Chapter 5
The Glory of Ancient Weapons and Armors: Neutron Imaging and Diffraction Methods in Modern Archaeology

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Abstract Neutron-imaging techniques are emerging as an innovative and attractive investigative approach to characterise ancient artefacts without the need for sampling or invasive procedures. In this chapter, a selection of case studies developed in collaboration with several Research Groups and Museum Institutions will show case the potential of neutron imaging technique to investigate a particular class of Heritage materials: metals. Despite neutron technique can be widely applied to different artefacts, i.e. pottery, sculpture, paintings, we decided to focus on this class since the application of neutron imaging technique have disclosed a unique insight into the investigation of their composition and microstructure. Until recently, this task was mainly fulfilled basing on standard analytical techniques like, for example, metallography. Traditional analysis, however, though very accurate, is not always suitable for rare and unique objects of high scientific and economic value.

5.1 Introduction

Due to their high penetration power in metals, neutrons are well suited for investigation of ancient metal weapons and armors. Imaging methods allow for visualization of the inner volume of the object which helps to reconstruct its 3D structure and gain information about the assembly of different components. Neutrons can be considered as particles but also as propagating wave due to their particle-wave nature. The wavelengths of low-energetic (cold and thermal) neutrons are in the same range as the distances between the atoms and atom planes in the crystal lattice of metals. In this way monochromatic neutrons with a defined wavelength have a

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N. Kardjilov and G. Festa (eds.), *Neutron Methods for Archaeology and Cultural Heritage*, Neutron Scattering Applications and Techniques,
DOI 10.1007/978-3-319-33163-8_5

sense to different crystallographic phases of a certain material, e.g. steel, which helps to separate these phases even in 3D and to obtain hints about the manufacturing process at different weapons and armors.

5.2 Used Experimental Methods

5.2.1 Neutron Imaging (Radiography and Tomography—NR and NT)

Similarly to traditional X-ray methods, radiography results in a two-dimensional image of the sample studied while tomography provides three-dimensional information.

However, as the interaction with material differs from X-ray to neutron for the same element, neutron imaging can produce complementary data. Whereas X-rays are more suitable to investigate dense materials inside light ones, neutrons are able to detect light elements such as hydrogen, carbon, and nitrogen, embedded into metallic or ceramic objects. Moreover, neutrons, thanks to their high penetration power in dense matter, represent an almost unique method for the non-invasive characterization of the microstructure of massive metal objects.

In particular NT can provide useful information to characterize the structure, morphology and composition of artworks through a three-dimensional reconstruction of the sample under investigation. From these data it is possible to detect hidden features inside objects, to understand ancient manufacturing technology, to evaluate the conservation status and identify past restauration works, to characterize new conservation methods.

Detailed description of the NR and NT techniques is presented in Chap. 16.

5.3 Examples of Neutron Studies of Ancient Weapons and Armors

5.3.1 Kabuto: Secret Technology Behind Beauty

5.3.1.1 Description of the Investigated Artefact

Kabuto refers to the helmet of traditional samurai armour. Among the various components of the warrior equipment, it assumes, for obvious reasons, considerable importance. Here, the technological skill of the craftsman might reach the best results in joining lightness and effectiveness to defend the most important organ of the samurai's body: the head. In addition, being the most visible part of the warrior from a distance, the helmet assumed also the role of the distinctive sign of a leader

Fig. 5.1 The picture shows the *suji-bachi* helmet that is a multiple-plate type of hachi (Japanese helmet bowl) with raised ribs on every plate and connection rivets hidden into the internal structure

in battle. Thus, not only effectiveness, but also elegance and visibility became necessary qualities for the samurai's helmet, the kabuto.

The kabuto became an important part of the traditional Japanese armour during the feudal period, and was initially worn only by high-ranking warriors (Sakakibara 1963; Sinclare 2004). Their construction and design evolved through time in ways peculiar to different manufacturing schools and modified under the influence of foreign culture and new fighting techniques (Sinclare 2004).

Among the three main categories kabuto can be classified according to its structure, the most complex typology consists of a large number (varying between 8 and 128) of lamellar plates arranged in a circular fashion around the crown on top of the head (Sinclare 2004).

The kabuto shown in Fig. 5.1 is an example of this type. The helmet is a signed Saotome *bachi* made in the 1st half of the 17th Century by Saotome Ienari. He was the third craftsman of the Saotome dynasty, so the helmet is one of the oldest surviving of this type. The sample is made of 64 lamellar plates, finished with a visor decorated with two facing gold dragons. It is a typical sujikabuto, or "flange helmet bowl", presenting ribs at the edge of each plate. The various components are joined by rivets invisible because the surface is lacquered on the inside and patinated on the outside. This helmet was kindly provided by a British private collector and was formerly part of the H. Russel Robinson Collection.

5.3.1.2 Scientific Question

These objects are quite rare and, when found in museums, are usually in an excellent state of conservation, being considered masterpieces representative of Japanese culture. For this reason, any detailed study of these artefacts must rely on non-invasive techniques and it was decided that NT should be employed. This

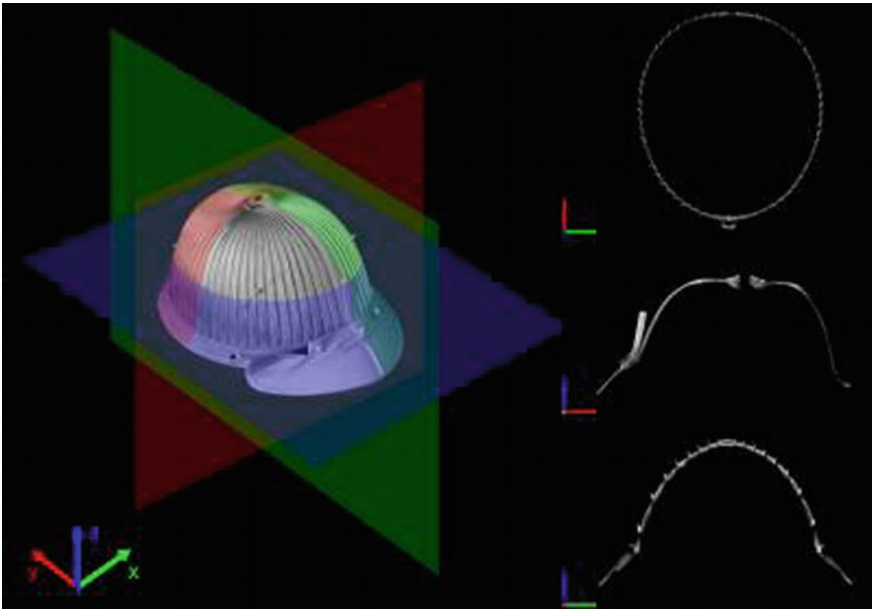

Fig. 5.2 On the left, perpendicular sections trough the helmet (*blue*, *green*, and *red* planes) and their orientation (x, y, and z axis) within the virtual 3D space. From *top* to *bottom* on the *right side*, axial, transversal, and normal views clearly show how the tomographic images were used to gain information on the kabuto inner structure. In the 3D reconstructed image, the *red square* marks the position of a hidden rivet. On the *right*, from *top* to *bottom*, the *red arrows* point its precise location in the axial, transversal and normal orthogonal views, respectively

investigation was conducted on NEUTRA[1] beamline at PSI (CH) (Lehmann et al. 2011).

