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Burnt Bones and Teeth: an Experimental Study of Color, Morphology, Crystal Structure and Shrinkage

Pat Shipman,[a] Giraud Foster[b] and Margaret Schoeninger[a]

Burnt osteological materials are one focus of interest in forensic, archaeological, and palaeontological studies. We document the effects of experimental, controlled heating on a sample of modern bones and teeth from sheep and goats. Four aspects of heating specimens to between 20 and 940°C were considered: color, microscopic morphology, crystalline structure and shrinkage. Our results show that changes in both color and microscopic morphology of burnt bones and teeth can be divided into five stages each of which is typical of a particular temperature range, although the stages based on color do not correlate exactly with those based on micromorphology. These stages can be used to determine (1) if specimens of unknown taphonomic history were burnt, and (2) the maximum temperature reached by those specimens. In addition, powder X-ray diffraction studies show that heating causes an increase in the crystal size of hydroxyapatite, the major inorganic component of bones and teeth. This fact in conjunction with the microscopic morphology can be used to confirm deduced heating to 645°C or more. The data on shrinkage are analyzed to yield a polynomial expression that summarizes percentage shrinkage as a function of the maximum temperature reached by bones. Thus, the original size of specimens can be reconstructed within limits since the maximum temperature reached by the bones can be deduced on the basis of color, microscopic morphology and/or powder X-ray diffraction patterns. Finally, because there is a discrepancy between the maximum heating device temperature and the maximum specimen temperature, caution must be exercised in distinguishing between the effects of man made and natural fires.

Keywords: BURNT BONES, COLOR CHANGES, MICROSCOPIC MORPHOLOGY, X-RAY DIFFRACTION, SHRINKAGE, TEMPERATURE.

Introduction

The analysis of burnt skeletal remains is a topic of importance to scientists in several fields. Forensic specialists are frequently confronted with deliberately or accidentally burnt human remains (e.g. Krogman, 1949; Stewart, 1979); archaeologists discover human cremations, animal sacrifices, and bones burnt during firing of cities or villages (e.g. Buikstra & Goldstein, 1973; Gejvall, 1963; Merbs, 1967; Ubelaker, 1978); and palaeontologists are faced with possible cases of cannibalism (Le Mort, 1981; Roper, 1969; Smith, 1976) or of earliest uses of fire (Gowlett *et al.*, 1981; Oakley, 1956). In the case of palaeontological remains, the acquisition of fire is regarded as a major technological breakthrough that probably led to substantial changes in lifestyle (Campbell, 1978; Dart, 1948; Oakley, 1961); thus, establishing accurately when this development occurred is a crucial task. Unfortunately, claims to date for the earliest uses of fire have been controversial. Dart's initial claims (1948) for early fire were based

[a]Department of Cell Biology and Anatomy, The Johns Hopkins University School of Medicine, 725 North Wolfe Street, Baltimore, Maryland 21205, U.S.A.
[b]Department of Obstetrics and Gynecology, The Johns Hopkins Hospital, Baltimore, Maryland 21205, U.S.A.

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on mistaking magnesium oxide stains for charcoal (Oakley, 1961). Gowlett *et al.* (1981) present evidence for fire at Chesowanja some 1·4 million years ago, based on burnt clay that shows a magnetic moment produced by heating to about 400°C—a normal campfire temperature (Tylecote, 1962: 25), but one only briefly produced by bush fires and exceeded by burning tree stumps. However, Isaac (1982) argues that the spatial association of the clay with the artifacts and bones is unclear because of fluvial disturbances and that the possibility of burning trees is inadequately explored (but see also Gowlett *et al.*, 1982).

This paper presents the results of a series of experiments involving the effects of burning sheep or goat remains under controlled conditions. Four aspects of the effects were considered: color changes; changes in microscopic morphology; crystalline structural changes; and shrinkage. The results of this study will enable researchers to identify with certainty skeletal remains that have been burned to deduce the maximum temperature to which those remains were heated, and to reconstruct, within limits, the original dimensions of the bone.

Knowledge of the maximum temperature reached by a skeletal element will, in turn, suggest the mode of heating employed, since various studies have documented the maximum temperature reached by fires or other heating devices. Buikstra & Swegle (n.d.) directly measured temperatures of 680–820°C for juniper and oak fires; the coals of an oak fire were measured at about 900°C. Tylecote (1962: 25) reports that 400°C is a normal temperature for a campfire and states that such fires rarely reach 700°C. On the other hand, Shepard (1956) has measured a maximum temperature of 962°C for wood stacked around pottery in the open. Objects heated in campfires may reach temperatures of 380–550°C (flints: Rowlett *et al.*, 1974) or even 600–900°C in pits or improvised ovens (ceramics: Shepard, 1956). Glass beads in cremation pyres have reached 850–940°C (Wells, 1960). Prairie fires reach a maximum of about 700°C, but the duration of temperatures in excess of 65°C is only 1–10 min (Stinson & Wright, 1969). Burning piled logs and slash produces higher maximum temperatures (1430°C) and sustains temperatures in excess of 800°C for at least 40 min (Wright & Bailey, 1982).

In previous studies, attempts have been made to derive criteria for deducing the duration, heat, or type of burning undergone by skeletal remains. Baby (1954), Binford (1963), Buikstra & Swegle (n.d.), Franchet (1933, cited in Le Mort, 1981), Krogman (1949), Merbs (1967) and others have described the gross appearance of bones burned under various conditions (kiln, oven, furnace, campfire, etc.), controlling for such factors as the condition of the bones at the onset of burning, the amount of soft tissue present on the bones, the duration and intensity of heating. Various authors agree that bones pass through a continuum of appearance ranging from unburnt (normal-colored) bones, to non-incinerated or smoked bone that shows blackening on the edges, to incompletely incinerated bone (blackened or dark brown in color), to completely incinerated or calcined bone that is usually described as bluish-white or grey in color (see summaries in Buikstra & Swegle, n.d.; Le Mort, 1981; Stewart, 1979; Ubelaker, 1978). The development of cracking, checking and warping of burnt bones is also widely described, although the conditions producing these effects are incompletely understood (Baby, 1954; Binford, 1963; Buikstra & Swegle, n.d.; Stewart, 1979). Unfortunately, there is considerable variability in the effects observed in these studies, due both to the factors enumerated above and to the subjectivity of judgments of the color or texture of burnt bones.

Herrman (1972, 1976, 1977) and Van Vark (1970) have investigated microscopic features of sections of burnt bones. Both agree that features such as osteons remain visible after heating to temperatures below 800°C, which means that age determination

techniques that rely on the density of osteons (Ahlqvist & Damsten, 1969; Kerley, 1965; Singh & Gunberg, 1970) can be applied. Herrman (1972, 1976, 1977) also notes that sintering or fusion of crystals occurs in bone heated to temperatures above 800°C. However, neither investigated the changes in natural surfaces of bones or teeth with heating, and it is unclear to what extent sectioning bones alters their surface texture. It would not, of course, alter the number or distribution of osteons. Van Vark (1970) also investigated shrinkage, finding that it varies with temperature.

