

Strain rate dependence of Work of Fracture tests on bone and similar tissues: reflections on testing methods and mineral content effects

JD Currey^{a,†}, K Brear^{a,††}, P Zioupos^{b,*},

^a Department of Biology, University of York, York, YO10 5YW, UK

^b Musculoskeletal & Medicolegal Research group, Cranfield Forensic Institute, Defence Academy of the UK, Shrivenham, SN6 8LA, UK

Abstract

This paper is concerned with the effect of different strain rate on the Work of Fracture (W_f) of various vertebrate mineralised tissues, controlling for the effect of mineral content and Young's modulus of elasticity. Using specimens of uniform shape and size values for the Work of Fracture of specimens tested at various deformation rates, and also the energy absorbed by notched specimens in impact, are reported. The results indicated that, of those tested, for most bone specimens the Work of Fracture measurements were constant like in the case for a 'material property'. Variations due to loading conditions (deformation rate) were small, with the exemption of antler, which is relatively poorly mineralised and in which the Work of Fracture values increased by a factor of 4 across the range from quasistatic loading to impact. The Tattersall and Tappin (1966) test has shown itself to offer some great advantages: if the quest is for a fracture toughness test for an unknown tissue it offers reliability, it is perhaps more forgiving to handling errors, it also suffers less of the influence of strain rate effects and uses relatively simple instrumentation. It is also able to demonstrate the remarkable toughness of antler bone which other more commonly used fracture toughness methods cannot do.

Keywords Bone, antler, dentine, work of fracture, impact.

† Deceased 18 December 2018;†† Deceased 4 November 2002

* Corresponding author at: Cranfield Forensic Institute, Cranfield University, Defence Academy of the UK, Shrivenham, SN6 8LA, UK

E-mail address: p.zioupos@cranfield.ac.uk

1 Introduction

Various mechanical properties of bone (and other bone-like mineralised tissues) can be partially 'explained' statistically by such variables as the amount of mineralisation, porosity, and histology of the tissue (hereafter called 'bone'). For instance, Young's modulus of elasticity is well explained by a combination of two variables, mineralisation and porosity [1]. Bending strength and Young's modulus are also highly correlated with each other [2]. Ultimate strain at failure is very well explained by mineralisation [3] as is impact energy absorption and notch sensitivity [4]. Quasi-static tensile strength, however, is not very well predicted by any simple combination of explanatory variables. Although there is in general an increase in strength with an increase in mineralisation, very highly mineralised bone is weak [5], and antlers – which are on the other side of the mineralization spectrum - are relatively strong [6-8]. The controversy has to do with the way energy is dissipated throughout the volume of a sample in tensile tests, some of it going into the elastic deformation, some in pre-fracture toughness (widespread microcracking absorbing energy at yield), some in actual fracture toughness in both stable and unstable crack growth [9]. In three studies of this lab the most characteristic cause-effect feature that was observed is that mineralisation affects mostly the post-yield ability of bones to absorb energy relatively to the pre-yield one [2.4.10].

There is also evidence that bone's fracture properties, as opposed to elastic properties, are particularly strain-rate dependent [11-16]. That is to say, fracture properties are not truly *material* properties, in that they depend to a considerable extent on the loading mode and imposed conditions; in other words, they are quite contextual. In general strength increases with strain rate up to a strain rate of about $\sim 0.1 \text{ s}^{-1}$ then, according to most recent studies, declines. Behiri and Bonfield [17,18] produced controlled cracks in bovine tibial compact tension specimens at various propagation velocities in the quasistatic range. As the crack velocity increased so the value of K_C also increased, about three-fold with a 12-fold increase in crack velocity. However, catastrophic crack travel, which eventually supervened as the deformation rate was increased further, was accompanied by a reduction in K_C . (These cracks travelled in the longitudinal direction relative to the long axis of the bone, which is not the direction in which dangerous tensile cracks usually travel.) More recent studies in tension and compression of human cortical bone have shown that here is an inflection of strength and toughness of bone above and below what we can call the physiologic strain rates (those for walking and running $0.01\text{-}0.1 \text{ s}^{-1}$) with bone being more brittle at high strain rates and for the very low ones, where creep rupture dominates [19,15]. The secret seems to be that bone demonstrates a ductile-to-brittle transition (DBT) [9,20-23] with tougher bones being those that delay the onset of DBT [9]. The key to bones toughness is the ability to avoid DBT for as long as possible during the deformation and in particular by avoiding damage localisation in the peri-yield process which when it occurs leads to low post-yield strains and low-energy absorption to failure [19]. The conundrum is that some of the exhibited 'toughness' is intrinsic deriving from the hierarchical organisation and architecture of bone [24-26] and some is extrinsic from the test conditions and therefore contextual. In this respect it is unfortunate when there isn't really a common point of reference for either the tissue or the testing method as the two interplay in this process. One toughness measuring test, which stands out for its simplicity and for producing reliable results in the authors' experience is the method introduced by Tattersall and Tappin [27] to determine the Work of Fracture (W_f) of brittle materials. It has been used quite often on mineralised tissues [28-36]. This method is similar to various fracture mechanics configurations in that, among other things, it aims to restrict damage and energy dissipation in the specimen to the tissue volume associated with the travelling fracture. Zioupos and Currey[37] have shown that, in human bone at least, the various methods of estimating toughness characteristics produce similar results when examined against the chronological age of the donors. However, once again the W_f tests were those which showed most prominently the drop in toughness with ageing in human bone and also those that correlated well with the organic phase of the material, which provides integrity and cohesion to the structure [38,39]. The present paper, therefore, is concerned with the effect of different strain rate (actually deformation rate, whose relationship to strain rate once a crack starts to travel is complex) on the Work of Fracture of various vertebrate mineralised tissues, controlling for the effect of mineral content and Young's modulus of elasticity. Tests were conducted using specimens of uniform shape and size. The results reported include the Work of Fracture of specimens tested at various deformation rates, and also the energy absorbed by notched specimens in impact. Finally, we consider the implications of the fact that for most bone Work of Fracture seems to be relatively constant, but for antler, which is relatively poorly mineralised, deformation rate affect W_f values very strongly.

2 Methods

2.1 Work of fracture

Specimens were obtained from the femur of a tiger *Panthera tigris*, bovine femur *Bos taurus*, the antler of a Red Deer *Cervus elaphus*, and the dentine of the tusk of a narwhal *Monodon monoceros*. These tissues were chosen because from other work we knew a considerable amount about their properties, and we wished to see whether our findings were generalisable between

varied vertebrate groups. Figure 1 shows in backscattered micrographs the histology of these bone analogues. Narwhal tusk dentine is at this magnification (scale bar or 200 μm common for all 4) amorphous with no discernible features; it possesses no osteocytes or osteons or lamellar structures such as seen in common bone. Bovine, antler, tiger and human (shown in (d) for illustration) are a mixture of fibrolamellar, laminar and osteonal histological types. The grey level in Fig 1 is directly related to the backscattered yield of electrons and therefore to the mineral content of the tissue, therefore, the more recently deposited osteons appear darker and the interstitial lamellae and some older osteons brighter.

FIGURE 1

Specimens were oriented in the longitudinal axis of the femurs and of the antler, and parallel to the helical grain of the Narwhal tusk [40]. The specimens had overall dimensions of 4 mm breadth, 2 mm depth and about 35 mm length. Before turning these blanks into Work of Fracture specimens we measured their Young's modulus of elasticity in bending, using a three-point bending test, at a head speed of 1 mm min^{-1} . We allowed for machine compliance. All specimens were kept thoroughly wet during preparation and testing.