5.3.1.3 Results

The tomographic reconstruction of the kabuto allowed us to acquire a wealth of information about the inner structure and assembly methods of this masterpiece, so that actual arrangement of the lamellar components in the kabuto was made visible (Fig. 5.2). The construction starts from the central plate, in the back of the helmet. The “S” shaped vertical plates overlap one another, leaving an empty space in the centre of the superimposed area. This arrangement was invented to absorb the energy of a blow and was probably evolved from a simpler structure and never reported in literature. The novelty is in the diagonal orientation of the rivets layout.

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[1] Neutron instruments are presented and described in part II “Experimental methods”.

This type of arrangement allows the helmet to be more resistant to blows. In fact, if the helmet is struck, every plate involved tends to transfer the impact to the next ones. This permits the differently curved parts to contribute to the absorption of the impact energy. Moreover, the external ribs located in the side end of the plates are facing towards the back of the helmet. Thanks to this arrangement, most of the impact energy is transferred to these ribs in case of a frontal or diagonal hit. The diagonal orientation of the rivets helps in keeping the structure stable under the blows offsetting their tendency to deform. It is amazing how a simple modification in the alignment of the boshi improves all the intrinsic advantages of the curved structure by adding stability and hence strength to the entire structure.

Moreover, all components contribute in holding together and stabilizing the two-layers structure. The rivets are arranged in six concentric rings running at different heights all around the helmet circumference joining adjacent plates that radiate vertically from a central opening at the top of the bowl. We also observed that the central plate located in the back of the kabuto is composed by a single metal sheet whose edges are completely covered by the two adjacent sectors. On the contrary, the frontal area was manufactured by superimposing a properly curved narrow metal sheet over a large 3-folded one that completes the round shape of the helmet, thus fixing and securing both sides of the hachi (helmet bowl) structure (Salvemini 2004).

5.3.2 Katana: The Tradition of Japanese Masters

5.3.2.1 Description of the Investigated Artefacts

It is since ancient times that Japanese swords are famous among all the others all over the world as the most effective in terms of hardness, resilience and, last but not least, aesthetic (Yumoto 1958; Kapp et al. 1998).

Their forging technique was almost unique; steel lumps, obtained from the furnace, were strongly pre-treated to obtain a homogenous and purified multilayered sheet. Distinctive carbon steels, characterized by different hardness, were shaped and specifically used for different parts of the blade components to optimize their mechanical feature (Yumoto 1958).

Since ancient time (Koto age, 10th–17th century) five different traditions developed distinctive construction techniques that evolved during the following historical periods (Nagayama 1997).

However, the actual techniques that were used by the early sword-smiths were never documented and the necessary information was orally transmitted from the master to his most skilled pupils. In spite of the large amount of studies, published on the subject, different manufacturing techniques are still not fully understood.

According to history, the different styles of Japanese sword-making are divided into four periods (Sato 1997; Nagayama 1997). Starting from the oldest one (Koto period; A.D. 987–1596), five traditions (Gokaden) evolved their own manufacturing techniques; the smelting and smithing procedures, the forging, and the final treatments, were different and specific of the particular schools that each tradition started, in the frame of every peculiar tradition, in a specific province and then diffused in the rest of the country. These provinces were either related to centres of political power or simply were located in geographic areas rich in iron ore.

5.3.2.2 Scientific Question

Until recently, only expendable samples were investigated basing on standard analytical techniques which manly require sampling or are based on surface analysis. Nowadays, neutron diffraction (Grazzi et al. 2011a, b, c) and neutron imaging methods (Squires 1996; Sears 1992; Lehmann and Hartmann 2010; Josic et al. 2010) have been demonstrated to be the most suitable tools to qualitatively and quantitatively characterize composition and micro-structural properties of metal artifacts in a non-destructive way, mandatory for well conserved museum exhibits (Piaskowski and Hist 1993; Nagayama 1997).

5.3.2.3 Results

NT was performed on five fragments of ancient swords (Fig. 5.3), broken approximately at 10–20 cm from the tang and already analysed through time of flight neutron diffraction (Grazzi et al. 2011a, b, c). All swords are assigned to a specific tradition: four of them are attributed to the Koto Age forging traditions, and one is a more recent blade attributed to the Shinto period (A.D. 1596–1781). The attributions were made through the analysis of the signatures (certain attribution)

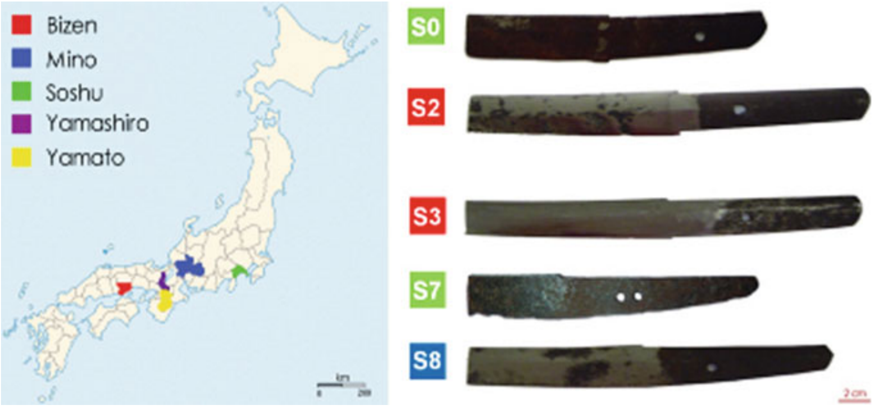

Fig. 5.3 Map of Japan (Honshu is the main island) showing the location of the provinces where the five Koto age sword-making traditions were originated (*left*) and a picture of the five blade fragments (*right*)

