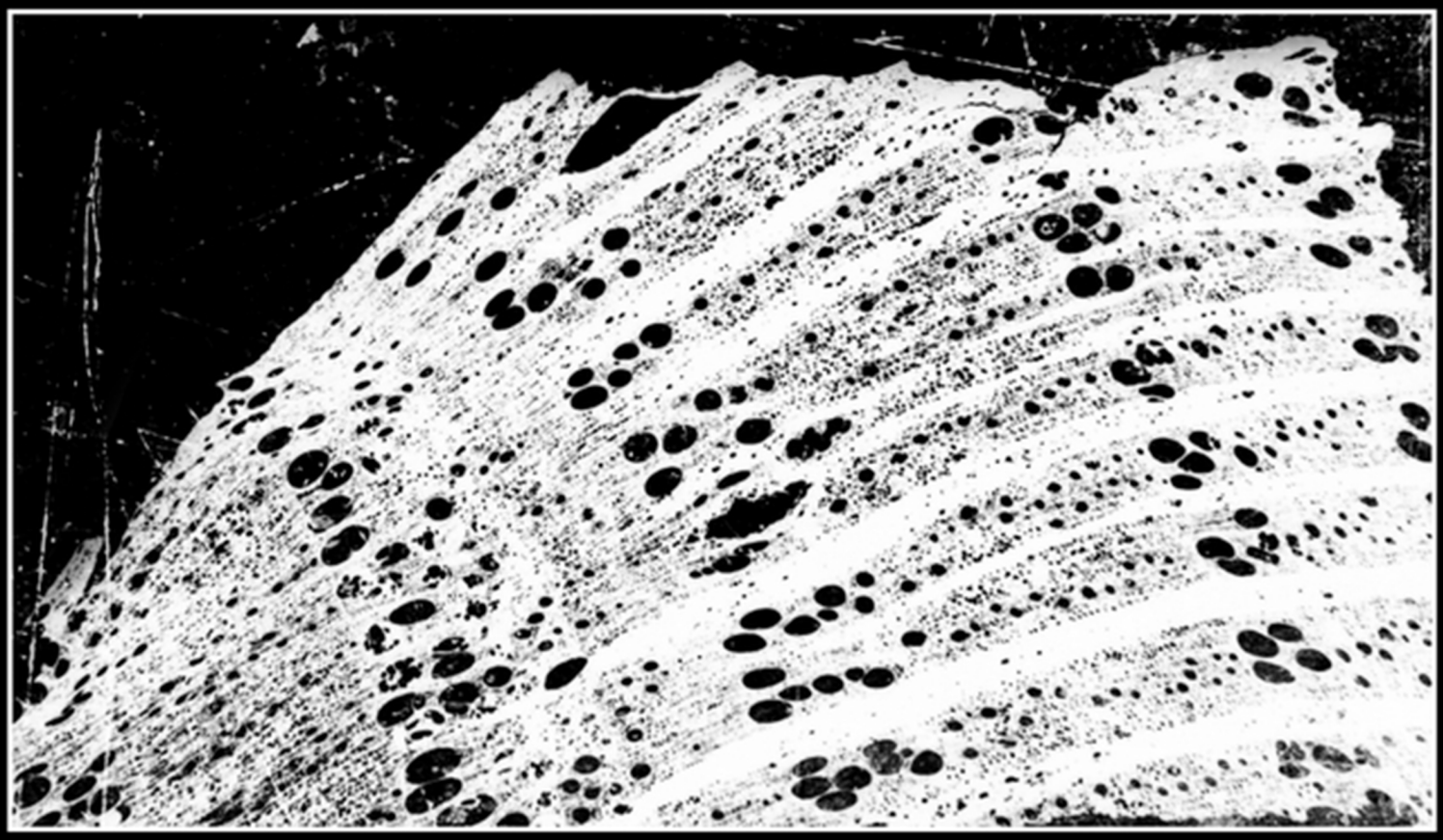

# Archaeological Soil and Sediment Micromorphology

Edited by
Cristiano Nicosia & Georges Stoops

WILEY Blackwell

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## Abbreviations

| | |
|---|---|
| BLF | blue light fluorescence |
| EDS | energy dispersive X-ray spectroscopy |
| EDX | energy dispersive X-ray spectroscopy |
| EPMA | electron probe X-ray microanalysis |
| FTIR | Fourier transform infrared spectroscopy |
| GC/MS | gas chromatography/mass spectrometry |
| IRMS | isotope ratio gas mass spectrometer |
| LC/MS | liquid chromatography/ mass spectrometry |
| Micro-CT | X-ray microcomputed tomography |
| OIL | oblique incident light |
| PPL | plane-polarized light |
| SEM | scanning electron microscope |
| UVF | ultraviolet light fluorescence |
| VPDB | Vienna Pee Dee belemnite |
| WDS | wavelength dispersive X-ray spectroscopy |
| WDX | wavelength dispersive X-ray spectroscopy |
| XPL | crosspolarized light |
| XRD | X-ray diffraction |
| μ-XRD | X-ray microdiffraction |
| XRF | X-ray fluorescence spectroscopy |

# Introduction

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## I.1 Aims and Structure of the Book

Progress in life and earth sciences (including geoarchaeology) is mainly carried out by collecting data, making comparisons and finally by interpreting differences and similarities. A systematic and synthetic overview of published results is, therefore, very important. Up to now this has not been done for the application of soil micromorphology to archaeological research in general, although several excellent books were published focusing exclusively (e.g., Courty *et al.* 1989) or partially (e.g., Barham & Macphail 1995; French 2003; Goldberg & Macphail 2006) on archaeological soil micromorphology. These publications, however, are based predominantly on case studies and the personal experience of the author(s). None is based on an exhaustive literature review and its systematic exploration by a large number of authors of different backgrounds and nationalities. The present multiauthored book aims to give a comprehensive state of the art, based on a combination of the authors' own experiences, the most up-to-date research, and the existing literature.

The book is divided into three parts. The first part contains relatively short entries, dealing mainly with inclusions or components relevant to archaeological deposits such as charcoal, excrements, bones or metal slags. The second part is composed of larger entries discussing complex combinations of features, such as those observed in ancient ploughsoils, animal gathering enclosures or urban dark earths. In the last part, short entries illustrate the use of (sub)microscopic analytical methods, which are increasingly being used directly on thin sections or on closely related samples.

## I.2 Definition and History of Soil Micromorphology

Soil micromorphology is the study of undisturbed, oriented samples with the aid of microscopic and / or ultramicroscopic techniques, to determine the composition of the constituents and their spatial relationship, with the aim of deducing their genetic and chronological relationships.

The most widely used technique is petrographic microscopy of thin sections (20–30 µm thick) prepared from undisturbed samples impregnated with resin. The petrographic microscope allows examination of the materials in transmitted polarized light (PPL) and under crossed polarizers (XPL). Studies in oblique incident light (OIL) and autofluorescence in blue or UV light are also possible. Apart from observations with the optical microscope, studies of thin sections or undisturbed samples by ultramicroscopic techniques, such as scanning electron microscopy (SEM), energy or wavelength dispersive X-ray analyses (EDXRA or WDXRA), micro X-ray diffraction, and so forth, are possible.

The discipline was developed in the 1930s by the Austrian scientist W. L. Kubiëna (1938). He called it micropedology, but later the term soil micromorphology became more popular. After the Second World War, several European, Russian and North American soil research centres were applying micromorphology, and systems of new concepts and terms were developed by Brewer (1964), FitzPatrick (1984, 1993), Bullock *et al.* (1985) and more recently by Stoops (2003) and Loaiza *et al.* (2015). Quantification of components and features started in the 1960s (Kubiëna 1967) but has been used only sporadically in archaeological studies.

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Interpretation of thin sections can be based, apart from literature studies, on deduction from known facts (e.g., the relationship between an increase in fine clay and the amount of fine clay coatings), or by experiments. In pedological soil micromorphology, experimental work is rather limited, whereas in archaeological micromorphology several interpretations rely on experimental work, especially regarding heating and combustion, activity areas, and land-management practices. The microscopic study of ethnoarchaeological and ethnographic analogues has also played a significant role.

It is important to realize that the micromorphology of natural soils and sediments and of archaeological deposits are strongly interrelated. Archaeologists need to understand the micromorphological features observed in natural soils in order to discern the influence of anthropic activities. On the other hand, several features are more strongly expressed in archaeological materials, and are well known by archaeologists but often overlooked by soil scientists in natural soils. This book has therefore been organized in such a way that soil scientists and Quaternary geologists will also be able to find information important for their research.

## I.3 Micromorphology and Archaeology

The increasing number of micromorphological publications in archaeology is related to the technique's evolution within archaeology, requiring a larger input from natural sciences, especially earth and material sciences. The first known record of the use of micromorphology in archaeology is found in Cornwall (1958). In his manual he explains how to take undisturbed soil samples, how to make thin sections, and the way micromorphology can help to characterize and classify surrounding or underlying soils, based on the systems of Kubiëna (1938 and 1953 – for a concise description of these systems see Stoops 2009).

Earth scientists first made thin sections of isolated archaeological deposits and objects out of curiosity, often in the frame of geological or pedological surveys. Amongst such early works one can cite the papers of Dalrymple (1958) on archaeological sediments and associated palaeosoils, Zachariae (1967) discussing the presence of earthworm excrements in Neolithic and Roman settlements, Masset & Van Vliet (1974) studying a white calcareous cover in graves, Mathieu & Stoops (1972) describing the heat-transformed soil around a medieval lime kiln, and the pioneering study of Romans & Robertson (1975) focussing on old cultivation techniques.

Systematic use of micromorphological techniques in archaeology dates back to the end of the 1970s: in Israel and the United States by Goldberg (since 1979) and in the United Kingdom by Macphail (since 1981). Early examples also include the works of Stoops (1984a and b) on archaeological deposits and mortars and plasters in the frame of excavations (1969–1973) in Anatolia (Turkey), and Haesaerts *et al.* (1983), determining the palaeoevironment of hominids in the Omo Valley (Ethiopia). The work of Courty *et al.* (1989) can be considered as a benchmark, illustrating the many uses of micromorphology in archaeology. A bibliometric study by Stoops (2014) shows an exponentially increasing number of micromorphological publications until the 1990s, followed by stagnation and even a small decrease, parallel with the diminishing interest in soil genesis and classification. It also shows that the relative proportion of archaeological publications using micromorphology strongly increased since 1990 (see Figure I.1). Archaeologists can now be counted amongst the most frequent users of soil micromorphology. It is, however, very difficult to measure the real impact of micromorphology in this way; quantification of publications in the field of archaeology is much more difficult than in most other natural sciences, because many journals used by archaeologists are not included in the Web of Science or similar research tools. Moreover, archaeologists often publish their results in local journals, or, still more difficult to trace, in excavation reports. This means also that many of these papers have escaped peer review.

## I.4 Use of Micromorphology in Geoarchaeology

Micromorphology is used currently to solve a variety of archaeological problems ranging from the identification of specific constituents to stratigraphy interpretation applied to the reconstruction of palaeoenvironments. Roughly, one can distinguish the following fields (i) archaeological materials (i.e., 'artefacts' and 'ecofacts'); (ii) reconstruction of ancient technology; (iii) reconstruction of the archaeological context, microstratigraphy, syn- and postdepositional changes; (iv) impact of man on the environment; (v) reconstruction of the palaeoenvironment by study of sediments and (palaeo) soils. It is, however, not possible to make a strict division of application fields, as they often overlap or are interdependent. For instance, the identification of an animal gathering enclosure (archaeological context) is not possible without the recognition of different excrement types (material studies), and the latter is not possible without a knowledge of plant remains, phytoliths and / or bone fragments.

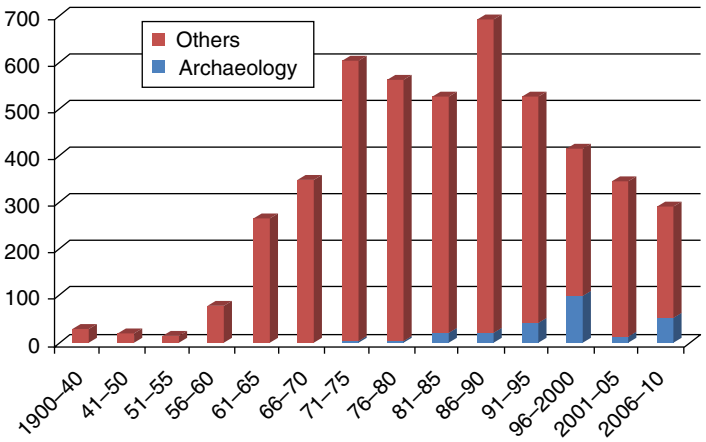


**Figure I.1** Evolution of the number of publications dealing with, or making use of, micromorphology since the beginning of the last century, and proportion of those related to archaeology. Based on Stoops 2014, extended and updated until 2010, totalling 5800 references. The lower numbers for the most recent decade may be the result of incomplete sampling of papers published in journals not retrieved by search programs.