The triangular ligament of the Work of Fracture specimens was 2 mm in height and had a base of about 3.8 mm. The specimens were loaded in three-point bending, with a gauge length of 28 mm at cross-head speeds varying between $8.3 \times 10^{-4} \text{ mm s}^{-1}$ and 3.3 mm s^{-1} , a 4,000-fold range. Because it is likely that Young's modulus has an effect on fracture behaviour we attempted, for any bone, to distribute specimens with different values for Young's modulus evenly between the different head speeds. The specimens were loaded in a water bath at room temperature, and were completely wet at all times. The work done in breaking the specimen was calculated from the area under the curve, normalised by dividing by *twice* the area of the ligament.

FIGURE 2

For the Work of Fracture test to give a realistic estimate of the work done in driving the crack through the specimen, it is important that the crack travel should be controlled. In the type of test we were using, the load-deformation rate was typically almost straight to near the greatest load, the curve then flattened slightly before declining gradually to a very low load. When the load had fallen to 5% of the maximum load, the test was stopped. On occasions, however, the load dropped suddenly, that is, the crack travelled in an uncontrolled way. We considered, arbitrarily, the test to have failed if the sudden loss of load was greater than 10% of the maximum load. These 'failures' are important, and are discussed below.

In successful tests the machine and the specimen are in equilibrium at all times, so all the work done by the machine is eventually used to drive the crack through the specimen, virtually none being lost as noise, the kinetic energy of the separated pieces flying apart, or the compliance of the testing apparatus. Any small amount of energy absorbed by the relatively stiff testing machine was recovered before the test finished.

The rate of crack travel was not measured. However, knowing the height of the triangle and the time taken to reduce the load borne to 5% of the greatest load, (by which time the crack will very nearly have separated the specimen in two) we estimate it as varying, over three orders of magnitude, between about 0.4 mm s^{-1} and $3 \times 10^{-4} \text{ mm s}^{-1}$. Because the specimens are all of nearly the same shape, the differences in cross-head speed (deformation rate) give a good idea of the differences in strain rate and, after crack initiation, of crack travel rate.

2.2 Impact energy

Specimens 40 mm long overall, with a gauge length of 35 mm, 3 mm wide and 4 mm deep, with a slot of 2 mm depth and 0.35 mm width cut in the middle of the tensile surface, were tested, wet, in a Hounsfield plastics impact tester. This is a falling pendulum, whose loss of energy after fracturing the specimen is measured. (Differences in Young's modulus between the impact specimens and the Work of Fracture specimens are not very secure because the rather high depth-to-length ratio of the impact specimens results in shear deformations becoming relatively large, and reducing the accuracy of estimates of Young's modulus obtained from beam equations.

3 Results

3.1 *The effects of deformation rate: static loading*

The main results are shown in Table 1 and summarised in Fig. 3. Many of the bovine and tiger specimens failed catastrophically at the higher head speeds (Table 2), and the values of Work of Fracture of such catastrophically failing specimens are ignored here. We shall, however, discuss these failures later. All the antler and Narwhal dentine specimens failed in a controlled way at all head speeds. Since, as already mentioned, the Young's modulus may affect fracture behaviour, we distributed the values of Young's modulus evenly across head speeds. The statistical analysis of this, and also of the distribution of calcium content and Work of Fracture between head speeds are shown in Table 3. This shows that, as we had endeavoured to arrange, there are no significant differences between the specimens tested at different deformation rates in respect of their Young's modulus or their calcium content. The differences that were seen, or were not seen, in the relationship between cross-head speed and Work of Fracture, were therefore not affected in any important way by confounding differences in Young's modulus or calcium content.

FIGURE 3

The relationship between the Work of Fracture and cross-head speed for the four types of material is shown in figure 3. Broadly speaking the common long bone tissue (bovine, tiger, and human added for comparisons) showed W_f values which were relatively constant across the range of speeds. Dentine tissue (some data added for Elephant tusk as well) shows itself to be much stronger in impact but not at lower rates. Antler bone was the only conventionally designed bone which is remarkably tough throughout the range. The W_f values shown are those for the part of the deformation process during which fracture was stable and there was a force driving the crack; and these W_f values are the ones we record and analyse vs mineral content and modulus of elasticity later on. Once the crack front became unstable no further energy absorption was recorded. Consequently, there are two aspects to consider here: (1) the work values produced for as long as fracture was stable and (2) the fraction of specimens which fractured in stable fashion before the crack growth became unstable (Ductile to brittle transition). The common femoral bones fractured in a stable manner for up to running speeds (loading rates $>0.008 \text{ s}^{-1} \sim \text{strain rate } >0.1 \text{ s}^{-1}$). No further tests were conducted on bovine specimens at cross head speeds greater than 0.083 mm s^{-1} , for at that speed six specimens out of ten failed catastrophically (Table 2). All five tiger specimens tested at 0.83 mm s^{-1} failed catastrophically and of those tested at a cross head speed of 0.083 mm s^{-1} eleven specimens out of fourteen failed catastrophically. The antler and the Narwhal dentine specimens all showed controlled crack travel at all head speeds. Therefore, it is a reasonable speculation that W_f values may have been climbing up with strain rate for all bones had they been able to stay in a stable fractured bone throughout the process.

Deer antler: On a log-log scale the deer antler show an almost linear increase in Work of Fracture, from a mean of 3760 J m^{-2} at a cross-head speed of $8.3 \times 10^{-4} \text{ mm s}^{-1}$, to a mean of 14690 J m^{-2} at a cross head speed of 3.3 mm s^{-1} . The slope of the linear regression (Table 4, equation 1) relating log Work of Fracture to log cross-head speed is 0.16, with an R^2 of 85%. If Young's modulus is used as an extra explanatory variable (equation 2) the fit is now remarkably good, the value of R^2 increasing from 85% to 93%. Equation 1, in its unlogged form is: $W_f = k \times \text{crosshead}^{0.16}$, and equation 2, which is a very good fit, is $W_f = k' \times \text{crosshead}^{0.16} \times E^{1.3}$. The implication of Young's modulus appearing as the 1.3 power in equation 2 is that Young's modulus has a positive effect on Work of Fracture.

Narwhal dentine: In contrast to the behaviour of antler, the Narwhal dentine Work of Fracture showed some tendency to decrease with deformation rate, from a mean of $9,150 \text{ J m}^{-2}$ at $3 \times 10^{-4} \text{ mm s}^{-1}$ to a mean of 4610 J m^{-2} at 3.3 mm s^{-1} . The linear regression was significantly negative, ($P = 0.023$), but the explanatory power of the equation was poor ($R^2 = 29\%$).

Bovine bone and tiger bone: These tissues showed no trend. There is considerable spread in the individual values of Work of Fracture but, for instance, in bovine bone the mean Work of Fracture is almost the same at $8 \times 10^{-4} \text{ mm s}^{-1}$ and $8 \times 10^{-2} \text{ mm s}^{-1}$ and in tiger they are about the same at cross head speeds of $8 \times 10^{-4} \text{ mm s}^{-1}$ and 0.83 mm s^{-1} . Over the same ranges of cross-head speeds the antler shows a 60% increase and a doubling, respectively, in the Work of Fracture.

3.2 The effects of mineral content and Young's modulus on Work of Fracture

The results so far have been concerned with the effect of deformation rate on the Work of Fracture of mineralised tissues. An original purpose of this work was to try to tease out the effect of stiffness and, probably more important, the effect of mineral content, on Work of Fracture by holding the specimen shape constant. As it turned out, deformation rate had such a marked effect, and in different directions in different tissues that simple comparisons are difficult. Figures 4a,b do not include the antler specimens, and show Narwhal only up to the head speed before its values of Work of Fracture started to decline. The figures show the relationship between log Work of Fracture and log calcium or Young's modulus. The mineral content of each specimen was determined colourimetrically using the method of Sarkar and Chauhan (1967) and is expressed as milligrams of calcium per gram dried defatted bone [41]. Although highly statistically significant the relationships are not strong (legend to figure 4), and are caused mainly by differences between the tissues as a whole, rather than showing any relationship within any tissue.

