technische Universität München provides several imaging methods that are employed in cultural heritage research. Neutrons will always come into play when X-rays cannot provide the desired information because they cannot penetrate X-rays, or just deliver little to no contrast on the materials involved. Neutrons can often provide complementary contrast to x-rays which makes them ideally suited to investigate samples like bones in chalk rock, or enamel and dentine in fossil teeth, but also moisture or wood impregnating chemical agents. Standard neutron radiography provides insight into metallic objects containing organic substances. 3D computed tomography provides three-dimensional information about the internal structure of samples, like fossils embedded in rock with a spatial resolution of the order of 15µm Bragg edge radiography and tomography is based on energy-selective scanning of samples which allows to detect different metallic phases e.g. in medieval swords, providing information on the forging technique. Neutron grating interferometry (nGI) allows to detect structures (e.g. different materials, porosity, inclusions, etc.) below the real space resolution limit of an imaging instrument by analyzing the ultra-small-angle scattering originating from these structures. Hence, this technique allows to indirectly localize structures in the size range from 15 [lm to 0.5 [lm. By analyzing these data information about the manufacturing process of e.g. pottery can be gained. The talk will illustrate all methods employed at ANTARES with specific examples and explain the basic principles of the methods, including nGI.

Access to the ANTARES facility is easy, and free of charge for public research. Scientific proposals can be submitted twice a year. The Imaging group has experience in handling of valuable samples.

# The Metallurgical Texture of gold artefacts from the Bronze Age Rampart of Bernstorf (Bavaria) Studied by Neutron Diffraction

Friedrich Wagner<sup>1,\*</sup>, R. Gebhard<sup>2</sup>, W.M. Gan<sup>3</sup>, M. Hofmann<sup>4</sup>

<sup>1</sup> Technical University of Munich, Germany

 $^{2}$  Archäologische Staatssammlung München, Germany

<sup>3</sup> German Engineering Materials Science Center at MLZ, Helmholtz-Centre Geesthacht

<sup>4</sup> Heinz Maier-Leibnitz Centre (MLZ), Technical University of Munich, Germany

\* fwagner@tum.de

The metallurgical texture of several pieces of the gold artefacts found in 1998 at a Bronze Age rampart near Bernstorf in Bavaria was studied by neutron diffraction using the STRESS-SPEC instrument at the FRM 2 reactor of the Maier-Leibnitz Center of the Technical University of Munich. The pieces consist of about 0.1 mm thick gold sheets embossed with complicated ornaments. The purpose of the studies was to obtain information on the technique used to produce these gold foils. All studied spots with about 5 mm diameter on the artefacts were found to exhibit a cube type {100}<100> texture that is typical for many cold rolled and subsequently annealed and recrystallized fcc metals. A comparison with laboratory made reference samples allowed us to rule out hammering with or without subsequent annealing or cross-rolling for the manufacture of the gold foils. Pole figures similar to those of the Bernstorf gold were obtained by rolling followed by annealing. Beyond the interest of the result for this specific example of archaeological interest, texture determinations using neutron diffraction could be shown to be an easy and completely non-destructive method for obtaining information on the production methods of archaeological gold artefacts.

## IMAT: A new neutron imaging and diffraction beamline at ISIS

## Winfried Kockelmann<sup>1\*</sup>, Triestino Minniti<sup>1</sup>

 $^{1}$  Science and Technology Facility Council - Rutherford Appleton Laboratory

\* winfried.kockelmann@stfc.ac.uk

A new neutron tomography and diffraction facility has recently been taken into operation at the accelerator-based neutron spallation source ISIS at the Rutherford Appleton Laboratory at Harwell, UK. Neutron radiography and tomography can be used for a noninvasive 3D visualisation of inner components and structures of archaeological objects and heritage science materials. The high penetration power of neutron allows assessing the preservation state of an object; organic material inside metal structures can be made visible, and distributions of hydrogenous materials can be determined. Selected regions of interest can additionally be characterised by time-of-flight neutron diffraction, for studying the material compositions and crystalline structures.

The instrument is currently being commissioned and prepared for user operation. Initially, the instrument allows for white-beam neutron radiography and tomography; energyselective and energy-dispersive imaging options are available for maximizing image contrasts between given materials and for mapping microstructure features in metals and alloys. IMAT is a new kind of neutron instrument that makes use of time-of-flight transmission techniques. A unique feature of IMAT is that neutron imaging and neutron diffraction are available on the same beamline. The installation of diffraction detectors is envisaged in the near future, for spatially-resolved crystallographic phase, residual strain and texture analysis. We report on the installation status of IMAT, and present results from the instrument commissioning. We will discuss the flexibility that a multi-purpose instrument offers for cultural heritage materials science.

# Neutron diffraction for cultural heritage studies: the Italian Neutron Experimental Station INES@ISIS

## Antonella Scherillo<sup>1,\*</sup>

<sup>1</sup> Science and Technology Facility Council – ISIS, UK

\* antonella.scherillo@stfc.ac.uk

The Italian Neutron Experimental Station INES, located at the pulsed neutron source ISIS (U.K.), is a general-purpose neutron diffractometer that was built with a special care aiming to focus its use on cultural heritage related studies. Thanks to the high penetration power of thermal neutrons, archaeometric measurements performed through neutron diffraction allow us to determine bulk properties of the sample in a non-destructive way, in particular regarding phase analysis and microstructure. This opens up the possibility of scientific investigation on objects otherwise unsuitable, due to their cultural and/or historical importance. Here we describe the INES instrument and present the results of some recent measurements, in particular on bronze objects from Sardinia, Celtic coins from northern Italy and Indian steel swords.

## O-24

# Neutron and laboratory X-ray characterization of excavated Napoleonic artefacts from the Berezina Battlefield in Belarus

<u>Jérôme Beaucour</u><sup>1,6,\*</sup>, Duncan Atkins<sup>1</sup>, T. Pirling<sup>1</sup>, U. Koester<sup>1</sup>, Nikolay Kardjilov<sup>2</sup>, F. Ott<sup>3</sup>, E. Ando<sup>4</sup>, A. Tangettini<sup>4</sup>, I. Tomand<sup>5</sup>, I. Groutso<sup>6</sup>, C. Montet-Beaucour<sup>6</sup>, I. Matuschevskaya<sup>6</sup>, V. Danilovitch<sup>7</sup>, S. Dernovitch<sup>7</sup>, V. Kochman<sup>7</sup>, V. Lakiza<sup>7</sup>

<sup>1</sup> Institut Laue Langevin (ILL), Grenoble, France

\* beaucour@ill.fr

An important collection of artefacts have been unearthed during preventative archaeological surveys from 2012 to 2014 around the village of Stoudienka in Belarus – the site of the Napoleonic army's passage during the retreat from their Russian campaign in November 1812. Recovered objects such as fragments of imperial eagles, brass plates and buttons with faint outlines of inscriptions and several lead bullets have resulted in historian's needs for further characterisation to obtain otherwise uncertain information, for example their mode of destruction, their composition and dating.

The Belarus Academy of Science and the Centre d'Etudes Napoleoniennes collaborated with the Institute Max Von Laue Paul Langevin in a series of experiments using nondestructive neutron diffraction techniques available at the ILL on selected artefacts: For instance, mechanical deformation measurements on the strain imager SALSA provided insights into object biographies and neutron activation analysis was used to obtain clearer knowledge of fabrication processes. Collaborations with other centres (HZB Germany and LLB France) in neutron radiography and tomography as well as complementary XCT analysis at the laboratory 3SR in Grenoble, along with recent experiments in the neutron imaging facility NEXT-Grenoble at the ILL have revealed hidden details in certain objects and the identity of hitherto unrecognizable artefacts. Another advanced non-destructive technique - prompt neutron activation analysis – was carried out at UJF (Czech Republic) on a series of musket shots from Berezina and compared with similar shot recovered from the Waterloo battle site.

<sup>&</sup>lt;sup>2</sup> Helmholtz Zentrum Berlin (HZB), Berlin, Germany.

<sup>&</sup>lt;sup>3</sup> Laboratoire Léon Brillouin, Centre d'Etudes de Saclay (LLB - CEA, CNRS), France

<sup>&</sup>lt;sup>4</sup> Laboratoire 3SR, UGA, CNRS, Grenoble, France

<sup>&</sup>lt;sup>5</sup> Nuclear Physics Institute of the CAS (UJF), Husinec, Czech Republic

<sup>&</sup>lt;sup>6</sup> Centre d'Etudes Napoléoniennes, Levallois, France

<sup>&</sup>lt;sup>7</sup> Institute of History, National Academy of Sciences of Belarus, Minsk, Belarus.

# Investigation of a Simulated Chinese Jade Dagger by Neutron Radiography and Prompt Gamma Activation Analysis

Richard Livingston<sup>1</sup>, Ariel O'Connor<sup>2</sup>, Jacob Lamanna<sup>3</sup>, <u>Heather Chen-Mayer<sup>4,\*</sup></u>

<sup>1</sup> University of Maryland, USA

<sup>2</sup> Smithsonian American Art Museum, USA

<sup>3</sup> National Institute of Standards and Technology, USA

 $^4$  NIST, USA

\* chen-mayer@nist.gov

In ancient China during the Shang dynasty, ca. 1600-1046 BCE, jade and bronze daggeraxes (ge) were used by the elite as ritual symbols of power and prestige. These ceremonial weapons consist of a nephrite jade [Ca2(Mg, Fe)5Si8O22(OH)2] blade mounted in a bronze haft. Several of these daggers are held by the Smithsonian Institution, and their conservators are interested in applying neutron radiography to image the jade tang hidden within the bronze haft and uncover early manufacturing techniques.

As a preliminary test of feasibility, a simulated dagger was made using a modern replica blade carved from nephrite from Wyoming, USA, and copper alloy plates. The simulated dagger was first imaged by X-ray and neutron radiography at the NIST Neutron Imaging Facility. The details of the jade tang within the copper alloy haft were clearly visible.

