

## Science for Cultural Heritage\*

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### 1. Introduction

We can find in the different regions of our planet a vast range of materials left by more than two thousand human generations. Some of these materials are in museums and private collections, some are still buried under meters of sediments and rocks or at the bottom of the sea. They are the product of *culture*, a unique character that appeared 50,000 years ago in ‘behaviourally modern humans’. The archaeological record shows at that time an explosive beginning of complex social behaviour and the broad use of symbolic expressions. The rock art of the Kimberley and the funerary burial remains of lake Mungo in Australia are outstanding remnant of that ancient culture. One possible explanation is that about 50,000 years ago a genetic mutation in *Homo sapiens* made possible the transition to speaking capabilities characterised by a fully syntactic language. This accelerated the evolutionary process and generated the ‘behaviour’ we still have today.

The products and signs of human culture in the past are broadly defined as ‘cultural heritage’. This term refers, in general, to human artefacts, such as buildings, statues, art objects, tools, weapons, etc., mostly made during the second half of the Holocene, after the development of large human settlements, following the diffusion of agriculture and animal domestication (people believe the biblical city of Jericho started as a village nine thousand years BC). The physical remains from prehistory, particularly the products of *Homo sapiens* culture, including rock art, stone tools and ornaments, are also part of our cultural heritage. There is a controversial debate on the culture of *Homo neanderthalensis*, extinct about 30 thousand years ago, but it was probably similar to that of ‘anatomically modern humans’ that emerged for the first time in the African fossil record 150-200 thousand years ago. Cultural heritage can be extended to include the first rudimentary stone tools produced by *Homo habilis* and *Homo erectus* during the Lower Pleistocene. Finally, more intangible categories related to human culture, such as dance, music and religious beliefs should be also considered as elements of our cultural heritage.

Considering the cultural heritage related to historic times, this is particularly significant for the identity of human societies, being closely related to their identity, an aspect of growing significance in the era of ‘globalisation’. Prehistoric cultural heritage is highly significant for indigenous groups, such as Aborigines in Australia and native populations in North America. For example, the age of their arrival and other archaeological findings has been linked to issues of land rights and other civil claims.

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In any case, it is very important to preserve cultural heritage, also for the benefit of future generations. Cultural heritage has a considerable economic value: it has been evaluated (see Norwegian/European project ‘Cultural Heritage Monuments and Historic Buildings as Value Generators’) that the turnover of cultural heritage related income in Europe is 335,000 M€ per year. On the other hand, cultural heritage loss due to pollution and aging is estimated at 14,000 M€ per year.

Cultural heritage is of paramount importance for developing country societies and economies. ICTP, under the aegis of UNESCO and IAEA, promotes the use of advanced methodologies in cultural heritage applications. In particular there are specific areas that require urgent attention, such protecting and restoring cultural heritage sites from natural and man-made disasters (climate change, earthquakes, air pollution, etc) and illegal trade in counterfeit art.

It is important to decipher the information carried by cultural heritage materials and their connection to the culture of the people who made them. This can be achieved using the scientific method – the body of techniques for investigating phenomena and acquiring new knowledge of the natural world. All the natural scientific disciplines contribute to understanding and preserving cultural heritage. Geology provides the concepts to model the change of landscape in the past and its effects on cultural heritage remains; Chemistry looks at the reactions modifying materials left by human activities, including the deteriorating effects of pollution; Biology has a growing role in the understanding of the past, particularly with information at the molecular level, including DNA and genes

We will focus on advanced scientific tools and procedures, mainly developed in physics research that can be used for the non-destructive characterisation of cultural heritage materials. These include new microscopes based on synchrotron radiation, neutrons, ion beams, lasers and other radiations, or particles that can reveal non-destructively the structure and composition of art objects and archaeological remains. The analyses are applied to a variety of ‘hard’ materials, such as artefacts in metal, ceramics, stone or fossil human teeth and ‘soft’ materials, such as textiles, wood and paper. Each kind of material requires a different analytical strategy and the use of a suitable probe.

