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## Examining histology to identify burned bone

Megan Hanson\*, Chester R. Cain

School of Geography, Archaeology and Environmental Sciences, University of the Witwatersrand, Johannesburg, South Africa

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## Abstract

Early research suggests that examination of the microscopic internal structure is a valid method for distinguishing burned from unburned bone in the archaeological record. This study compares burned and unburned modern bones with archaeological bones from Sibudu Cave (*ca.* 60,000 years ago) and Cave of Hearths (over 200,000 years ago) to more fully describe the heat-induced histological changes to bone. We also explore the effects of diagenesis on those changes to determine if histological evidence of burning preserves through fossilisation and diagenetic processes and can be successfully used to identify ancient burning. Application of this technique can help in addressing various issues, including the origins of controlled use of fire and understanding animal butchery and disposal. © 2007 Elsevier Ltd. All rights reserved.

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## 1. Introduction

The identification of hominin-controlled fire and cooking in the archaeological record is an important line of evidence in unravelling ancient behaviour. Fire provides light, heat, protection from predators, assistance in hunting, and a means of cooking meat and plant materials (Bellomo and Harris, 1990; James, 1989; Oakley, 1961). Researchers, therefore, have long been concerned with identifying the early use of fire at archaeological sites (Clark and Harris, 1985); however, many assertions for the earliest use of fire still remain controversial (Binford and Stone, 1986; Dart, 1948; Goldberg et al., 2001; Oakley, 1961; Weiner et al., 1998; Yu-Zhu, 1986).

Researchers employ a range of analyses to demonstrate or disprove hominin use of fire (Barbetti, 1986; Bellomo and Kean, 1994; Brain, 1993; Schiegl et al., 1996; Shipman et al., 1984), and, as an abundant material that preserves in most archaeological sites, bone is often evaluated for thermal

E-mail address: meganleighhanson@msn.com (M. Hanson).

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alterations in attempts to identify use of fire. Burned bone is a good indicator of fire because it is very susceptible to heat, and changes in bone are frequently distinctive and are usually not affected by recovery techniques.

Thermally altered bone displays surface colour and texture changes (Brain, 1993; David, 1990; Shipman et al., 1984), which some argue can be good indicators of the temperature to which the bone was heated, as well as to the duration of the heating (Brain, 1981, 1993). When burned, bone surface first darkens to brown (at less than 400 °C), then carbonises, turning black (typically around 400-500 °C), and once the organic components are burned, the bone will become grey to grey-blue in colour (at 600-900 °C) (Bonucci and Graziani, 1975; Brain, 1993; Correia, 1997; Shipman et al., 1984). In the final stage, the bone becomes calcined, developing a chalky consistency and white colour (Brain, 1993). Heated bones also display observable cracking and occasional longitudinal fractures (Correia, 1997; David, 1990). Unfortunately, visual surface assessments can be misleading as organic and mineral staining can mimic colour changes (Oakley, 1961; Shahack-Gross et al., 1997), and the surface of weathered bone sometimes resembles that of burned bone (Sillen and Hoering, 1993; Stiner et al., 1995; Taylor et al., 1995; White, 1992).

<sup>\*</sup> Corresponding author. 1252 Maryland Avenue NE Apt. B, Washington, DC 20002, USA. Tel.: +1 202 445 1106.

Several researchers have therefore turned to more rigorous analyses of surface and internal structure to identify burned bone.

After examining the most superficial layers of experimentally burned bones at  $55,000 \times$  magnification, Bonucci and Graziani (1975) found that hydroxyapatite crystals thickened as temperature increased until the orderly crystalline structure of the bones disintegrated at 900 °C, with the first noticeable indication of burning appearing at 350 °C. Using this experimental data, Bonucci and Graziani (1975) were able to identify similar crystal changes in 2500-year-old cremated human bone, but they had more difficulty identifying such changes in older, apparently burned fossilised bone from Mousterian levels at an Italian cave site. (Bonucci and Graziani (1975) also conducted thermogravimetry studies to identify changes in burned bone, but they were inconclusive when applied to ancient bone and have seldom, if ever, been used since.)

