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We have studied toughening mechanisms in mineralised tissues. We have demonstrated the importance of microdamage as a universal toughening mechanism . This is clear from all the mineralised tissues we have examined. The tougher tissues have a variety of mechanisms to increase elongation before fracture, and these are rather different in antler and in dentine. (Antler has fibers that behave telescopically and also has a structure that forces microcracks to depart from the most dangerous orientation; dentine has a structure that initially induces yield at 45° to the loading direction, whatever that is, but at higher strains induces clouds of microcracks in the direction of the loading, and finally the macrocrack is diverted by the predominant fiber direction.) We have examined such toughening mechanisms by a combination of mechanical testing, (including static, impact and fatigue loading) optical examination, scanning confocal microscopy, acoustic emission, fractal analysis of fracture surfaces, and some finite element analysis.

The work on the grant has resulted in 5 papers published or in press in refereed journals, two chapters in books published or in press, and one large paper (on fatigue) which has just been submitted. We expect two more papers based on AFOSR work to appear.

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# TOUGHENING MECHANISMS IN BIOLOGICAL HARD TISSUES

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The period covered by this report covers work done on the grant from 15 June 1993 until termination.

***Fractals*** We have extended our analysis of fracture surfaces to surfaces of specimens broken in impact. We have examined two variables in a number of bones of very different toughnesses: the nature of the specimen (mineral content, histology etc.) and whether the impact specimen was notched or un-notched. In general, as might be expected, brittle bone shows a much greater decrease in impact energy when a notch is introduced than happens with the tougher bones. We find that, rather surprisingly, the fractal dimensions are very similar for all the specimens, whether tough or brittle, notched or un-notched. What this implies (somewhat indirectly) is that although toughness is increased with a general increase in the work it takes to create two new fracture surfaces, at high strain rates toughness is not simply a function of the energy required for fracture *travel*, but is probably a matter of the elastic and microdamage energy absorbed collaterally before the fracture actually initiates. This is different from what happens at low strain rates (see paragraph on strain rate effects).

***Microcrack morphology*** This paper in press in the *Journal of Materials Science* at the time of the last report (**'The extent of microcracking....'**)has now been published and is enclosed.

***Off-axis loading*** The paper in press in the *Journal of Biomechanics* at the time of the last report (**'Dependence of mechanical properties**...')has now been published and is enclosed.

***Acoustic emission and development of damage*** The paper being considered for publication at the time of the last report has now been published in the journal *Medical Engineering & Physics* and is enclosed (**'An examination of the micromechanics**...'). The main points it makes are: i) the tensile cyclic traces for both bone and antler have underlying similarities, which can be attributed to the occurrence of damage in the form of microcracks (less than 100 μm long); ii) visual information (by microscopically observing the microcracks) and the acoustic emission information point to the same thing, that it is the kind and distribution of microcracking that makes the difference between these two materials.

*Bone*, and *antler* (and a great number of mineralised tissues) are biological 'composites' consisting of a mineral reinforcing element (calcium phosphate) and an organic matrix (mainly collagen) capable of undergoing considerable extension (strain 10%). Although the mineral exists in the form of particles (tiny plates) mineralised tissues are not strictly particulate composites, but 'hybrids', because the matrix itself is fibrous. Bone and antler also show differences in both their constitution and their structure and architecture. The mineral, by its presence, induces stiffness in the matrix and cements the fibres together. The fibres also, of course, keep the mineral together, and by keeping the mineral discontinuous enhance the composite's toughness. The matrix itself, through its fibrous make-up, brings anisotropic mechanical properties to the structure.

*Microcracking.* Laminar bone has a more regular architecture and is more mineralised, and therefore more stiff, but it is also affected more by the occurrence and development of microcracks than antler bone. Antler has considerable local variation of mineralisation, which may help to divert its cracks and thus consume more energy. In addition, antlers' osteons consist of lamellae with fibrous elements at an angle to each other, so upon longitudinal stretching the lamellae unwind, detach and collapse inwards as in wood. This constitutes a telescopic mechanism by which higher strain and toughness can be achieved. In general antler has more specialised elements and increased heterogeneity throughout.

