Chapter 1
Probing Our Heritage with Neutrons—One Successful Story

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Abstract Probing our heritage reminds us of a variety of issues in Heritage Science: from the correct determination of historical and cultural time-frame of artefacts, to their location and method of production, to the choice of best treatments and environmental conditions for their restoration. A large variety of chemical, physical and microstructural techniques are employed by Museums and art experts to characterize objects of cultural significance. Most of these methods are invasive and probes like X-rays and charged particles have limited penetration power. Neutrons penetrate thick layers, depending on their energy, without substantial attenuation, a quality which makes them ideal to study and visualize the interior (bulk) properties of materials in a totally non-destructive and non-invasive way. The high sensitivity to specific light elements (e.g. H) is an additional special property of the neutron probe. Neutron techniques are increasingly used for the quantitative, non-invasive analysis of many aspects of cultural heritage preservation in a broad sense: museum collections, artefacts, books, manuscripts, musical instruments, archaeological findings.

1.1 Historical Background

> The experimental results were very difficult to explain on the hypothesis that the beryllium radiation was a quantum radiation, but followed immediately if it were supposed that the radiation consisted of particles of mass nearly equal to that of a proton and with no net charge, or neutron. (…) When such neutrons pass through matter they suffer occasionally close collisions with the atomic nuclei and so give rise to the recoil atoms which are observed.

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The citation is from Chadwick's 1932 breakthrough article 'Existence of a Neutron' (Chadwick 1932) where he showed that the physical properties of radiation obtained by hitting a beryllium target with α-particles could be explained by invoking a neutral particle of one atomic mass unit: the neutron.

Already in 1920, Rutherford had postulated the existence of a new neutral and massive particle in the nucleus of atoms. Such assumption originated from the observation of a disparity between an element's number of protons, represented by the atomic number, and its atomic mass. Such disparity appears as a mass excess compared to the mass due to the already known proton particles. Rutherford called these uncharged particles *neutrons*. In 1931, Bothe and Becker found that if alpha particle radiation from polonium fell on beryllium, boron or lithium, a penetrating radiation was produced. In fact, when they hit a beryllium target with alpha particles presumably electrical neutral radiation that could penetrate 200 mm of lead was emitted. They assumed the neutral radiation to be high-energy gamma rays.

In the same year, several similar experiments were carried out in Europe. One experiment in particular caught Chadwick's attention: Joliot and Curie discovered that, when a beam of this yet unknown radiation hits a substance rich in protons (in their case paraffin), protons were emitted and easily detected by a Geiger counter. Joliot and Curie believed the radiation hitting the paraffin target must have been high-energy gamma photons. In 1932 Chadwick repeated their experiments with the goal of searching for a neutral, uncharged particle with about the same mass as a proton. He realized that the emitted radiation could not be gamma rays by measuring the emitted proton energy and discovering that to generate such an effect it should have been in the 50 MeV order of magnitude, therefore much larger than gamma ray energy (typically few MeV).

> When a sheet of paraffin wax about 2 mm thick was interposed in the path of the radiation just in front of the counter, the number of deflections recorded by the oscillograph increased markedly. This increase was due to particles ejected from the paraffin wax so as to pass into the counter (Chadwick 1932).

Chadwick also tried different targets, including helium, nitrogen, and lithium, which helped him determine that the mass of the new particle was just slightly larger than the mass of the proton. In 1935 Chadwick won the Nobel Prize in Physics for the discovery of neutrons. Scientists soon realized that the newly discovered neutron, as an uncharged but massive particle, could be used to probe other nuclei. In 1934 Fermi (Nobel prize for Physics in 1938) obtained induced radioactivity in high atomic number elements by hitting them with neutrons. In 1938 Hahn (Nobel prize for Chemistry in 1945), Meitner and Strassmann discovered nuclear fission, the splitting of uranium nuclei into light elements, induced by neutron bombardment. The discovery of nuclear fission was the first step towards the development nuclear energy (Fig. 1.1).

After the discovery of the neutron, it became clear that the nucleus contains protons and neutrons. The nucleus is a compact body with dimension of about 10^{-15} m. The free neutron is unstable and cannot be stored. Therefore, it must be made free by certain nuclear reactions to be available for experiments.