Subsequently, the nephrite blade and the copper alloy plates were analyzed by cold prompt gamma neutron activation (PGAA) to evaluate the feasibility of this technique for identifying the nephrite source. The PGAA was performed at the Cold Neutron PGAA station at NIST. Three nephrite specimens in the Smithsonian collection from China, Siberia and Taiwan, characterized previously by electron microprobe analysis, were used as comparative standards. The major nephrite elements – Ca, Mg, Fe and Si – were analyzed with uncertainties in the range of 0.3-0.4%. Three of the trace elements conventionally used for sourcing, Cr, Mn and Ni, were analyzed with similar uncertainties. In addition, other trace elements possibly useful for sourcing – B, Cl, Zn, Sr, Sm, and Gd – were detected.

### O-25

# Investigating beads from Chalcolithic funerary cremation contexts of Perdigões, Portugal

## <u>M. Isabel Dias<sup>1,\*</sup></u>, Zsolt Kasztovszky<sup>2</sup>, M. Isabel Prudêncio<sup>1</sup>, Ildikó Harsányi<sup>2</sup>, Imre Kovács<sup>3</sup>, Zoltán Szőkefalvi-Nagy<sup>3</sup>, Judit Mihály<sup>4</sup>, György Káli<sup>3</sup>, Antonio C. Valera<sup>5</sup>

<sup>1</sup> Centro de Ciências e Tecnologias Nucleares, Campus Tecnológico e Nuclear, Instituto Superior Técnico, Loures, Portugal

<sup>2</sup> Centre for Energy Research, Hungarian Academy of Sciences, Budapest, Hungary

<sup>3</sup> Wigner Research Centre for Physics, Hungarian Academy of Sciences, Budapest, Hungary

<sup>4</sup> Research Centre for Natural Sciences, Hungarian Academy of Sciences, Budapest, Hungary

<sup>5</sup> Era Arqueologia, Núcleo de Investigação Arqueológica – NIA. Cç. de Santa Catarina, 9C, 1495-705 Cruz Quebrada – Dafundo – Portugal. Interdisciplinary Center for Archaeology and Evolution of Human Behavior (ICArHEB, Universidade do Algarve, Campo de Gambelas, Faro, Portugal

\* isadias@ctn.tecnico.ulisboa.pt

Beads appear to have been one of the earliest examples of personal adornments. The analyzed beads come from Pit 40, a funerary secondary deposition of cremated remains of more than a hundred individuals. The pit is located in the center of Perdigões (Évora, Portugal) ditched enclosures and is dated from the third quarter of the 3rd millennium BC. The beads were burned and were part of the funerary votive assemblages, also composed by arrowheads ivory figurines, marble idols and pots, phalanx idols, copper awls, Pecten shells and pottery sherds. These beads have between 5 and 10.5 mm in diameter, with a central perforation up to 5 mm and maximum thickness of 3 mm, they are gray or black and of unknown nature. We report the use of prompt-gamma activation analysis (PGAA), external milli-beam particle induced X-ray emission spectroscopy (PIXE) and high-resolution time-of-flight diffractometer (ToF-ND) at the Budapest Neutron Centre, to a large dataset demonstrating that composition can help on identification of beads nature by using non-destructive techniques. Infrared spectroscopic measurements were also performed on some beads. In one broken bead a micro-destructive - Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectrometry (SEM/EDX) analysis was applied at IST. Among the chemical elements obtained by PGAA, calcium (53%<CaO<74%) is the element with major concentrations. Sr, Si and Fe are also present in the majority of the samples. Sr contents can be explained by calcium substitution within the lattice; Si and Fe can be related with soils particles that are still in the beads. The chemical results obtained by PIXE show that the surface is more contaminated with soils particles due to high Si, Fe and K contents, and phosphorous was found in higher proportion, certainly originated from bones. FTIR patterns show that the major part of the beads is calcium carbonate crystallized in calcite form, contaminated with silicates and calcium phosphate. The ToF-ND results also indicate the crystal lattice structure of calcite. Aragonite, a polymorph formed in the biomineralisation process of shells, was not detected, indicating a total phase transformation to calcite due to heating processes during funerary practices. The low amount of Mg, usually present in shells below the detection limits of applied methods, as well as, eventual alterations of elemental proportion derive by the burning and burial processes, may compromise the identification of the marine / estuarine origin of the shells. The SEM-EDS results clearly show the shell nature of the archaeological beads, with the characteristic shell macrostructure, with inner, intermediate and outer layers of calcium carbonate. Thus, the results obtained showed that shells were the raw materials used for the production of these beads, found in funerary contexts and burnt together with bones during funerary practices in Perdigões.

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# Multi-technique archaeometallurgical investigations of metal objects from the Marche Region, Italy

Massimo Rogante<sup>1,\*</sup>, Adél Len<sup>2</sup>, Jacek Milczarek<sup>3</sup>, László Rosta<sup>2</sup>

1 Rogante Engineering Office, Italy

2 Wigner Research Centre for Physics, Hungarian Academy of Sciences, Budapest, Hungary

3 Narodowe Centrum Badań Jądrowych

\* main@roganteengineering.it

Small angle neutron scattering (SANS) and dynamic neutron radiography (DNR) investigations have been performed for the non-destructive analysis of different ancient and new textiles. SANS allowed comparing the nanostructure of ancient and modern linen fabrics, as well as studying the neutron scattering properties of different types of modern wool fibres, providing new data useful to understand the structural basis for their characteristics and progressing technological and material description. DNR consented studying the hydrogenous liquid migration within new fabric samples, investigating the spontaneous imbibition of single sheets of different textiles. Such experiment proved that DNR is sensitive enough for studies of the kinetics of water migration, determining the dependence of the wetting front motion on time and the changes in spatial water distribution along the fabric. This kind of analysis can contribute to solving problems of conservation and preservation. The considered neutron characterization has also provided scientific data for further and more comprehensive analyses in the Cultural Heritage field.

# K-04

# Chemical imaging of paintings: X-rays and ions as complementary probes to neutrons

## <u>Thomas Calligaro</u><sup>1,\*</sup>

 $^1$ Centre de Recherche et de Restauration des Musées de France C2RMF

\* thomas.calligaro@culture.gouv.fr

Beams of X-rays and, to a lesser extent, ions and neutrons (with the neutron autoradiography technique) have been successfully applied to collect elemental maps of historical paintings. Such probes can notably reveal the pigment distribution within entire paintworks through their elemental fingerprints without contact and in a non-destructive way. The comparison of the recorded images with pictures taken with routine imaging techniques (e.g. visible, UV, IR photography and X-ray radiography) provide curators, art historians and restorers information concerning the painter techniques and choices, and can help in the preparation and the monitoring of restorations.

The presentation focus on two elemental imaging techniques developed at the C2RMF. The first relies on the use of a scanning sub-millimetre X-ray beam of a transportable, versatile and cost-effective XRF scanner designed and built at the C2RMF. Initially developed at the synchrotron, such technique has been transposed to the laboratory under the name MA-XRF [1]. The second approach is based upon a scanning external millibeam of protons of a few MeV in PIXE mode at the AGLAE accelerator facility [2]. The merits and limitations of both methods will be discussed. For example, excitation protons confers to PIXE a probing depth that vary with incident energy, an improved yield for light elements compared to heavier ones, and an easy beam scanning. On the other hand, a scanning X-ray beam is probing deeper, exhibits unique features such as Compton scattering and is less susceptible to induce modifications in paint materials than ion beams. Finally, the penetration of neutrons and gamma-rays permits to image isotopes independently of the paint layer thickness and stratigraphy.

The capabilities and limitations of MA-XRF and PIXE imaging of will be illustrated with case studies carried on paintworks. The Saint John the Baptist by Leonardo da Vinci (muse du Louvre) was studied using MA-XRF and paintings from the 16th and 19th c. were investigated using both MA-XRF and scanning-PIXE.

- E. Ravaud, L. Pichon, E. Laval, V. Gonzalez, M. Eveno, T. Calligaro, Development of a versatile XRF scanner for the elemental imaging of paintworks, Appl. Phys. A122 (2016) 17
- [2] T. Calligaro, V. Gonzalez, L. Pichon, PIXE analysis of historical paintings: Is the gain worth the risk?, Nucl. Instr. and Meth. B363 (2015) 135

# POSTER PRESENTATIONS

# Non-destructive evaluation of the microstructure of iron parts of traditional Japanese sword manufacturing process by pulsed neutron transmission spectroscopy

<u>Genki Hori</u><sup>1,\*</sup>, Yoshiaki Kiyanagi<sup>1</sup>, Akira Uritani<sup>1</sup>, Manako Tanaka<sup>2</sup>, Atsushi Yamazaki<sup>1</sup>, Kenichi Watanabe<sup>1</sup>

<sup>1</sup> Nagoya University, Japan

 $^{2}$ Showa Women's University, Japan

\* hori.genki@d.mbox.nagoya-u.ac.jp

#### Introduction

In the case of iron cultural properties such as Japanese sword and matchlock gun, manufacturing techniques and material characteristics are not clear. To get detailed information about crystallographic characteristics of those iron artefacts is desired for elucidating them. In the analysis of precious iron cultural properties, non-destructive evaluation is preferable. Neutron has high penetrating power, and it is possible to get inside information non-destructively. Moreover, by using pulsed neutron transmission spectroscopy, we can obtain crystallographic information by analyzing the low-energy neutron transmission spectra including Bragg edges. The position and the shape of the Bragg edge give information such as strain, crystallite size, texture and density. It is considered to be useful to study such crystallographic parameter change of iron parts during making process to consider the effect of the making process to the crystallographic information. We performed pulsed neutron transmission experiments for iron parts at different making stages and analyzed the transmission data by using the RITS code [1] to obtain crystallographic information.

## Experimental

Neutron experiments were performed at NOBORU beam line at J-PARC Material and Life Science Experimental Facility in Japan. Ten iron samples at different making stages were cutout from Japanese swords made with the same process. The sample was placed in front of a two-dimensional position sensitive detector, and the transmission spectra of the pulsed neutrons were measured by the time-of-flight method.

