Spondylus in Prehistory

New data and approaches

Contributions to the archaeology of shell technologies

Edited by

Fotis Ifantidis
Marianna Nikolaidou

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TABLE OF CONTENTS

INTRODUCTION

A Volume on *Spondylus*
Marianna Nikolaidou & Fotis Ifantidis
3-8

I – SPANNING SPACE AND TIME IN *SPONDYLUS* STUDIES: ARTIFACTS, SYMBOLS, IDENTITIES

II – VIEWS FROM THE "THRESHOLD": *SPONDYLUS* TECHNOLOGIES IN THE AEGEAN

III – Reconstructing Lives: Archaeometric and Experimental Analyses

Concluding Commentary

LIST OF CONTRIBUTORS

Esteban Álvarez-Fernández
Departamento de Prehistoria, Historia Antigua y Arqueología, Universidad de Salamanca
C. Cerrada de Serranos S/N, E-37002 Salamanca, Spain
E-mail: epanik@usal.es; estebanalfer@hotmail.com

Álvaro Arrizabalaga
Departament of Geography, Prehistory & Archaeology, University of Basque Country
C/ Francisco Tomás y Valiente s/n. 01006, Vitoria, Spain
E-mail: alvaro.arrizabalaga@ehu.es

Maria Angelica Borello
Département de Géographie, Faculté des Sciences Economiques et Sociales, Université de Genève
Uni Mail, 40 Bd du Pont-d'Arve, CH-1211 Genève 4, Switzerland
E-mail: borelloarch@yahoo.fr

Benjamin P. Carter
Department of Sociology & Anthropology, Muhlenberg College
2400, Chew St. Allentown, PA 18104-5586, Pennsylvania, USA
E-mail: bcarter@muhlenberg.edu

John C. Chapman
Department of Archaeology, Durham University
DH1 3LE, Durham, United Kingdom
E-mail: j.c.chapman@dur.ac.uk

Piroska Csengeri
Herman Ottó Museum
Görgey Artúr u. 28, H-3529, Miskolc, Hungary
E-mail: csengerip@gmail.com

Katerina Douka
Research Laboratory for Archaeology & the History of Art, University of Oxford
Dyson Perrins Building, South Parks Road, OX1 3QY, Oxford, United Kingdom
E-mail: katerina.douka@rlaha.ox.ac.uk

Bisserka I. Gaydarska
Department of Archaeology, Durham University
DH1 3LE, Durham, United Kingdom
E-mail: b_gaydarska@yahoo.co.uk

Šárka Hladilová
Institute of Geological Sciences, Faculty of Science, Masaryk University
Kotlářská 2, 611 37, Brno, Czech Republic
Department of Biology, Faculty of Education, Palacky University
Purkrabska 2, 77140, Olomouc, Czech Republic
E-mail: sarka@sci.muni.cz

Fotis Ifantidis
Aristotle University of Thessaloniki; 16th Ephoreia of Prehistoric & Classical Antiquities – Thessaloniki Metro
94, Theagenous Charisi str., 54453, Thessaloniki, Greece
E-mail: fotisif@hotmail.com; ifantidi@hist.auth.gr

María-José Iriarte
Departament of Geography, Prehistory & Archaeology, University of Basque Country
C/ Francisco Tomás y Valiente s/n. 01006 Vitoria, Spain
E-mail: mariajose.iriarte@ehu.es

Jan John
Department of Archaeology, University of West Bohemia in Pilsen
Sedláčkova 15, 30614, Plzeň, Czech Republic
E-mail: jjohn@kar.zcu.cz

NINA KYPARISSI-APOSTOLIKA
Ephoreia of Palaeoanthropology & Speleology of Southern Greece
34b, Ardittou str., 11636, Athens, Greece
E-mail: nkyparissi@hotmail.com

ROBERTO MICHELI
Soprintendenza per i Beni Archeologici del Friuli Venezia Giulia
9, Viale Miramare, I-34135, Trieste, Italy
E-mail: roberto.micheli@beniculturali.it

MARIANNA NIKOLAIDOU
Cotsen Institute of Archaeology, University of California, Los Angeles
1748, Orangewood Ln, Arcadia, CA 91006, California, USA
E-mail: marianna@ucla.com

MARIA PAPPA
16th Ephoreia of Prehistoric & Classical Antiquities
Megalou Alexandrou (opposite to Poseidonion) str., 54646, Thessaloniki, Greece
E-mail: mpappa@culture.gr

ZSUZSANNA SIKLÓSI
Eötvös Loránd University, Institute of Archaeological Sciences
Múzeum krt. 4/B, H-1088, Budapest, Hungary
E-mail: siklosizs@gmail.com; siklosi.zsuzsanna@btk.elte.hu

