The Tensile Strength of Archaeological Bone

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The tensile strength of fresh and archaeological bovine bone has been determined perpendicular to its long axis indirectly by the diametral compression of disc-shaped specimens. Measured values of strength and bulk density ranged from ~ 60 MPa and $\sim 2 \cdot 0$ g cm^{-3} for the modern samples to only ~ 4 MPa and $0 \cdot 8$ g cm^{-3} for archaeological bone in the poorest states of preservation, respectively. Two mechanisms are suggested to be involved in the diagenetic degradation of bone. The initial loss of strength is attributed to hydrolysis of collagen although the precise detail of the attack is not known. This is followed by predation of tunnelling micro-organisms leading to an increase in pore volume and further loss of mechanical strength.

Keywords: ARCHAEOLOGICAL BOVINE BONE, TENSILE STRENGTH, DIAGENESIS, COLLAGEN HYDROLYSIS, BULK DENSITY, PORE VOLUME.

Introduction

Although recent years have seen a rapidly expanding body of published work concerning the chemical composition and mineral nature of archaeological bone, there has been little evidence of any significant investigation of its mechanical properties. Compared to the dietary, environmental and genetic information that is potentially preserved in ancient bone, studies of its mechanical properties apparently offer few rewards. Nevertheless, bone tissue constitutes a composite material comprising a biomineral (a stoichiometrically imperfect carbonate containing hydroxyapatite) intimately bonded to an organic matrix of highly cross-linked collagen fibres. The study of its mechanical properties, composition and microstructure in various states of degradation therefore has considerable potential for the interpretation of the diagenetic processes. There has been extensive work on the mechanical properties of ''living'' bone, both human and animal, in which the relationships between tensile strength, modulus of elasticity, composition and microstructure have been explored (Yamada, 1970; Vincent, 1982; Currey, 1984, 1988). Experiments on fresh bone have indicated that tensile and compressive strengths and Young's modulus can vary with a number of different factors. It has been suggested that bone is weakened by the presence of Haversian systems but it has not been clearly estab-

lished whether this is due to a general reduction in the amount of bone tissue present or a lower concentration of bone mineral (Currey, 1969). Subsequent work established that the Young's modulus of bone when tested wet depended upon both porosity and total calcium content (Currey, 1988). Several researchers (reported in Burr, 1980) have observed that other parameters, such as crushing strength, increase with increasing ash or mineral content (Bartley *et al.*, 1966; Burstien *et al.*, 1975). Carter & Hayes (1977) noted that the strength of human femur and tibia varied with ''apparent local density'' squared, whilst its compressive modulus varied with ''apparent density'' cubed. Bone also exhibits distinct anisotropy in the arrangement of its structural components so that, for example, in the mid-shafts of long bones the collagen fibres and associated apatite crystals run approximately parallel to their long axes. As a consequence, the measured tensile strength of bone depends on the direction of the applied force which is resisted most effectively in directions parallel to the collagen fibres (Vincent, 1982).

The brittleness of archaeological bone presents significant practical problems when trying to determine its strength. Direct tensile testing of brittle materials, although desirable, is notoriously unreliable due to difficulties associated with sample preparation and alignment. Torsional or shear loads superimposed on the sample during testing may cause premature failure. In the examination of Roman and Viking bovine bone, together with fresh specimens, many of these problems

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were overcome by static three-point bend tests used to evaluate flexural modulus and strength (Battaglia, 1985). These results were correlated with calcium content, weight loss on ignition and the histological appearance of each specimen. Although individual test specimens were relatively small ($\sim 2 \times 4 \times 30$ mm^3), they were sufficiently large to contain local inhomogeneities due to differential diagenetic degradation. Many of these potential problems can be avoided if an indirect method of tensile strength determination is used such as the "Brazilian test" (Hondros, 1959). In its simplest form, this test consists of the compression of a thin disc along its vertical diameter where failure eventually takes place. The tensile stress (σ) can be calculated from the compressive load at failure (P) using the formula:

$$\sigma = \frac{2P}{\pi dt},$$

where d is the diameter of a disc of thickness t. The correlation between results obtained using this and more direct methods has been shown to be improved if concave loading anvils are used. Premature specimen collapse at the contact edges can be delayed by superimposing a Hertzian contact stress distribution on the loading system (Awaji & Sato, 1979). A loading anvil radius to specimen radius ratio of ~ 1.25 was suggested to minimize this risk. This modified technique has been used sucessfully to measure indirectly the tensile strength of a brittle ceramic (Parry *et al.*, 1989) and transverse properties of glass and carbon fibre reinforced composite materials (Parry & Wronski, 1990) using relatively small specimens.