In this study, we have assessed changes in color, microscopic features, crystal structure and shrinkage induced by heating on natural surfaces of bones and teeth. Major aims of this project were to provide an effective approach to deducing past events involving heating or burning of skeletal materials as well as a means of reconstructing the original dimensions of burnt bones.

Both dental and osteological remains were used in this study. Although several authors remark that whole burnt teeth are infrequently recovered, only roots or fragments being found (e.g. Buikstra & Goldstein, 1973; Wells, 1960), the extreme usefulness of teeth in species identification, age estimation and sex determination warrants their inclusion in this study. Further, our results show that highly fragmented teeth are as useful for determining the extent of heating as are complete teeth or bones.

Materials and methods

Two skeletal elements, differing widely in preservation potential, ultrastructure and placement in the body were chosen for study: the mandible and the astragalus. We decided to use the bones and teeth of goats and sheep because these species, or other similar small bovids, are frequently present at archaeological sites. In many parts of the world, sheep and goats are a major food item, a major member of the animal community, and/or a sacrificial animal of major importance. There is no evidence to suggest major structural differences among bones of different mammalian species.

We manually cleaned 60 mandibles and astragali of goats and sheep aged approximately 2–6 months (first molars had erupted). Three astragali and two mandibles were placed at a time in a Norman test Kiln (model T-8), which was heated with the specimens inside for 4 h until a given maximum temperature was reached by both the specimens and the furnace. Maximum temperatures chosen corresponded to the settings on the muffle furnace: 1 = 185°C; 1·5 = 285°C; 2 = 360°C; 2·5 = 440°C; 3 = 525°C; 3·5 = 645°C; 4 = 675°C; 4·5 = 745°C; 5 = 800°C; 5·5 = 870°C; 6 = 940°C. The furnace and specimens were then allowed to cool for 4 h before the specimens were removed. We recorded temperatures at 1 h intervals throughout the experiment (Figure 1). Two mandibles and three astragali were retained in their unburnt condition as control samples.

The colors of bone and tooth of each specimen were recorded using the *Munsell Soil Color Charts* (Munsell Color Company Inc., 1954) after burning or, in the case of the controls, before burning. The Munsell system records colors by hue, value and chroma, and so offers a standardized, reproducible system of describing or quantifying colors. We recorded both predominant and minor colors of bones and teeth in our sample.

Microscopic changes of different tissues were monitored at several locations. We inspected bone samples from both the lateral side of the mandible and the articular surface of the astragalus. Enamel surfaces were inspected at two points, one located well above the gingival line (location A) and one located just apical to the gingival line (location B). We inspected the dentinal surface inside the pulp cavity, to avoid any differences in morphology that might be produced by differences in fracture planes.

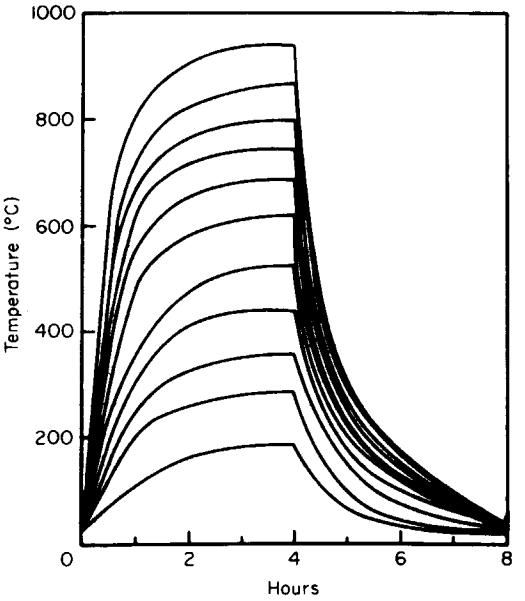

Figure 1. Temperature of samples plotted against time. Each line represents the temperature curve for several specimens heated to a particular temperature for 4 h and then cooled for 4 h.

All dental tissue samples were taken from both lingual and labial surfaces of the first mandibular molar, but no difference was found between these samples.

Samples of the various tissues were cleaned initially with a mild detergent in an ultrasonicator. We cleaned the surfaces further with alcohol and acetone applied with a soft artist's brush. Specimens were then mounted on scanning electron microscope (SEM) stubs and coated with 200 Å of gold-palladium to render them conductive (Hayat, 1978). We inspected the samples under the SEM using various orientations and tilts at magnifications ranging from 15× to 15,000×. Care was taken during microscopic inspection to avoid areas covered by remnants of carbonized or intact soft tissue.

Bone samples were prepared for X-ray diffraction analysis by grinding them to a fine powder with an agate mortar and pestle. A small amount of the sample was mixed with acetone and then applied in an even layer to a glass slide. This procedure minimizes variations in patterns that may result from analysis of a range of particle sizes or of samples of varying thickness and assures random orientation of crystal planes. The diffractometer used copper *k* alpha radiation and the machine parameters were: rate = 1×10^3, 2 degrees min^{-1}, 1 inch min^{-1}, voltage = 40. Ground samples were prepared from goat astragali heated to each temperature setting between 185 and 675°C and from one sample heated to 940°C ($N = 9$).

Size reduction or shrinkage was assessed by taking four different measurements, three on each astragalus and one on each mandible (Table 1), prior to and after heating. The percentage shrinkage was calculated for each specimen at each temperature as follows:

$$[(\text{original dimension} - \text{altered dimension})/\text{original dimension}] \times 100$$

Table 1. Measurements used in assessing shrinkage

Bone	Measurement	Description
Astragalus	a	Maximum length of the lateral aspect
Astragalus	b	Maximum length of the medial aspect
Astragalus	c	Minimum longitudinal circumference, as taken near the midline
Mandible	d	Minimum corpus circumference as measured in the diastema

