Materials Science and Engineering: C. Volume 6, Issue 1, September 1998, pp. 33-40
DOI: 10.1016/S0928-4931(98)00033-2

Recent developments in the study of failure of solid biomaterials and bone: 'fracture' and 'pre-fracture' toughness.

P. Zioupos,

Department of Materials & Medical Sciences, Cranfield University, Shrivenham, SN6 8LA, UK

Abstract[1]

Our understanding of the stress and energy requirements for the fracture of solid bio(logical)-materials has improved recently as a result of a number of investigations which highlighted some important new aspects of the failure process. The present short review article shows that under normal circumstances structural 'damage' precedes the creation of a fatal macrocrack and the onset of 'failure'. The externally perceived material 'toughness' is a function of both: i)the degree of prefailure accumulation of damage and ii)of the properties of the final fracture surface. These two situations can be distinguished as 'pre-fracture' toughness and 'fracture toughness' and the relative contribution of each one in the overall toughness of the tissue is crucial for the final outcome of the failure process. Inevitably materials which show remarkable toughness have to enhance their performance in respect to both quantities.

Keywords: biomaterial(s), bone(s), failure, damage, fracture, toughness.

1. Introduction

The mechanical performance of biological tissues has attracted continuous attention over the years. There is a general interest to know how tissues transfer forces and moments, how they self repair (homeostatic process), how far they are from failure in life and under various loading conditions (safety factors) and how they avoid failure if and when they do. There is also more specific need to know the mechanical soundness of tissues in some

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1 ***Corresponding author***: Dr P. Zioupos, Department of Materials & Medical Sciences, Cranfield University, RMCS Shrivenham, SN6 8LA, UK; tel:+44(0)1793-785932; fax:+44(0)1793-785772; email:zioupos@rmcs.cranfield.ac.uk

situations where they are used as biomaterials (e.g. bone and heart valve tissue for tissue banks) or after human intervention inside the body (near prostheses). More recently biomimetic engineers have focused their attention on the remarkable properties and architectural features of natural biological material in order to achieve a better understanding and ultimately the ability to replicate them.

In-vivo, the toughness of a tissue is of vital importance for the survival of the organ or the organism. However, the measurement of toughness *in-vitro*, in the laboratory, is not always straightforward. Researchers usually focus their attention on the properties of the final fracture surface. This is reasonable for 3-dimensional solid materials, but it poses problems when semi-solid, one or two dimensional tissues are examined. In softer tissues, in particular, we also encounter a different kind of material failure caused mostly by a disintegration of the structure where the final 'fracture surface': a)is difficult to define and b)is accompanied by extensive collateral damage. The externally perceived 'toughness' is then a factor of three phases as seen in Fig. 1. In phases I-II, the elastic-continuum damage mechanics (ECDM), toughness is enhanced by producing diffuse damage at the expense of stiffness and residual strength. In phase III, the fracture mechanics (FM) realm, energy is absorbed at and next to the final fracture surface; the amount of energy depending crucially on the properties of the final fracture plane and the overall number of such planes and/or fragments.

1.1 Variety in fracture mechanics (FM) approaches

FM experimenters usually have two questions to consider. The first is a question of *method*: the so called stress vs. energy approach. Stress based criteria, such as the stress intensity factor (K_c) postulate that fracture is initiated when the concentration of stress at the crack tip reaches a critical value. Energy based approaches, which either measure the critical strain energy release rate G_c (or J, for non-linear effects) or the work to fracture of a specimen W_f, determine critical levels of energy per unit area necessary for fracture. In this field it has become quite clear recently that modern composites (and for that matter bone[1] and other biological hard tissues) show weak interlamellar interfaces, which are able to absorb energy and/or divert a crack and in this way deter the onset of fracture. The second is a question of *emphasis*: the importance of initiation vs. propagation of the crack. Brittle single phase homogeneous materials find it difficult to slow down and divert a crack once it is started. However, most biological materials utilise a number of tricks commonly seen in modern composites like crack diversion/deflection, fibre pull-out, crack and/or matrix bridging [2,3] and by these means they increase the required amount of energy to fracture. Increasingly, nowadays, emphasis is placed in studying the route of propagation of major cracks [4,5].

1.2 The damage mechanics (DM) approach

Unlike fracture mechanics, which considers the effects of a single dominant defect, damage mechanics considers the presence and effects of a large number of microdefects distributed widely and randomly throughout the structure [6]. In engineering materials manufacturing microdefects can be dislocations, pores or decohesions. Newly created imperfections may further modify the mechanical behaviour of a material. In biological materials, *a priori*, natural homeostatic and repair processes usually take care of 'microdefects'. Nevertheless, natural materials contain a multitude of microarchitectural patterns and local inhomogeneities which can act as 'engineering microdefects'.