EVANGELIA SKAFIDA
Archaeological Museum of Volos
1, Athanassaki str., 38001, Volos, Greece
E-mail: eskafida@gmail.com

STELLA SOUVATZI
Hellenic Open University
2, N. Plastira str., 13561, Athens, Greece
E-mail: stellasouvatzi@hotmail.com

TATIANA THEODOROPOULOU
The Wiener Laboratory, The American School of Classical Studies at Athens
54, Souidias str., 10676, Athens, Greece
Equipe de Protohistoire Egéenne UMR7041 (Archéologie et Sciences de l'Antiquité)
Maison R. Ginouvès, 21, allée de l'Université, 92023, Nanterre, France
E-mail: tatheod@hotmail.com

NORMA VALENTÍN MALDONADO
Subdirección de Laboratorios y Apoyo Académico del INAH
Moneda 16, colonia Centro, México D.F. 06060, Mexico
E-mail: nvalentinm@hotmail.com

ADRIÁN VELÁZQUEZ CASTRO
Museo del Templo Mayor
Seminario 8, colonia Centro, México D.F. 06060, Mexico
E-mail: adrianveca@yahoo.com

RENA VEROPOULIDOU
Aristotle University of Thessaloniki; Museum of Byzantine Culture, Thessaloniki
25, Solonos str., 54644, Thessaloniki, Greece
E-mail: verren@hist.auth.gr

BELEM ZÚÑIGA ARELLANO
Proyecto Técnicas de manufactura de los objetos de concha del México prehispánico, Museo del Templo Mayor
Seminario 8, colonia Centro, México D.F. 06060, Mexico
E-mail: belemzu@yahoo.com

C H A P T E R   1 2

THE CONTRIBUTION OF ARCHAEOMETRY TO THE STUDY OF PREHISTORIC MARINE SHELLS

Katerina Douka

Marine shell exoskeletons are influenced by environmental stimuli, which cause incremental banding, annual growth rings and isotopic variations. When humans interact with shellfish by gathering and eating them, or shaping their shells into ornaments, these archaeological remains can, in the hands of scientists, provide rare insights into prehistoric lives and adaptation.

In the past fifty years, shells from archaeological sites have been used in deciphering site chronologies, understanding trade and exchange networks, examining symbolism, body decoration and display, and estimating human dietary adaptation. Additionally, they have provided important information related to the seasonal round of prehistoric activities.

This paper broadly reviews the contribution of science in archaeology through the study of marine shells. One important example is the application of isotope biochemistry to Spondylus *sp. artifacts from Neolithic Europe. The pitfalls associated with these techniques are mentioned and some suggestions for future research are briefly discussed.*

The fusion of advanced scientific methods with material from the archaeological record has radically transformed our understanding of the past. Major research questions, such as inter- and intra-site chronologies; subsistence and dietary patterns; trade and exchange systems; movements of people, artifacts and ideas across space and time; all framed within an interdisciplinary context can be successfully addressed with the help of well-formed scientific approaches. One must note, however, that science itself is not, and will never be, able to resolve any archaeological questions if not supported by well-contexted and carefully excavated archaeological material. The incorporation of this "prior-information" is what makes scientific models successful and reproducible.

Molluscan remains from archaeological sites are archives of significant information whose potential was recognized rather late amongst the archaeological community, but at a time –not surprisingly– broadly contemporaneous with the beginning of scientific archaeology. Brothwell and Higgs's major reference work, *Science in Archaeology: A Survey of Progress and Research* (1969), included four chapters on archaeomalacology and drew attention to the need for further research. This found immediate response in the next decades, which witnessed the publication of papers on shellfish exploitation, either from an archaeological (e.g. Bailey 1975) or ethno-archaeological (Meehan 1982; Trubitt 2003) point of view, on shell provenancing, shell dating, and the elucidation of seasonal patterns in shellfish exploitation (references below).

Analysis of Molluscan Remains

Molluscan shells have been described as "*biological chart recorders*" (Goodwin *et al.* 2003), containing climatic, chronological, metabolic, and other information in the form of elemental and isotopic variation. They are polycrystalline biominerals composed principally of calcium carbonate ($CaCO_3$) with a distinct structure and micromorphology, shape, and texture. The shell carbonate is found in the form of low-magnesium calcite, high-magnesium calcite and aragonite. The latter is the most common form of all, while former is normally a secondary addition, byproduct of diagenetic alteration. As with most types of archaeological remains, the study of the molluscan shell revolves around the examination of different levels of structures. These may be classified at the macroscopic, microscopic, molecular and atomic level, each of which contains information crucial for the interpretation of the finds either as an entity or as part of a larger assemblage.

