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# Neutron imaging for cultural heritage at ANSTO

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**Abstract** Since commencing operation in 2014, the Dingo neutron imaging facility has expanded capabilities of investigating materials of cultural heritage significance at the Australian Nuclear Science and Technology Organisation. This paper offers an overview of the diverse range of artefacts investigated on the beamline during the last decade. A selection of case studies established through national and international collaborations showcases how neutron imaging can provide university and museum users with a powerful analytical tool for non-invasive real-space evaluation of a variety of ancient artefacts with properties complementary to other nuclear methods.

#### **1** Introduction

Neutron methods are a well-established scientific analytical tool for the investigation of a broad range of materials and phenomena. These techniques exploit the peculiar interaction mechanism of the neutron with the atomic nuclei to gain information on the material properties of a sample from macroscopic to nanoscopic scale [1, 2].

Although the fields of application are broad, the ability to survey the bulk of complex system in a non-invasive and non-destructive way has established their use in the field of cultural heritage and conservation sciences [3].

In fact, a major challenge in the investigation of materials of cultural, historical, and archaeological significance is the need of non-invasive procedures that can characterise the object while preserving their unique value and integrity for transmission to future generations. Although laboratory-based techniques can provide valuable information, in some cases these conventional methods present limitations in terms of penetration depth and representativeness.

Among the suit of neutron methods, neutron imaging has found broad applicability in the field of cultural heritage science [4, 5]. This analytical method can provide a real space representation of an object or phenomenon in one or more dimension by measuring the transmitted intensity of the probing beam that decreases exponentially along the path length through the sample, depending on the material elemental composition and density. Neutron imaging can give a unique insight, at a macroscopic scale, about the composition, manufacture, and preservation of a variety of materials that is complementary to other nuclear methods [6].

The range of objects that can be investigated is quite diverse: arms and armours, musical instruments, numismatics, votive items, potteries, stone materials, paintings, wood artefacts, meteorites. The method can be also a useful tool for the assessment of the status of conservation and the evaluation of new conservation approaches [3, 7].

Alongside the classic methods of neutron radiography and tomography, advanced neutron imaging techniques have been also exploited in the field of cultural heritage research, i.e. Bragg-edge neutron transmission [8–10], neutron resonance transmission imaging [11], prompt gamma-ray activation imaging [7], bimodal imaging [4], autoradiography [3].

It should be pointed out that in archaeometry studies, neutron imaging is often complement by neutron diffraction methods to probe matter beyond the macroscopic bulk features and provided a more robust insight by looking at a smaller scale: the crystalline structure of the metal [12–17]. Neutron diffraction can provide qualitative and quantitative data about phase composition, crystal and magnetic structures of different constituent phases, (residual) microstrains and macrostrains, and crystallographic texture [18]. This information can be used to read the imprint left at different level of the crystalline structure by distinctive manufacturing processes or by other actions that occurred during the life cycle of an item—i.e. mechanical and thermal impact, reuse, burial, and conservation works.

Finally, for the sake of completeness, it should be mentioned that neutron activation analysis [19] and neutron small-angle scattering [20] also offer a robust complementary tool for bulk isotopic composition and investigation at a nanoscale, respectively [7].

The Australian Nuclear Science Organisation (ANSTO) operates a range of cutting-edge nuclear techniques for the non-invasive characterization of cultural heritage materials, supported by dedicated research staff who are experts in the field. The synergy of

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**Fig. 1** Layout of Dingo beamline. On the left, the in-pile collimator is facing the OPAL research reactor through the thermal neutrons beam port HB2. Upstream, a selector wheel/shutter unit enables to select the beam size, followed by a sapphire filter for reduction of fast neutrons background. The tertiary shutter regulates access to the sample area via a safety interlock system. A slits system is installed on the wall of the instrument enclosure to further adjust the beam size and reduce irradiation of the sample area. The neutron beam is transported from the in-pile collimator to the sample position through two sections of helium-filled flight tubes. At the sample position, an *xyz*-translation table can host air-bearing rotation stages of different accuracy and load capacity. The detector box is fixed on a motorised *z*-translation stage. Both sample stage and detector are mounted on a rail system. A permanent beam stop is located behind the detector. The whole beamline is surrounded by biological shielding and accessible via a sliding door

nuclear techniques enables correlative and complementary analysis to answer a diversity of questions over a wide range of materials without the need for invasive procedures.