A better understanding of the mechanics of a material may be obtained if one considers carefully the contribution of a few primary deformation (i.e. elastic, plastic or brittle) modes [7]. *Elastic* deformation is a process in which no new 'microdefects' are nucleated while all existing 'microdefects' translate/rotate with the surrounding mass without growing in size and without changing the residual properties of the material. *Plastic* deformation consists of an irreversible re-arrangement of the microstructure during which the material flows primarily by dislocation motions and in which the total number of atomic bonds remains constant. In plasticity the stiffness and the unloading trace of the stress/strain curves remain unaltered and there is a residual irrecoverable strain as the material settles in some new dimensions. By contrast during *brittle* deformation points of cohesion of the microstructure (atomic bonds, matrix elements) break, the material stiffness itself changes, but there is no flow of the lattice of the material as such and, therefore, no plastic strain. A perfectly brittle material fails as soon as a few microcracks appear. A *quasi-brittle* material will sustain a greater number of cracks in a distributed manner and it will externally/phenomenologically 'flow' as if it were capable of plastic deformation. However, there is no residual plastic strain associated with brittle deformation and in this case the relative reduction in the material stiffness is the only reliable means of quantifying the effects of damage.

In this manner, significant progress has been made lately in the field of bone-mechanics [8,9]. Undamaged bone (a mineral filled polymer matrix material) shows viscoelastic behaviour [10] at low levels of stress (below 20 MPa in quasi-static loading, [11]) or strain.

The viscous contribution of the organic matrix and collagen is present in both damaged and undamaged bone and in fact becomes more pronounced in the damaged (microcracked) tissue [12-14]. The bone structure has a number of sites where under the application of a damaging level of stress the material 'gives in' and develops 'fresh' microcracks [9,15]. Damaged bone shows phenomenologically a non-linear behaviour, some kind of flow [16] and looses some of its stiffness and strength [17]. When looked at in terms of its elastic, viscous, plastic and brittle behaviours [9,16] bone shows initially a (visco-) elastic, later quasi-brittle (elasto-damaged) behaviour with little evidence of plasticity (permanent flow) as such. Studies examining the reduction in stiffness of damaged bone [12-14] and tendons [18-20] on their way to fracture help in understanding this kind of 'gradual failure process' and have established DM alongside FM in the characterisation of materials failure.

The present article reviews some recent studies which examine the failure of a solid biomaterial as a function of both: i) the degree of prefailure accumulation of damage and ii) of the properties of the final fracture surface. In the discussion some commonly used materials science terms are redefined in accordance with the previous arguments. The term 'ductile' is used to denote a gradual fracture process as it applies for quasi-brittle (elasto-damaged) materials. The term 'brittle' is used for sudden, abrupt and catastrophic events.

2. Recent Experiments

2.1 The K_R principle

The stress based FM answer for the increased toughness of some biological materials (like deer antler [21]) is the introduction of the crack growth resistance curves K_R. It consists of measuring the critical stress intensity factor not only at the start, when the macrocrack is initiated, but afterwards as it makes its way through the material. In brittle materials a K_R curve is flat, K is constant and, therefore, there is little to deter the crack in its growth. In tough solids, especially by microcracking at the crack tip, K_R increases with the crack length, the crack finding considerable resistance to its growth.

Vashishth *et al.*[21] prepared 15 compact tension specimens machined wet from the antlers of red deer. Crack propagation gauges were attached onto the specimens to monitor the crack length while testing. Five specimens were kept as controls for microscopic observations, five were used to study K_R behaviour and five just to determine the K_c initiation values. By use of Scanning Electron Microscopy (SEM) the number of microcracks viewed from the surface of lengths between 100-250 μm were counted in all control and experimental compact tension specimens.

These workers found that K_R increased linearly with crack length, a 20% increase in the length of the crack nearly doubled the required stress intensity factor (Fig. 2). The authors observed that the number of microcracks were in accord with the results on fracture toughness. Many more microcracks were present in their fracture propagation (K_R) specimens than in the fracture initiation (K_c) ones. Microcracks were seen both ahead and behind the tip of the propagating macrocrack. The authors explained the increase toughness of antler bone by the nucleation, growth and coalescence of the observed microcracks, which were responsible for the stable progress of fracture by absorbing energy away from the main crack itself.