In this study the use of the diametral compression test was extended to the determination of the tensile strength of fresh and diagenetically degraded bovine bone samples.

Materials and Methods

Although it is generally accepted that collagen fibres provide the bone's resistance to tensile forces, it was decided to determine strength perpendicular to the long axes of bone specimens. It was anticipated that it would be easier to pull adjacent fibres apart transversely than to break the collagen strands themselves. Directions normal to the presumed orientation of the collagen fibres should therefore present planes of potential interfacial weakness. Samples of fresh (recently dead) bovine bone and archaeological bone from a variety of burial environments were selected for investigation. The near cylindrical sections of long bones were chosen to ensure that any directionality in the samples could be predicted and preserved during testing. Cow femur was adopted for the fresh bone samples due to its ready availability. Bovine metapodials were preferred for testing from the archaeological specimens since these are common in the

archaeological record in North West Europe and often survive intact whereas the larger long bones were often broken for marrow extraction.

The mid-shafts of bone samples were sawn into 50–60 mm lengths which were then cut longitudinally into three sections. Fresh bone and archaeological bone exhibiting exceptional preservation could generally be cut and shaped without cracking or crumbling. However, the majority of the archaeological material was too friable to machine without splintering and consequently was consolidated by vacuum impregnation with 10% Paraloid B72 (an ethyl methacrylate/methyl acrylate copolymer) in a 50:50 mixture of acetone and toluene. After clearly marking the long axes on each sample several small, cylindrical specimens were drilled using a purpose made tubular saw. Water was used as both coolant and lubricant during drilling which produced cores nominally 6.5 mm in diameter. Parallel faces were polished on these cylinders using a metallographic polishing wheel and a stainless steel holder resulting in thicknesses ranging between 0.8 and 4.8 mm. Care was taken to preserve the orientation of each specimen throughout and samples were finally washed in two changes of acetone which also served to remove the consolidant. All discs were then oven-dried at 105°C, transferred to a vacuum desiccator and weighed. The mean diameter and the thickness of each disc were determined using a vernier micrometer.

To determine pore volume a series of experiments was undertaken on discs drilled from archaeological bone specimens. These were prepared, dried, measured and weighed in the same way as before but were additionally weighed wet. To ensure that water penetrated the entire pore structure the discs were immersed in distilled water and left under vacuum overnight to exclude any entrapped air. On removal from the water each disc was blotted briefly to remove any surface water and re-weighed. The pore volume was calculated from the difference between the wet and dry weights and the volume of the discs, assuming unit density for water at room temperature. Bulk density was calculated using the oven dried weights.

Mechanical testing was carried out using a Lloyd 30 kN 6000R computer controlled testing machine fitted with concave loading anvils of radius 5 mm, shown schematically in Figure 1. A loading rate of 0.1 mm s^{-1} was used to test 105 specimens from 20 different bone samples. Load and cross-head displacement were recorded autographically for each test and a typical set of traces is shown Figure 2. Fracture surface examination of gold coated specimens of interest was carried out using a Cambridge Instruments S600 scanning electron microscope.