In order to avoid repetition or overlapping with the other chapters of this book, reference is generally restricted here to review papers in this and other books, containing relevant reference material.

### I.4.1 Archaeological Materials

The study of archaeological materials deals with the identification of constituents observed in thin sections. A difference with 'classic' soil micromorphology is that archaeological constituents are produced and often modified by human action, mainly by fire (see for instance Canti 2017a and b and Röpke & Dietl 2017, this book). Especially important are constituents of plant and animal origin. Plant remains (see Ismail-Meyer 2017, this book) are rather similar in natural and archaeological contexts, unless they are charred (see Canti 2017b, this book). Phytoliths of silica, oxalates or carbonates do occur in natural soils (Gutiérrez-Castorena & Effland 2010) but are often more concentrated in archaeological sediments (see Canti & Brochier 2017a and b, and Vrydaghs *et al.* 2017, this book), and can be transformed by heat (Canti & Brochier 2017b, this book). Constituents of biological origin are found in natural soils and are common in archaeological deposits. Examples are bones and teeth (see Villagran *et al.* 2017, this book), biospheroids (Canti 2017c, this book) avian and mollusc shells (see Canti 2017d and e). Shells are concentrated in middens and are often transformed by heat (see Canti 2017a, this book).

Other constituents are, in reality, almost exclusive to archaeological deposits, such as worked stone fragments (Angelucci 2017, this book), metal slags and metallurgy byproducts (Angelini *et al.* 2017, this book), ceramic materials (Maritan 2017, this book) and coal (Canti 2017f, this book).

More specific components, almost unknown in natural soils, are the excrements of herbivores, omnivores and carnivores (Brönnimann *et al.* 2017a and b, this book) and the parasite ova found within them (Pümpin *et al.* 2017, this book). Excrements can sometimes be transformed by heat (Mallol *et al.* 2017, this book; Canti & Brochier 2017a, this book). They yield information on the feeding habits of humans and their domestic animals. Guano is often related to archaeological sites in rock shelters and caves (Karkanas & Goldberg 2010; Karkanas 2017, this book; Mallol & Goldberg 2017, this book).

Descriptions of coarse mineral and rock components of the groundmass are very important in the case of provenance studies, if soils and sediments in the environment were sampled and studied for comparison. A petrographic study of the neighbouring geological resources is often needed to understand which raw materials were available for construction, ceramic manufacturing, metallurgy, and so forth.

### I.4.2 Ancient Technology

One of the aims of archaeological investigations is to gain an insight into ancient manufacturing techniques. This is especially important for periods and / or areas not covered by written documents. Examples of micromorphological studies that contribute to our knowledge of ancient construction are the study of earth building materials (Friesem *et al.* 2017, this book) or turf (Huisman & Milek 2017, this book). The study of calcareous and gypsic mortars and plasters (Stoops *et al.* 2017a and b, this book) contributes not only to our knowledge of building techniques but also to the way mortars and plasters were prepared, starting from limestone or gypsum rock. In the same way, the investigation of metal slag yields information both on metallurgical processes and raw materials (Angelini *et al.* 2017, this book). The study of combustion features gives an insight into the use of fuel and fire (Mallol *et al.* 2017, this book).

For many of these studies a good knowledge of the local natural resources (stones, clay, limestone, gypsum rock and ores) is necessary. A precise provenance of raw

materials can often be determined from their microscopic study. This requires a more-than-basic knowledge of petrography.

### I.4.3 Reconstruction of the Archaeological Context, Microstratigraphy, Syn- and Postdepositional Changes

In many cases this research is not possible without a good understanding of the nature of the fabric components (see section I.4.1). Examples of archaeological context studies are the investigation of animal enclosures (Shahack-Gross 2017, this book) and caves and shelters (Mallol & Goldberg 2017, this book). Cave sediments, because of their protected location, generally contain, besides archaeological materials, a wealth of information on environmental and climatic changes, such as the occurrence of cold periods, illustrated by frost features (Van Vliet-Lanoë 2010).

Well known examples of anthropogenic soils or deposits are the plaggen soils, common in parts of Belgium, Germany and the Netherlands, and the Amazonian dark earths (Arroyo-Kalin 2017, this book). Both occur generally at the surface in rural areas and are still subject to pedological processes. European dark earths (Nicosia *et al.* 2017, this book) are found predominantly in urban environments. They show a large variety of characteristics depending upon period and location. Somewhat comparable are middens, heaps of shells, long since known in northern Europe (e.g. the so called køkkenmødding), but recently also studied in South America (Villagran *et al.* 2009; Correa *et al.* 2011).

### I.4.4 Impact of Man on the Environment

Throughout history, mankind has had an ever-increasing impact on the environment (Davidson *et al.* 1992). The most striking examples are without doubt forest clearance and agriculture (Deák *et al.* 2017, this book).

When studying archaeological sites, it is important to develop insight into the natural environment of the civilization, as there is always a clear relation between civilization, position in the landscape, natural resources and climate. An understanding of the surrounding soils can give information on the possible land use, land quality and possible output, often allowing estimation of the maximum size of the population or need for migration due to changing conditions. Such investigations are not developed in this book, as they concern recent, subrecent soils and palaeosoils, whose micromorphology is discussed in detail elsewhere (e.g., Stoops *et al.* 2010). Micromorphological studies of local sediments can, however, yield valuable information on alluvial dynamics, denudation and related erosion and colluvial processes.

## I.5 Techniques

As with all types of analyses, the relevance of the results depends in the first place upon the quality of the samples. This means that a clear sampling strategy has to be followed, as significant as correct sampling techniques. The latter is especially important in micromorphology, as samples should, by definition, be undisturbed and oriented (Stoops & Nicosia 2017, this book).

As mentioned in the second paragraph of this chapter, the most commonly used method in micromorphology is the study of thin sections with the help of a polarizing microscope (also called a petrographic microscope). These microscopes can also be equipped with a system for UV or blue light fluorescence (Stoops 2017, this book), a tool for the study of organic and phosphatic material (Karkanas & Goldberg 2010; Karkanas 2017, this book). Cathodoluminescence (Stoops 2017, this book) is especially helpful for investigations of calcareous materials. Reflected light microscopy, requiring polished uncovered sections, is necessary for the study of coal, opaque constituents and organic particles (Ligouis 2017, this book).

Optical microscopy, however, does not allow identification of all components, especially when they are microcrystalline, cryptocrystalline or amorphous. Then, additional techniques are needed, if possible on the (uncovered) thin section, or on corresponding impregnated samples (by microdrilling) or on spare parts of the monolith samples.

Several physical methods yield information on the chemical composition of the constituents. The most important are the different microprobe techniques (Wilson 2017, this book) and micro X-ray fluorescence (Mentzer 2017a, this book), which can be applied directly to uncovered thin sections, and isotope mass spectrometry (Mentzer 2017b, this book) on microdrilled samples. Other microtechniques focus on molecular or crystallographic structures, such as gas chromatography combined with mass spectrometry (Shillito 2017, this book), micro Fourier transformed IR spectrometry (Berna 2017, this book) and micro X-ray diffraction (Berthold & Mentzer 2017, this book).

Finally, some other methods allow a more detailed insight into the fabric than is possible with the optical microscope. Scanning electron microscopy (SEM) on fresh fracture surfaces is the most commonly used method, applied in soil micromorphology since the end of the 1960s, to observe morphology, e.g. crystal shapes that can assist in identification, or the etching of grains, giving information on the environment. SEM-EDXRA (Wilson 2017, this book) techniques can be used on uncovered thin sections to determine the elemental composition of components that cannot be identified by

transmitted light observation alone. A more sophisticated, new method is the micro CT scanning (see Ngan-Tillard & Huisman 2017, this book) of small fragments, showing in 3D their internal fabric, mainly the microstructure.

## I.6 Concluding Remarks

As mentioned before, identification of constituents is mainly based on their optical properties. In recent research, Shahack-Gross (2016) analysed a self-evaluation exercise performed amongst micromorphologists working in archaeological contexts. She concluded that mineralogy and petrography are generally poorly understood by the participants, and encouraged students in micromorphology to study core geology courses. As a lecturer of many intensive training courses in Europe, and as a referee of several papers, the first author also experienced that many archaeologists have insufficient knowledge of earth sciences in general (especially of soil science, mineralogy and petrography). Such knowledge is not only necessary to identify materials and features of archaeological origin but also to distinguish natural features from those resulting from human action. A typical example is the dusty clay coatings that are immediately considered by many archaeologists as a proof of human activity, disregarding all possible natural origins (see Deák *et al.* 2017, this book). Sufficient knowledge of mineralogy and petrography is, moreover, necessary to understand the source of materials found in archaeological contexts and the possible alteration processes (weathering, diagenesis). Not only archaeologists, but also the younger generation of soil scientists is struggling with these general problems as their training in geology has been reduced very much in recent decades.

The interpretation of micromorphological features is, in the first place, based on comparing collected data with data from literature or those of colleagues. This requires a clear, unambiguous description of the observations, which can be realized using one of the existing systems mentioned in section I.2, or a personal terminology, provided that all terms are clearly, unambiguously explained and defined. What should be avoided in particular, but has often been noticed in publications, is the use of a mixture of terminologies from different systems, without taking into consideration the different concepts on which they are based.

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# Part I

# Inclusions in Archaeological Soils and Sediments

# 1

# Bone and Other Skeletal Tissues

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## 1.1 Introduction

Bone, teeth and other hard tissues derived from animals are a major artefact class of many archaeological sites. Fragments of these biological components can often constitute a significant portion of the coarse fraction of archaeological deposits (e.g., Schiegl *et al.* 2003; Dibble *et al.* 2009; Goldberg *et al.* 2012; Mentzer *et al.* 2015). The study of larger fragments of archaeological bone recovered during excavation is a central aspect of zooarchaeological and anthropological analysis. Geoarchaeologists encounter bones and teeth when conducting micromorphological analysis; however, the size of the bone fragments found during this type of study is often smaller than the size of those incorporated into more traditional zooarchaeological studies. Bones in thin sections of archaeological deposits therefore provide a different, but complementary, perspective on the archaeological remains of animals (Estévez *et al.* 2014). By studying these small-scale remains of bone in a thin section, micromorphologists can address a wide range of archaeological issues related to human behaviour, site formation processes, taphonomy and chemical diagenesis.

## 1.2 Micromorphology

### 1.2.1 Composition and Structure of Bone and Related Materials

**Bone** is a biological material that is produced by vertebrate animals. In living bodies, bone functions as both an organ and as a rigid skeleton that supports and contains soft tissues. Fresh bone is composed of inorganic calcium phosphates precipitated in an organic collagen matrix. More specifically, bones are generally composed of 20–30% collagen (protein) and 60–70% calcium phosphates (bone mineral), with the remaining <10% comprising a combination of other components such as complex sugars, lipids, carbonates, Mg, Na, trace elements and metal ions (White & Hannus 1983; Posner *et al.* 1984; Pate & Hutton 1988; Linse 1992; McCutcheon 1992; Currey 2002). The mineral component of bone is commonly referred to as: (i) hydroxylapatite or hydroxyapatite ($Ca_5(PO_4)_3(OH)$); (ii) bioapatite (a poorly crystalized calcium phosphate resembling hydroxylapatite); or (iii) carbonate hydroxylapatite ($Ca_5(PO_4CO_3)_3(OH)$) also known as dahllite (Pate & Hutton 1988; Linse 1992; McCutcheon 1992; White & Hannus, 1983; Stiner *et al.*, 1995; Karkanas *et al.* 2000; Hedges, 2002; Berna *et al.* 2004; Trueman *et al.* 2004; Smith *et al.* 2007). In reality, bone mineral is difficult to characterize. Mineralogical analyses reveal that pure hydroxylapatite is never actually found in bone or teeth, thus Weiner (2010) argues that this term should be reserved for geogenic, noncarbonated forms of apatite. Furthermore, the mineral dahllite is no longer recognized by the International Mineralogical Association. Therefore, despite the general inconsistencies found in the literature regarding bone mineral, the terms bioapatite, carbonate hydroxylapatite, or carbonate apatite are most appropriate.

The same combination of collagen and bioapatite occurs in mammal, bird, reptile and fish bones. The only exceptions are fish of the elasmobranch type (sharks, skates and rays), whose skeletons consist of cartilage containing a different type of collagen (type II) and no bioapatite (Szpak 2011).