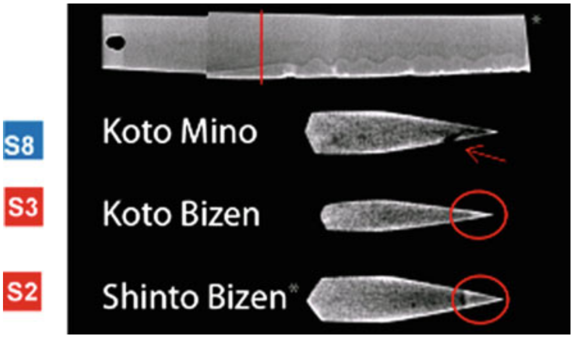

Fig. 5.4 Selected cross sections from the NT are reported for 3 samples: a crack is clearly visible along S8 (*red arrow*), while the martensitic structure is visible at the cutting edge of the swords S3 and S2 (*red circles*). The approximate location of the cross section is indicated by the *red line* in the *upper* 3D rendering model

and, in absence of signature, through the stylistic analysis by our museum experts (hypothetical attribution).

The overall study was carried on ICON[2] beamline at PSI (CH) (Kaestner et al. 2011) and full results were published at Salvemini et al. (2012); here a selection of the most important data is reported.

In particular, NT is able to provide morphological inner features, such as amount, distribution and shape of defects, porosity and slag inclusions, that are related to the manufacturing process(i.e. quenched microstructure along the edge, defects, slag inclusions, internal cracks). In addition, important information on the conservation status can be obtained from the determination of the mineralization phases and the corrosion products than can be easily mapped due to the high neutron attenuation cross-section for H-base compounds (Fig. 5.4).

Sample S2 is characterized by a homogeneous inner structure of the blade cross section. No trace of cracks or slags has been evidenced by the tomographic technique. Instead, we were able to detect a clear space pattern at the cutting edge where the crystalline structure of the steel changes from hard martensite to pearlite, giving rise to the hamon, clearly delimited in the Fig. 5.5a. The tomographic method also allowed mapping several rust spots that extend well below the sample surface (Fig. 5.5b).

Standard neutron tomographic technique can be further improved, to achieve materials discrimination, by a proper selection of the neutron energy. In fact, monochromatic neutron beams give the possibility of modifying the image contrast for different phases taking advantage of the abrupt change of the attenuation coefficients in the proximity of the so-called Bragg cut-off (Josic et al. 2010). This method is able to provide a map of the phase distribution phases. This information can be related to the use of different types of steel and can help to define the forging procedure of the samples.

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[2] Neutron instruments are presented and described in part II “Experimental methods”.

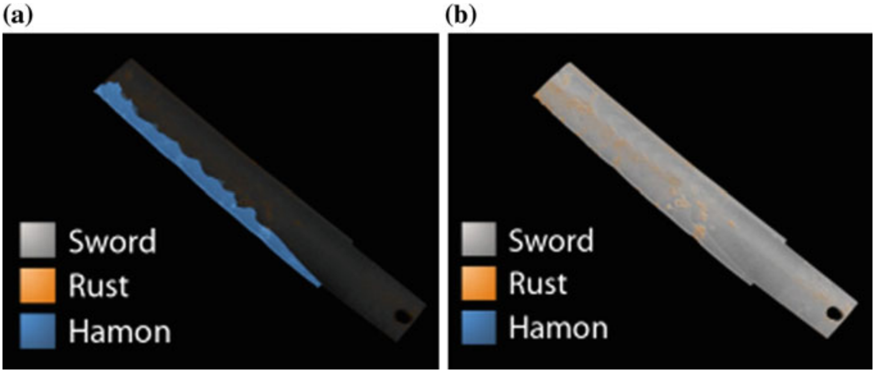

Fig. 5.5 **a** The 3D segmentation of blade S2 shows the progress of the martensitic microstructure, here reported in blue colour, outlining the hamon pattern along the cutting edge. **b** The 3D surface projection evidences the presence of rust spots (*orange* colours) diffusing below the surface of the fragment S2

Fig. 5.6 The white beam NT of the sample S3 has allowed mapping the distribution of the slag inclusions along the blade, here mapped indicated in *red*

Referring to Fig. 5.7, we observe that the inner volume of the fragment is characterized by different distributions of dark areas (cementite) and light areas (ferrite). Thus, the present method allows us identifying the typical structure of a Japanese sword, which is known to be composed of two distinct sections of different types of steel (Kapp et al. 1987). According to Kapp et al. (1987), Japanese medium- and full-size swords are composite structures made of a kawagane and shingane components. Kawagane (jacket steel) is the component forming the outer surface of the blade, including the sharp edge and the hamon. Shingane (core steel) is a slightly softern, low-carbon steel, which is embedded or wrapped into the high-carbon jacket steel along the entire length of the sword. Being more ductile than kawagane, it helps protecting the blade from cracking and breaking under stress. Still according to Kapp et al. (1987), Japanese smiths assemble the different pieces into a block, weld it together, and then draw out the steel bar into a sword. More complex structures might use four or more different pieces of steel for the