Table 2. Munsell colors on burnt bones and teeth

	Tissue	Temperature (°C)	Predominant color	Minor colors
Stage I	Bone	20	5Y8/2	
	Tooth	20	5Y9/2	
	Bone	185	10YR8/4	2·5Y8/4, 10YR7/8
	Tooth	185	2·5Y8/4	2·5Y8/2,6, 2·5Y9/4
Stage II	Bone	285	N2·75/0	2·5YR3/2, 7·5YR4/2
	Tooth	285	5YR3/4	5YR4/3
	Bone	360	N2·75/0	7·5YR6–7/2
	Tooth	360	N3/0	N2,4–5/0
	Bone	440	7·5YR8/3	10YR2,4/1
	Tooth	440	10YR3/1	5Y8/1, 7·5YR6/6, 10YR4/1,4
Stage III	Bone	525	10YR7/2	N6·75,8/0, 10YR5/3, 10YR6/2
	Tooth	525	5PB3/1	7·5YR6/2, 10YR7/3, 5PB7/6
Stage IV	Bone	645	N9/0	10YR6/2, 10YR7–8/1
	Tooth	645	5PB7/4	N4·5,7,8·25,9·25/0, 5PB7/6, 5PB9/1
	Bone	675	N9·5/0	N6·5/0, 10YR6–7/1, 5PB4/4, 5PB6/2
	Tooth	675	5PB7/4	N8·5/0, 5PB8/1
	Bone	745	N9·5/0	10YR6/1–2, 10YR8/2
	Tooth	745	N9·5/0	2·5Y7/1, 10YR2/1, 10YR9/2, 2·5PB8/2,4,6
	Bone	800	N9·5/0	7·5YR8/4
	Tooth	800	N9·5/0	N3,7/0, 2·5PB8/2, 5PB9/1
	Bone	870	N9·5/0	7·5YR8/4
	Tooth	870	N9·5/0	N3,3·25/0, 2·5YR6/2
Stage V	Bone	940	N9·5/0	7·5YR8/6
	Tooth	940	N9·5/0	N3·5,4/0, 5YR7/6, 10YR8/3

Different percentages were averaged for all specimens heated to a given temperature (three astragali and two mandibles for each temperature). No measurements were taken on teeth because we expected them to crack and fragment rather than shrink (but see Krogman, 1949).

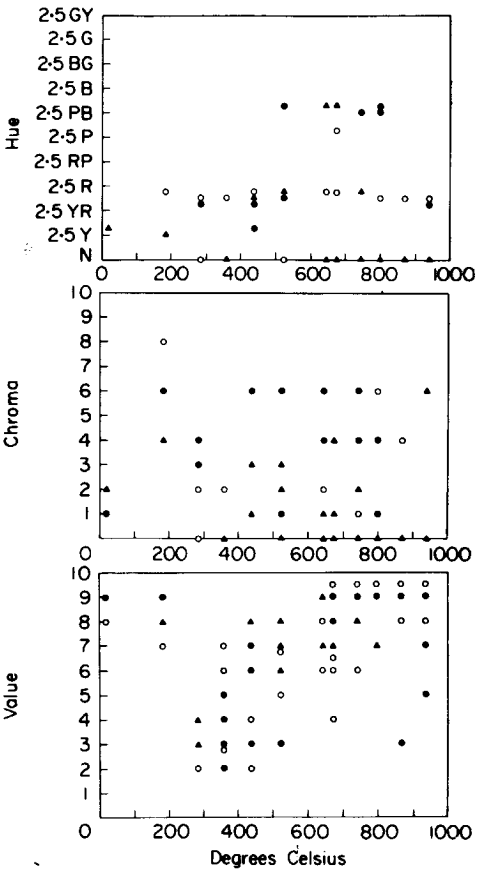

Figure 2. Changes in the hue (a), chroma (b) and value (c) of bones and teeth with increasing temperature. Note the abrupt diversification of hue, chroma and value between 360–525°C. N = neutral; Y = yellow; YR = yellow-red; R = red; RP = red-purple; P = purple; PB = purple-blue; B = blue; BG = blue-green; G = green; GY = green-yellow. Hue, chroma and value are assigned according to the Munsell Color Charts. ●, Teeth; ○, bones; ▲, teeth and bones.

Results

Color

Table 2 presents the predominant and minor colors observed on the samples at each temperature in Munsell notation. As can be seen from Figure 2, both dental and osteological materials follow a similar pathway of color changes. Hue starts in the yellow range, passes through reds and purples, and changes to diverse neutral hues above about 400°C. Value and chroma both start high, drop abruptly, and diversify to high and low values above about 400°C. Thus, all three components of color show progressive diversification with heating and all show the first appearance of low or neutral values at about 400°C.

The color data from our samples fall into five distinct stages, with the verbal descriptions given here supplementing the numerical Munsell data given in Table 2. In Stage I (20–<285°C), specimens are commonly neutral white, pale yellow and yellow;

Table 3. Heating stages of dentin: microscopic morphology

Stage I
Temperature 20–<185°C. The dentinal surface of the pulp cavity is normal and unaltered [Figure 3(a)]. The calcospherites typical of the growing dentinal surface are clearly visible and are pierced by a regular array of smooth-edged, circular openings to the dentinal tubules
Stage II
Temperature 185–<285°C. The peritubular matrix is visibly shrunken and separated from the intertubular matrix [Figure 3(b)]. The surface of the intertubular matrix shows many small asperities that give it a roughened texture
Stage III
Temperature 285–<440°C. The asperities that appeared in Stage II have melted and smoothed out [Figure 3(c)]. The division between peritubular and intertubular matrix is only infrequently visible. The openings of the dentinal tubules are elongated, rather than being circular, and the intertubular matrix forms a network of bars between the openings
Stage IV
Temperature 440–<800°C. This stage is marked by the appearance of many particles which give the surface a frothy or fleecy texture and by the increasing elongation and enlargement of the tubule openings [Figure 3(d)]. Some areas of glassy texture, perforated by irregularly-shaped openings, are visible
Stage V
Temperature 800–940°C. The frothy protuberances typical of Stage IV have coalesced into globules that fuse into nodular spikes arranged in a staggered array [Figure 3(e),(f)]. The spaces between the spikes are the remnants of the tubules themselves and the spikes are the remnants of the intertubular bars

Table 4. Heating stages of enamel tissues: microscopic morphology

Stage I
Temperature 20–<185°C. As with dentin, Stage I enamel is normal and unaltered. In location A, the typical honeycomb pattern is seen and in location B, the sinuous arrangement of fibers is visible [Figures 4(a) and 5(a)]
Stage II
Temperature 185–<285°C. Location A & B enamel develops dimples, but the overall surface texture is smoother than in Stage I [Figures 4(b) and 5(b)]
Stage III
Temperature 285–<440°C. Rounded particles appear, covering the surface [Figures 4(c) and 5(c)]
Stage IV
Temperature 440–<800°C. Both enamel surfaces are marked by the appearance of vitrified or glassy particles separated by many pores and fissures [Figures 4(d) and 5(d)]. Location B enamel breaks up into smaller fragments during Stage IV
Stage V
Temperature 800–940°C. The fine particles of Phase IV coalesce into larger, smooth-surfaced globules that fuse into an irregularly-shaped mass pierced by rounded holes [Figures 4(e) and 5(e),(f)]

in Stage II (285–<525°C), common colors are reddish brown, very dark grey-brown, neutral dark grey, and reddish-yellow; in Stage III (525–<645°C), specimens are neutral black, with medium blue and some reddish-yellow appearing; in Stage IV (645–<940°C), neutral white predominates, with light blue-grey and light grey also present; in Stage V (940+°C), specimens are neutral white with some medium grey and reddish-yellow. It is apparent that color alone is insufficient to identify precisely