In 2015, a strategic scientific research project on cultural heritage was formally established to promote access and encourage utilization by cultural institutions and researchers to the suite of nuclear methods available across ANSTO's campuses [21].

The focus of this paper is on the use of conventional polychromatic neutron tomography over last decade of collaborative research experience, bridging science and humanities, on the Dingo beamline at ANSTO.

# 2 Instrument layout

Dingo is a neutron imaging instrument (Fig. 1) fed by the thermal neutrons located on the beam port HB2 at the OPAL research reactor, featuring a thermal neutron spectrum [22]. The instrument can be operated into two different modes, high-resolution beam and a high-intensity beam, depending on the chosen pin-hole positioned at in-pile collimator. These correspond to a geometrical collimator of the instrument of L/D (where L is the distance between the collimator to the image plane, and D the diameter of the collimator) of 1000 and 500, respectively. The neutrons are transported through a direct flight path from the pin-hole to the sample position, where the maximum beam size spreads out to about  $200 \times 200 \text{ mm}^2$ .

Approximately 2.5 m away from the in-pile collimator the selector wheel/shutter unit enables to select one beam at the time while shielding the other. The selector wheel offers different apertures to reduce beam size down to  $50 \times 50 \text{ mm}^2$ .

Upstream of the selector wheel, a sapphire filter composed of several super optical quality crystals with the [001] axis parallel to the incoming beam and of 90 mm  $\times$  90 mm  $\times$  30 mm in size is framed on a motorised translation stage. The filter can be driven in and out of the beam for reduction of fast neutrons background.

A slits system is installed on the wall of the instrument enclosure. Mounted on aluminium frame, the four motorised slits made of  $B_4C$  can be translated to further adjust the beam size and reduce irradiation of the sample area.

The neutron beam is transported through helium-filled flight tubes. The first section is located between the sapphire filter unit and the tertiary shutter. The second section is located between the slit assembly and sample position. This section can be adjusted in length depending on the sample position.

At the sample position, an *xyz*-translation stage with a loading capacity of up to 500 kg in radiography setup (without rotation stage) and up to 250 kg in tomography mode (with rotation stage) allows sample positioning within a range of > 500 mm in the *x*-and *y*-directions and 400 mm in the *z*-direction. A set of high-accuracy air-bearing rotation stages provides different load capacity.

The detector box consists of an active-pixel sensor camera mounted out of the beam at an angle of 90 degrees and looking at a scintillation screen via a mirror positioned at 45 degrees. Over the course of the last 10 year in operation, a range of different cameras has been used and replaced due to radiation damage. At the time of writing, the cooled camera ZWO ASI2600MM is in operation. The scintillation screen (<sup>6</sup>LiF/ZnS (Ag) or Gd<sub>2</sub>O<sub>2</sub>S: Tb) converts neutron radiation into visible light that is focused to the camera sensor. Two different lenses, with 50 mm and 100 mm fixed focal lengths, are available to tune the field of view up to full beam size. The detector assembly is fixed on a motorised *z*-translation stage featuring a vertical range of approximately 100 mm to alternatively align with the high-intensity or high-resolution beam.

Behind the detector at end of the instrument is a permanent beam stop design in a layered structure of  $B_4C$ , borated polyethylene, and steel shielding mix. The whole instrument is surrounded by heavy concreate and roofed with standard concrete slabs with the interior cladded with borated polyethylene to maximize shielding ability for gamma and neutron radiation.

# 3 Case studies

3.1 Rediscovery the ancient technology of arms and armor: a multi-techniques approach

Metallurgy has played a substantial role in the rise of modern civilisation. The techniques of metal working developed in antiquity encompass an understanding or intuition of the properties of materials. Especially the design and manufacture of arms and armours embody the highest advancement of knowledge and sophistication in material technology developed within a multi-dimensional cultural contest in the past.

A striking example is embodied by the Samurai sword. The traditional curved steel blade dominated the battlefield of Japan over thousands of years and nowadays is mostly regarded as work of art, combining unrivalled aesthetic and functionality. Its signature features were obtained through a unique manufacturing process that consisted in applying a specific thermomechanical treatment to a forge-welded body composed of iron and steel laminae. The crafting methods were never documented, and the necessary information was orally transmitted from the master to his most skilled pupils and varied across different schools and traditions [23].