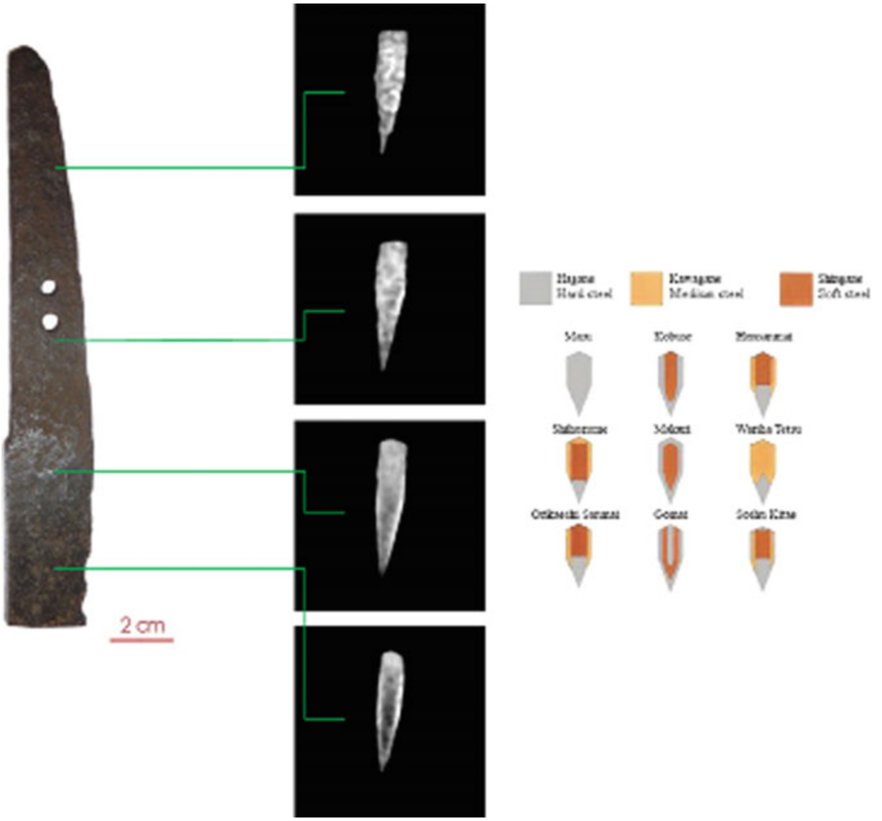

Fig. 5.7 Reconstructed slices at different height along the sword S7. Energy resolved tomography have been able to evidence light areas of ferrite and dark areas of cementite. On the *right*, we report some different sword configurations, using steels with different carbon contents, typical of Japanese swords (Kapp et al. 1987)

core, the edge, the side, and the back of the blade. This great variety of forging methods reflects the way the various schools developed, in isolation from each other, over the country. However, we should point out here that this interpretation of the inner structure is not unique and other determinations have been proposed following a metallographic analysis (Tawara 1953; Hoshi and Sasaki 2005; Notis 2000).

The tomographic slices, reported in Fig. 5.7, suggest that the S7 blade has been probably manufactured by assembling a “jacket” (side and back) of low-carbon steel (shingane) around a very hard steel (the so-called hagane) composing the “core” and the “edge” of the sword. Thus, the present structure appears to be inconsistent with the one suggested in Ref. 21. Nonetheless, it is still possible to identify the present configuration among the various possibilities given by Kapp et al. (1987) (cf. right picture in Fig. 5.7) and, consequently, the sword should be assigned to one among the OrikaeshiSanmai, SoshuKitae, WarihaTetsu or Honsanmai configurations (Kapp et al. 1987).

5.3.3 Koshirae: Decorative Motifs of a Social Status

5.3.3.1 Description of the Investigated Artefacts

Japanese sword mountings are the various housings and associated fittings that hold the blade of a Japanese sword when it is being worn or stored. In particular koshirae refers to the ornate mountings of a Japanese sword.

The sword-fittings that are most admired today were considered to be adequate if practical function, and also aesthetic criteria, were fulfilled. These features were applied to their design or manufacture, thus leading to the evolution of more elaborate decorative techniques and the emergence of specialist sword-fitting makers (Sato 1997).

The commonest basic material for sword-mounts, especially among the earlier examples, is iron, usually of fine quality. The other metals employed, commonly known as soft metals, include silver, bronze, brass, copper, and the three special copper alloys which are peculiar of Japan (Sugimori 2004).

Several decorative methods were employed: surface pattern and finishing, piercing and openwork, relief-modelling and etching, inlay, overlay, and incrustation, engraving and chasing, to mention a few. Finally, the artefact was treated with special etching baths, whose result is to produce a rich palette of pigmentation for the different alloys. Although these colours are only skin-deep, they are practically permanent, as long as they are not subjected to scratching or rubbing with abrasives, which will rapidly destroy them and reveal the raw metal beneath.

5.3.3.2 Scientific Question

For the present study a set of two hand-guards of Japanese sword (tsuba) were investigated by means of NT on CONRAD2[3] Beamline at HZB (DE) (Kardjilov et al. 2011). The study aimed to understand the manufacturing process used in the making of the artefacts.

5.3.3.3 Results

The first tsuba (Fig. 5.8a) is a slightly elliptical iron tsuba with a rounded rim. The tsuba is carved with a tendril design (sukashi technique), including facing dragons,

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[3] Neutron instruments are presented and described in part II “Experimental methods”.

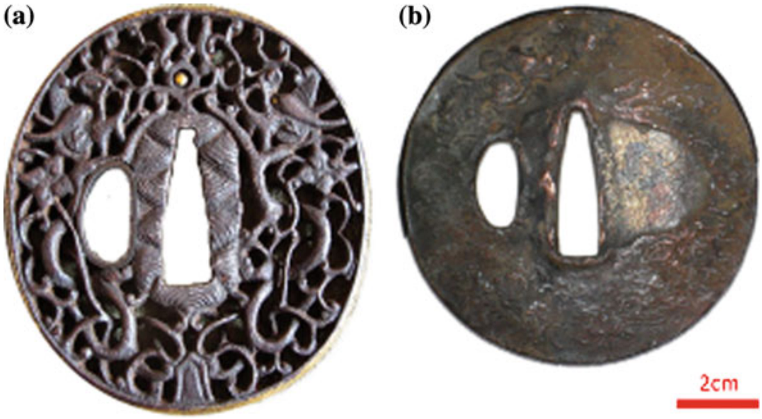

Fig. 5.8 **a** The iron tsuba, **b** the copper tsuba

a jewel, and stylized flowers. The rim is rounded and gilded and there are traces of gilding in other areas. It is not signed but can be dated to the first half of XVIII century, probably from Nagasaki (Hizen) area, in namban style. The Japanese word namban, translated as “southern barbarian”, was used by Japanese not only to indicate any foreigner who entered their country, but also to define a large number of artworks in which the decorations reflected a foreign influence in their appearance. The diameter of 8 cm leads us to presume that this hand-guard was created for a long sword (katana). It was equipped with a nakagohitsu, the central hole for fitting the tang of the blade, and a kozukahitsu, the left side hole for accommodating the handle of a small knife called kozuka.

The second Japanese hand-guard was made of copper and copper alloys (Fig. 5.8b) and is of sanmai type: this is composed by three layers with the central one made of a different alloy with respect to the others. The upper and lower layers are decorated through hammering. Decoration was typically made acting on the back of the layer and then fastening it to the central layer with adhesive or lacquer. A fukurin, i.e. soft metal, type rim was hammered around to hold the three layers together. The rim profile is rounded and not decorated. The decoration theme is made of waves and imaginary animals (dragons). The piece can be dated either to the end of Momoyama period or to the beginning of Edo period. It was probably made in the Kyoto area around the beginning of XVII century. A relief decoration is visible on the surface even though it is in poor conditions of conservation. Detachments are recognizable at the edge and on the decorated surface. As in the previous sample, there is a nakagohitsu and a kozukahitsu.