Results

Tables 1–4 show the calculated bulk density (g cm^{-3}) and diametral compressive strength (MPa) of

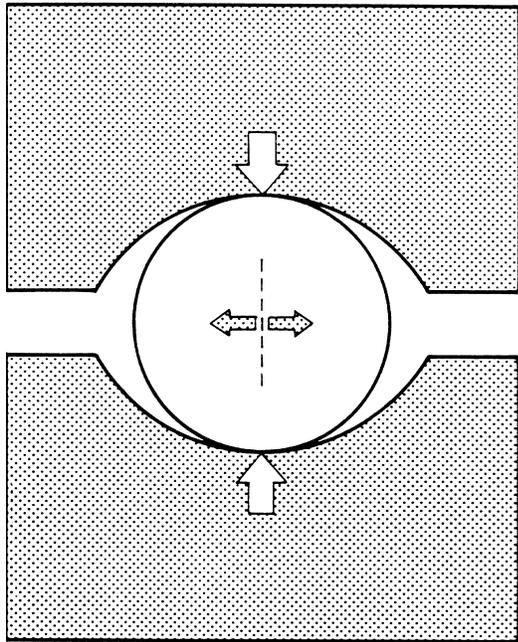Figure 1. Principal forces and loading geometry showing concave loading anvils.

Table 1. Density and diametral compressive strength results of samples taken from fresh bone together with chemical analysis where available

ρ (g cm^{-3})	σ (MPa)	% Carbon	% Hydrogen	% Nitrogen
1·97	47·7	11·53	2·13	3·62
2·01	54·8	11·24	2·04	3·50
1·99	55·3			
2·30	57·8			
2·01	65·9			

Table 2. Density and diametral compressive strength results of samples taken from "good" archaeological bone together with chemical analysis where available

ρ (g cm^{-3})	σ (MPa)	% Carbon	% Hydrogen	% Nitrogen
1·88	19·1			
1·95	22·6			
1·84	23·1			
1·88	24·3	12·73	2·35	4·21
1·90	24·6			
1·78	25·3	13·29	2·30	4·47
1·84	25·4			
1·88	27·5			
1·91	29·6			
1·90	31·5			
1·95	32·3	11·31	2·22	3·69
1·90	38·2			
1·88	38·2			

Table 3. Density and diametral compressive strength results of samples taken from "moderate" archaeological bone together with chemical analysis where available

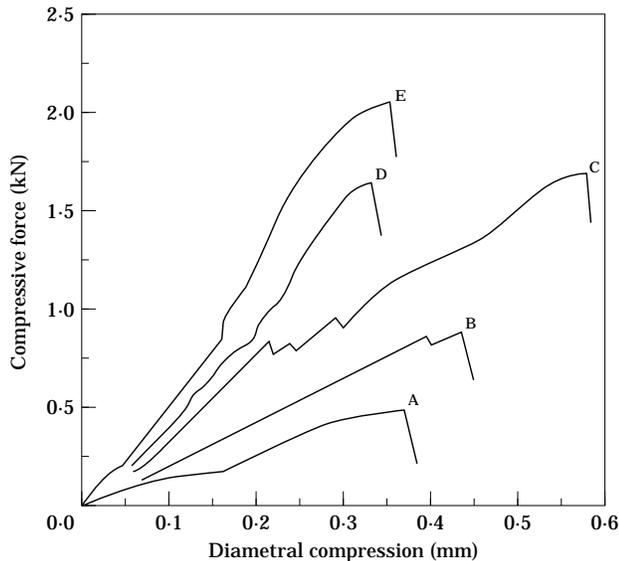| ρ (g cm^{-3})	σ (MPa)	% Carbon	% Hydrogen	% Nitrogen
1·37	8·40			
1·30	8·47			
1·28	8·64			
1·39	8·72			
1·42	8·80			
1·81	8·88			
1·37	10·3			
1·33	10·9	5·36	1·08	1·13
1·36	11·4			
1·37	11·6			
1·37	12·2			
1·39	12·5	5·32	1·19	0·82

Figure 2. Compressive force—displacement traces, corrected for differences in thickness, for poorly preserved samples of, A, medieval human femur and, B, bovine bone dating from the Iron Age. Well preserved samples of Roman origin are shown in C and D whilst the behaviour of fresh bovine bone is demonstrated by E.