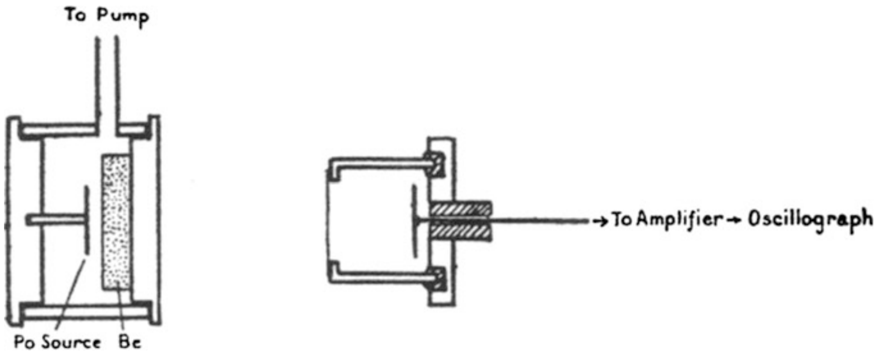

Fig. 1.1 Chadwick device

Research focused on ways to actually produce and use them in a series of different applications. Between the 1940s and the 1970s three kinds of methods were developed and applied for the production of neutrons: *radioactive decay* (for example in the spontaneous fission that occurs in very heavy chemical elements with atomic mass unit greater than 92), *nuclear reactions* (such as natural alpha and gamma bombardment of nuclides) and *induced nuclear fission* (as it occurs in nuclear reactors). Chicago Pile-1 (1942) was the first artificial nuclear reactor that went critical. It was built in the framework of the Manhattan Project, and carried out by the Metallurgical Laboratory at the University of Chicago under the supervision of Enrico Fermi. Chicago-Pile was made of a large amount of graphite and uranium, with control rods of cadmium, indium, and silver. Soon after, reactor technologies rapidly evolved becoming the most productive neutron sources and were used for power production and research as well as for military applications during World War II.

In the 1970s research started to focus on the spallation process, where neutrons are produced through disintegration of target nuclei (Carpenter 1986). Earlier research activity addressed to produce spallation facilities had been carried out between the years 1940s–1960s (Carpenter 1986). Lawrence's proposed 'cyclotron' in 1940 was able to produce neutrons by hitting a beryllium target, paving the way to the use of accelerators for neutron production. The term 'spallation' was coined by Sullivan and Seaborg in 1959 (Harvey 1959). During the 1960s there was an increased interest for spallation neutron production as an alternative to other techniques (Tunnicliffe et al. 1966). In 1974 the ZING-P neutron source prototype (developed by Jack Carpenter at Argonne National Laboratory) demonstrated the feasibility of a spallation neutron source (Carpenter 1986).

With the discovery of neutrons and the development of methods and technology in neutron production, the new research area of neutron science started to develop and neutron scattering and absorption (capture) rapidly became routine techniques for the non-invasive and non-destructive study of materials in the bulk.

Key research to establish the basic principles of neutron diffraction techniques was carried out in the 1940s by Wollan and Shull at Oak Ridge National Laboratory (formerly Clinton Laboratories USA) (Johnson and Schaffer 2008). In 1955 Brockhouse confirmed the quantum theory of solids through the first measurements of phonons using a prototype triple-axis neutron spectrometer. These were the seminal experiments for which, in 1994, Shull and Brookhouse were granted the Nobel Prize in Physics. The ‘special’ way neutrons interact with matter allows to ‘see’ where atoms are and how atoms move, making the neutron a unique probe for the studying the structure and dynamics of materials at the nanoscale.

1.2 Neutron Techniques

One can use nuclear reactions induced in a specimen by a particle beam (usually a neutron beam) to render certain constitutive elements of a material radioactive, permitting their analysis by identification of the radioactive decay products by detecting radiation emitted during such reactions or by measuring the portion of a neutron beam that traverses an object without any interaction.

1.2.1 Activation Analysis

The most common technique to create a nuclear reaction is the neutron activation, discovered in 1936 by Hevesy and Levi. They discovered that certain rare earth elements, contained in the irradiated samples, became radioactive as a consequence of neutron beam exposure (Zeisler et al. 2003). The neutron flux creates unstable nuclei in the specimen by the process of neutron capture. The resulting nuclear transition produce γ-ray emission, K-capture and other processes which then enables identification of the isotopic species present and of the elements. This phenomenon was then employed in Neutron Activation Analysis (NAA) for the identification of major, minor, trace and rare elements (Zeisler 2003).