FIGURE 4

3.3 Impact energy absorption

The bone specimens broken in impact had the same relative positions as the Work of Fracture specimens, but the differences were greater (Figure 3). The bovine specimens had by far the lowest values, the tiger specimens absorbing about three times as much energy. Whereas the ranking of the Narwhal and antler were less consistent across the different tests. In impact the Narwhal specimens absorbed about six times more energy than the tiger specimens, and the antler specimens, in turn, considerably more than the Narwhal. In fact, the values for the antler specimens are minimum values; it proved impossible to fracture them completely; they always distorted into a bow and slipped between the blocks holding them. Most of their deformation was, however, permanent, the bowed specimens did not spring back straight when they had passed through the blocks. Therefore, most of the energy reported here was used up in damaging the specimens, but more energy would be required to break them in two. The relationship between log impact energy and log calcium is extremely strong (Figure 5).

FIGURE 5

4 Discussion

The bone examples used here are only few of the many bone adaptations present in nature. As figure 6 shows 'bone' is able to achieve a wide range of properties at the tissue level by a simple interplay of its three main ingredients, namely water, organic and mineral. A range of modulus of elasticity values and hardness (indicative of yield) can be achieved by this adaptation which is driven by composition alone. The figures include common long bones which act with the usual purpose to transfer loads in life. This concept is simple as far as commonly encountered bones increase their material stiffness and hardness in line with mineral content at the tissue level. This reflects on their performance in quasistatic conditions. Toughness, experienced as energy absorption, when it matters for survival, is a bit more complicated. It appears that a decrease in mineral content is invariably good for the ability to absorb energy during fracture and for stable fractures. It also confers notch insensitivity and damage tolerance [4]. However, in life, other bone tissue fashioned differently, like in dentine, also seem to possess toughness benefits. The bone of the tusk of the Narwhal (and the similarly made elephant tusk) are tough by a combination of lower mineral content and a laminar microstructure, which shows damage

absorbing capacity at a sub-microscopic level [42]. Without a doubt some insightful studies can be written for tooth dentine alone and this is remarkable for a tissue which does not possess the remodelling processes of normal bone. Nature equipped dentine, this material which is only produced once, with enough toughness from the start to sustain service for life and without the need for a later self-repair, which is not able to perform.

FIGURE 6

Of the four materials examined here, only antler bone shows an *increase* in Work of Fracture with deformation rate in the static tests. The other tissues show an unchanged, or even a somewhat decreased Work of Fracture. However, this tendency cannot be taken at face value, because the two mammalian long bones also showed an increased tendency to fracture catastrophically as deformation rate increased (Table 2). The successful tests are presumably, therefore, biased towards the higher values that the bones would achieve at the relevant deformation rates. It is interesting that, overall, at the two lowest deformation rates antler specimens have a Work of Fracture significantly *lower* than that of Narwhal dentine (Mann-Whitney U : $p = 0.012$).

Carter and Caler [15] proposed that bones fracture after they have accumulated a 'certain amount of damage', and that the rate of damage accumulation is proportional to a very high power of the stress. Fracturing after a certain amount of damage does make this 'accumulated damage' value a 'material property' very much like the material property we seek to establish here. However, the accumulated damage was defined as a modulus reduction, in effect the upper limit of change in a material property value. On the other hand the present experiments, which quantify the *work* done on the specimens, they measure in some way a product of the applied load and the deformation over the fracture area. That is the energy density during controllable fracture of bone.

Worth pointing out that the resistance to deformation is the responsibility of the narrow triangular ligament. The configuration of the test pieces is such that high stresses tend to be confined to this small piece of the specimen. Therefore, the ability of antler bone to undergo *widespread* damage before failing, thereby increasing their toughness [8], might seem in the present context to be more or less irrelevant. However, it is probable that, even if the high stresses are found only locally, antler has a greater ability to spread the damage further away from the crack tip than the more highly mineralised tissues. In bovine femur a two orders of magnitude increase in deformation rate produced no change in Work of Fracture. In Tiger femur three orders of magnitude increase in deformation rate was unaccompanied by change in Work of Fracture. Our results show that the Tattersall and Tappin tests indeed produce toughness values which can be taken to be a deformation rate insensitive 'material property' *only if* the test can be made to work non-catastrophically. However, despite the unchanging mean Work of Fracture with deformation rate seen in the bovine and tiger specimens, Table 2 suggest that the two more highly mineralised bones, bovine and tiger are in fact deformation-rate sensitive. In a more fine-grained experiment, the bovine specimens would presumably start to undergo catastrophic failures somewhere between 8×10^{-4} and $8 \times 10^{-3} \text{ mm s}^{-1}$, and tiger specimens at about $8 \times 10^{-3} \text{ mm s}^{-1}$. This also suggests that most of the strain rate-dependent *increase* in strength found by Carter and others workers is in fact due to pre-failure damage widespread through the specimen.

The Narwhal tusk shows some decrease in Work of Fracture at higher deformation rates, but never becomes brittle. However, unlike the other materials, the extraordinary antler manages, at high deformation rates, greatly to increase the toughness of near-fracture-surface material by complex fracture mechanisms employing widespread interlamellar damage [8].

Perhaps is worth adding that the strain rate sensitivity we explore here, can be easily explained in non-fracture behaviour on the basis of material viscoelasticity alone. Bone tissue is viscoelastic but this is not the sole reason for strain rate sensitivity. In material science strain rate sensitivity exists even for truly elastic materials such as metals brought about by their atomic level organisation ('body centred' vs 'face centred' systems). The point the present paper is making is

that although Fracture is a contextual and circumstantial process, there may be some test, which derives a 'material property'. Where, by 'material property' we mean a value which is or more less constant and defines this material. That in itself would be valuable to know.

In conclusion the present results have shown that for most bones Work of Fracture (W_f) values seem to be a material property which is unaffected by deformation rate. This fact can be invaluable for strange bone analogues (i.e. antler and tusks) which work mostly at high strain rates and it is only in this domain that exhibits their full mechanical potential. At the high strain rate domain classical fracture toughness methodology is difficult to apply and in terms of instrumentation alone much more difficult to implement.

Acknowledgements, contributions & context

The main idea for this work belongs to Dr Andy Sedman (currently working for *DSTL* Porton Down UK) following discussion with the rest of us on the obvious shortcomings of the conventional established FT methods to quantify and demonstrate the incredible toughness of antler bone compared to this of other common bone analogues [7]. Neither K_C , nor K_R/J_R , or indeed CMOD are able to show why antler is so damage tolerant, notch insensitive and absorbs so much more energy to fracture than the rest [4,6,8,9]. However, W_f tests express these mechanical advantages clearly and they can also be applied over a wide range of strain rates, which conventional FT methods are not able to. Kevin Brear took it upon himself to carry out the tests using a range of bones, which over some time created the report presented here. Early results were discussed in an ESB congress in 1996 [44] and contextualised in two review articles that followed [9,45]. John Currey wrote most of the present materials, methods and results by giving it his unique flavour and commenting on what he always considered crucial in bone tissue in nature, namely the effect and influence of the mineral content in determining properties and function. Peter Zioupos acted as a curator of the work and contextualised some of it in introduction and discussion in view of recent developments in basic bone biomechanics.