#### Results

Here, we concentrate on the crystal lattice spacing since it will be affected by hammering and quenching. In the sample before quenching, the crystal lattice spacing shows minimal around the centre between the cutting edge and the back of the blade. The trend may be due to hammering. In the sample after the quenching, the lattice spacing around the cutting edge became larger with approaching the end of the edge. This increase is clearly attributed to content of martensite phase. We obtained such spatial dependent change from 'pilling and melting' to 'Kaji Togi (Adjusting the shape).

[1] H. Sato, T. Kamiyama, Y. Kiyanagi, Materials Transactions, Vol. 52, No. 6, pp. 1294-1302, (2011)

#### P-02

## Detector Development for Mobile Fast-Neutron Radiography

<u>Florian Reisenhofer</u><sup>1,\*</sup>, John Kettler<sup>1</sup>, Andreas Havenith<sup>2</sup>

<sup>1</sup> RWTH Aachen University – Nukleare Entsorgung und Techniktransfer, Germany

<sup>2</sup> Aachen Institute for Nuclear Training GmbH, Germany

\* reisenhofer@net.rwth-aachen.de

One of the priorities of the PERTINAX project ('periodic testing by imaging with neutrons in addition to X-rays') is the development of a detector system for fast-neutron radiography. The aim of the project is the development of a mobile fast-neutron radiography system. Such systems allow the Non-Destructive Assay (NDA) of large volumes with high density materials and shielded components, such as radioactive waste containers. Applications include, for example, the detection of local water accumulations or the combination with neutron activation analysis methods for improved results. The project started in November 2016 and is funded by the Federal Ministry of Economic Affairs and Energy (BMWi) under the funding code 1501534. Previous work and research was done in the project 'neutron imaging system for radioactive waste analysis' (NISRA) [1].

Feasibility study: scintillator materials / single-pixel-detector

For the detector system, different scintillator materials as well as different detectors for scintillator readout come into consideration. In a first feasibility study the properties of organic scintillators like trans-stilbene, EJ-299-33A/34 from Eljen Technology [2] and others will be tested in combi- nation with a standard photomultiplier tube. These scintillators allow pulse-shape discrimination (PSD) which can be used to distinguish  $\gamma$ -radiation from neutrons. This becomes appealing to avoid  $\gamma$ -fogging in an environment where  $\gamma$ -radiation is present. For processing the signals, NIM-based electronics will be used in this feasibility study. In a further experiment radiographs shall be produced using a remotecontrollable coordinate table, which is applied to scan samples irradiated by a 10 Ci Am-Be neutron source.

### Readout detectors that allow spatial resolution

The major challenge of PERTINAX is the implementation of a detector system that offers sufficient spatial resolution. Different detector types for scintillator readout, which fulfil this criterion and also offer the opportunity to apply PSD, are available. Detectors like multi-anode photomultiplier tubes or arrays of silicon photomultipliers are included in this category. These detectors are already being used in the field of medical imaging. Since PSD has to be applied for every channel/pixel, suitable electronics which is able to observe multiple channels has to be developed. Electronics which meets these requirements is under development or already being used at the RWTH Aachen University, for example, in the field of astrophysics. Parts thereof can be modified and are suitable for an application in fast neutron radiography.

Perspective - neutron radiography in combination with neutron activation analysis

Knowledge of sample geometry and homogeneity are important issues performing neutron activation analysis. The presence of  $\gamma$  and neutron shields or hydrogen-containing substances can lead to uncertainties through their impact on self-shielding factors, neutron flux etc. Taking into account structural information about the sample, achieved by neutron radiography, offers the opportunity to improve the results of neutron activation analysis of large waste packages. References

- [1] J. Kettler et al., NINMACH 2013 Conference Abstract Booklet
- NEUTRON/GAMMA PSD PLASTIC SCINTILLATOR EJ-299-33A, EJ-299-34 Datasheet, Eljen Technology, Revision Date 2/8/2016

#### P-03

# Microstructural characterization of European historical swords through neutron imaging

<u>Francesco Grazzi</u><sup>1,\*</sup>, Burkhard Schillinger<sup>2</sup>, Alan Williams<sup>3</sup>, Dominik Bausenwein<sup>4</sup>, Raphael Beuing<sup>2</sup>, Joachim Kreutner<sup>4</sup>

<sup>1</sup> Consiglio Nazionale Ricerche (CNR), Italy

<sup>2</sup> Heinz Maier-Leibnitz Zentrum (FRM II), Germany

<sup>3</sup> The Wallace Collection, London, UK

<sup>4</sup> Bayerisches Nationalmuseum, Munich, Germany

\* francesco.grazzi@isc.cnr.it

Historical metallurgy is one of the most interesting fields of archaeometry, especially concerning the production and use of steel for arms and armour. It is evident from several analyses performed on steel samples that the production of arms and armour used cutting edge technology of that time so a study of such artefacts can give fundamental details about the technological skill of a specific area or period. In order to correlate similar samples of a specific age or provenance, it is important to build trustworthy classification parameters rather than simply relying on aesthetic features. Neutron imaging techniques allow us to determine the morphology and microstructure of composite steel artefacts thus allowing us to characterize the composition, the quality of the steel, the presence of welds and the thermal treatment during manufacture. We intend to start a systematic study to characterize the production methods of European swords in Europe from the early Middle Ages to late Renaissance and to 17th century in order to learn about the evolution of the production techniques: the smelting process to produce iron and steel was better understood and so the quality of the steel increased enormously with a substantial change in the composition and in the reduction of the number and size of slag inclusions and structural defects. The thermal treatments and the structure of the blades changed as well according to the requirements of different fighting techniques made possible by a better knowledge of steel metallurgy. All these characteristics directly influence the morphology and phase distribution of the blades and can be easily observed through neutron imaging. On this purpose we started analyzing three swords of great historical and artistic importance now belonging to the Bayerisches Nationalmuseum with white beam and energy selective methods. The details of the swords are listed here. - Longsword, produced in Tyrol in the late 15th century, made of steel, enamel, leather. From Ambras Castle, Innsbruck. This sword is generally considered to be one of the most beautiful medieval swords in existence today. - Hunting sword, produced by Melchart Diefstetter, Munich, c. 1550, made of steel, wood, mother of pearl, bone, leather. From the collections of the House of Wittelsbach. Only the lower part of this sword blade has been sharpened on both sides. It is likely that this 'long knife' was used for hunting. - Sword, produced in Northern Italy, possibly Milan, c. 1560, made of steel, enamel. From the Kunstkammer of the margraves of Brandenburg-Ansbach. This majestic sword is considered to be one of the most important objects ever to have been produced by an Italian weaponsmith.

White beam tomography allowed to detect the presence and shape of several morphological features in the bulk of the blades as multilayered structures, cracks, defects, and to determine the width and the shape of the martensitic hardened edges. Energy selective analysis allowed us to determine details of the steel composition and microstructure as well as mapping the different low and high carbon areas.

# A Feasibility Study of Cultural Properties in Hidden Relics using Neutron and X-ray Imaging Technique

<u>TaeJoo Kim<sup>1,\*</sup></u>, JinMan Kim<sup>1</sup>, Jongyul Kim<sup>1</sup>, Changee Lee<sup>1</sup>

<sup>1</sup> Korea Atomic Energy Research Institute

\* tj@kaeri.re.kr

Cultural heritages are from the past and must be passed on to future generations. Information of cultural heritages, especially the internal structure and composition, is important for its conservation. Such Information of cultural heritages is also important source for the study of ancient manufacturing technologies and its function. Interestingly, some heritages contain other historic relics inside. Many researchers have attempted to analyze these hidden relics. However, most of them remain unexplored because an investigation is rarely possible without a destruction of the samples or containers, which are also precious heritage items. Among the many non-destructive tests, the X-ray has come to be widely used for studies on the majority of archaeological objects. Generally, X-ray cannot give detailed information inside of a thick sample because of the attenuation characteristics. Neutrons, as opposed to X-rays, are the best type of probe for examining the interior of a thick sample. However, neutrons are used far less frequently in this field because of accessibility issues. In this study, we investigated the hidden relics using neutron and X-ray imaging technique with different types of source. In addition, preliminary results obtained from measurements on cultural heritage samples are discussed.

# Examining pottery forming techniques through combined petrographic analysis and neutron tomography

John Gait<sup>1,\*</sup>, Katalin Bajnok<sup>2,3</sup>, Zoltán Kis<sup>4</sup>

<sup>1</sup> Fitch Laboratory, British School at Athens, Greece

<sup>2</sup> Wigner Research Centre for Physics, Hungarian Academy of Sciences, Budapest, Hungary

<sup>3</sup> Institute of Archaeological Sciences, Eötvös Loránd University, Budapest, Hungary

<sup>4</sup> Centre for Energy Research, Hungarian Academy Sciences, Budapest, Hungary

\* john\_gait@hotmail.com

This poster presents the preliminary results of an investigation of pottery forming techniques combining petrographic analysis of ceramic thin sections with two-dimensional and threedimensional neutron imaging.

Within the context of archaeological and anthropological studies of material culture, it has become increasingly recognised that the manner in which artefacts were produced may have been influenced by a range of social as well as cultural factors, and as such may be as informative as more traditional typological or stylistic approaches to interpreting objects. With regard to pottery, the choice of primary forming techniques (e.g. wheel throwing, coiling, percussionforming etc.) have tended to be regarded as conservative practices, being passed on through successive generations within a close social network. Consequently, pottery forming techniques may provide insight into past social and cultural dynamics that may be obscured by more overt stylistic changes, which themselves may be more readily influenced by external factors. However, in many instances, actual physical traces of primary forming techniques are not preserved, or are difficult to discern without causing significant damage to the objects in question.

In certain instances evidence of pottery forming techniques may be inferred from the orientation of particles or voids within the ceramic structure. In order to investigate the potential application of neutron imaging, and in particular three-dimensional tomography, as a nondestructive analytical tool for the investigation of forming techniques, two sherds of archaeological pottery that had previously been studied by thin section petrography, were analysed at the Budapest Neutron Centre. The sherds in question belonged to the Lower Nubian A-Group (c. 3700-2800BC) and C-Group (c. 2300-1600BC) cultures, and were known to contain a large proportion of fibrous-organic temper.