The morphological, elemental and isotopic composition inferred from the analyses of these materials is important to art history, archaeology, anthropology and other areas of research relevant to cultural heritage. This in-depth characterisation can be used to develop appropriate strategies for the conservation of cultural heritage sites and objects. The scientific community involved in cultural heritage studies is producing a growing amount of information that can be shared globally using virtual networks, databases and all the tools made available by the information/communication technology revolution.

## **2. Characterisation of cultural heritage materials**

Cultural heritage materials need to be characterised in the four dimensions of time and space, spanning scales of many orders of magnitude. Chronologies from decades to million years can be measured by ‘clocks’ based on radioactivity and other phenomena characterised by predictable changes with time. Satellite imaging and laser scans provide tools of increasing sophistication for prospecting cultural heritage sites on space

dimensions from kilometres down to centimetres. Composition and structure of cultural heritage materials is analyzed down to the nanometres scale using new microscopes based on synchrotron radiations and high energy ions.

The analysis of isotopic ratios for elements such as carbon, oxygen, nitrogen, calcium, strontium and other elements provides useful information to reconstruct migration patterns and diet of ancient human populations.

Genetic science offers novel approaches to cultural heritage studies. For example, the analysis of 'ancient DNA' has been applied to investigations of Egyptian mummies and Neanderthal remains.

In the following we will discuss more details on advanced instruments and methods used in studies of cultural heritage.

### 3. Dating

An important objective in cultural heritage studies is to order chronologically past events by analysing materials associated with past human activities. Relative chronologies can be deduced from circumstantial evidence, such as change of style and manufacturing technique. Relative chronological information can also be obtained using methods based on time-dependent geological and chemical changes (e.g., stratigraphy, sedimentation rate, weathering, hydration and magnetism). Certain kinds of cyclic phenomena, such as tree ring or varve formation, will yield very precise chronologies if stringent precautions are followed. Finally, many methods providing absolute chronologies are based on time-dependent phenomena related to natural radioactivity, and include:

1. decay of long-lived radionuclides produced by cosmic rays, as in the radiocarbon method;
2. *in-situ* production by cosmic rays of long-lived radionuclides, such as  $^{10}\text{Be}$ ,  $^{26}\text{Al}$  and  $^{36}\text{Cl}$ , which can be used for dating rock surfaces and stone artefacts;
3. build-up of radiation exposure effects, in thermoluminescence (TL), optically stimulated luminescence (OSL), electron spin resonance (ESR) and fission track dating;
4. build-up of a radiogenic daughter from a primordial radionuclide, in K-Ar, Ar-Ar and U-series dating.

$^{14}\text{C}$  is the most widely used of these chronometers. In the late 1940s, the development of radiocarbon dating by detection of the  $^{14}\text{C}$  residual activity revolutionised archaeology providing a precise and direct measurement of the time scale for the development of human activities during the late Quaternary. In particular, radiocarbon dating had a strong impact on the understanding of European prehistory, previously dated only by correlation with the historical chronology of the Near East.

In the late 1970s, the development of direct atom counting by AMS enhanced more than a million-fold the sensitivity of  $^{14}\text{C}$  analysis. Extensive AMS work followed, particularly in the analysis of radiocarbon and other cosmogenic radionuclides for archaeological, geological and environmental applications (Tuniz *et al.*, 1998). Through

the non-invasive analysis of famous artefacts and findings such as the Shroud of Turin (Damon *et al.*, 1989) and the Ice Man (Prinooth-Fornwagner and Niklaus, 1994), AMS has gained widespread public recognition as a dating technique. Also  $^{10}\text{Be}$  has been used for dating in archaeology (Boaretto *et al.*, 2000).