References

- [1] Currey, J.D. The effect of porosity and mineral content on the Young's modulus of compact bone. *Journal of Biomechanics* 21: 131-9, 1988.
- [2] Currey JD. What determines the bending strength of compact bone? *J Exp Biol.* 202: 2495-503, 1999.
- [3] Currey, J.D. Physical characteristics affecting the tensile failure properties of compact bone. *Journal of Biomechanics* 23: 837-44, 1990.
- [4] Currey JD, Brear K, Zioupos P. Notch sensitivity of mineralised tissues in impact, *Proceedings of the Royal Society of London. – B*, 271 (1538): 517-22, 2004.
- [5] Zioupos, P., Currey, J.D., Casinos, A. and De Buffrénil, V. Mechanical properties of the rostrum of the whale *Mesoplodon densirostris*, a remarkably dense bony tissue. *Journal of zoology, London.* 241: 725-37, 1997.
- [6] Currey, J.D. Strain rate dependence of the mechanical properties of reindeer antler and the cumulative damage model of bone fracture. *Journal of Biomechanics* 22: 469-75, 1989.
- [7] Sedman A. Mechanical failure of bone and antler: the accumulation of damage. PhD dissertation, Dept. Biology, Univ. of York, Sept 1993.
- [8] Zioupos, P., Currey, J.D. and Sedman, A.J. An examination of the micromechanics of failure of bone and antler by acoustic emission tests and laser scanning confocal microscopy. *Medical engineering and physics.* 16: 203-12, 1994.
- [9] Zioupos P. Recent developments in the study of failure of solid biomaterials and bone : 'fracture' and 'pre-fracture' toughness, *Materials Science and Engineering: C*, 6 (1): 33-40, 1998.
- [10] Currey JD. Tensile yield in compact bone is determined by strain, post-yield behaviour by mineral content. *J Biomech.* 37(4): 549-56, 2004.

- [11] McElhaney, J.H. Dynamic response of bone and muscle tissue. *Journal of Applied Physiology* 21: 1231–36, 1966.
- [12] Crowninshield, R.D., Pope, M.H. The response of compact bone in tension at various strain rates. *Annals of Biomedical Engineering* 2: 217–25, 1974.
- [13] Currey, J.D. The effects of strain rate, reconstruction and mineral content on some mechanical properties of bovine bone. *Journal of Biomechanics* 8: 81–6, 1975.
- [14] Carter, D.R., Hayes, W.C. Bone compressive strength: the influence of density and strain rate. *Science* 194: 1174–6, 1976.
- [15] Carter, D.R. and Caler, W.E. A cumulative damage model for bone fracture. *Journal of orthopaedic Research* 3: 84-90, 1985.
- [16] Hansen U, Zioupos P, Simpson R, Currey J & Hynd D. The effect of strain rate on the mechanical properties of human cortical bone, *Journal of Biomechanical Engineering*, 130 (1) 2008. No. 011011 - 1
- [17] Behiri, J.C. and Bonfield, W. Crack velocity dependence of longitudinal fracture in bone. *Journal of materials Science* 15: 1841-9, 1980.
- [18] Behiri, J.C. and Bonfield, W. Fracture mechanics of cortical bone. In: *Biomechanics: Principles and applications*. Huiskes, R., Van Campen, D. and De Wijn, (Eds). J. Martinus Nijhoff, The Hague. 1982, pp 247-51.
- [19] Zioupos P, Hansen U & Currey JD. Microcracking damage and the fracture process in relation to strain rate in human cortical bone tensile failure, *Journal of Biomechanics*, 41 (14): 2932-9, 2008.
- [20] Kirchner, H. Ductility and brittleness of bone. *International J. Fracture*, 139: 509–16, 2006.
- [21] Peterlik, H, Roschger, P, Klaushofer, K., Fratzl, P. From brittle to ductile fracture of bone. *Nature Materials*, 5: 52–5, 2006.
- [22] Ural A, Zioupos P, Buchanan D & Vashishth D. The effect of strain rate on fracture toughness of human cortical bone: a finite element study, *Journal of the Mechanical Behavior of Biomedical Materials*, 4 (7): 1021-32, 2011.
- [23] Ural A, Zioupos P, Buchanan D & Vashishth D. Evaluation of the influence of strain rate on Colles' fracture load, *Journal of Biomechanics*, 45 (10): 1854-57, 2012.
- [24] Rho JY, Kuhn-Spearing L & Zioupos P. Mechanical properties and the hierarchical structure of bone, *Medical Engineering & Physics*, 20 (2): 92-102, 1998.
- [25] Zioupos P, Kaffy, C & Currey JD. Tissue heterogeneity, composite architecture and fractal dimension effects in the fracture of ageing human bone, *International Journal of Fracture*, 139 (3-4): 407-24, 2006.
- [26] Zioupos P, Gresle M & Winwood K. Fatigue strength of human cortical bone: Age, physical, and material heterogeneity effects, *Journal of Biomedical Materials Research Part A*, 86 (3): 627-36, 2008.
- [27] Tattersall, H.G. & Tappin, G. The work of fracture and its measurement in metals, ceramics and other materials. *Journal of materials Science* 1: 296-301, 1966.
- [28] Alman, B. and Frasca, P. Fracture failure mechanisms in patients with osteogenesis imperfecta. *Journal of orthopaedic Research* 5: 139-43, 1987.
- [29] Brear, K., Currey, J.D., Pond, C.M. and Ramsay, M.A. The mechanical properties of the dentin and cement of the tusk of the narwhal *Monodon monoceros* compared with those of other mineralised tissues. *Archives of oral Biology* 35: 615-21, 1990.
- [30] Moyle, D.D. and Bowden, R.W. Fracture of human femoral bone. *Journal of Biomechanics* 17: 203-13, 1984.
- [31] Rasmussen, S.T. and Patchin, R.E. Fracture properties of human enamel and dentin in an aqueous environment. *Journal of dental Research* 63: 1362-8, 1984.
- [32] Rogers, L.L. and Moyle, D.D. Effect of specimen size on work-of-fracture measurements. *Journal of Biomechanics* 21: 919-26, 1988.

- [33] Yan, J, Clifton, K. Mecholsky, J, Reep LR. Fracture toughness of manatee rib and bovine femur using a Chevron-notched beam test. *Journal of Biomechanics*. 39: 1066-74, 2006.
- [34] Sharma, NK. Pal, R. Sehgal, DK, Pandey RL. Application of Elastic-Plastic Fracture Mechanics to Determine the Locational Variation in Fracture Properties of Cortical Bone. *Materials Performance and Characterization*. 3: 429-47, 2014.
- [35] Chittibabu, V., Santa Rao, K., and Govinda Rao, P. Factors affecting the mechanical properties of compact bone and miniature specimen test techniques: a review. *Advances in Science and Technology Research Journal*, 10: 169-83, 2016.
- [36] Ast, J. Ghidelli, M. Durst, K. Göken, M. Sebastiani, M. Korsunsk. AM. A review of experimental approaches to fracture toughness evaluation at the micro-scale, *Materials & Design*, 173: 107762, 2019.
- [37] Zioupos P & Currey JD. Changes in the stiffness, strength, and toughness of human cortical bone with age. *Bone*, 22 (1): 57-66, 1998.
- [38] Zioupos P, Currey JD & Hamer AJ The role of collagen in the declining mechanical properties of aging human cortical bone. *J Biomedical Materials Research Part A*, 45 (2): 108-16, 1999.
- [39] Fantner GE1, Hassenkam T, Kindt JH, Weaver JC, Birkedal H, Pechenik L, Cutroni JA, Cidade GA, Stucky GD, Morse DE, Hansma PK. Sacrificial bonds and hidden length dissipate energy as mineralized fibrils separate during bone fracture. *Nature Materials*. 4(8): 612-6, 2005.
- [40] Currey, J.D., Brear, K. and Zioupos, P. Dependence of mechanical properties on fibre angle in narwhal tusk, a highly oriented biological composite. *Journal of Biomechanics* 27: 885-97, 1994.
- [41] Sarkar, B.C. & Chauhan, U.P. A New Method for Determining Micro Quantities of Calcium in Biological Materials. *Analytical Biochemistry*, 20: 155-166, 1967.
- [42] Zioupos P & Currey JD. Pre-failure toughening mechanisms in the dentine of the narwhal tusk: Microscopic examination of stress/strain induced microcracking, *J Materials Science Letters*, 15 (11): 991-94, 1996.
- [43] Zioupos P, Currey JD & Casinos A. Exploring the effects of hypermineralisation in bone tissue by using an extreme biological example., *Connective Tissue Research*, 41 (3): 229-48, 2000.
- [44] Currey JD, Brear K, Zioupos P. Strain rate dependence of Work-of-Fracture tests on bone. 10th Conf. Eur Soc Biomech, Leuven, Belgium Aug 28-31, 1996.
- [45] Zioupos P & Gupta H. Fracture of Bone Tissue: the 'hows' and the 'whys', *Medical Engineering and Physics*, 30 (10): 1209-26, 2008.