specimens taken from samples of fresh bone and archaeological bone in good, moderate and poor states of preservation, respectively. It can be seen that the tensile strength of fresh bone, measured transverse to the long axis of the bone, ranges between 47 MPa and 66 MPa for this testing technique. Where available, the tables also indicate the results of carbon, hydrogen and

nitrogen analyses determined using a Carlo Erba Strumentazione Elemental Analyser. Figure 3 shows a plot of tensile strength against bulk density for all samples. Despite some scatter in the case of discs of highest density, which correspond to archaeological bone taken from water-logged deposits, the values show a dramatic fall in strength after only modest losses in bone density. Another interesting feature of Figure 3 is that the data appear to fall into two distinct groups, those discs with bulk densities between 1·72 g cm^{-3} and 2·0 g cm^{-3} and discs with bulk densities between 0·8 g cm^{-3} and 1·4 g cm^{-3}. The first group exhibits a wide range of tensile strengths and shows a

Table 4. *Density and diametral compressive strength results of samples taken from ''poor'' archaeological bone together with chemical analysis where available*

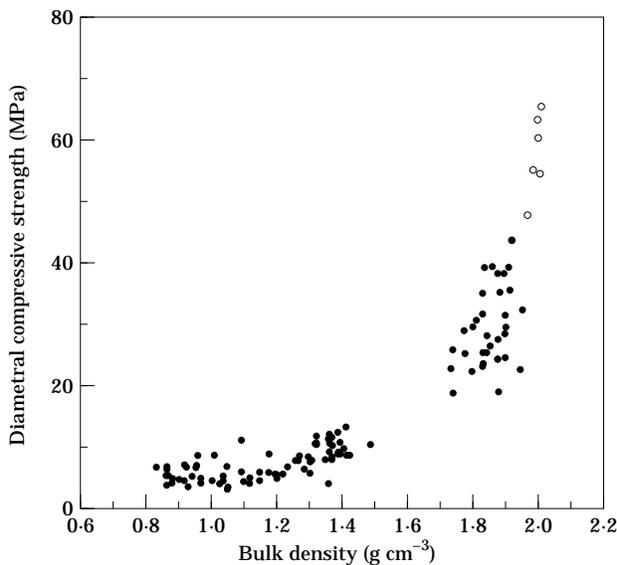| ρ (g cm^{-3})	σ (MPa)	% Carbon	% Hydrogen	% Nitrogen
1·05	3·25	5·11	1·11	0·85
1·06	3·61			
0·97	4·15	6·10	1·24	1·27
1·12	4·19			
1·10	4·52			
1·04	4·55			
1·15	4·60	5·85	1·23	0·91
1·00	4·70			
1·12	5·02	4·16	0·95	0·62

Figure 3. Plot of indirect tensile strength against bulk density for samples of fresh and archaeological bovine bone. ●, Cow metatarsals (archaeological); ○, cow fermur (modern).

rapid loss of strength for small reductions in bulk density. By contrast, the second group shows almost negligible decrease in strength even though the bulk density varies by a factor of almost two. None of the samples tested showed bulk densities between 1·5 g cm^{-3} and 1·7 g cm^{-3}. Figure 4 shows a plot of pore volume against bulk density for the samples investigated. Many of the discs lie on a straight line suggesting that there is a direct (linear) relationship between bulk density and pore volume.

To explore some of the factors which may influence strength, 20 samples that had failed cleanly along the vertical diameter were selected for carbon, hydrogen and nitrogen analysis. These results are grouped together in Table 5. To examine the relationship between mechanical strength and protein content in the archaeological samples, strength is plotted against nitrogen content in Figure 5. Although it is possible to see a general trend of increasing tensile strength with higher nitrogen content the correlation is poor when
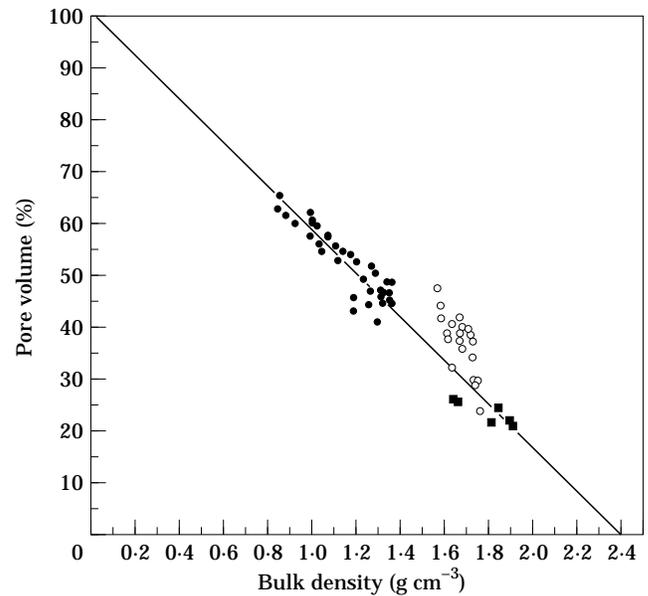
Figure 4. Plot of porosity against bulk density for samples of cow metatarsals from aerated (●) and waterlogged (○) soils. Note the intercept at a bulk density of $\sim 2·4$ g cm^{-3}. ■, Cow femur (modern).

