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Differential Burning, Recrystallization, and Fragmentation of Archaeological Bone

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This paper presents research on the conditions under which progressive levels of burning may occur to archaeological bone, and how burning damage changes bones' crystal structure and susceptibility to fragmentation (a.k.a. friability). Experiments were conducted to simulate common patterns of high-temperature bone diagenesis and fragmentation previously documented in Paleolithic shelter sites. Bones buried up to 6 cm below the coal beds of the experimental fires were carbonized, but calcination occurred only with direct exposure to live coals. Analysis by infra-red spectroscopy reveals that marked changes in crystallinity accompany the macroscopic transformations in colour and friability of modern, fire-altered bone; specifically, a monotonic, non-linear decrease in mean fragment length across six colour categories was observed when samples were agitated or trampled, and a concordant decline in bone identifiability, first with respect to skeletal element and ultimately the recognizability of bone tissue itself. These findings help qualify the behavioural and taphonomic implications of fragmented, burned bones in archaeological sites, especially with regard to potential stratigraphic associations between artefacts and hearth features in sites and the intensity of space use by human occupants. The identification of burning damage on archaeological bone is a separate issue, however. It was found that the molecular signatures of recrystallization in modern burned bones partly overlap with recrystallization caused by weathering after only 1 to 2 years of exposure in an arid setting and by partial fossilization of archaeological bones over the long term. While infra-red and X-ray diffraction techniques effectively describe heat-induced changes in modern bone mineral and are an important aid for modelling diagenetic processes, these techniques did not reliably identify burning damage to archaeological bones. Cross-referencing readily visible colour phases with HCl-insoluble fraction data proves much more effective and economically feasible for the latter purpose.

Keywords: HEARTH FEATURES, PALEOLITHIC CAVES, BONE DIAGENESIS VIA BURNING, WEATHERING AND FOSSILIZATION, MINERAL RECRYSTALLIZATION, INFRA-RED SPECTROSCOPY, CARBON/NITROGEN RATIO.

Introduction

Today fire seems little more than a simple pleasure and convenience. Yet regular access to the benefits of fire once irreversibly changed hominid ways of life. The mastery of fire almost certainly expanded the human ecological niche, for example, providing light beyond the close of day, warmth beyond the seasons, and protection against all animals that fear it. The topic of fire in prehistory therefore has been visited by many archaeologists. Most prominent in the literature are works on how bones are altered by fire and how burning damage to archaeological bones or sediments can be reliably identified (e.g. Shipman *et al*., 1984; Bellomo & Harris, 1990; Brain, 1993; Nicholson, 1993; Sillen & Hoering, 1993), how traditional peoples build, use, and maintain fires and the ways that these ‘‘living’’ features are transformed into archaeological remains (e.g. Goodale, 1957, 1971: 169–172; Gifford, 1977; Yellen, 1977; Binford, 1978; Brain, 1981; Walters, 1988), and development of and elaborations in the use of fire as technology over the course of prehistory (e.g. Howell, 1965; Perlès, 1977; Binford & Ho, 1985; Clark & Harris, 1985; Richards, 1987: 277; Brain & Sillen, 1988; James, 1989).

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Whatever one wants to know about fire, the criteria for its recognition remain largely the same—burned objects and earth—and one commonly burned material is bone. The question of what burned bones reveal about prehistoric human behaviour is a more complex problem however, requiring both accurate identification of burning damage on archaeological materials and reliable assessments of the contexts in which such damage may occur. The latter issue concerns, for example, the spatial associations between objects in sediments and how burning and fragmentation of bone relate to the rates and intensities with which humans use domestic space.

Here, we explore the relationships between four phenomena pertinent to interpreting burning damage on bones from archaeological sites: (1) visible changes in bone colour; (2) changes in bone mineral and matrix; (3) alterations in the mechanical properties of bone that promote fragmentation; and (4) the extent to which soil insulates buried bones from fires on the ground surface. We are ultimately concerned with how burned bones become part of the fabric of archaeological records inside shelters, the spatial associations between the live fire and the material it normally damages, and the influence of fire on differential bone preservation. Our main goal is to establish some physical baselines for learning how human activities around a fireplace might be expected to translate into the patterns we see in archaeological deposits hundreds or thousands of years later.

A second, but related, group of observations from our experiments is that some signatures of bone recrystallization caused by heat overlap with those arising from weathering and fossilization. Shipman *et al.* (1984) have previously shown clear-cut relationships between bone discoloration, microscopic morphology, crystalline structure, and shrinkage due to heating in modern bone samples. A central implication of their study is that burned bones should be recognizable in the archaeological record by these same criteria. Our comparisons of archaeological and modern bone expose some perplexing and often contradictory observations regarding the identification of burning damage by infra-red spectroscopy and, probably, X-ray diffraction techniques. These contradictions offer new insights on the nature of fossilization, weathering, and heat-induced transformations of archaeological bone.

We begin this presentation by documenting some common patterns of size-mediated burning damage on bones and lithic artefacts in Paleolithic shelter sites. We then turn to the experiment results on modern bones, obtained in controlled settings, that are most pertinent to understanding the archaeological observations. The experiments address four questions in succession: how does mineral recrystallization relate to visible changes in bone colour as specimens are heated by fire? To what extent does burning alter a bone's strength, long-term durability, recognizability, and preservation potential? Under what conditions might bones buried in the sediment beneath a fire be burned by that fire? And can the effects of low-temperature diagenesis (e.g. weathering, demineralization, fossilization) on bone microstructure mimic or overlap those caused by heat? The implications of the experiment data for identifying burning damage, inferring temporal associations between hearth features and artefacts, and humans' use of domestic space in archaeological sites are discussed in the final section.

Archaeological Background: Relationships Between Burning and Artefact Size in Shelter Sites

Archaeological sites frequently contain burned artefacts, bones, and/or other materials such as ash, charcoal, or mollusc shells. We have noted recurring, simple relationships between fragment size and the frequency and intensity of burning damage in these circumstances, whereby the smallest pieces are the most likely to exhibit damage. Riparo Mochi, a rockshelter in northern Italy is a good example by virtue of the long time span it represents, lasting from roughly 38,000 to 8000 years ago (Kuhn & Stiner, 1992). Figure 1 shows median fragment lengths for burned and unburned bones from the early Aurignacian through late Epigravettian levels of this site, excavated in 1959; these faunal assemblages were retrieved by fine-screening the sediments, and all non-identifiable fragments were retained along with the identifiable pieces. Clearly, smaller bones more often show evidence of burning in this rockshelter, regardless of time period. Median fragment lengths for burned bones seldom exceed 1·2 cm (pieces significantly smaller than 0·4 cm were probably lost in the excavators' sieves).

Table 1 reveals a generally analogous situation for samples of completely recovered bone from one Epigravettian and one Middle Paleolithic (Mousterian) coastal shelter in Latium, Italy (from Stiner, 1990). The faunas from Ripara Salvini are ^{14}C dated to about 12,500 years ago (Bietti, 1984; Avellino *et al.*, 1989; Bietti & Stiner, 1992). Those from Grotta Breuil are dated to about 37,000 years ago, based on ESR technique (Schwarcz *et al.*, 1990/91; see also Bietti *et al.*, 1988, 1990/91). The mean fragment lengths and size ranges for burned bones from Riparo Salvini and Grotta Breuil are significantly smaller than those for unburned bones. Indeed, the results for the two sites are quite similar.

The relationship between the incidence of burning damage and bone fragment length holds true across Middle, early Upper, and late Upper Paleolithic cave assemblages. Hence, we can be reasonably sure that continuity in human culture does not account for the fact that burned bone fragments tend to be small. It also is interesting that, while many of the smaller bones are nearly or fully *carbonized*, calcined bones are rare in all of these cases.