Table 5. Heating stages in bone tissues: microscopic morphology

Stage I
Temperature 20–<185°C. Normal bone texture is present. In both types of bone, the surface is gently undulating. The subchondral bone, in particular, is pierced by many vascular canals at regular intervals. The bone surface is intact and continuous [Figure 6(a)]
Stage II
Temperature 185–<285°C. The bone surface becomes more irregular as small, granular asperities, separated from each other by tiny pores and fissures, appear [Figure 6(b)]. The bone surface remains intact and continuous
Stage III
Temperature 285–<440°C. The pores and asperities typical of Stage II disappear; the bone surface becomes glassy and smoother than it was in Stage I. Areas of bone may take on a vitrified appearance [Figure 6(c)]. At temperatures greater than or equal to 285°C, subchondral bone develops a distinctive polygonal cracking pattern not found in bone subjected to other taphonomic events (Shipman,1981*a*, *b*). These plates are formed by the propagation of cracks between vascular canals, apparently as a consequence of dehydration. The edges of the plates are sharply demarcated and roughly perpendicular to the bone surface
Stage IV
Temperature 440–<800°C. The bone surface becomes highly particulate in the early parts of this stage, and rapidly turns frothy [Figure 6(d)]
Stage V
Temperature 800–<940°C. The particles of the previous stage melt and coalesce into larger structures. In the astragalar sample, a blanket of rounded polygonal structures with a smooth and nearly featureless upper surface was observed [Figure 6(e),(f)]. In the mandibular samples, these structures have a distinctive nodular appearance similar to that observed in Stage V dental samples [Figure 6(g)]

the temperature to which a tooth or bone has been heated, although it can be used to deduce a range into which the temperature of heating falls.

Microscopic morphology

High power SEM inspection of dental and osteological samples revealed dramatic changes, although these were not always visible at lower power (less than 1,000×). Our study indicates that the most useful magnification for distinguishing among bones and teeth heated to different temperatures are in the range of 1000–10,000×, since lower magnifications often failed to reveal the changes that are most diagnostic. Light microscopy revealed only the development of cracks in the bones and teeth, which was judged of little use in assessing temperature of burning, especially since fossils or buried bones frequently develop cracks from other causes.

The changes undergone by dentin, enamel and bone surfaces can be divided into five stages. Although these stages seem to occur at the same temperature thresholds in all three tissues, they do not correlate exactly with the color stages described above. Further, the stages described below probably occur as a continuum of morphological changes rather than in abrupt steps; thus, it is not uncommon to find a specimen with patches of two or more stages. The most advanced stage represented on an area of more than 300 μm^2 is used here as the stage reached by a specimen.

The types of morphological changes observed at each stage were very similar in dentin, the two areas of enamel surface, and the cortical and subchondral bone. They are fully described and figured in Tables 3–5 and Figures 3–6. A general description of the stages is: in Stage I (20–<185°C), tissues are normal; in Stage II (185–<285°C),

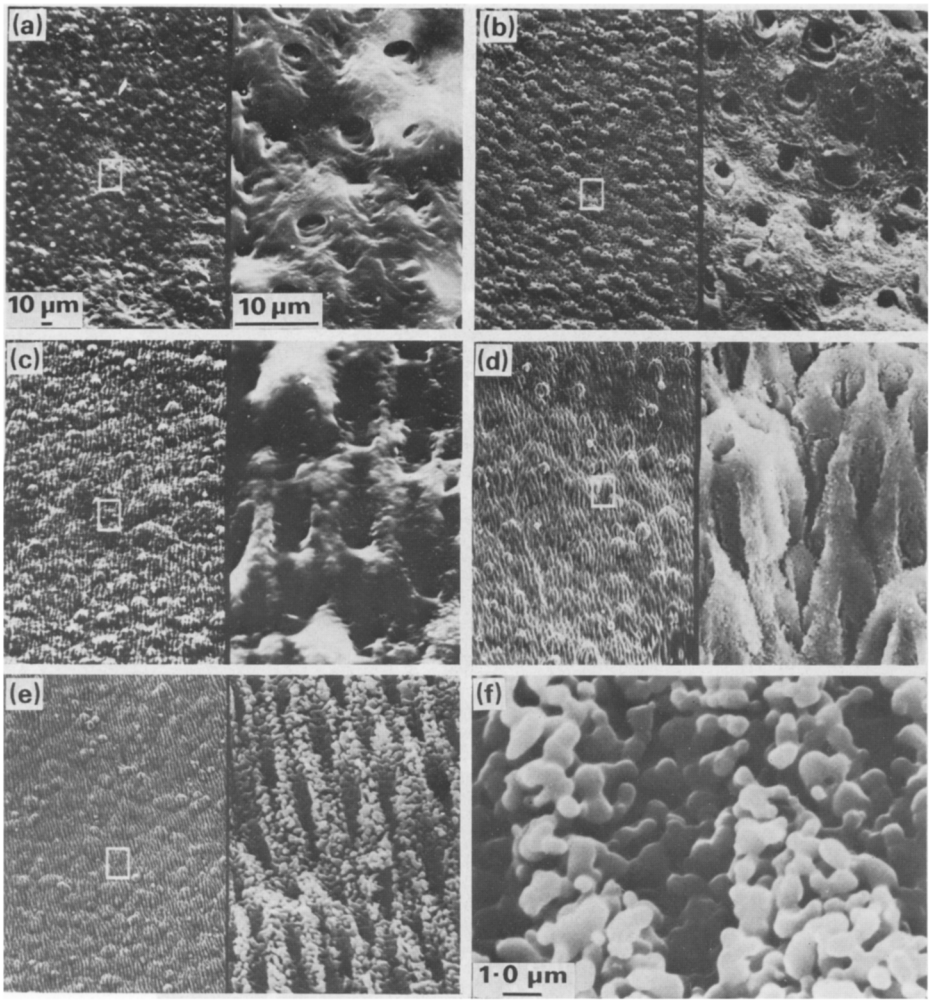

Figure 3. Microscopic morphological stages in dentin. See Table III for a description of the stages. (a) Stage I; (b) Stage II; (c) Stage III; (d) Stage IV; (e) Stage V; (f) Stage V, at higher magnification.

these tissues show increased surface roughness; in Stage III (285–<440°C), they become glassy and very smooth; in Stage IV (440–<800°C), they acquire a frothy or fleecy appearance; and in Stage V (800–940°C), these frothy protuberances coalesce into smooth-surfaced globules or nodules.