2.2 K_c and J integral during initiation

The *J* integral is the non-linear equivalent to the strain energy release rate *G* in cases where plasticity or events resembling 'plasticity' (i.e. microcracking) at the crack tip absorb some energy which is in addition to the energy that is used to create the new fracture surfaces. Kasapi and Gosline [22] examined the fracture toughness of the material of the equine hoof wall, a very tough and a wear resistant tissue. The authors examined the behaviour of both *K* and *J* values required to initiate the fracture with the additional element of looking into strain rate dependent effects. The *J* integral proved to have remarkably high values (10-15 kJ m^{-2}) and was constant over a strain range spanning 5 orders of magnitude. This indicated that the *J* values were something of a material property, somewhat independent of the testing conditions. By contrast, the initiation stress intensity factor *K* showed an increase, doubling its value over the same strain rate range (Fig. 3). This study was concerned with the initiation of fracture and *K* reflected in essence the behaviour of the maximum attainable critical load *P*.

In a second paper Kasapi and Gosline [23] considered both the directionality in the architecture of the tissue and the level of hydration of the keratin which both changed with the region. These workers showed that a combination of hierarchy, structural organisation, constitution, level of hydration and constituent material (keratin) properties can work synergetically to produce a tougher material. Viscoelasticity appeared to help toughening in particular in relation to strain rate effects and this reinforces earlier studies on the toughness of bovine horn material [24] which has been shown to be much tougher when wet aided by the increased ductility of the interfibrillar matrix.

2.3 Strain energy release rate in propagation

Peterlik *et al.*[25] examined the behaviour of bovine cortical bone during the *propagation* of a crack along its three principal architectural directions (longitudinal, radial, tangential). Fourteen specimens for fracture toughness determination in a 3-point bending configuration were prepared in total. The strain energy release rate was measured by loading/unloading and reloading the same specimen which allowed characterisation of the travelling crack mode and the amount of energy put into the fracture process. This study demonstrated *directional* effects in the toughness of bone. The fracture process dependent on the direction of travel of the crack, being either brittle (in the longitudinal direction) or deflected (in the tangential direction) or toughened by microcracking (in the radial direction).

The previous qualitative outcome reflected clearly in the graphs showing the strain energy release rate as a function of crack length. Brittle fracture showed that the energy required was constant, deflected fracture showed the energy required to increase only moderately, while the fracture accompanied by microcracking showed an impressive increase in the magnitude of the energy absorbed with the current crack length (Fig. 4).

These results provide evidence of an energy based understanding of a self toughening (crack growth resistant) fracture process (during propagation) similar to the one described for antler bone [21]. However, the energy based approach of Peterlik *et al.*[25] has certain conceptual and practical advantages over the previous stress related one. Firstly, the question is toughness and consequently the capacity to absorb *energy* is the main issue. Secondly, the events at the crack tip which give rise to the stress field and are expressed by the stress intensity factor are mostly influenced by the elastic condition of the material (stiffness, viscoelastic nature) and much less by the deterioration of it (i.e. as by microcracking). Microcracking was shown in this way to constitute an important toughening mechanism.

2.4 Slow/fast crack growth in fatigue

Fractography is a useful tool for engineers keen to unravel the mysteries of failure because the fracture surface provides a time record of the complete fracture process. However, fractography is always open to interpretation. Fracture surfaces of bone, for instance, invariably show some areas of considerable roughness and others which are considerably flat and smooth. In the past this phenomenon was [1] attributed to a ductile/brittle transition of the fracture whereby the architecture had a dominant role and perhaps influenced this so called transition.

Recent fractographic examination of surfaces of human specimens of two different ages broken in zero to tension fatigue showed similar effects [13]. At a first glance the test showed that i) rough ductile fractures were more common in the older human bone tissue; and ii) they were more frequent in the endosteal side of the tissue than in periosteal side of it. Careful observation of the fractured elements, however, revealed that while there were osteons that fractured fully either in a brittle or in a ductile ('telescopic') mode, there were also a few osteons which showed both types of behaviour separated halfway by a sharp delimiting ridge (Fig. 5). These osteons serve as an example to indicate that the ductile & brittle behaviours are only the result of the imposed conditions and not some inherent property related to the osteon structure itself. In other words the average osteon seems capable of undergoing a ductile energy absorbing fracture under certain conditions, but it also fractures in a brittle fashion when there is a runaway crack in advance.