The analysis revealed a homogeneous body for the iron tsuba, probably filed starting from a single piece of iron (Fig. 5.9). As a matter of fact, starting from the Muromachi period, the sukashi technique evolved over time. The iron plate prepared as the ground metal was very uniformly forged and relatively soft in order to cut out the fine designs successfully but hard enough to avoid breakage during use.

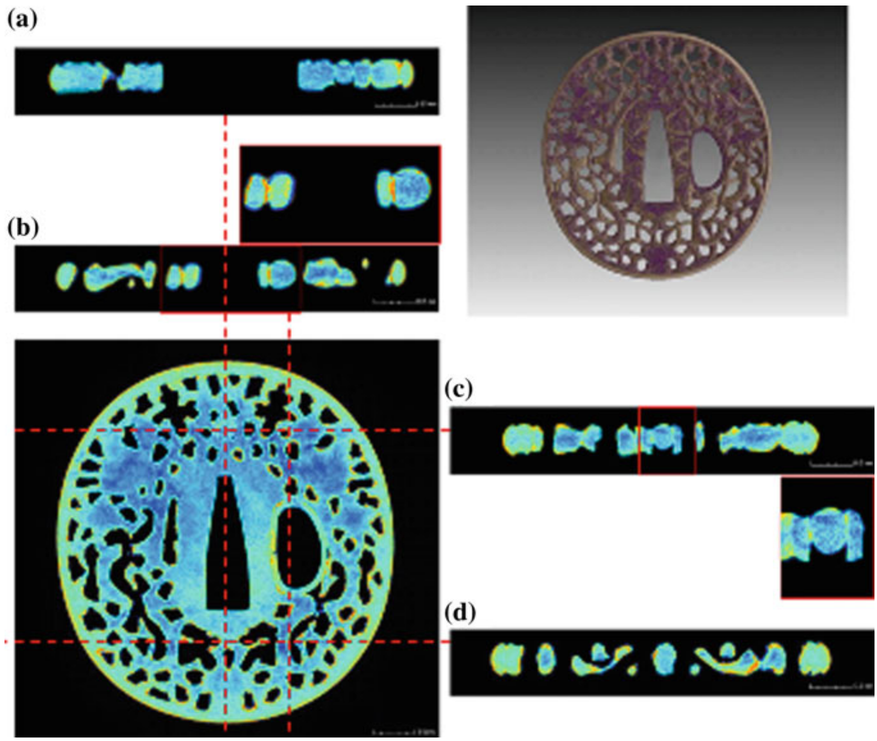

Fig. 5.9 On the *bottom left side* of the figure, a virtual cross section of the iron tsuba is reported in false colours (brighter areas relate to higher attenuation cross sections). The *red dotted lines* define the position of the orthogonal views shown alongside (**a**, **b**, **c** and **d**). The *red squares* map the area of interest inside the views and their respective enlargements. In the cross section **a**, the evidence of soldering are visible in *red-orange* tone at the edge of the ring. While images **c** and **d** allow observing the absence of such soldering and the *brighter spots* can be related to the patination or alteration of the surface. The view **d** gives an idea of the complicate arabesque-structure made by piercing a single metal disc. The *top right* image refers to a 3D rendering of the sample

Any void or significant irregularity in the iron body would have made the shaping of continuous decorative lines impossible. Extra effort in the preparation of the plate would have been necessary, as observed in inner volume of the samples which was free from any cracks, pores and inclusions (Sato 1997). Evidences of forge-welding were not identified in the design motifs involving plants and animals figures, apart from the gilded ring outlining the profile of the kozukahitsu. While the inner brighter areas are reconstruction artefacts, the ones detected on the surface were probably due to the application of a patina or to the early formation of products of alteration (Sugimori 2004).

Virtual cross sections taken at different heights of the copper tsuba revealed three layers of copper and copper alloys (Fig. 5.10). According to ancient metal working technique described in literature (Robinson 1970), the external sheets of metal were

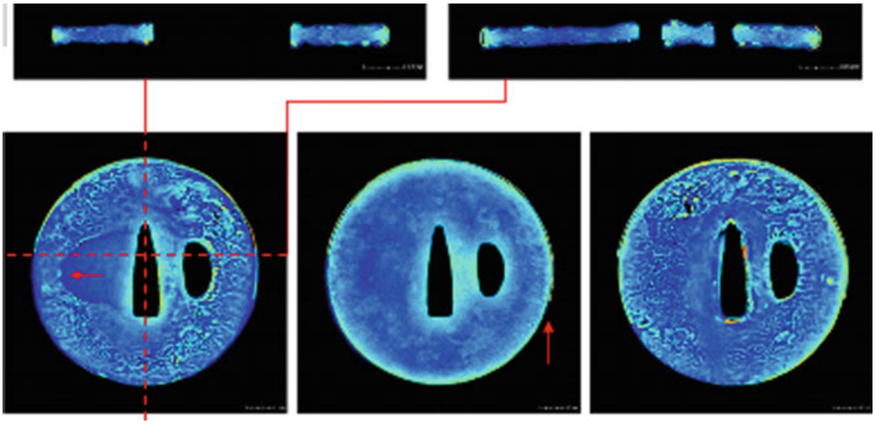

Fig. 5.10 The three *bottom* images refer to cross sections taken at different depths of the copper tsuba, starting from one side of the metal disk and progressively emerging on the other one. The layers closer to the surface (the first and the third radial cross sections) feature the decorative pattern and the lacking area (frontal cross sections), enhanced by the false colour visualization. Moreover, the remaining of a ring around the border of the tsuba is visible (*red arrow*, second image) The *red dotted lines* map the position for the orthogonal views, reported above, where the decorated layers joined to the bulk sheet and the enclosing ring can be observed

probably applied on the two sides of the inner core by heating and slightly hammering, while their rather soft surface was worked with a punch to give texture. All around the rim of the tsuba and the pierced hole, the composite structure was finally tightened by applying a thin ring appearing detached and lost in some areas (Salvemini et al. 2015).

5.3.4 *The European Swords*

5.3.4.1 Description of the Investigated Artefacts

The study of historical technologies allows revealing methodological process that leads to the conception, development, and evolution of a specific artefact, trying to establish the relationship existing among people that implemented it, as well as the socio-economical context that characterized the circumstances of its production.