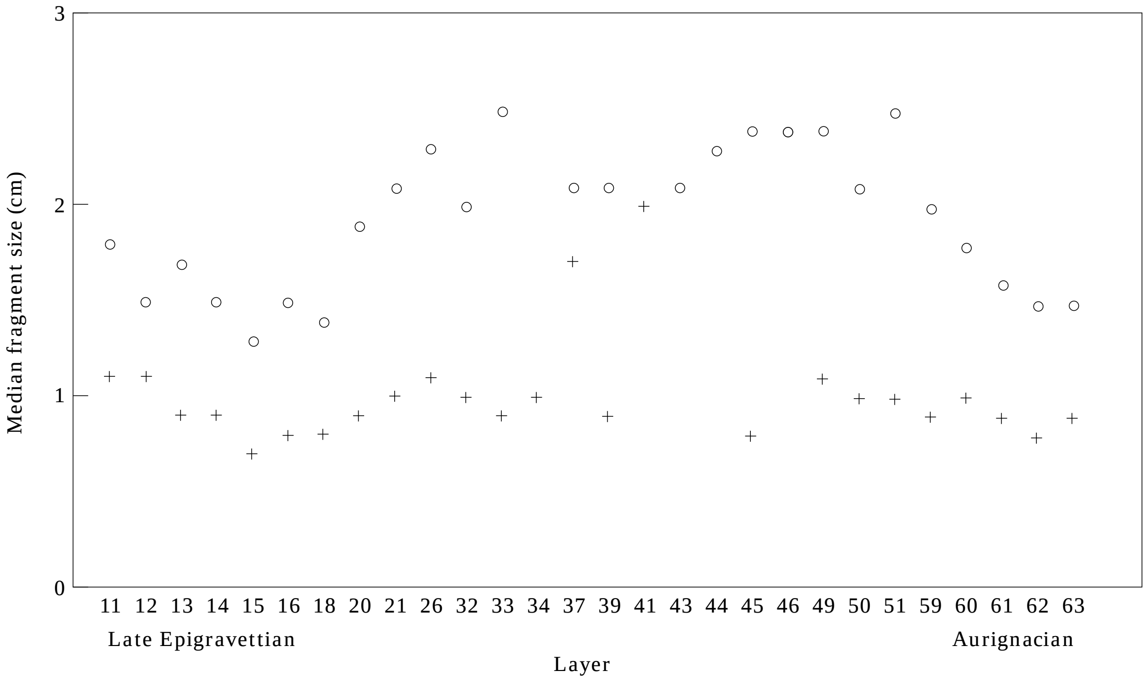

Figure 1. Median size differences for burned and unburned large mammal bone fragments from Middle Paleolithic through early Mesolithic levels of Riparo Mochi, Italy. The completely recovered material is from the east trench of the 1959 excavations; (○) unburned fragments, (+) burned fragments. The aberrant burned samples from layers 37 and 41 contain exceptionally small quantities of material.

Table 1. Fragment size (cm) data for burned and unburned bones from Grotta Breuil and Riparo Salvini, Italy

Not burned		Burned	
Riparo Salvini sample (late Upper Paleolithic/Epigravettian):			
N observations	297	*N* observations	124
Minimum length	1·0	Minimum length	0·4
Maximum length	8·3	Maximum length	5·0
Mean length	2·3	Mean length	1·4
S.D.	1·0	S.D.	0·6
Statistical significance of length differences between burned and unburned bone specimens:			
t statistic = 8·731		$P<0·001$	
Grotta Breuil sample (Middle Paleolithic):			
N observations	347	*N* observations	44
Minimum length	1·0	Minimum length	0·2
Maximum length	9·5	Maximum length	3·3
Mean length	3·0	Mean length	1·4
S.D.	1·5	S.D.	0·7
Statistical significance of length differences between burned and unburned bone specimens:			
t statistic = 6·636		$P<0·001$	

Source: Stiner (1990).
Note: Excavation procedures at these sites ensured complete recovery of bone fragments. Each sample therefore consists of identifiable and non-identifiable bones.

Because fire figures prominently in the food preparation technologies of most prehistoric and modern human cultures, it is reasonable to expect that at least some burning of bones stems directly from cooking activities. However, examination of the lithic artefacts from Grotta Breuil and three other Latium Mousterian caves shows that the overall potential of such an explanation is minimal. Table 2 compares the percentage of identifiable burned bones and lithic artefacts found in the cultural levels of the Mousterian caves (see also Kuhn, 1990, 1995; Stiner, 1994). Lithic artefacts—tools, flakes, cores, and debris—are burned as often or more often than identifiable bones. Deliberate heat-treating of stone raw materials to enhance their utility is known for many prehistoric cultures, but not in the Italian Mousterian; the burning damage noted here is insensitive to differences in raw material, and the artefacts are burned nearly to the point of destruction. The Mousterian lithic data instead are another example of the size effect: large mammal bone fragments bearing anatomically recognizable features generally tend to be larger than lithics bearing identifiable technological features. Differences in average specimen size are behind the variation in burning frequencies, not cooking *per se*.

We recently encountered patterns similar to those described above in the Paleolithic levels of Hayonim Cave in northern Israel (Belfer-Cohen & Bar-Yosef, 1981; Bar-Yosef, 1991). Hayonim contains many well preserved, shallow fireplaces, as does Kebara Cave (see Bar-Yosef *et al.*, 1986, 1992; Meignen *et al.*, 1989). Unburned bones in the deposits of Hayonim Cave usually exhibit green fractures, pointing to humans as the primary cause of breakage. The causes of fracture

Table 2. Percentages of identifiable ungulate bones and lithic artefacts burned in various cultural levels of four Mousterian cave sites, Italy

Site and provenience	Ungulate bone NISP %	Lithic artefacts Tools %	Flakes %	Cores %	Debris %
Grotta dei Moscerini:					
M2	52	42	30	38	44
M3	32	53	39	58	42
M4	7	32	18	40	41
M6	7	41	17	41	32
Grotta Guattari:					
G1	*	11	0	3	15
G2	*	5	0	11	11
G4	*	12	3	9	27
G5	*	8	12	9	27
Grotta di Sant'Agostino:					
S0	3	11	—	—	—
S1	3	10	13	14	25
S2	6	14	12	14	28
S3	1	12	8	4	16
Grotta Breuil:					
Br	2	20	14	16	24

—, No information available; *, burned bone chips are present, but relative frequencies are not known.
Sources: Kuhn, unpublished data on lithic artefacts; Stiner (1990) on bones.
Note: Percentage is calculated for bones identifiable to ungulate species or order (NISP) and does not include tiny fragments; percentage value is a fraction of the total number of items in each level and artefact category.

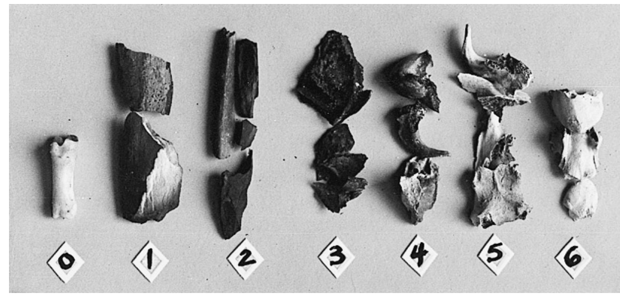

Figure 2. Modern examples of burning colour codes 0–6. Light shades on left are cream-coloured and represent fresh or lightly burned bone; light shades on right instead are pure white and represent the calcined (most advanced) phase of burning, at which point bones are most easily reduced to powder.

Table 3. Burning damage categories based on macroscopic appearance and colour

Burn colour code	Description
0	Not burned (cream/tan)
1	Slightly burned; localized and <half carbonized
2	Lightly burned; >half carbonized
3	Fully carbonized (completely black)
4	Localized <half calcined (more black than white)
5	>half calcined (more white than black)
6	Fully calcined (completely white)

on smaller, burned pieces is less clear, however. Questions about the behavioural and post-depositional circumstances in which these archaeological features and associated material formed prompted our investigations of burning phenomena through controlled experiments.

Materials and Methods

The effects of burning on bone in archaeological and experimental settings are examined here at both the macro- and microscopic levels. We deliberately burned bones to varying extents in controlled fires, and subsequently examined their susceptibility to fragmentation (a.k.a. friability) and their mineral properties by infrared spectroscopy. To learn about possible overlap in the effects of different diagenetic processes, we also compared the mineral properties of modern fresh and burned bones to those of modern weathered specimens, as well as to semi-fossilized archaeological bones of burned and unburned appearance.