Table 5. *Results of chemical analyses carried out on selected samples*

σ (MPa)	% Carbon	% Hydrogen	% Nitrogen
3·25	5·11	1·11	0·85
3·84	6·13	1·19	1·23
4·15	6·10	1·24	1·27
4·60	5·85	1·23	0·91
5·02	4·16	0·95	0·62
7·91	4·43	1·00	0·54
10·5	3·72	0·89	0·44
10·9	5·36	1·08	1·13
11·2	8·72	1·59	2·32
12·5	5·32	1·19	0·82
13·4	3·82	0·95	0·54
16·4	9·21	1·66	2·72
23·6	11·32	2·14	3·73
24·3	12·73	2·35	4·21
25·3	13·29	2·30	4·47
29·6	11·01	2·04	3·62
32·3	11·31	2·22	3·69
42·6	11·93	2·17	3·94
47·7	11·53	2·13	3·62
54·7	11·24	2·04	3·50

compared to Figure 4. Scanning electron fractographs of specimens of interest are presented in Figures 6–8.

Discussion

If it is assumed that the collagen fibres are aligned predominantly parallel to the long axis, values of tensile strength of ~ 60 MPa determined in this work for fresh bone should be compared with reported values of ~ 116 MPa for the tensile strength of osteons measured at 90° to the orientation of the fibres
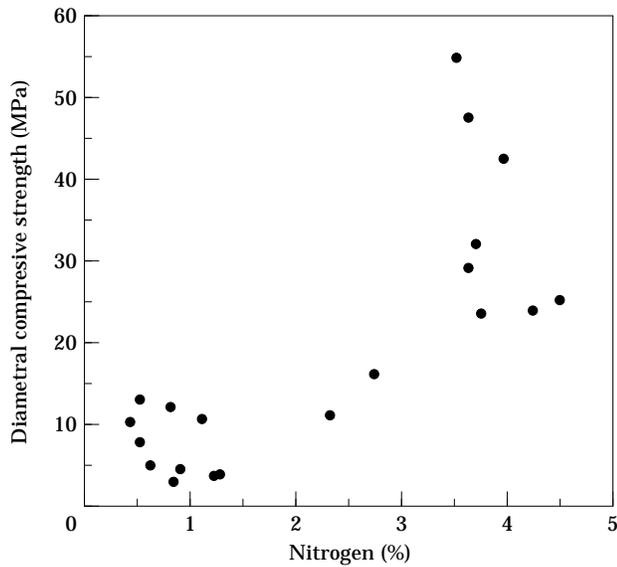
Figure 5. Plot of indirect tensile strength against nitrogen content of samples.