At the **macroscopic** level, species identification, shape measurements, allometric calculations, quantification (MNI and NISP), observations on the preservation state (color, shape, surface), or technology (when dealing with humanly-modified shell), can be made.

Analysis of structures at the **microscopic** level usually relates to attempts of revealing more detailed information on the age and season-at-death of the remains, their preservation state, taphonomic processes (beach-wear, chemical, and biological attack), technological evidence (e.g. perforation techniques) and use-wear (polishing, striation), as well as the presence of surficial and other residues (i.e. ochre traces) on the shell.

The organic component of the exoskeleton is usually very low (1-5%), thus **molecular** fingerprinting has not been widely used in archaeomalacology, except in the case of the amino-acid racemization (AAR) dating method (e.g. Demarchi *et al.* 2010).

On the other hand, **atomic**-scale data deriving from shell structures have been extremely profitable to archaeological enquiry. The presence and abundance of chemical elements and isotopes provide a valuable tool for deciphering the absolute

age of a specimen, its provenance, the temperature at which it precipitated, as well as metabolic and other physiological activities associated with the animal. Usually, methods of isotopic geochemistry are utilized, and the data are normalized based on international reference standards. Shell biogeochemistry is dictated by the physical and chemical conditions under which the mollusc lived; by biological factors controlling the mineralization process; and, very importantly in the case of archaeological specimens, by post-mortem diagenetic alterations which often occur during deposition (Pirrie & Marshall 1990). This paper is a general review of the status of research on archaeological molluscan shells, at the atomic level, with special reference to the isotopes of carbon (C), oxygen (O), and strontium (Sr).

Carbon

Carbon is found in nature in the form of three main isotopes (^{12}C, ^{13}C, ^{14}C), the first two being stable while ^{14}C (radiocarbon) is radiogenic and will decay at a constant rate, the ^{14}C half-life. During marine shell formation, or biomineralization, all three C isotopes are incorporated within the shell structure. The carbon fixed in the marine shell derives from:

- dissolved inorganic carbon (or DIC) which is directly incorporated by the molluscs during the precipitation of skeletal carbonate,
- respiratory CO_2 stemming from food metabolism.

The extent of environmental versus metabolic contribution to the final carbon isotopic signature of the shell is still incompletely understood, and this has been the focus of much recent biological research (McConnaughey & Gillikin 2008, and references). In biological and environmental studies, ^{13}C is a regularly investigated isotope used to shed light on biomineralization processes, feeding, and other metabolic activities or climatic data contained in the carbonate skeleton (McConnaughey *et al.* 1997).

In archaeology, however, radiocarbon (^{14}C) is by far the most-commonly exploited isotope, used for the direct dating of molluscan remains. The principles of radiocarbon dating have been extensively described in the literature since its development in the late 1940s, and its first application in archaeology shortly thereafter (Aitken 1990; Bronk Ramsey 2008; Taylor 1987; Taylor *et al.* 1994). In brief, the radiocarbon isotope is produced in the upper levels of the atmosphere. Shortly after formation it is oxidized and as $^{14}CO_2$ it enters the Earth's biosphere through exchange with the oceans and photosynthesis of plants and decay begins. All living organisms, including molluscs, take up carbon atoms during ontogeny and reach isotopic equilibrium with the ambient environment. When an organism dies both uptake and equilibrium cease to exist since no carbon, including radiocarbon, is incorporated in the shell any longer. After death the stable isotopes remain unaffected, yet the unstable ^{14}C atoms continue to decay at a constant rate without being replenished. Radiocarbon laboratories measure residual ^{14}C and calculate an age based on the radiocarbon decay equation, using the half-life mentioned previously.

Shell remains from archaeological sites provide valuable substrates for radiocarbon dating. In some contexts, shells may be the only available datable material, for example, in sites along the Mediterranean Rim where bone collagen and charcoal preservation is often extremely poor, or in the case of shell middens. There are, however, several problems that must be addressed prior to obtaining reliable radiocarbon ages from shell carbonates. Some of these include the "marine reservoir effect", the "hard-water effect", the "old-shell problem" as well as the analytical, laboratory-related uncertainties, which are normally accounted and corrected for. Below I will briefly explain some of these.