An idealised model for antler is as follows: soft matrix (1.5 GPa) in i) fibrous form, ii) the fibres themselves being woven in cylindrical form as to be able to act telescopically and iii) the fibre direction being used to induce directional properties (anisotropy) in the structure as a whole. This matrix is stiffened by impregnation with mineral crystals (110 GPa), which also help to cement the fibres together. There may exist an additional local variation in the concentration of the crystals The level of mineralisation seems to affect the way in which different tissues react so as to increase heterogeneity. In addition, the crystals are themselves elongated, and aligned crystals in the form of rods or platelets, even in a uniform continuous isotropic matrix, will produce anisotropic properties.

A third material whose toughening mechanism is of great interest is the *Narwhal tusk*. This has been examined by the same methods (cyclic loading, microscopic examination and acoustic emission) and the results are currently being analysed. Acoustically it appears to be a remarkably 'silent' material at low stresses and in the yield region and we believe that is due to microdamage according to *three* distinct modes. The narwhal tusk material is of a laminated design where successive laminae have fibres almost perfectly aligned in different directions. The mineral crystals in general follow the direction of the fibres, but they are aggregated locally in spheres, which touch each other. Characteristic of this design is that the mineral phase, through its globular pattern is isotropic but lies within a very anisotropic fibrous architecture. A consequence of this design is that: i) tension at any direction initially induces yield at a 45° angle through the interglobular spaces; afterwards the microcracks coalesce into clouds at a lesser angle to the tensile direction; ii) at a later stage the fracture proceeds along the fibre direction (split in-between the fibres); iii) at an even higher stress level the macrocrack is partially deflected by the variably oriented fibrous layers. As a result there is a yield stress of a low value which is almost independent of the angle of loading, although the structure itself is highly anisotropic in both its elastic and toughness properties.

***Fatigue behaviour of Bone and Antler***. In the acoustic emission and damage paper we suggested that the occurrence of damage is the main determinant of the shape of the stress/strain curve for all mineralised tissues and consequently of the remarkable strength and toughness characteristics of some of these tissues. Quantification of damage requires the performance of cyclic tensile tests. Pioneering work in this field by Dennis Carter and co-workers suggested that, provided that damage increases linearly with the cycle number, then the determination of a single exponent parameter allows the description of the behaviour of a range of materials in simple loading tests to failure, (the effect of the strain rate on strength, see Currey, J.D. (1989 *J.Biomech.* 22 469-4751989), or the behaviour in creep rupture tests (Mauch, M., Currey, J.D. and Sedman, A.J. 1992 *J. Biomech.* 25 11-16), or in fatigue tests (a number of papers by Carter and others). However, these two publications from this lab showed that this single parameter appears to take very similar values for two materials (bone and antler) which behave quite differently. Something is wrong. We think the answer is that the quantification of damage is a *function of* and/or *requires the determination of* a greater number of damage parameters so as to reflect the pronounced differences in the failure properties of these two tissues.

We have completed a series of fatigue tests and are currently analysing the results, which indeed suggest that i) damage accumulates *non-linearly* with the cycle number and ii) the form of the non-linearity is dependent on the level of the stress.

Preliminary treatment of the results show that the rate of development and accumulation of damage in antler slows down with every increase in the level of accumulated damage. On the other hand in bovine bone the rate of damage accumulation increases with the level of damage. What this means in practical terms is that the fracture process in antler becomes controllable since further damage is 'discouraged' from happening, something which indicates that the effect of the microcracks is beneficial rather than deleterious for the structure as a whole. In other words the larger, and more especially the more numerous the microcracks the less the stress concentrating effect they produce. The reason for this remarkable behaviour in antler is almost certainly micromechanical. At the level of the osteons microcracks open with an inclination to the principal tensile stress field and not normal to it. Consequently the material continues to soften without becoming weaker at the same time. The peculiar course of the rate of accumulation of damage had been predicted in part in the paper on the acoustic emission of bones (*'An examination of the micromechanics.....'*) where also the micromechanical disintegration of the microstructure was observed. While the softening of antler without the weakening of its structure was also observed in a series of tests described in the paragraph under: 'A Damage Paradox' in the chapter we have submitted in the forthcoming book on *Biomimetic* materials.

A publication including this piece of work on fatigue and our conclusions is currently in preparation.