The bone samples used in this study therefore originate from both modern and archaeological sources. Fresh bones for the burning experiments were collected by the authors in the northern Galilee (Israel), near Hayonim Cave, and are primarily from goats (but also one cow). The animals died the previous autumn or winter, and their remains were largely skeletonized, but still greasy, by early summer, when we collected them. The archaeological samples were obtained from Paleolithic (Natufian, Kebaran, Aurignacian & Mousterian) levels of Hayonim Cave. Pairs of archaeological bones, one of burned appearance and the other not, were selected on the basis of colour from samples of ungulate cortical splinters recovered during the 1992–3 excavations. The archaeological samples were chosen by a non-archaeologist trained only to use the burn colour codes (defined below) and darkening of the bone interior. The latter criterion is important, since burning damage on bone normally extends deep into the cortex, and distinguishes burning damage from common types of superficial mineral staining.

Observations on experimentally burned fresh bones included data on the skeletal element fragments used, visible burning intensity, specimen sizes at each step in an experiment, and the proximity of bones to the hottest part of the firebed. Visible stages of burning on bone were classified by colour on an ordinal scale from 0 to 6; these stages are illustrated in Figure 2. The visible grades of burning, described in Table 3, range from unburned (code 0) to intermediate burning stages centering on carbonization (100% carbonized or pure black = code 3) to the most advanced phase known as calcination (100% calcined or pure white = code 6). These colour criteria are generally consistent with previous experimental studies of bone burning (Shipman *et al.*, 1984; Nicholson, 1993) and with many archaeologists' informal typologies of such damage, although we have simplified the gradations in order to make the comparisons manageable.

Fire experiment set-ups

The data reported here originate from an ongoing programme of experiments linked to current excavations of Hayonim Cave, an archaeological project focusing primarily on evidence of Middle Paleolithic hominid behaviour. Four experimental fires (called 2, 3, 4 and 5) are discussed; these fires were relatively informal arrangements intended to simulate real campfires, and the set-up was similar for each experiment. The fireplace was a well-ventilated, exposed dry limestone surface, adjacent to a sheer rock wall. Some fires were built directly atop the bald rock surface, whereas later ones were built on a base of sieved cave sediments. Each fire used about 6 kg of local Mediterranean hardwoods—carob, oak, olive, or a mixture thereof. No piece of wood was greater than 7 cm in diameter, and most were smaller. The peak temperature of each fire was reached within 5–10 minutes of setting (Bellomo & Harris, 1930: 326 noted similar patterns of heat build-up), and temperatures of 900–1000°C were recorded with a thermocouple. Each setting was allowed to burn down naturally, with minimal banking of the coals. Fires were lit in the morning, and if not dead by early evening, coals were collected in a large can and allowed to smoulder through the night.

Premeasured fresh goat bones were poured directly into the control fires (Fires 2 and 3) and/or were buried in 1 to 15 cm of soil beneath the firebed (Fires 4 and 5); a total of 30 litres of fine dry-sifted sediment, taken from the backdirt of the Hayonim excavations, served as the matrix in which bones were buried in the later experiments. The bone material was retrieved once each fire cooled by gently sieving the mass through a 0·1 cm mesh screen, avoiding further breakage. Bones were remeasured and classified according to the intensity of visible burning damage with each new step of an experiment; the recognizability of anatomical elements was also recorded.

To see how burning damage affects bone friability under mild pressure, we induced fragmentation by vigorously shaking the burned bones, sorted by colour category, in a cardboard box for 60 s, or by having two adult men trample buried bones *in situ* for several minutes. Fragmentation is measured in terms of median fragment length and the volume (ml) of powder generated for each burn colour category.

Infra-red spectra of bone

The microscopic structure of bones was checked throughout the experiments by infra-red spectroscopy and HCl organic extraction. The surfaces of archaeological bones were first cleaned mechanically with a scalpel. Representative fractions from each bone were collected and ground with an agate mortar and pestle. A few tens of µg were then mixed with KBr, and a 7-mm diameter pellet was prepared using a Qwik Handipress (Spectratech, Warrington, U.K.). The Fourier transform infra-red spectrum was obtained using a Midac Corporation (Costa Mesa, U.S.A.) spectrometer operated by Spectracalc software (Galactic Industries Corp., Salem, New Hampshire, U.S.A.). Many of the analyses were performed on-site at Hayonim Cave in Israel (see Weiner *et al*., 1993).

An expanded spectrum in the 425–900 cm^{-1} range was used for measuring the mineral crystallinity based on the so-called *splitting factor* (or crystallinity index), following Weiner & Bar-Yosef (1990). The ratios of the absorption band of carbonate at 874 cm^{-1} to that of 565 cm^{-1} for phosphate were used to semi-quantitatively estimate relative carbonate contents of the mineral phase. This follows Featherstone *et al*. (1984), who, by using synthetic standards, showed that the ratio of the carbonate 1415 cm^{-1} absorption band to the same phosphate band estimates ($\pm$ 10%) carbonate content. We used the 874 cm^{-1} carbonate absorption rather than the 1415 cm^{-1} absorption, because the latter was affected by the presence of organic matrix absorption bands in the relatively well-preserved bones.

The *insoluble organic matrix* fraction was extracted by dissolving 100–200 mg of powdered bone in 4 ml of 1 N HCl at room temperature. The insoluble fraction was separated from the supernatant by centrifugation (14,000 ***g*** for 2 min), and then washed twice in the same way with deionized water. The dried pellet was used to obtain an infra-red spectrum, as described above, and a carbon/nitrogen (C/N) ratio using a Carlo-Erba (Milan, Italy) model 1108 elemental analyser.

Mineral Recrystallization as a Function of Visible Burning on Modern Bones

Fresh bone normally comprises 60–70% by weight of dahllite (carbonated apatite) crystals. At ambient temperatures, the crystals are gradually altered by diagenesis, wherein large crystals tend to grow at the expense of small ones. This process may occur over millennia, as one of the changes that normally occurs in fossilization, or through the more rapid transformations of weathering over a few months or years. By contrast, high temperature diagenesis is nearly instantaneous. At temperatures below about 650°C, the changes that occur in the crystals of heated bone are probably similar to those occurring at low temperatures, but above this temperature, a solid state recrystallization occurs (Shipman *et al*., 1984).

Part of the information provided by infra-red spectra of bone relates to the degree of crystallinity of the dahllite crystals (see also Shipman *et al*. (1984) on the use of X-ray diffraction for a similar purpose). This is a function of the extent of splitting of the two