Crystal structure

The X-ray diffraction patterns of all bone samples indicate that the mineral component of the specimens remains a hydroxyapatite at all temperatures used in this study. Even so, there is a distinct difference in crystal size between the bone heated to the higher temperatures (≥645°C) and that heated to lower temperatures (≤525°C). All of the patterns from the lower range exhibit line broadening typical of small crystals,

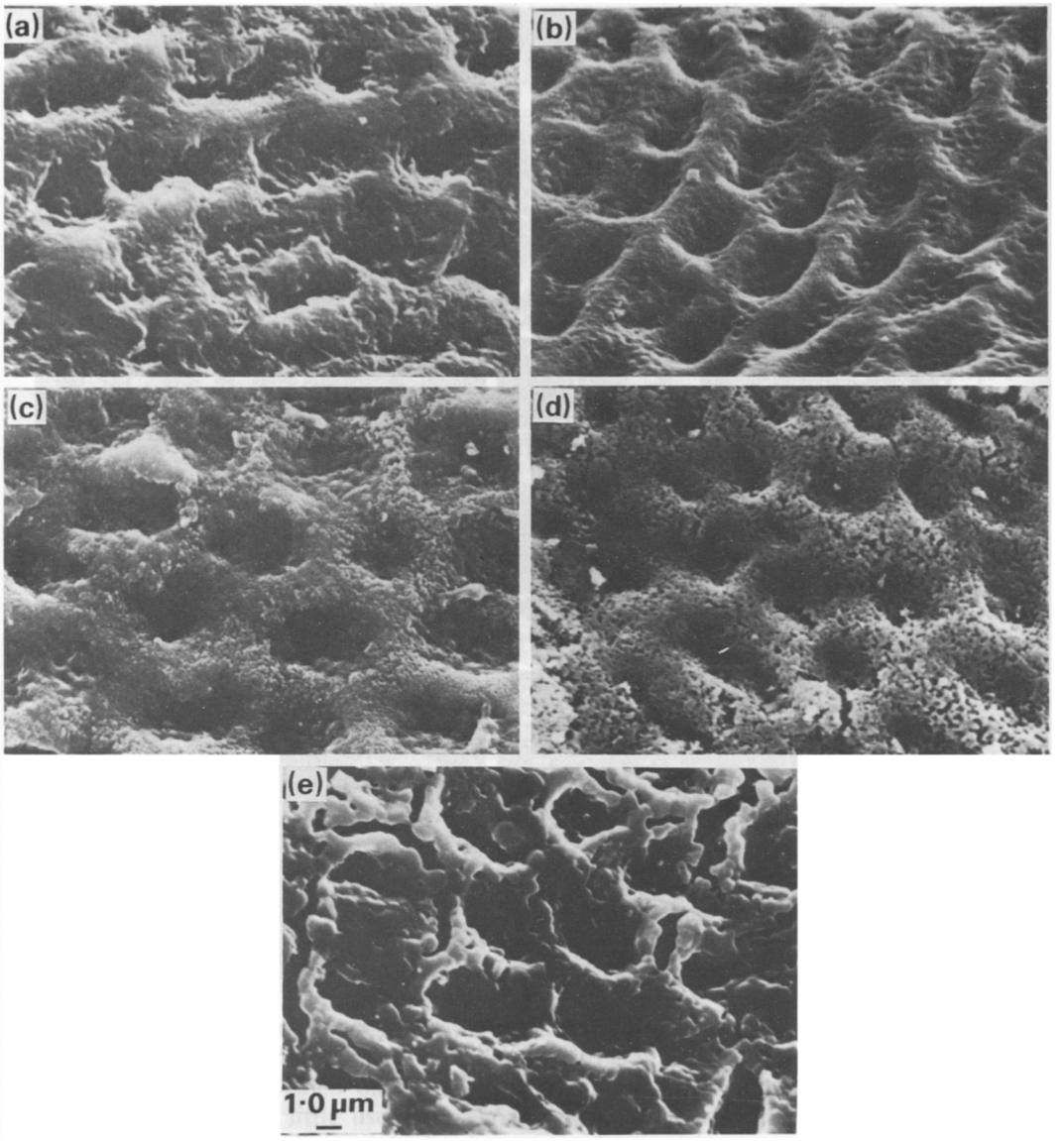

Figure 4. Microscopic morphological stages of location A enamel. See Table IV for a description of the stages. (a) Stage I; (b) Stage II; (c) Stage III; (d) Stage IV; (d) Stage V.

although there is a gradual increase in hydroxyapatite crystal size associated with increased temperature. This size increase is indicated by a sharpening and slight narrowing of peaks particularly in the 32–34° 2θ region and the 26–29° 2θ region of the X-ray diffraction patterns (Figure 7). The bone that was burned at 645°C produced an X-ray diffraction pattern markedly different from that produced by bone heated to $\leqslant$525°C. All of the peaks listed in the standard geological powder diffraction file for hydroxyapatite (card no. 9-432) are obvious, narrow, and completely separated from

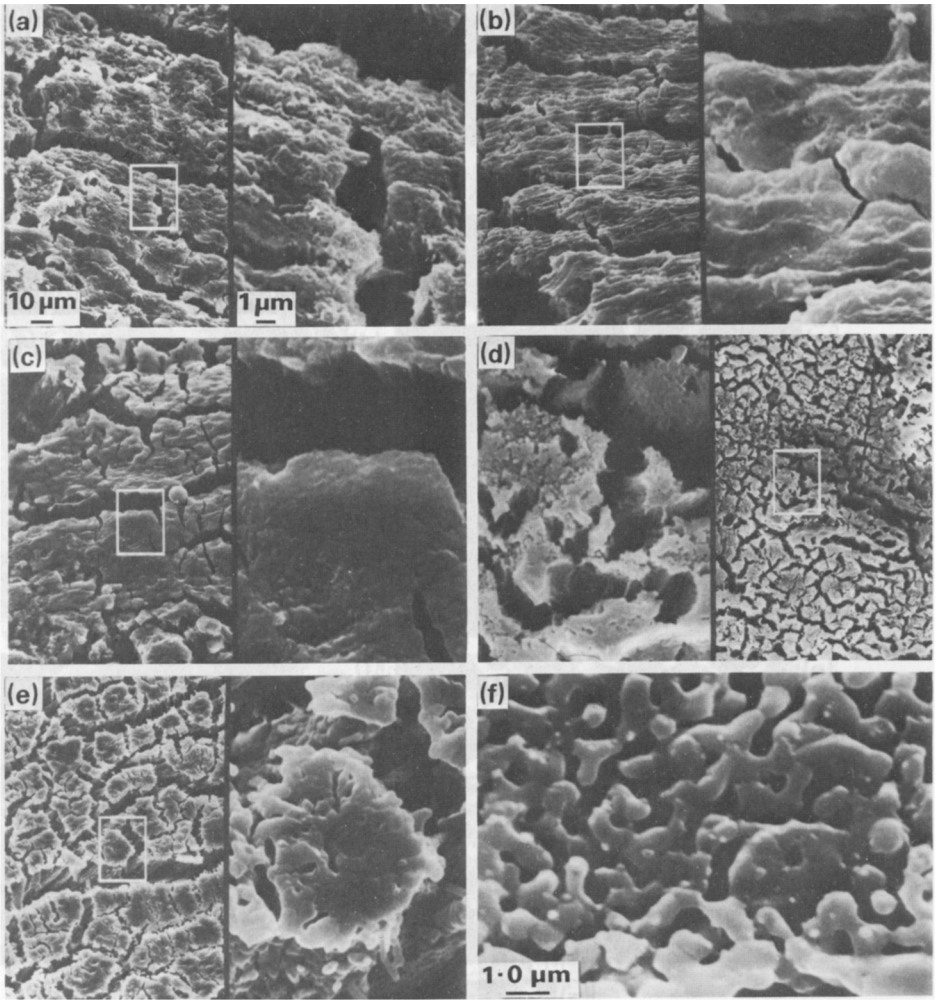

Figure 5. Microscopic morphological stages of location B enamel. See Table IV for a description of the stages. (a) Stage I; (b) Stage II; (c) Stage III; (d) Stage IV; (e) Stage V; (f) Stage V, at higher magnification.