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Besides bone, other hard biological tissues that can appear in the archaeological record are antler, teeth and keratin structures like horn and hair (of the aforementioned tissues, hair is the only nonskeletal one). **Antler** is a bony extension of the skulls of deer that has the same composition as bone (bioapatite, collagen, noncollagenous proteins and water – Currey 2002). **Teeth** are composed of three different hard biological materials: enamel, dentin and cementum. These materials have the same general composition as bone, but differ in the relative proportions of mineral to other components; enamel, dentin and cementum contain >95%, 75%, and 45% carbonate hydroxylapatite, respectively (Provenza & Seiber 1986; Weiner & Wagner 1998; Francillion-Viellot *et al.* 1990; Weiner 2010). **Horn** exists on animals from the Bovidae family (cattle, sheep, goat, etc.). It differs from bone in that it contains keratin (alpha-keratin, a fibrous protein also found in hair, nails, wool and claws) with minor amounts of bioapatite (Hashiguchi & Hashimoto 1995; Salamon 1999; Hashiguchi *et al.* 2001; O'Connor *et al.* 2015) or no crystalline phase at all (Tombolato *et al.* 2010). The **hair** fibre is made of hard keratin, water, lipids, pigment and trace elements (Wilson & Tobin 2010). Horn, hair and other keratinous tissues rarely survive in the archaeological record unless burial conditions impede biological activity (Wilson *et al.* 2007; Wilson & Tobin 2010; O'Connor *et al.* 2015).

In addition to having different compositions, hard biological tissues differ in their macroscopic structure. **Bones** can be divided into two different structures according to their porosity. Compact or cortical bone has low or null porosity. Spongy bone, also known as trabecular or cancellous bone, has high porosity. The boundary between both types is diffuse since compact and spongy represent a continuum (Weiner & Wagner 1998; Francillion-Viellot *et al.* 1990; Currey 2002; Weiner 2010). Spongy bone is frequently, though not exclusively, found in bone epiphysis or inside vertebrae, whereas dense compact bone is found in the shaft of long bones (bone diaphysis) or as part of flat bones (e.g., skull, scapula). **Antler** is composed of a combination of exterior compact bone and interior spongy bone (Goss 1983). **Teeth** exhibit a specific structural arrangement of enamel, dentin and cementum, from which only dentin is porous. The crown portion of the tooth is composed of enamel, which is thickest next to the crest of the cusp. The outer portion of the root is composed of cementum. The inner portions of the root and crown are composed of dentin (Carlson 1990). **Horns** contain fibrous keratinous tissue, and exhibit a gradient of porosity that is higher in the exterior and lower in the interior. They are attached to the skull by a short bony core made of spongy bone and covered with skin. Horns exhibit a hollow core when detached from the animal skull (Tombolato *et al.* 2010). The **hair** shaft is macroscopically smooth, but can have various shapes and thickness depending on the animal species and individual characteristics (Brunner & Coman 1974; Tridico *et al.* 2014).

### 1.2.2 Optical and Microstructural Properties of Bone and Other Skeletal Tissues

The optical properties of bone, teeth (including ivory), antler and keratin tissues (horn, hoof and hair) are summarized in Table 1.1. Different types of **bone** structures are visible in thin section. Compact and spongy bone can be easily recognized, despite the angle of the cut, due to the massive appearance that characterizes the former, and the large, closed-packed pores that characterize the latter (Figure 1.1a). At magnification, four fabric types of bone can be described. The basic component of bone material is the mineralized collagen fibril, which constitutes a combination of bioapatite crystals ($5 \times 25 \times 2–4\,nm^3$ plate-shaped crystals) and collagen fibrils (80–100 nm in diameter) (Lowenstam & Weiner 1989; Weiner 2010). The fibrils can be packed in four different ways (Currey 2002): woven bone (fibrils are randomly arranged) (Figure 1.1b); parallel-fibred bone (close-packed, parallel fibrils that have the same orientation); lamellar bone (fibrils are arranged in thin lamellae or sheets, like plywood, where each sheet differs in orientation of the fibrils) (Figure 1.1c); and fibrolamellar bone, which is a combination of woven and lamellar and/or parallel-fibred bone (Figure 1.1d, f). Different histological structures may be characterized by one or more type of fibril packing. For example, Haversian systems, which house blood vessels or nerves, consist of longitudinal vascular canals surrounded by concentric lamellar bone (Figure 1.1e). The appearance of bone in thin section depends both on the macrostructure, the arrangement of the fibrils and the angle at which the bone was cut during the preparation of the slide. For example, lamellar zones of compact bone show a pattern of intercalating dark and light bands in XPL (Figure 1.1f).

Both components of fibrils, the (quasicrystalline) collagen and the bioapatite, contribute to the appearance of bone under crossed polars. Both components, on their own, have low-order white to grey interference colours (Courty *et al.* 1989; Bromage *et al.* 2003); however, bone exhibits form birefringence, in which the birefringence of two materials influence each other. Both collagen and bioapatite are uniaxial, and their optical axes lie parallel to the axes of the collagen fibrils (Watson 1975; Wolman 1975; Stoops 2003). Collagen has a positive elongation sign, while bioapatite (and all apatite isomorphs), has a negative elongation sign (Bourne 1956; Watson 1975; Bromage *et al.* 2003). Although Courty *et al.* (1989) state that the mineral component is responsible for the

**Table 1.1** Optical characteristics of bone, teeth, ivory, antler and keratin tissues (horn, hoof and hair). Note that pleochroism is absent in all the hard tissues.

| Tissue | Colour (PPL) | Interference colour (XPL) | Autofluorescence (blue and UV light) | Figure | References |
|---|---|---|---|---|---|
| **Bone** | Light yellow | Low-order white to grey | Blue (UV), yellowish to green (blue light) | 1.1a, 1.1f, 1.2a | Courty *et al.* 1989; Stoops 2003; Karkanas & Goldberg 2010 |
| **Antler** | Light brown | Low-order white to grey | Yes | 1.3a, b, c, d | Rolf & Enderle 1999; Skedros *et al.* 2014 |
| **Teeth** | | | | | |
| ***Enamel*** | Light brown | Low-order white to grey | Blue light: no<br>UV and red light: yes | 1.3e, f | Schmidt & Keil 1971 |
| ***Dentin*** | Pale brown and grey (with parallel fibers) | Grey and light orange (polarization cross visible in transversal sections of tooth) | Yes | 1.3e, f,<br>1.4a, b, c, d | Schmidt & Keil 1971 |
| ***Cementum*** | Pale brown | Low-order white with varying brightness | No | 1.4a, b, c, d | Schmidt & Keil 1971 |
| **Ivory (fossil)** | Brown | Low-order grey | Yes | 1.4e, f | Su & Cui 1999; Heckel 2009; Virág 2012 |
| **Keratin tissues** | | | | | |
| ***Horn*** | Light brown | Low-order grey | Yes | 1.5a, b, c, d, e, f | Tombolato *et al.* 2010 |
| ***Hoof*** | Yellowish brown | Low-order grey to high order | Yes | 1.6a, b | Kasapi & Gosline 1997 |
| ***Hair*** | Colourless to pale yellow | High order | Yes | 1.6c, d, e, f | Wilson 2010; Wilson *et al.* 2010; Dejmal *et al.* 2014; Tridico *et al.* 2014 |

low-order interference colours of bone, Karkanas & Goldberg (2010), based on the work of Watson (1975), indicate that the interference colour of fresh bone is mostly due to collagen. Watson's conclusion is based on observations of bones that, after losing their collagen matrix, changed sign of elongation from positive (dominated by collagen) to negative (dominated by bioapatite). This change is also seen in the experimental heating of a fish vertebra at 100 °C and 500 °C (Figure 1.2).

In practice, the determination of elongation sign in bone must take into account the orientation and distribution of the collagen fibres, which may vary in different bones according to their biomechanical properties (Bromage *et al.* 2003). In the example given in Figure 1.2, if we consider the bioapatite and collagen fibres to be transversal to the medullary cavity, then there is a change from positive to negative elongation after heating. This fits Watson's proposal that collagen interference colour is dominant in fresh bone. However, if the fibrils are radially oriented, then the opposite situation is described (from negative to positive). Moreover, Bromage *et al.* (2003) state that the different interference colours seen in fresh bone are also determined by the orientation of its collagen, with transversal collagen fibres appearing white, longitudinal fibres appearing black and fibres with intermediate orientation showing different levels of grey under crosspolarizers.

The autofluorescence of bone can be attributed to the bioapatite component, although there is also a small contribution from collagen (Altermüller & Van Vliet-Lanoe 1990). Autofluorescence may be partly or completely lost as a result of decay or heating (see below) and its presence can be taken as indirect evidence of the degree of bone weathering (Hoke *et al.* 2011; Hollund 2013).

**Antler** has the same optical properties as bone with macrostructure comparable to that of long bones (an outer rim of compact bone and a core of spongy bone; Figure 1.3a–d; Table 1.1). **Teeth** can be identified by their overall morphology, as well as optical properties of the three components (dentin, enamel and cementum) (Table 1.1). In transversal cuts of the cusp, all vertebrate teeth show an outer layer of enamel, a core of dentin with radial orientation of fibrils, and polarization cross under XPL (see Figure 1.3e, f). The hollow centre of the tooth (pulp cavity) may be expressed as a void. In fossil or archaeological teeth, the dentine may have weaker

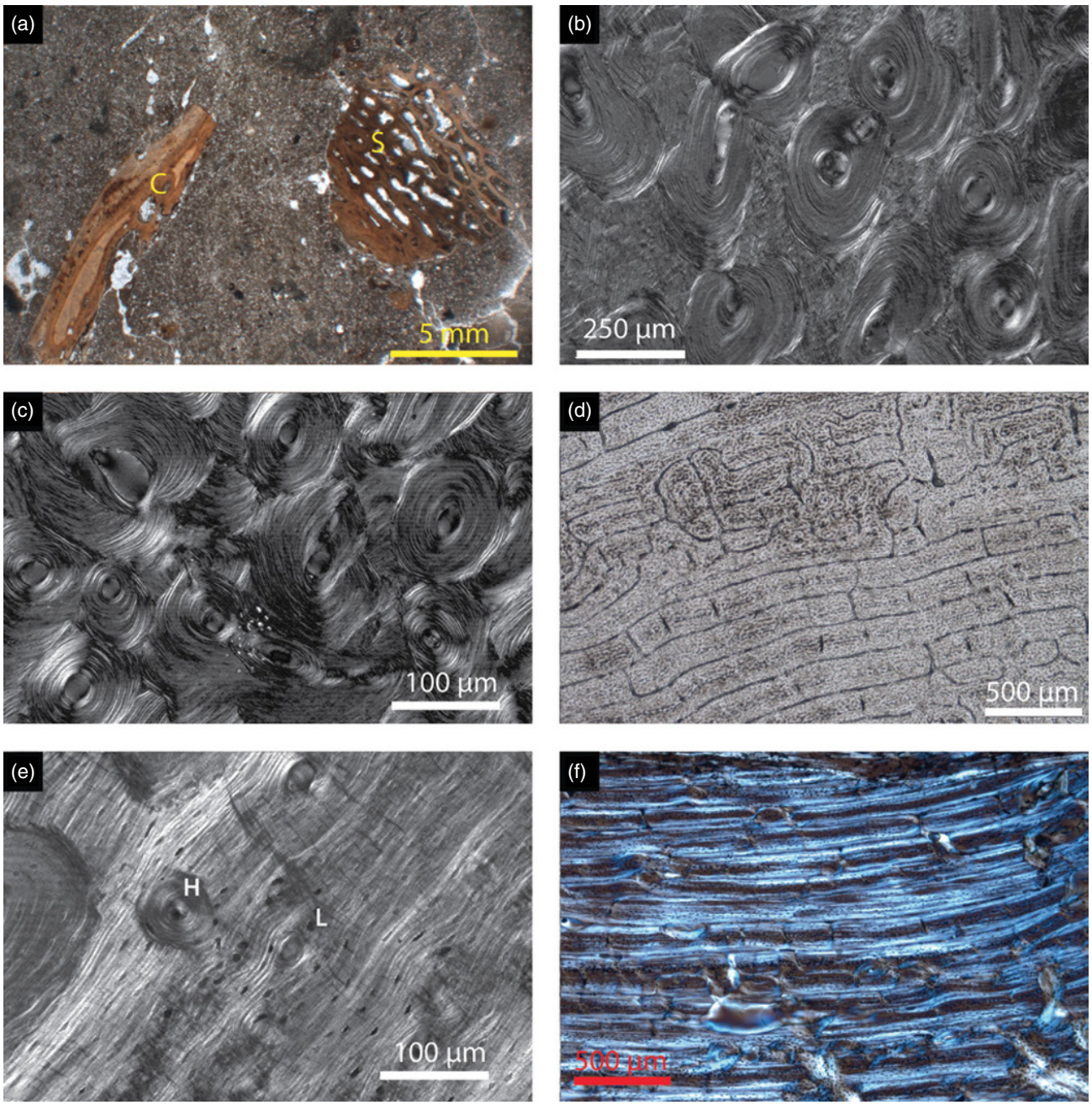


**Figure 1.1** Examples of bone microstructures. (a) Fragments of spongy (S) and compact (C) bone fragments in a medieval deposit. PPL. (b) Microstructure of human bone (transversal cut) with osteons (circular canals with concentric lamellar bone) surrounded by woven bone. PPL. (c) Thin section of human bone (transversal cut) showing clear pattern of lamellar bone within and surrounding osteons. XPL. (d) Microstructure of fibrolamellar (plexiform) bone tissue (transversal cut) in deer bone section. PPL. (e) Haversian channel (H) formed in lamellar bone (L). (f) Fibrolamellar (plexiform) bone microstructure (transversal cut) of unknown mammal, probably ungulate. XPL.