One of the primary assumptions in radiocarbon dating was that the concentration of ^{14}C within an organism has remained in equilibrium with that of the atmosphere. Of course, this is now known not to be true. In the case of the ocean, complex circulation patterns and variable rates of mixing mean that water may reside in the deep ocean for centuries, becoming highly ^{14}C-depleted (Mangerud 1972). This "old", deep water eventually mixes with modern surficial water and the ^{14}C-deficiency is passed on marine organisms affected by it. As a result, contemporaneous organisms that incorporate marine-derived carbon in their body through diet, metabolic or biomineralisation processes show depleted –older– radiocarbon age (apparent age) with respect to the (true) age of species that derive their carbon solely from the constantly renewed and relatively homogenous terrestrial environment (Ascough 2005). This offset between the true age and the apparent ^{14}C age is called *marine radiocarbon reservoir effect*. A correction is required for all affected species and this was calculated to be around 400 years (Stuiver *et al.* 1986). The global correction, however, does not account for regional oceanic conditions (*local reservoir*) and thus a further adjustment is also applied (e.g. Siani *et al.* 2000).

In addition, brackish species that incorporate dissolved CO_2 or bicarbonates leached out from ^{14}C-depleted sources, such as limestone-dominated areas, demonstrate the *hard-water effect* (Keith & Anderson 1963). This may add several hundred to few thousand years (in extreme cases) to the age of a sample (Bezzera 2000; Hogg *et al.* 1998; Pilcher 1991).

The "old shell" problem (Rick *et al.* 2005), equivalent to the "old wood" problem, is not, strictly-speaking, a deficiency in the radiocarbon method *per se*. This discrepancy is not due to any analytical error but a result of the inbuilt age of a shell that may have been collected long after the death of the mollusc, from fossil outcrops, long-dead beach assemblages or thanatocoenoses. The measured age will reflect the time the organism died and not the period the shell was used and deposited and will, therefore, disagree with the stratigraphic position. If such an issue is suspected or identified, then it can be usually resolved by dating more than one sample from the same context/archaeological site, ideally a short-lived terrestrial material.

Glossary

Biomineral: The product of biomineralization. Biomineralization results in the synthesis and precipitation of inorganic phases in the structures of living organisms. In marine molluscan shells, calcium carbonate ($CaCO_3$) is the dominant biomineral and is usually precipitated within an organic, proteinaceous matrix.

Diagenesis: In palaeontology and archaeology, diagenesis describes all of the biological, chemical and physical processes that occur within the burial environment after the death of an organism. Diagenesis results in the alteration of the original physical and chemical structure of the organism.

Minimum Number of Individuals (MNI): A quantification method for counting the faunal remains found in a site in such a way that only the minimum number of specimens is determined. In zooarchaeology, a simple method of MNI analysis is based in the assumption that two similar bone elements, which do not duplicate a unique skeletal part and do not derive from a demonstrably different size or age group, belong to the same individual.

Number of Individual/Identified Specimens (NISP): The absolute number of identified archaeological remains in a collection. Apart from the absolute number of entities, NISP is influenced greatly by the degree of fragmentation and recovery probability at each site.

Molecular Fingerprinting: The spectrometric examination of molecules and their abundance in a specific environment, in order to identify molecular species, such as proteins, and their diagenesis.

Molluscan Shell Biochemistry: The analysis of the presence and abundance of chemical elements and their isotopic ratios in the molluscan shell. Such elements are usually C, O, Sr, Mg, Ca, Mn and Ba.

Isotope: Atoms of the same element which vary in their mass. They contain the same number of electrons and protons (same atomic number) but different number of neutrons (hence different mass number) in their nuclei. Most elements are found naturally as two or more isotopes, in either a stable or unstable form. Variations in the isotope abundance of an element are caused either by radioactive decay of the unstable isotopes or by isotopic fractionation.

Isotope Fractionation: Refers to the alteration of the natural isotopic ratios of a chemical element. Isotope fractionation occurs through various physical, chemical or biochemical processes as a result of differences in the reaction rates and bond strength of the molecular species. In the case of marine shells, preferential isotope fractionation of the C atoms may occur during the transfer of ^{14}C from atmospheric CO_2, to oceanic HCO_3- and to the incorporation of those atoms into marine plants and subsequently to marine organisms. The fractionation of ^{14}C is twice that of ^{13}C in these processes and since ^{13}C is stable, ^{14}C enrichment will result in younger radiocarbon age unless corrected. This is achieved using measured stable isotope ^{13}C ratios and normalization based on international standards.

Radiocarbon Half-life: Radioactive isotopes decay at a regular rate, which is expressed as the "half-life" constant. Two values are often quoted for the rate of ^{14}C decay. The first is the Libby half-life of 5568 ± 30 years and the second the Cambridge half-life of 5730 ± 40 years. The former is used in the calculation of radiocarbon ages for historic continuity.

Thanatocoenosis: An undisturbed fossil assemblage containing the remains of plants or animals that have once lived in the same biotope and which died and accumulated simultaneously or over a short period of time from a nearby, restricted geographic locality. Such assemblages are often used to infer the composition and species abundance of past habitats amongst a contemporaneous group by comparison against modern analogs.