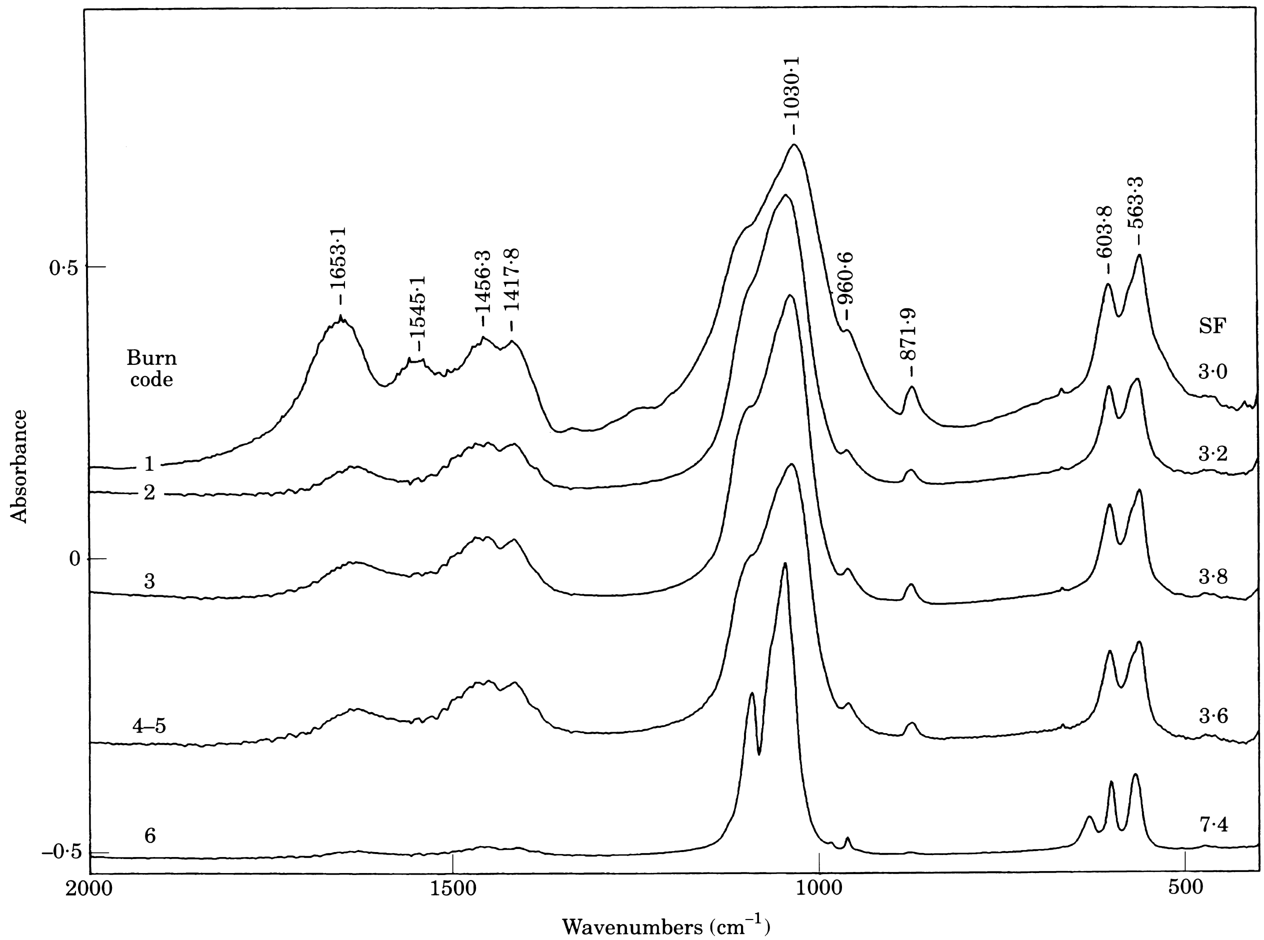

Figure 3. Infra-red spectra of modern bones burned in the fire experiments. Burn colour codes are shown on the left, and splitting factors (SF) on the right. Note that the collagen peak (1653 cm^{-1}) that is still prominent in the burn code 1 sample is greatly reduced in all of the others, and that the recrystallized sample (burn code 6) contains almost no carbonate, as evidenced by the absence of the carbonate peaks at 1456 cm^{-1}, 1417 cm^{-1} and 872 cm^{-1} on the wave-number axis.

absorptions at 603 and 565 cm^{-1} (Termine & Posner, 1966) and reflects a combination of the relative sizes of the crystals and the extent to which the atoms in the lattice are ordered. As recrystallization proceeds, these two absorption peaks become increasingly separated from one another. The extent of this process is therefore measured by the splitting factor (SF), an average of the heights of the two peaks (baseline drawn between 495–750^{-1}) divided by the height of the low point between them (Weiner & Bar-Yosef, 1990: 189–190). The higher the SF value, the larger and/or more ordered are the crystals: hence, this crystallinity index is always lowest for fresh bone (e.g. SF = 2·5–2·9) and highest for calcined (or highly fossilized) bone (e.g. SF $\simeq$ 7·0). By way of example, Figure 3 shows spectra for modern bones burned to colour codes 1–6; the progressive splitting at around 603 and 565 cm^{-1} can be seen at the right end of the wave-number axis.

Figure 4 traces the changes in splitting factor (SF) and carbonate (CO_3) values, the latter of which is also obtained from the infra-red spectra, as a function of visible changes in burn colour on bones from the control fires. The SF values (Figure 4(a)) increase, and the carbonate values (Figure 4(b)) decrease, as burning damage intensifies. The differences in SF and CO_3 content for bone colour values 1, 2 and 3 are minimal, however, when contrasted with the changes associated with more advanced burn colour codes 4, 5 and 6, representing partial and complete calcination. It therefore is easy to identify the calcined phase in bones by infra-red spectroscopy, because the original dahllite lattice recrystallizes, and in so doing loses carbonate to form hydroxyapatite (in one case, the bone recrystallized to (presumably) β-tricalcium phosphate).

Analyses of the 1 N HCl-insoluble matrix fractions of the modern goat and cow bones from the experimental fires (Table 4; see Appendix for more information) show that collagen is still preserved in bones with a burn colour value of 1. This is apparent from the infra-red pattern itself (see DeNiro & Weiner, 1988*a*) and from the C/N ratio around 3·0 (for a related C/N study, see Brain & Sillen, 1988). Bones

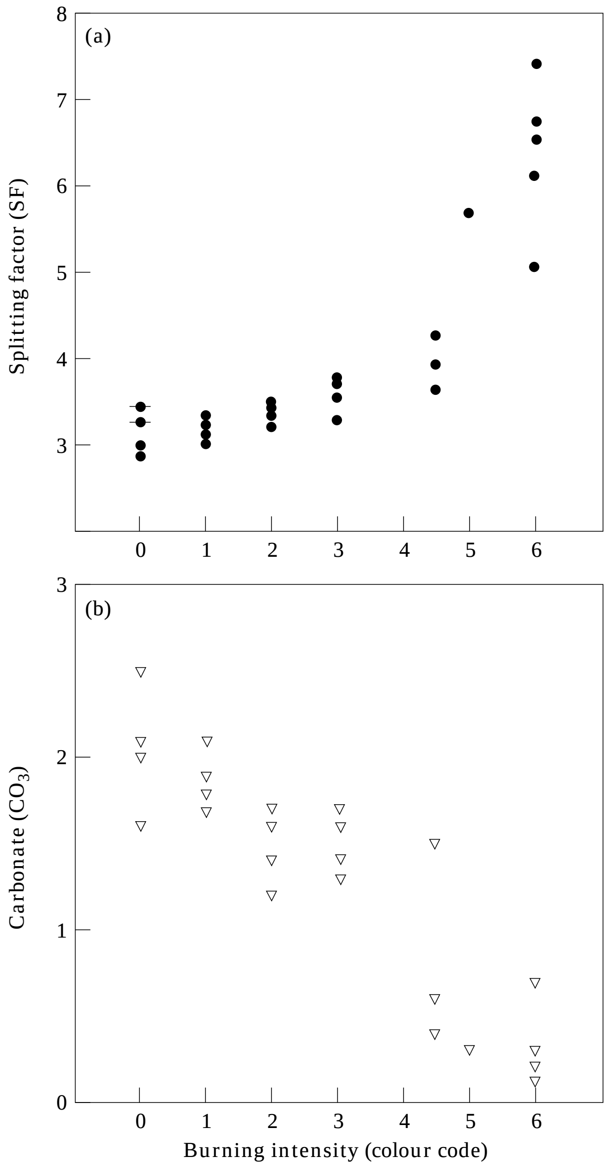

Figure 4. Splitting factor (a) and carbonate (b) values for experimental bone by burn colour code. ●, SF; ▽, CO_3; —●—, SF for slightly weathered exteriors of otherwise fresh bone specimens (see text for discussion of this phenomenon).

with burn colour values 2, 3 and 4–5 all produce insoluble fractions with infra-red spectra characteristic of pyrolyzed material. However, fully calcined bone (colour code 6) has no insoluble matrix; the HCl solution is clear, presumably because all of the internal matrix had been destroyed by heat.