The first experiment where NAA was applied to cultural heritage dated back to 1960 when Emeleus and Simpson published the first results of a study of a Roman pottery fragments analyzed through NAA and γ-ray spectrometry. The results of this experiment allowed them to distinguish between production factories (Emeleus & Simpson 1960). The experiment was carried out using the thermal neutron beam from the Bepo reactor in Harwell (UK). In the following decades, a series of research studies applying thermal neutrons to cultural heritage were carried out. NAA was applied to a large variety of samples such as fossil bones (Eisenbarth and

Hille 1977) where nitrogen and fluorine contents were measured relative to main constituents of the inorganic bone material (principally phosphorus and calcium). Measurements gave determination of the human remain dating in a non-destructive way. In 1987 Mommsen et al. studied ancient potteries from archaeological excavations through a new methodology where neutron-induced radiation in low energy region (10–135 keV) was measured with a high-resolution semiconductor detector. NAA is largely applied to the study of trace elements to determine provenance and manufacturing techniques. The principle of the NAA technique is presented in detail in Chap. 10.

1.2.2 Prompt Gamma Activation Analysis

Another technique that uses neutron absorption is Prompt Gamma Activation Analysis (PGAA) (Molnar 2004). This nuclear analytical technique utilizes the characteristic prompt γ-ray spectrum measured during the irradiation of the sample for the non-destructive determination of elemental and/or isotopic composition of samples.

PGAA technique is best suited for the investigations of materials containing light elements such as H, S, P and K.

A PGAA measurement campaign was carried out on so called ‘black boxes’ in 2008. The aim of this study, performed as ANCIENT CHARM European project activity, was to identify strengths and weaknesses of neutron techniques applied to the study of archaeological objects and to develop a best practice for a combined use of neutron analysis methods (such us Prompt Gamma Activation Analysis, Time of Flight Neutron Diffraction and neutron tomography) for different combinations of materials. For this purpose, 17 samples were used, known as *black boxes*, consisting of closed cubes containing geometrical arrangements of materials such as metals, minerals, ceramics, and organic matter. Result on a black box is shown in Fig. 1.2.

Nr.	Nominal comp.	PGAA results
1 + 2	Ag in talc	H, Si, Cl, Mn, Fe, Cu, Ag
3	Sand	H, B, Na, Al, Si, Cl, K, Ti, Mn, Fe, Cu
4	Iron grit	H, B, Al, Cl, Mn, Fe
5	Salt	Na, Al, Cl, Cu
6	Al sheet	–
7	Cu sheets	H, Na, Al, Si, Cl, Mn, Fe, Cu, Zn, Ag

The principle of the PGAA technique is presented in detail in Chap. 11. Examples of application of the PGAA method can be found in Chap. 6.

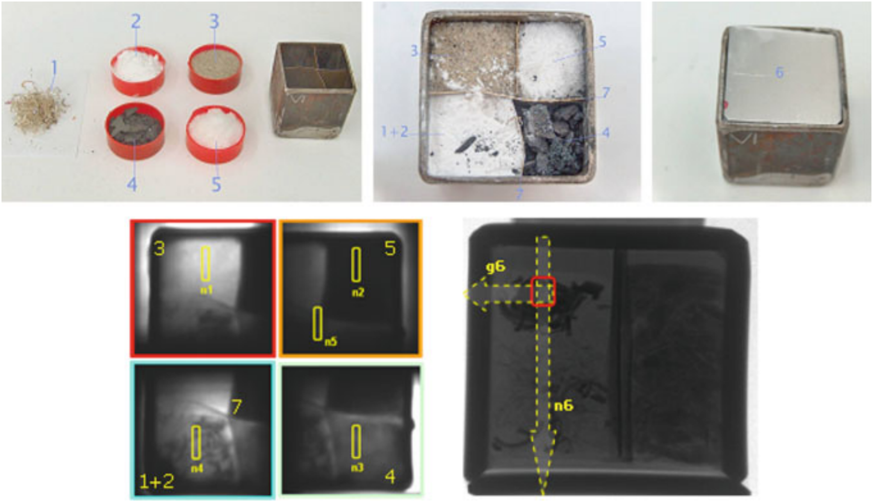

Fig. 1.2 *Iron box*: the black box internal feature—segments divided with two crossed sheets. With PGAA, the fiber-like material is identified as Ag chippings in regions (1 + 2). Predominantly Si was found in section 3 and Fe in section 4, whereas Na and Cl (in a molar ratio 1:1) in section 5. At the crossing point of the sheets the presence of Cu was confirmed