The results are presented as two three-dimensional models showing the internal structure of the sherds. This preliminary study has indicated that both inorganic and organic inclusions (whether carbonised or relic voids) can be detected using neutron imaging techniques, and with sufficient resolution to enable particle orientations to be discerned. The patterns of particle orientation observed in these models largely matches that inferred from earlier petrographic studies, in turn indicating the probable use of coil-building forming techniques. Neutron tomography may therefore represent an effective, non-destructive, means in which to investigate forming techniques in fibrous-organic-tempered pottery. Future analysis and interpretation of the data, together with analysis of experimental pottery, aim to further refine the technique.

# Preliminary result of investigation of the metal composition of coins from Phanagoria's treasure by method of neutron resonance capture analysis

<u>Almat Yergashov<sup>1,\*</sup></u> Nina Bazhazhina<sup>2</sup>, Pavel Sedyshev<sup>2</sup>, Shakir Zeynalov<sup>2</sup>, Yuri Mareev<sup>2</sup>, Saltanat Mazhen<sup>3</sup>, Irina Saprykina<sup>4</sup>, Mikhail Abramzon<sup>5</sup>, Valery Shvetsov<sup>2</sup>

<sup>1</sup> Joint Institute for Nuclear Research, Dubna, Russia

<sup>2</sup> Frank Laboratory of Neutron Physics, Joint Institute for Nuclear Research, Dubna, Russia

<sup>3</sup> State University "Dubna", Russia

<sup>4</sup> Institute of Archaeology Russian Academy of Sciences

<sup>5</sup> Nosov Magnitogorsk State Technical University

\* almat\_16.93@mail.ru

The method of neutron resonance spectroscopy – Neutron Resonance Capture Analysis (NRCA) is developed in the Laboratory of Neutron Physics for the purpose of determination of the element composition of samples. Identification of elements and isotopes is carried out by measuring the energy of neutron resonances in the reaction of radiative capture, and their content in the sample is determined by measuring the gamma quanta yield in the observed resonances. The research is conducted on the beams of pulsed resonance neutron source IREN of FLNP. A cylindrical multisection detector with liquid scintillator is used as a detector of gamma quanta. NRCA has a number of advantages: non destructiveness, practically absent induced activity, principle possibility of investigation of samples of any shape and size, sensitivity to isotope composition of the sample. This makes it an efficient tool for investigation of archaeological artefacts, cultural heritage objects. Such investigations were carried out in collaboration with Institute of Archaeology Russian Academy of Sciences for the ancient coins from the Phanagoria's treasure. The main part of a treasure (more than two thirds) is formed by staters of Reskuporid V (3 century AD). These coins are of special interest for studying of economic climate and inflationary processes which are followed by degradation of coinage alloy of staters. Preliminary result of the investigation of the element and isotope composition of the coins will be presented in the work.

# Production and use of bronze spirals in the 2nd Millennium BC Carpathian Basin

## <u>Géza Szabó</u><sup>1,\*</sup>, Viktória Kiss<sup>2</sup>

<sup>1</sup> Wosinsky Museum, Szekszárd, Hungary

<sup>2</sup> Institute of Archaeology, Research Centre for Humanities, Hungarian Academy of Sciences

\* kaladeaa@gmail.com

The discovery of the gigantic bronze spiral at Abaújdevecser (North-eastern Hungary) in 2002 startled Hungarian archaeology: the 43 cm long, 4 cm thick, 12 kg object was absolutely unprecedented that time. Due to some other fortunate 'discoveries' latterly, fragments of similarly large bronze spirals has come to the forefront of archaeological research in the last two years. In the presentation we collect information regarding either several similarly large bronze spirals that recently came to light from depots or their normal size versions discovered from burial contexts. The presentation focus on the chronology, distribution, production, context of use and deposition of the spiral arm and ankle rings, with special attention to the so-called 'megaspirals'.

## Neutron characterization of ancient and modern textiles

## Massimo Rogante<sup>1,\*</sup>, László Rosta<sup>2</sup>

<sup>1</sup> Rogante Engineering Office, Italy

<sup>2</sup> Research Centre for Physics, Hungarian Academy of Sciences, Budapest, Hungary

\* main@roganteengineering.it

This review article is related to multi-technique approaches and complementary techniques adopted to investigate various metal archaeological objects of the Marche Region, Italy, belonging to different contexts. In particular, prompt gamma activation analysis (PGAA), time-of-flight neutron diffraction (TOF-ND), neutron radiography (NR), small angle neutron scattering (SANS) and particle induced X-ray emission (PIXE) were successfully employed, obtaining new information on material microstructure and origin of the investigated objects. The obtained results contribute to the identification, valorisation and evaluation of conservation posed by these Cultural Heritage (CH) artefacts, addressing diverse real case studies and including different art-historical and archaeological questions (e.g., manufacturing methodologies and dating). The considered advanced physical characterization techniques, finally, supply a major help in increasing scientific examinations and diffusion of CH findings.

P-08

# PIXE investigation of ancient linen fabrics dated from Old Kingdom to Ptolemaic ages (2200-300 B.C.)

Massimo Rogante<sup>1,\*</sup>, Imre Kovács<sup>2</sup>, Zoltán Szőkefalvi-Nagy<sup>2</sup>

<sup>1</sup> Rogante Engineering Office, Italy

<sup>2</sup> Research Centre for Physics, Hungarian Academy of Sciences, Budapest, Hungary

\* main@roganteengineering.it

External milli-beam particle induced X-ray emission spectroscopy (PIXE) has been used as non-destructive technique to investigate ancient linen fabrics from pre-dynastic and Ptolemaic ages belonging to the archaeological collections of the Egyptian Museum of Turin and to the Civic Archaeological Museum of Bologna, Italy. Nine linen fabric samples dated from Old Kingdom to Ptolemaic ages (2200-300 B.C.) were investigated, as well as 5 modern linen fabric samples, added for comparison. The primary goal of this work has been to advance the correct material description of the fabrics providing scientific data for further and more comprehensive comparative analyses. PIXE has provided quantitative analyses for the major components (Si, Cl, K, Ca and Fe) and minor or trace elements as P, Ti and Mn, supplying information on the near-surface elemental composition complementary to the nano-structural data obtained from SANS analyses. The achieved results contribute to comprehend the structural basis for the chemical properties of the considered material, as well as to set up a classification according to the chemical composition, allowing in general a better understanding of the studied linen cloths.

# Application of Prompt Gamma Activation Analysis to Provenance Study of the Korean Obsidian Artefacts

<u>Yong-joo Jwa<sup>1,\*</sup></u>, Ildikó Harsányi<sup>2</sup>, Seonbok Yi<sup>3</sup>, Mi-Eyn Jin<sup>1</sup>, Zsolt Kasztovszky<sup>2</sup>, Gwanq-Min Sun<sup>4</sup>

<sup>1</sup> Gyeongsang National University

<sup>2</sup> Center for Energy Research, Hungarian Academy of Science, Budapest, Hungary

<sup>3</sup> Seoul National University

<sup>4</sup> Korea Atomic Energy Research Institute

\* jwayj@gnu.ac.kr, tungma@snu.ac.kr

Provenance study of prehistoric obsidian artefacts provides a clue to understanding various aspects of the ancient culture in consideration. So far, estimation of the source of the obsidians found in archaeological context has usually been made by analyzing their geochemical characteristics. Geochemical information has been acquired by such analytical methods as pXRF, PIXE, ICP, ICP-MS, etc. However, as far as obsidian sourcing is concerned, there occur two serious problems in geochemical interpretation of the data obtained by these methods [1]. One is from the quality of the data (i.e., accuracy and precision) for some analytical methods while the other has something to do with the multivariate analysis applied to the interpretation of the data. Also, we reported that in obsidian there exist lots of microlites which were produced from quenching of acidic magma under disequilibrium condition [2]. It seems likely to be a critical factor in the geochemical analysis for obsidian how many microlites are distributed and what kinds of them are occurred in obsidian. To avoid the side effect of microlites in geochemical consideration of obsidian source, it is necessary to get bulk data and use it for discussion.

The PGAA method is proved to be a successful geochemical bulk analysis for obsidian samples, based on the major and on some trace elements [3, 4]. We analyzed some obsidian samples from Mount Baekdusan, Korea-China border and Kyushu island, Japan using the PGAA method at the Centre for Energy Research, Hungarian Academy of Sciences, Hungary. The obsidians from both areas have been known for the contrasting sources of the prehistoric obsidian artefacts in Korean Peninsula. The acquired data reveal that the two kinds of obsidians are discriminated on their provenance. We also compare the data from the PGAA method with those from the other geochemical methods such as WD-XRF, EPMA, and so on.

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### P-11

## PGAA as an analyzing tool for glass archaeological samples

Bogdan Constantinescu<sup>1,\*</sup>, Daniela Stan<sup>1</sup>, Zsolt Kasztovszky<sup>2</sup>, Ildikó Harsányi<sup>2</sup>

<sup>1</sup> National Institute for Nuclear Physics and Engineering "Horia Hulubei", Bucharest, Romania

<sup>2</sup> Centre for Energy Research, Hungarian Academy of Sciences, Budapest, Hungary

Prompt Gamma Activation Analysis (PGAA) is a perfect tool to determine the bulk elemental concentrations of valuable artefacts, without the destruction of the objects. PGAA was applied at the Budapest Neutron Centre to determine the bulk elemental composition for some glass archaeological samples from Romanian museums. Glass archaeological samples were fragments of coloured Byzantine glass bracelets (XI century, found on an archaeological site on Danube border in Dobroudja) and of medieval artefacts from Bucharest area. Na (natron) and K (plant ash) contents give information on fluxes used in glass production; Al content could indicates the sand deposit, Mg content also suggests natron or plant (wood) ash flux procedure, Ca content indicates the stabilizer from glass technology. All these data give information on workshops, technologies and commercial relations existing in South-Eastern Europe. A parallel investigation using milli-PIXE was performed for colorants and pigments (some of the bracelets were painted) identification. Despite PIGE-PIXE combination is probably the best one for glass analysis, our PGAA-PIXE methods proved to be adequate complementary tools to determine many chemical elements from glass composition.