Head Speed mm/s	BOVINE			TIGER			NARWHAL			DEER		
	E	Calcium	Wf	E	Calcium	Wf	E	Calcium	Wf	E	Calcium	Wf
8 x 10 ⁻⁴	17.2	261.8	1130	14.1	242.5	3000	12.4	244.1	12659	10.6	230.7	4140
	19.7	252.7	2100	15.7	244.3	4420	11.5	236.7	4940	10.2	229.6	4290
	22.6	256.7	3310	16.3	241.3	5470	11.3	233.9	9840	11.2	225.5	3770
	19.0	261.0	3950	16.3	242.8	6050				9.4	228.8	2850
	15.4	259.7	2450	12.2	242.0	4500						
	13.7	262.0	870									
8 x 10 ⁻³	19.2	258.9	1840				11.2	243.7	7801	9.3	228.1	5120
	20.8	257.6	720				11.5	238.7	10112	11.2	233.0	6210
	11.4	256.0	2330				11.9	247.5	7592	9.1	230.0	5200
	21.0	263.3	1830									
	18.7	239.3	2000									
8 x 10 ⁻²	16.1	233.7	2401	13.7	238.7	4940	13.0	243.5	11027	9.8	231.7	5920
	16.0	246.8	3340	15.0	243.3	4520	11.1	244.0	9152	9.3	232.7	5350
	17.5	271.1	1042	17.0	247.8	5010	10.8	241.6	4777	8.5	233.6	6670
	17.5	242.6	2000							8.8	233.4	5720
8 x 10 ⁻¹				17.6	240.8	6360	10.7	240.1	4742	8.7	228.0	9170
				16.0	238.9	4521	11.1	234.2	3299	7.5	223.0	8180
				17.3	233.9	4327	11.9	237.4	7965	9.6	235.0	12000
										11.3	217.0	13440
									10.7	232.0	11360	
3.3							12.2	246.7	8286	10.0	227.9	14960
							13.8	238.8	2907	11.1	224.5	17980
							11.4	241.2	2526	9.8	238.3	11130
Impact	21.6	270.7	1657	14.9	246.6	6276	6.8	227.6	24079	9.3	223.6	44026
	22.1	269.7	1555	15.6	245.4	4714	7.1	231.6	36287	8.7	223.8	41751
	22.6	269.3	1362	14.9	247.7	5260	7.2	229.1	32152	10.1	223.3	46664
				14.3	252.9	4299				9.3	219.9	52479

Table 1: Dataset for Wf, Calcium content and Young's modulus.

Crosshead speed (mm s⁻¹)					
Tissue	8.3×10^{-4}	8.3×10^{-3}	8.3×10^{-2}	8.3×10^{-1}	3.3
Bovine	6/6	5/10	4/10	-	-
Tiger	5/5	3/4	3/14	0/5	
Narwhal	3/3	3/3	3/3	3/3	3/3
Antler	4/4	3/3	4/4	5/5	3/3

Table 2 Fraction of samples that showed ductile controllable fractures for the four different tissues at different head speeds: (successful tests/total tests)

Comparison	Degrees of Freedom	F	P
Young's modulus			
Bovine	12,2	0.27	0.771
Tiger	8,2	1.89	0.213
Narwhal	10,4	0.87	0.515
Antler	15,4	0.84	0.521
Calcium			
Bovine	12,2	1.34	0.298
Tiger	8,2	3.10	0.101
Dentine	10,4	1.28	0.312
Antler	15,4	0.85	0.515
Work of fracture			
Bovine	12,2	0.47	0.633
Tiger	8,2	0.14	0.874
Dentine	10,4	1.46	0.284
Antler	15,4	24.88	<0.001

Table 3 Analysis of variance of three properties of the specimens (Young's modulus, calcium and Work of Fracture) with respect to deformation rate.

Log Wf = 4.03 + 0.156 log crosshead. $t = 10.16$ P<0.0001; $R^2 = 85.0\%$	(1)
Log Wf = 2.73 + 1.63 log crosshead + 1.32 log E $t = 15.21$, P<0.0001; $t = 4.44$, P < 0.0001; $R^2 = 92.9\%$	(2)

Table 4. Relationships in antler between the crosshead speed, Young's modulus and Work of fracture.

FIGURE CAPTIONS

Figure 1. Back scattered SEM micrographs of (a) Narwhal tusk dentine. Note that in this magnification the material looks amorphous, no discernible features exist, this is because it comprises of mineralised fibrils which do not form motifs like lamellae or osteons; these would have produced a mosaic of bone tissue compartments familiar to us from human bone histology. (b) Bovine Femur – The tissue is laminar and the lamellae are circumferential around the cortex of this long bone. (c) Red Deer antler – Fully osteonal it possesses no lamellar bone throughout its life from inception. The osteons are primary, not a result of remodelling, and consist of lamellae which are loosely bound to each other, so during fracture they detach [8] and absorb energy in a telescopic manner. (d) Human femoral bone of a 56M from a previous study [37] added in for illustration of the difference in structure – A mosaic of different compartments of varying tissue ages, old and new and with remnants of circumferential lamellae in between. New secondary osteons are darker in this micrographs because they are relatively less mineralised than the older tissue material. Scale bar size common for all four micrographs.

Figure 2. Samples were produced in the form of beams (35x4x2 mm) and notched in a chevron pattern as for the Tattersall & Tappin test configuration (shown here for antler). The triangular notch helps in stabilising the fracture even in semi-brittle materials because the advancing fracture front meets with an increasing width of ligament as it moves through the tissue.

Figure 3. Work of Fracture (W_f) values vs. loading rate (mean \pm SD, note logged scales). Extra data points have been added from later work for elephant tusk (dentinous tissue similar to Narwhal tusk) and human femoral bone [45]. Common long bone tissue (bovine, tiger, human) shows broadly the same behaviour with W_f values staying relatively constant across the range of speeds. Dentine tissue shows itself to be much stronger in impact but not in lower rates. Antler bone is the only conventionally designed bone which is remarkably tough throughout the range. The percentages illustrate the fraction of specimens which fractured in stable fashion before the crack growth became unstable (Ductile to brittle transition) The common femoral bones fractured in a stable manner for up to running speeds (loading rates $>0.008\text{ s}^{-1}$ ~ strain rate $>0.1\text{ s}^{-1}$).

Figure 4: (a) Relationship between Work of Fracture (J m^{-2}) and calcium content (mg g^{-1}). Note log scales. Linear regression on logged properties is highly significant ($P < 0.001$) but weak ($R^2 = 40\%$). The linear regression line, with its 95% confidence intervals, is shown. (b) Relationship between Work of Fracture (J m^{-2}) and Young's modulus (GPa). Note log scales. Linear regression on logged properties is highly significant ($P < 0.001$) but weak ($R^2 = 32\%$). The linear regression line, with its 95% confidence intervals, is shown.

Figure 5. Relationship between Impact strength and Calcium (log scales). Linear regression with 95% confidence interval.

Figure 6. (a) the three main ingredients of bone composition in a ternary diagram. For bone tissues across nature the course of increased mineralisation is clearly prescribed along a trajectory (arrow), modified from [43]; (b) moving along the same mineralisation trajectory has two main consequences: an increase in both stiffness and hardness.