1.2.3 Neutron Resonance Analysis Methods

These methods are based on resonance absorption of neutrons with epithermal energies (Postma and Schillebeeckx 2005): absorption (capture and scattering) of neutrons by nuclei as a function of neutron energy shows sharp peaks (resonances) specific for elements (isotopes). The analysis of resonances allows the identification and quantification of elements and isotopes within an object in a non-destructive manner. This is the basis of Neutron Resonance Capture Analysis (NRCA), in which resonances are observed by detecting the prompt γ-radiation emitted directly after neutron capture as a function of the neutron energy. In NRCA, neutrons with energies corresponding to energy levels of the compound nucleus are captured preferentially. Resonances identify the nuclides present in the material whereas the areas under the resonance peaks provide information about their relative amount. They can be visualized in the neutron capture spectrum as a function of neutron energy. Another method based on neutron resonances, Neutron Resonance Transmission Analysis (NRTA), is the transmission of a neutron beam through a sample. Resonances are seen as dips in the transmission curve as function of neutron energy. A schematic representation of NRCA and NRTA measurements is shown in Fig. 1.3.

NRCA is particularly effective for samples composed of copper and its alloys because of the significant sensitivities for copper itself and for a range of elements such as Ag, As, Sb, Sn, and Zn (that are likely to be found in Cu alloys), and for Fe, which is also relevant for archaeological applications. It has to be stressed that activation in

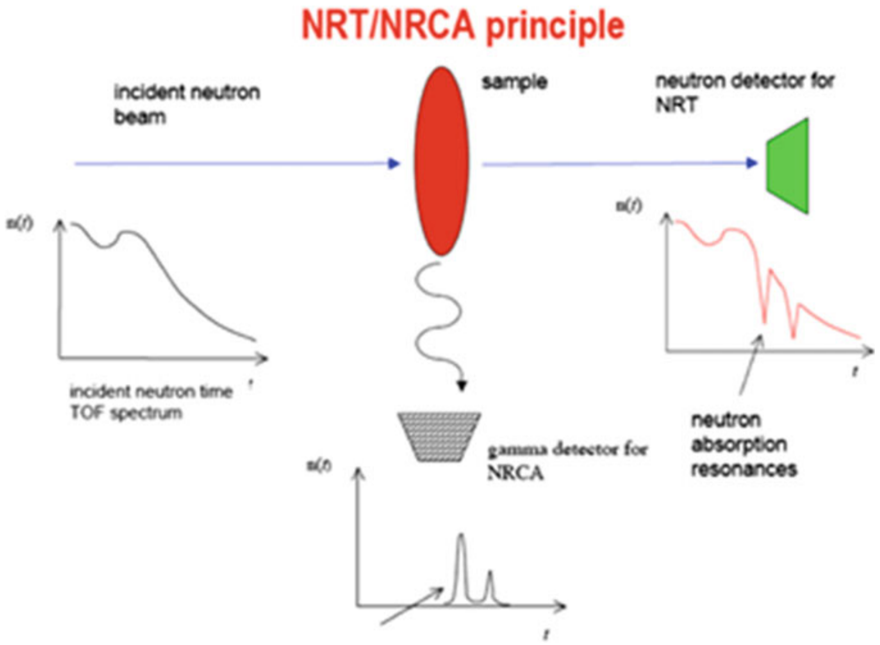

Fig. 1.3 Scheme of a set-up for Neutron Resonance Transmission Analysis (NRTA) and Neutron Resonance Capture Analysis (NRCA)

materials is mainly due to thermal neutrons, which can be removed from the beam with the aid of a Cd-filter. Hence activation is low after an NRCA run and normally negligible after a short waiting period. Thus artefacts can be returned to owners quickly. The principles of NRCA and NRTA are presented in detail in Chap. 12.

A technique beyond NRCA and NRTA is Neutron Resonance Transmission Imaging (NRTI). The potential of NRTI was studied at e.g. the J-PARC facility (Tremsin et al. 2014) and within the Ancient Charm project at the spallation neutron source ISIS of the Rutherford Appleton Laboratory (Gorini et al. 2007). It relies on the use of position sensitive neutron detectors. NRTI has been applied to produce 2D (Perelli et al. 2011) and 3D elemental contrast figures of archaeological samples (Festa et al. 2015) at ISIS.