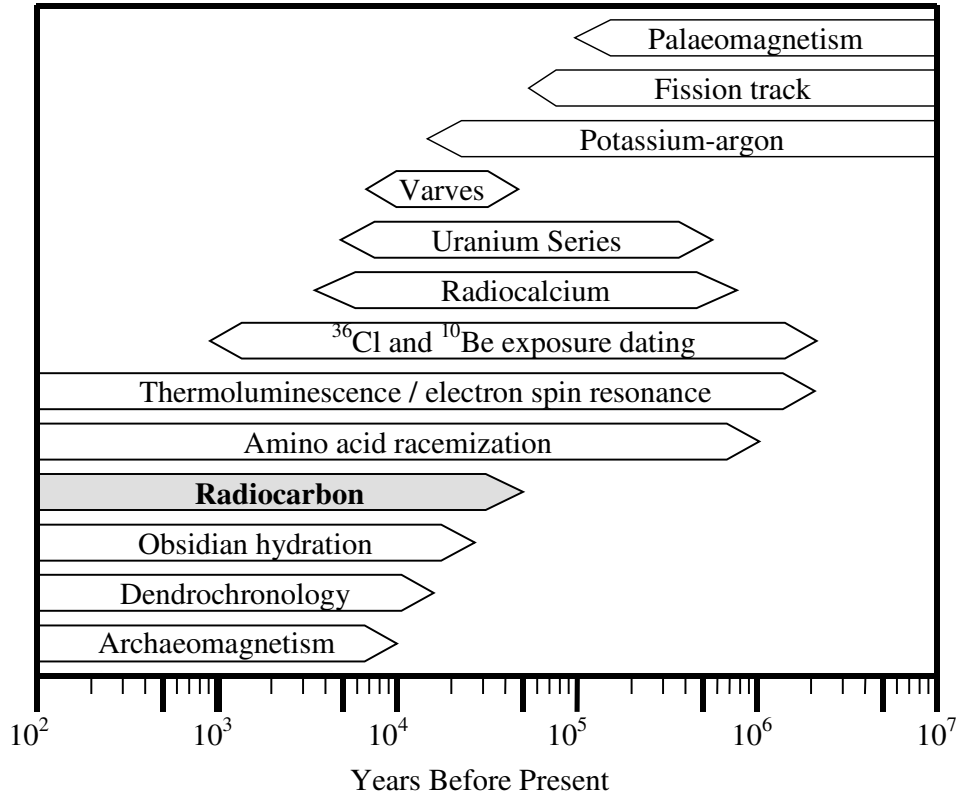


Fig. 1. Comparison of the datable time span of different dating techniques (Tuniz *et al.* 2004).

#### 4. Accelerator microanalysis

Particle accelerators were developed more than seventy years ago for basic research. There has been a major shift in the past twenty years towards their use in the analysis of materials composition and structure for interdisciplinary applications, including cultural heritage. Low-energy ion accelerators, originally constructed for nuclear physics, were turned to other uses as the effort in their initial application faded. They have evolved into specialised tools for ion beam analysis (IBA) and accelerator mass spectrometry (AMS) [Tuniz *et al.*, 1998]. An IBA facility totally devoted to cultural heritage studies has been operational for nearly 20 years at the Louvre museum (Menu, 1990). A laboratory was recently established in Florence with the main purpose of performing applications of nuclear techniques to solve problems related to cultural heritage (<http://labec.fi.infn.it>). Synchrotron accelerators have become dedicated facilities, optimised for emission of bright electromagnetic radiation, an ideal microanalytical probe. Finally, high-energy

proton accelerators are used in spallation sources for producing pulsed beams of neutron to characterise the structure of materials.

#### *4.1 Ion beam analysis*

Ion beams lose energy by ionisation of the atoms composing the target material caused by the interaction of the Coulomb field of the projectile with the atomic electrons and also by nuclear scattering from the nuclei of the atoms. The range of ion beams in materials is short, with relatively well defined end point. By comparison, x-rays are attenuated according to an exponential law and sample a much greater amount of material.

Ion beams are used for trace element determination using the characteristic x-rays produced in the ionization process. Ion beams can also interact directly with atomic nuclei. Nuclear reactions, including elastic and inelastic scattering or Coulomb excitation, are useful to identify specific elements and nuclides in the sample. Concentration of individual elements or isotopes as a function of depth is possible using narrow nuclear resonances and energy loss of ions as they travel in the material.

Detection methods for x-rays,  $\gamma$ -rays, charged particles and neutrons have been developed in parallel with the development of accelerators, ion sources and other instruments necessary for the production of ion beams.