birefringence or even appear completely isotropic (Schmidth & Keil 1971). Transverse cuts of the root exhibit outer layers of cementum and inner cores of dentin (Figure 1.4a–d). Longitudinal cuts may contain all three components. Depending on the cut, cementum may exhibit a scalloped morphology where it attaches to bone. Ivory, a specific type of dentin found in tusks of the order proboscidea, has distinct internal structures with parallel 'lines of Owen' (found in any dentin), and characteristic chevron-shaped 'Schreger patterns' that are only visible at low magnifications under PPL (see Su & Cui 1999; Heckel 2009; Virág 2012) (Figure 1.4e, f;

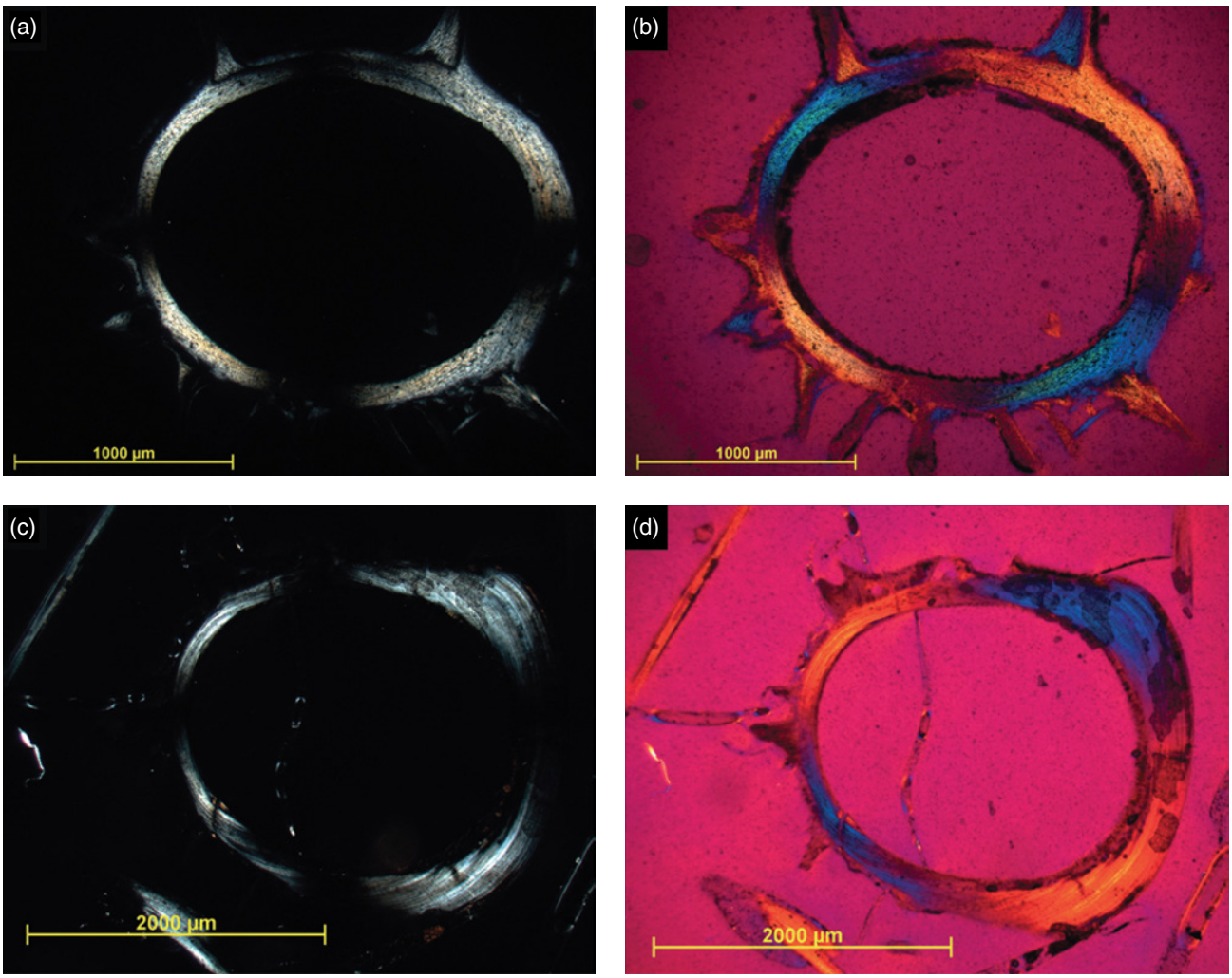


**Figure 1.2** Thin sections from the experimental heating of fish vertebrae under oxidising conditions. (a) Transversal cut of vertebra heated at 100 °C in XPL. (b) Same as Figure 1.2a after insertion of gypsum plate, note positive elongation of bone, characteristic of collagen, when collagen fibres are transversally arranged. (c) Transversal cut of a vertebra heated at 500 °C in XPL, note lowering in interference colours. (d) Same as (c) after insertion of gypsum plate, note negative elongation, characteristic of apatite (see also Figure 1.5).

Table 1.1). These structures produce preferred cracking and delamination patterns that yield rectangular to equant fragments. The optical properties of horn, hoof and hair are those of keratin; however, they differ in morphology and microstructure (Table 1.1). The microstructure of **horn** is lamellar, with the lamellae extending along the length of the horn following the direction of growth (Tombolato *et al.* 2010). In transversal sections, the layering of the growth pattern is clearly visible, with darker banding parallel to the surface (PPL) and a speckled pattern in XPL (Figure 1.5a, c). The stacked bundles of lamellae can be seen in longitudinal sections of horn under PPL. Differences in the orientation of the lamellae result in a banded pattern with first to second-order interference colours (Figure 1.5b, d). The darker bands are autofluorescent under UV and blue light. However, longitudinal sections show little or no autofluorescence (Figure 1.5e, f). The keratinous wall of the horse **hoof** in thin section (Figure 1.6a, b) has a characteristic woven or cross-hatch fabric with regularly spaced channels visible under XPL. Its matrix is similar to that of compact bone, with circular lamellae of keratin around a hollow tubule (the medullary cavity) (Bertram & Gosline 1986; Kasapi & Gosline 1997; Tombolato *et al.* 2010). **Hair** (Figure 1.6c–f; see also Dejmal *et al.* 2014, Figure 10) has internal stratigraphy composed of an outer cuticle, an inner cortex and a central medulla (Brunner & Coman 1974; Wilson & Tobin 2010). Depending on the orientation of the hair, the rough morphology of the cortex may be visible. When affected by fungal tunnelling, the typical green fluorescence of hair turns red (Wilson *et al.* 2007; Wilson & Tobin 2010).

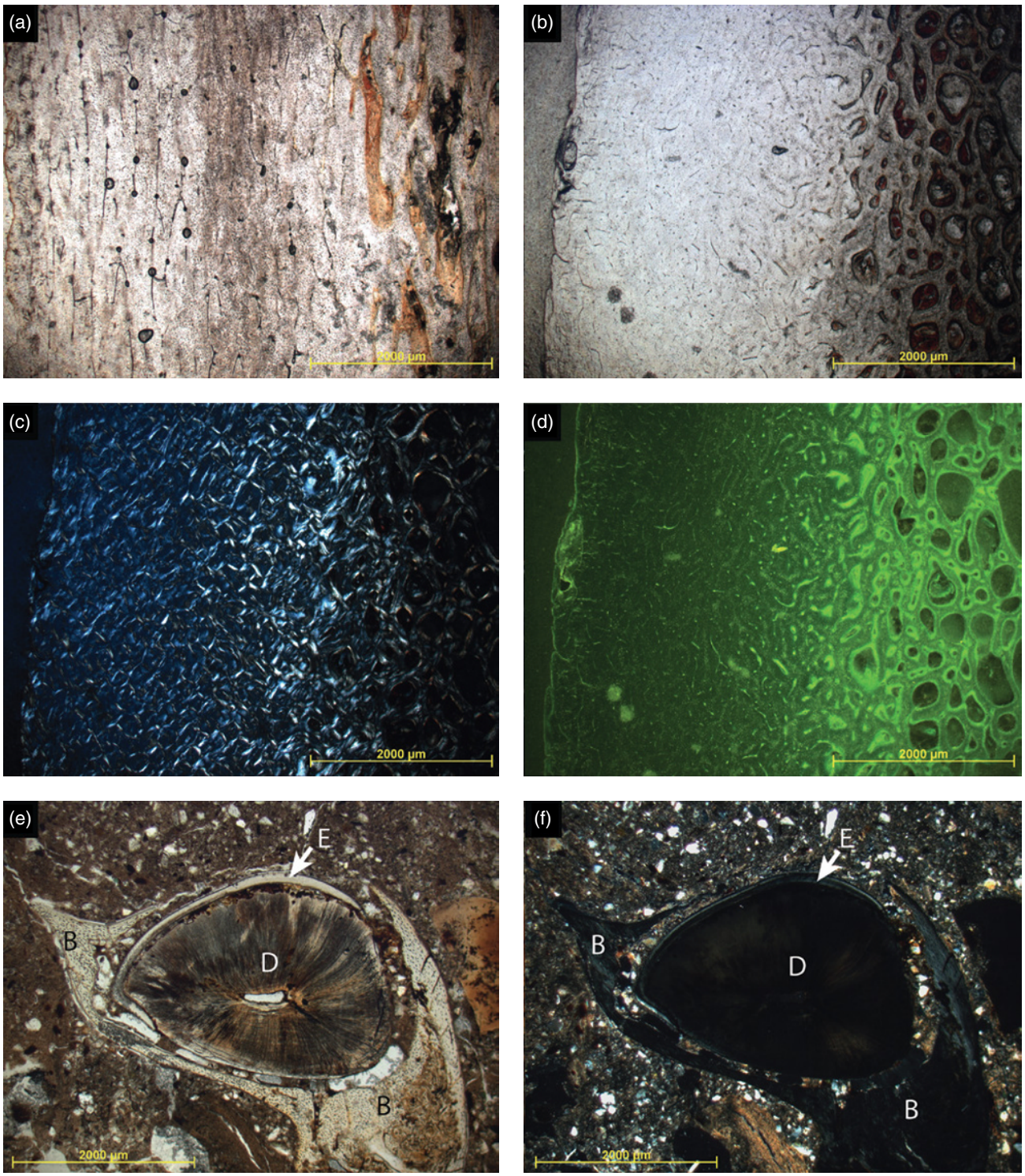


**Figure 1.3** Microstructure of teeth, antler and ivory. (a) Longitudinal section of modern red deer antler, the spongy bone is seen as elongated structures (right of the image) next to the massive compact bone with vascular channels. PPL. (b), (c) and (d) Transversal section of red deer antler. Note transition from spongy (core) to compact bone (edges), as commonly seen in long bones. Spongy bone has stronger autofluorescence, possibly due to higher organic content. (b) PPL; (c) XPL; (d) BLF. (e) and (f) Transversal cut of a rodent tooth – still embedded in bone (B) from the skull or a jaw fragment – showing radial dentin (D) in the core and outer layer of enamel (E). Medieval occupation deposit (Achlum, The Netherlands). (e) PPL; (f) XPL.