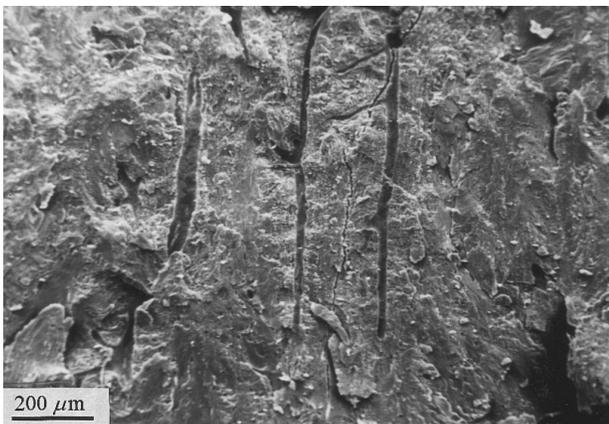Figure 6. Scanning electron micrograph of disc showing fracture along Haversian canals.
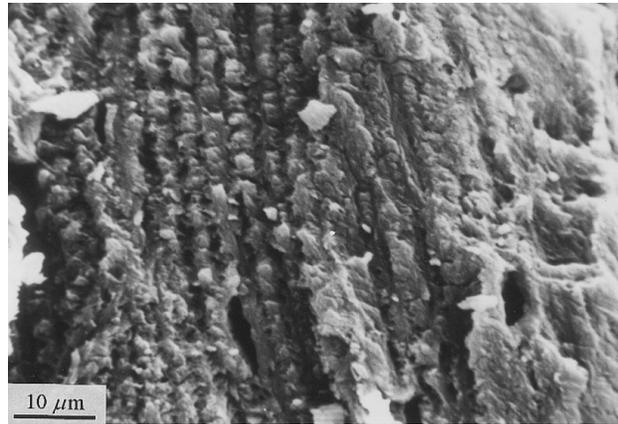

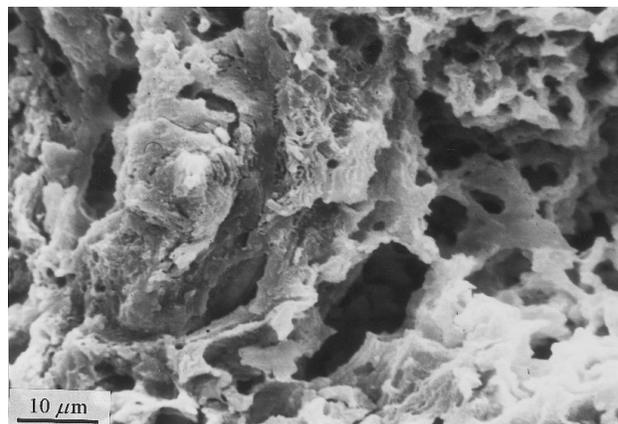Figure 7. Scanning electron micrograph of well preserved archaeological bone showing plywood-like lamellae.

Figure 8. Scanning electron micrograph of poorly preserved archaeological bone showing porous spongy texture.

(Vincent, 1982). The technique used here therefore appears to underestimate the value measured, possibly more directly, by other techniques. These differences have also been observed in the mechanical testing of other brittle materials (Parry *et al.*, 1989) and can be attributed to the fact that the diametral compressive test produces a biaxial stress field as well as differences in the volume of the specimens subjected to the maximum stress.

The apparent density calculated by dividing the mass of each specimen by the volume enclosed within each disc is termed here bulk density. This should not be confused with the true or microstructural density of bone tissue since the measured volume will also contain a considerable amount of free space in the form of voids. This "empty" space or pore volume may be expressed as a percentage of the total volume. Some researchers (e.g. Currey, 1988) have used the term volume fraction which is defined as [1 − porosity]. It can be seen from Tables 1–4 that there is a general trend of increasing tensile strength (measured perpendicular to the long axis) with increasing bulk density. These data, plotted in Figure 3, have a bimodal appearance suggesting that two mechanisms may be responsible for differences in both the bulk density and tensile strength of ancient bones. Alternatively, the absence of intermediate specimens may suggest that bones buried in even moderately aggressive environments suffer a rapid loss in density and a corresponding reduction in strength shortly after deposition.

However, Figure 4 suggests that for well preserved bone (i.e. bone with nitrogen contents above 3·5%) there is a wide range of samples of very similar strength and nitrogen and therefore protein content. If bulk density against nitrogen content is examined it can be seen that all the well preserved samples have bulk densities of around 2·0 g cm^{-3}. Thus, for well preserved bone, the major factor controlling tensile strength would appear to be protein or collagen content rather than porosity level.