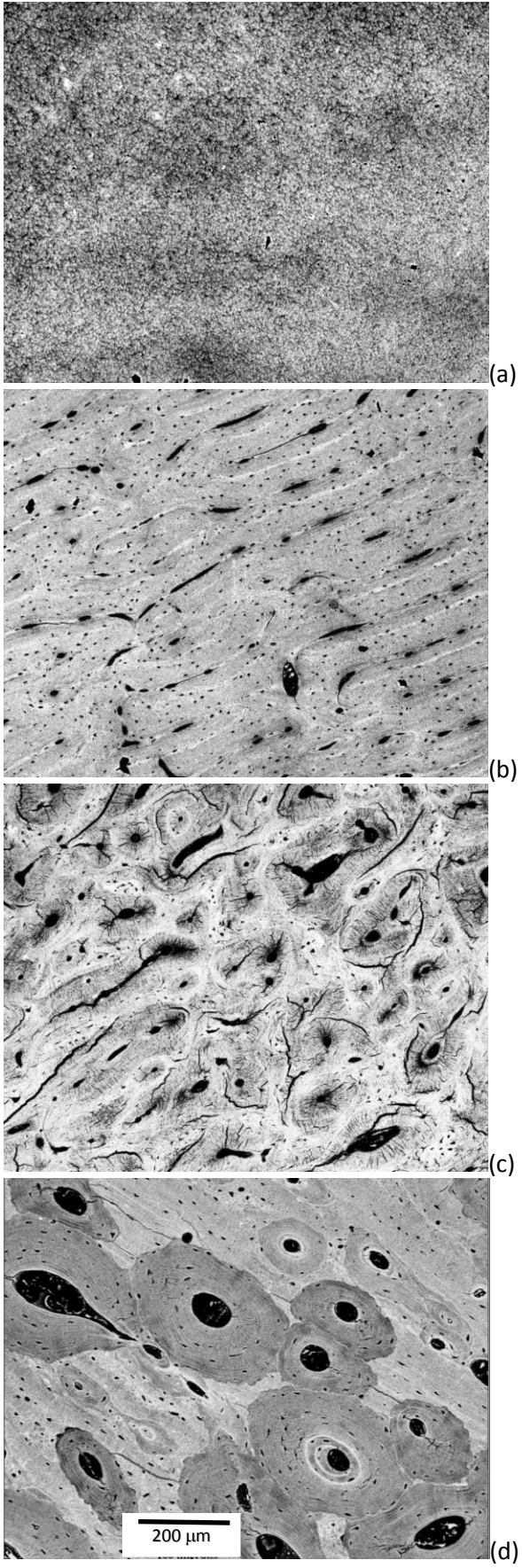


Figure 1.

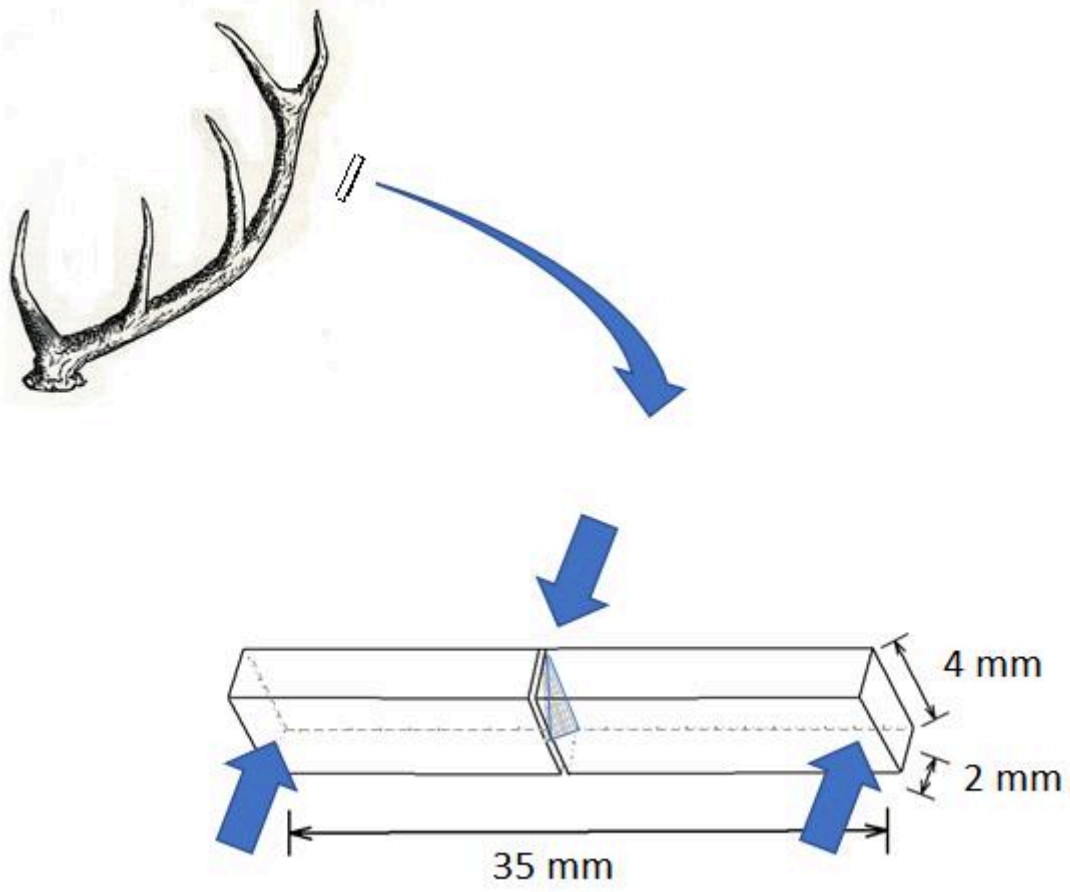


Figure 2.

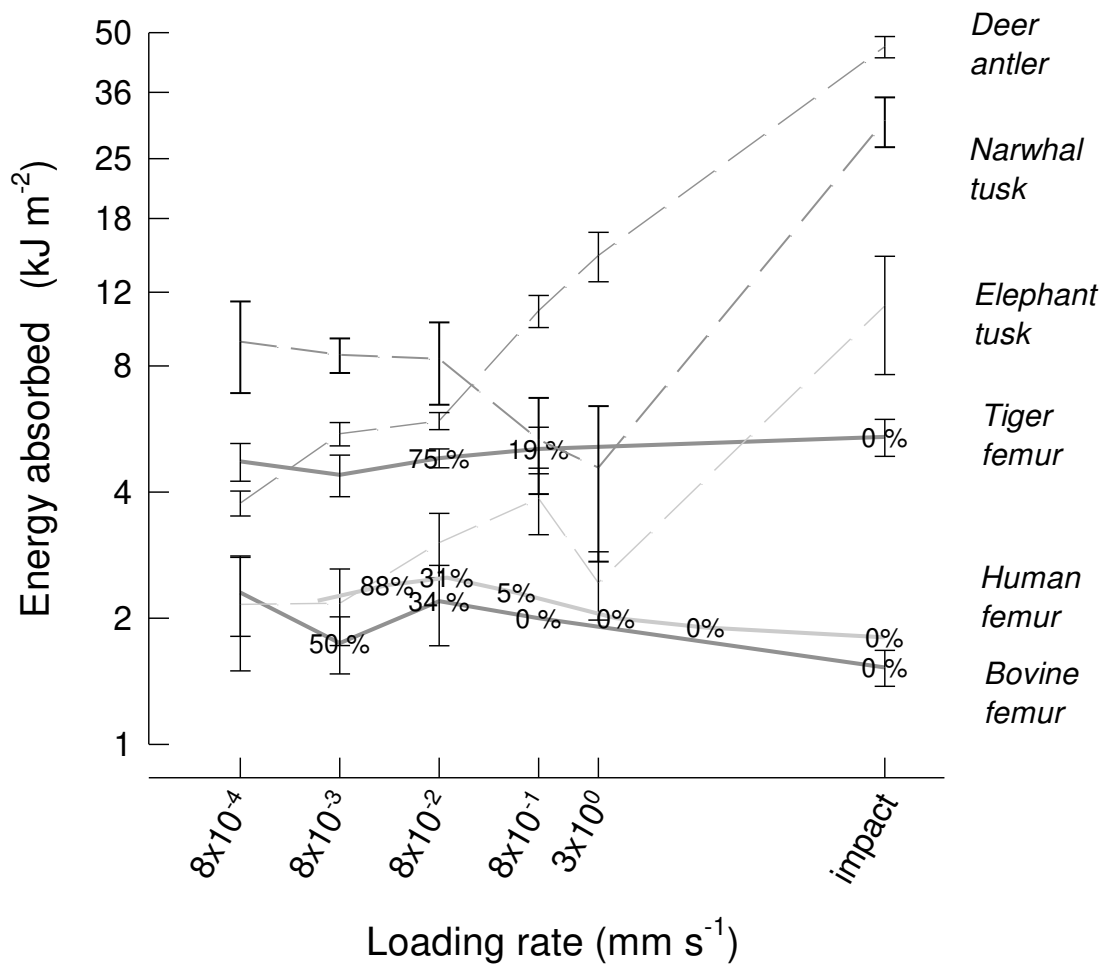


Figure 3.