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<sup>\*</sup> bconst@nipne.ro

### P-12

## PGAA as an analyzing tool for obsidian archaeological samples

Bogdan Constantinescu<sup>1,\*</sup>, Daniela Stan<sup>1</sup>, Zsolt Kasztovszky<sup>2</sup>, Ildikó Harsányi<sup>2</sup>

<sup>1</sup> National Institute for Nuclear Physics and Engineering "Horia Hulubei", Bucharest, Romania

<sup>2</sup> Centre for Energy Research, Hungarian Academy of Sciences, Budapest, Hungary

\* bconst@nipne.ro

Prompt Gamma Activation Analysis (PGAA) is a perfect tool to determine the bulk elemental concentrations of valuable artefacts, without the destruction of the objects. PGAA was applied at the Budapest Neutron Centre to determine the bulk elemental composition for obsidian archaeological samples from Romanian museums. Archaeological obsidian items especially from Transylvania and geological samples were analyzed to determine the provenance of material used for Neolithic tools. The raw material supposed to originate mainly from Carpathian 1 and Carpathian 2 sources in the Tokaj Mountains. Reference data of previously measured geological material [1] have been used for comparison Concentrations of major components H, Na, Al, Si, K, Ca, Ti, Mn and Fe as well as the trace elements of B, Cl, Sm and Gd were used to discriminate between geological sources.

The authors would acknowledge the support from the CHARISMA and IPERION CH projects of the EC.

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# Determination of the chlorine content of archaeological iron artefacts by prompt gamma activation analysis

<u>Friedrich Wagner<sup>1,\*</sup></u>, Rupert Gebhard<sup>2</sup>, Zsolt Révay<sup>3</sup>, P. Albert<sup>2</sup>, K. Kleszcz<sup>3</sup>, H. Hess<sup>2</sup>, Petra Kudějová<sup>2</sup>, Christian Stieghorst<sup>3</sup>

<sup>1</sup> Physics Department, Technical University of Munich, Garching, Germany

<sup>2</sup> Archäologische Staatssammlung München, Germany

<sup>3</sup> Heinz Maier-Leibnitz Centre (MLZ), Technical University of Munich, Garching, Germany

\* fwagner@tum.de

Archaeological iron artefacts often suffer from severe corrosion after their excavation, and the presence of chlorine in the corrosion layer is known to play a major role in this process. Efforts to conserve such artefacts therefore often include treatments to remove the chlorine, for instance by leaching in alkaline solutions or by heating in inert atmospheres. To assess the necessity for and the efficiency of such treatments it is therefore important to determine the chlorine content of the finds before and after treatment in a nondestructive manner. A unique method for this is prompt gamma activation analysis (PGAA). We report on such experiments performed at the PGAA facility of FRM-2, which allows the investigation of rather large objects. A survey of about 50 objects excavated in Bavaria between the late 19th century and 2012 yielded Cl contents between several tens of ppm and 3000 ppm, but reveals no obvious correlation between the date or site of excavation and the chlorine content, which sheds doubt on the idea that the agricultural use of fertilizers during the past decades has contributed to the Cl content in archaeological artefacts. The chlorine is, however, very inhomogeneously distributed even in single objects. Experiments in which samples are leached or heated in different atmospheres showed that the efficiency of the Cl removal by leaching is incomplete even after long leaching times, while heating requires temperatures well above 600 °C.

## P-13

#### P-14

# Time and frequency domain analysis of pulsed cold neutron beam PGAA

Danyal Turkoglu<sup>1,\*</sup>, Rolf Zeisler<sup>1</sup>, <u>Heather Chen-Mayer<sup>1</sup></u>

#### <sup>1</sup> NIST, USA

\* danyal.turkoglu@nist.gov, chen-mayer@nist.gov

Isotopes emit characteristic prompt gamma  $(\gamma)$  rays immediately after neutron capture. In prompt gamma-ray activation analysis (PGAA), a high-resolution spectrometer measures  $\gamma$ -ray energies and intensities that are used for quantitative analysis of elemental composition of a bulk sample. In addition to the prompt  $\gamma$  rays, decay  $\gamma$  rays can be used for elemental assay.

To enhance the measurement of the relatively-weak decay  $\gamma$  rays, a beam chopper that cyclically blocks the cold neutron beam – thus eliminating the prompt  $\gamma$  rays when the beam is off – has been implemented. Unlike traditional instrumental neutron activation analysis (INAA) that transports the sample post-irradiation for counting, the stationary sample and detector configuration in chopped-beam PGAA enables measurement of very-short lived activation products with half-lives (T1/2) less than one minute. Many, possibly thousands, of chopper cycles allow for accumulation of signal. Irradiation and decay lengths, ti and td, respectively, in the beam chopper cycle are tunable parameters for maximizing the signal-to-background ratio for a half-life. The effect of Ti and Td can be determined with analytical modeling of irradiation and decay cycles.

Short-lived activation products from commonly-determined elements include: 20F, 24Na, 28Al, 38Cl, 56Mn, 46Sc, 77mSe, 80Br, 82Br, 127I, 179m1Hf, 187W, 108Ag, 110Ag, as well as a huge number of isotopes produced from fissile elements. Determinations of 77mSe and unfolding of interfering  $\gamma$  rays of Dy 515.467 keV (T1/2= 75.4 s) and Yb 514.868 keV (T1/2= 0.068 s) by their half-life have been demonstrated. The decay spectra, with lower noise baselines and fewer  $\gamma$ -ray lines, are valuable for samples producing complex  $\gamma$ -ray spectra, potentially lowering detection limits and increasing selectivity for some elements. Current developments for routine application include an optimized detector geometry, and energy and time stamps of every detected  $\gamma$  ray saved in list mode to memory.

These time-stamped, list-mode (TLIST) data present the opportunity for investigating the temporal behavior of signals and optimizing for signal-to-background ratio in particular time windows depending on the sample matrix. TLIST data are sparse when binned to a time histogram with 1 ms bins, making it necessary to combine data from many cycles together to view decay properties. However, frequency domain analysis by Fourier transform of the list mode data has shown signature peaks above the noise background at the chosen chopper operation frequency. We report preliminary results of frequency domain analyses for the 24mNa (T1/2= 0.020 s) and the Dy+Yb TLIST data. Simulation of ideal TLIST data in the time and frequency domain has been performed to deconvolve the excitation function (neutron beam irradiation) and the response function (decay emission). This technique might be useful in identifying weak signatures of short-lived radionuclides in TLIST data for chopped-beam PGAA, while further development could lead to quantitative analysis.

# Lithic material of Tata-Porhanyó investigated by promptgamma activation analysis and petrographical methods

<u>Viola T. Dobosi</u><sup>1</sup>, Katalin T. Biró<sup>1,\*</sup>, Zsolt Kasztovszky<sup>2</sup>, Boglárka Maróti<sup>2</sup>, Sándor Józsa<sup>3</sup>

<sup>1</sup> Hungarian National Museum, Budapest, Hungary

<sup>2</sup> Centre for Energy Research, Hungarian Academy of Sciences, Budapest, Hungary

<sup>3</sup> Department of Petrology and Geochemistry, Eötvös Loránd University, Budapest, Hungary

\* tbk@ace.hu

Tata-Porhanyó is one of the classical Middle Palaeolithic sites in Hungary. The recognition of the site as a palaeontological locality dates back to the 18th century. Later on, it was one of the earliest Palaeolithic excavations in Hungary (1909), published by the excavator in 1912. The site was re-discovered and excavated again by L. Vértes and his team in 1958 and 1959. The results were published in a monograph [1]. About 40 years later, new excavations were started here by V. Dobosi and J. Cseh (1995-2001), basically for cleaning the site which terminated in a National Scientific Research Fund program yielding material comparable in size and importance to the previous excavation campaigns. Currently we are working on the new monographic publication. More than 43,000 stone artefacts, among them 2935 finished tools are currently registered from the Tata-Porhanyó site. On a selected set of siliceous raw material samples (about 20 pieces), petrographical and geochemical studies were made. Besides the microscopic studies, the elemental compositions of the samples have been determined by prompt-gamma activation analysis (PGAA). The non-destructive method is suitable to differentiate between the major raw material types, on the basis of the major- and some trace elements concentrations [2]. The aim of the study was to investigate, if we can perform provenance analysis of the archaeological objects. The interpretation of the results is still in progress; showing the variety of lithic resources utilised by the Palaeolithic inhabitants of the site.

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### Erősd and related sites investigated by prompt-gamma activation analysis and petrographical methods

Sándor Sztáncsuj<sup>1,\*</sup>, Sándor Józsa<sup>2</sup>, Katalin T. Birő<sup>3</sup>, Zsolt Kasztovszky<sup>4</sup>, Ildikó Harsányi<sup>4</sup>

<sup>1</sup> Székely National Museum, Sfântu Gheorghe, Romania

<sup>2</sup> Department of Petrology and Geochemistry, Eötvös Loránd University, Budapest, Hungary

<sup>3</sup> Hungarian National Museum, Budapest, Hungary

<sup>4</sup> Centre for Energy Research, Hungarian Academy of Sciences, Budapest, Hungary

\* sztancsuj sandor@yahoo.com

Ariuşd (Erősd)-Tyiszk-hegy is a multi-layered (tell) settlement of the Copper Age, eponym site of the Ariuşd Group, the South-Western branch of the Cucuteni-Tripolye cultural complex. The investigation of lithic material from the site (including a number of 778 lithics) was recently started by the authors. Following the first results [1], petrographical and geochemical study of the lithic artefacts was continued in the framework of the CHARISMA and IPERION EU projects in the Budapest Neutron Center. 94 artefacts were subjected for analysis, provening from the sites of Ariuşd, Boroşneu Mare (Kisborosnyó)-Borzvára, Doboșeni (Székelyszáldobos)-Borvízoldal, Malnaş Băi (Málnásfürdő)-Füvenvestető and Olteni (Oltszem)- Vármegye [2]. Furthermore, the investigations included also the analysis of some comparative raw material pieces received from the Museum of the Eastern Carpathians, deposited in the Lithotheca Collection of the Hungarian National Museum. This poster will present results of PGA analyses and petrographical thin section studies made on the Erősd material and related sites.