1.2.4 Time Resolved Prompt Gamma Activation Analysis

Bi-parametric time-energy acquisition Prompt Gamma Activation Analysis (T-PGAA) is a technique that combines prompt gamma-ray analysis (PGA) and neutron resonance analysis (NRA). It was recently applied for the study of cultural heritage artefacts at ISIS (Festa et al. 2013a, b) and to study a meteorite sample at

J-PARC (Toh et al. 2014) facility. Time-resolved Prompt Gamma Activation Analysis consists in the measurement of gamma energy spectrum induced by radiative capture as a function of incident neutron Time of Flight (Festa et al. 2013a, b; Miceli et al. 2013, 2014).

T-PGAA enhances the capabilities of both the NRCA and PGAA techniques, providing a more general sensitivity for elements (Festa et al. 2013a, b, 2016; Toh et al. 2014) (Fig. 1.4).

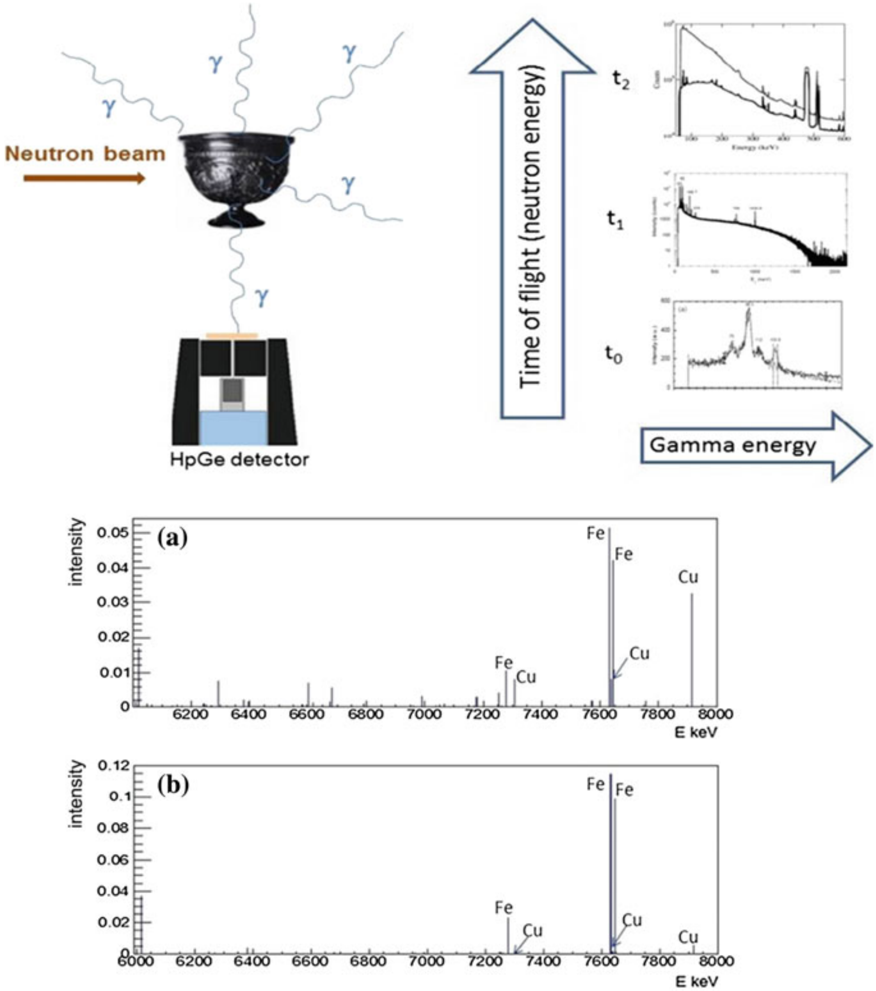

Fig. 1.4 Layout of the designed experiment recording gamma spectra at different neutron energy frames on the top. On the bottom, simulated gamma spectra are reported for (**a**) neutron resonance energy range from 200 to 900 eV (**b**) total neutron energy range from 0 to 1.2 keV

1.2.5 Neutron Diffraction

In 2001 first test measurements using a Time of Flight Neutron Diffraction on archaeological samples were performed on pottery fragments at the spallation source ISIS, (UK) (Kockelmann et al. 2001). Neutron Diffraction (ND) is based on elastic scattering of thermal neutrons by periodic, long-range ordered (crystalline) or non-periodic, short-range ordered (glass) arrangements of atoms. Nowadays ND is routinely used for the phase and structure analysis, texture analysis, microstructure analysis and residual stress analysis of artefacts with a minimum volume of 1 × 1 × 0.5 mm. An important issue for curators is the assessment of the condition of material concerning production techniques. In this context through ND characterisation of an artefact one gains insight about the materials and technologies used by the craftsmen.