Trace Elements: The term "trace" refers to those chemical elements which are found in minute quantities in the Earth's crust, and in this case in molluscan shells. Their concentration may be in the order of few 1000s ppm (parts per million) or less.

Set aside the aforementioned issues most of which are well-understood, the reliability of radiocarbon dates from biogenic carbonate depends heavily on the preservation condition of the dated shell and the presence or not of post-depositional contamination. Identifying and measuring the effect of diagenesis, which in most naturally aragonite shells appears in the form of secondary calcite, is extremely important (Chappell & Polach 1972; Grant-Taylor 1972; McGregor & Gagan 2003 and references therein). The ways to do this will be discussed later on.

Oxygen

Oxygen is abundant in the Earth's crust, hydrosphere, atmosphere, and biosphere where it is found in the form of three stable isotopes. ^{16}O is the most common of the three with 99.76% natural abundance, whereas ^{17}O and ^{18}O account for the rest of it (0.038% and 0.2% respectively). The ratio of the most abundant isotopes, $^{18}O/^{16}O$, is widely applied in an array of disciplines ranging from hydrogeology to palaeoclimatology and archaeological science. This ratio varies significantly around the globe due to its direct dependence on parameters such as latitude, longitude, and altitude.

In marine biogenic carbonates, two interconnected factors principally control oxygen isotopic composition; water temperature and water isotopic ratio (Keith *et al.* 1964). Since H_2O evaporation is temperature-dependent, changes in the evaporation rate will lead to $^{18}O/^{16}O$ variation of the water mass too. In that, higher sea surface temperatures (SSTs) will cause higher evaporation rates which will lead, in turn, to isotopically heavier oceanic water. Urey (1947) and co-workers (Epstein *et al.* 1951, 1953; McCrea 1950) were able to demonstrate that since marine molluscs build their shells in constant isotopic equilibrium with their ambient environment, temperature variations of the oceanic water will be reflected in their shells as oxygen isotope variation. Shackleton (1973) and other workers reported that an increase of 1°C in sea-water temperature produces an approximate 0.2‰ decrease of the $^{18}O/^{16}O$ abundance ratio in the molluscan shell. In other words, a variation of 1‰ of the δ^{18}O shell value implies a change of ambient water temperature by about 5°C.

In archaeomalacology, oxygen isotope variability has been used in two ways:

(1) in seasonality studies and palaeotemperature determination. Since molluscan shell grows seasonally, a plot of oxygen isotopic ratios taken at close intervals across the direction of shell growth will reveal the fluctuations in water temperature over the period represented (ideally the lifespan of the organism) and may be correlated to the seasonal cycle (Deith 1985). The information from the shell's last growth band is then correlated to the seasonal profile and will indicate which period of the year the animal was harvested (season-at-death). By analyzing archaeological shell remains it is possible to determine the season(s) during which shellfish were consumed and, by extension, when humans were present at a site (Deith 1988). Following the theoretical and instrumental developments in

the 1950s, Emiliani *et al.* (1964) used archaeological *Trochus* and *Patella* sp. shells from two cave sites, Arene Candide (Italy) and Haua Fteah (Libya), to estimate palaeotemperature based on $^{18}O/^{16}O$ ratios. They found that the climatic change as reflected in shell $CaCO_3$, paralleled that obtained from the fossil fauna and flora and the lithology of the sediments (Bowen 1966). Since then, a large number of investigations have been published worldwide exploring the seasonal character of shell middens and other types of occupation based on oxygen isotopic data and supplementing traditional shell growth ring analyses (among others: Deith 1983, 1985, 1986; Deith & Shackleton 1988; Higham 1996; Killingley 1981; Koike 1973, 1979; Mannino *et al.* 2003; Mannino *et al.* 2007; Shackleton 1973).

(2) in provenancing specimens deriving from isotopically-distinct localities (meteoric vs. oceanic water influence). Keith *et al.* (1964) suggested that local water isotopic variation as reflected in shell carbonates (marine, riverine, and lacustrine shell) could be used to determine the origin of a shell artifact. Shackleton (1970) used the method to determine the provenance of *Cardium edule* specimens found at the Neolithic site of Nea Nikomedeia and, later, Shackleton and Renfrew (1970) explored the source of Neolithic *Spondylus* sp. artifacts again by means of oxygen isotope variation (for a discussion see below). Few similar publications have emerged since (Eerkens *et al.* 2005). This may well be due to the limited precision of the method, the complex nature of its application, the high level of expertise needed, and the high cost.