one another in the bone burned at 645°C. Although the actual parameters of the crystal cannot be measured accurately, the pattern obviously reflects a more highly crystalline material of larger individual crystal size than existed in the bone heated to $\leqslant 525$°C. The most likely explanation is that the larger crystals have grown at the expense of the original smaller crystals. The bone heated at temperatures >645°C produced X-ray diffraction patterns that are virtually identical to the pattern from bone ashed at 645°C. Although it has been observed previously that synthetic calcium-deficient hydroxyapatite similar to bone converts to B-tricalcium phosphate when heated to temperatures above 800°C (Posner, 1969), we found no evidence of this conversion. In hydroxyapatite, but not in tricalcium phosphate, a peak with an intensity 60% of the largest peak occurs at 32·2° 2θ (powder diffraction file cards no. 9-432 and no. 18-303).

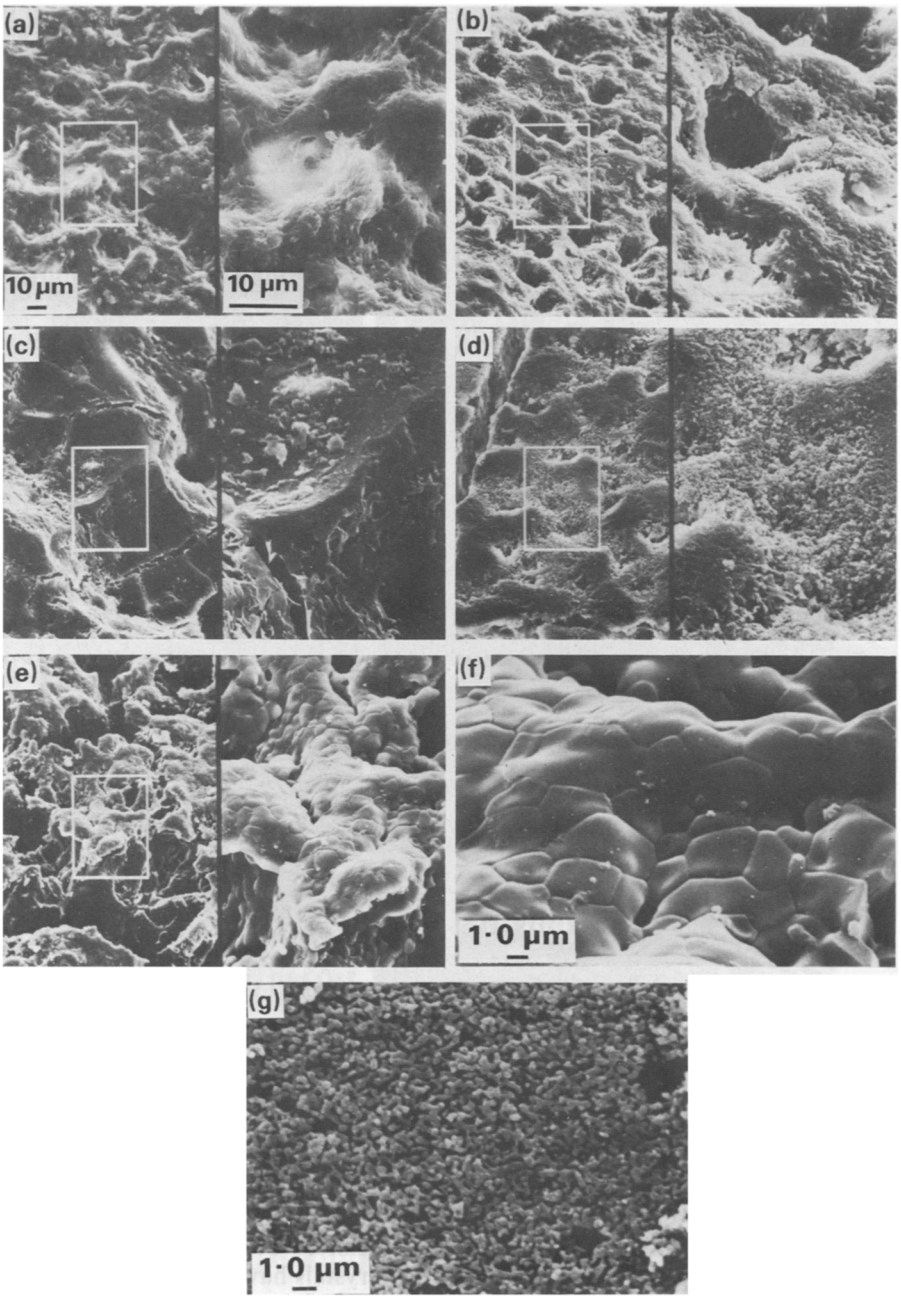

Figure 6. Microscopic morphological stages in bone. See Table V for a description of the stages. (a) Stage I, subchondral bone; (b) Stage II, subchondral bone; (c) Stage III, subchondral bone; (d) Stage IV, subchondral bone; (e) Stage V, subchondral bone; (f) Stage V, subchondral bone, at higher magnification, closely resembles artificial hydroxyapatite sintered at 1000°C (Rao *et al.*, 1974; Rootare *et al.*, 1978); (g) Stage V, cortical bone.