Combination of different ion beam analysis techniques such as PIXE (particle induced x-ray emission) and NRA (nuclear reaction analysis) can be used to determine elemental composition for elements from hydrogen to transuranic elements.

**PIXE** is by far the most widely applied of all ion-beam related techniques used in analysis of cultural heritage materials. It is used for routine detection of elements with atomic numbers greater than perhaps 13, using simple energy-dispersive x-ray detectors. The detection limits are not constant across the periodic table, but are extremely good in many critical regions such as for the transition elements and for heavy elements such as lead and mercury. It can be used in different modes: broad beam for analysis of bulk samples and microbeam for measurement of individual features. Maps of the composition of heterogeneous samples can be obtained by rastering the beam across the sample and making a point-by-point determination of the element present.

**NRA** is used to make sensitive determinations of many specific isotopes. In general, nuclear reactions and elastic scattering are used for detecting specific elements/isotopes throughout the periodic table. However, nuclear reaction analysis is particularly helpful for elements with  $Z < 20$  since the sensitivity of PIXE decreases rapidly for smaller atomic numbers. Strongly resonant nuclear reactions induced by  $^{15}\text{N}$  and  $^{19}\text{F}$  beams are used to probe the concentration of hydrogen as a function of depth. Inelastic scattering of protons (PIGE - proton induced  $\gamma$ -ray emission) is used to detect Li, B, F, Na, Mg and Al. (d,p) reactions can determine the concentration of oxygen and carbon.

**Ion microbeam** analysis uses an ion beam focussed to micron dimensions. Imaging can be performed using the secondary radiation induced by the primary beam, such as in PIXE and NRA, or using the energy loss of transmitted primary ions.

**External beam analysis** is important in cultural heritage studies for the analysis of samples that, for their composition or size, cannot be inserted in vacuum chambers. In this case, the ion beam passes from the vacuum through a few-micron thick polymer window into the room atmosphere. Samples placed at the beamline exit window can be x-ray analyzed in air. This system is very practical for non-invasive and non-destructive analyses of precious artefacts.

#### 4.2 Accelerator Mass Spectrometry

AMS is the analytical technique of choice for the detection of long-lived radionuclides that cannot be practically analysed with decay counting or conventional mass spectrometry (MS). Its advantage is that the ambiguities in ion identification are practically removed, enabling the analysis of isotopic ratios as low as  $10^{-15}$ , a factor  $10^6$  lower than in most MS systems.

Since the atoms and not the radiation resulting from their decay are directly counted, the sensitivity of AMS is unaffected by the half-life of the isotope being measured and detection limits at the level of  $10^6$  atoms are possible. Compared to the decay counting technique, the efficiency of AMS in detecting long-lived radionuclides is  $10^5 - 10^9$  times higher, the size of the sample required for analysis can be  $10^3 - 10^6$  times smaller and the measurement can be performed 100 to 1000 times faster. To highlight the difference between decay counting and atom concentration analysis, consider 1 g of modern carbon containing  $6 \times 10^{10}$  atoms of  $^{14}\text{C}$ , which can be measured by decay counting with 1% precision ( $10^4$  decays detected) in 1000 minutes hours. With a high-intensity ion source, AMS can count  $10^4$   $^{14}\text{C}$  atoms in one minute, consuming only 100 mg of the source material.

Van de Graaff tandem electrostatic accelerators are the optimum choice for a variety of AMS applications. Tandem accelerators working between 0.5 and 3 MV have been specifically designed for  $^{14}\text{C}$  analysis (e.g. <http://www.pelletron.com/AMS.htm>). Large tandem accelerators, originally developed for nuclear physics research, are also used to analyse a variety of rare radionuclides, including  $^{41}\text{Ca}$  and  $^{36}\text{Cl}$  (Tuniz *et al.* 1998).