The bulk density of archaeological bone may be influenced by a number of factors. The first and probably most important of these will be the micro-architecture of the bone itself, i.e. the size and distribution of osteocyte lacunae, canaliculi and Haversian canals in the specimen. Because the region from which discs were cored was controlled as closely as possible during sample preparation, each disc can be assumed to have a very similar micro-architecture. Differences in bulk density cannot therefore be attributed solely to this variability. A potentially significant contributing factor must also be the amount of additional porosity produced as a result of microfocal destruction or "tunnelling" by micro-organisms (Hackett, 1981). Other influences include variability in the chemical composition of the bone tissue itself, which will include both natural differences in the degree of mineralization during life and those brought about by diagenetic change. Even in essentially neutral soils there is considerable evidence from carbon, hydrogen and nitrogen analyses to show that slow hydrolysis of collagen takes place resulting in its inevitable loss from buried bones. Similarly analysis of phosphorous and calcium content has shown that acid soils result in a loss of bone apatite leading to collagen rich areas (Turner-Walker, 1993). Infiltration by metal salts and diagenetic minerals may also further influence the bulk density of archaeological specimens.

The plot of pore volume against bulk density (Figure 4) intersects the pore volume axis at 100% for zero bulk density and extrapolates to a density of $\sim 2 \cdot 4$ g cm^{-3} at zero porosity. This figure, it is suggested, represents the true or microstructural density of bone tissue in the samples examined. Those data that lie off this straight line and appear to show an increase in pore volume for no additional increase in bulk density, derive exclusively from waterlogged burial conditions. This deviation from the general behaviour may therefore be interpreted in terms of water being absorbed by collagen which could be accommodated by swelling of the fibres as opposed to filling pores in the structure.

The results of the chemical analyses presented in Table 5 are consistent with an initial rapid decrease in the mechanical strength of bone without appreciable loss of collagen or increase in porosity. In the majority of burial environments this initial rapid modification of bone properties appears to be followed by a much slower weakening of the structure brought about by an increase in porosity rather than a gradual loss of its organic fraction. This gradual loss of strength is probably attributable to an increase in the number and size of microscopic voids that can act as failure initiation. Furthermore, the high number of natural channels (e.g. Haversian canals) running parallel to the long axes of bones will facilitate crack propagation in these directions. This is consistent with scanning electron fractographs of disc surfaces. Figure 6 shows a disc in which failure was initiated by, or followed, a plane of weakness caused by several tunnels or canals. From their geometry it is most probable that these channels represent Haversian canals and canals of Volkman rather than tunnels created by the action of micro-organisms.

As can be seen from Figure 7, the fracture surface appearance of archaeological bone with a high collagen content (excavated from Iron Age deposits at Stanwick, North Yorkshire) shows a distinctly grained "plywood-like" appearance. Higher magnification clearly shows the lamellar structure of bone (for comparison, see Figure 1 of Weiner et al., 1991). By contrast, Figure 8 shows the fracture surface of a disc cut from archaeological bone from the same site which was considerably degraded showing both a low bulk density and high porosity. At higher magnification it can be seen that there has been complete loss of microstructure and that the originally dense bone tissues have been replaced by a very spongy texture. There were no obvious signs of any pathological condition when examined in bulk. In this specimen the structure of the bone has been considerably weakened by the numerous voids, any one of which could act as crack initiation sites.

Conclusions

This work has indicated that two different, but not necessarily independent, mechanisms are involved in the diagenetic degradation of bone leading to a loss of strength when measured perpendicular to the collagen fibres. The initial loss of strength is suggested to result from hydrolysis of collagen although it is not known whether this involves the attack of peptide bonds, cross-links between adjacent collagen molecules or the protein-mineral bond. Degraded fragments are likely to be restricted within the bone structure since strength loss occurs without a significant reduction in the nitrogen content.

Sufficiently weakened bone tissue will allow predation by micro-organisms and a ready protein source will permit the proliferation of the organisms responsible for the microfocal destruction of buried bones. It is suggested that the tunnelling action of these organisms is responsible for the large increase in pore volume observed in archaeological bone and its associated loss in mechanical strength. Microbial action will decline with accelerated hydrolysis of the remaining collagen due to its increased surface area. An equilibrium will then be established between the deproteinized bone and its burial environment allowing the groundwater to remodel the remaining inorganic component. This well either lead to total loss of the sample or consolidation of the remaining apatite crystallites by dissolution and re-precipitation leading ultimately to fossilization.

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