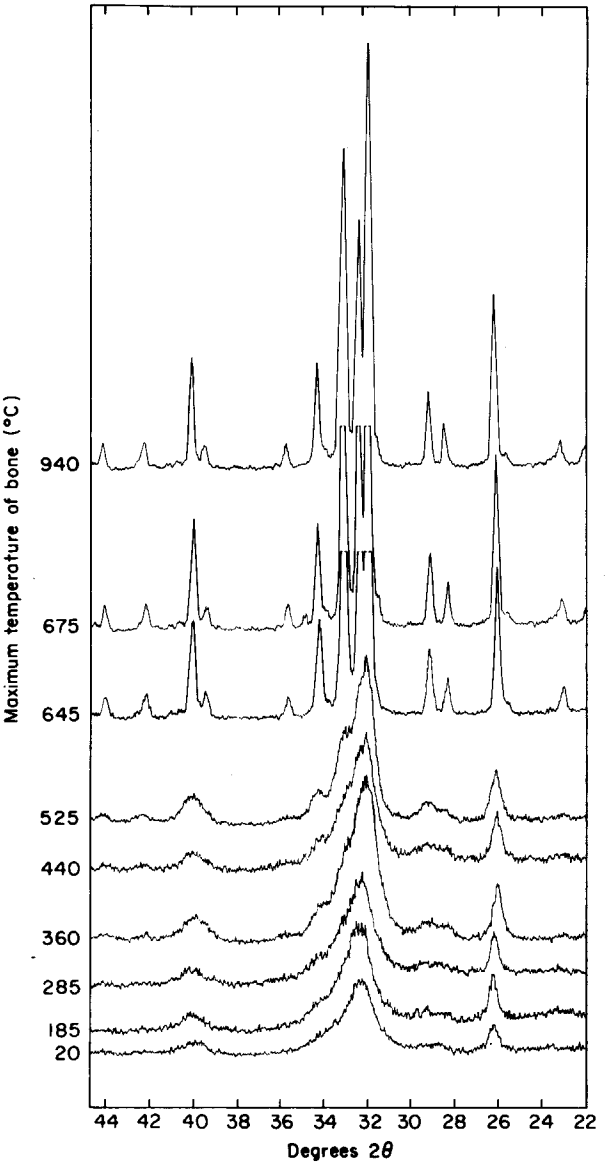

Figure 7. Powder X-ray diffraction patterns of goat astragali heated to different temperatures. The temperatures increase from the lower to higher portion of the figure. The lines produced by bone samples heated to $\leqslant 525°C$ represent hydroxyapatite of small crystal size yet even at these lower temperatures the crystal size is correlated with temperature. Crystal size increases with increasing temperature as indicated by a narrowing and increasing definition of the peaks. There is a major change in the pattern at 645°C. The peaks are sharply defined and well separated indicating that the crystals are much larger although the actual parameters cannot be measured from the pattern. Minimal change occurs with increasing temperature above 645°C. All patterns are consistent with that expected for hydroxyapatite.

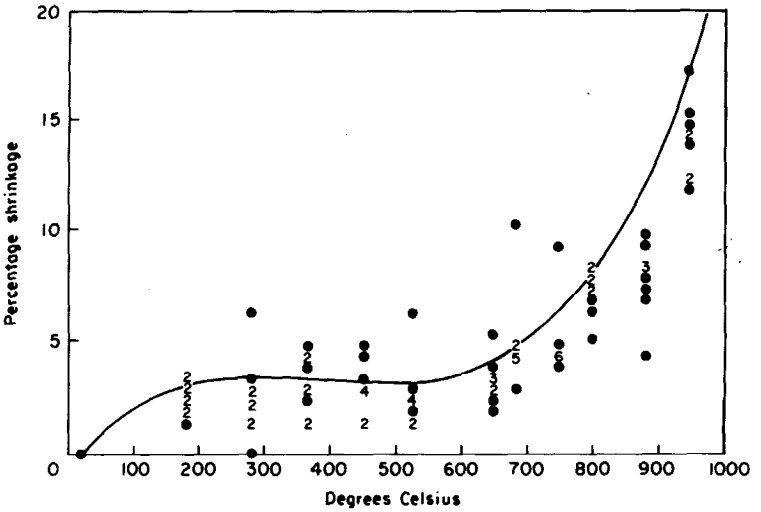

Figure 8. Mean percentage shrinkage with increased temperature, based on three measurements on astragali and one on mandibles. The polynomial expression that best fits a line to the points is $y = 0{\cdot}302x^3 + 0{\cdot}0000826x^2 + 0{\cdot}0000000704x - 0{\cdot}688$. This yields a correlation coefficient of $0{\cdot}775 < P < 0{\cdot}001$ with 106 d.f.).

Since all of our patterns show a strong peak at 32·2° 2θ, we assume that no conversion to tricalcium phosphate has occurred. In sum, the major changes we observed in these patterns occurred between 525° and 645°C. A gradual increase in crystal size occurred between room temperature and 525°C, a large increase in size occurred between 525° and 645°C, and virtually no further change occurred above 645°C.

Shrinkage

Figure 8 shows the mean percentage shrinkage for each of the measurements taken on our specimens as a function of temperature. The values shown on this graph do not represent the effects of progressive heating of a single set of specimens. Rather, the point on each line at each temperature is the mean percentage shrinkage of the several specimens heated to that temperature. It is apparent from this graph that the mean percentage shrinkage is not constant at all temperatures.

Discussion

The changes in the three components of color suggest that these are produced by alterations in the chemical composition of the specimens with heating. For judging temperature, color alone is an especially imprecise criterion both because of individual differences in the ability to perceive fine color distinctions and because burnt bones may change color if they are buried (Svenson & Wendel, 1965). In the case of bones known to have been buried, such as fossils or deliberate burials, the microscopic changes are likely to be more useful in determining temperature than are the color changes. Similarly, the color of fossils is often strongly influenced by trace elements in the sediments (Oakley, 1954) and is not a reliable indicator of pre-fossilization color.

The microscopic morphological changes appear to be the result of a reaction caused

by the progressive heating, recrystallization and possible melting of hydroxyapatite. This interpretation is strongly supported by three main points. First, we observe a striking, general similarity in the morphological changes seen in different tissues. Thus, although the different bone and tooth tissues have initially different appearances, the type of change they undergo is apparently dictated by the response of the constituent material to heating. Second, this consistent pattern of morphological changes occurs at similar temperatures in various tissues. Again, the simplest explanation for such consistency is that it is controlled by the substance (hydroxyapatite) and not by the skeletal element (mandible, tooth, astragalus) or tissue (dentin, enamel, subchondral bone, cortical bone). Finally, our conclusion is also supported by the extraordinary similarity of the appearance of subchondral bone heated to 940°C in our sample [see Figure 6(f)] to micrographs of artificial hydroxyapatite sintered at 1000°C for 30 min (Rootare *et al.*, 1978, figure 2; Rao & Boehm, 1974, figure 4). Although these researchers used artificial hydroxyapatite compressed into rods under high pressure and then heated, whereas we used natural bones and teeth, their results are similar to ours. The most logical explanation is that we are observing a physical reaction of hydroxyapatite during heating rather than anything else.

In conjunction with another study, X-ray diffraction patterns were determined for Mesolithic bones that are not believed to have been burned. These bones showed the same large hydroxyapatite crystals as the specimens heated to high temperatures in this study. Although these data show that diagenesis may produce changes in crystal structure similar to those documented here for burnt specimens, SEM-inspection of those same specimens did not reveal the morphological changes that accompany heating to high temperatures. Therefore, the X-ray diffraction patterns alone cannot be used as evidence of burning of fossil specimens.