The toughening contribution of slow fracturing ductile osteons and their energy absorbing capacity were demonstrated in the study of Stover *et al.*[26]. These workers loaded sample beams taken from equine bones (in pairs) in either simple monotonic loading to failure or fatigue cycling to failure in a 4-point bending mode. Notably *only* the fatigue-cycled specimen surfaces exhibited areas of the so called 'osteonal pull out' effect and not the monotonically loaded ones. They also observed that the osteonal pull-out areas (what earlier was defined as osteons fractured in a ductile mode) was proportionally related to the fatigue lifetime (N_f number of cycles) of the specimens. Specimens with a long lifetime had a greater osteonal pull out area and also exhibited a greater loss of material stiffness from the beginning till the end of the tests. In addition to these findings they observed that there was some correlation between the initial stiffness of the beams and the osteonal pull out areas and consequently the capacity for toughness of the individual bone samples. Although the authors did not make anything of this observation, this result brings to mind the usually observed trade off between stiffness on the one hand and strength/toughness on the other that is often seen in composite materials studies.

2.5 Work of fracture versus deformation rate tests

Work of fracture measurements offer certain advantages in quantifying the fracture toughness of some composite materials [27]. They measure not simply the energy that is needed to propagate a self similar crack, but all the non-linear energy that is actually needed to produce a new fracture surface. In the method used by Tattersall and Tappin [28] a certain specimen geometry allows a stabilised fracture to be obtained for all kinds of brittle and quasi-brittle materials. It has been used successfully for bone by Moyle and Bowden [29] who also later examined how the specimen size and/or geometry may influence the results [30].

W_f measurements were used recently to demonstrate the reduced toughness of human bone material with age [31]. W_f data were obtained from specimens 4x2 mm in cross section with a chevron notch at a low cross head speed of 0.5 mm min^{-1}. The work for breaking each specimen was divided by *twice* the ligament area. Measurements for the energy absorption capacity of similar specimens at a high speed in impact were obtained by use of a Hounsfield plastics impact tester. In impact, some of the fracture energy goes into creating the fracture surfaces (proportional to the final number of fragments), some goes into collateral damage next to each surface, and some goes into kinetic energy in each of the fragments. The two data sets showed a very good correlation (Fig. 6) over a wide range of ages. The young bone in particular, which is less mineralised was especially tough in impact. Although the energy consumed in impact is overall much higher than the specific energy in W_f tests the two values increased hand in hand showing that an appreciation of toughening effects at high strain rates can result from simple studies by use of much slower fracture toughness methods.

W_f tests have also been used to examine deformation rate effects in a group of different animal bone materials [32]. Specimens were obtained from a bovine femur (*Bos taurus*, of typical plexiform architecture), from the femur of a tiger (*Panthera tigris,* of typical osteonal architecture) and from the naturally tough material [33,9] of the antlers of red deer (*Cervus elaphus*) which in life experiences increased levels of loading in impact. The tests were performed at cross-head speeds varying between 0.05 mm min^{-1} and 200 mm min^{-1} in a materials testing machine and the data was supplemented by tests in impact.

Two aspects of the materials' toughness are evident in Fig. 7. The naturally tough antler bone showed a tendency to dissipate more energy to fracture per unit area as the strain rate increased. Antler dissipated an order of magnitude more energy in impact than in quasi-static loading. However, the two quasi-brittle 'ordinary' bones (bovine and tiger) produced the same value for W_f at all deformation rates, including impact. One way of looking at this data is that the W_f test in this respect produced a material property for the 'ordinary' bones, but it showed a rate depended property for the naturally tough bone. However, one other aspect of toughness was shown by the percentages of successfully completed tests. There, once again antler bone showed no ductile to brittle transition and was able to fail non-catastrophically even in impact. On the contrary bovine bone started showing catastrophic failures at rates above 5 mm min^{-1} and the tiger bone above 50 mm min^{-1}.

The absence of a transition into a brittle behaviour and the impressive increase in the magnitude of the W_f values with the strain rate are both two very remarkable toughening aspects of this material behaviour.

2.6 Toughening by accumulation of microcracks

In the absence of any artificially induced stress concentrators, when the stress field is rather uniform, bones like bovine femur, antler material, human femur, and dentine [8,9,12-17] are capable of producing extensive and widespread microcracking. Microcracking is able to soften a tissue in terms of both strain and stiffness. This has beneficial results both *locally*, whereby tougher tissues 'yield' around deleterious cracks (and by means of microcracking blunt the sharp edges of these cracks) and also *globally*, since the ability to avoid: a) the initiation and growth of a macrocrack (fig.1, phase-II) and b) uncontrollable fracture, means that the material is still intact and able to perform its duties.

Quantification of the degree of inflicted damage can be obtained by either 'listening' to the cracks developing (acoustic emission method, [9]) or by measuring the reduction in the residual stiffness of the tissue. These methods have produced some encouraging results. In acoustic emission recordings in simple monotonic tensile loading tests antler has shown a tendency to register acoustic events (=cracks) at a rate which decreased with the applied uniaxial strain [9].