In a pilot study originated in collaboration with the museum of applied arts and sciences (MAAS) in Sydney (AU) and later expanded to the collection of the Queen Victoria art gallery and museum in Launceston (AU), neutron methods were exploited to develop a multi-technique analytical protocol to non-destructively characterize the laminated structure of a set of 25 Samurai's swords.

Neutron tomography was applied to characterize bulk structural and morphological features of the blades (i.e. amount, distribution and shape of defects, porosity, and non-metallic inclusions). Complementary neutron powder diffraction full pattern analysis enabled to non-invasively quantify metal and non-metal phases composing the samples that relate to the smelting and smithing procedures. On the other hand, neutron diffraction stress measurement determined residual stress as an imprint of the manufacturing process (Fig. 1) [3, 21].

The data were first collected on samples of well-known origin, period, and authorship to create a reference database on the main manufacturing methods developed by Japanese swordsmiths. These benchmark data were cross-matched with results obtained from a group of *mumei* (no-signature) blades in the attempt to attribute their manufacturing tradition basing on qualitative and quantitative data rather than stylistic criteria.

Analysis and comparison of all collected results allowed identifying differences and commonalities in the production process. Certainly, as in the case of any other forensic research methods, a statistical approach based on a broader set of samples of different origins is required in order to bring more reliability and robustness to the conclusions made on the basis of this neutron-based non-destructive analytical method (Fig. 2) [21, 24, 25].

Recently, the same non-invasive multi-methodological approach based on neutron techniques has been employed to examine rare swords from the Kingdom of Dahomey, dating from the mid-nineteenth century. The investigation aimed to determine whether the swords were made in Dahomey (West Africa) or in Europe. Neutron tomography aided by powder diffraction full pattern analysis, and diffraction residual stress measurement was applied to pinpoint peculiarities indicative of either European or indigenous manufacturing processes. Our findings have significant implications for our understanding of iron use and production in this region and period, as well as into specific local sword forging techniques, offering valuable new data on a time period and place with limited primary sources for scholars [26].

#### Sadatsugu Bitchu. 1346 - 1370



**Fig. 2** Example of neutron data on sword attributed to Sadatsugu Bitchu, 1346–1370, collection of the museum of applied arts and sciences (MAAS) in Sydney (AU). On the top: photographic image of the sample. In the middle: longitudinal cross sections along the tomographic reconstruction. The bar indicates the correspondence between grey tone and neutron attenuation coefficient. At the bottom: a generic mesh in the transverse cross section of the sword for neutron diffraction mapping and a real example of the longitudinal stress,  $d_0$  and peak width maps. Images adapted from references [7, 24, 25]

### 3.2 Under the rust: characterisation of archaeological findings

Metal artefacts excavated in archaeological site are often heterogeneous not only in their stylistic features but also in structure and composition.

Although the severe state of degradation, especially metal artefacts, can be found, neutron tomography can extract useful archaeometric information. This has been the case of the investigation of ancient totally corroded ferrous artefacts from the early Iron Age site of Saruq al-Hadid, Dubai. Neutron tomography enabled to investigate features associated to the manufacturing process of the object from surface to bulk: irregularities on the surface induced by plastic deformation, i.e. hammering; variation in type and distribution of corrosion products in relation to secondary recycling activities; structural inhomogeneities such as pierced holes, incisions, and ex-welding lines. The most interesting aspect identified in every artefact has been the ex-welding lines. Corrosion preferentially evolves along these lines, and those can be used for comparing the manufacture of different ferrous artefacts (Fig. 3). The sensitivity of neutron tomography was demonstrated by complementary invasive investigation of selected ferrous artefacts via analyses of remnant carburized areas by optical microscopy, and analyses of slag inclusions by scanning electron microscope coupled with energy-dispersive spectroscopy (SEM–EDS). These findings helped better understand the socio-technological contest of the early iron technologies developed in the past societies of the ancient near east [27–29].