Strontium

In addition to the study of major elements, trace element geochemistry of biogenic carbonates is a commonly employed area of research. Strontium (Sr) has been one of the most studied trace elements and is widely used as an indicator for climatic conditions and geographic origin. Sr is closely associated with calcium (Ca) and may therefore be found within living tissues, such as shell, teeth, and bone.

Sr is found in nature in the form of four isotopes ^{88}Sr, ^{87}Sr, ^{86}Sr and ^{84}Sr. ^{88}Sr is the most abundant (82%) whilst ^{87}Sr is the only radiogenic isotope produced by beta decay of ^{87}Rb. The abundance of Sr concentration in geological or biogenic samples is measured through the $^{87}Sr/^{86}Sr$ ratio, which essentially utilizes a radiogenic and a stable Sr isotope. The ratio usually falls between 0.703 and 0.740 and although the variations seem to be negligible, modern instruments in the measurement of $^{87}Sr/^{86}Sr$ (mass spectrometers: LC-IPC-MS, TIMS etc.) employed can routinely reach a precision of 0.00002 or ±0.1 ‰. The difference in the $^{87}Sr/^{86}Sr$ ratio observed at different areas of the Earth is mainly a result of bedrock age and composition, that reflect the variations in the original Rb/Sr ratio of the geological setting: older rocks, such as granites, will give values as high as 0.740 whereas younger volcanic rocks will be around 0.703 (Sealy 2005). During rock weathering processes, Sr from the bedrock leaches into the local soil and is subsequently incorporated into the plant and animal tissues growing in the area by substituting for Ca. It has

174

been suggested that no fractionation occurs during the transfer of Sr into the biosphere, thus the relative values can be used, more or less, directly to identify the location where an organism subsisted and grew. The $^{87}Sr/^{86}Sr$ ratio of seawater has varied significantly in geological time and an isotopic trend curve for the Phanerozoic has been compiled (Elderfield 1986; McArthur *et al.* 2001; Veizer 1989; Veizer *et al.* 1997). Modern seawater has an average value of 0.709, influenced by the input of ^{87}Sr-enriched continental byproducts and underwater volcanic action. $^{87}Sr/^{86}Sr$ values of marine molluscs reflect the isotopic composition of the ambient water during growth. Yet, the values may not be necessarily exactly the same, since Sr uptake mechanisms directly depend on shell mineralogy (aragonitic structures are usually enriched in Sr in comparison to calcitic ones that discriminate against the element) and other factors such as water salinity, temperature, diet, and growth rate (Anadon *et al.* 2002; Pilkey & Goodell 1963).

In archaeology, application of $^{87}Sr/^{86}Sr$ analysis aims at reconstructing residential mobility (Grupe *et al.* 1997; Price *et al.* 1994; Bentley 2006 for a review), palaeodietary (Sealy *et al.* 1991; Sillen & Kavanagh 1982) and material exchange patterns, where the $^{87}Sr/^{86}Sr$ value of a human tissue or an artifact is compared to the baseline values of the regional geology and hydrology. If the values agree, the individual or the object is considered local; if not, exotic. In archaeomalacology, $^{87}Sr/^{86}Sr$ has been used as an indicator of climate conditions, particularly water temperatures and palaeosalinity at the time of shell formation (Ingram & Sloan 1992; Widerlund & Andersson 2006). In addition, Sr ratios have been used in sourcing the geographic origin of a specimen (Shackleton & Elderfield 1990) (see below). Recently, Vanhaeren *et al.* (2004) used the method as a chronological proxy and, ultimately, a tool for provenancing five *Dentalium* sp. beads found in association with the La Madeleine child burial (10,190±100 BP). By measuring the isotope ratios of the beads and comparing them to values of shells from nearby fossil sources, they found that the ornaments used in the burial were most probably of recent origin and must have been collected from distant beaches and not from Miocene outcrops situated much closer, in the periphery of the site.

Pitfalls

Most archaeometric approaches based on the isotope variation of molluscan shells rest on the assumption that the elements within the sample have precipitated in vivo and no post-mortem alteration has occurred. However, this needs always to be tested prior to performing modern scientific analyses since the preservation state and integrity of an archaeological shell are of paramount importance in obtaining reliable and reproducible data from this type of material. This is because physical, chemical and biological factors cause post-mortem (diagenetic) changes in marine carbonates. Diagenetic alteration normally involves (i) the preferential leaching of certain elements to the depositional environment which results in alteration of the original biochemical composition and/ or (ii) the incorporation of exogenous material either on the surface or within the shell structure, in the form of secondary $CaCO_3$.

