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EXPERIMENTAL MICROMECHANICS AND VISCOELASTICITY OF BIOLOGICAL AND BIO-PROTECTIVE MATERIALS

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Abstract

The properties of composite materials including those of biological origin depends very much upon *structure*. We consider here *viscoelastic* properties in which the stiffness depends on time or frequency, and *microelastic* properties in which there is dependence of stress upon spatial gradients of strain. The complex structural hierarchy of composite materials, particularly biological ones, gives rise to several viscoelastic processes, however the microelastic response is principally governed by the largest size structural elements.

1 Viscoelastic properties

1.1 INTRODUCTION

Viscoelasticity includes phenomena such as creep, relaxation and dynamic response. The loss angle δ is the phase angle between stress and strain during oscillatory (dynamic) loading, as illustrated in Fig. 1. Dynamic viscoelasticity is referred to as internal friction, and recoverable viscoelasticity as anelasticity.

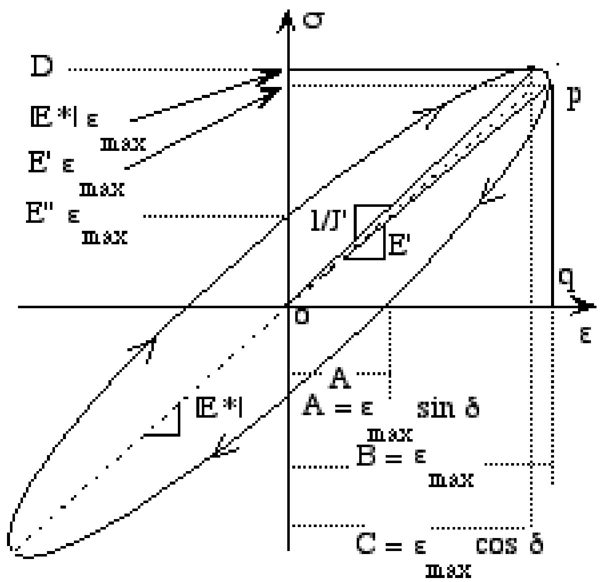

Fig. 1 Stress σ vs. strain ε for a linearly viscoelastic material under oscillatory loading, after Lakes (1998). Illustration of slopes and intercepts. A material with a rather large value of tan $\delta \approx 0.4$ is shown for illustration. The material could be a viscoelastic rubber.

Viscoelastic phenomena bring to mind polymers since such effects are very pronounced in many polymeric materials. Specifically large viscoelastic effects (damping as quantified by the loss tangent, tan δ, from 0.1 to 1 or more) are common in polymers at ambient temperature. By contrast in structural metals such as steel, brass, and aluminum, viscoelastic effects are usually small: tan δ is 10^{-3} or less; some aluminum alloys may exhibit very small loss, e.g. tan $\delta = 3.6 \times 10^{-6}$. A comparison of viscoelastic properties of some materials is presented in Fig. 2. In hierarchical solids which contain structure at multiple length scales, viscoelasticity can arise from multiple processes at the different scales. Viscoelasticity in biological materials is of particular interest since it is causally linked to a variety of microphysical processes and can be used as an experimental probe of those processes. Viscoelasticity in biomaterials and bio-protective materials is of interest because it can beneficially or adversely affect the performance of these materials.