The principle of the ND technique is presented in detail in Chap. 9 and examples of ND studies can be found in Chap. 4.

1.2.6 Neutron Radiography and Tomography

Neutron radiography (NR) is a real space imaging method based on capture and scattering of thermal and cold neutrons (Domanus 1992). A neutron beam impinging onto any heterogeneous object is differently transmitted depending on thickness, density, chemical composition and total cross section of the material along the line of sight. Through NR one can obtain information on the internal view of materials with a spatial resolution down to 100 microns. By recording the transmitted beam it is possible to reconstruct the internal feature of the studied object. The attenuation of a neutron beam passing through objects provides contrast for different elements. Contrast is also achieved by variation of neutrons energies. Additional techniques are the Phase Contrast Radiography, based on neutron refraction (Lewis and Schutz 1937) and Neutron Tomography, providing 3D visual information about the internal structure of the sample (Kardjilov et al. 2004). Example for neutron tomographic investigation of Osiris bronze statue is shown in Fig. 1.5 (Agresti et al. 2015).

1.2.7 Developing of Neutron Techniques for Cultural Heritage Research in the Frame of the "Ancient Charm" Project

The Ancient Charm ("Analysis by Neutron resonant Capture Imaging and other Emerging Neutron Techniques: new Cultural Heritage and Archaeological Research Methods") project was an international and interdisciplinary collaborative

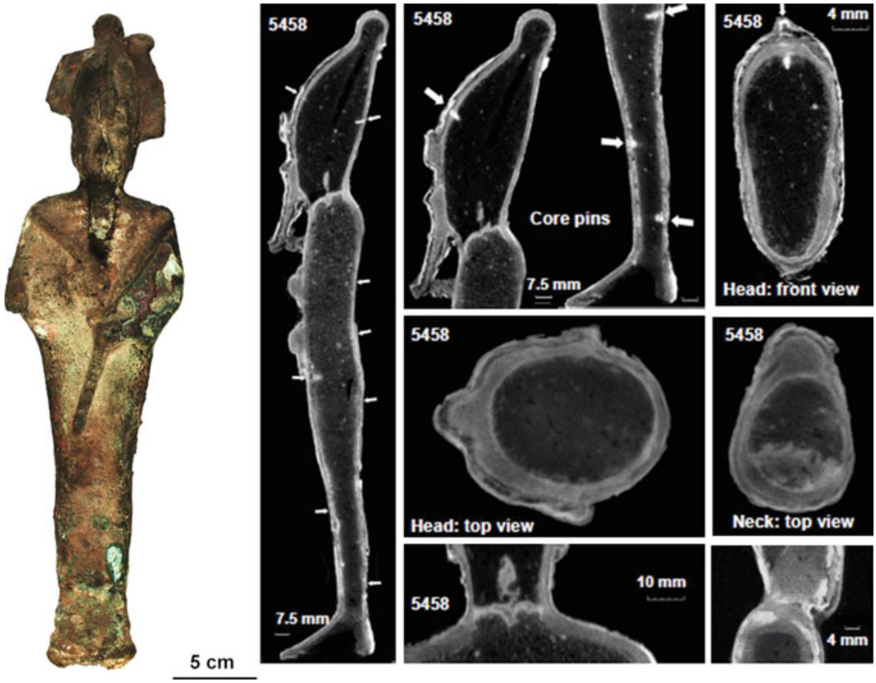

Fig. 1.5 Osiris bronze figure (*left*) was investigated by neutron tomography. The digital sections through the sample volume are shown on the right. The thickness of the bronze material as well as the position of the core pins reveal information about the casting ancient technology (Agresti et al. 2015—reproduced by permission of The Royal Society of Chemistry)

project, financed by the European Commission "New and Emerging Science and Technology" program. The idea of this project followed the exploitation and development of γ-detector for neutron spectroscopy with eV neutrons of the VESUVIO beamline at the ISIS spallation neutron source in 2006 (Gorini et al. 2007).