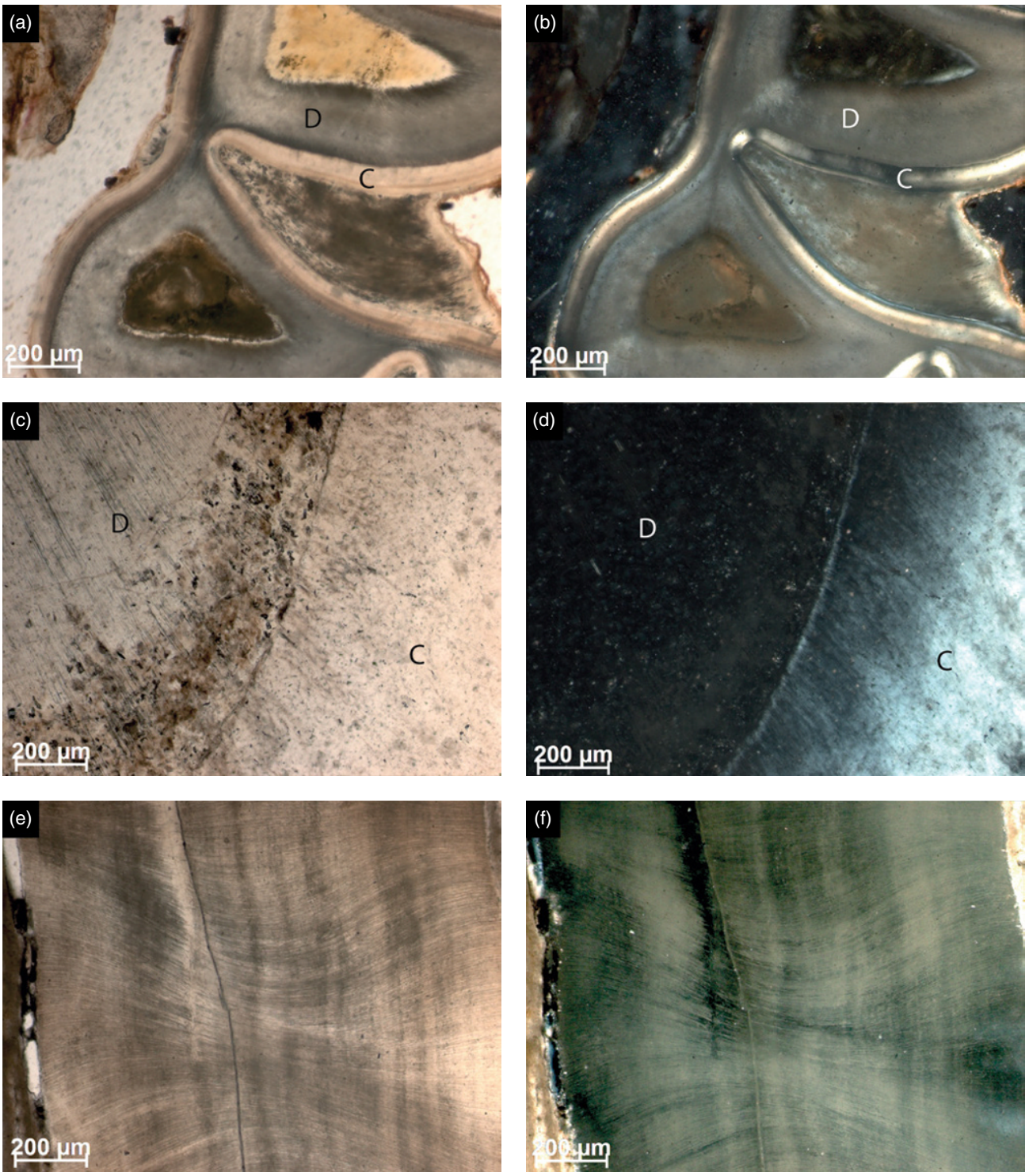


**Figure 1.4** (a) and (b) Transversal cut through rodent molar, with dentin (D) and cementum (C). Note bone attached to the cementum: (a) PPL; (b) XPL. (c) and (d) Close-up of cementum (C) and dentin (D). (c) PPL; (d) XPL. (e) and (f) Fossil mammoth ivory: (e) PPL; (f) XPL.

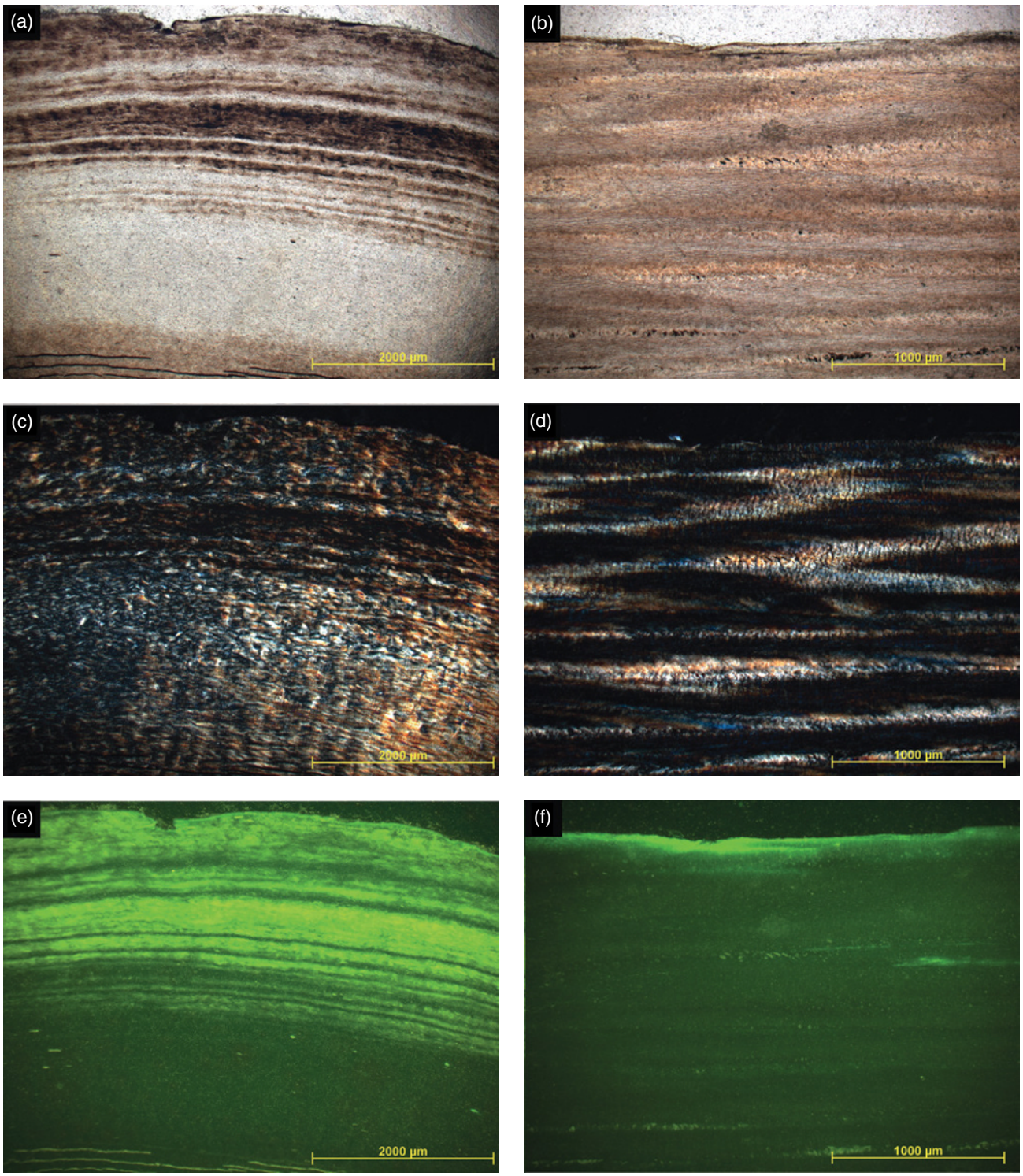


**Figure 1.5** Horn and other keratinous tissue. Parts (a), (c) and (e) are transversal sections of modern cow horn: (a) PPL; (c) XPL; (e) BLF. Parts (b), (d) and (f) are longitudinal sections of the same horn: (b) PPL; (d): XPL; (f) BLF. Note growth lines in the transversal section and lamellae extending parallel to the length of the horn in the longitudinal section.

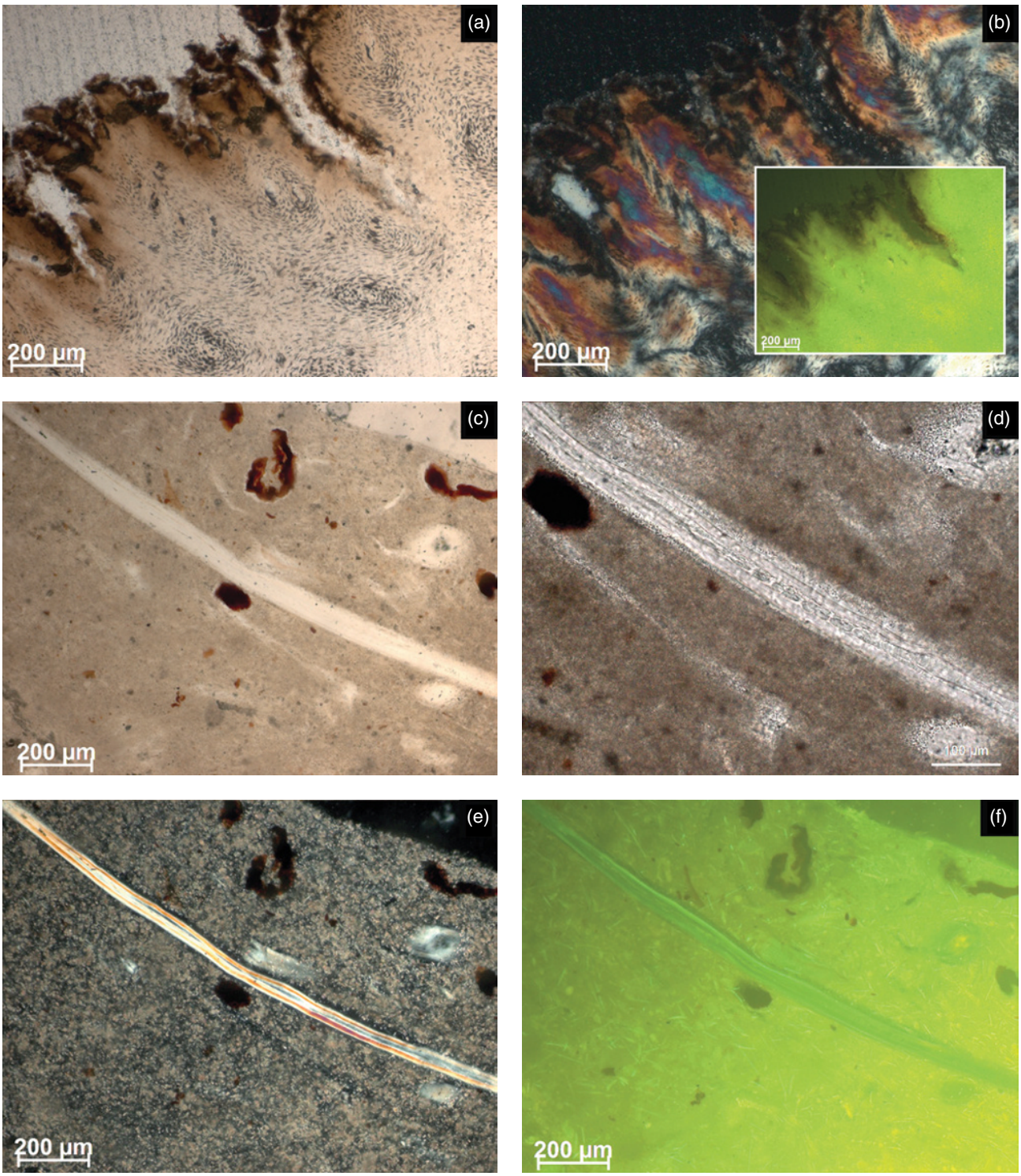


**Figure 1.6** (a) and (b) Horse hoof. Inset in H shows BL image: (a) PPL; (b) XPL. (c), (d), (e) and (f) Hair in coprolite: (c) PPL; (d) PPL; (e) XPL; (f) BLF.

### 1.2.3 Identification of Bone Element and Animal Species from Thin Sections

In micromorphological samples it is rare to be able to identify the skeletal element – let alone determine the animal group or species – due to the small size of most bone fragments in thin section. For fragments of large bones it may be possible to distinguish between spongy and compact tissue in thin section (Figure 1.1a). In other situations, fortuitous orientations or sections may aid in identification of the element, especially if the element is whole or characteristic features are visible (e.g., the mandible pictured in Figure 1.3e, f).

Bones from humans and nonhuman mammals can be differentiated by morphology, DNA, proteins, or histological analysis. Plexiform bone is a specific type of compact / cortical fibrolamellar bone. Humans do not produce plexiform bone, so its presence in a large bone fragment may suggest that the source is nonhuman (Figure 1.1d). However, other primates and most small mammals also lack plexiform bone, which in the case of small fragments can prevent a straightforward identification. Furthermore, plexiform bone can be removed from the bones of large animals by weathering. In these ambiguous cases, measurements of certain histological structures (e.g., the diameters of Haversian systems) in oriented sections are necessary (Hillier & Bell 2007) (Figure 1.1c, d, f).

Although identification of most animal groups or species is difficult in micromorphological samples, an exception is seen in certain bones that are clearly diagnostic of fishes. Fish scales can be easily distinguished in thin section by their denticulate surface (Figure 1.7a, b) and fish vertebrae are unmistakably identified by their cross-shaped or rounded morphology (Figure 1.7c, d). Different bones that comprise the fin rays also show particular shapes that allow for their identification in thin section. The proximal portion of fin rays is made of thin, elongated bones with thicker and rounded extremities (Figure 1.7e), while the lepidotrichia (the distal portion of the fin) is made of successive paired small bones, which exhibit shapes similar to parenthesis (Figure 1.7f) (Francillion-Vielot *et al.* 1990). In addition, recent studies demonstrate that micro CT (microcomputed tomography) scanning of micromorphological blocks provides three-dimensional information about bone fragments and may facilitate species determination when samples contain small bones (Huisman *et al.* 2014b; Ngan-Tillard & Huisman 2017, this book).