Although this observation may have been of no consequence it has been corroborated by later fatigue tests in tension [12] after monitoring the reduction in stiffness for bone and antler. By using an early DM expression [34] capable of following the progress of damage-D (percentage reduction in the elastic modulus of the material) separately as a function of:

stress (σ), effective stress (σ/1-D), and the fraction of the remaining intact cross sectional area (1-D):

$$dD/dN = \{\sigma / B(\sigma)\ (1-D)\}^{\beta}\ \{1-D\}^{-p} \qquad (1)$$

values were produced for the parameters β and p, as functions of the level of stress. This way, the rate of damage (expression 1) occurring during cycling loading was quantified from 0 at the start of the tests to its maximum value 1 at fracture of the specimens.

The exponent β, which is also the exponent of the Paris's expression in fatigue ($N_f=A\ \sigma^{-\beta}$, N_f:cycles to failure, A:power law coefficient) showed on average similar values in ordinary bone and antler with a slight tendency to increase at higher stresses. However, p, which is the exponent of the expression (1-D) had positive values in bone, but negative in antler (Fig. 8). This produces the following extraordinary toughening trick. Imagine that damage increases from 0 towards the value of 1. Expression (1) shows that as the level of incipient damage increased during fatigue the rate of damage accelerated in bone (p>0) and slowed down in antler (p<0). This result, when considering the implications for failure, is the same as in monotonic loading [9]: an altogether more stabilised fracture process for the antler material.

3. Concluding remarks.

This review of recent experiments shows that we are gradually obtaining a better understanding of the fracture process for a number of solid biomaterials. All these biomaterials/tissues are made of metabolically cheap elements and, therefore, their impressive toughness properties or even the manner of their failure is a very intriguing phenomenon.

If we were to summarise the fracture process and the necessary conditions to initiate and propagate failure we could say that tough biological composites have the following 'behavioural' patterns (Fig. 9):

i) the material must be capable of achieving a great degree of damage by generating energy absorbing microdefects. These have to be able to disperse and also not-interact with each other. The material benefits if the defects are generated in many hierarchical levels and come to a halt each time they hit a different kind of a barrier. This as a whole will probably point towards a condition where the rate in which additional damage (or number of defects) is generated slows down with the total amount of damage already present in the structure (section 2.6).

ii) when a major macrocrack is initiated, it is advantageous if it can be slowed down as it goes through the structure and stay in slow growth as long as possible (section 2.4). This can be helped by either requiring the necessary stress intensity factor (section 2.1), or the energy needed for the growth of the crack (sections 2.2, 2.3) to increase with the crack length.

iii) in the unfortunate case that the crack shows a tendency to accelerate it is helpful that the energy associated with producing a unit of fractured area increases disproportionately (section 2.5) so as to counterbalance the runway effects of the crack. It is desirable that the material avoids a ductile to brittle transition as long as possible.

Tough biological composites achieve a good compromise between 'pre-fracture' and 'fracture' toughness characteristics. Both features are equally crucial for the final outcome of the failure process. Materials of remarkable toughness have to enhance their performance with respect to both these two defined quantities.

Acknowledgements

This article was the theme of a seminar given by the author in the IRC for Biomaterials in London in November 1996. The *W_f .vs. strain rate* tests were performed by Mr K.Brear of the Department of Biology of the University of York, UK, the original idea resulted from a discussion with Dr A.J.Sedman of the CBDE in Salisbury, UK. I thank Dr R.F.Ker of the Dept of Biology, Univ. of Leeds for peering critically over this manuscript. Special thanks are due to Dr Herwig Peterlik and his colleagues in Vienna for letting me have yet unpublished data as personal communication.