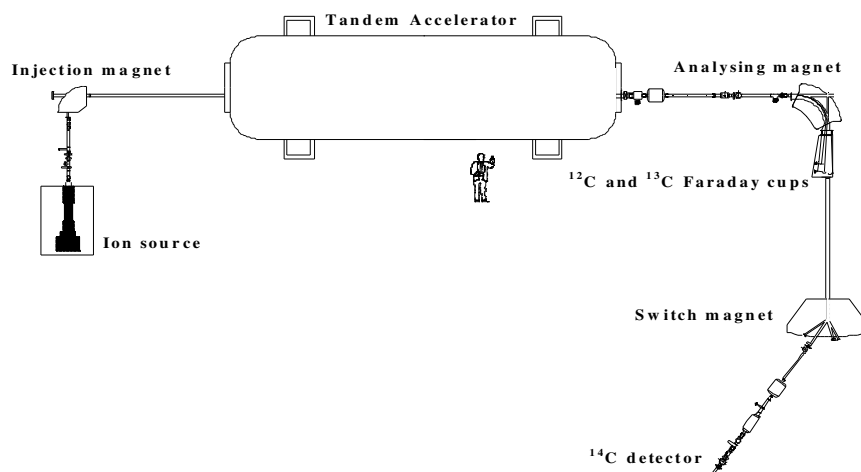


Fig. 2. Scheme of the ANTARES AMS facility as used for  $^{14}\text{C}$  AMS (Tuniz *et al.*, 1997).

## 5. X-ray analysis

The unique properties of synchrotron radiation (SR) make available a portfolio of imaging and spectro-microscopy techniques, using the light spectrum from the infra-red to multi-keV x-rays, of high interest in cultural heritage studies. It is beyond the scope of this paper to review all the available techniques. We will only discuss briefly the tools that are having a large impact in the analysis of art and archaeology, such as microtomography and x-ray fluorescence.

In SR computed micro-tomography ( $\mu$ -CT) the sample is rotated in front of the detector, and several different projections are acquired. A 3D image is then reconstructed by back-projecting the profiles collected in this way with specific mathematical recipes. As for planar imaging,  $\mu$ -CT can be performed both in absorption or phase-sensitive modality (in edge detection or in holography regime), depending on the sample-to-detector distance.

Three-dimensional imaging of specific elements can be obtained by combining microtomography with x-ray fluorescence induced by a scanning micron size beam (XRF).

The development of these methodologies is based not only on the availability of third generation x-ray facilities, but also on recent advances in computer power, both in terms of calculation speed and memory, considering that images corresponding to many gigabytes of data can be collected in few minutes of irradiation time.

A vast range of portable systems for x-ray analysis is becoming commercially available thanks to progress in compact x-ray detectors and electronics, allowing fast, in-situ and non-destructive analysis of materials in art and archaeology. Novel instrumentation includes miniature x-ray sources, pocket-size multichannel analyzers and compact CZT (cadmium-zinc-telluride) detectors. Portable systems can be designed for multiple analyses, including x-ray fluorescence, x-ray diffraction and microtomography. They are increasingly used in museums, galleries and restoration institutions for precious and non movable objects. Leonardo's Mona Lisa was recently studied with an x-ray portable system by a the group from the Centre de Recherche et de Restauration des Musees de France. This master work is taken out of the protecting glass box only once a year for restoration studies and the group had three hours for the experiment. The objective was to study Leonardo's methods to create perceptions of depth and volume, using different layers of translucent paint.

## 6. Neutron analysis

Neutrons can penetrate materials deeply, making them valuable analytical probes for non-destructive analysis of cultural heritage objects. They are produced by fission in nuclear reactors or by spallation reactions in high energy proton accelerators. New portable neutron sources are becoming available and make neutron-based methodologies more accessible to museum curators, restoration specialists and archaeologists. The activation induced during neutron irradiation increases the risks compared to x-rays, particularly in the analysis of certain metals such as gold and silver.

Digital radiation detection systems such as neutron sensitive imaging plates and CCD camera systems are advantageous compared to film methods: exposure time can be much shorter and imaging algorithms can be used for imaging improvements.

Neutrons have higher penetration than x-rays for many materials relevant to cultural heritage, such as metals. The most widespread imaging methods are neutron radiography (NR) and computed tomography.