By the year 1300, after nearly four thousand years of continuous development, the sword in Europe reached its apogee as an instrument of war. The evolution of the sword and the dagger throughout the Renaissance period presents a fascinating and complex picture. Long-cherished forms were retained side by side with forms which changed from year to year, making exact chronologies sometimes difficult to establish.

In response to change in body armour, new military tactics or simply the dictates of fashion, many sword types flourished from the mid 15th century when the sword, and in particular the light and fast rapier, became an accepted part of soldier civilian dress and battlefield equipment.

The 17th century was a most important one in the evolution of the sword in Europe; from the 1600s the fashion for rapier and dagger play in fencing had considerably less popularity. A small version rapidly become the most commonly worn sword in Europe and in some countries continued its career until its decline during the 19th century (Coe et al. 2012).

Coeval documentation regarding the manufacturing and commerce of sword blades includes references revealing how blades were valued depending on their place of manufacture, with Toledo blades scoring on top, and the other ones priced down to three times less. The "old" Toledo masters (16th and 17th Century) made their best sword blades by means of forge-welding two strips of steel sandwiching a third strip of wrought iron, that formed the core. The composite block was forged and shaped into a blade. Finally the blade was heated and quenched only in the upper 80 % of its length, while the remaining 20 % was simply tempered. The purposefully composite assembly and the obtained microstructure found their explanation into the need of containing the cost related to the purchase of steel, the typology of rapier blades, the kind of fighting techniques adopted at that time, the need of avoiding fragile fractures, and the easy fitting of the mounting (Gener 2009).

5.3.4.2 Scientific Question

The present study was undertaken with the aim to determine differences in actual manufacturing technology among different European sword blades. To this aim, two rapier blade fragments from the 17th and 18th century have been investigated (Fig. 5.11).

5.3.4.3 Results

The first blade (labelled HE-4) is marked as being made in Solingen (actual Germany), while the second one (labelled HE-5), though subject to some debate, is marked as being made in Toledo (Fig. 5.11). We should point out that reasonable doubts could be raised about the second one's origin and that this blade too is probably from Solingen.

The samples were not considered precious; therefore complete and half cross-sections were cut in different points of the longitudinal direction. Consequently, it was also possible to investigate the variations in construction and microstructure along the blade samples through standard metallography. The same

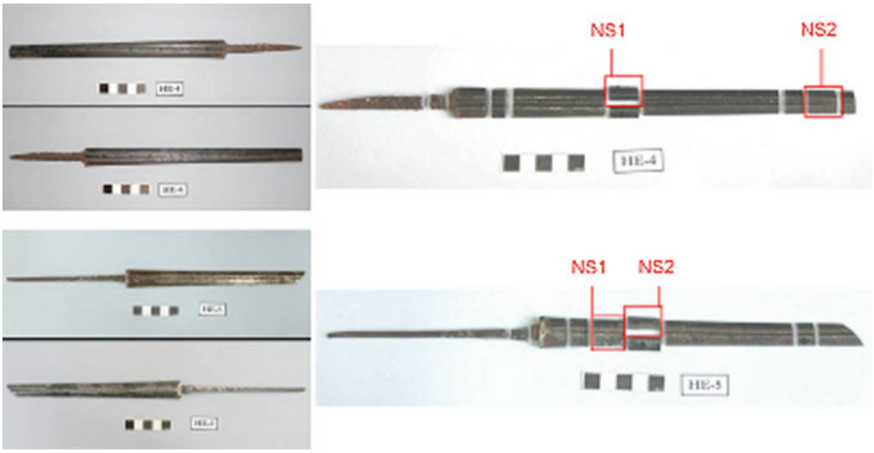

Fig. 5.11 On the *left*, the integer sword blades are shown on their both sides. On the *right* image, the *red frames* map the original position of the analyzed cross section

samples were analysed by means of neutron imaging to observe and to explore advantages and limits of the techniques, through validation based on previous metallographic investigation (Gener 2009).

The neutron imaging study was performed at the ICON[4] beamline at the Paul Scherrer Institut (Giller et al. 2008).

Preliminarily, conventional NT was carried out to provide 3D information about the structure inside the sample. Then, in order to obtain information about the composition and material distribution, an energy-selective neutron imaging analysis was carried out too.

Conventional Tomography

In the fragment NS1 of Solingen blade HE4, the reconstructed volume of the two cross sections, evidences a very homogeneous body (Fig. 5.12, left side). As a matter of fact, the metallographic study of fragment HE4-3, the twin half of sample NS1 and longitudinally cut along the central channel, confirmed a very homogeneous microstructure. The inclusions appear elongated in the direction of the longitudinal axis of the blade, showing the direction in which the original metallic block was forged, and they are distributed in layers, which can be due either to the construction of the core using various fragments of steel welded together or to the consolidation processes that were part of the production of steel (Fig. 5.12a).

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[4] Neutron instruments are presented and described in part II “Experimental methods”.