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Figures

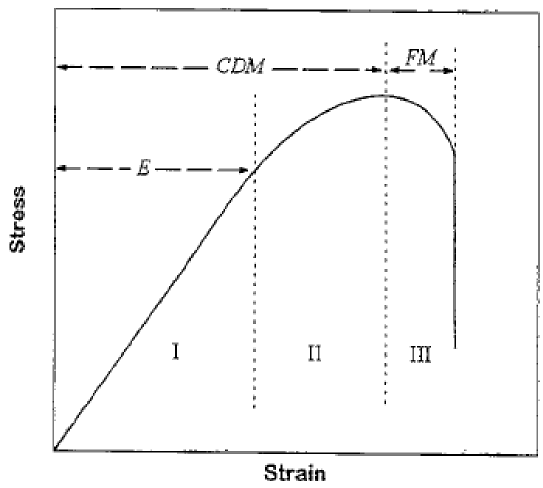

FIGURE 1: On its way to failure a 'quasi-brittle' solid (not just bone) exhibits a stress/strain behaviour, which can be split in three major domains: the elastic range (E), the continuum damage mechanics range (CDM), and the fracture mechanics (FM) one. In phase-II some of the input energy goes to increase the elastic strain energy of the body and some is dissipated in the generation of tiny cracks. In phase-III the product of $d\sigma^*d\varepsilon$ (increments of stress and strain) is negative and the ability of the material to increase its strength is rapidly counterbalanced by the reduction in the effective cross sectional area caused by the growth of a major crack. Mechanical tests for toughness are designed to take advantage of and enhance the effects of each phase. Depending on the kind and combination of applied stresses, the geometry of the specimen (which determines the stress field) and perhaps some specialised material properties (anisotropy, regional heterogeneity) the relative length of the three regions can vary widely and at will by the experienced experimenter who aims to isolate one or the other kind of behaviour.

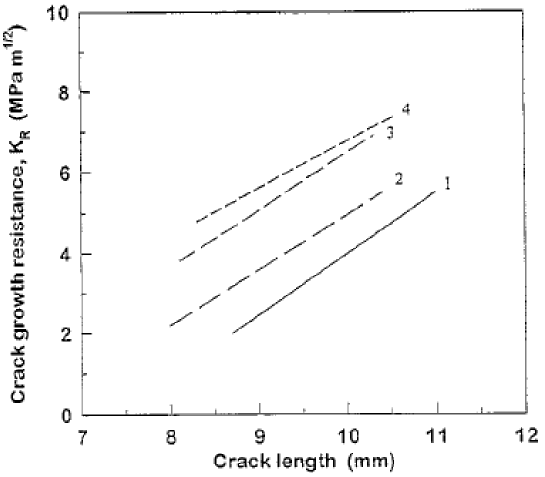

FIGURE 2: K_R, crack growth resistance curve, for antler bone. *K* increases linearly with the length of the crack. Four examples are shown. (Redrawn from [21])

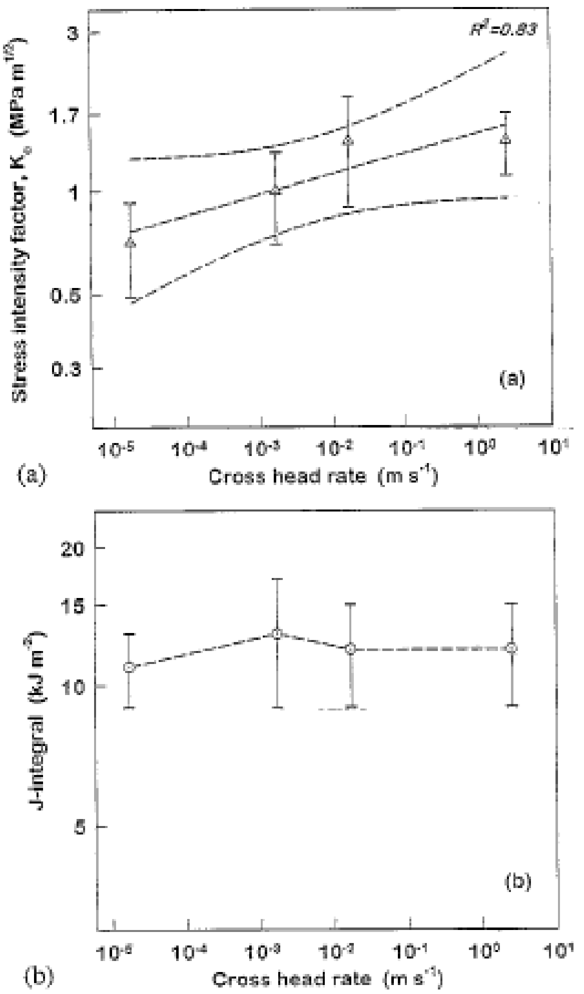

FIGURE 3: Crack initiation. K_c and J integral behaviour as a function of strain rate for equine hoof wall material. (a) Mean K_c values (±SD) and regression line (with 95% confidence interval). Viscoelastic effects at the tip of the crack are responsible for a significant increase in the stress intensity factor with strain rate. (b) Mean J integral values (±SD). (Redrawn from [22])