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## Monte Carlo Simulation of Compton Imaging Tomography for Prompt Gamma Activation Analysis

<u>Benjamin Riley</u><sup>1</sup>, Danyal Turkoglu<sup>2</sup>, Heather Chen-Mayer<sup>2,\*</sup>, Haijian Chen<sup>3</sup>, Jerimy Polf<sup>8</sup>

<sup>1</sup> University of Kentucky

 $^{2}$  NIST

<sup>3</sup> University of Maryland School of Medicine

\* chen-mayer@nist.gov

Prompt gamma activation analysis (PGAA) is an elemental analysis technique using gamma rays emitted upon neutron capture by nuclei contained within a sample. It provides quantitative information for the elemental composition while maintaining the sample integrity. The primary limitation of PGAA, however, is that it is a bulk technique giving average concentrations, without spatial information. This work explores Compton Imaging tomography of the prompt gamma rays, with the goal of developing a new multidimensional method that can provide both quantitative and spatial information for the elemental distributions within a sample. We report preliminary work on computer simulation of Compton imaging of prompt gamma emissions and reconstruction to localize the source origin. Simulation was conducted using the Monte Carlo code GEANT4 on a series of simple systems to evaluate the feasibility of applying Compton imaging tomography to the element of interest at a scale of interest for elemental analysis. The reconstructions were performed using back projection and a statistical method. A stack of alternating disks of Ti and H (in the form of water) of 2 cm in diameter and 0.5 mm thick was placed perpendicularly to a set of two detector planes made with CZT material. Prompt gamma rays produced by a beam of incident neutrons were tracked, with those scattered from the first detector plane and absorbed by the second recorded. The GEANT4 simulated energy histograms were windowed by the main Ti prompt gamma peak at 1381 keV and H peak at 2223 keV to produce energy and position information as input to the reconstruction code to recover spatial information at the source origin. The Ti and H emission images were recovered and can be shown separately or as a composite. The result from the back-projection method and the statistical reconstruction method are compared. The 5.5-mm spacing of the disks is fully resolvable. In addition, it is worthwhile noting that the statistical reconstruction was about 50 times faster than the backprojection method for this example. The statistical reconstruction was done in full 3D, whereas the back-projection was performed slice by slice. Further refinement of the model to reflect experimental setup will be carried out. The result of this study will facilitate improvement of the reconstruction process for the data obtained earlier on the NCNR PGAA instrument.

#### Inverse Iteration Algorithm for Neutron Depth Profiling

Cong Shi<sup>1,\*</sup>, Caijin Xiao<sup>2</sup>. Yonggang Yao<sup>2,\*</sup>, Bangfa Ni<sup>2</sup>, Xiangchun Jin<sup>2,\*</sup>

<sup>1</sup> Chengdu University of Technology

<sup>2</sup> China Institute of Atomic Energy

\* cjshaw@ciae.ac.cn, yaoyonggang1985@126.com, winnie\_jin\_cn@163.com

This work presents a Monte Carlo code to get response spectrum of ions for the Neutron Depth Profiling (NDP) technique that simulates the behaviour of ions transmitted through sample matrix and generates the energy spectrum for a specified detector. Based on the method of Monte Carlo simulation and probabilistic inversion for neutron depth profiling, the depth concentration distribution of element B in Si matrix material is calculated. According to NDP experimental equipment at CARR, energy spectra of standard sample SRM2137 are simulated using MCNP and Geant4 software, and the concentration-depth diagram of elements in SRM2137 is achieved adopting inverse iteration method through MATLAB software. It shows that the inverse iteration calculation in NDP is feasible.

#### P-19

# NeXT-Grenoble, a novel Neutron and X-ray Tomography characterisation facility

Duncan Atkins<sup>1,\*</sup>, A. Tengattini<sup>2</sup>, B. Giroud<sup>1</sup>, E. Ando<sup>2</sup>, J. Beaucour<sup>1</sup>, G. Viggiani<sup>2</sup>

<sup>1</sup> Institut Laue-Langevin, Grenoble, France

<sup>2</sup> Université Grenoble Alpes, CNRS, Grenoble INP, Institute of Engineering Univ., France

\* atkins@ill.fr

NeXT-Grenoble is a Neutron and x-ray imaging facility developed and now running in Grenoble, born from a collaboration between the University Grenoble Alpes and the European Neutron Source the Institut Laue-Langevin (ILL). This instrument will ultimately combine the uniquely intense neutron flux offered by the ILL with laboratory x-ray tomography in order to obtain simultaneous bi-modal imaging, thus allowing the complementarity of these methods to be exploited. As a preliminary step, a medium resolution neutron radiography/tomography instrument has been implemented in 2016 allowing cultural heritage, academic and industrial users to perform successful material characterisations in a wide range of applications such as porous/geomaterials, hydrogen fuel cells, medical prosthetics and archaeological artefacts. In several cases tomographic acquisitions were obtained during in-situ hydro-thermo-chemo-mechanical tests, for example using bespoke high confinement triaxial or tensile loading rigs, or imposing hightemperature conditions, running multi-phase permeability tests as well as inducing electrochemical reactions. The planned addition in 2017/2018 of an integrated x-ray tomograph, as well as the implementation of a newly developed high-resolution neutron detector, is expected to push boundaries still further and permit the visualisation of interactions between complex phenomena in areas such as geomaterial permeability, hydraulic fracturing and concrete behaviour under extreme conditions, as well as complicated cultural heritage artefacts containing organic and in-organic matter.

# Neutron texture analyses of Bronze Age swords from the Alpine region: benchmarking neutron diffraction against laboratory

#### ${\it methods}$

Marianne Mödlinger<sup>1</sup>, Evelyne Godfrey<sup>2</sup>, <u>Winfried Kockelmann<sup>3,\*</sup></u>

 $^1$ Excellence-Cluster TOPOI, Deutsches Archäologisches Institut, Eurasien Abteilung, Berlin, Germany

 $^2$  Uffington Heritage Watch

<sup>3</sup> STFC-Rutherford Appleton Laboratory

\* winfried.kockelmann@stfc.ac.uk

Bronze Age swords represent the peak of craftsmanship in terms of prehistoric weapon production. Following an extensive study on the manufacture and usage of Bronze Age swords from the Alpine region [1], we have used time-of-flight performed neutron diffraction on the General Materials Diffractometer (GEM) at the ISIS Facility, UK, to analyse ten swords in order to highlight the advantages and drawbacks of the diffraction analysis. The phase compositions and crystallographic textures of the copper tin alloys reveal information about the mechanical and thermal treatment at different parts of the sword blades. Knowledge about different working intensities of cold working in various areas of the sword blades can provide information about the usage of the swords as mainly thrusting or as cut-and-thrust used weapons. The neutron diffraction results are compared to results from bronze replica samples and to X-ray fluorescence and metallographic analyses applied to the same objects.

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## Determination of firing temperature of clay pottery wares by Small Angle Neutron Scattering

Katalin Bajnok<sup>1,\*</sup>, Adél Len<sup>1,\*</sup>, János Füzi<sup>1</sup>, László Rosta<sup>1</sup>

<sup>1</sup> Wigner research Centre for Physics, Hungarian Academy of Sciences, Budapest, Hungary

\* bajnok.katalin@winger.mta.hu; len.adel@winger.mta.hu

Small Angle Neutron Scattering measurements on archaeological ceramic samples have been performed at the Yellow Submarine and FSANS instruments at Budapest Neutron Centre. The main objective of the present study was to deepen our knowledge about the limits and usefulness of the SANS method regarding ceramic samples, therefore a series of reference samples and also Late Roman / Early Medieval archaeological samples and from the late Roman fortification of Keszthely (Hungary) were also measured.

Tempered and non-tempered control samples with known firing temperatures were prepared, and measured by SANS. The intensity versus scattering vector curves obtained from the detector neutron counts showed power-law type scattering, what was attributed to the scattering from the crystallites, pores, voids, amorphous inclusions. The influence of the sample thickness on the power-law exponent was also checked.

A correlation between the firing temperature and the SANS curves was set up, showing that this method – as complementary measurements among e.g. X-ray and Neutron Diffraction, Scanning Electron Microprobe, X-ray Fluorescence etc. – is suitable for analysing the high temperature nanostructural behaviour of ceramics.