Using experimentally heated bone, Shipman et al. (1984) delineated a series of colour and surface changes, visible with both light and scanning electron microscopy, that occur as heating progresses to higher temperatures, with the first indication of burning appearing between 185 °C and 285 °C. The applicability of these schemes to archaeological assemblages is preliminarily confirmed by Nicholson's (1993) work. However, as both Nicholson (1993) and White (1992) have noted, similar surface texture and colour changes can occur on unburned bones diagenetically altered by weathering and fossilisation. The high-powered microscopic surface analvses performed by Bonucci and Graziani (1975) and Shipman et al. (1984), in the end, possess many of the same problems as macroscopic surface analyses-burning, weathering, and diagenesis all cause similar changes to the bone surface and the causes of these changes can be confused.

Chemical analyses of reportedly burned bone can test for the presence of organic carbon, the carbon: nitrogen ratio, ammonia (NH<sub>3</sub>) levels or amino acid patterns; all of which are associated with burning in bone (Brain and Sillen, 1988; Sillen and Hoering, 1993; Taylor et al., 1995). While organic carbon analyses can distinguish between bone that is black in colour due to heating and bone that is black due to staining by manganese oxide, they cannot work on bone after the carbon has oxidised, which usually occurs between 600 °C and 900 °C (temperatures that are within the range of human campfires). Sillen and Hoering (1993) note that the data from carbon: nitrogen analyses can be difficult to interpret and Taylor et al. (1995) conclude that the ammonia and amino acid analyses might not work on bones that had undergone long periods of diagenesis. All of these chemical analyses fail to provide information on the intensity of heating, including temperature or duration.

Experimental work has shown that x-ray diffraction and infra-red spectrometry can be used to identify heat-induced crystallographic transformations in modern bones (Shipman et al., 1984; Sillen and Hoering, 1993; Stiner et al., 1995), but applications to archaeological material have had mixed results. Stiner et al. (1995) found that when the technique was used on Middle Palaeolithic archaeological bone from Hayonim Cave, Israel, the signatures displayed no significant differences between burned and unburned specimens, whereas Shahack-Gross et al. (1997) were able to differentiate between infra-red spectra from burned and unburned bones from the same site. Shahack-Gross et al. (1997) did note that it is possible that other forms of diagenesis could result in bones with similar spectra to that of pyrolised collagen. Others have noted that it is difficult to recognise changes in crystal size in archaeological bone that was heated to less than 650 °C (Harsanyi, 1993; Stiner et al., 1995).

Because weathering and fossilisation do not significantly affect the internal structure of the bone, examining bone histology (the internal structure as visible with light microscopy) is at this stage a reliable indicator of burning without the problems of equifinality seen in the techniques previously mentioned. Much of the microscopic analysis of the internal structure of burned bone to date has been conducted by forensic anthropologists, who have identified important heatinduced changes to human bone histology (Bradtmiller and Buikstra, 1984; Cattaneo et al., 1999; Forbes, 1941; Harsanyi, 1993; Herrmann, 1977; Hummel and Schutkowski, 1993; Nelson, 1992). Only a few archaeologists have pursued such investigations. Schiegl et al. (1996) examined thin soil sections with in situ bone as part of a study of hearths and chemical and other signatures of burning. They reported that the colour of the apparently burned bones in thin section varied from pale brown to dark brown and deep black.

Brain (1993) documented accentuation of the lamellar structure from carbon deposition (which he attributed to carbonised organic components), cracks spreading through the matrix, and a general loss of microstructure at high temperatures. In his experiment, Brain (1993) burned bone sections to seven different temperatures in a kiln for 30 min. His results are presented in Table 1. Brain used his experimental observations to assign approximate temperatures to four reportedly burned fossil specimens from the early hominid (1.0-1.5 MYR BP) site of Swartkrans. The descriptions in this research were limited, however, and there were no descriptions of the histological features of unburned fossils.

Table 1

Changes observed in microstructure of burned bone, as described by Brain (1993)

Sustained temperature (°C)	Observed changes
200	Carbon accumulated in the lacunae, but most of the structure looks unchanged
300	Lamellar structure has more carbon and is accentuated with a brown-black colouration; cracks spread outwards from the haversian canals through the matrix
400	Cracks spread outwards from the haversian canals through the matrix
500	Most of the carbon has oxidised, leaving the matrix a pale colour with many cracks
600	Cracks are numerous and prominent throughout the matrix, but structure is still visible
700	The matrix has shrunk, making the cracks even wider
800	Microstructure has largely disappeared due to the fusion of the apatite crystals

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