Neutron tomography can also contribute to elucidate the famed excellent corrosion resistance of ancient Indian iron artefacts, still not fully explained by scholar. The iron manufactured using the traditional iron making process followed by Indian tribes known as 'Agaria' was investigated using a range of correlative microscopic, spectroscopic, diffraction, and tomographic techniques to postulate the hidden mechanisms of superlative corrosion resistance. Neutron tomography helped to evaluate size and distribution of pores as well as to determine the extent depth of the corrosion products. This information was contextualised to the manufacture, ingredients involved in Agaria iron making process, and post-metal treatment using metal-working operation called hot hammering (forging). It was postulated that consolidation of pores, expected removal of inclusions, and thick passive corrosion product layer formation have supported the iron in attaining the excellent corrosion resistance [30].

3.3 Investigating the authenticity of votive items: a synergy of nuclear methods

Neutron and X-ray feature complementary contrast mechanism are in interaction with matter. This factor can be exploited when investigating composite artefacts. An exemplary application is investigating the content of Egyptian votive mummies.

Animal mummies make up the largest category of objects we have from ancient Egypt and yet, until recently, are one of the least researched. Votive mummies are a feature of the first millennium B.C., gaining great popularity in the Late Period and increasing even more in the Ptolemaic and Roman Periods [31, 32].

As a non-invasive scientific method, tomographic investigation of mummies from ancient Egypt has been used since 1896 and provided information on mummification techniques, age and state of health of the individual at the time of death as well as any post-mortem damage to the mummy [33]. Medical computed tomography (CT) is commonly employed for these studies, but proved

Fig. 3 On the top, comparative investigation of corrosion products by neutron tomography (a) and optical microscopy (b). Magnetite/maghmite (Mt) and goethite (Gth) are highlighted. At the bottom, structural inhomogeneities evidenced by neutron tomography



to have limited capabilities in differentiating between skeletal and desiccated soft tissues due to the limitation imposed by inbuilt safety mechanisms concerning radiation dose in human CT.

Neutron tomography can offer a complementary tool to study soft tissues, embalming substance, and bandage of the mummies non-invasively due to high sensitivity to organic materials. The high penetration power of the neutron enables to investigate the mummified bundle when encased into ceramic or metallic containers that is not always achievable with X-ray tomography [34, 35].

Two ibis mummies from the Egyptian antiquities collection of the Nicholson Museum in Sydney were investigated to clarify their content and enable further the scholarly discussion on the purpose of ancient Egyptian fake mummies. The study also aimed to slightly narrowing the date range of production as Ptolemy VI Philometer (180–168 BC), during an investigation into corrupt practices of the ancient manufacturers of ibis mummies, decreed that, from that time, only one bird per bundle was permitted.

While X-ray is indisputably the analytical method of choice to investigate the contest of such material in a non-invasive way, neutron tomography demonstrated some advantages. Neutrons offered a better contrast to detect the presence of feathers, to map the distribution of the resinous balm, and to identify individual layers of the linen bandage. On the other hand, the penetration power was quite limited in the thickest portion of the item, thus affecting the image quality (Fig. 4).

The investigation provided the museum with valuable information about the content and composition of the mummified packages. The condition of the artefacts was also ascertained; this will greatly assist in the museum's ongoing care and conservation of each of the collection items.

Neutron tomography can be a valuable forensic tool to gather indirect proof about the authenticity of ancient objects, especially in case of materials for which a direct dating method is yet to be discovered, i.e. bronzes. In this study [37], a bronze wine vessel attributed to Shang Dynasty (1600–1046 B.C.) in China, part of the East Asian Collection of the museum of applied arts and sciences in Sydney (Australia), was investigated. A synergic combination of nuclear techniques was applied to gain a better insight into the structural and compositional features of the artefact as proof of authenticity.

The risk of excessive sample activation induced by long exposure to the neutron beam and to determine the bulk elemental composition was first assessed via gamma spectrometry assurance.

The bulk structural and morphological features of the vase were then reconstructed via neutron tomography. The evaluation of size, shape, and distribution of porosities as well as the thickness of the metal wall was found consistent with the piece-mould casting technology typical of Shand dynasty craftsman in China.

Finally, particle-induced X-ray emission (PIXE) analysis determined the metal to be a ternary (copper-tin-leaded) alloy, another feature consistent with the traditional manufacture of bronze vase of that time, thus suggesting a genuine artefact [37].