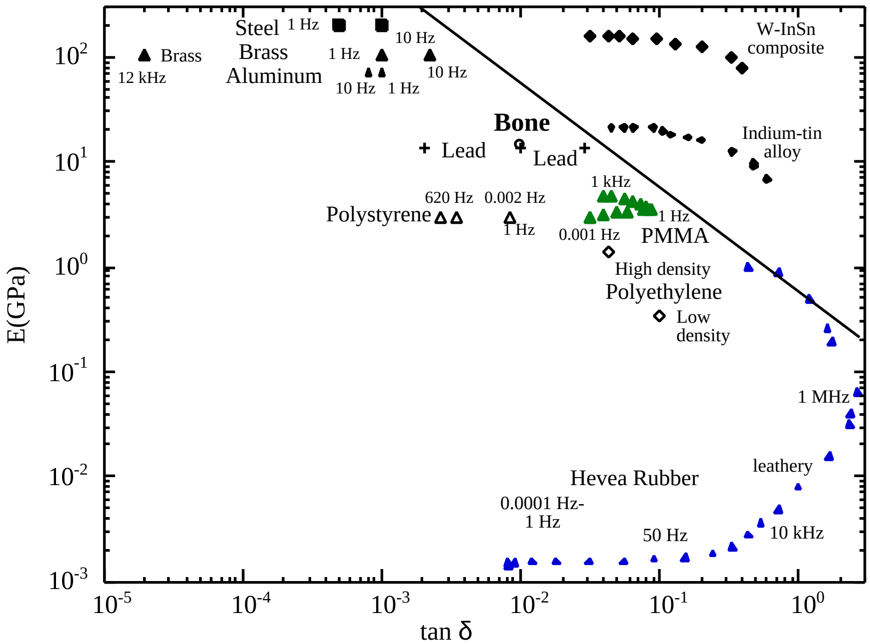

Fig. 2 Stiffness-loss map for some materials. Temperature is near room temperature. Adapted from Lakes (1998). Data are replotted from several sources, including Brodt, et al. (1995) for indium-tin alloy.

1.2 EXPERIMENT

We experimentally examine viscoelastic behavior in materials with microstructure including biological materials and composites which could protect the body from vibration. Viscoelastic behavior depends on material microstructure. We have characterized several materials isothermally over 11 decades of time and frequency with a novel instrument. The rationale is as follows. For some materials, particularly some amorphous polymers, it is possible to infer material properties over a wider range from test results taken at different temperatures. Materials for which such a procedure is possible are called thermorheologically simple. Many examples covering 12 or more decades are known. Many materials, particularly composites, biological materials, and materials in which multiple viscoelastic mechanisms are active, are not thermorheologically simple. Direct measurement of properties over many decades is required for a full characterization of the material.

Viscoelastic measurements were performed in torsion at ambient temperature using apparatus developed by Brodt, *et al*. (1995). This device permits measurements over an unusually wide range of time and frequency, under isothermal conditions. Such capability is particularly useful in composites and other materials which are not thermorheologically simple. The wide frequency range is obtained by eliminating resonances from the devices used for loading and for displacement measurement, by minimizing the inertia attached to the specimen, and by use of a geometry giving rise to a simple specimen resonance structure amenable to simple analysis. Higher frequencies (1 kHz to 100 kHz) became accessible following design modifications permitting study of higher harmonic modes. Torque (sinusoidal for dynamic studies and step function for creep studies) was produced electromagnetically by a Helmholtz coil acting upon a high intensity neodymium iron boron magnet at the specimen free end. Angular displacement was measured via laser light reflected from a small mirror upon the magnet to a split-diode light detector. At resonant frequencies, tan δ was inferred from the width of the dynamic compliance curve or from free decay of vibration, and in the subresonant domain, from the phase angle between torque and angle.

1.3 PROTECTIVE MATERIALS FOR REDUCTION OF IMPACT AND VIBRATION

Indium tin alloy exhibits favorable combinations of stiffness and damping over a wide range of frequency. It is used as a solder and may find application in vibration damping to protect the human body. Tan δ followed a ν^{-n} dependence over many decades of frequency ν for these alloys. Results are attributed to a dislocation-point defect mechanism. The combination of damping and stiffness is higher than that of common materials, as shown in Fig. 2. Composite materials specifically designed to maximize the product of damping and stiffness exhibit even higher values as shown in Fig. 2. Composite materials with such properties have a high figure of merit for damping vibration. In comparison, compact bone falls among the common materials. It appears that the 'design' of bone does not incorporate optimization of damping.

Foams, considered as composite materials with a void phase, are currently used in protecting the human body. Novel foam materials with a negative Poisson's ratio are under study as seat cushions, wheelchair cushions, and elbow pads. Negative Poisson's ratio foams (Lakes, 1987) exhibit higher damping than foams of conventional structure, when both foams are pre-compressed as they would be in an automotive cushion. It is desirable in this setting to achieve an appropriate compliance.

Viscoelastic elastomer materials are of use as shoe insoles in protecting the body from impacts during running. Ordinary rubber exhibits high damping at ultrasonic frequencies, as shown in Fig. 2. High damping viscoelastic rubber compositions exhibit significant damping (Shipkowitz, et al. 1988) at frequencies (1 to 100 Hz) associated with activities such as running.