Of the three main calcium carbonate polymorphs found in shells, aragonite and high-magnesium calcite are metastable and are expected to become reduced to the most stable form, low-magnesium calcite, under certain depositional conditions. This simply means that when conditions are favorable, marine shells composed of the former two minerals will slowly dissolve and be replaced by neomorphic calcite. The isotopic composition of the newly precipitated mineral is expected to be different than that of the biogenic carbonate (Maliva 1998) since it was added post-mortem, under different environmental and geological conditions and without the influence of biomineralization controls. The data obtained from diagenetically altered archaeological shells will be different than those of pristine specimens and most possibly erroneous since they will no longer reflect the seawater composition in which they grew, but that of the depositional environment. Diagenesis has an effect on all three chemical elements (C, O, Sr) discussed above.

In the case of C isotopes, and particularly in radiocarbon dating, modification of the original isotopic ratio it simply means erroneous dates being produced. The issue was identified at the very early stages of shell radiocarbon dating (Berger *et al.* 1964; Chappell & Polach 1972; Grant-Taylor 1972; Mangerud 1972; Thommeret & Thommeret 1965) and is still investigated today (e.g. Bezzera *et al.* 2000; Chiu *et al.* 2005; Webb *et al.* 2007; Douka *et al.* 2010). Meteoric diagenesis also impinges on the reliability of $^{18}O/^{16}O$ values from marine shells and will strongly affect any proxies (SSTs, season-at-death) derived from the study of their oxygen isotope composition. The concentration of Sr in shells is also affected by post-depositional processes and the problem is more pronounced when the values of the original and diagenetic environments are significantly diverse (Brand 1991; Enmar *et al.* 2000). Isotope exchange with Sr from percolating groundwater, the deposition of secondary minerals with a different 87Sr/86Sr ratio from the host biogenic carbonate and the diffusion of radiogenic ^{87}Sr from clay minerals and other Rb-rich phases into the shell are processes that will alter the authentic Sr concentration (Tremba *et al.* 1975). It has been suggested that Sr is preferentially removed from aragonite (Siegel 1960), the most common mineral phase in marine shells, which adds a further complication.

It becomes clear, that any post-mortem alteration of the molluscan shell will mask the original isotopic information archaeologists and geochronologists seek to extract. Precise identification of the presence and the extent of diagenesis are therefore vital in any modern archaeometric attempt concerning this type of material. Combination of X-Ray diffraction (XRD) and Scanning Electron Microscopy (SEM) is the best approach for the detection of diagenesis and the assessment of the mineralogical and structural integrity of the molluscan shell prior to further analyses. Once diagenesis has been identified, a further step is to isolate any exogenous contaminant, normally in the form of low-magnesium calcite, and remove it from the original phase. A new pretreatment protocol (CarDS: Carbonates Density Separation) was recently developed at the Oxford Radiocarbon Accelerator Unit (University of Oxford,

175

U.K.) (Douka *et al.* 2010) to separate aragonite and high-magnesium calcite from secondary calcite using their specific gravity differences.

The application of geochemical analyses on archaeological shells has great potential for archaeology but it is not always straightforward and the intrinsic limitations posed by the material need to be taken into account. Surely, further work will elucidate aspects of shell diagenesis and the most efficient ways to obtain reliable isotopic data, be it C, O or Sr.

Case Study: *Spondylus* sp. Artifacts and Isotope Analysis

In the European Neolithic, scientific approaches have helped to expose trading patterns in regions that are now regarded as interlinked local systems, with economies not necessarily closely coupled together but where networks of trade and exchange emphasized their cultural identities. *Spondylus* artifacts played a significant role in the formation of this notion, despite the fact that our knowledge of many aspects surrounding this type of artifacts is rather limited. Recently, Dimitrijević and Tripković (2006) very eloquently spelled out the majority of the issues pertinent to the full understanding of the "*Spondylus* phenomenon". Assumptions on the chronological contemporaneity of artifacts, attribution to an extensive pan-European exchange network, even single "workshop" derivation, have often been suggested, but they have never been adequately tested or proven.

Two publications relevant to Neolithic *Spondylus* artifacts have appeared in the literature, published twenty years apart. They revolve around the examination of this type of material using isotope techniques and, although technically superseded, are still considered seminal.

The first, by Shackleton and Renfrew (1970), titled *Neolithic trade routes re-aligned by Oxygen Isotope Analyses*, intended to trace the origin of *Spondylus* artifacts in the Balkans. The most likely water bodies were the Aegean or the Black Sea. The authors measured the isotopic compositions of nine archaeological samples from four archaeological sites (Varna, Sitagroi, Vinča, Gradeshnitsa) and compared them to the theoretically estimated or previously published $^{18}O/^{16}O$ values from each locality. In order to increase the validity of their results, three modern shells from the Black Sea were analyzed and their $^{8}O/^{16}O$ ratios were compared to the values of the archaeological specimens. The results suggested that the slightly enriched values from the archaeological *Spondylus* artifacts were not likely to derive from the Black Sea. Therefore, according to the authors, the Mediterranean and specifically, the Aegean is a more likely source. This suggestion had a significant impact upon the field, and has since been used widely as an example of the large-scale trading routes operating in the Neolithic and, essentially, the construction of the "*Spondylus* culture".