The group at the Paul Scherrer Institute, in collaboration with Swiss museums, is involved in the neutron analysis of 150 bronze Roman sculptures. The high lead content of these objects make neutron the only probe to study internal structures (Lehmann *et al.*, 2005). A program of NR analyses has been carried out by the National Museum of Slovenia using the Ljubljana TRIGA Mark II research reactor. The scope of these analyses is for conservation purposes and to study the manufacturing technology (Rant *et al.*, 2006).

## 7. Some case Studies

### *Works of art*

The analysis of paintings at the micro- and nano-metre scale using a combination of methodologies such as transmission electron microscopy, atomic force microscopy, ion beam analysis and synchrotron radiation spectroscopy showed the innovative methodologies developed by Dutch artists in the 17<sup>th</sup> century in their representations of Nature (Menu, 2006). In another study, SR has been used for K-edge imaging of paintings. In this method, two images are taken at two different energies, above and below the threshold (K-edge) energy of the element of interest. The subtraction of the two images provides the elemental map. The hidden brush strokes of the great French painter Eduard Manet were revealed using this method to image barium, lead and mercury, ingredients of a pigment lengthener used by painters during the 19<sup>th</sup> and 20<sup>th</sup> century. Scientist could re-construct Manet's techniques to reach the final effect (Krug, 2006).

PIXE and SR-XRF have been used to study the drawing book of Albrecht Durer, created by the German painter in 1521. Trace elements detected with PIXE provide information on the genesis of his creation, including the origin of the materials used and the chronology of his creations. For example, all drawings show the same chemical composition, related to the special silver point used (90% silver, 10% copper and traces of zinc), but one of the drawings, the 'portrait of a young man with a fur hat' was made with another point (83% silver, 12% copper, 5% zinc) (Menu, 2006).

The Bradshaws are Australian Aboriginal rock paintings with a unique style characterised by elegant and graceful figures with many ornaments and accoutrements. An example is shown in figure 3. Paintings of this style are found in the Kimberley region in the north west of the state of Western Australia. The paint colour is usually a light mauve or mulberry. These figures were first reported by early explorer Joseph Bradshaw who, accompanied by his brother, surveyed this region in 1891. The paintings are so unusual and distinctive that there has been much speculation and debate concerning their origins and meanings. Some researchers have gone as far as to suggest origins other than



the ancestors of modern indigenous Australians. Recent fieldwork in the region started in 1994 with the aim of providing absolute dates for the Kimberley rock art sequence. Small samples of pigments, beeswax, and associated mineral crusts have been collected and are being dated by AMS, providing the first age estimates for the well-known Bradshaw painting style. The pollen contained in mudwasp nests, which overlie or underlie Kimberley rock paintings, can be dated by AMS, providing minimum or maximum ages for rock paintings (Roberts *et al.*, 1994). Two measurement techniques were involved in this investigation – radiocarbon AMS and OSL, finding a minimum age of 17,000 BP. So the origins of the Bradshaw paintings are still unknown and further measurements to establish their ages will be required.