Bone Fragmentation as a Function of Burning Intensity

Living bone tissue is very strong and is adapted to withstand a variety of stresses (e.g. Currey, 1984). This property of fresh bone is lost to some extent in

Table 4. Summary of 1N HCl-insoluble fraction results by burn colour code for bones from experiment fires

Burn colour code	Infra-red spectrum reading (and observable colour)	C/N ratio
Fresh bone (0)	Collagen (cream)	3·03
1	Collagen (yellow)	3·00
2	Pyrolyzed matrix (partly black)	n.d.
3	Pyrolyzed matrix (completely black)	4·36
4–5	Pyrolyzed matrix (black and white)	4·20
6	No insoluble fraction	—

n.d., Not determined; —, no insoluble matrix.
Note: See Appendix for complete information on the mineral properties of the bone samples, replicated in four sample series.

diagenesis. Hence, significant rearrangements in the crystal lattice of bone caused by burning should greatly affect its potential resistance to pressure. While we can expect that burned bone is more likely to crumble than fresh bone as a rule, we presently know little of the character of the damage progression.

Bone samples representing the full range of burn phases were generated in the experimental fires. The susceptibility of the bones to fragmentation in relation to burning intensity was observed in three ways: (1) as a direct product of heat alone, without added pressure (see also Nicholson, 1993); (2) vigorous agitation of fragments sorted by burn colour inside a box for 60 s; and, finally, (3) trampling premeasured whole bones buried beneath a cooled firebed. Agitating burned specimens in a box permits complete recovery of all fractions including fine powder, whereas the trampling experiments better typify human contexts, specifically the effects of foot traffic on shallowly buried, burned bones.

Figure 5 is a composite of the bone fragmentation results from the experiments, illustrating the overall form of the damage curve. Pressure or agitation quickly reduces the material to smaller sizes, and eventually powder (Table 5). These data reveal a monotonic, non-linear decrease in median fragment length for the agitated/trampled bones as burning intensity advances from code 0 to code 5–6. There is a concordant decline in bone identifiability, initially with respect to skeletal element and ultimately to bone tissue itself.

Table 5 shows that agitation of burned bones in code class 3 resulted in approximately 6% fine powder. The powder fraction builds up appreciably after the bones reach code 4; the same amount of agitation produced 10% and 11% powder fractions for burned bones in code classes 4, 5 and 6, respectively. In the most extreme conditions, wherein ungulate bones are fully calcined, we might expect them to be nearly or completely reduced to a powder by soil compaction and trampling. Similarly, unburned bones buried under hearths were hardly affected by trampling, while burned ones were extensively fragmented. The contrast

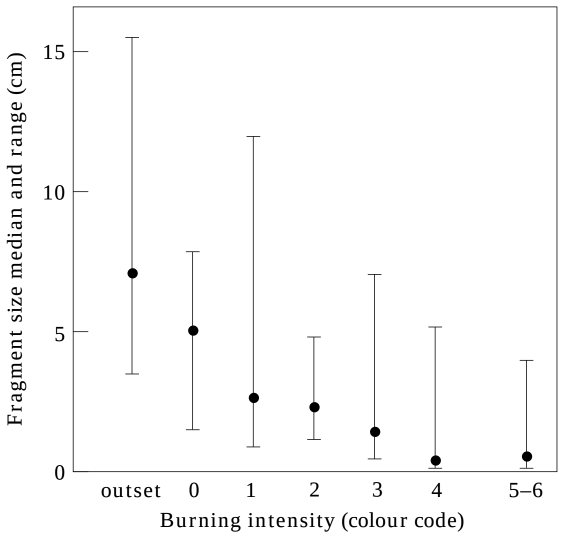

Figure 5. Bone fragment size medians and ranges by burn code before and after pressure-induced fragmentation. (outset) represents fresh bones selected for the experiments. The data represent a composite, though compatible, set of results from Fires 2 and 5.

between burned and unburned bones with regard to brittleness is clear from visual inspection of the material and from the fragment size and volume data.

The rates at which these diagenetic changes occur are also of interest from the archaeological perspective. Because the effects of heat are nearly instantaneous, the transformations and increased susceptibility to fragmentation caused by burning may take place within the same time frame as human activities at a site. This is less true of weathering, and not at all true of fossilization.

Burning of Bones Buried Beneath an Open Campfire

The next question we set out to investigate was whether bones buried in the sediment upon which a fire is built can be burned by that fire and, if so, to what extent. Having long recognized that fires heat the earth immediately beneath them, archaeologists routinely check for reddened (oxidized iron) soils as one means for verifying the existence of hearth areas in sites. Bellomo & Harris (1990: 323) further note that surface fires permanently affect the magnetic properties of iron-rich sands and clays as heat penetrates the sediments, especially within the first 5 cm below the heat source.

In two of our experiments, bones were placed at various depths in sieved dry sediment, and fires built on top. The bones were later retrieved from the firebeds by re-excavating and sieving the sediment. Table 6 shows that, although bones were buried as deep as 15 cm below the coal bed, only those specimens in the first 5 cm were affected much by heat from the fire. Moreover, these shallowly buried bones were burned only to the point of carbonization (burn code 3). This strong contrast between buried and unburied material is clear in Figure 6. Goat ribs buried at 5 cm below the firebed are visibly altered, whereas bones buried 10 cm deep were largely unmarred by heat. Interestingly, the heads of curved goat ribs, which protruded upward through the soil, were locally burned in a way that resembles roasting damage observed in ethnographic contexts (e.g. Gifford, 1977; Yellen, 1977; Gifford *et al.*, 1980).

The buried bones underwent corresponding alterations in colour and mineralogical characteristics, as

Table 5. Bone fragment size as a function of burn code before and after agitation or trampling, organized by burn colour codes

Agitation or trampling?	Burn colour code	*N*obs	% power volume	Median size (cm)	Size range (cm)
Bones buried 1 to 4 cm below firebed, trampled half only (Fire 5):					
(outset)	Fresh (0)	14	—	7·0	3·5–15·5
Trampled	0	6	—	5·0*	1·6–7·8
Trampled	1	11	—	2·7	0·9–12·0
Trampled	2	27	—	2·3	1·2–4·8
Trampled	3	87	—	1·5	0·5–7·0
Bones placed partially in fire, later agitated in box (Fire 3):					
(outset)	Selected† (3)	80	—	2·0	0·4–6·4
Agitated	3	741	6	0·4	0·2–5·8
Bones placed directly in fire‡, later agitated in box (Fire 2):					
(outset)	Fresh (0)	74	—	3·5	1·2–9·2
No agitation	3	6	—	2·9*	2·6–3·4
No agitation	4	32	—	3·1	0·4–5·6
No agitation	5	55	—	2·2	0·4–7·0
No agitation	6	41	—	1·4	0·4–7·0
Agitated	4	228	10	0·4	0·2–5·1
Agitated	5–6	536	11	0·6	0·2–4·0

*Note that this sample is exceptionally small.
†Burning to colour code 3 was produced deliberately by placing the bones partly in or atop the fire; a range of carbonized fragments were selected from the burned material, measured, and then agitated in a box.
‡Bones were placed in the centre of the fire; this produced advanced burning damage only.

*Table 6. Numbers of bones (*Nobs*) in each burn category by depth below the firebeds in Fires 2, 4, and 5*

Depth buried in sediment	Burn colour code 0	1	2	3	4	5	6
0 cm, in firebed *N*obs = 134	—	—	2	4	32	55	41
1–4 cm in soil *N*obs = 142	5	11	34	92	—	—	—
5 cm in soil *N*obs = 17	2	2	4	9	—	—	—
10 cm in soil *N*obs = 8	5	2	1	—	—	—	—
15 cm in soil *N*obs = 4	4	—	—	—	—	—	—

Note: The samples for bones buried 5–15 cm below experiment firebeds are small, primarily because large whole goat bones were used. The data none the less make the point that burning damage is minimal to bones lying below 5 cm in the firebed.