Within the Ancient Charm project new neutron-based analysis and imaging techniques and non-invasive methods for 3D tomographic imaging of the elemental and phase composition of cultural heritage objects were designed and realized. New techniques and methods were applied to the study of selected real archaeological objects and copies to test effectiveness of obtained reconstruction information (Gorini 2007). Results from this project can be found in Chaps. 13 and 14.

Today neutron techniques are routinely used to provide valuable and additional information to restores for the development of tailored procedures to prevent deterioration. They are employed to obtain a variety of information spanning from the elemental composition, the inner structure of materials and its morphology, the making techniques, the processing and provenance, the dating, the state of conservation and restoration and finally whether the materials is genuine or fake.

In the following Chaps. 4–7 some selected examples of application of most recent neutron analysis techniques are revised. The principle of the Neutron Radiography and Tomography techniques is presented in detail in Chap. 16.

1.3 Selected Highlights

Neutron methods have been used for investigation of prominent objects providing strong resonance in the scientific community and in the society. Some examples of selected research projects and important results are presented below.

- **Investigation of Napoleon's hairs**
  Napoleon Bonaparte died on May 5th 1821 in exile on the island of Saint Helena maybe of arsenic poisoning. Instrumental Neutron Activation Analysis study was performed on several of Napoleon's hair, two samples cut the day after his death and two samples cut seven years earlier (1814) during his first exile on the island of Elba. Results show that all of the samples of Napoleon's hair have a high arsenic concentration. These results disfavor the arsenic poisoning theory (Lin et al. 2004). Details of this study are presented in Chap. 10.
- **NAA at the British Museum**
  Neutron Activation Analysis was routinely used at the British Museum from 1978 to 2002 for provenance studies on ceramics and marble. Data obtained were compared with results coming from other laboratories and are the content of an extended database (Hughes 2007). Similar examples can be found in Chap. 6.
- **NRCA on bronze statuette**
  Neutron Resonance Capture Analysis was carried out on bronze Etruscan statuettes from the National Museum of Antiquities (NMA) in Leiden (NL) to obtain their elemental compositions. Experiments shown different composition of the samples and could identify fakes (Postma et al. 2004). Examples of the application of this method are shown in Chap. 12.
- **Bronze statuettes at the Rijksmuseum**.
  Renaissance bronze statuettes, collection preserved at Rijksmuseum of Amsterdam, were studied with the aim of obtaining fundamental information on manufacturing production techniques of these fine art objects. Sculptures were investigated through neutron tomography at the Paul-Scherrer Institut, Villigen, Switzerland. This method allowed studying the internal shape of the bronze sculptures and provided clues of different material compositions. The most interesting areas of the objects, identified through tomographies, were studied to obtain material compositions and crystalline structures using time-of-flight neutron diffraction on the ENGIN-X instrument at the ISIS facility (Rutherford Appleton Laboratory, UK) (van Langh et al. 2011). For more details, see Chaps. 2 and 4.

- **Painting autoradiography at the Berlin Gemäldegalerie**
  Thanks to a more than two-decade-long collaboration between the Berlin Gemäldegalerie and the Helmholtz-Zentrum Berlin, a large number of paintings were examined by a consistent and systematic use of neutron-activation autoradiography (NAAR) and gamma-ray spectroscopy. The combination of both non-destructive methods provides insight into the pigments used, the brushwork, the state of preservation of the various layers of paint, and the many stages that went into creating the work. The NAAR method is used on regular base at the Painting Gallery in Berlin for taking a detailed look at a painter's creative process. Obtained information helps to gain insight into the artists and time periods of the paintings (Fischer et al. 1987). Examples of this method with detailed description are presented in Chaps. 3 and 15.
- **Ghiberti Heads**
  The study of the gilded bronze artefacts, performed within the Ancient Charm project, was performed using integrated neutron techniques. The reliefs are part of the monumental Doors, *The Gates of Paradise* and *the North Gate,* located at the *Battistero di Firenze* by *Lorenzo Ghiberti*, a masterpiece of the Florentine Renaissance. The reliefs presented critical aspects regarding state of conservation and shows a re-melting whose extension and composition were unknown. The analysis of the Ghiberti Heads were performed using Prompt Gamma Activation Imaging (PGAI)—a technique which integrates the collection of structural information (neutron imaging) and elemental information (PGAA)—to reconstruct the elemental distribution of the elements with a few-mm resolution, Neutron Diffraction and Radiography to obtain valuable information on the manufacturing technique and state of conservation of the reliefs, and Neutron Resonance Transmission to map the gold layer distribution under the pollution deposits to assess the cleaning techniques used by the curators during the restoration process (Festa et al. 2009, 2011) (Fig. 1.6).
- **Metallic Ancient Flute**
  The investigated sample was a small metallic duct flute from *Accademia Nazionale di Santa Cecilia* (Festa 2013b). It is a vertical metal whistle 'in D' with a standard conical bore slightly contracting towards the foot and six finger holes on the front (Fig. 1.7). The object was studied by means of integrated and simultaneous neutron-based techniques such as neutron diffraction, neutron radiative capture analysis and neutron radiography. The aim of the experiment was to characterize elemental and phase compositions of the material, at deriving information on the construction and restoration techniques, and on the role of chemical-physical material properties on the emitted sound.