***Remodelling: the efficient stimulus.*** The work of this AFOSR program is concerned mainly with the in-vitro stress/strain related behaviour of naturally occurring mineralised tissues. The damage mechanics aspects of these tissues are extremely interesting in that they show us ways by which the damage tolerance, the strength, the toughness and the stiffness can be altered in order to achieve materials tailor-made for the requirements of a specific job. The possible benefits and advantages brought about in the construction of biomimetic materials by such in-vitro studies are clear.

However, living tissues go a step further; they repair their damage constantly and also optimise both their structure and shape according to the continuously varying operational needs of their mechanical environment. Bone is such a 'smart' material. A question of considerable importance at the moment is whether bone remodelling is a response to *strains* or to *damage*. We have started to analyse this in bone by using finite element analysis to explore the strain patterns and yield patterns that one would expect from a tissue of bone's particular symmetry and anisotropy (which we have had to determine), and comparing these with the pattern of microdamage that we have observed in the specimens themselves. We find, disappointingly from the analytical point of view, that observed patterns of remodelling in bone could be in response, either to damage or to strain, though some types of strain can be excluded as efficient signals.

A paper including our work in this field (**'Experimentally determined microcracking....'**) is in press in the *Philosophical Transactions of the Royal Society-B*. A copy of the ms is included with this report.

***Strain rate effects*** It is our general finding that the main characteristic allowing mineralised tissues to be tough is their ability to develop damage *in a controlled way*. There two aspects of this, of course; i) the ability to develop diffuse damage at all, and ii) the ability to prevent this damage from spreading. The pattern of development of microdamage depends on the mineral content. In general the toughest mineralised tissues have lower mineral contents. This is not the sole reason though, the microstructure is also important. This is obvious from comparison of antler and Narwhal tusk, which have similar mineral contents by very different microstructures. This somehow influences their toughness behaviour at different strain rates.

At very low strain rates narwhal tusk is tougher than antler. As the strain rate increases antler becomes tougher and tougher, while narwhal remains about the same.

Finally in impact, antler cannot be broken across the grain whereas narwhal, though having a high impact strength, can. Other, more highly mineralised tissues have a toughness lower than either tusk and antler at all loading rates and of a rather constant value. This difference of behaviour has clear implications concerning the micromechanics of toughening in the two tissues. A paper on the effect of strain rate on toughness is being prepared from these experiments.

Finally, as *jeu d'esprit* I include a paper **'The validation of algorithms...'** borne of a sense of frustration at the last world congress of biomechanics in Amsterdam. It makes, I think, an important point.

***Conclusions*** The work reported here, along with what has been written in our earlier reports, concludes what has been done during the term of the AFOSR contract. However, there are a number of unanswered questions we are actively exploring, more data to be analysed and more papers to be written. We are doing this using odds and ends of funding.

What we have been able to show, which is new or at least has not been shown at all clearly before, is the quantitative importance of microdamage as a toughening mechanism in these tissues. This is clear for all the mineralized tissues we have looked at. We have also shown that the *tougher* mineralised tissues have several mechanisms to increase the elongation occurring before fracture, and these are rather different in antler and in narwhal tusk. Unfortunately, because of difficulties with the availability of a confocal microscope, which has now being sorted out, we as yet have been unable to tie down completely the toughening mechanism in narwhal. However, we are actively working on this now.

Dr P Zioupos and I are extremely grateful to AFOSR for producing the funding to make this work possible. It has been extremely interesting for us, and we think we have advanced the subject. We hope that some of the work, in particular the different types of toughening shown by antler and narwhal tusk may, despite occurring in very biological materials, give food for thought for people trying to think of new ways of toughening engineering materials.

***Papers published or in press since the last report***

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**Currey, J.D., Brear, K. and Zioupos, P.** 1994 Dependence of mechanical properties on fibre angle in narwhal tusk, a highly oriented biological composite. *Journal of biomechanics* **27** 885-897

**Zioupos, P. and Currey, J.D.** 1994 The extent of microcracking and the morphology of microcracks in damaged bone. *Journal of materials science* **29** 978-986

**Zioupos, P., Currey, J.D. and Sedman, A.J.** 1994 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** 203-212

**Zioupos, P., Currey, J.D., Mirza, M.S. and Barton, D.C.** 1995 Experimentally determined microcracking around a circular hole: comparison with predicted stresses. *Philosophical Transactions of the Royal Society of London-A.* (In Press)