1.4 BIOLOGICAL MATERIALS: STRUCTURE AND VISCOELASTICITY

In composite materials of biological origin, such as bone, the presence of proteinaceous or polysaccharide phases can give rise to significant viscoelasticity. The mineral phase of bone is crystalline hydroxyapatite which is virtually elastic; it provides the stiffness of bone. Human compact bone is a natural composite which exhibits a rich hierarchical structure. On the microstructural level are the osteons, which are large (200 micron diameter) hollow fibers composed of concentric lamellae and of pores. The lamellae are built of fibers, and the fibers contain fibrils. At the ultrastructural level (nanoscale) the fibers are a composite of the mineral hydroxyapatite and the protein collagen. Specific structural features have been associated with properties such as stiffness via the mineral crystallites, creep via the cement lines between osteons, and toughness via osteon pull-out at the cement lines. Lacunae are ellipsoidal pores with dimensions on the order 10 micron which provide spaces for the osteocytes (bone cells) which maintain the bone and allow it to adapt to changing conditions of stress.

Observe in Fig. 3 that the loss tangent in shear of wet compact bone attains a broad *minimum* over the frequency range associated with most bodily activities. Observe in Fig. 2 that the viscoelasticity of bone at a frequency of 1 Hz lies within the range of the stiffness-loss map associated with 'normal' materials. There is no evidence that compact bone has any level of extremal damping. The loss tangent of dry compact bone is less than that of wet bone at high and at low frequencies, as shown in Fig. 3. In contrast to the above results, at physiological strains or below, creep under sufficiently large load, giving rise to an initial strain in the range 0.003 to 0.007, terminates in fracture (Mauch, Currey, and Sedman, 1992).

Bamboo has an interesting microstructure and macrostructure with hierarchical features which contribute to its structural integrity. Specifically, bamboo contains fiber-like structural features known as bundle sheaths as well as oriented porosity along the stem axis. Bamboo, moreover, has functional gradient properties in which there is a distribution of Young's modulus across the culm (stem) cross section. Dynamic viscoelastic properties of bamboo were determined in torsion and bending. Damping, measured by tan δ, in dry bamboo was relatively small, about 0.01 in bending and 0.02 to 0.03 in torsion, with little dependence on frequency in the audio range. In wet bamboo, damping was somewhat greater: 0.012 to 0.015 in bending and 0.03 to 0.04 in torsion. The anisotropy in damping implies a purely cellular model is insufficient; there is large scale molecular orientation or at least two distinct solid phases.

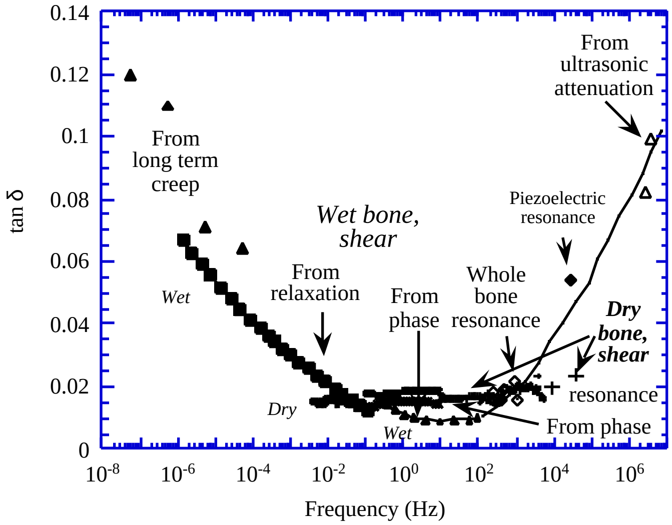

Fig. 3 Tan δ for human compact bone, adapted from data of Lakes et. al. (1979) for wet human bone at 37°C and data of others in vibration and wave modalities as assembled by Lakes (1982). Results for dry bone at 22°C adapted from Garner, et al. (1998). Damping at low frequency inferred from long term creep by Park and Lakes (1986).

1.5 BIOLOGICAL MATERIALS: CAUSAL MECHANISMS

Viscoelasticity in bone arises from a variety of mechanisms. High damping at low frequencies and creep at long times is associated with viscous-like deformation at interfaces such as the cement lines (Lakes and Saha, 1979). Thermoelastic coupling may account for some of the damping between 0.01 Hz and 10 Hz (Lakes and Katz, 1979).