# 3.4 Numismatic: understanding early coinage

In the sixth century B.C., the manufacture of coins differentiated across mints in mainland Greece and in the Greek colonies in Southern Italy. In Greece coins were produced from a piece of cast metal of predetermined weight (a 'blank' or flan) that was struck between two engraved bronze dies [38, 39]. Colonies in Magna Graecia, instead, developed a unique minting technique [40]. Today known as incuse coinage, these issues feature the image of the reverse type rendered as a 'negative' or 'incuse' image sunk into the flan on the obverse. The study of this technique is part of a collaborative project initiated by the Australian Centre for Ancient



Fig. 4 Votive animal mummie NM62.585 from the Egyptian antiquities collection of the Nicholson Museum in Sydney (AU). **a** Photographic images of the wrapped ibises; sections across the **b** X-ray and **c** neutron tomographic reconstructions and corresponding detailed view of the regions of interested **d** and **e** are shown. The scale bar at the bottom of each tomographic section indicates the colour code for the attenuation coefficient. Please note that the X-ray CT was acquired partially. The provided cross sections highlight the external wrapping enveloping the remains of the ibises, visible in the core area, and the difference in contrast that neutron and X-rays can provide for such components. **f** The photographic image of the museum item is overlapped to the segmentation based on neutron and X-ray data. The animal remains, distribution of the embalming material in the bandage, feathers, and the stomach content are rendered as separated volume in **g**, **h**, **i**, and **j**. Images adapted from reference [36]

Numismatic Studies (ACANS) at Macquarie University and the Australian Nuclear Science and Technology Organisation (ANSTO) in Sydney since 2014. The research program aims to investigate the distinct technique of minting developed by Greek city states in Southern Italy during the sixth and fifth centuries B.C. Metallurgical research based on neutron tomography, texture measurement, full-pattern powder diffraction, and SEM–EDS was combined to numismatic and historical studies for the systematic analysis of silver coins from the focus area and period [41–43].

Neutron tomography demonstrated to be a valuable non-invasive analytical tool to extract critical information from numismatic material (Fig. 5). The morphology, porosity, inclusions, and the presence of composite structures of coins can be reconstructed with acceptable accuracy and linked to the manufacturing process. Neutron tomography was able to identify (unexpected) plated coins, due to the difference in attention coefficients between the material typically composing the core (copper) and the wrapping lamina (silver). Individual structural components can be virtually separated, and their volume estimated, as well as the thickness of the external plating, could be mapped in three dimensions. Additionally, variation of the neutron attenuation coefficients can be quantified to identify presence of corrosion products and inform about the status of conservation of the coins [44].



**Fig. 5** a Photographic image of the obverse of coin 07GS527, Metapontum, 510-470 B.C. from the ACANS collection at Macquarie University (AU); **b** the position of the planes cropping the reconstructed volume of the coin is shown together with the corresponding cross sections. The scale bar at the bottom indicates the colour code for the attenuation coefficient; **c** identified structural components are separated and rendered in different colour as indicated by the legend at the bottom; **d** the three-dimensional map shows the distribution of porosities. A scale bar at the bottom indicates the coding colour volume; **e** the thickness of the silver plating is reported in false colour for both obverse and revere of the coin. A scale bar indicates the coding colour thickness. Image adapted from reference [44]

# 4 Conclusions

The neutron imaging instrument Dingo has significantly enhanced the research capabilities of the neutron science facilities at ANSTO in the field of cultural heritage. The presented selection of case studies has demonstrated effective application of polychromatic neutron imaging for non-destructive real-space evaluation of the bulk structure of a variety of ancient artefacts, especially metals, at a macroscopic scale. The technique can provide users with a powerful analytical tool to investigate different aspect of cultural heritage materials: morphology, bulk structure, qualitative composition, features indicative of manufacture (i.e. defects, porosities, welding, etc.), and inform about the status of conservation of a range of different artefacts.

The process of beamtime request and allocation, samples packaging and transportation, experiment execution, and data evaluation may require additional effort and time compared to portable and laboratory-based techniques. However, these extra challenges are compensated by the higher sensitivity, the accuracy, and the non-invasiveness of the method. Additionally, long-term involvement and collaboration between users' groups and beamline scientists has facilitated engagement with the cultural heritage research community, streamlined procedures, and success rate.

Finally, research outcome can be measured through peer-reviewed scientific publications but can also reach the public. Studies from Dingo have been showcased in newspapers [45] and more recently displayed in museum exhibitions [46], thus capitalising on the public-relations benefits and raising ANSTO's reputation in the field of cultural heritage research.

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