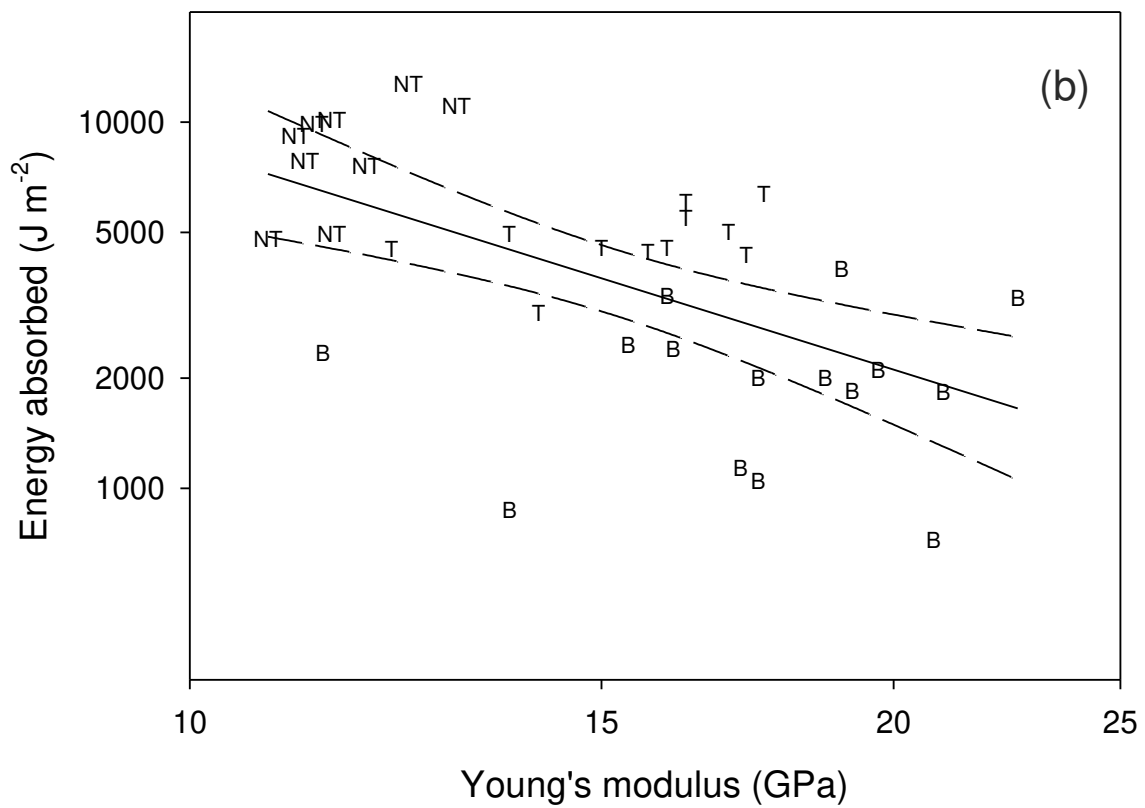
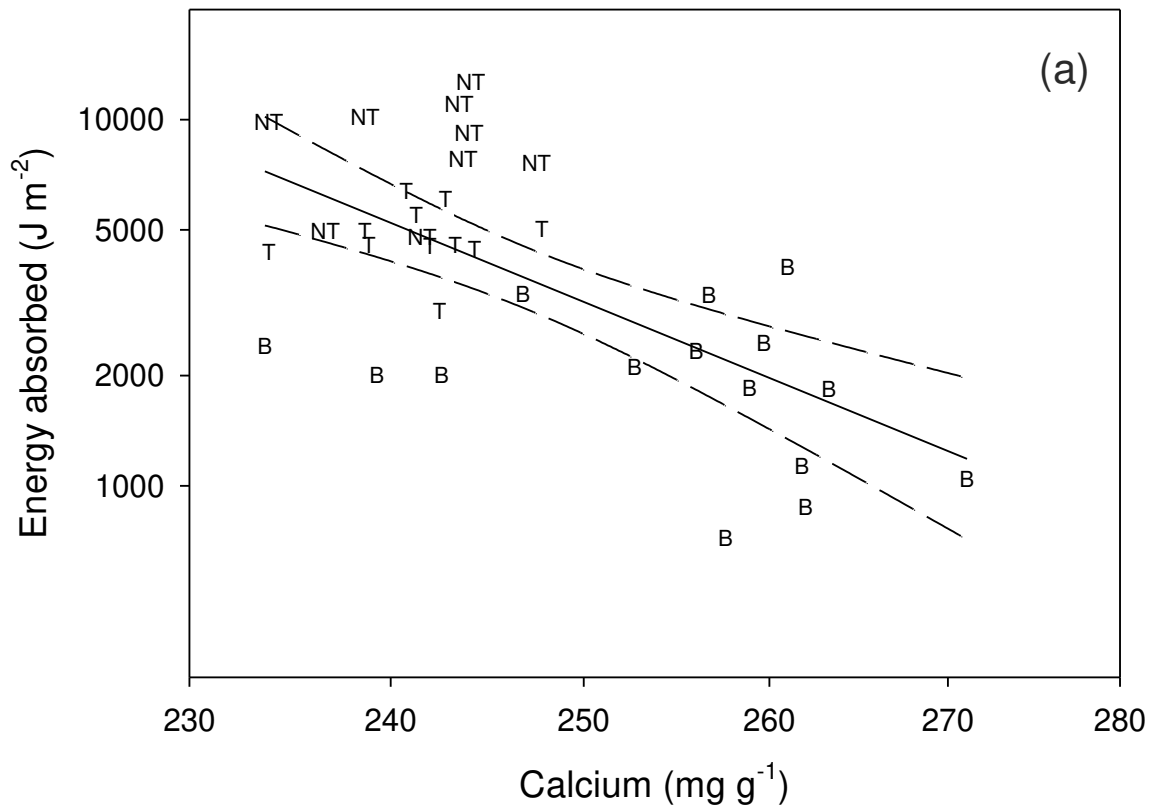


Figure 4.