Stress-induced fluid flow in channels within bone is particularly interesting as an energy transfer process because it is an hypothesized mechanism for explaining the stimulus delivered to bone cells to trigger bone adaptation. Since compact bone has a hierarchical microstructure, such fluid flow occurs in bone on a spectrum of length scales during mechanical excitation. Such fluid flow can have biological significance via transport of nutrients to cells, via direct pressure effects or indirectly via streaming potentials. Stress-induced fluid flow can be explored via the viscoelastic effects which occur as a result. For example, finite-element analysis of fluid flow in the Haversian systems of bone suggests a peak tan δ of less than 0.0005 for bending of bone oriented in the longitudinal direction (Stewart, et al., 1988) as a result of fluid flow. Larger damping is expected in the bending of bone oriented in the transverse direction: maximum tan $\delta \approx 0.025$. Flow in the Haversian systems would not cause any damping in torsion under the assumption of parallel fluid filled tubes in an isotropic solid. Bone, however, is heterogeneous on the scale of osteons, and it is possible that damping peaks may occur in torsion due to fluid flow. Experiments are in progress.

The loss tangent of a specimen of dry human compact bone (Garner, et al., 1998) is less than that of several kinds of wet human bone at high and at low frequencies, as shown in Fig. 3. The difference is not necessarily due to the absence of fluid flow in dry bone; drying also can alter the mobility of groups in the collagen macromolecules in bone.

2 Microelastic properties

2.1 INTRODUCTION

As for and *microelastic* properties there is dependence of stress upon spatial gradients of strain. This can arise due to additional freedom which is possible in solids. For example, the idea of a couple stress (moment per unit area) can be traced to Voigt in the late 1800's during the formative period of the theory of elasticity, and it was developed further by the Cosserats in 1909. Many theoretical studies were conducted, beginning in the 1960's. In Cosserat elasticity, one of the simplest generalized continuum theories, there are characteristic lengths as additional engineering elastic constants. There are a total of six independent elastic constants in an isotropic Cosserat solid. Recent experimental work discloses a variety of cellular and fibrous materials, including bone, to exhibit such freedom, and the characteristic lengths have been measured. As for structural hierarchy, hierarchical solids are solids in which structural elements themselves have structure. Generalized continuum effects are primarily the result of the largest size structural elements in the material. Cosserat elasticity and the related microstructure elasticity theory have a natural characteristic length scale associated with the theory, in contrast with classical elasticity in which there is no such length scale. Generalized continuum theories are therefore of interest in connection with structured materials such as foams and natural and synthetic composites, in which the microstructure size is not negligibly small.

The constitutive equations for a linear isotropic Cosserat elastic solid (Mindlin, 1964) also known as a micropolar solid (Eringen, 1968) are:

$$\sigma_{kl} = \lambda \varepsilon_{rr} \delta_{kl} + (2\mu + \kappa)\varepsilon_{kl} + \kappa \varepsilon_{klm}(r_m - \varphi_m) \qquad (1)$$

$$m_{kl} = \alpha\, \varphi_{r,r} \delta_{kl} + \beta \varphi_{k,l} + \gamma\, \varphi_{l,k} \qquad (2)$$

The usual summation convention for repeated indices is used throughout and the comma denotes differentiation with respect to spatial coordinates. σ_{kl} is the force stress, which is a symmetric tensor in classical elasticity but it is asymmetric here. m_{kl} is the couple stress or moment per unit area, ε_{kl} is the small strain, u_k is the displacement, and e_{klm} is the permutation symbol. The microrotation φ_k in Cosserat elasticity is kinematically distinct from the macrorotation r_k which depends on the displacements. In three dimensions, the isotropic Cosserat elastic solid requires six elastic constants λ, μ, α, β, γ, and κ for its description. The following technical constants derived from them are beneficial in terms of physical insight. These were discussed by Eringen (1968) and Gauthier and Jahsman (1975): Young's modulus $E = (2\mu+\kappa)(3\lambda+2\mu+\kappa)/(2\lambda+2\mu+\kappa)$, shear modulus $G = (2\mu+\kappa)/2$, Poisson's ratio $\nu = \lambda/(2\lambda+2\mu+\kappa)$, characteristic length for torsion $l_t = [(\beta+\gamma)/(2\mu+\kappa)]^{1/2}$, characteristic length for bending $l_b = [\gamma/2(2\mu+\kappa)]^{1/2}$, coupling number $N = [\kappa/2(\mu+\kappa)]^{1/2}$ (dimensionless) and polar ratio $\Psi = (\beta+\gamma)/(\alpha+\beta+\gamma)$ (dimensionless).