LIST OF AUTHORS

ABRAMZON, Mikhail	Nosov Magnitogorsk State Technical University;	P-06
	abramzon-m@mail.ru	
ANDREY, Laura	Site et Musée Romains Avenches; laura.andrey@vd.ch	O-03
ATKINS, Duncan	Institut Laue Langevin; atkins@ill.fr	<u>P-19</u> , O-24
AVDEEV, Maxim	ACNS-ANSTO; maxim.avdeev@ansto.gov.au	O-08
BACKS, Alexander	Heinz Maier-Leibnitz Zentrum (FRM II);	K-03
	alexander.backs@frm2.tum.de	
BAJNOK, Katalin	Wigner Research Centre for Physics, Hungarian Academy of	P-05, <u>P-21</u>
	Sciences; bajnok.katalin@wigner.mta.hu	
BAUSENWEIN, Dominik	Heinz Maier-Leibnitz Zentrum (MLZ), Garching, Germany;	P-03
	dominik.bausenwein@frm2.tum.de	
BAZHAZHINA, Nina	Frank Laboratory of Neutron Physics, Joint Institute for Nuclear	P-06
	Research; bazhazhina@mail.ru	
BEAUCOUR, Jérôme	Institut Laue Langevin; beaucour@ill.fr	O-24, P-19
BELGYA, Tamás	Centre for Energy Research, Hungarian Academy of Sciences;	I-03
,,	belgya.tamas@energia.mta.hu	
BEUING, Raphael	Bayerisches Nationalmuseum, Munich, Germany;	P-03
blonto, itapiaci	raphael.beuing@bnm.mwn.de	1 00
BIRÓ, Katalin T.	Hungarian National Museum; tbk@ace.hu	I-01, P-15, P-16
BÜCHERL, Thomas	Technische Universität München, ZTWB Radiochemie München	<u>1-01</u> , 1-15, 1-10 O-20
BUCHERL, Thomas		0-20
MALEDA A C	RCM; thomas.buecherl@tum.de	0.00.0.00
VALERA, Antonio C.	Era Arqueologia, Núcleo de Investigação Arqueológica;	O-06, O-26
	antoniovalera@era-arqueologia.pt	
CALLIGARO, Thomas	Centre de Recherche et de Restauration des Musées de France;	<u>K-04</u>
	thomas.calligaro@culture.gouv.fr	
CANTINI, Francesco	Universitá di Firenze; francesco.cantini.fi@gmail.com	O-05, O-09
CHEN, Haijian	University of Maryland School of Medicine;	P-17
	haijianchen@som.umaryland.edu	
CHEN-MAYER, Heather	NIST; chen-mayer@nist.gov	<u>O-25, P-14</u> , P-17
COCO, Davide	Università di Roma "La Sapienza"; cocodavide@gmail.com	O-05
CONSTANTINESCU, Bogdan	National Institute for Nuclear Physics and Engineering;	<u>P-11, P-12</u>
	bconst@nipne.ro	
CREANGE, Sara	Rijksmuseum; s.creange@rijksmuseum.nl	<u>O-09</u>
DAVIS, Joel	Nuclear Materials-ANSTO; joel.davis@ansto.gov.au	O-08
DIAS, M. Isabel	Centro de Ciências e Tecnologias Nucleares, CTN, IST; U. Lisboa;	O-06, <u>O-26</u>
	isadias@ctn.tecnico.ulisboa.pt	
DOBOSI, Viola T.	Hungarian National Museum; t.dobosi.viola@hnm.hu	P-15
DUVAUCHELLE, Anika	Site et Musée Romains Avenches; anika.duvauchelle@vd.ch	O-03
EDGE, David	The Wallace Collection; david.edge@wallacecollection.org	0-05, I-04
ERDMANN, Robert	Rijksmuseum, University of Amsterdam, Radboud University	O-09
	Nijmegen; r.erdmann@rijksmuseum.nl	
LINDELOF, Poul Erik	University of Copenhagen, Copenhagen, Denmark; lindelof@it.dk	O-02
FEDRIGO, Anna	STFC; anna.fedrigo@fi.isc.cnr.it	O-02
FÜZI, János	Wigner Research Centre for Physics, Hungarian Academy of	P-21
,	Sciences; fuzi.janos@wigner.mta.hu	
GAIT, John	Fitch Laboratory, British School at Athens;	P-05
Gitti, soim	john gait@hotmail.com	1_00
GEBHARD, Rupert	Archäologische Staatssammlung München;	O-13, I-05, P-13
GEDITAILD, Rupert	rupert.gebhard@extern.lrz-muenchen.de	0-13, 1-05, 1-15
GILLIOZ, Sandra	Site et Musée Romains Avenches; sandra.gillioz@vd.ch	O-03
GODFREY, Evelyne	Uffington Heritage Watch; egodfrey@heritage-watch.com	P-20
GRAZZI, Francesco	Consiglio Nazionale delle Ricerche - Istituto dei Sistemi Complessi;	· · · · ·
HADGING THE	francesco.grazzi@isc.cnr.it	04, <u>P-03</u>
HARSÁNYI, Ildikó	Centre for Energy Research, Hungarian Academy of Sciences;	O-06, O-26, P-10,
	harsanyi.ildiko@energia.mta.hu	P-11, P-12, P-16
HAVENITH, Andreas	Aachen Institute for Nuclear Training GmbH;	P-02
	havenith@nuclear-training.de	0.1-
HIRATA, Yuho	Nagoya University; hirata.yuuho@h.mbox.nagoya-u.ac.jp	0-17
HORI, Genki	Nagoya University; hori.genki@d.mbox.nagoya-u.ac.jp	O-17, <u>P-01</u>
JIN, Mi-Eyn	Gyeongsang National University; jin4860228@gmail.com	P-10
JIN, Xiangchun	China Institute of Atomic Energy; winnie_jin_cn@163.com	P-18

JOLIE, Jan	Institute of Nuclear Physics of the University of Cologne;	O-19
	jolie@ikp.uni-koeln.de	
JÓZSA, Sándor	Department of Petrology and Geochemistry, Eötvös Loránd University; sandor-jozsa@caesar.elte.hu	P-15, P-16
JWA, Yong-Joo	Gyeongsang National University; jwayj@gnu.ac.kr	<u>P-10</u>
KAESTNER, Anders	Paul Scherrer Institut, Neutron Imaging and Activation Group,	O-05
	Villigen, Switzerland; anders.kaestner@psi.ch	
KAI, Tetsuya	Japan Atomic Energy Agency; tetsuya.kai@j-parc.jp	O-17
KÁLI, György	Wigner Research Centre for Physics, Hungarian Academy of	O-16
	Sciences; kali.gyorgy@wigner.mta.hu	
KARDJILOV, Nikolay	Helmholtz-Zentrum-Berlin; kardjilov@helmholtz-berlin.de	O-09, <u>I-04</u> , O-24
KASZTOVSZKY, Zsolt	Centre for Energy Research, Hungarian Academy of Sciences;	I-01, O-06, I-03, O-
	kasztovszky.zsolt@energia.mta.hu	12, O-26, P-10, P-
VETTLED Labor	DW/TH Asshar University Nulleons Enternment and	11, P-12, P-15, P-16 P-02
KETTLER, John	RWTH Aachen University - Nukleare Entsorgung und Techniktransfer; kettler@net.rwth-aachen.de	F-02
KIM, JinMan	Korea Atomic Energy Research Institute; kjm5@kaeri.re.kr	P-04
KIM, Jongyul Kim	Korea Atomic Energy Research Institute; kjongyul@kaeri.re.kr	P-04
KIM, TaeJoo	Korea Atomic Energy Research Institute; tj@kaeri.re.kr	P-04
KIS, Zoltán	Centre for Energy Research, Hungarian Academy of Sciences;	I-02, I-03, P-05
,	kis.zoltan@energia.mta.hu	,,
KISS, Viktória	Institute of Archaeology, research Centre for the Humanities,	P-07
,	HAS; kiss.viktoria@btk.mta.hu	
KIYANAGI, Yoshiaki	Nagoya University; kiyanagi@phi.phys.nagoya-u.ac.jp	<u>P-01</u> , O-17, P-01
KLUGE, Eschly Jan	Institute of Nuclear Physics at the University of Cologne;	<u>O-19</u>
	ekluge@ikp.uni-koeln.de	
KOCKELMANN, Winfried	STFC-Rutherford Appleton Laboratory;	<u>O-22</u> , P-20
	winfried.kockelmann@stfc.ac.uk	
KOVÁCS, Imre	Wigner Research Centre for Physics, Hungarian Academy	O-06, O-26, P-09
KODUTNED I I	of Sciences, Budapest, Hungary; kovacs.imre@wigner.mta.hu	D 00
KREUTNER, Joachim	Bayerisches Nationalmuseum, Munich, Germany; ioochim kreutner@hum mum do	P-03
KRIEG, Myriam	joachim.kreutner@bnm.mwn.de Site et Musée Romains Avenches SMRA; myriam.krieg@vd.ch	O-03
KUDĚJOVÁ, Petra	Heinz Maier-Leibnitz Zentrum (MLZ), Technical University of	O-13, O-14, P-13
ROBLOOVII, Felia	Munich; petra.kudejova@frm2.tum.de	0 10, 0 14, 1 10
LAA <sup>§</sup> , Michael	University Duisburg/Essen; michael.laass@gmx.de	O-16
LAMANNA, Jacob	National Institute of Standards and Technology;	O-15
3 -	jacob.lamanna@nist.gov	
LANDSBERGER, Sheldon	University of Texas; s.landsberger@mail.utexas.edu	0-11
LEE, Changhee	Korea Atomic Energy Research Institute; leech@kaeri.re.kr	P-04
LEFMANN, Kim	Nanoscience Centre, Niels Bohr Institute, University of	O-02
	Copenhagen, Copenhagen, Denmark; lefmann@nbi.ku.dk	
LEHMANN, Eberhard	Paul Scherrer Institut; eberhard.lehmann@psi.ch	<u>K-01</u> , O-03, <u>O-15</u>
LEN, Adel	Wigner Research Centre for Physics, Hungarian Academy of	O-27, P-21
	Sciences; len.adel@wigner.mta.hu	
LIVINGSTON, Richard	University of Maryland; rliving1@umd.edu	O-25
LUZIN, Vladimir	ACNS-ANSTO, Sydney; vladimir.luzin@ansto.gov.au	O-28
GAN, Weimin M.	German Engineering Materials Science Centre at MLZ, Helmholtz-	O-05
	Zentrum Geesthacht, Garching, Germany; weimin.gan@hzg.de	
MAKOWSKA, Malgorzata	FRM II, MLZ; malgorzata.makowska@frm2.tum.de	O-20
MANNES, David	Paul Scherrer Institut; david.mannes@psi.ch	O-03, <u>O-15</u>
MAREEV, Yuri	Frank Laboratory of Neutron Physics, Joint Institute for Nuclear	P-06
witten v, run	Research; mareev@nf.jinr.ru	1 00
MARIANNE, Moedlinger	Excellence-Cluster TOPOI, Deutsches Archäologisches Institut,	P-20
. , 6	Eurasien Abteilung, Berlin, Germany;	
	marianne.moedlinger@gmail.com	
MARIN, Julio	Centro Atomico Bariloche - Comision Nacional de Energia	<u>O-10</u>
	Atomica; jhmarin@cab.cnea.gov.ar	
MARÓTI, Boglárka	MTA Centre for Energy Research;	O-06, I-03, P-15
	maroti.boglarka@energia.mta.hu	
MAZHEN, Saltanat	State University "Dubna"; saltanat5.06.95@mail.ru	P-06