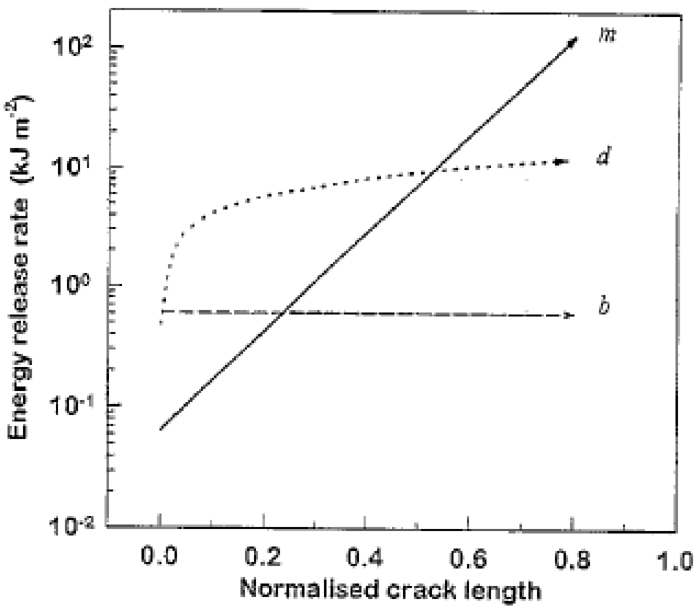

FIGURE 4: Crack propagation. Schematic showing the energy required to propagate a crack in the radial (*m*:microcracking damage), tangential (*d*:deflected crack) and circumferential (*b*:brittle fracture) directions in bovine bone. The fracture accompanied by microcracking damage (m) ahead of it is highly toughened [25].

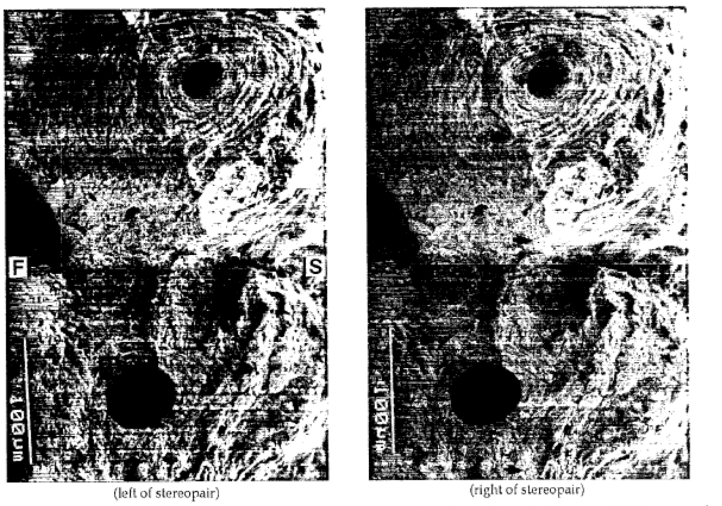

FIGURE 5: Slow-fast transition. SEM stereophotograph of the fracture surface of a human specimen fractured in tensile fatigue tests [13]. There was a slow (S) growth of the crack from the endosteal side, which at the ridge (arrows) jumped into a fast (F) uncontrollable/brittle propagation towards the periosteal surface of the bone. Compare the roughness of the two areas.

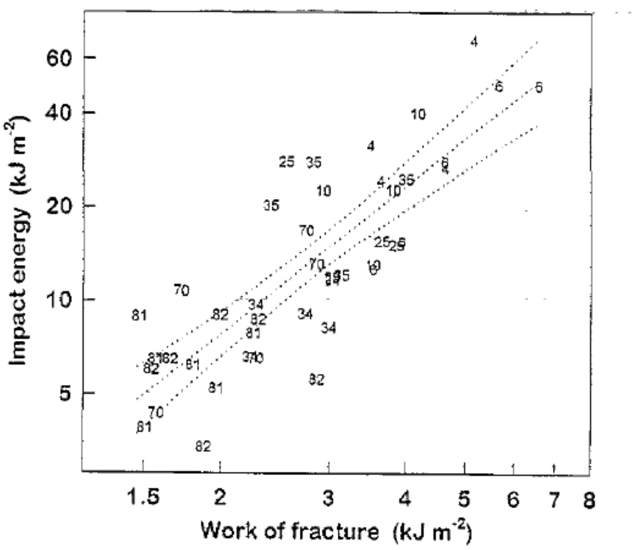

FIGURE 6: W_f versus *impact energy* absorption. Results shown are for human bones of various ages (age shown as symbol); regression line and 95% confidence interval, R^2=0.69. Both toughness measurements also showed very consistent reduction with age in log-log relationships (R^2=0.85, and 0.91 respectively). (Redrawn from [31])