## 1.3 Taphonomy of Bone

Taphonomic processes that impact bones include combustion and heating, biological activity, mechanical fracturing and chemical weathering. Identification of the traces of these processes in bones visible in thin section can aid in reconstructing site formation processes, including syndepositional human activities such as burning and trampling and disturbance of sites by animals. Other processes can be indicative of past environments and burial settings. Micromorphology can also be integrated to the solution of taphonomic problems raised by zooarchaeological analyses (the microtaphonomic approach, *sensu* Estévez *et al.* 2014).

### 1.3.1 Combustion and Heating of Bone

Burning can occur incidentally, for example when bones are located underneath a hearth, or intentionally when they are burnt for fuel (Schiegl *et al.* 2003), used to manage specific properties of the fire (Théry-Parisot 2002), or subjects of site-maintenance practices (Clark & Ligouis 2010). Only the organic constituents (i.e., fat and collagen) of bone truly burns; however, the mineral fraction of bone also becomes altered by heat, providing a means for identifying and tracking heating in the past. Other types of heating, such as roasting, baking and boiling, produce characteristic compositional and structural changes, including loss of collagen at low temperatures (Roberts *et al.* 2002).

According to Ellingham *et al.* (2015) bone can undergo four stages of transformation related to burning or incineration: (i) dehydration, (ii) decomposition, (iii) inversion and (iv) fusion. Several experimental studies have shown that bones progress through predictable stages of colour alteration corresponding to degree of heating (e.g., Shipman *et al.* 1984; Stiner *et al.* 1995; Bennett 1999; Hanson & Cain 2007). These stages include: fresh, unburnt bone, which appears ivory or tan in colour; partially carbonized or charred bone, which can appear brown or reddish; fully carbonized or charred bone, which appears black; and white calcined bone, in which all organic material and moisture is removed. Despite these numerous studies, some controversy remains as to what temperature corresponds to which colour change (Ellingham *et al.* 2015). Some researchers note that the initiation of the different stages of colour change vary between bones derived from mammals, birds and fish (Nicholson 1993), whereas others point out the amount of flesh and fat covering a bone can also significantly influence the onset of colour changes due to heating (Symes *et al.* 2008).

Table 1.2 summarizes experimental data produced from modern bones burnt or heated at a variety of temperatures. Two sets of burning experiments were conducted on ungulate and fish bones, which were then processed into thin sections (see also Figures 1.8, 1.9, 1.10). Petrographic observations of these bone samples are included; however, it is important to stress that many

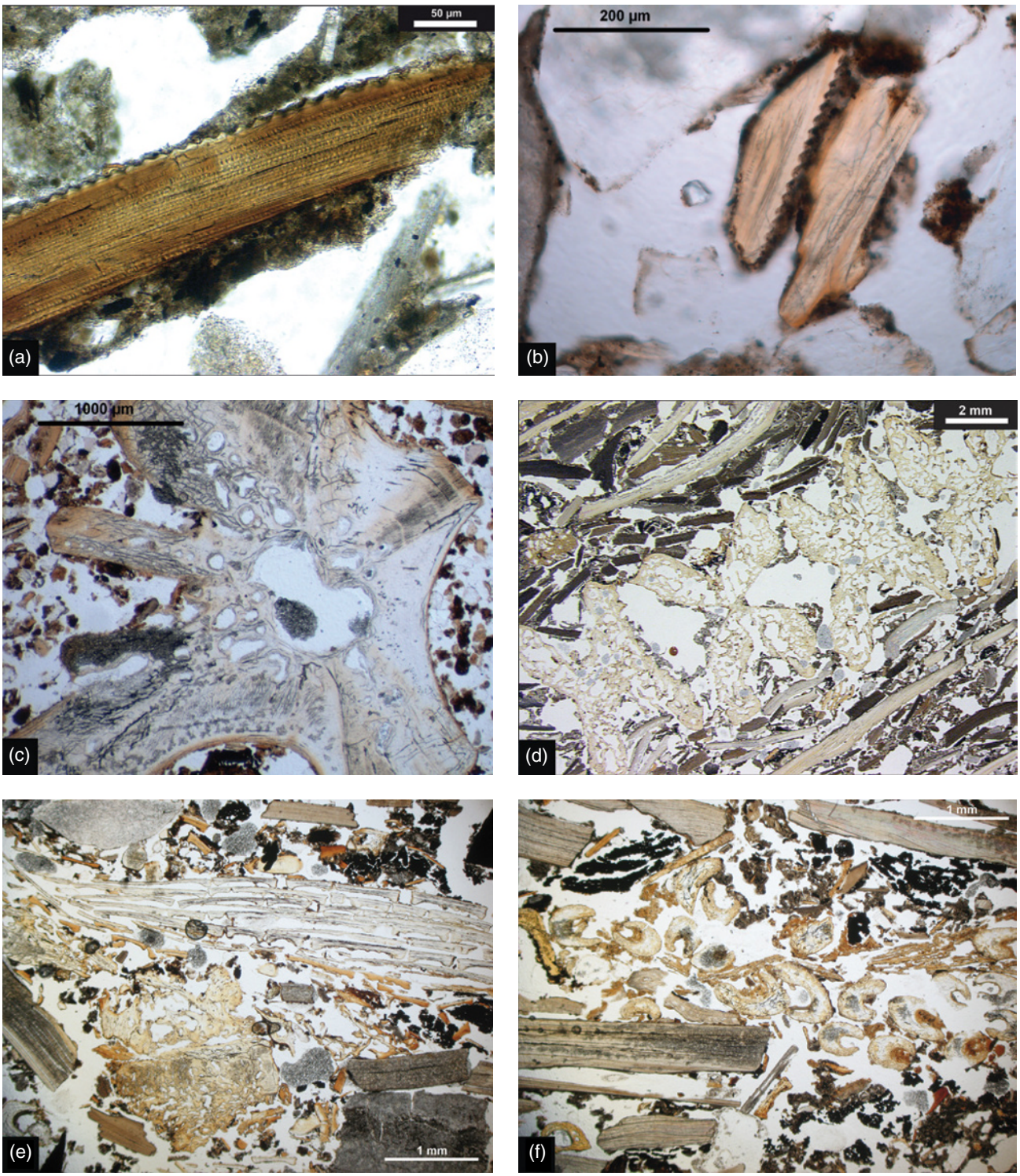


**Figure 1.7** Fish bones. (a) Fish scale with typical denticulated edge. Caipora shell mound (c. 7440–6110 cal. yr BP, Santa Catarina, Brazil – see Estevez *et al.* 2014; Villagran 2014). PPL. (b) Small fragment of fish scale. Same site as Figure 1.5a. (c) Section of a fish vertebra, Santa Marta 8 fish mound (c. 1550 cal. yr BP, Santa Catarina, Brazil – see Villagran 2014. PPL. (d) Section of four articulated fish vertebrae, Cubatão shell mound (c. 3000 BP, Santa Catarina, Brazil). PPL. (e) Articulated bones from a fin ray (fr). Sernambetiba shell mound (c. 2000 BP, Rio de Janeiro, Brazil). PPL. (f) Dispersed bones from fin ray lepidotrichia (fr). Sernambetiba shell mound (Rio de Janeiro, Brazil). PPL.

**Table 1.2** Micromorphological characteristics of experimentally heated bone in thin section. n.d. = no data. Thin sections of pig and cow bones: the unburnt samples (bone and teeth) are from the personal reference collection of P. Goldberg; the sample heated at 100 °C is a boiled reindeer bone collected by L. Binford from an ethnographic context. Thin sections of fish bones courtesy of M. Canti.

| Temperature in °C (heated to +/– 10 °C) | Micromorphological characteristics of *pig and cow bones* in thin section (temperatures held for 45 minutes) | Micromorphological characteristics of *fish bones* in thin section (temperatures held for 30 minutes) |
|---|---|---|
| Unburnt | Colourless (transparent) in PPL. Strong low-order interference colours (white, grey, black). Strong fluorescence under blue and UV light. | n.d. |
| 100 | Colourless (transparent) in PPL. Strong low-order interference colours (white, grey, black), strongly contrasting. Weak to moderate fluorescence under blue light; moderate under UV light. | Colourless (transparent) to light grey in PPL. Strong birefringence in XPL. Low order interference colours. |
| 200 | Colourless (transparent) in PPL. Strong low-order interference colours (white, grey, black), strongly contrasting. Moderate fluorescence under blue light; moderate to strong under UV light. | As above. |
| 300 | Light to medium yellow, ranging to dark reddish brown in PPL. Weak abnormal olive brown, to grey brown interference colours. Moderate to strong fluorescence under blue and UV light, except in areas with reddish brown colour in PPL. | Light to medium yellow in PPL. Weak to moderate grey to white interference colours. |
| 400 | Dark reddish brown to black (opaque) in PPL. Weak interference colours (reddish in areas that are reddish brown in PPL). Absent to very weak fluorescence under blue light; absent to weak fluorescence under UV light. | Reddish brown to black (opaque) in PPL. Weak abnormal interference colours, reddish in areas that are reddish brown in PPL. |
| 500 | Pale brown to black (opaque) in PPL. Weak abnormal olive brown to dark blue-grey interference colours. Weak to moderate fluorescence under blue light in areas that are brown in PPL, absent to moderate fluorescence under UV light. | Pale brown in PPL. Moderate blue-grey interference colours. |
| 600 | As above. | Colourless (transparent) to brown in PPL. Moderate blue interference colours. |
| 700 | Pale brown in PPL. Weak to moderate low order greys. Interference colours are with low contrast. Absent to very weak fluorescence in blue and UV light. | As above. |
| 800 | Brown to brownish grey in PPL, with visible internal fissures. Moderate to strong bluish grey interference colours with an overall milky cast (accentuated by the substage condenser). Small crystal aggregates with higher interference colours are visible within the bone tissue. Absent to weak fluorescence in blue and UV light. | As above. |
| 900 | Brown to brownish grey in PPL, with visible internal fissures. Strong white to grey interference colours with an overall milky cast (accentuated by the substage condenser). Absent to very weak fluorescence in blue and UV light. | Pale brown in PPL. Weak to moderate low order grey to blue-grey interference colours. |
| 1000 | As above | As above. |
| 1100 | n.d | Opaque in PPL. Moderate low-order grey interference colours with an overall milky cast. |
| 1200 | n.d. | Translucent in PPL. Weak to moderate low order grey interference colours. |

of the observed features, such as colour in PPL and interference colours can be impacted by a variety of sample preparation parameters and taphonomic conditions. These include the angle at which the bone is cut, slide thickness, microscope light source and use of the substage condenser, as well as chemical and biological alteration (see section 1.3.3).

The data presented in Table 1.2 are broadly consistent with published observations from archaeological samples. Many researchers have noted the distinctive milky

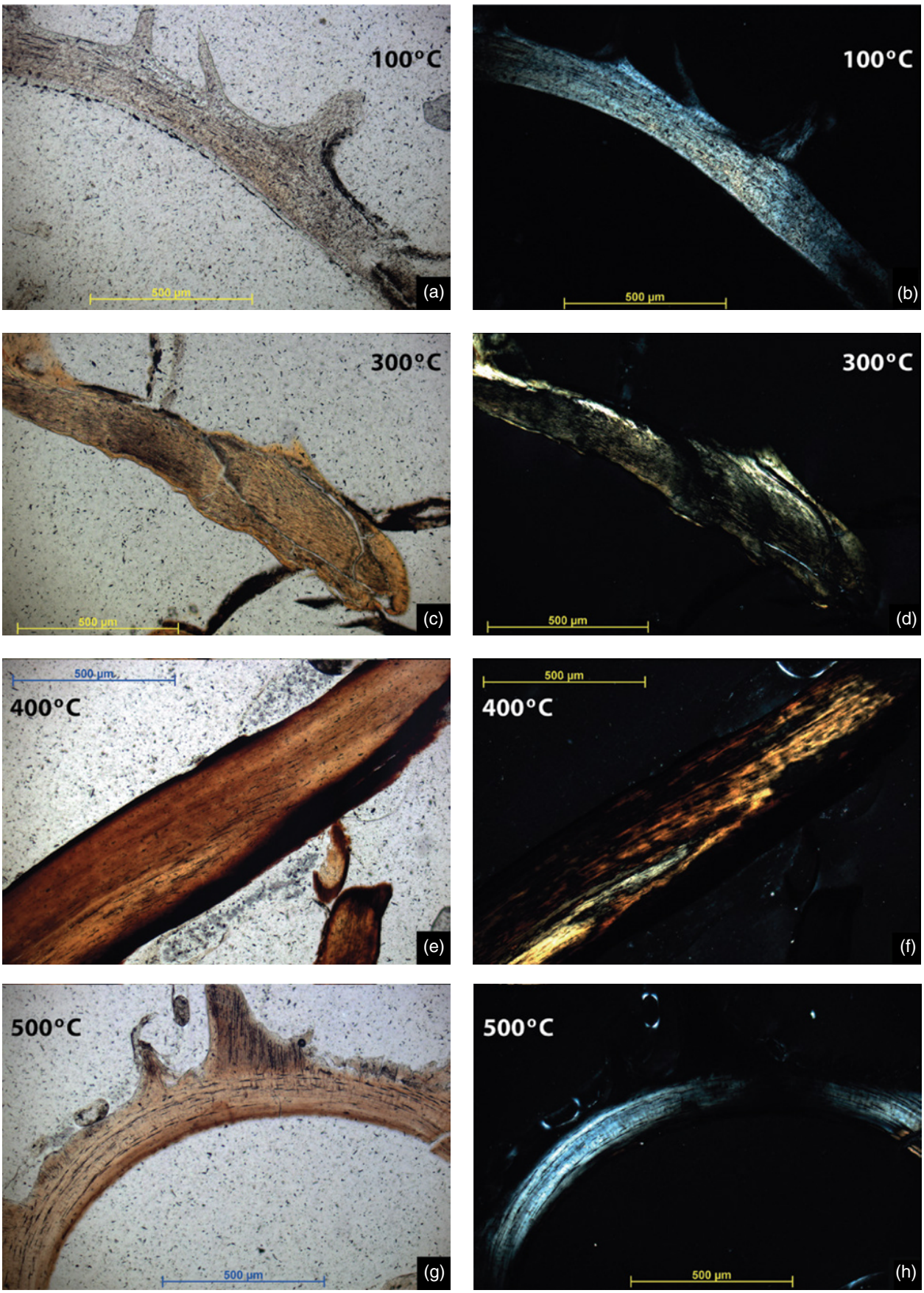


**Figure 1.8** (a)–(h) Thin sections of herring bone heated from 100 °C to 500 °C under oxidising conditions. Images in PPL (left) and XPL (right). From 100 to 400 °C, the colour of bone changes from light grey to yellowish orange and reddish brown to black (PPL). Interference colours go from first-order yellow and white between 100 and 300 °C. Second-order reddish interference colours appear at 400 °C (XPL). Thin sections courtesy of M. Canti.