Figure 6. Differential burning on bones buried at 5 cm and 10 cm below the coal bed in Fire 4.

shown in Table 7. The SF and carbonate values for buried bones burned to colour codes 1–3 are analogous to values for similarly burned bones that had been directly exposed to fires (see Figure 4). It is significant that we were unable to induce calcination on bones buried by any amount of soil, despite the fact that our control fires were comparatively hot (minimally 900°C; cf. Shipman *et al*., 1984).

Overlapping Signatures of Burning, Weathering, and Fossilization

The above experiments concern only the signatures of burning damage in a modern setting, where splitting factor and carbonate data are useful for tracking changes in the microstructure of bones subjected to heat. While appropriate for experimental studies on modern bones, and for developing and refining diagenetic models more generally, we wondered whether processes of weathering and fossilization might partly mask patterns of bone recrystallization caused by fire.

Bone weathering normally results from exposure to wind, sun, and/or freeze–thaw cycles (see Behrensmeyer, 1978). The smooth cortex of fresh bone soon degenerates under these conditions, cracking, splitting, and eventually flaking away from the outside inward. As far as we know, weathering occurs only to bones that lie exposed on the ground surface, but the rates at which weathering damage progresses vary enormously with local conditions. The bones used in our burning experiments were from animals that perished within the previous year, and, though defleshed, all of the bones had a ''fresh'' appearance. We nonetheless encountered some small-scale variation in relative dryness and microstructure between the exterior and interior surfaces of the same specimens (goat bones in Table 8). In Figure 4(a), the exterior surfaces of the control bones show SF values significantly higher than the interior portions of the same bones (see also Appendix). This variation clearly represents subtle weathering.

Having noted this discrepancy in the control specimens, we expanded our comparisons to include a variety of conspicuously weathered bones from two arid environments: dog bones exposed for approximately 2 years in Israel, and cow bones exposed in a controlled setting for exactly 9 years in New Mexico (U.S.A.). Taken together, our samples span the lightest and most severe degrees of weathering; the cow bones exposed to the sun for 9 years (semi-protected by vegetation) were severely deteriorated.

Table 8 compares surface and interior SFs for the weathered bones. The interior SF value is lower in every case, generally equivalent to values for genuinely fresh bone (see also Weiner & Bar-Yosef, 1990). The SFs for the outer cortex of all samples range between 3·15 and 3·43, regardless of exposure time, and they

Table 7. Mineralogical and colour properties of bones burned while buried in sediment beneath Fire 5

SF	CO_3 (874/565)	Burn colour code	Visible colour
3·01	0·15	1	Yellow/brown
3·12	0·15	1	Yellow/brown
3·09	0·19	2	Yellow/grey
3·31	0·19	2	Brown/grey
3·26	0·17	3	Black
3·18	0·19	3	Black
3·50	0·13	3	Black

Note: The maximum degree of burning that could be achieved on buried bones was carbonization (burn colour code 3).

Table 8. Splitting factor values of the surfaces and interior portions of modern weathered bones

Sample description	Exposure time	SF	
		Surface	Interior
Goat bone, Israel*	<1 year	3·26	2·88
Goat bone, Israel*		3·43	2·98
Dog calcaneum, Israel†	~2 years	3·32	3·08
Dog phalanx, Israel†		3·36	3·11
Cow phalanx, New Mexico‡	9 years	3·15	2·90
Cow femur, New Mexico‡		3·27	2·95

*Specimens were skeletonized but somewhat greasy when collected for analysis. The bones had been exposed to full sun and were from individuals that perished the previous winter in the northern Galilee, Israel.

†The two dog bones are from one individual, collected from parkland outside Jerusalem, Israel. Estimated time of exposure is 2 years, but is not absolutely certain.

‡Samples are from a long-term control study by Stiner, conducted in Albuquerque, New Mexico, U.S.A. The time of exposure is certain, and both specimens were semi-protected by vegetation throughout the 9 years of exposure. The degree of deterioration by weathering varies among the 9-year-old samples with the amount of plant cover, only two of which are analysed here, but all may be described as severely damaged.

indicate some reduction of collagen content (e.g. dog calcaneum in Figure 7). The SF data suggest that some microscopic transformations caused by weathering occur rapidly, apparently within the first year or two of exposure, and then stabilize. These microscopic changes therefore *do not* correspond directly to the visible degrading of bone that zooarchaeologists and paleontologists normally associate with weathering damage. The changes in modern bone crystallinity caused by weathering also partly overlap with those caused by heat between burn colour codes 0 and 3 (i.e. up to complete carbonization).

The crystal lattice of bones may also change if bones are buried in sediments for long periods of time. Indeed, some degree of alteration is to be expected in any archaeological or paleontological deposit (e.g. Weiner & Bar-Yosef, 1990). Thirteen pairs of apparently burned and unburned bones, found together in Natufian or Paleolithic strata of Hayonim Cave, were analysed for splitting factor, carbonate content, and HCl-insoluble fraction. The two specimens in each archaeological pair presumably were subjected to roughly the same diagenetic history, because they were

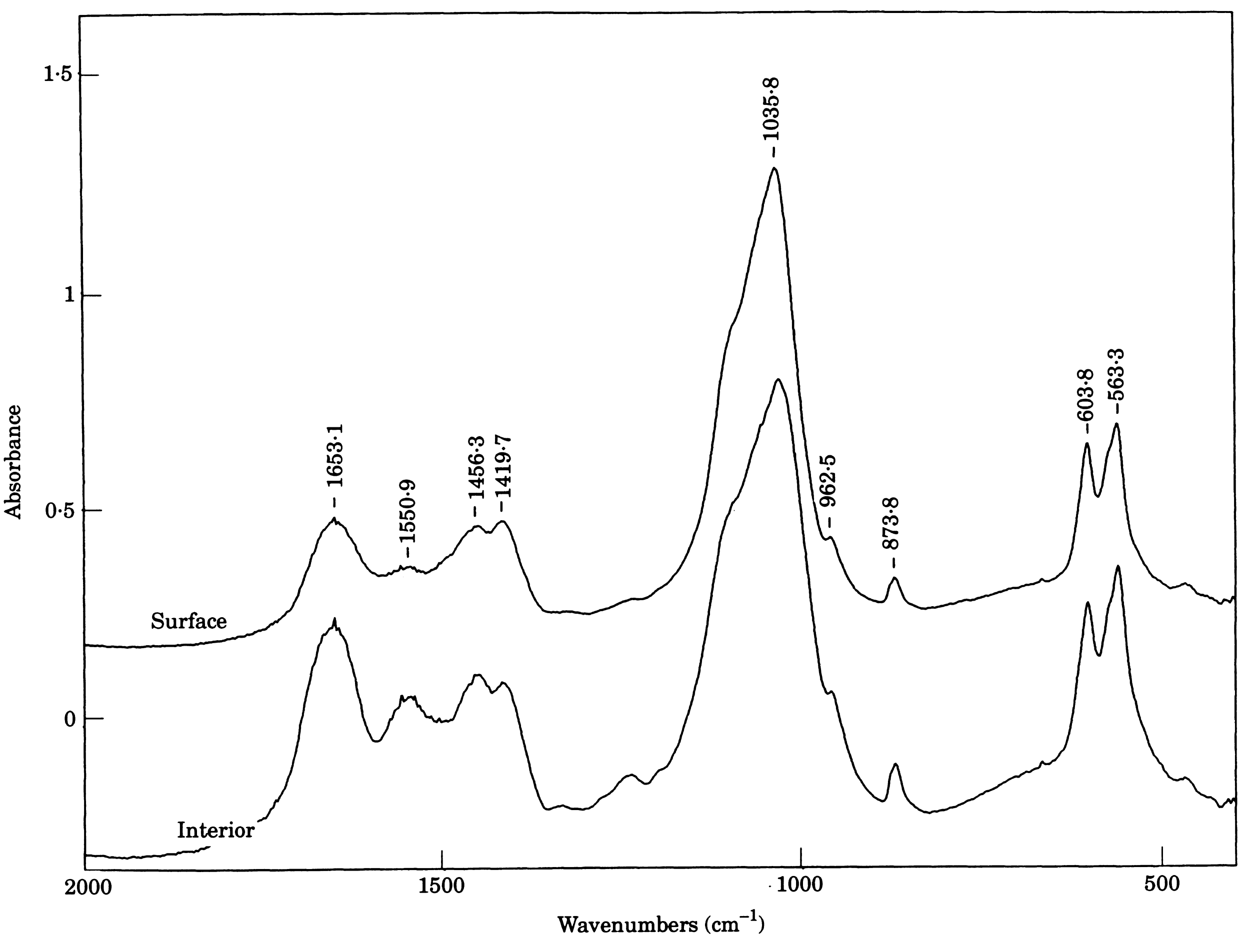

Figure 7. Infra-red spectra of the surface and interior of a modern weathered dog calcaneum, collected in Israel after being exposed for approximately 2 years. Note the reduction in collagen (peak 1653 cm^{-1}) in the surface of the specimen as compared to the interior.