Results of similar investigations can be found in Chap. 4.

Fig. 1.6 Decorative element (Prophet Head) cleaned by a chemical solution and by laser

1.4 General Remarks

The successful application of neutron based investigation methods to study cultural heritage objects nowadays is a result of a long-term collaboration between the institutions taking care for the objects (museums, institutes and private collections) and the research infrastructures. In general it is not obvious that such collaboration could be possible and even successful. The aim of the restores and curators is to preserve the objects with as less as possible damage and impact through the time. Therefore any invasion related to research attempts is considered very carefully and as conservative as possible. From the other side the neutron researchers are claiming that the experiments with neutrons are non-destructive and non-invasive but this is not always sufficient to convince the cultural heritage specialists. Questions related to sample activation, change of the isotope balance and radiation damage are often neglected or wrongly interpreted. In addition the neutron sources are large-scale facilities and the investigated objects should be brought to the experimental installations. In this case important details like insurance issues, lack of safe storage places or acclimatized room are preventing the transportation of the samples to the research facilities. Nevertheless the unique properties of the neutrons providing results, which are impossible with other experimental methods, motivated the cultural heritage and the neutron researchers to collaborate. The active collaboration between researchers from the two sides made possible to solve all the problems and to establish protocols and good practices for investigation of cultural heritage objects. Nowadays neutron investigation methods are used on a regular base for samples from museum and private collections. There is also a trend to

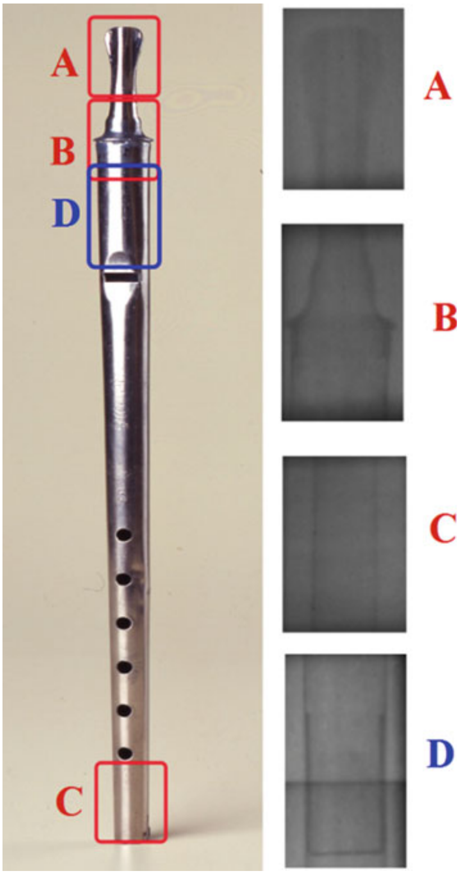

Fig. 1.7 Photograph of the investigated whistle flute preserved at the *Accademia di Santa Cecilia* and neutron radiographies of selected regions (Festa 2013b)

present the results from the investigation as a media show beside to the exposed object in the museums. Example for this is the investigation of several bronze statues from the Rijksmuseum by neutron tomography of which the data are available in online media format. The neutron based methods are already recognized by the cultural heritage society as reliable and distinctive non-destructive research tools and many new collaborations and initiatives were started in the last few decades. They are supported on national and international levels by initiating scientific projects for popularization of the neutron research and for providing of easy access of the relevant objects to the neutron facilities. For example the "Ancient Charm" project was originated by the EU commission in which ten partners from six countries collaborated over four years on investigating cultural heritage objects.

The aim of the current book is to demonstrate the experience gained in the last decade of the successful application of neutron research methods in several cultural heritage fields and to help for further collaborations and joint research initiatives.

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