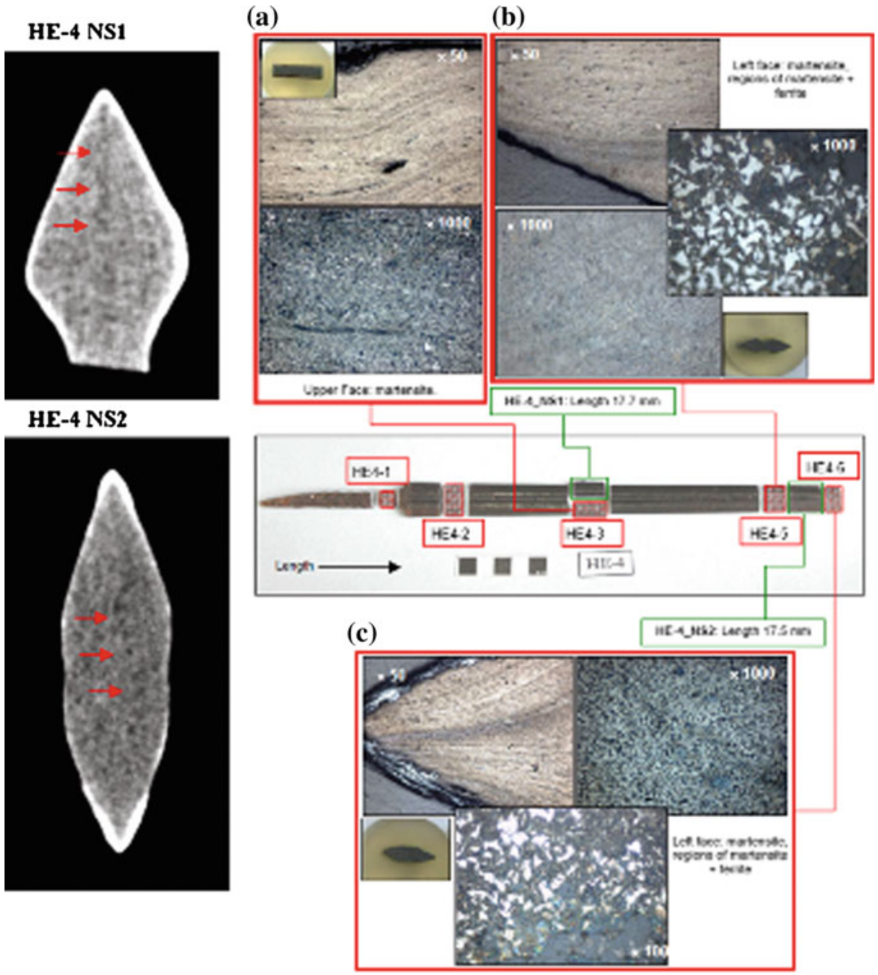

Fig. 5.12 On the *left* side, selected slices from the reconstructed volume of the HE-4 fragments are shown. In sample NS1, the *red arrows* point out the mark of a welding, while in sample NS2 the distribution of slag inclusions. On the *right* side, the central image maps the original location of the cut fragments; the cross sections analyzed by means of metallography are framed in *red*, and the samples analyzed through neutron imaging are reported in *green squares*. The metallographies are shown. The windows correspond to overviews of the samples **a** HE4-3 Detail of the area around a mark (**a** letter, hot-punched inside the central groove—or “fuller”—of the blade), showing the layered structure of the slag and how it conforms to the deformation of the mark (×50) with detail of the microstructure (×1000) **b** HE4-4 Detail of the edge, showing the weld (×50) with detail of the microstructure (×1000) **c** HE4-5 Detail of the edge, showing the weld (×50) with detail of the microstructure (×1000)

Moreover, along the two cutting edges, the presence of martensitic microstructure was revealed by the detection of a bright superficial area in neutron tomographies and confirmed by standard analysis where tempered martensite was observed (Fig. 5.12).

In the cross sections taken before and after the fragment NS2, the metallographic results were again coherent with neutron tomographic investigation (Fig. 5.12, left side). At this point we concluded that the blade is also composed of a single type of material, a heat-treated steel associated to tempered martensite microstructure (Fig. 5.12b, c).

The fragment NS1, pertaining to the sword blade HE-5, featured a composite construction instead, already visible in the white beam neutron tomographic reconstruction (Fig. 5.13, left side). These evidences have been confirmed by the metallographic investigation (Gener 2009).

The metallogarphied sample HE5-2, cut closer to the tang, respect to the fragment NS1, features the same construction. At this point, though, the external layer has been ground off at the edges of the blade, to the point of exposing the internal core in one of them. In this sample, the external strips feature ferrite, and the internal core shows a pearlite and ferrite microstructure corresponding to an approximate proportion of carbon around 0.5 %, with the pearlite also partially spheroidised (Fig. 5.13a) (Gener 2009).

Sample NS2 is also from a longitudinal cut done along the central channel. At the end of the sample closer to the tang, two wedges of iron are visible, marking the end of the iron wrapping, which only reaches this point. It can be observed and confirmed by the metallographic analysis conducted on the twin cross section HE5-3 (Fig. 5.13b).

Energy-Selective Imaging

In the second part of the experiment, energy selective neutron imaging analyses were performed.

Owing to the symmetry of the sample and the homogeneity in their inner structure, only radiographies were acquired (Fig. 5.14).

The radiography of the HE 4 cross sections confirmed the presence of a homogeneous body. In particular, sample NS2 (Fig. 5.14) evidenced a core that was probably made joining various bits of metal in order to have a piece of suitable steel big enough for the task (Gener 2009).

The presence of a dark region, outlining both fragments of the HE-4 sword blade can be related to the presence of martensitic structure, well detected and most extended with respect to the HE-5 samples. It is not to be excluded that this effect can also be relate the presence of corrosion products on the surface of the sample and slightly diffusing inside the body.

In the fragment HE5, a variation in contrast has been recognized, as evidenced in sample NS1 of Fig. 5.14. Crossing the 110 ferrite Bragg-edge (i.e. changing wavelength), the inversion of grey tones is visible and the obtained contrast