Table 9. Splitting factors (SF), carbonate ratios (CO_3), and 1N HCl insoluble fractions for burned and unburned archaeological bone pairs from Hayonim Cave, Israel*

Square	Cultural affiliation	"Unburned" SF	"Unburned" CO_3	"Burned" SF	"Burned" CO_3	Insoluble fraction, burned only	C/N ratio, burned only
F27d	K	3·62	0·18	3·45	0·16	0·8	4·11
J25b	N	3·35	0·17	3·07†	0·20	absent	—
C27a	K	4·00	0·12	3·66	0·15	2·1	4·65
J25d	N	3·19	0·16	3·72	0·15	0·6	n.d.
K26a	N	3·31	0·17	3·26†	0·14	absent	—
E28b	K	3·23	0·18	3·33	0·16	2·0	4·39
H18c	M	3·21	0·18	3·78	0·13	0·3	n.d.
M25c	N	3·21	0·19	3·39	0·18	4·8	4·98
K18d	A/M	3·18	0·19	3·44	0·18	1·0	7·90
K26b	N	3·31	0·18	3·82†	0·12	trace	n.d.
D26d	K	3·50	0·18	3·39	0·20	1·0	4·84
C27b	K	3·26	0·19	3·39	0·18	2·3	4·54
G27b	M	3·35	0·18	3·30	0·21	2·1	5·25

n.d., Not determined; N, Natufian cultural affiliation; K, Kebaran; A, Aurignacian; M, Mousterian.
*CO_3 (874/565).
†Only trace amounts of 1N HCl insoluble fraction.
Note: All archaeological bone samples are cortical specimens from ungulates, mostly limb bone fragments. Quotation marks indicate that the samples were classified as burned or unburned strictly on the basis of specimen colour. Pairs of fragments were obtained from the same Paleolithic levels, subsquares, and 5 cm cuts to control for variation in preservation conditions within the cave deposits.

found within the same $50 \times 50 \times 5$ cm units. The samples were selected conservatively on the basis of colour and with the knowledge that mineral and organic stains may be mistaken for burning.

Surprisingly, the infra-red data reveal no significant differences in SF or carbonate content among the apparently burned and unburned archaeological samples (Table 9). The results are remarkably uniform; all specimens have SF values between 3·0 and 4·0, apparently the usual condition for bones in Hayonim Cave. The infra-red spectra—at least with respect to SF and carbonate content—did not differentiate between the mineral phase of apparently burned and unburned bones, although the same infra-red data keyed to colour codes readily distinguish the two states in modern bones. Moreover, we found no correspondence between infra-red readings and the age of the cultural material in our sample (Natufian through Mousterian), in general agreement with Sillen's (1981) findings on calcium/phosphate ratios for Natufian and Aurignacian samples from Hayonim Cave.

It is conceivable, particularly in light of our observations about weathering, that diagenetic processes can achieve the same effects in unburned bones buried for thousands of years that burning achieved instantly in the experimental fires. If this is the correct explanation, then another question arises. The apparently unburned bones from these strata all have rather low SF values, ranging between 3 and 4. Why has the crystallinity of the burned bones not continued to increase with time and, in so doing, maintained the expected difference in SF values between bones of burned and unburned appearances?

One possible explanation for the similarities in SF and carbonate values is that the darkened archaeological bones were not actually burned. To address this question, the 1N HCl-insoluble organic matrix fractions were extracted from the darkened bone of each pair and their C/N ratios and infra-red spectra analysed (following DeNiro, 1985; DeNiro & Weiner, 1988*a*,*b*,*c*). Collagen, an important component of fresh bone, is burnable. Well-preserved bones in geological sediments may retain fair amounts of collagen or its combustible products, most of which show up in the HCl-insoluble fraction (Master, 1987). Table 9 shows the C/N ratios of the 1N HCl-insoluble fractions. The C/N values are for the "burned" members of the archaeological bone pairs from Hayonim Cave only; no insoluble organic phase generally is recoverable for unburned bones. All of the C/N values are above the range that is characteristic of collagen (i.e. 2·9 to 3·6, De Niro, 1985). Table 9 also shows that all of the 1N HCl-insoluble fractions of the so-called burned bones from Hayonim Cave have essentially the same infra-red spectra, and that this group is quite different from those for unburned bone tissues in different diagenetic states (Figure 8).

At least 11 of the archaeological specimens classified as burned on the basis of colour were in fact burned. The colour criterion, so readily discernible by the naked eye, worked quite well at Hayonim Cave. The HCl-insoluble procedure could well be used as an additional criterion for positively identifying burned bones in archaeological deposits. While visually-based assessments of burning appear to have been correct most of the time (11 of 13), we note that two other