2.2 BASIS FOR EXPERIMENTS

Salient consequences of Cosserat-type theories are as follows. These consequences may be used as a basis for interpreting experiments, as well as for predictive purposes.
(i) A size-effect is predicted in the torsion of circular cylinders of Cosserat elastic materials. The effective shear modulus associated with such cylinders increases as their size decreases. A similar size effect is also predicted in the bending of plates and of beams. No size effect is predicted in tension.
(ii) Calculation of stress concentration factors around a circular hole, taking into account couple-stresses, results in lower values than accepted heretofore. Stress concentration around a rigid inclusion in an elastic medium is greater in a Cosserat solid than in a classical solid. Stress concentration near cracks and elliptic holes is reduced in comparison to classical predictions .
(iii) Dilatational waves propagate non-dispersively, i.e. with velocity independent of frequency, in an isotropic Cosserat elastic medium. Shear waves propagate dispersively in the presence of couple-stress. A new kind of wave associated with the micro- rotation is predicted to occur in Cosserat solids. Dispersion of dilatational waves can be accounted for in the more general Cosserat-type theories known as microstructure elasticity or micromorphic elasticity.
(iv) Strain and stress distributions are modified in a Cosserat solid. For example, in the torsion of a square cross section bar, peak strain and stress are reduced but strain spills over into the corners of the cross section in which classically the strain is zero (Park and Lakes, 1986, 1987).

2.3 BEHAVIOR OF BONE

Experiments on bone may be interpreted within the continuum view in which the forces in the micro-structure are averaged, and concepts of stress and strain are used. In that vein, size effects in the rigidity of bone samples were interpreted via a generalized continuum theory, Cosserat elasticity, which allows a moment per unit area as well as the usual force per unit area. Slender specimens had a higher apparent stiffness than thick ones. The stiffening effect was noticeable even for specimens as thick as 5 to 6 mm. As for the magnitude of the ratio in stiffness of osteons to that of whole bone, a factor of 3.5 stiffening effect was observed by Lakes and Yang in microsamples as small as about 0.5 mm in diameter. High osteon stiffnesses were recently reported by Ascenzi et. al. (1994). The shear moduli, 23 GPa for osteons with longitudinal fibers, and 17 GPa for osteons with lamellae of alternate orientation, were more than four times higher than moduli reported in the literature for macroscopic specimens of bone tissue. These results are in harmony with the torsional size effects observed by others, as shown in Fig. 4. They may be understood in terms of Cosserat elasticity, in torsion, assuming technical elastic constants to be as given in Fig. 4, which also shows for comparison the classical behavior of a homogeneous material, solid PMMA, and the Cosserat elastic behavior of a polymer foam.