Probably bones and teeth undergo at least some of the progressive stages of heating cited by Shepard (1956, p. 28) for minerals in ceramics: dehydration, oxidation, reduction, inversion, decomposition, and fusion. Dehydration occurs due to breakage of hydroxyl bonds in hydroxyapatite crystals, removal of water molecules adhering to the surface of the crystals, and removal of water molecules bound to the organic material (collagen and ground substance) during heating. The loss of water from all areas causes the extensive cracking, checking and warping noted by ourselves and others (e.g. Binford, 1963; Buikstra & Goldstein, 1973; Buikstra & Swegle, n.d.; Stewart, 1979; Ubelaker, 1978). Decomposition of the organic component of the specimens probably occurs between 360 and 525°C, when the variability in hue, chroma and value increases. Most or all of the organic material in the bone is burned off in this range (Ubelaker, 1978). The change we observe in crystal size at 645°C is similar to changes documented in other bone samples at about 750°C (Bonucci & Graziani, 1975; Posner, 1969). Such changes have been interpreted as indicating inversion of hydroxyapatite into tricalcium phosphate but our work suggests that larger crystals of hydroxyapatite are formed instead. This temperature is lower than the reported melting point of pure geological apatite (1200°C: Dennen, 1959), a fact that can be explained by the presence of other substances that may act as a flux for hydroxyapatite. Fusion, or melting of the hydroxyapatite crystals, apparently occurs at temperatures of 800+ °C, as shown by our SEM work.

Shrinkage

Several important points are raised by the shrinkage data presented here. First, the percentage shrinkage is a function of the temperature to which the specimens were heated. Correlation coefficients for measurements a, b and c (see Table 1 for definitions)

are all significant at the $P < 0{\cdot}02$ level (10 d.f.). This finding substantiates previous suggestions (Buikstra & Swegle, n.d.; Herrman, 1977; Van Vark, 1970) that variance in percentage shrinkage is attributable to the extent of incineration (degree of heating) of the bone. Second, the values for the different astragalus measurements are highly correlated with each other throughout the temperature range investigated (a with b, $r = 0{\cdot}975$, $P < 0{\cdot}001$; b with c, $r = 0{\cdot}960$, $P < 0{\cdot}001$; a with c, $r = 0{\cdot}970$, $P < 0{\cdot}001$; 10 d.f. in all cases). One possible explanation previously suggested by Herrman (1977) is that ultrastructure—probably the relative proportion and distribution of spongy and compact bone relative to the plane of measurement—dictates the amount of shrinkage that is possible at a given temperature. Finally, the maximum mean percentage shrinkage in this study (about 15%) is comparable in magnitude to that measured on bone fragments heated to 800°C or more by Van Vark (1970). This figure exceeds that reported by Buikstra & Swegle (n.d.) and others for smoked or calcined bones. However, maximum temperatures on Buikstra & Swegle's samples, as measured directly or inferred from the duration of heating, were usually below the maximum temperatures for our study or Van Vark's. Given that we have demonstrated that percentage shrinking covaries with temperature, these discrepancies are hardly surprising. Our suggestion is that, rather than setting a standard correction factor as did Buikstra & Swegle (n.d.), a sliding scale based on a polynomial expression generated from our data (Figure 8) or from comparable data from other skeletal elements should be used, once the temperature to which the bones or teeth have been heated has been established on other grounds.

Conclusions

These experiments have shown that different bony and dental tissues undergo similar changes in color, microscopic morphology and size at various temperatures. These changes occur despite the differences in initial structure or tissue type and are concluded to be a function of changes in the hydroxyapatite and collagen common to these tissues. One such change is the growth in crystal size of hydroxyapatite, shown by powder X-ray diffraction studies. The change in color is most likely attributable to decomposition of the organic component. The systematic documentation of the colors, microscopic morphology and X-ray diffraction patterns of bones and teeth burned at different temperatures presented here will enable researchers to determine (1) if skeletal remains have been burned, and (2) the maximum temperature to which they were heated, by matching the color, microscopic morphology, and X-ray diffraction patterns with our data. Because several color and morphological stages encompass a range of temperatures, and because diagenesis may produce changes in hydroxyapatite crystal size, we suggest that the most accurate results will be obtained by using all three lines of evidence on as many different tissues as are available. Of course, since the color of burnt bones may be altered by burial, care must be taken to apply this criterion only in appropriate cases.

Another important result of our study is that it emphasizes that the maximum temperature of the skeletal elements is the variable that is causally related to the changes we observe. To our knowledge, only Buikstra & Swegle (n.d.) have measured bone temperatures directly during experimental heating. Their data show clearly that neither fleshed nor defleshed remains reach the maximum temperature of the heating device in less than 2 h. For example, in one experiment they monitored the heat of the oak fire they were using (*c.* 900°C), the flesh of a dog carcass as it burned (580–650°C), and the bones of that animal, which smoked at 400–500°C and calcined between 400 and 650°C. When heating is of relatively short duration, bones are likely to survive

intact or mostly intact and cannot be expected to have reached the maximum temperature of the heating device.

These discrepancies between maximum bone temperature and maximum heating device temperature mean that extreme caution must be exercised in using data such as ours to distinguish between accidental fires and those made and controlled by humans or early hominids. Some of the problems in demonstrating that a particular fire was controlled by hominids have already been articulated by others (Barbetti *et al.* in Harris, 1978; Gowlett *et al.*, 1981, 1982; Isaac, 1982). Although our approach can reveal the maximum temperature reached by a skeletal element, the most cautious interpretation of such a result is that it is also the minimum temperature for the fire or heating device. Thus, to demonstrate controlled fire, the maximum bone temperature (minimum fire temperature) must exceed that documented for the most probable types of accidental fire. It seems reasonable to eliminate altogether grass or savannah fires as a cause of detectably burnt bones in most cases, since Stinson & Wright (1969) report that such fires produce surface temperatures exceeding 65°C for fewer than 10 min. Because of the higher temperatures generated by forest fires, and the longer duration of those temperatures (Wright & Bailey, 1982), greater caution must be used in interpreting data from forested areas.

Finally, it is important to note that the preparation of meat with fire by early hominids, whenever it first occurred, may not have raised the bones to a temperature sufficient to be detected using our approach (see also Laloy, 1980, cited in Le Mort, 1981). Because evidence of containers suitable for boiling or structures suitable for baking is lacking, roasting remains the most probable form of cooking in the Lower and Middle Pleistocene. However, since meat insulates bones from heating (Rosenthal & Shinagel, 1981), and since early hominids may not have cooked their meat very fully, evidence for roasting in the Lower and Middle Pleistocene may not be forthcoming regardless of the cooking practices of hominids during this time. It is more likely that the earliest detectable use of fire will be the result of hominids occasionally throwing bones back into the fire once the meat has been eaten, rather than the result of cooking *per se*.

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