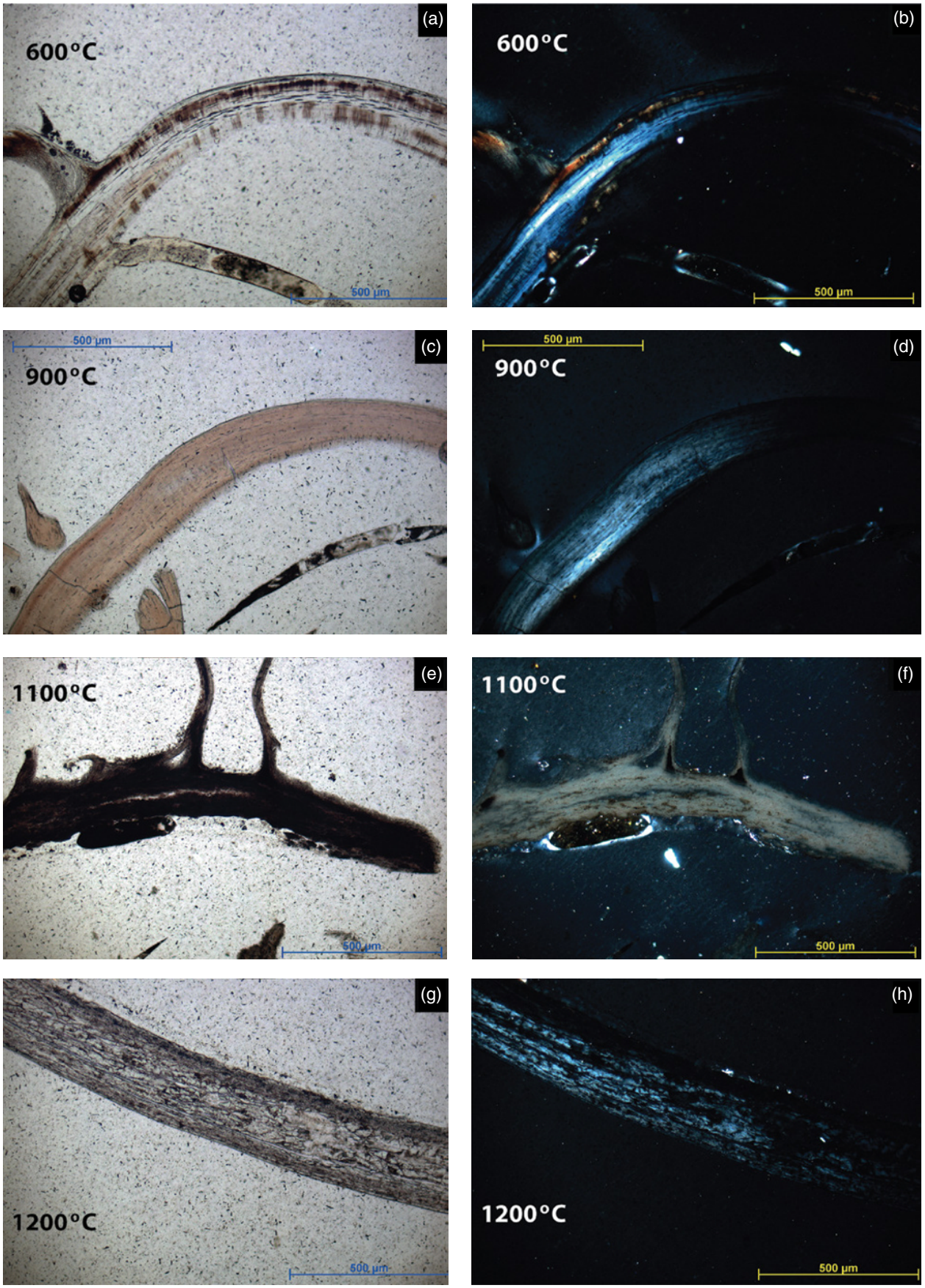


**Figure 1.9** (a)–(h) Thin sections of herring bone heated from 600 °C to 1200 °C under oxidising conditions. Between 500 °C (Figure 1.8) and 900 °C bones turn pale brown with blue-grey and blue interference colours (XPL). Bone heated to 1100 °C is opaque (PPL) with cloudy or milky appearance (XPL). At 1200 °C, the bone is again pale brown (PPL) and shows lower order white and grey interference colours (XPL). Thin sections courtesy of M. Canti.

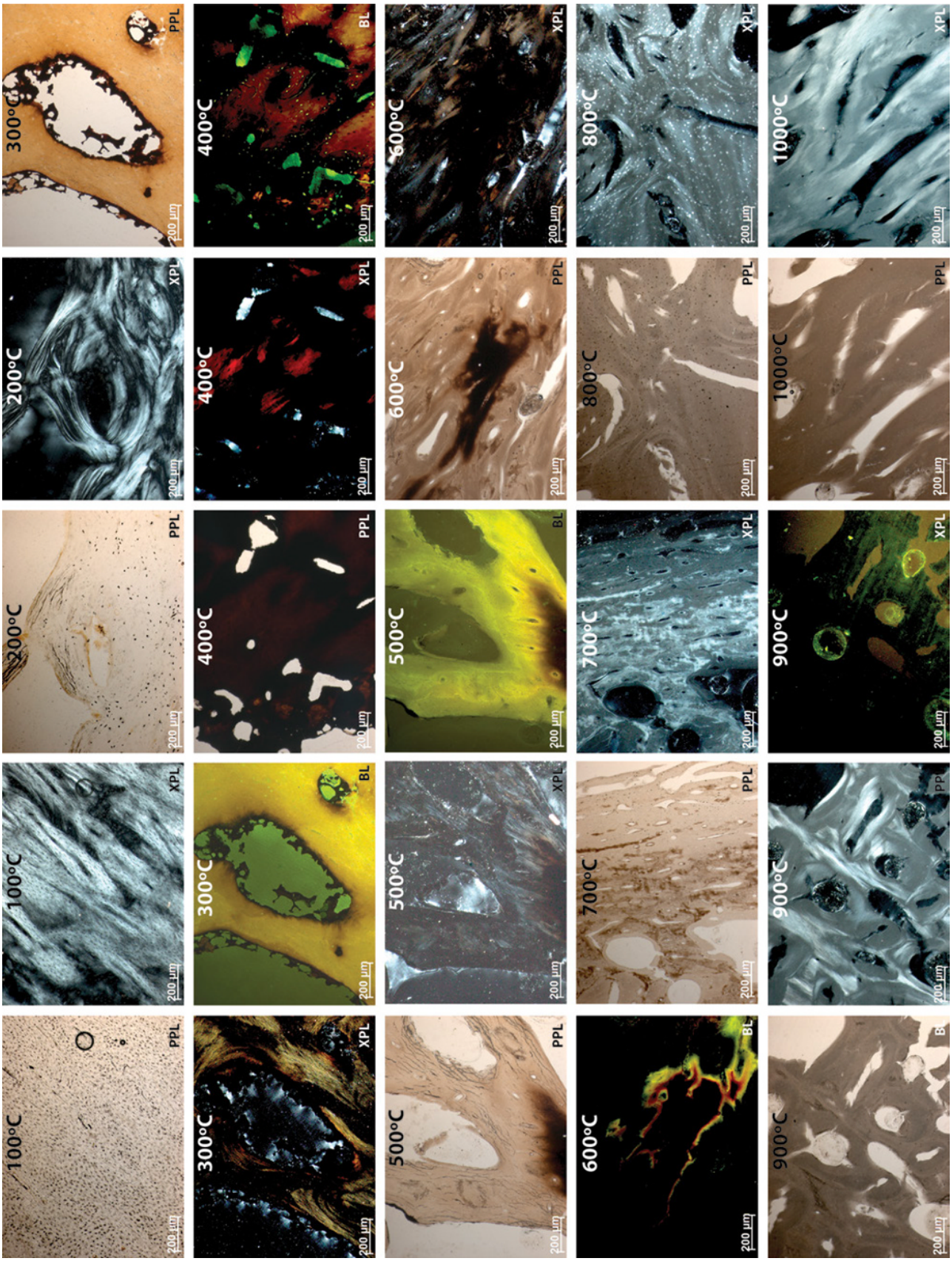


**Figure 1.10** Thin sections of ungulate bone heated from 100 °C to 1000 °C under oxidising conditions. At 100 °C sample corresponds to boiled reindeer bone, at 300–600 °C and 800–900 °C samples come from heated cow bones, and at 700 °C and 1000 °C samples are pig bones. No major difference is seen between bones heated at 100 °C and 200 °C. At 300 °C bones turn light to medium yellow (PPL) with olive brown to grey brown interference colours (XPL) and moderate to strong fluorescence (BLF). At 400 °C bones turn dark reddish brown (PPL) with red interference colours with opaque areas (XPL). Colour changes to pale brown (PPL) between 500 and 700 °C. Interference colours are olive brown to dark blue-grey between 500 and 600 °C, turning to low order grey at 700 °C (XPL). Fluorescence is now weak to moderate (BLF). Between 800 and 1000 °C bones are brown to brownish grey (PPL) with strong birefringence (XPL) and low order interference colours from bluish grey to grey with milky cast typical of calcined bone. Fluorescence is weak to absent (BLF).

cast to the birefringence of calcined bone under XPL (e.g., Schiegl *et al.* 2003; Mentzer *et al.* 2015). Schiegl *et al.* (2003), in a study of a burnt bone deposit, suggested that bones exhibiting darker, opaque colours of brown and black in PPL are likely carbonized. They also argue that bones that appear almost colourless in PPL, but with first-order interference colours in XPL, suggest a lack of collagen and recrystallization of apatite or calcination.

Several studies have reported a decrease or loss of autofluorescence under UV light as a common sign of heating in bones due to loss of collagen (Courty *et al.* 1989; Schiegl *et al.* 2003; Karkanas *et al.* 2007) (see Figure 1.11a). The results from experimental heating of pig and cow bone described in Table 1.2 and Figure 1.10 are broadly consistent with this observation. Autofluorescence under ultraviolet (UV) and blue light decreases significantly above 500 °C. In contrast, the heated fish bones described in Table 1.2 retained autofluorescence throughout the complete experimental temperature range. This difference may be due to the duration of heating (30 min). For instance, Thompson *et al.* (2009) found that, although molecular changes began to occur in bones heated for 15 , they recommended 45 min of heating for future experiments.

When heating occurs in nonoxygenated environments (e.g., bones buried in the soil underneath a fire) bones turn black macroscopically (Stiner *et al.* 1995). In thin sections, this may be seen as a coat of sootlike charred organic material on the surface of the heat-altered bone (Figure 1.11b). In larger bones, lack of oxygen during heating may result in charring of fat or marrow or in the accumulation of soot inside the bone. This results in the formation of opaque black precipitates in the bone microstructure that mask the optical properties of the bone mass (Figure 1.11c).