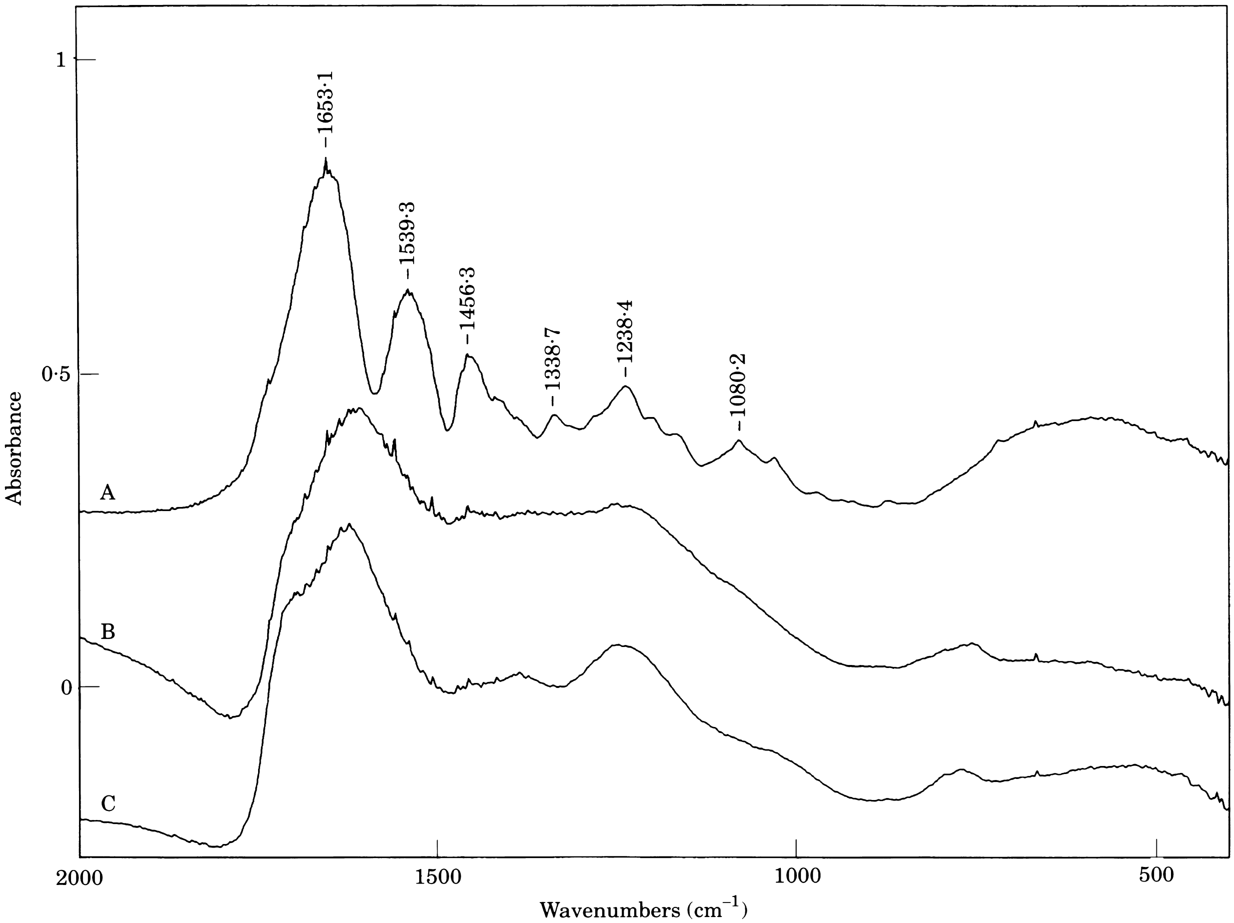

Figure 8. Infra-red spectra of the 1N HCl-insoluble fractions of (A) modern fresh goat bone, (B) modern bone burned to colour code 3 in the fire experiments, and (C) a fossil bone of burned appearance from square F27d in Hayonim Cave (370 cm below datum). Spectrum (A) is essentially that of collagen, characterized by the Amide I band at 1653 cm^{-1}, Amide II at 1539 cm^{-1}, and the proline absorption at 1456 cm^{-1}. The modern (B) and fossil burned bone (C) spectra are similar to one another and very different from that of fresh bone (A). The burned bone spectra can also be easily distinguished from the spectra of the insoluble fractions extracted from unburned fossil bones (see DeNiro & Weiner, 1988*a*). Note that, because the spectra represent only the collagen extract from the samples, their contours do not resemble spectra for collagen plus mineral content shown in the other figures.

bones identified as burned on the basis of colour did not yield insoluble fractions; these specimens either were erroneously identified as burned, or the burning damage was sufficient only to change their colour but not their internal matrixes.

In sum, our comparisons show that the signatures of crystallinity of bones altered by weathering, burning, and fossilization partly overlap. Infra-red spectrometry effectively measures heat-induced changes in the crystallinity of modern bone mineral, in a manner analogous to the information obtained by Shipman *et al.* (1984) using X-ray diffraction. The two methods do not monitor exactly the same physical parameters (Ziv & Weiner, 1994), but they can be expected to produce related readings of modern and fossil bones. We find that the HCl-insoluble techniques noted above, along with macroscopic changes in *internal* bone colour, more reliably diagnose burning damage on archaeological bones.

Discussion

The results of the experiments provide a few rather straightforward answers to some of the puzzling archaeological patterns documented earlier in this paper. We observed that burned bones are more fragile or brittle than unburned bones in an experimental context, and that their mechanical strength varied as a function of the extent to which the bones were burned. We also showed that bones buried in sediments prior to when a fire is lit can be burned by that fire, implying that bone deposition and bone burning potentially represent unrelated events during the formation of archaeological sites. Evidence of burning reveals only that fires and bone debris were in close proximity to one another at some moment in the past. Moreover, a threshold in burning intensity is evident in the potential of fire to burn bones within as opposed to buried below the coal bed: bones were easily calcined in open campfires fed with Mediterranean hardwoods, but bones buried just a few centimetres below the surface were burned only to the point of carbonization.

Because burning renders bones more susceptible to fragmentation, many of the identifiable features of burned bones may be lost soon after deposition. We find that the greatest decline in macroscopically identifiable bone occurs between burn colour codes 0 and 3, a gradient that contrasts the great strength of fresh bone with that of completely carbonized (blackened) bone; as burning damage approaches complete calcination, bones may be reduced to ever finer fragments. Finally, we demonstrated that weathering can rapidly induce changes in bone mineral crystallinity and loss of bone matrix, in a manner resembling the effects of fire. *In situ* diagenesis, associated with fossilization, may also lead to analogous loss of bone matrix over the course of many years.

Burned bones in archaeofaunas generally are smaller than unburned bones because burned bones are more easily broken. As fragile as burned bones may become, however, it is not heat alone that breaks them, but pressure (at least prior to the complete calcination phase; see also Knight's pressure-loading experiments (1985)). Trampling is probably the foremost, or most immediate, cause of breakage in cultural contexts, especially if people use a place for prolonged periods or visit it repeatedly. This kind of situation is especially likely to come about in natural shelters, and possibly also in masonry rooms. Beyond the basic question of whether hominids used fire, some of the greatest potential of burning data for human behavioural studies may lie in the realm of site structure and spatial analysis (for a related discussion, see Rosen, 1989). Indeed, factors such as fragmentation and insulation by soils are directly relevant to how hearths and their contents are transformed from magnets of human activity to stains in sediments and/or collections of charred objects. The archaeological signatures of burning considered here arise from the *interaction* of heat, burial, and pressure, the latter of which commonly arises from foot traffic in an occupied space. Hence, patterns of bone fragmentation and burning intensity may inform us about the intensity of place use by human beings, if qualified by data on sedimentation rates.

Infra-red and X-ray diffraction techniques applied to bone in modern controlled settings are valuable tools for understanding and modelling the character of diagenetic progressions. However, these techniques probably are not appropriate for confirming burning damage on archaeological bones. Our infra-red data on splitting factor and carbonate content did not reliably diagnose burning on prehistoric bones, contra Shipman *et al*.'s (1984) suggestion that measures of bone crystallinity (X-ray diffraction in their example) should accurately reflect such damage in archaeological contexts. While these criteria certainly diagnose burning damage in modern circumstances, many things can happen in the interval between bone discard by prehistoric humans and excavation by archaeologists. If anything, archaeologists' colour-based methods, supplemented by HCl-insoluble fraction analyses, are more reliable for identifying burning damage in archaeofaunas.

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Appendix

Mineral properties of modern goat and cow bones burned in control fire experiments

Source animal	Burn code	SF	565/603 (cm^{-1})	CO_3 (874/565) (cm^{-1})	PO_1* (cm^{-1})
Control set:†					
goat (core)‡	0	2·88	1·23	0·25	1032
goat (core)‡	0	2·98	1·22	0·21	1030
goat (surface)§	0	3·26	1·20	0·20	1032
goat (surface)§	0	3·43	1·20	0·16	1032
Set 1:					
goat	1	3·14	1·20	0·19	1032
goat	2	3·41	1·16	0·17	1032
goat	3	3·74	1·22	0·14	?
goat	5	5·70	1·06	0·03	?
goat	6	6·78	1·09	0·03	?
Set 2:					
cow	1	3·23	1·18	0·18	1032
goat	2	3·43	1·08	0·12	1041
goat	3	3·56	1·15	0·13	1038
goat	4–5	3·95	1·03	0·06	1042
goat	6	6·58	1·09	0·02	1045
Set 3:					
cow	1	3·03	1·20	0·21	1030
goat	2	3·23	1·07	0·14	1041
cow	3	3·75	1·15	0·16	1037
cow	4–5	3·64	1·11	0·15	1037
goat	6	7·44	1·10	0·02	1045
Set 4:					
cow	1	3·36	1·23	0·17	1032
cow	2	3·36	1·19	0·16	1033
cow	3	3·30	1·08	0·17	1043
goat	4–5	4·30	1·05	0·04	1043?
goat‖	6	5·08	1·08	0·07	1047
goat	6	6·12	1·10	0·01	1047

*This is the location of the major phosphate absorption peak. We note that it varies considerably, but we do not understand the significance of these variations.
†Four separate sets of samples analysed from the burning experiments.
‡Control sample taken from bone interior or core, representing a freshly skinned animal (first entry) and a skeletonized but somewhat greasy individual (second entry).
§Control sample taken from surface of skeletonized, somewhat greasy bone of animal that perished previous winter near experiment site in northern Israel.
‖Problematic result, run twice with second result shown below.