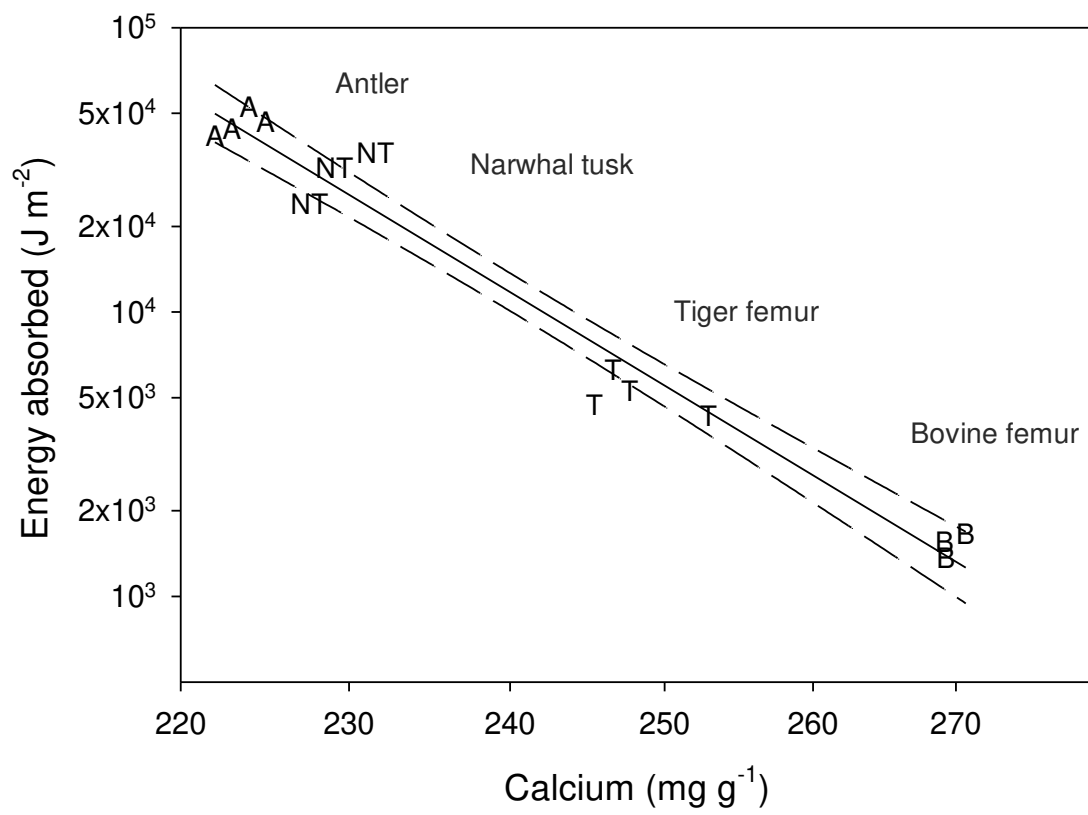


Figure 5.

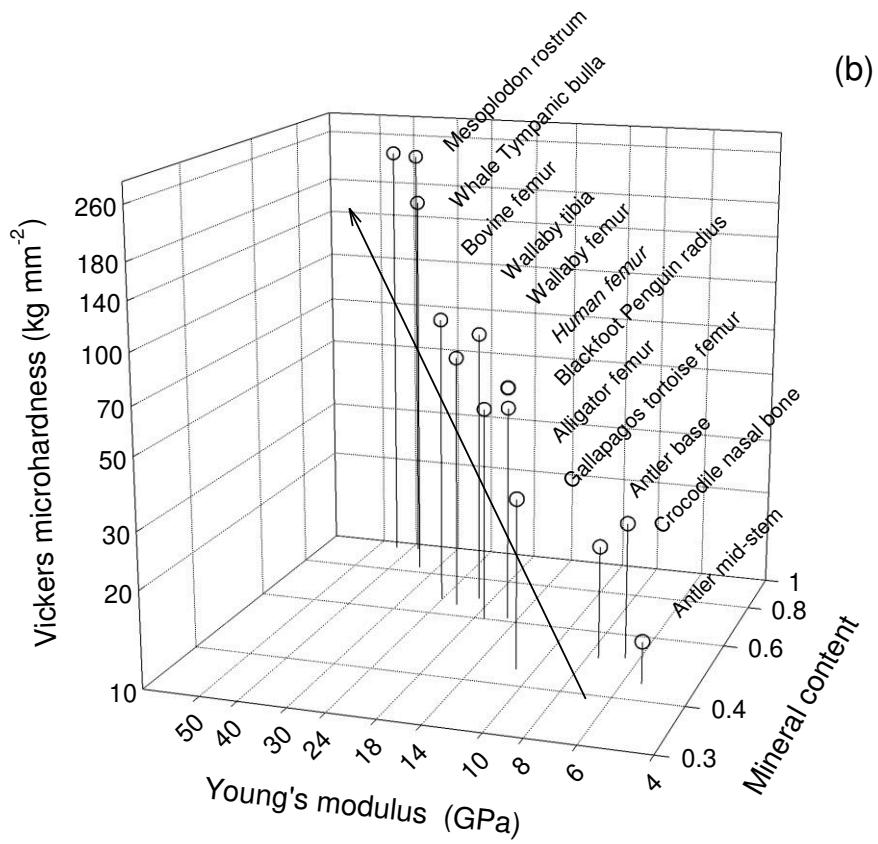
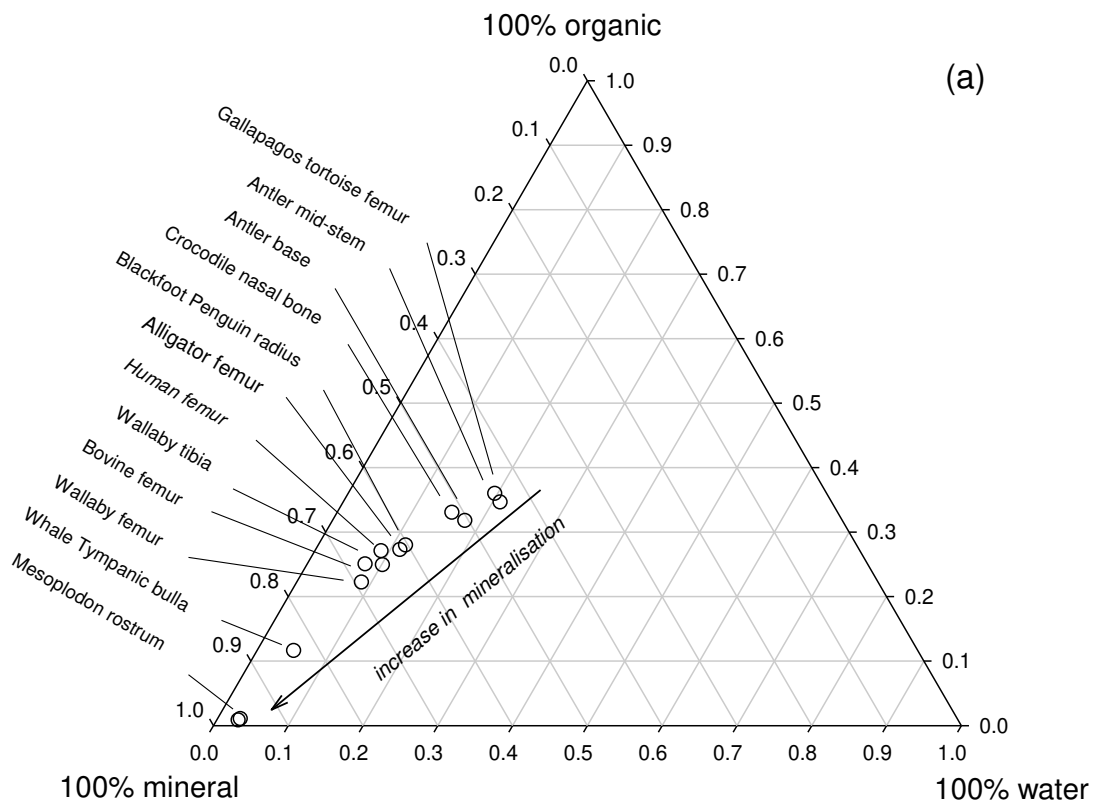


Figure 6.

Strain rate dependence of work of fracture tests on bone and similar tissues: reflections on testing methods and mineral content effects

Currey, John D.

2019-08-22

Attribution-NonCommercial-NoDerivatives 4.0 International

Currey JD, Brear K, Zioupos P. (2019) Strain rate dependence of work of fracture tests on bone and similar tissues: reflections on testing methods and mineral content effects. *Bone*, Volume 128, November 2019, Article number 115038

<https://doi.org/10.1016/j.bone.2019.115038>

Downloaded from CERES Research Repository, Cranfield University