Two decades later, J. Shackleton and H. Elderfield (1990) used $^{87}Sr/^{86}Sr$ ratios to define whether the *Spondylus* artifacts from prehistoric Europe were of recent (Holocene) or fossil age, hence of distinct geographic origin. As previously mentioned, $^{87}Sr/^{86}Sr$ values of marine shell change with geological time and heavily depend on the local geology during growth. The authors analyzed three archaeological shell artifacts from Central Europe (Eggenburg, Emmersdorf a.d. Donau, Mórágy–Tüzködomb) and a fossil specimen of *Spondulus* crassicosta, from Middle Miocene deposits. Their results indicated that the *Spondylus* artifacts were of recent origin, isotopically distinct from the fossil shell material. This find corroborated the results of the first study, as it showed that *Spondylus* from the Mediterranean were being used and transported through long exchange networks.

Despite their success and originality, these studies are not without potential problems. The datasets used in both papers were very small which makes the results difficult to evaluate and, of course, invites further testing of their important conclusions. In addition, although both studies explicitly mention the possible sources for error deriving from diagenesis, in fact they do not account for it. If diagenesis has occurred, both O and Sr values may be slightly shifted towards the values of meteoric groundwater, possibly affecting the final conclusion. Since *Spondylus* shell bodies are found in nature mainly in the form of aragonite, the application of XRD analysis coupled with SEM imaging could have easily ruled out this possibility. While these technologies may not have been widely available few decades ago, especially without involving serious damage to the artifacts, currently they are readily accessible and easy to perform. XRD analysis requires only few milligrams of powder material and SEM imaging can be entirely non-destructive, especially when using low-vacuum SEM (LV-SEM) or environmental SEM (ESEM) that do not require sampling of the analyzed specimen or coating with a conductive material.

Summing up, with regards to the case of Neolithic *Spondylus* artifacts, the first scientific attempts to contribute to the issue were successful but not many have followed up on this example. According to the suggestions put forward by Shackleton & Renfrew (1970) and Shackleton & Elderfield (1990), new direct radiocarbon dates, stable isotopes and trace element measurements of *Spondylus* artifacts could well shed light on issues such as (i) the timing and starting point(s) of the "*Spondylus* culture", (ii) its geographic and chronological distribution throughout the Balkans and Central Europe, (iii) inter-site and inter-regional contemporaneity; all of which will clarify the nature of these objects. Are we, for example, dealing with independent, chronologically and stylistically, inventions across space and time or with long-established trade patterns and transcultural fashions?

Conclusions

This article has attempted to outline the major applications of carbon (C), oxygen (O) and strontium (Sr) isotopes on archaeological marine shell. The main areas of relevance are dating, sourcing and seasonality studies, as well as palaeoenvironmental and palaeoclimatic reconstructions. In no small part, the success or failure of these approaches relies on the

careful detection and removal of diagenetic alteration from the specimens under examination. Failure to achieve this will result in data that are not reliable or reproducible. With the improvement of screening methods (XRD, SEM) as well as mass spectrometric and other analytical techniques (AMS, TIMS, LA-ICP-MS, PIXE) for measuring the isotopic signal of shells, archaeometric approaches pertinent to shell artifacts have become almost non-destructive, forming with curatorial concerns and museum collections managing.

Future developments in analytical and instrument precision, screening methods and even smaller sample size requirements, will certainly result in further improvements in the application of these types of techniques on archaeological molluscan remains. An important area of future work is in the mapping of isotopic baselines for comparison with archaeological samples. At present many of these are coarse or poorly modeled, and geographically restricted in many cases.

Returning to the *Spondylus* artifacts, their ubiquitous presence across exceptionally broad geographic areas of Neolithic Europe (e.g. Séfériadès 2009: fig. 8.3), makes the construction of a comparable, pan-European working framework to investigate this phenomenon difficult to achieve. The exact meaning and value of Neolithic *Spondylus* artifacts may be long lost in time, but several aspects underlying the mode of shell procurement and manufacture, chronology, routes of circulation, use, and discard, remain to be fully explored in a systematic way. It is to be hoped that well-formulated scientific questions and interdisciplinary approaches will unravel this fascinating phenomenon of cultural "invention" and/or "invasion" across prehistoric Europe.

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