Despite general trends in presence and absence of certain optical properties with temperature, these properties alone are not the best approach to identifying burned bone in thin section. Mineral staining can mimic colour change in PPL caused by low temperature heating. Similarly, small fragments of charred bone can resemble other charred organic remains, particularly a black amorphous substance identified as 'fat-derived char' (Berna & Goldberg 2008; Goldberg *et al.* 2009) (Figure 1.11d). This material is produced from burning of flesh, bone and / or animal fat and has been identified in diverse archaeological contexts (Goldberg *et al.* 2012; Miller *et al.* 2013; Villagran *et al.* 2013). It exhibits numerous vesicles with small fissures or cracks radiating from the walls (Figure 31.6e in Mallol *et al.* 2017, this book) which, when the fragments are sufficiently large, should allow for distinction between it and charred bone, or 'bone char'. The appearance of pale brown or orange fragments of bone that exhibit variations in the interference colour and fluorescence can be due either to low temperature heating or to collagen decay after microbial alteration or chemical dissolution (Schoeninger *et al.* 1989; Trueman & Martill 2002; Trueman *et al.* 2004). The lack of equifinality in the optical characteristics of burnt bone and bone modified by other processes requires the use of other microanalytical techniques to determine if bones found in thin section have been subjected to heating. The application of FTIR, FTIR microscopy, XRD, histomorphometry, measurements of crystallite size and organic petrography to loose samples and thin sections has been shown to be helpful in distinguishing between bones subjected to heating and those subjected to other alteration processes (Shahack-Gross *et al.* 1997; Karkanas *et al.* 2007; Piga *et al.* 2008; Dibble *et al.* 2009; Thompson *et al.* 2009; Clark & Ligouis 2010; Goldberg & Berna 2010; Lebon *et al.* 2010; Reiche 2010; Squires *et al.* 2011; Berna *et al.* 2012; Ellingham *et al.* 2015).

### 1.3.2 Mechanical Fracturing of Bone

There are numerous causes of bone fragmentation: intentional human practices, such as butchery and burning; unintentional trampling; and syn- and post-depositional physical processes, including wetting and drying, freezing and thawing, formation of secondary salt crystals and overburden pressures. The effects of these processes have been widely investigated through taphonomic studies; however, most cannot be distinguished using micromorphology alone. Some processes, described below, can be clearly recognized in thin section, whereas others can be identified from aspects of the sedimentary matrix. Therefore micromorphology of fragmented bone can provide a valuable data set that complements more traditional zooarchaeological studies.

Trampling of bones, whether by humans or animals, can cause distinctive fractures that are readily identifiable in thin section. Experimental work conducted by Miller *et al.* (2009) determined that trampling can cause *in situ* snapping of bone, leading to articulated, accommodating fragments of slightly displaced bone (Figure 1.11e). Bones with significant pore space, such as spongy bone, will often exhibit lower degrees of accommodation and appear 'crushed'. Fracture patterning similar to that found in the experiments has been reported in archaeological thin sections and attributed to human trampling (Dibble *et al.* 2009; Goldberg *et al.* 2009). Similar types of *in situ* fracturing could be caused by pressure related to sediment overburden, an interpretation offered for fractured bone at the site of Tönchesberg (Conard 1992). However, as far as we are aware, no micromorphological studies of bones fractured by sediment overburden have been conducted. Trampling can also cause horizontal displacement of

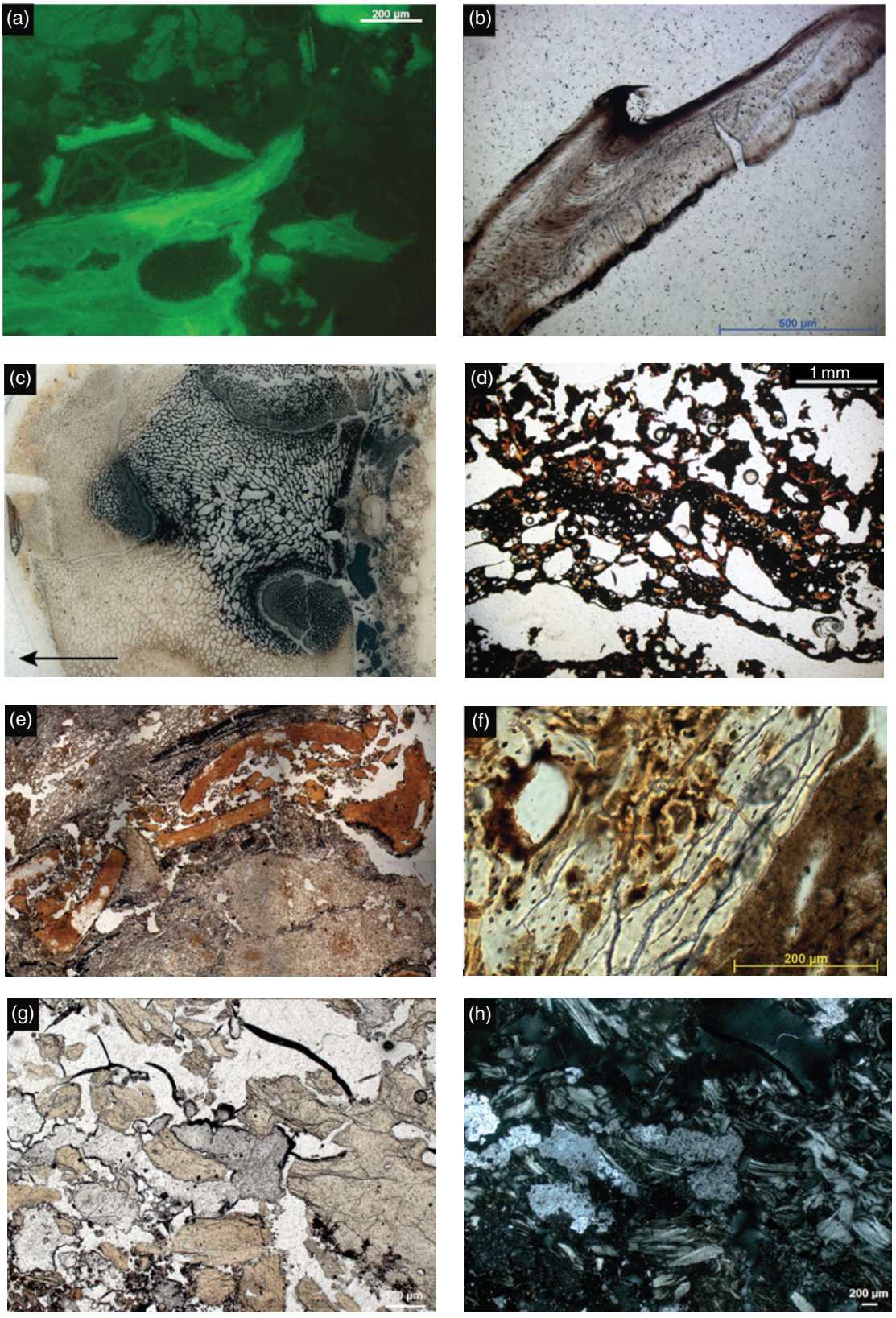


**Figure 1.11** Various effects of heating on bone (a–d). (a) Burnt and fresh bones from the Santa Marta 8 fish midden (c. 1550 cal. years BP, Santa Catarina State, Brazil). Note the auto-fluorescence of fresh bone fragments compared to the weak to absent fluorescent burned fragments. BLF. (b) Herring bone experimentally heated to 700 °C in absence of oxygen. Note the dark edges due to the presence of soot. PPL. (c) Thin section scan showing a fragment of horse bone experimentally burnt for 8 hours in an open hearth with average temperatures below 300 °C. Note the dark colours on the inside of the bone due to charring. Image width 68 mm. Image courtesy: C. Mallol. (d) Fat-derived char from a combustion feature at the seventeenth–eighteenth century site of Sealer 4, Livingston Island (Antarctica – see Villagran *et al.* 2013). Note its characteristic homogeneous black colour and vesicular shape that can easily be mistaken of charred spongeous bone (see Figure 1.8c). PPL. Bone cracking and fragmentation (e–h). (e) The breakage pattern, with accommodating fragments in articulated position or with little displacement indicates *in situ* breakage, probably due to trampling. Iron Age midden-like deposit (Oosterbeintum, the Netherlands). PPL. (f) Localized zone of cracking in surface of rodent skull (see Figure 1.3a, b). This pattern occurs only in the outer surface layers of the bone, and is probably due to preburial exposure. PPL. (g) and (h) Secondary minerals forming in-between bone fragment and breaking it apart. Middle Stone Age deposits in Sibudu (South Africa): (g) PPL; (h) XPL.

materials, including fragments of bone. In this case, the bone fragments do not exhibit articulation or accommodation but they appear subrounded and incorporated within deposits composed of a heterogenous mix of various components (Goldberg *et al.* 2009; Miller *et al.* 2013).

Bone exposed on the surface or subsurface for extended periods can be affected physically by repeated drying and wetting and / or freeze-thaw cycles. Such cycles can result in physical damage characterized by patterns of fine fissures, as seen in Figure 1.11f. Schiegl *et al.* (2003) and Miller (2015) report a deposit composed almost exclusively of sand-sized fragments of bone at the cave site of Hohle Fels; the bone fragments appear subrounded and exhibited laminated coatings of calcareous silt, which the researchers interpret as having formed by cryoturbation.

Moisture containing dissolved ions within archaeological deposits can collect within pre-existing fissures and pores in bones where, upon evaporation, crystals can form. As the crystals grow they exert pressure on the surrounding bone, which leads to fracturing and fragmentation (Figure 1.11g, h). This process is most common in sites that are relatively dry, and have alkaline burial conditions with sources for dissolved ions that preferentially form crystals (Behrensmeyer 1978). Crystal-induced fragmentation of bone due to gypsum formation has been reported from the rockshelter sites of Sibudu (Goldberg *et al.* 2009) and Diepkloof (Miller *et al.* 2013) in South Africa. A similar process has been observed with secondary calcite at Qesem (Karkanas *et al.* 2007) and Obi-Rakhmat (Mallol *et al.* 2009).

### 1.3.3 Alteration through Biological Processes

Biological activity can contribute to the mechanical and chemical transformation of bone in archaeological sites. The type and scale of impacts depend on the organism responsible. For example, fungi and plant roots produce characteristic chemical weathering patterns on bone surfaces (Behrensmeyer 1978). Aside from mechanical fragmentation due to root activity, the effects of plants on bone are difficult to identify in thin section. In contrast, other nonhuman biological taphonomic agents are more recognizable. These include macrofauna, mesofauna and microbes.

#### 1.3.3.1 Macro- and Mesofaunal Decay

Carnivores and omnivores scavenge and consume fresh and ancient bones from surface and subsurface deposits in archaeological sites. These activities are evidenced by macroscopic damage as well as bone fragments visible in coprolites (see Brönniman *et al.* 2017, this book). Macphail & Goldberg (2010) note that leaching of bones during their passage through the digestive channels of dogs, humans and birds results in loss of both birefringence and autofluorescence. However, this may not always happen. For example, bones in dog or human coprolites from the site of Swifterbant S4 (Huisman *et al.* 2009) do not show evidence of dissolution. Alteration by soil mesofauna can sometimes be documented in thin section. For example, in temperate environments, earthworms and insects may consume bone fragments and excrete them in their faecal pellets (see for example Figure 1.12a). In tropical settings, decay of bones by termites is a well-known phenomenon (e.g., Huchet *et al.* 2011; Backwell *et al.* 2012).

#### 1.3.3.2 Microbiological Decay

Bone collagen is rich in nitrogen, which makes it a desirable potential resource for microflora and fauna. Furthermore, if bone is buried as part of a complete body, putrefaction and decay of soft tissue can promote decay of bone by bacteria. Microbiological decay of bone does not usually occur in environments that are waterlogged, arid, or permanently frozen, as these setting impede bacterial activity. Microbial decay results in specific decay patterns, known as microscopical focal destructions (MFDs). There are different types of MFDs, which, based on their morphology, are named linear longitudinal, budded or lamellate tunnelling. Some of these types may be visible in thin section (Figure 1.12b). It can be unclear whether the different morphologies of alteration indicate different species, different circumstances or different stages in bacterial attack (Jans 2014; see also Hedges & Millard 1995), although some MFD morphologies are characteristic of type or species of bone-degrading mechanisms (Trueman & Martill 2002; Jans 2005).

In noncalcareous environments with available moisture and oxygen, saprophytic fungi can colonize and degrade bone (see Forancelli *et al.* 2012). Fungal degradation can be recognized in thin section by visible fungal hyphae, or the occurrence of branching tunnels ('Wedl tunnels') that run through the bone tissue (Figure 1.12c) although some tunnelling ('Wedl type 2') may be of bacterial origin (Trueman & Martill 2002). In waterlogged settings, fungal decay is not possible; however, traces of fungal decay in these environments can indicate that the decay occurred prior to saturation, providing evidence for a change in soil environment. Bone submerged in shallow, clear water may become colonized by cyanobacteria. These microbes tend to tunnel into the outer bone layer (Figure 1.12d), sometimes leaving small spheres of hydroxylapatite behind in their cavities (Turner-Walker & Jans 2008; Turner-Walker 2012).

### 1.3.4 Chemical Weathering

The combination and special arrangement of collagen and carbonated hydroxylapatite in bones makes bone frequently more resistant to decay than other materials in the archaeological record (Collins *et al.* 2002).