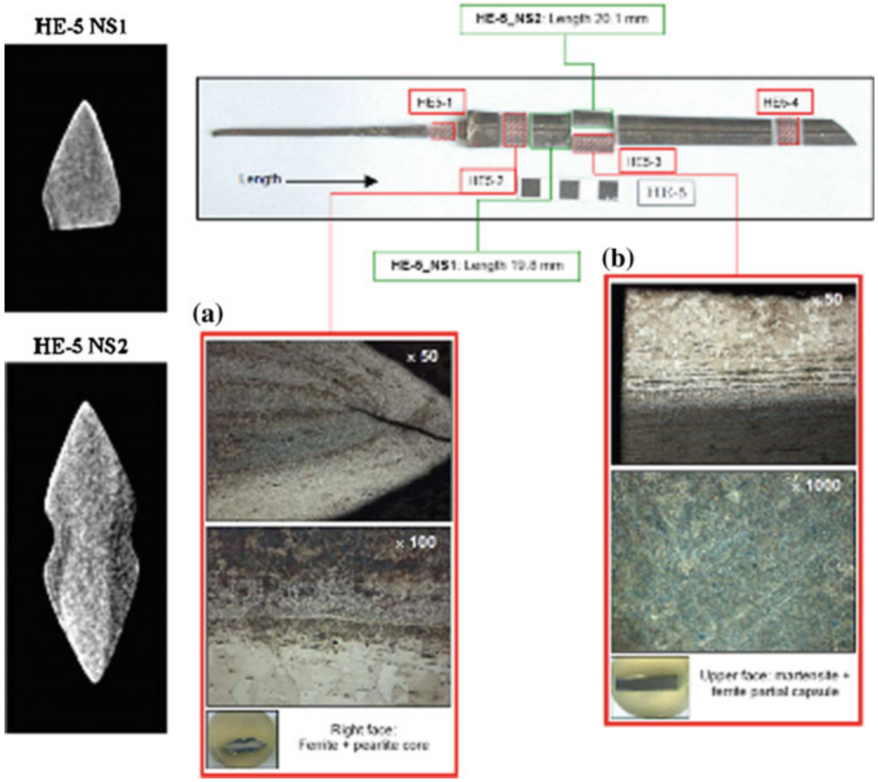

Fig. 5.13 The inner composite volume of the HE-5 fragments is visible from the tomographic slices. The stretching of the dynamic range allows to enhance the contrast and to discriminate between iron (*dark*) and steel (*light*). On the *right* side, the original location of the cut fragments is documented by the *upper* image; the cross sections analyzed by means of metallography are *framed in red*, and the samples analyzed through neutron imaging are reported in *green squares*. The respective metallographies are shown in the *bottom*. The windows correspond to overviews of the samples **a** HE5-2 Detail of the edge area, showing the crack developed there (×50) with core microstructure, partially spheroidised pearlite (×100) **b** HE5-3 Construction detail, showing the weld area between the iron external strip and the core (×50) and microstructure of the steel core, tempered martensite (×1000)

enhancement allowed discerning between iron and steel. On the contrary, fragment HE-5 NS2 did not evidence any variation in composition of the used steel.

Gray gradient in the image can be attributed to the shift in the incoming neutron wavelength distribution over the acquisition window. Due to the small field of view, the limited side of the analyzed sample and the required neutron energy resolution, the wavelength shift can be ignored (Peetermans et al. 2013).

Small blurring in the contour (i.e. sample HE-5 NS2) are caused by misalignment in the positioning of the sample respect to the neutron beam axis.

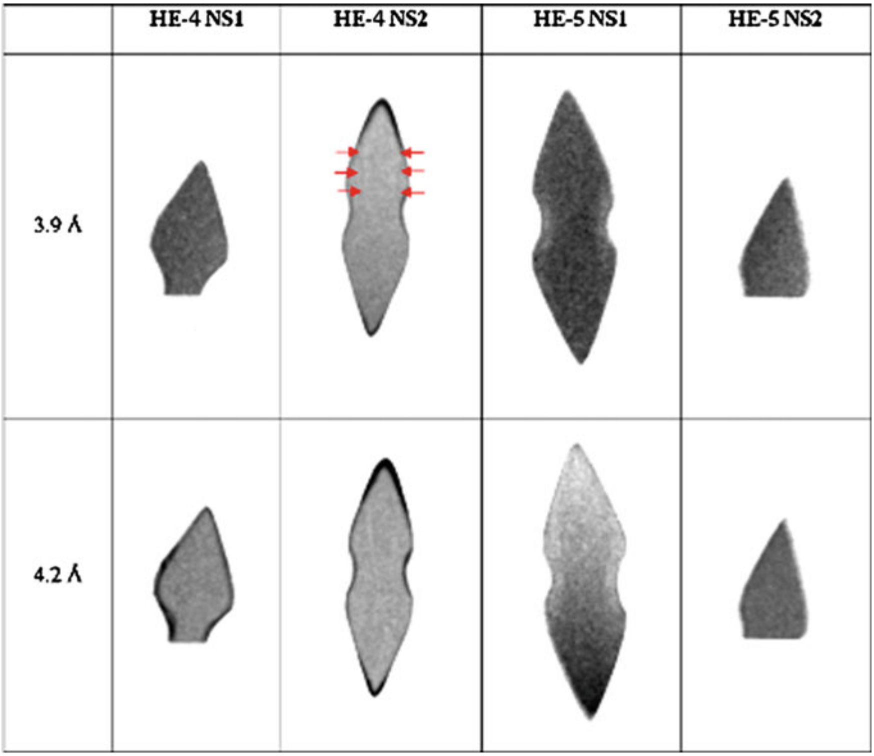

Fig. 5.14 Monochromatic neutron radiographies taken at 3.9 and 4.2 Å are reported for each fragment that was labelled at the *top* of each image. In the case of sample HE-4 NS2, the *red arrows* indicate the weld of pieces of metal

However, the result obtained from the energy-selective imaging study, gave coherent data with respect to the metallographic analysis. We want to stress that the composite structure of European Renaissance blade, described in historical documents and detected through standard destructive analysis, was recognized using a non-destructive method. Thanks to the energy-selective radiography, the volume of samples was qualitatively and quantitatively characterized, allowing detecting the forge-welding strips of wrought iron sandwiching a third strip of steel that formed the core (Salvemini 2014).

5.4 Conclusion

The case studies presented in this chapter clearly demonstrated the potential of neutron imaging techniques for the characterization of metal artefacts of historical, archaeological and cultural interest.

In particular, neutron tomographic methods allowed identifying structural components and morphological features that can be related to the manufacturing process, the life cycle, and the state of conservation of artefacts. For instance:

- the presence, location, direction and arrangement of structural components can be determined;
- inclusions and porosity can be mapped, localized and quantified;
- phase of different composition and microstructure can be evidenced;
- presence of possible welding, soldering or binding materials can be clarified;
- patination of the surface can be verified;
- defects and corrosion products can be identified for assessing the conservation status.

From the archaeometallurgical standpoint, this information can be extremely useful to establish interesting relationships and cross-matching among technologies developed by different material cultures. Especially considering arms and armours, NT can reveal their composite assembling that evolved over time in order to obtain specifically desired technical and mechanical performance as well as aesthetical design.

On the big picture, these studies could reveal a sympathetic overview of the lost traditional manufacturing procedures, mainly transmitted orally to the present, and often recovered by contemporary craftsman on a personal level, referring to partial information reported in written documents not always fully reliable.

A new interesting possibility has been disclosed by neutron imaging methods and further work will be needed to gain a more thorough understanding of the ancient manufacturing methods.

The present analysis could not have been carried out using traditional (invasive) analytical methods, which cannot be applied to such ancient and rare artefacts where a non-invasive, non-destructive experimental approach is mandatory.

In our study, neutron imaging allowed revealing unexpected features with respect to the current literature report and some of the obtained results were reported in the international literature for the first time.

Despite neutron-imaging techniques, in a combination with other method, especially neutron diffraction, provide an attractive approach, it is very rarely applied to help conservation and diagnostic of museum objects. By presenting the obtained positive results, we hope this work will ideally encourage further study and application of neutron techniques to different types of objects or a more in-depth look at their application to metal artefacts, whose current problems, or open questions, cannot be solved with the current, often invasive, commonly used techniques (Salvemini 2014).

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