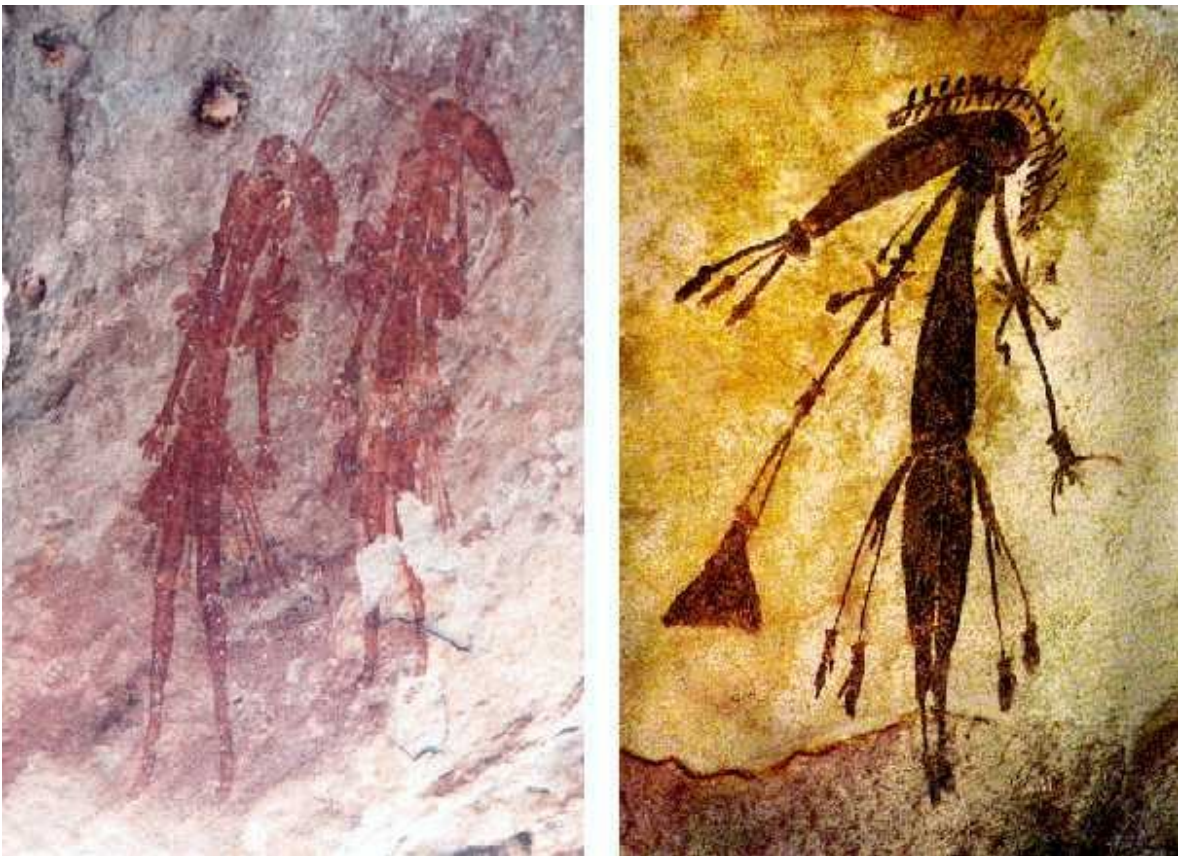


Fig. 3. Computer enhanced “Tassel” Bradshaw figures showing their ornate form of dress with rear triple tassels, bangles, elbow bands, cummerbund waistbands with pubic aprons and complicated head-dresses. A mud-wasp nest can be seen on the rock face just above the left hand figure. Photo: courtesy of Graham Walsh, Takarakka Rock Art Research Centre (Tuniz *et al.*, 2000).

## *Controversial archaeology*

### The Venafro chessmen

The Venafro chessmen, discovered in 1932 in the southern Italian necropolis of Venafro, are among the most controversial chess-related archaeological finds of this century. For more than 60 years, archaeologists have formulated a variety of hypotheses to explain how bone chess pieces of Arabic shape were discovered in a tomb of Roman age. Some scholars claimed that the chessmen were indeed of Roman origin. The chess pieces are preserved in the Archaeological museum of Naples, where a bone fragment of 2 grams was collected for AMS analysis. AMS radiocarbon measurements yielded a calibrated age of 885-1017 AD (68 % confidence level) (Terrasi *et al.*, 1994), supporting the view that this game was introduced to Central Italy during the Saracen invasions of the 10<sup>th</sup> century AD.

### The Iron Crown

The Iron Crown (see figure 4) of the first Holy Roman Emperor, Charlemagne, is held in the Cathedral at Monza, near Milan in Italy. The origin and age of the crown, later used to crown Napoleon Bonaparte, are uncertain. Historical records place its origin between the Roman and Middle Ages, a spread of several centuries. In 1996, it was discovered that the precious stones were held in place by a mixture of clay and beeswax, which provided enough carbon for AMS radiocarbon dating. The analysis performed at ANTARES yielded an age between 700 and 780 AD (Milazzo *et al.*, 1997), consistent with the historical date for the crowning of Charlemagne, 800 AD, on Christmas Night.