MIHALY, Judit	Research Centre for Natural Sciences, Hungarian Academy of	O-26
	Sciences; mihaly.judith@ttk.mta.hu	0.07
MILCZAREK, Jacek	Narodowe Centrum Badan Jadrowych;	O-27
MINNITI, Triestino	jacek.milczarek@ncbj.gov.pl STFC-Rutherford Appleton Laboratory;	O-22
MININII, Illestillo	triestino.minniti@stfc.ac.uk	0-22
MÜHLBAUER, Martin	Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität	O-20
	München; martin.muehlbauer@frm2.tum.de	0 20
NAZAROV, Kuanysh	Joint Instinute for Nuclear Research; quanysh@list.ru	O-04
NEUWIRTH, Tobias	Heinz Maier-Leibnitz Zentrum (FRM II);	K-03
*	tobias.neuwirth@frm2.tum.de	
NI, Bangfa	China Institute of Atomic Energy; bfni@ciae.ac.cn	P-18
O'CONNOR, Ariel	Smithsonian American Art Museum; oconnora@si.edu	O-25
OIKAWA, Kenichi	Japan Atomic Energy Agency; kenichi.oikawa@j-parc.jp	<u>O-17</u>
OLSEN, Scott	ACNS-ANSTO, Sydney; scott.olsen@ansto.gov.au	O-08
PAPPAS, Catherine	TUDelft / fac. Appl. Sci. / dep. RadiationScienceTechn.;	O-18
	c.pappas@tudelft.nl	
PENTZ, Peter	The National Museum of Denmark, Copenhagen, Denmark;	O-02
	peteremil.pentz@natmus.dk	
KUHNEN, Hans Peter	Institut für Altertumswissenschaften, Johannes Gutenberg	O-13
	University Mainz; kuhnen@uni-mainz.de	
PLOMP, Jeroen	TUDelft / fac. Appl. Sci. / dep. RadiationScienceTechnology;	O-18
	j.plomp@tudelft.nl	
POLF, Jerimy	University of Maryland School of Medicine;	P-17
	jpolf@som.umaryland.edu	0.00.0.00
PRUDÊNCIO, M. Isabel	Centro de Ciências e Tecnologias Nucleares, CTN, IST; U. Lisboa;	<u>O-06</u> , O-26
WILLIAMS Alon D	iprudenc@ctn.tecnico.ulisboa.pt Welless Collection, eler millions@mellessesllestion, em	0.02 0.05 1.04
WILLIAMS, Alan R.	Wallace Collection; alan.williams@wallacecollection.org	O-02, O-05, I-04, P-03
REHREN, Thilo	Cyprus Institute, STARC; th.rehren@ucl.ac.uk	K-02
REISENHOFER, Florian	RWTH Aachen University - Nuclear Engineering and Technology	P-02
REDEENIOF ER, Florian	Transfer; reisenhofer@net.rwth-aachen.de	1-02
RÉVAY, Zsolt	Maier-Leibnitz Center, Technical University of Munich;	O-13, O-14, O-19,
101 111, 25010	zsolt.revay@frm2.tum.de	P-13
RILEY, Benjamin	University of Kentucky; bkri223@g.uky.edu	P-17
ROGANTE, Massimo	Rogante Engineering Office; main@roganteengineering.it	<u>O-27, P-08, P-09</u>
ROSTA, László	Wigner Research Centre for Physics, Hungarian Academy of	O-21, O-27, P-08,
	Sciences; rosta.laszlo@wigner.mta.hu	P-21
SALVEMINI, Filomena	ACNS-ANSTO; filomens@ansto.gov.au	O-05, <u>O-08</u>
SÁNCHEZ, Fernando	Centro Atómico Bariloche, CNEA; sanchezf@cab.cnea.gov.ar	0-10
SAPRYKINA, Irina	Institute of Archaeology Russian Academy of Sciences;	P-06
,	dolmen200@mail.ru	
SCHERILLO, Antonella	STFC; antonella.scherillo@stfc.ac.uk	O-05, <u>O-23</u>
SCHILLINGER, Burkhard	Heinz Maier-Leibnitz Zentrum (FRM II), Technische Universität	O-02, O-05, <u>O-16</u> ,
	München; burkhard.schillinger@frm2.tum.de	<u>K-03</u> , P-03
SCHMUTZLER, Britta	State Academy of Art and Design Stuttgart;	O-13, <u>O-14</u>
	britta.schmutzler@gmx.de	
SCHULZ, Michael	Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität	O-20, K-03
	München; michael.schulz@frm2.tum.de	
SCIARRETTA, Francesca	IUAV University of Venice; scifra@iuav.it	<u>I-02</u>
SEDYSHEV, Pavel	Frank Laboratory of Neutron Physics, Joint Institute for Nuclear	P-06
	Research; sedyshev@nf.jinr.ru	
SEIFERT, Marc	Heinz Maier-Leibnitz Zentrum (FRM II);	K-03
	marc.seifert@frm2.tum.de	0.00
SHEEDY, Kenneth	Australian Centre for Ancient Numismatic Studies-Macquarie	O-08
CHIL C.	University, Sydney; ken.sheedy@mq.edu.au	<b>D</b> 10
SHI, Cong	Chengdu University of Technology; shicong1992@gmail.com	P-18 O-17
SHINOHARA, Takenao SHVETSOV, Velow	Japan Atomic Energy Agency; takenao.shinohara@j-parc.jp Frank Laboratory of Nautron Physics Joint Institute for Nuclear	
SHVETSOV, Valery	Frank Laboratory of Neutron Physics Joint Institute for Nuclear Research; shv@nf.jinr.ru	P-06
STAN, Daniela	National Institute for Nuclear Physics and Engineering "Horia	P-11, P-12
STILL, Duncia	Hulubei", Bucharest, Romania; daniela@nipne.ro	1 11, 1-12
	, Paonacov, romana, annonesinpheno	

STARNINI, Elisabetta	School of Humanistic Sciences, Department of Historical Studies, University of Torino, Italy: estarnini@hotmail.com	O-12
STIEGHORST, Christian	TUM   Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM	O-13, O-14, O-19,
	II); christian.stieghorst@frm2.tum.de	P-13
STROBL, Markus	markus.strobl@esss.se	O-02
SUN, Gwang-Min	Korea Atomic Energy Research Institute; gmsun@kaeri.re.kr	P-10
SÜRMELIHINDI, Gül	Institute of Geosciences, Johannes Gutenberg University Mainz,	O-13
	Germany; surmelhi@uni-mainz.de	
SZABÓ, Géza	Wosinsky Museum, Szekszárd, Hungary; kaladeaa@gmail.com	P-07
SZAKMÁNY, György	Department of Petrology and Geochemistry, Eötvös Loránd	O-12
	University, Budapest, Hungary; gyorgy.szakmany@geology.elte.hu	
SZENTMIKLÓSI, László	Centre for Energy Research, Hungarian Academy of Sciences,	I-02, <u>I-03</u>
	Budapest, Hungary; szentmiklosi.laszlo@energia.mta.hu	
SZŐKEFALVI-NAGY, Zoltán	Research Centre for Physics, Hungarian Academy of Sciences,	O-06, O-26, P-09
	Budapest, Hungary; szokefalvi-nagy@wigner.mta0.hu	
SZTÁNCSUJ, Sándor	sztancsuj_sandor@yahoo.com	<u>P-16</u>
TANAKA, Manako	Showa Women's University, Japan; ma-tanaka@swu.ac.jp	P-01
TARBAY, Gábor János	Hungarian National Museum, Archaeological Department,	<u>O-07</u>
	Prehistoric Collection, Budapest, Hungary;	
	tarbayjgabor@gmail.com	
TURKOGLU, Danyal	NIST, USA; danyal.turkoglu@nist.gov	<u>P-14</u> , P-17
URITANI, Akira	Nagoya University, Japan; uritani@energy.nagoya-u.ac.jp	O-17, P-01
VÁCZI, Benjámin	Department of Petrology and Geochemistry, Eötvös Loránd	<u>O-12</u>
	University, Budapest, Hungary; vbeni9305@gmail.com	
VAN EIJCK, Lambert	TUDelft / fac. Appl. Sci. / dep. RadiationScienceTechnology;	<u>O-18</u>
	l.vaneijck@tudelft.nl	
WAGNER, Friedrich	fwagner@tum.de	O-13, <u>I-05</u> , <u>P-13</u>
WATANABE, Kenichi	Nagoya University, Japan; k-watanabe@energy.nagoya-u.ac.jp	O-17, P-01
PASSCHIER, Cornelis Willem	Institute of Geosciences, Johannes Gutenberg University Mainz,	O-13
	Germany; cpasschi@uni-mainz.de	
XIAO, Caijin	China Institute of Atomic Energy; cjshaw@ciae.ac.cn	P-18
YAMAZAKI, Atsushi	Nagoya University, Japan; a-yamazaki@energy.nagoya-u.ac.jp	P-01
YAO, Yonggang	China Institute of Atomic Energy; yaoyonggang1985@126.com	P-18
YELLIN, Joseph	The Hebrew University of Jerusalem, Israel; joe.yellin@gmail.com	0-11 D.00
YERGASHOV, Almat	Joint Institute for Nuclear Research, Dubna, Russia;	<u>P-06</u>
YI, Seonbok	almat_16.93@mail.ru Seoul National University; tungma@snu.ac.kr	P-10
ZEISLER, Rolf	••• ••	P-14
ZEYNALOV, Shakir	NIST, USA; rolf.zeisler@nist.gov Frank Laboratory of Neutron Physics, Joint Institute for Nuclear	P-06
ZETIMLOV, SHAKII	Research, Dubna, Russia; zeinal@nf.jinr.ru	r-00
ZHOU, Zhou	TUDelft / fac. Appl. Sci. / dep. RadiationScienceTechnology;	O-18
ZHOU, Zhou	z.zhou-1@tudelft.nl	0-16
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