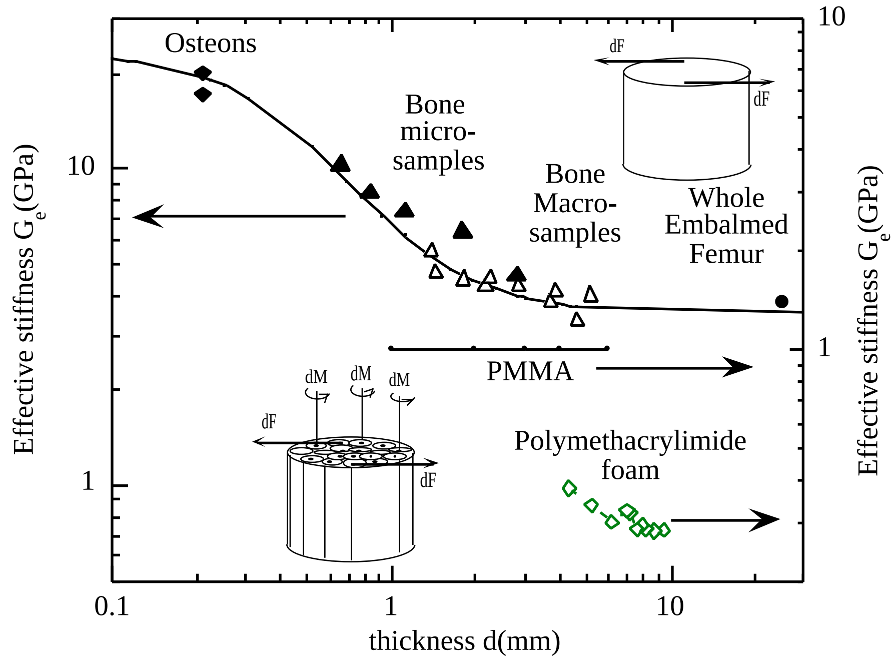

Fig. 4 Effective torsional stiffness of bone and polymethacrylimide foam vs. diameter, adapted from Lakes (1995) and Anderson et al. (1994).

♦ Osteons, fresh, wet, after Ascenzi, et. al., (1994).

Δ Bone macro-samples, fresh, round, wet, after Yang and Lakes (1981).

Δ Bone micro-samples, fresh, round, wet, after Lakes and Yang (1983).

• Whole embalmed femur, after Huiskes, et. al., (1981).

___ Solid curve, Cosserat elasticity, torsion, assuming technical elastic constants to be:
G = 3.5 GPa, l_t = 0.22 mm, l_b = 0.44 mm, Ψ = 1.5, N = 0.62.

◊ Polymethacrylimide foam, Rohacell WF300, square cross section.
G = 0.28 GPa, l_t = 0.8 mm, N = 0.2: technical Cosserat elastic constants after Anderson and Lakes (1994)

• Solid PMMA (polymethyl methacrylate) behaves classically.

Cosserat elastic constants derived from size effects in human bone have been used to predict surface strain distributions around holes in a strip under tension and on prismatic bars under torsion. For the holes, reasonable qualitative agreement was found, but it was not perfect owing to the neglect of the anisotropy of bone. Good quantitative agreement was obtained for strain distributions in square cross section bars of bone in torsion since the same elastic constants, specifically the torsion characteristic length and coupling number N are relevant in this geometry as in the torsion size effect study (Park and Lakes, 1986).

2.4 BEHAVIOR OF SYNTHETIC CELLULAR SOLIDS

In selected isotropic cellular solids *all six* of the Cosserat elastic constants have been measured by Lakes (1986) and Anderson et al. (1994). In particular, a dense polyurethane foam and closed cell polymethacrylimide foams of different densities exhibited Cosserat elasticity as manifested by size effects in torsion and bending. Selected results for foam and PMMA are compared with bone in Fig. 4. PMMA is purely classical in its behavior. Polymethacrylimide foam exhibits Cosserat elastic effects but the magnitude of the size effects is less than in bone. Specifically, the change of apparent stiffness with diameter is less in foams than in bone since the coupling number N is less in foam than in compact bone.

Several of these constants have been verified by further experiments in geometries different from those used in the original measurements. Holographic studies show that strain can spill over into regions which are classically forbidden, specifically the corners of a square cross-section prism in torsion. Moreover the warp in such a bar was shown by holography to differ from the classical prediction (Anderson, et. al, 1995). The effect of stress redistribution is to reduce the stress concentration factor around holes in bone and related materials.

2.5 SIGNIFICANCE OF MICROELASTIC BEHAVIOR

Behavior describable by generalized continuum representations such as Cosserat elasticity is important in that stress concentration factors are ameliorated in comparison with classical materials. Bone is less vulnerable to the stress concentrating effects of drilled holes than is expected on the basis of classical elasticity (Brooks, et. al., 1970). The ability of bone to redistribute stress around such defects (prior to any remodeling) is associated with the alternate stress pathway of Cosserat elasticity (Park and Lakes, 1986) for which the physical mechanism is the array of local moments transmitted through the stiff osteons.

3. Summary

Material microstructure can give rise to both viscoelastic (dependent on time, rate, or frequency) effects and microelastic (dependent on spatial gradients of strain) effects. Hierarchical structure in biological materials such as bone gives rise to a multiplicity of viscoelastic mechanisms. Microelastic effects, however, are governed by the largest size structural elements in the material. Even so, the degree of heterogeneity of the Haversian structure of compact bone confers substantial microelastic effects including redistribution of strain away from stress concentrators. These effects are more pronounced than they are in synthetic foams.

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