Fig. 4. The Crown of Charlemagne

### Donatello's glue

The Annunciazione Cavalcanti (Cathedral of Santa Croce, Florence) is one of the best known creations of the Italian sculptor Donatello (1386-1466 AD). The sculpture is decorated with a group of terra-cotta cherubs. The base of one of these figures has large cracks that had been subsequently repaired with a resin glue. It is not known when the

accident occurred. Our date for the glue, 1331-1429 AD (68 % confidence level), proved that the restoration had been performed during the lifetime of the artist. The breakage and repair may therefore have happened when the work of art was created. Italian scholars believe that the cherub cracked because it was not hollowed out before firing and that the repair was carried out by Donatello himself, after damaging the statue in the kiln.

### The conquest of Peru

The manuscripts “*Historia et Rudimenta Linguae Piruanorum*” and “*Exsul Immeritus Blas Valera populo suo*”, which were found in the family papers of Neapolitan historian Clara Miccinelli, are commonly known as the “Miccinelli documents”. They discuss events and people associated with the Spanish conquest of Peru.

In addition to details about reading literary *quipus* - Inca documents which were written using a combination of textile ideograms and knots - “*Historia et Rudimenta Linguae Piruanorum*” (History and Rudiments of the Language of the Peruvians; Laurencich Minelli *et al.*, 1995) includes the incredible claims that Pizarro conquered the region after having Inca generals poisoned with arsenic-tainted wine and condemned the Inca emperor, Atahuallpa, to death instead of granting him an audience with the King of Spain. The account departs markedly from the long-held version of the event – that Atahuallpa was put to death for ordering the execution of his brother and rival. a letter from Francisco de Chavesa *conquistador* and chronicler on Pizarro’s expedition: The letter, dated August 5, 1533, was addressed to Charles V, King of Spain, and is the source of the accusations already suggested in “*Historia et Rudimenta Linguae Piruanorum*” and “*Exsul Immeritus Blas Valera populo suo*”.

The historical significance of the discovered material is immense and historians considered it to be of primary importance to verify the authenticity. As a part of a worldwide research collaboration we performed the radiocarbon dating of five samples associated with the Miccinelli documents (Zoppi *et al.*, 2000). The results showed that, with a high degree of confidence, the wax used to seal the letter to the King of Spain originated earlier than 1533, the date on the letter. Similarly, the wax used for the box containing the agreement allowing Valera to write “*Nueva Corónica y Buen Gobierno*” under a false name, most probably originated before 1618, the accepted completion date of this important document.

### *Prehistory and human evolution*

Palaeoanthropologists have recently discovered the power of synchrotron radiation analysis. The use of x-ray microtomography to determine non-destructively the internal structure of skulls, teeth and other fossil remains is revolutionising the field of palaeoanthropology and opening the possibility of studies that were not possible until recently.  $\mu$ CT analyses are increasingly used to provide less subjective data related to the anatomical characters of human remains. The image contrast can be dramatically improved using new imaging methodologies including phase contrast radiography, microtomography and holotomography. The intense monochromatic beams from third

generation SR facilities allow fast data acquisition with micron resolution and avoiding beam hardening effects (Tafforeau *et al.*, 2006; Mazurier *et al.*, 2006)).

Human teeth are one of the best preserved types of evidence available for human evolution studies. They provide crucial information on development, diet and health. The use of synchrotron radiation  $\mu$ CT was applied to the teeth of *S. tchadensis* to demonstrate his morphological affinities with the human clade ( Brunet *et al.*, 2005).

SR microtomography has recently provided for the first time information on internal tooth microstructure for *Homo nenderthalensis*. The results show that the timing of molar crown and root completion in Neanderthal is similar to that of modern humans, but they have more complex enamel-dentine junction morphology (Macchiarelli *et al.*, 2006).

## 8. Conclusions

Scientists apply tools and methods of increasing sophistication in studies of cultural heritage, but we should not disregard the intangible aspects of human culture that fall under the scrutiny of art historians, philosophers, ethnographers and other scholars in the humanities. Scholars from these different branches of human knowledge should work together, with the *caveat* that many of the models proposed by the humanities scholars are difficult to verify with the scientific method.

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