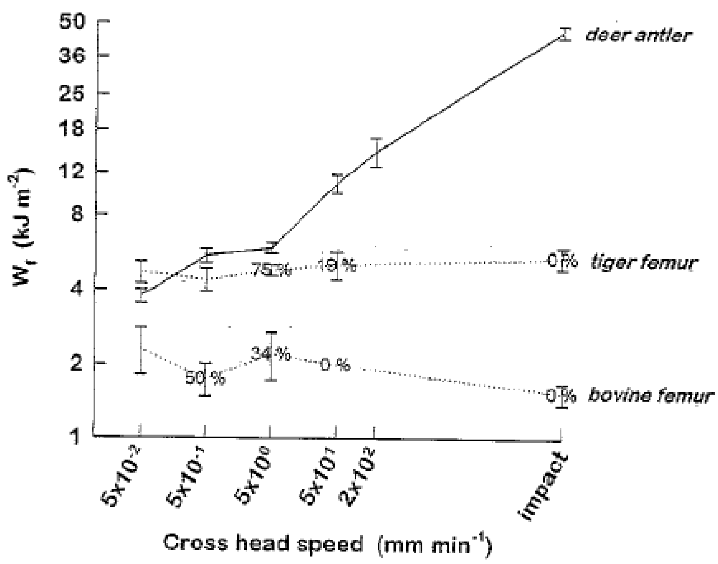

FIGURE 7: W_f versus *cross head speed* tests. The W_f (mean±SD) for ordinary bones (bovine and tiger femoral material) stays at constant values over a five orders of magnitude increase in the deformation rate. However, the 'tough' material of antler showed W_f to increase by nearly an order of magnitude over the same span of deformation rates. Antler also showed stabilised fractures at all rates and for all specimens. In bovine and tiger bone increasingly more specimens fractured in a 'brittle' mode at higher rates. The numbers show the percentage of the total number of specimens that achieved a stabilised fracture. (Redrawn from [32])

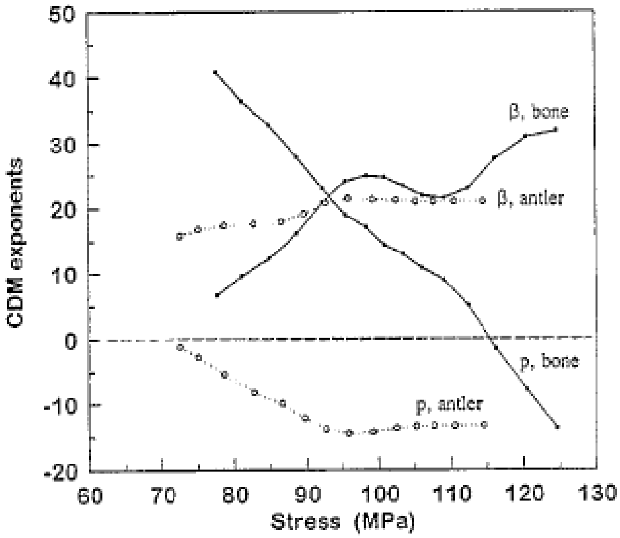

FIGURE 8: Microcrack toughening. The behaviour of exponents β and p of equation-1 for bone (continuous traces) and antler (dotted traces) as a function of the fatigue stress level. β the exponent of stress (σ) and also effective stress (σ/1-D) was on average the same for bone and antler. However, p the exponent of (1-D) had positive values in bone, but negative in antler. With the level of incipient damage increasing during fatigue the rate of damage accelerated in bone (p>0) but slowed down in antler (p<0). (Redrawn from [12])

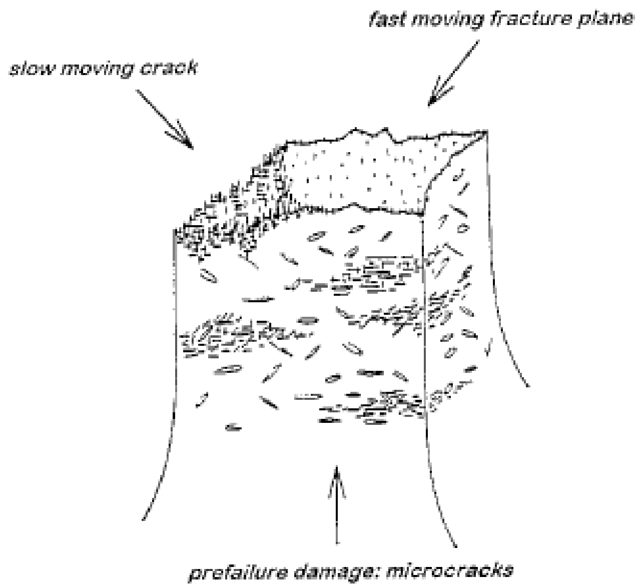

FIGURE 9: In the fracture of a solid-biomaterial specimen, and in accordance with fig.1, energy is dissipated in a number of ways: either as prefailure damage (microcracks), or to grow a slow moving crack with its collateral damage, or, to grow a fast moving fracture plane.